U.S. ARMY CORPS OF ENGINEERS
HUNTSVILLE DIVISION

TRIAL BURN PLAN

DEACTIVATION FURNACE

SENECA ARMY DEPOT
ROMULUS, NEW YORK

VOLUME 2 OF 2

PREPARED FOR

U.S. ARMY CORPS OF ENGINEERS
HUNTSVILLE, ALABAMA

PREPARED BY

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1893

APPENDIX D

CALCULATIONS

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CALCULATIONS

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Appendix D-1

Calculation of Maximum Hourly Impacts Associated with State Ambient Air Guidance

These calculations follow the methodology described in **Section 4.4**, calculation of allowable emission rates based on NYSDEC Guidance. Equations 4.4.1 and 4.4.2 are used to calculate emission rates which are associated with state ambient air quality guidance for 1 hour (SGC) and annual (AGC) impacts. Where necessary, allowable impacts are converted from one time basis to another by the factors provided to SEAD by NYSDEC **(see Table 4-3)**. The results of these calculations are emission rates which comply with both the hourly and annually averaged allowable impacts. This data is summarized in **Table 4-4** and is the basis by which allowable pollutant feed rates are calculated. **Section 4.6** describes the methodology used to back calculate allowable pollutant feed rates. The resulting pollutant feed rates are also summarized in **Table 4.4.**

Basis: The hourly average impact for 1 lb/hr emission of a constituent was found to be 18.55 mg/m^3 for the deactivation furnace at SEAD. This was modelled as shown in Appendix J.

SGC	=	Max Ave Hourly Impact	(ug/m^3)
AGC	=	Max Ave Annual Impact	(ug/m^3)

Step 1: If impacts are not on correct time basis convert by:

To convert From	To	Multiply by
1 hr impact	24 hour impact	0.4
1 hr impact	3 month impact	0.2
1 hr impact	1 year impact	0.1

Step 2: Calculate Maximum Hourly Emission Rate by EQ 4.4.1

$$EQ\ 4.4.1 \qquad Qs = SGC\ \frac{1\ lb/hr}{18.55\ ug/m^3}$$

Step 3: Calculate Maximum Annual Emission on an Hourly Basis by Eq 4.4.2

$$EQ\ 4.4.2 \qquad Qa = Qs\ x\ \frac{AGC}{0.10\ x\ SGC\ x\ (2080/8760)}$$

Simplified EQ 4.4.2 becomes

$$Qa = Qs\ x\ \frac{42.12\ AGC}{SGC}$$

Step 4: In order to satisfy the SGC and AGC, use the lower of Qa and Qs as the allowable emission rate.

Calculate allowable hourly emissions using equations 4.4.1 and 4.4.2 for all metals known to be present in munition PEP and which may be emitted from the deactivation furnace. Example calculations for each material of importance follow.

Antimony

SGC = 120 ug/m^3
AGC = 1.2 ug/m^3

Step 1: No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 120 \times 1/18.55 = 6.47 \ lb/hr$$

Step 3:

$$Qa = \frac{6.47 \times 1.2}{0.1 \times 120 \times (2080/8760)} = 2.72 \ lb/hr$$

Step 4: Qa < Qs; therefore the maximum hourly emission of Antimony = 2.72 lb/hr.

Particulate

NAAQS = 150 ug/m^3 (24-hr impact)
NAAQS = AGC = 50 ug/m^3 (annual impact)

Assumptions: (1) 50% of the allowable impact is already taken up by background particulate matter
(2) All particulate matter is assumed to be PM-10

Step 1: Calculate SGC and AGC values for particulate matter (PM-10) accounting for background particulate.

SGC = (150-0.5 (150)) ug/m^3 /0.4 = 187.5 ug/m^3

AGC = (50-0.5 (50)) ug/m^3 = 25 ug/m^3

Step 2:

$$Qs = 187.5 \times 1/18.55 = 10.11 \ lb/hr$$

Step 3:

$$Qa = 42.12 \times \frac{25}{187.5} \times 10.11 = 56.78 \ lb/hr$$

Step 4: Qs < Qa; therefore the maximum hourly emission of particulate = 10.11 lb/hr.

Lead

Allowable ambient lead concentrations are governed by a NAAQS which is a 3-month average. Because it is a standard and is a 3 month average, we did not derive an allowable 1-hr and annually averaged impact. Therefore, the calculation of allowable feed emission follows a slightly different format. If SEAD were to derive 1-hr and annual numbers, calculated allowable munition rates would have been higher. SEAD has based their calculations on conservative estimates.

NAAQS = 1.5 ug/m^3 (hourly impact averaged over 3 months)

Step 1: For the generic 1 lb/hr emission convert the hourly impact to a 3-month impact.

$$\frac{18.55\ ug/m^3\ (1\text{-}hr\ impact)}{1\ lb/hr\ emission} \times \frac{0.2\ (3\text{-}month\ impact)}{(1\text{-}hr\ impact)} = \frac{3.71\ ug/m^3(3\text{-}month\ impact)}{1\ lb/hr}$$

Step 2: Set up a proportion of the generic 1 lb/hr emission over the associated 3-month impact and compare it to the allowable lead emission rate over the allowable lead impact (3-month NAAQS).

$$\frac{1\ lb/hr\ emission}{generic\ 3\text{-}month\ impact} = \frac{X\ lb/hr\ allowable\ emission\ rate}{3\text{-}month\ NAAQS}$$

$$\frac{1\ lb/hr}{3.71\ ug/m^3} = \frac{X\ lb/hr}{1.5\ ug/m^3} = 0.404\ lb/hr\ lead$$

However, the furnace will not operate 24 hours a day. The furnace will only be permitted to operate 2080 hours/yr (8 hr/day x 5 day/week x 52 week/yr). Thus, over a quarterly period the furnace will operate 2080/4 = 520 hrs out of a possible 8760/4 = 2190 hrs.

Since the NAAQS reflects continuous (24 hr/day) operation, the furnace can operate at a higher emission rate (equal to the ratio of total hours to hours of operation) and meet the NAAQS, as long as it operates no more than 520 hours per quarter and less than 2080 hours per year.

Step 3: Taking into account the reduced furnace operation, calculate the maximum allowable lead emission rate in lbs/hour.

Max allowable hourly emission of lead =

$$0.404\ lb/hr\ x\ \frac{2190\ hours/quarter}{520\ operational\ hours/quarter} = 1.70\ lb/hr$$

Nickel

SGC = 1.5 ug/m^3
AGC = 0.02 ug/m^3

Step 1: No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 1.5 \times 1/18.55 = 0.081 \ lb/hr$$

Step 3:

$$Qa = 0.02 \times \frac{42.12 \ (0.02)}{0.08} = 0.21 \ lb/hr$$

Step 4: Qs < Qa, therefore, the maximum hourly emissions of Nickel = 0.081 lb/hr.

Chrome (VI) Compounds

SGC = 0.10 ug/m^3
AGC = 2 x 10^{-6} = ug/m_3

Step 1: No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 0.10 \times 1/18.55 = 0.0054 \ lb/hr$$

Step 3:

$$Qa = 0.0054 \times \frac{42.12 \ (2 \times 10^{-5})}{0.10} = 4.5 \times 10^{-5} \ lb/hr$$

Step 4: Qa < Qs; therefore, the maximum hourly emission of Chrome VI = 4.5 x 10^{-5} lb/hr.

HCL

SGC = 150 ug/m^3
AGC = 7 ug/m^3

Step 1: No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 150 \times 1/18.55 = 8.08 \ lb/hr$$

Step 3:

$$Qa = 8.08 \times \frac{42.12\ (7)}{150} = 15.89 \ lb/hr$$

Step 4: Qs < Qa; therefore, the maximum hourly emission of HCl = 8.08 lb/hr.

However, 40 CFR 264.343.A,(2)(b) restricts HCL Emissions to 4.0 lb/hr. In addition SEAD will limit the munition feedrate so as to restrict HCl Emissions to less than 3.0 lb/hr.

Barium

SGC = 120 ug/m^3
AGC = 0.5 ug/m^3

Step 1: No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 120 \times 1/18.55 = 6.47 \ lb/hr$$

Step 3:

$$Qa = 6.47 \times \frac{42.12 \ (0.5)}{120} = 1.14 \ lb/hr$$

Step 4: Qa < Qs; therefore, the maximum hourly emission of Barium = 1.14 lb/hr.

There are no SGCs or AGCs recorded in NYSDEC's Air Guide-1 for Aluminum and Tin. Therefore, in order to permit the furnace interim valves must be developed using the methodology described in AG-1 (IV A.2.a.1. and IV.A.2.b.1.). According to these sections, interim values can be assigned only if the contaminant has a recognized occupational exposure limit; and if it does not fit the definition of high toxicity contaminant in Appendix C of AG-1. Neither Aluminum or Tin are defined as having high toxicity.

The AGCs were calculated using the recommended equation:

$$\frac{ACGIH:\ TWA\text{-}TLV\ or\ NIOSH:\ TWA\text{-}REL}{420} = Interim\ AGC$$

where:

ACGIH	-	American Conference of Governmental Industrial Hygenists
TWA	-	Time Weighted Average
TLU	-	Threshold Limit Value
NIOSH	-	National Institute of Occupational Safety and Health
NEL	-	Recommended Exposure Limit

The SGCs were calculated using the recommended equation:

$$\frac{Lesser\ of\ ACGIH:\ TWA\text{-}TLV\ or\ NIOSH:\ TWA\text{-}REL}{4.2} = Interim\ SGC$$

NIOSH and ACGIH Exposure Limits

COMPOUND	NIOSH[1] TWA-REL	ACGIH[2] TWA-TLV
Aluminum	-	2 mg/m^3
Tin	2 mg/m^3	2 mg/m^3

1: NIOSH - Pocket Guide June 1990

2: ACGIH - Threshold Limit Valves 1990-1991

$$Tin:\ Interim\ AGC = \frac{2000\ ug/m^3}{420} = 4.76\ ug/m^3$$

$$Tin:\ Interim\ SGC = \frac{2000\ ug/m^3}{4.2} = 476\ ug/m^3$$

$$Aluminum:\ Interim\ AGC = \frac{2000\ ug/m^3}{420} = 4.76\ ug/m^3$$

$$Aluminum:\ Interim\ SGC = \frac{2000\ ug/m^3}{4.2} = 476\ ug/m^3$$

As no occupational exposure limits are available for strontium, deminimis values must be used in accordance with AG-1 (IV.4.2.a.2.). **Appendix K** presents a review of toxicological data available for Strontium. This data shows that strontium is a low toxicity metal.

Aluminum

SGC = 476 ug/m^3
AGC = 4.76 ug/m^3

Step 1: The SGC and the AGC are interim values as described in AG-1. No conversion to hourly on annual impacts is necessary.

Step 2:

$$Qs = 476 \times \frac{1}{18.55} = 25.66 \ lb/hr$$

Step 3:

$$Qa = 25.66 \times \frac{42.12 \ (4.76)}{476} = 10.81 \ lb/hr$$

Step 4: Qa < Qs; therefore, the maximum hourly emission of aluminum = 10.81 lb/hr.

Tin

SGC = 476 ug/m^3
AGC = 4.76 ug/m^3

Step 1: The SGC and the AGC are interim values as described in AG-1. No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 476 \; x \; \frac{1}{18.55} = 25.66 \; lb/hr$$

Step 3:

$$Qa = 25.66 \; x \; \frac{42.12 \; (4.76)}{476} = 10.81 \; lb/hr$$

Step 4: Qa < Qs; therefore, the maximum hourly emission of aluminum = 10.81 lb/hr

Zinc

SGC = 150 ug/m^3
AGC = 50 ug/m^3

Step 1: No conversion to hourly or annual impacts is necessary.

Step 2:

$$Qs = 150 \times \frac{1}{18.55} = 8.09 \ lb/hr$$

Step 3:

$$Qa = 8.09 \times \frac{42.12 \ (50)}{150} = 113.53 \ lb/hr$$

Step 4: Qs < Qa; therefore, the maximum hourly emission of zinc = 8.09 lb/hr.

Strontium

No data in Air Guide - 1
No NIOSH - REL, or ACGIH - TLV

See Appendix K for the toxicological review of strontium compounds and the designation of strontium as a low toxicity constituent. The low toxicity deminimis value is used.

Deminimis AGC for a low toxicity metal = 1.0 ug/m^3

Step 1:

$$Calculate\ a\ SGC = \frac{1.0\ ug/m^3}{0.1} = 10\ ug/m^3$$

Step 2:

$$Qs = 1\ x\ \frac{10}{18.55} = 0.54\ lb/hr$$

Step 3:

$$Qa = 0.54\ x\ \frac{42.12(1.0)}{10} = 2.27\ lb/hr$$

Step 4: Qs < Qa; therefore, the maximum deminimis hourly emission of strontium = 0.54 lb/hr.

Sulfur Dioxide (SO_2)

NAAQS = 365 ug/m^3 (24-hr impact)
NAAQS = AGC = 80 ug/m^3 (annual impact)

Step 1: Calculate SGC and AGC values for SO_2

SGC = 365 ug/m^3 /0.4 = 913 ug/m^3

AGC = 80 ug/m^3

Step 2:

$$Qs = 913 \ x \ 1/18.55 = 49.2 \ lb/hr$$

Step 3:

$$Qa = 42.12 \ x \ \frac{80}{1000} \ x \ 49.2 = 182 \ lb/hr$$

Step 4: Qs < Qa;, therefore the max hourly emission of SO_2 = 49.2 lb/hr.

Note: Maximum potential emission rate of SO_2 in tons per year assuming every pound of sulfur fed to the furnace is converted to SO_2.

$$49.2 \ lb/hr \ x \ 2080 \ \frac{Hrs}{Year} x \frac{Ton}{2000lb} = 51.2 \ ton/year \ SO_2$$

Nitrogen Dioxide (NO_2)

NAAQS = AGC = 100 ug/m^3

Step 1: No conversion to hourly or annual is necessary. Only the NAAQS for an annual impact is used.

Step 2:

$$Qa = NAAQS(Annual\ Impact)\ x\ 1/1.855\ x\ 8760/2080 = Allowable\ NO_2\ Emission\ lb/hr$$

$$Qa = 100\ x\ \frac{1}{1.855}\ x\ 8760/2080 = 227\ \ lb/hr\ NO_2$$

Note: Maximum potential emission rate of NO_2 in tons per year assuming a conservative 5% conversion of Nitrogen to NO_2. The source for the conversion was USATHAMA report DRXTH-TE-6R-84277, which delt with incineration of explosive compounds in soil.

$$227\ lb/hr\ x\ 2080\ hrs/year\ x\ \frac{ton}{2000\ lb}\ x\ 0.05 = 11.8\ ton/year\ NO_2$$

APPENDIX D-2

CALCULATION OF THE MAXIMUM ALLOWABLE IMPACT FOR CHROME VI UNDER FEDERAL GUIDANCE

D.2 Calculation of the Maximum Allowable Impact for Chrome IV Under Federal Guidance

The only carcinogenic metal identified in the PEP was Chromium. Therefore, the allowable cumulative additional risk to the MEI (1 cancer case per 100,000) exposed over a life time will be due soley to chromium (as Cr^{+6}).

The unit risk and the allowable life time risk to the MEI were obtained from Volume IV of the incinerator Guidance series.

$$(1)\ Cr^{+}6\ Unit\ Risk = \frac{1.2x10^{-2}}{ug/m^3}$$

$$(2)\ Allowable\ associated\ lifetime\ risk = 1x10^{-5}$$

Calculate the impact which is associated with life time risk of 1 x 10^{-6} to the MEI.

$$1\ x\ 10^{-5}\ x\ \frac{1\ ug/m^3}{1.2\ x\ 10^{-2}} = 8.3\ x\ 10^{-4} ug/m^3$$

APPENDIX D-3

SAMPLING DURATION CALCULATIONS

APPENDIX D-3

SAMPLING DURATION CALCULATIONS

D.3 SAMPLING DURATION CALCULATIONS

D.3.1 DNT Sample Duration Calculation

Assumptions
Feed item - item 49
DNT Feedrate - 44.39 lb/hr
Sample method - STEM
DRE - 99.99%
Stack Gas Flow Rate: 4000 scf/min..
Moisture: 7%.
Sample rate: 30 dscf/hr.
Minimum Sample Needed for Detection: 15 ug.
Desired Sample: 50 ug.

Sample Duration - 1hr

STEP 1 Calculate POHC Emission Rate (lb/hr):

POHC Emissions (w/DRE of 99.99%), lb/hr = [POHC Feed Rate, lb/hr X (1 - 0.9999)]

DNT Emissions = 44.39 lb/hr X (1 - 0.9999) = 0.004439 lb/hr.

STEP 2 Calculate POHC Concentration in Stack Gas (lb/scf):

POHC concentration (lb/scf) = [POHC Emission Rate (lb/hr)] / [stack gas flow rate (scf/min) X 60 min/hr].

DNT Conc. = [0.004439 lb/hr] / [4000 scf/min X 60 min/hr] = 1.80×10^{-8} lb/scf.

STEP 3 Calculate POHC Concentration corrected for 7% Moisture (lb/dscf):

POHC concentration (lb/dscf) = [POHC Conc. (lb/scf)] X [(1 scf) / (1 - 0.07) dscf]

DNT Conc. (lb/dscf) = [1.80×10^{-8} lb/scf] X [(1 scf) / (1 - 0.07) dscf] = 1.90×10^{-8} lb/dscf

STEP 4 Calculate POHC Collection Rate (ug/hr):

POHC Collection Rate (ug/hr) = [POHC Conc. (lb/dscf)] X [Sampling Rate (dscf/hr)] X [453,592,370 (ug/lb)]

DNT Collection Rate (ug/hr) = 1.90 X 10^{-8} lb/dscf X 30 dscf/hr X 453,592,370 ug/lb = 263.4 ug/hr.

Test length requires a minimum of 50 ug sample available in one hour.

Test length needed for DNT = 50 ug/263.4 ug/hr = 0.19 hr.

Therefore, one hour test run will be sufficient.

D.3.2 NG Sample Duration Calculation

Assumptions:

Feed Item - Item 143
NG Feed Rate - 28.86 lb/hr

Other Assumptions - See D.3.1 above

STEP 1 - Calculate POHC Emission Rate (lb/hr):

NG Emmissions = 28.86 lb/hr x (1-0.9999)

NG Emissions = .002886 lb/hr

STEP 2 - Calculate POHC Concentration in Stack Gas (lb/scf):

NG Concentration = [.002886 lb/hr] / [4000 scf/min X 60 min/hr] = 1.20 X 10^{-8} lb/scf

STEP 3 - Calculate POHC Concentration corrected for 7% Moisture (lb/dscf):

NG concentration (lb/dscf) = [1.20 X 10^{-8} (lb/scf)] X [(1 scf) / (1 - 0.07)dscf] =
1.30 X 10^{-8} lb/dscf

STEP 4 - Calculate POHC Collection Rate(ug/hr):

NG Collection Rate (ug/hr) = 1.30 X 10^{-8} lb/dscf X 30 dscf/hr X 453,592,370 ug/lb = 177 ug/hr

Test length requires minimum of 50 ug sample available.
Test length needed for NG = 50 ug/177 ug/hr = 0.28 hr.

Therefore, a one hour test run will be sufficient

D.3.3 HCB Sample Duration Calculation

Assumptions
Feed item - HCB Spiked Item 182
HCB Feedrate - 4.11 lb/hr
Sample method - MM5
Analytical Method - SW846; Method 8120A
DRE - 99.99%
Stack Gas Flow Rate: 4000 scf/min.
Moisture: 7%.
Sample rate: 30 dscf/hr.
Detection limit 10 ug/l (8270)
Final Analytical Volume: 1 ml
Desired Sample: 20 ug.

STEP 1 Calculate POHC Emission Rate (lb/hr):

POHC Emissions (w/DRE of 99.99%), lb/hr = [POHC Feed Rate, lb/hr X (1 - 0.9999)]

HCB Emissions = 4.11 lb/hr X (1 - 0.9999) = 0.000411 lb/hr = 4.11 x 10^{-4}.

STEP 2 Calculate POHC Concentration in Stack Gas (lb/scf):

POHC concentration (lb/scf) = [POHC Emission Rate (lb/hr)] / [stack gas flow rate (scf/min) X 60 min/hr].

HCB Conc. = [4.11 x 10^{-4} lb/hr] / [4000 scf/min X 60 min/hr] = 1.7 x 10^{-9} lb/scf.

STEP 3 Calculate POHC Concentration corrected for 7% Moisture (lb/dscf):

POHC concentration (lb/dscf) = [POHC Conc. (lb/scf)] X [(1 scf) / (1 - 0.07) dscf]

HCB Conc. (lb/dscf) = [1.7 X 10^{-9} lb/scf] X [(1 scf) / (1 - 0.07) dscf] = 1.8 X 10^{-9} lb/dscf

STEP 4 Calculate POHC Collection Rate (ug/hr):

POHC Collection Rate (ug/hr) = [POHC Conc. (lb/dscf)] X [Sampling Rate (dscf/hr)] X [453,592,370 (ug/lb)]

HCB Collection Rate (ug/hr) = 1.8 X 10^{-9} lb/dscf X 30 dscf/hr X 453,592,370 ug/lb = 25.06 ug/hr.

Test length needed for HCB = 20 ug/25.06 ug/hr = 0.8 hr.

D.3.4 TCE Sample Duration Calculation

Assumptions
Feed item - TCE Spiked Item 1B2
TCE Feedrate - 4.11 lb/hr
Sample method - VOST
Analytical Method - SW846, 5040A & 8240
DRE - 99.99%
Stack Gas Flow Rate: 4000 scf/min..
Moisture: 7%.
Sample rate: 1 liter/min.
Minimum Sample Needed for Detection: 20 ng.
Desired Sample (includes 10x Safety Factor): 0.2 ug.
Sample Duration - 20 minutes

STEP 1 Calculate POHC Emission Rate (lb/hr):

POHC Emissions (w/DRE of 99.99%), lb/hr = [POHC Feed Rate, lb/hr X (1 - 0.9999)]

TCE Emissions = 4.11 lb/hr X (1 - 0.9999) = 0.000411 lb/hr = 4.11×10^{-4} lb/hr.

STEP 2 Calculate POHC Concentration in Stack Gas (lb/scf):

POHC concentration (lb/scf) = [POHC Emission Rate (lb/hr)] / [stack gas flow rate (scf/min) X 60 min/hr].

TCE Conc. = [4.11×10^{-4} lb/hr] / [4000 scf/min X 60 min/hr] = 1.7 X 10^{-9} lb/scf.

STEP 3 Calculate POHC Concentration corrected for 7% Moisture (lb/dscf):

POHC concentration (lb/dscf) = [POHC Conc. (lb/scf)] X [(1 scf) / (1 - 0.07) dscf]

TCE Conc. (lb/dscf) = [1.7 X 10^{-9} lb/scf] X [(1 scf) / (1 - 0.07) dscf] = 1.8 X 10^{-9} lb/dscf

STEP 4 Calculate POHC Collected in 20 minutes:

POHC Collection Rate (ug) = [POHC Conc. (lb/dscf)] X [(Ft^3/liter) x Sample Volume (liters)] X [453,592,370 (ug/lb)]

TCE Collection Rate (ug) = 1.8 X 10^{-9} lb/dscf X Ft^3/28.3 liter x 20 liter X 453,592,370 ug/lb = 0.58 ug.

Test length requires a minimum of 0.2 ug sample available in 20 minutes.

Therefore, one 20 minute test run will be sufficient.

APPENDIX D-4

MATERIAL BALANCES

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	22.41 %
WEIGHT % H =	12.50 %	1.36 %
WEIGHT % O =	0.00 %	37.26 %
WEIGHT % N =	0.00 %	9.16 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	28.42 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	14,013 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	4,553 Btu/lb
FEED RATE TO KILN =	62.00 lbs/hr	11.31 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	260.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS		
KILN INSIDE DIAMETER =	2.54	feet
KILN LENGTH =	20.00	feet
KILN INSIDE VOLUME =	102	ft3
KILN SLOPE =	0.188	ft/ft
KILN ROTATIONAL VELOCITY =	1.00	rpm
SCC LENGTH =	4.67	feet
SCC WIDTH =	4.67	feet
SCC HEIGHT =	15.00	feet
SCC INSIDE VOLUME =	327	ft3
COMBUSTION INLET AIR CONDITIONS		
PRIMARY AIR TEMPERATURE =	50	degF
PRIMARY AIR REL HUMIDITY =	70	%
SECONDARY AIR TEMPERATURE =	50	degF
SECONDARY AIR REL HUMIDITY =	70	%
INCINERATOR OPERATING CONDITIONS		
WASTE FEED TEMPERATURES =	50	degF
KILN OPERATING TEMPERATURE =	450	degF
SCC OPERATING TEMPERATURE =	1,600	degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	800	degF
LOW TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	240	degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES		
PARTICULATE REMOVED AS ASH IN KILN =	0.0	%
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0	%
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0	%
PARTICULATE REMOVAL IN CYCLONE =	30.0	%
PARTICULATE REMOVAL IN BAGHOUSE =	95.0	%
STACK GAS CONDITIONS		
STACK GAS TEMPERATURE =	240	degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	11.31 lbs/hr	
AUXILIARY FUEL FEED =	62.00 lbs/hr	260.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	910 lbs/hr	3,717 lbs/hr
COMBUSTION AIR REQUIRED =	2,277 scfm	858 scfm
	11,038 lbs/hr	4,159 lbs/hr
EXCESS AIR REQUIRED =	1,107 %	12 %
FLUE GAS PRODUCED =	11,110 lbs/hr	15,528 lbs/hr
	386 lbmol/hr	539 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	8.0 minutes	
MAX GAS RESIDENCE TIME =	1.42 seconds	1.45 seconds
FLUE GAS VELOCITY =	14.0 ft/sec	10.3 ft/sec

ENERGY BALANCES

HEAT RELEASE RATE =	1,256,154 Btu/hr	5,051,800 Btu/hr
OPERATING TEMPERATURE =	450 degF	1,600 degF
RADIATION HEAT LOSSES =	2,788 Btu/hr	15,377 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	51,494 Btu/hr	
AUX FUEL HEAT INPUT =	1,204,660 Btu/hr	5,051,800 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,253,366 Btu/hr	6,289,789 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,376 Btu/hr/ft3	19,274 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	0.07 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	0.15 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	0.22 lbs/hr

ENERGY BALANCES

HEAT REMOVED BY HI TEMP COOLER =	3,478,359 Btu/hr
COOLING AIR TEMP OUT =	158 degF
HEAT REMOVED BY LO TEMP COOLER =	2,434,851 Btu/hr
COOLING AIR TEMP OUT =	168 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER	EMISSIONS AFTER HI TEMP GAS COOLER
FLUE GAS FLOWRATE :		
SCFM :	3,224 scfm	3,224 scfm
ACFM :	13,504 acfm	8,260 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,528 lbs/hr	15,528 lbs/hr
LBS/HR DRY :	15,087 lbs/hr	15,087 lbs/hr
FLUE GAS TEMPERATURE :	1,600 degF	800 degF
FLUE GAS SATURATION TEMP :	171 degF	149 degF
FLUE GAS ACTUAL HUMIDITY :	0.0293	0.0293
FLUE GAS SAT HUMIDITY :	0.4319	0.2003
FLUE GAS COMPOSITION :		
N2 :	76.86 %	76.86 %
O2 :	14.17 %	14.17 %
CO2 :	4.38 %	4.38 %
H2O :	4.55 %	4.55 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	3.31 lbs/hr	3.31 lbs/hr
CONCENTRATION :	169 ppmv	169 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.93 lbs/hr	1.93 lbs/hr
CONCENTRATION :	56 ppmv	56 ppmv
NOx :		
FLOWRATE :	2.13 lbs/hr	2.13 lbs/hr
CONCENTRATION :	86 ppmv	86 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	0.23 lbs/hr	0.22 lbs/hr
CONCENTRATION :	0.01 gr/dscf	0.01 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	3,224 scfm	3,224 scfm
ACFM :	4,589 acfm	4,589 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,528 lbs/hr	15,528 lbs/hr
LBS/HR DRY :	15,087 lbs/hr	15,087 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	112 degF	112 degF
FLUE GAS ACTUAL HUMIDITY :	0.0293	0.0293
FLUE GAS SAT HUMIDITY :	0.0609	0.0609
FLUE GAS COMPOSITION :		
N2 :	76.86 %	76.86 %
O2 :	14.17 %	14.17 %
CO2 :	4.38 %	4.38 %
H2O :	4.55 %	4.55 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	3.31 lbs/hr	3.31 lbs/hr
CONCENTRATION :	169 ppmv	169 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.93 lbs/hr	1.93 lbs/hr
CONCENTRATION :	56 ppmv	56 ppmv
NOx :		
FLOWRATE :	2.13 lbs/hr	2.13 lbs/hr
CONCENTRATION :	86 ppmv	86 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	0.22 lbs/hr	0.16 lbs/hr
CONCENTRATION :	0.0084 gr/dscf	0.0059 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,224 scfm	3,224 scfm
ACFM :	4,589 acfm	4,589 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,528 lbs/hr	15,528 lbs/hr
LBS/HR DRY :	15,087 lbs/hr	15,087 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	112 degF	112 degF
FLUE GAS ACTUAL HUMIDITY :	0.0293	0.0293
FLUE GAS SAT HUMIDITY :	0.0609	0.0609
FLUE GAS COMPOSITION :		
N2 :	76.86 %	76.86 %
O2 :	14.17 %	14.17 %
CO2 :	4.38 %	4.38 %
H2O :	4.55 %	4.55 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	3.31 lbs/hr	3.31 lbs/hr
CONCENTRATION :	169 ppmv	169 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.93 lbs/hr	1.93 lbs/hr
CONCENTRATION :	56 ppmv	56 ppmv
NOx :		
FLOWRATE :	2.13 lbs/hr	2.13 lbs/hr
CONCENTRATION :	86 ppmv	86 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	0.01 lbs/hr	0.01 lbs/hr
CONCENTRATION :	0.0003 gr/dscf	0.0003 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0007 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 1
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	79 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	442 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	442 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	0.23 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	0.23 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	0.23 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	0.23 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		21 degF
CRITICAL HUMIDITY =		0.0029 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00012 X T 0.00038

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-10-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	19.85 %
WEIGHT % H =	12.50 %	1.64 %
WEIGHT % O =	0.00 %	37.26 %
WEIGHT % N =	0.00 %	9.16 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	30.69 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	14,013 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	4,364 Btu/lb
FEED RATE TO KILN =	62.00 lbs/hr	11.31 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	260.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS		
KILN INSIDE DIAMETER =	2.54	feet
KILN LENGTH =	20.00	feet
KILN INSIDE VOLUME =	102	ft3
KILN SLOPE =	0.188	ft/ft
KILN ROTATIONAL VELOCITY =	1.00	rpm
SCC LENGTH =	4.67	feet
SCC WIDTH =	4.67	feet
SCC HEIGHT =	15.00	feet
SCC INSIDE VOLUME =	327	ft3
COMBUSTION INLET AIR CONDITIONS		
PRIMARY AIR TEMPERATURE =	50	degF
PRIMARY AIR REL HUMIDITY =	70	%
SECONDARY AIR TEMPERATURE =	50	degF
SECONDARY AIR REL HUMIDITY =	70	%
INCINERATOR OPERATING CONDITIONS		
WASTE FEED TEMPERATURES =	50	degF
KILN OPERATING TEMPERATURE =	450	degF
SCC OPERATING TEMPERATURE =	1,600	degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	800	degF
LOW TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	240	degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES		
PARTICULATE REMOVED AS ASH IN KILN =	0.0	%
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0	%
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0	%
PARTICULATE REMOVAL IN CYCLONE =	30.0	%
PARTICULATE REMOVAL IN BAGHOUSE =	95.0	%
STACK GAS CONDITIONS		
STACK GAS TEMPERATURE =	240	degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	11.31 lbs/hr	
AUXILIARY FUEL FEED =	62.00 lbs/hr	260.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	907 lbs/hr	3,717 lbs/hr
COMBUSTION AIR REQUIRED =	2,273 scfm	861 scfm
	11,019 lbs/hr	4,173 lbs/hr
EXCESS AIR REQUIRED =	1,108 %	12 %
FLUE GAS PRODUCED =	11,090 lbs/hr	15,523 lbs/hr
	385 lbmol/hr	539 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	8.0 minutes	
MAX GAS RESIDENCE TIME =	1.43 seconds	1.45 seconds
FLUE GAS VELOCITY =	14.0 ft/sec	10.3 ft/sec

ENERGY BALANCES

HEAT RELEASE RATE =	1,254,017 Btu/hr	5,051,800 Btu/hr
OPERATING TEMPERATURE =	450 degF	1,600 degF
RADIATION HEAT LOSSES =	2,788 Btu/hr	15,377 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	49,357 Btu/hr	
AUX FUEL HEAT INPUT =	1,204,660 Btu/hr	5,051,800 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,251,229 Btu/hr	6,287,652 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,355 Btu/hr/ft3	19,267 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	0.07 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	0.15 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	0.22 lbs/hr

ENERGY BALANCES

HEAT REMOVED BY HI TEMP COOLER =	3,477,225 Btu/hr
COOLING AIR TEMP OUT =	158 degF
HEAT REMOVED BY LO TEMP COOLER =	2,434,058 Btu/hr
COOLING AIR TEMP OUT =	168 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER		EMISSIONS AFTER HI TEMP GAS COOLER	
FLUE GAS FLOWRATE :				
SCFM :	3,223	scfm	3,223	scfm
ACFM :	13,500	acfm	8,257	acfm
LBMOL/HR :	539	lbmol/hr	539	lbmol/hr
LBS/HR :	15,523	lbs/hr	15,523	lbs/hr
LBS/HR DRY :	15,081	lbs/hr	15,081	lbs/hr
FLUE GAS TEMPERATURE :	1,600	degF	800	degF
FLUE GAS SATURATION TEMP :	171	degF	149	degF
FLUE GAS ACTUAL HUMIDITY :	0.0293		0.0293	
FLUE GAS SAT HUMIDITY :	0.4319		0.2003	
FLUE GAS COMPOSITION :				
N2 :	76.85	%	76.85	%
O2 :	14.17	%	14.17	%
CO2 :	4.38	%	4.38	%
H2O :	4.56	%	4.56	%
EXCESS AIR :	227	%	227	%
FLUE GAS CONTAMINANTS :				
HCl :				
FLOWRATE :	3.58	lbs/hr	3.58	lbs/hr
CONCENTRATION :	182	ppmv	182	ppmv
HF :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
Br2 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
P2O5 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
SO2 :				
FLOWRATE :	1.93	lbs/hr	1.93	lbs/hr
CONCENTRATION :	56	ppmv	56	ppmv
NOx :				
FLOWRATE :	2.13	lbs/hr	2.13	lbs/hr
CONCENTRATION :	86	ppmv	86	ppmv
CO :				
FLOWRATE :	0.43	lbs/hr	0.43	lbs/hr
CONCENTRATION :	29	ppmv	29	ppmv
PARTICULATES :				
FLOWRATE :	0.23	lbs/hr	0.22	lbs/hr
CONCENTRATION :	0.01	gr/dscf	0.01	gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	3,223 scfm	3,223 scfm
ACFM :	4,587 acfm	4,587 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,523 lbs/hr	15,523 lbs/hr
LBS/HR DRY :	15,081 lbs/hr	15,081 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	112 degF	112 degF
FLUE GAS ACTUAL HUMIDITY :	0.0293	0.0293
FLUE GAS SAT HUMIDITY :	0.0609	0.0609
FLUE GAS COMPOSITION :		
N2 :	76.85 %	76.85 %
O2 :	14.17 %	14.17 %
CO2 :	4.38 %	4.38 %
H2O :	4.56 %	4.56 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	3.58 lbs/hr	3.58 lbs/hr
CONCENTRATION :	182 ppmv	182 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.93 lbs/hr	1.93 lbs/hr
CONCENTRATION :	56 ppmv	56 ppmv
NOx :		
FLOWRATE :	2.13 lbs/hr	2.13 lbs/hr
CONCENTRATION :	86 ppmv	86 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	0.22 lbs/hr	0.16 lbs/hr
CONCENTRATION :	0.0084 gr/dscf	0.0059 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,223 scfm	3,223 scfm
ACFM :	4,587 acfm	4,587 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,523 lbs/hr	15,523 lbs/hr
LBS/HR DRY :	15,081 lbs/hr	15,081 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	112 degF	112 degF
FLUE GAS ACTUAL HUMIDITY :	0.0293	0.0293
FLUE GAS SAT HUMIDITY :	0.0609	0.0609
FLUE GAS COMPOSITION :		
N2 :	76.85 %	76.85 %
O2 :	14.17 %	14.17 %
CO2 :	4.38 %	4.38 %
H2O :	4.56 %	4.56 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	3.58 lbs/hr	3.58 lbs/hr
CONCENTRATION :	182 ppmv	182 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.93 lbs/hr	1.93 lbs/hr
CONCENTRATION :	56 ppmv	56 ppmv
NOx :		
FLOWRATE :	2.13 lbs/hr	2.13 lbs/hr
CONCENTRATION :	86 ppmv	86 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	0.01 lbs/hr	0.01 lbs/hr
CONCENTRATION :	0.0003 gr/dscf	0.0003 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0007 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 2
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	79 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	442 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	442 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	0.23 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	0.23 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	0.23 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	0.23 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		21 degF
CRITICAL HUMIDITY =		0.0029 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00012 X T 0.00038

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	20.92 %
WEIGHT % H =	12.50 %	2.31 %
WEIGHT % O =	0.00 %	60.34 %
WEIGHT % N =	0.00 %	15.85 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	5,808 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	5,343 Btu/lb
FEED RATE TO KILN =	34.00 lbs/hr	110.98 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	255.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS		
KILN INSIDE DIAMETER =	2.54	feet
KILN LENGTH =	20.00	feet
KILN INSIDE VOLUME =	102	ft3
KILN SLOPE =	0.188	ft/ft
KILN ROTATIONAL VELOCITY =	1.50	rpm
SCC LENGTH =	4.67	feet
SCC WIDTH =	4.67	feet
SCC HEIGHT =	15.00	feet
SCC INSIDE VOLUME =	327	ft3
COMBUSTION INLET AIR CONDITIONS		
PRIMARY AIR TEMPERATURE =	50	degF
PRIMARY AIR REL HUMIDITY =	70	%
SECONDARY AIR TEMPERATURE =	50	degF
SECONDARY AIR REL HUMIDITY =	70	%
INCINERATOR OPERATING CONDITIONS		
WASTE FEED TEMPERATURES =	50	degF
KILN OPERATING TEMPERATURE =	400	degF
SCC OPERATING TEMPERATURE =	1,450	degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	800	degF
LOW TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	240	degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES		
PARTICULATE REMOVED AS ASH IN KILN =	0.0	%
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0	%
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0	%
PARTICULATE REMOVAL IN CYCLONE =	30.0	%
PARTICULATE REMOVAL IN BAGHOUSE =	95.0	%
STACK GAS CONDITIONS		
STACK GAS TEMPERATURE =	240	degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	110.98 lbs/hr	
AUXILIARY FUEL FEED =	34.00 lbs/hr	255.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	727 lbs/hr	3,646 lbs/hr
COMBUSTION AIR REQUIRED =	2,536 scfm	883 scfm
	12,291 lbs/hr	4,277 lbs/hr
EXCESS AIR REQUIRED =	1,582 %	17 %
FLUE GAS PRODUCED =	12,407 lbs/hr	16,939 lbs/hr
	431 lbmol/hr	587 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	5.3 minutes	
MAX GAS RESIDENCE TIME =	1.35 seconds	1.44 seconds
FLUE GAS VELOCITY =	14.8 ft/sec	10.4 ft/sec

ENERGY BALANCES		
HEAT RELEASE RATE =	1,253,586 Btu/hr	4,954,650 Btu/hr
OPERATING TEMPERATURE =	400 degF	1,450 degF
RADIATION HEAT LOSSES =	2,664 Btu/hr	13,786 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	592,966 Btu/hr	
AUX FUEL HEAT INPUT =	660,620 Btu/hr	4,954,650 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,250,922 Btu/hr	6,191,787 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,350 Btu/hr/ft3	18,970 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.02 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	0.27 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	0.60 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	0.89 lbs/hr

ENERGY BALANCES	
HEAT REMOVED BY HI TEMP COOLER =	3,082,832 Btu/hr
COOLING AIR TEMP OUT =	146 degF
HEAT REMOVED BY LO TEMP COOLER =	2,655,978 Btu/hr
COOLING AIR TEMP OUT =	178 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER		EMISSIONS AFTER HI TEMP GAS COOLER	
FLUE GAS FLOWRATE :				
SCFM :	3,515	scfm	3,515	scfm
ACFM :	13,649	acfm	9,004	acfm
LBMOL/HR :	587	lbmol/hr	587	lbmol/hr
LBS/HR :	16,939	lbs/hr	16,939	lbs/hr
LBS/HR DRY :	16,504	lbs/hr	16,504	lbs/hr
FLUE GAS TEMPERATURE :	1,450	degF	800	degF
FLUE GAS SATURATION TEMP :	168	degF	149	degF
FLUE GAS ACTUAL HUMIDITY :	0.0263		0.0263	
FLUE GAS SAT HUMIDITY :	0.3811		0.1966	
FLUE GAS COMPOSITION :				
N2 :	76.86	%	76.86	%
O2 :	15.01	%	15.01	%
CO2 :	3.91	%	3.91	%
H2O :	4.11	%	4.11	%
EXCESS AIR :	277	%	277	%
FLUE GAS CONTAMINANTS :				
HCl :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
HF :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
Br2 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
P2O5 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
SO2 :				
FLOWRATE :	1.73	lbs/hr	1.73	lbs/hr
CONCENTRATION :	46	ppmv	46	ppmv
NOx :				
FLOWRATE :	29.36	lbs/hr	29.36	lbs/hr
CONCENTRATION :	1,086	ppmv	1,086	ppmv
CO :				
FLOWRATE :	0.41	lbs/hr	0.41	lbs/hr
CONCENTRATION :	25	ppmv	25	ppmv
PARTICULATES :				
FLOWRATE :	0.93	lbs/hr	0.91	lbs/hr
CONCENTRATION :	0.03	gr/dscf	0.03	gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	3,515 scfm	3,515 scfm
ACFM :	5,002 acfm	5,002 acfm
LBMOL/HR :	587 lbmol/hr	587 lbmol/hr
LBS/HR :	16,939 lbs/hr	16,938 lbs/hr
LBS/HR DRY :	16,504 lbs/hr	16,504 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	110 degF	110 degF
FLUE GAS ACTUAL HUMIDITY :	0.0263	0.0263
FLUE GAS SAT HUMIDITY :	0.0582	0.0582
FLUE GAS COMPOSITION :		
N2 :	76.86 %	76.86 %
O2 :	15.01 %	15.01 %
CO2 :	3.91 %	3.91 %
H2O :	4.11 %	4.11 %
EXCESS AIR :	277 %	277 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.73 lbs/hr	1.73 lbs/hr
CONCENTRATION :	46 ppmv	46 ppmv
NOx :		
FLOWRATE :	29.36 lbs/hr	29.36 lbs/hr
CONCENTRATION :	1,086 ppmv	1,086 ppmv
CO :		
FLOWRATE :	0.41 lbs/hr	0.41 lbs/hr
CONCENTRATION :	25 ppmv	25 ppmv
PARTICULATES :		
FLOWRATE :	0.91 lbs/hr	0.63 lbs/hr
CONCENTRATION :	0.0314 gr/dscf	0.0220 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,515 scfm	3,515 scfm
ACFM :	5,002 acfm	5,002 acfm
LBMOL/HR :	587 lbmol/hr	587 lbmol/hr
LBS/HR :	16,938 lbs/hr	16,938 lbs/hr
LBS/HR DRY :	16,504 lbs/hr	16,504 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	110 degF	110 degF
FLUE GAS ACTUAL HUMIDITY :	0.0263	0.0263
FLUE GAS SAT HUMIDITY :	0.0582	0.0582
FLUE GAS COMPOSITION :		
N2 :	76.86 %	76.86 %
O2 :	15.01 %	15.01 %
CO2 :	3.91 %	3.91 %
H2O :	4.11 %	4.11 %
EXCESS AIR :	277 %	277 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.73 lbs/hr	1.73 lbs/hr
CONCENTRATION :	46 ppmv	46 ppmv
NOx :		
FLOWRATE :	29.36 lbs/hr	29.36 lbs/hr
CONCENTRATION :	1,086 ppmv	1,086 ppmv
CO :		
FLOWRATE :	0.41 lbs/hr	0.41 lbs/hr
CONCENTRATION :	25 ppmv	25 ppmv
PARTICULATES :		
FLOWRATE :	0.03 lbs/hr	0.03 lbs/hr
CONCENTRATION :	0.0011 gr/dscf	0.0011 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0029 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO . 3
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	86 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	434 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	434 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	0.93 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	0.93 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	0.93 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	0.93 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		18 degF
CRITICAL HUMIDITY =		0.0026 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00011 X T 0.00066

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	32.66 %
WEIGHT % H =	12.50 %	2.64 %
WEIGHT % O =	0.00 %	47.68 %
WEIGHT % N =	0.00 %	13.53 %
WEIGHT % S =	0.30 %	0.62 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	1,232 ppm
PPM Na K B Ca Mg =	0 ppm	14,257 ppm
PPM HEAVY METALS =	0 ppm	13,217 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	5,864 Btu/lb
FEED RATE TO KILN =	37.00 lbs/hr	92.00 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	260.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS		
KILN INSIDE DIAMETER =	2.54	feet
KILN LENGTH =	20.00	feet
KILN INSIDE VOLUME =	102	ft3
KILN SLOPE =	0.188	ft/ft
KILN ROTATIONAL VELOCITY =	1.80	rpm
SCC LENGTH =	4.67	feet
SCC WIDTH =	4.67	feet
SCC HEIGHT =	15.00	feet
SCC INSIDE VOLUME =	327	ft3
COMBUSTION INLET AIR CONDITIONS		
PRIMARY AIR TEMPERATURE =	50	degF
PRIMARY AIR REL HUMIDITY =	70	%
SECONDARY AIR TEMPERATURE =	50	degF
SECONDARY AIR REL HUMIDITY =	70	%
INCINERATOR OPERATING CONDITIONS		
WASTE FEED TEMPERATURES =	50	degF
KILN OPERATING TEMPERATURE =	450	degF
SCC OPERATING TEMPERATURE =	1,600	degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	800	degF
LOW TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	240	degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES		
PARTICULATE REMOVED AS ASH IN KILN =	0.0	%
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0	%
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0	%
PARTICULATE REMOVAL IN CYCLONE =	30.0	%
PARTICULATE REMOVAL IN BAGHOUSE =	95.0	%
STACK GAS CONDITIONS		
STACK GAS TEMPERATURE =	240	degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	92.00 lbs/hr	
AUXILIARY FUEL FEED =	37.00 lbs/hr	260.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	897 lbs/hr	3,717 lbs/hr
COMBUSTION AIR REQUIRED =	2,279 scfm	855 scfm
	11,044 lbs/hr	4,144 lbs/hr
EXCESS AIR REQUIRED =	1,124 %	11 %
FLUE GAS PRODUCED =	11,152 lbs/hr	15,555 lbs/hr
	387 lbmol/hr	539 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	4.4 minutes	
MAX GAS RESIDENCE TIME =	1.42 seconds	1.45 seconds
FLUE GAS VELOCITY =	14.1 ft/sec	10.3 ft/sec

ENERGY BALANCES		
HEAT RELEASE RATE =	1,258,398 Btu/hr	5,051,800 Btu/hr
OPERATING TEMPERATURE =	450 degF	1,600 degF
RADIATION HEAT LOSSES =	2,788 Btu/hr	15,377 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	539,488 Btu/hr	
AUX FUEL HEAT INPUT =	718,910 Btu/hr	5,051,800 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,255,610 Btu/hr	6,292,033 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,398 Btu/hr/ft3	19,281 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.07 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	1.02 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	2.25 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	3.34 lbs/hr

ENERGY BALANCES	
HEAT REMOVED BY HI TEMP COOLER =	3,484,320 Btu/hr
COOLING AIR TEMP OUT =	159 degF
HEAT REMOVED BY LO TEMP COOLER =	2,439,024 Btu/hr
COOLING AIR TEMP OUT =	168 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER	EMISSIONS AFTER HI TEMP GAS COOLER
FLUE GAS FLOWRATE :		
SCFM :	3,225 scfm	3,225 scfm
ACFM :	13,511 acfm	8,264 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,555 lbs/hr	15,555 lbs/hr
LBS/HR DRY :	15,120 lbs/hr	15,120 lbs/hr
FLUE GAS TEMPERATURE :	1,600 degF	800 degF
FLUE GAS SATURATION TEMP :	171 degF	149 degF
FLUE GAS ACTUAL HUMIDITY :	0.0288	0.0288
FLUE GAS SAT HUMIDITY :	0.4315	0.1994
FLUE GAS COMPOSITION :		
N2 :	76.78 %	76.78 %
O2 :	14.17 %	14.17 %
CO2 :	4.47 %	4.47 %
H2O :	4.48 %	4.48 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.92 lbs/hr	2.92 lbs/hr
CONCENTRATION :	85 ppmv	85 ppmv
NOx :		
FLOWRATE :	20.90 lbs/hr	20.90 lbs/hr
CONCENTRATION :	843 ppmv	843 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	3.46 lbs/hr	3.39 lbs/hr
CONCENTRATION :	0.13 gr/dscf	0.13 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	3,225 scfm	3,225 scfm
ACFM :	4,591 acfm	4,591 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,555 lbs/hr	15,554 lbs/hr
LBS/HR DRY :	15,120 lbs/hr	15,120 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	112 degF	112 degF
FLUE GAS ACTUAL HUMIDITY :	0.0288	0.0288
FLUE GAS SAT HUMIDITY :	0.0605	0.0605
FLUE GAS COMPOSITION :		
N2 :	76.78 %	76.78 %
O2 :	14.17 %	14.17 %
CO2 :	4.47 %	4.47 %
H2O :	4.48 %	4.48 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.92 lbs/hr	2.92 lbs/hr
CONCENTRATION :	85 ppmv	85 ppmv
NOx :		
FLOWRATE :	20.90 lbs/hr	20.90 lbs/hr
CONCENTRATION :	843 ppmv	843 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	3.39 lbs/hr	2.37 lbs/hr
CONCENTRATION :	0.1283 gr/dscf	0.0898 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,225 scfm	3,225 scfm
ACFM :	4,590 acfm	4,590 acfm
LBMOL/HR :	539 lbmol/hr	539 lbmol/hr
LBS/HR :	15,552 lbs/hr	15,552 lbs/hr
LBS/HR DRY :	15,120 lbs/hr	15,120 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	112 degF	112 degF
FLUE GAS ACTUAL HUMIDITY :	0.0288	0.0288
FLUE GAS SAT HUMIDITY :	0.0605	0.0605
FLUE GAS COMPOSITION :		
N2 :	76.78 %	76.78 %
O2 :	14.17 %	14.17 %
CO2 :	4.47 %	4.47 %
H2O :	4.48 %	4.48 %
EXCESS AIR :	227 %	227 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.92 lbs/hr	2.92 lbs/hr
CONCENTRATION :	85 ppmv	85 ppmv
NOx :		
FLOWRATE :	20.90 lbs/hr	20.90 lbs/hr
CONCENTRATION :	843 ppmv	843 ppmv
CO :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	29 ppmv	29 ppmv
PARTICULATES :		
FLOWRATE :	0.12 lbs/hr	0.12 lbs/hr
CONCENTRATION :	0.0045 gr/dscf	0.0045 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0102 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 4
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	79 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	435 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	435 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	3.46 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	3.46 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	3.46 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	3.46 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		20 degF
CRITICAL HUMIDITY =		0.0028 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00012 X T 0.00043

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	13.63 %
WEIGHT % H =	12.50 %	1.03 %
WEIGHT % O =	0.00 %	27.33 %
WEIGHT % N =	0.00 %	8.29 %
WEIGHT % S =	0.30 %	0.35 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	690 ppm
PPM Na K B Ca Mg =	0 ppm	208,404 ppm
PPM HEAVY METALS =	0 ppm	284,701 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	5,856 Btu/lb
FEED RATE TO KILN =	25.00 lbs/hr	130.91 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	260.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS	
KILN INSIDE DIAMETER =	2.54 feet
KILN LENGTH =	20.00 feet
KILN INSIDE VOLUME =	102 ft3
KILN SLOPE =	0.188 ft/ft
KILN ROTATIONAL VELOCITY =	1.50 rpm
SCC LENGTH =	4.67 feet
SCC WIDTH =	4.67 feet
SCC HEIGHT =	15.00 feet
SCC INSIDE VOLUME =	327 ft3
COMBUSTION INLET AIR CONDITIONS	
PRIMARY AIR TEMPERATURE =	50 degF
PRIMARY AIR REL HUMIDITY =	70 %
SECONDARY AIR TEMPERATURE =	50 degF
SECONDARY AIR REL HUMIDITY =	70 %
INCINERATOR OPERATING CONDITIONS	
WASTE FEED TEMPERATURES =	50 degF
KILN OPERATING TEMPERATURE =	450 degF
SCC OPERATING TEMPERATURE =	1,600 degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	800 degF
LOW TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	240 degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES	
PARTICULATE REMOVED AS ASH IN KILN =	0.0 %
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0 %
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0 %
PARTICULATE REMOVAL IN CYCLONE =	30.0 %
PARTICULATE REMOVAL IN BAGHOUSE =	99.0 %
STACK GAS CONDITIONS	
STACK GAS TEMPERATURE =	240 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	130.91 lbs/hr	
AUXILIARY FUEL FEED =	25.00 lbs/hr	260.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	649 lbs/hr	3,717 lbs/hr
COMBUSTION AIR REQUIRED =	2,249 scfm	868 scfm
	10,900 lbs/hr	4,206 lbs/hr
EXCESS AIR REQUIRED =	1,570 %	13 %
FLUE GAS PRODUCED =	11,051 lbs/hr	15,517 lbs/hr
	380 lbmol/hr	535 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	5.3 minutes	
MAX GAS RESIDENCE TIME =	1.43 seconds	1.46 seconds
FLUE GAS VELOCITY =	14.0 ft/sec	10.3 ft/sec

ENERGY BALANCES

HEAT RELEASE RATE =	1,252,359 Btu/hr	5,051,800 Btu/hr
OPERATING TEMPERATURE =	450 degF	1,600 degF
RADIATION HEAT LOSSES =	2,788 Btu/hr	15,377 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	766,609 Btu/hr	
AUX FUEL HEAT INPUT =	485,750 Btu/hr	5,051,800 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,249,571 Btu/hr	6,285,994 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,338 Btu/hr/ft3	19,262 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	1.96 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	28.86 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	66.67 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	97.50 lbs/hr

ENERGY BALANCES

HEAT REMOVED BY HI TEMP COOLER =	3,475,350 Btu/hr
COOLING AIR TEMP OUT =	158 degF
HEAT REMOVED BY LO TEMP COOLER =	2,432,745 Btu/hr
COOLING AIR TEMP OUT =	168 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER	EMISSIONS AFTER HI TEMP GAS COOLER
FLUE GAS FLOWRATE :		
SCFM :	3,200 scfm	3,200 scfm
ACFM :	13,487 acfm	8,248 acfm
LBMOL/HR :	535 lbmol/hr	535 lbmol/hr
LBS/HR :	15,517 lbs/hr	15,515 lbs/hr
LBS/HR DRY :	15,106 lbs/hr	15,106 lbs/hr
FLUE GAS TEMPERATURE :	1,600 degF	800 degF
FLUE GAS SATURATION TEMP :	171 degF	149 degF
FLUE GAS ACTUAL HUMIDITY :	0.0272	0.0272
FLUE GAS SAT HUMIDITY :	0.4288	0.1978
FLUE GAS COMPOSITION :		
N2 :	76.98 %	76.98 %
O2 :	14.51 %	14.51 %
CO2 :	4.15 %	4.15 %
H2O :	4.27 %	4.27 %
EXCESS AIR :	244 %	244 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.62 lbs/hr	2.62 lbs/hr
CONCENTRATION :	77 ppmv	77 ppmv
NOx :		
FLOWRATE :	18.28 lbs/hr	18.28 lbs/hr
CONCENTRATION :	743 ppmv	743 ppmv
CO :		
FLOWRATE :	0.41 lbs/hr	0.41 lbs/hr
CONCENTRATION :	27 ppmv	27 ppmv
PARTICULATES :		
FLOWRATE :	98.17 lbs/hr	96.21 lbs/hr
CONCENTRATION :	3.74 gr/dscf	3.66 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER		EMISSIONS AFTER CYCLONE	
FLUE GAS FLOWRATE :				
SCFM :	3,200	scfm	3,200	scfm
ACFM :	4,582	acfm	4,574	acfm
LBMOL/HR :	535	lbmol/hr	535	lbmol/hr
LBS/HR :	15,515	lbs/hr	15,486	lbs/hr
LBS/HR DRY :	15,106	lbs/hr	15,106	lbs/hr
FLUE GAS TEMPERATURE :	240	degF	240	degF
FLUE GAS SATURATION TEMP :	111	degF	111	degF
FLUE GAS ACTUAL HUMIDITY :	0.0272		0.0272	
FLUE GAS SAT HUMIDITY :	0.0589		0.0589	
FLUE GAS COMPOSITION :				
N2 :	76.98	%	76.98	%
O2 :	14.51	%	14.51	%
CO2 :	4.15	%	4.15	%
H2O :	4.27	%	4.27	%
EXCESS AIR :	244	%	244	%
FLUE GAS CONTAMINANTS :				
HCl :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
HF :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
Br2 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
P2O5 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
SO2 :				
FLOWRATE :	2.62	lbs/hr	2.62	lbs/hr
CONCENTRATION :	77	ppmv	77	ppmv
NOx :				
FLOWRATE :	18.28	lbs/hr	18.28	lbs/hr
CONCENTRATION :	743	ppmv	743	ppmv
CO :				
FLOWRATE :	0.41	lbs/hr	0.41	lbs/hr
CONCENTRATION :	27	ppmv	27	ppmv
PARTICULATES :				
FLOWRATE :	96.21	lbs/hr	67.35	lbs/hr
CONCENTRATION :	3.6646	gr/dscf	2.5652	gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,200 scfm	3,200 scfm
ACFM :	4,554 acfm	4,554 acfm
LBMOL/HR :	535 lbmol/hr	535 lbmol/hr
LBS/HR :	15,419 lbs/hr	15,419 lbs/hr
LBS/HR DRY :	15,106 lbs/hr	15,106 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	111 degF	111 degF
FLUE GAS ACTUAL HUMIDITY :	0.0272	0.0272
FLUE GAS SAT HUMIDITY :	0.0589	0.0589
FLUE GAS COMPOSITION :		
N2 :	76.98 %	76.98 %
O2 :	14.51 %	14.51 %
CO2 :	4.15 %	4.15 %
H2O :	4.27 %	4.27 %
EXCESS AIR :	244 %	244 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.62 lbs/hr	2.62 lbs/hr
CONCENTRATION :	77 ppmv	77 ppmv
NOx :		
FLOWRATE :	18.28 lbs/hr	18.28 lbs/hr
CONCENTRATION :	743 ppmv	743 ppmv
CO :		
FLOWRATE :	0.41 lbs/hr	0.41 lbs/hr
CONCENTRATION :	27 ppmv	27 ppmv
PARTICULATES :		
FLOWRATE :	0.67 lbs/hr	0.67 lbs/hr
CONCENTRATION :	0.0257 gr/dscf	0.0257 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0615 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 5
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	79 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	411 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	411 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	98.17 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	98.17 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	98.17 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	98.17 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		19 degF
CRITICAL HUMIDITY =		0.0027 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00011 X T 0.00058

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	6.98 %
WEIGHT % H =	12.50 %	1.00 %
WEIGHT % O =	0.00 %	25.67 %
WEIGHT % N =	0.00 %	19.07 %
WEIGHT % S =	0.30 %	5.96 %
WEIGHT % Cl =	0.00 %	5.38 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	23,258 ppm
PPM Na K B Ca Mg =	0 ppm	59,358 ppm
PPM HEAVY METALS =	0 ppm	276,780 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	2,158 Btu/lb
FEED RATE TO KILN =	61.00 lbs/hr	33.17 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	174.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS	
KILN INSIDE DIAMETER =	2.54 feet
KILN LENGTH =	20.00 feet
KILN INSIDE VOLUME =	102 ft3
KILN SLOPE =	0.188 ft/ft
KILN ROTATIONAL VELOCITY =	1.20 rpm
SCC LENGTH =	4.67 feet
SCC WIDTH =	4.67 feet
SCC HEIGHT =	15.00 feet
SCC INSIDE VOLUME =	327 ft3
COMBUSTION INLET AIR CONDITIONS	
PRIMARY AIR TEMPERATURE =	50 degF
PRIMARY AIR REL HUMIDITY =	70 %
SECONDARY AIR TEMPERATURE =	50 degF
SECONDARY AIR REL HUMIDITY =	70 %
INCINERATOR OPERATING CONDITIONS	
WASTE FEED TEMPERATURES =	50 degF
KILN OPERATING TEMPERATURE =	400 degF
SCC OPERATING TEMPERATURE =	1,200 degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	800 degF
LOW TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	240 degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES	
PARTICULATE REMOVED AS ASH IN KILN =	0.0 %
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0 %
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0 %
PARTICULATE REMOVAL IN CYCLONE =	30.0 %
PARTICULATE REMOVAL IN BAGHOUSE =	95.0 %
STACK GAS CONDITIONS	
STACK GAS TEMPERATURE =	240 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	33.17 lbs/hr	
AUXILIARY FUEL FEED =	61.00 lbs/hr	174.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	959 lbs/hr	2,488 lbs/hr
COMBUSTION AIR REQUIRED =	2,553 scfm	566 scfm
	12,372 lbs/hr	2,746 lbs/hr
EXCESS AIR REQUIRED =	1,183 %	10 %
FLUE GAS PRODUCED =	12,455 lbs/hr	15,374 lbs/hr
	432 lbmol/hr	533 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	6.6 minutes	
MAX GAS RESIDENCE TIME =	1.34 seconds	1.82 seconds
FLUE GAS VELOCITY =	14.9 ft/sec	8.2 ft/sec

ENERGY BALANCES		
HEAT RELEASE RATE =	1,256,802 Btu/hr	3,380,820 Btu/hr
OPERATING TEMPERATURE =	400 degF	1,200 degF
RADIATION HEAT LOSSES =	2,664 Btu/hr	11,135 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	71,572 Btu/hr	
AUX FUEL HEAT INPUT =	1,185,230 Btu/hr	3,380,820 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,254,138 Btu/hr	4,623,824 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,382 Btu/hr/ft3	14,168 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.29 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	4.24 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	9.39 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	13.92 lbs/hr

ENERGY BALANCES	
HEAT REMOVED BY HI TEMP COOLER =	1,721,839 Btu/hr
COOLING AIR TEMP OUT =	104 degF
HEAT REMOVED BY LO TEMP COOLER =	2,410,575 Btu/hr
COOLING AIR TEMP OUT =	166 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER		EMISSIONS AFTER HI TEMP GAS COOLER	
FLUE GAS FLOWRATE :				
SCFM :	3,190	scfm	3,190	scfm
ACFM :	10,778	acfm	8,181	acfm
LBMOL/HR :	533	lbmol/hr	533	lbmol/hr
LBS/HR :	15,374	lbs/hr	15,374	lbs/hr
LBS/HR DRY :	15,028	lbs/hr	15,028	lbs/hr
FLUE GAS TEMPERATURE :	1,200	degF	800	degF
FLUE GAS SATURATION TEMP :	161	degF	148	degF
FLUE GAS ACTUAL HUMIDITY :	0.0230		0.0230	
FLUE GAS SAT HUMIDITY :	0.3027		0.1921	
FLUE GAS COMPOSITION :				
N2 :	77.26	%	77.26	%
O2 :	15.83	%	15.83	%
CO2 :	3.24	%	3.24	%
H2O :	3.60	%	3.60	%
EXCESS AIR :	336	%	336	%
FLUE GAS CONTAMINANTS :				
HCl :				
FLOWRATE :	1.84	lbs/hr	1.84	lbs/hr
CONCENTRATION :	95	ppmv	95	ppmv
HF :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
Br2 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
P2O5 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
SO2 :				
FLOWRATE :	5.36	lbs/hr	5.36	lbs/hr
CONCENTRATION :	157	ppmv	157	ppmv
NOx :				
FLOWRATE :	10.72	lbs/hr	10.72	lbs/hr
CONCENTRATION :	437	ppmv	437	ppmv
CO :				
FLOWRATE :	0.32	lbs/hr	0.32	lbs/hr
CONCENTRATION :	22	ppmv	22	ppmv
PARTICULATES :				
FLOWRATE :	14.42	lbs/hr	14.13	lbs/hr
CONCENTRATION :	0.55	gr/dscf	0.54	gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER		EMISSIONS AFTER CYCLONE	
FLUE GAS FLOWRATE :				
SCFM :	3,190	scfm	3,190	scfm
ACFM :	4,545	acfm	4,544	acfm
LBMOL/HR :	533	lbmol/hr	533	lbmol/hr
LBS/HR :	15,374	lbs/hr	15,369	lbs/hr
LBS/HR DRY :	15,028	lbs/hr	15,028	lbs/hr
FLUE GAS TEMPERATURE :	240	degF	240	degF
FLUE GAS SATURATION TEMP :	108	degF	108	degF
FLUE GAS ACTUAL HUMIDITY :	0.0230		0.0230	
FLUE GAS SAT HUMIDITY :	0.0549		0.0549	
FLUE GAS COMPOSITION :				
N2 :	77.26	%	77.26	%
O2 :	15.83	%	15.83	%
CO2 :	3.24	%	3.24	%
H2O :	3.60	%	3.60	%
EXCESS AIR :	336	%	336	%
FLUE GAS CONTAMINANTS :				
HCl :				
FLOWRATE :	1.84	lbs/hr	1.84	lbs/hr
CONCENTRATION :	95	ppmv	95	ppmv
HF :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
Br2 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
P2O5 :				
FLOWRATE :	0.00	lbs/hr	0.00	lbs/hr
CONCENTRATION :	0	ppmv	0	ppmv
SO2 :				
FLOWRATE :	5.36	lbs/hr	5.36	lbs/hr
CONCENTRATION :	157	ppmv	157	ppmv
NOx :				
FLOWRATE :	10.72	lbs/hr	10.72	lbs/hr
CONCENTRATION :	437	ppmv	437	ppmv
CO :				
FLOWRATE :	0.32	lbs/hr	0.32	lbs/hr
CONCENTRATION :	22	ppmv	22	ppmv
PARTICULATES :				
FLOWRATE :	14.13	lbs/hr	9.89	lbs/hr
CONCENTRATION :	0.5359	gr/dscf	0.3751	gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,190 scfm	3,190 scfm
ACFM :	4,541 acfm	4,541 acfm
LBMOL/HR :	533 lbmol/hr	533 lbmol/hr
LBS/HR :	15,360 lbs/hr	15,360 lbs/hr
LBS/HR DRY :	15,028 lbs/hr	15,028 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	108 degF	108 degF
FLUE GAS ACTUAL HUMIDITY :	0.0230	0.0230
FLUE GAS SAT HUMIDITY :	0.0549	0.0549
FLUE GAS COMPOSITION :		
N2 :	77.26 %	77.26 %
O2 :	15.83 %	15.83 %
CO2 :	3.24 %	3.24 %
H2O :	3.60 %	3.60 %
EXCESS AIR :	336 %	336 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	1.84 lbs/hr	1.84 lbs/hr
CONCENTRATION :	95 ppmv	95 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	5.36 lbs/hr	5.36 lbs/hr
CONCENTRATION :	157 ppmv	157 ppmv
NOx :		
FLOWRATE :	10.72 lbs/hr	10.72 lbs/hr
CONCENTRATION :	437 ppmv	437 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
PARTICULATES :		
FLOWRATE :	0.49 lbs/hr	0.49 lbs/hr
CONCENTRATION :	0.0188 gr/dscf	0.0188 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0573 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 6
DATE : 03-29-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	79 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	345 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	345 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	14.42 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	14.42 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	14.42 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	14.42 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		14 degF
CRITICAL HUMIDITY =		0.0022 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00009 X T 0.00091

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	1.00 %
WEIGHT % H =	12.50 %	0.03 %
WEIGHT % O =	0.00 %	4.85 %
WEIGHT % N =	0.00 %	22.13 %
WEIGHT % S =	0.30 %	1.79 %
WEIGHT % Cl =	0.00 %	1.59 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	6,808 ppm
PPM Na K B Ca Mg =	0 ppm	30,540 ppm
PPM HEAVY METALS =	0 ppm	648,716 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	1,005 Btu/lb
FEED RATE TO KILN =	64.00 lbs/hr	2.79 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	172.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS	
KILN INSIDE DIAMETER =	2.54 feet
KILN LENGTH =	20.00 feet
KILN INSIDE VOLUME =	102 ft3
KILN SLOPE =	0.188 ft/ft
KILN ROTATIONAL VELOCITY =	1.20 rpm
SCC LENGTH =	4.67 feet
SCC WIDTH =	4.67 feet
SCC HEIGHT =	15.00 feet
SCC INSIDE VOLUME =	327 ft3
COMBUSTION INLET AIR CONDITIONS	
PRIMARY AIR TEMPERATURE =	50 degF
PRIMARY AIR REL HUMIDITY =	70 %
SECONDARY AIR TEMPERATURE =	50 degF
SECONDARY AIR REL HUMIDITY =	70 %
INCINERATOR OPERATING CONDITIONS	
WASTE FEED TEMPERATURES =	50 degF
KILN OPERATING TEMPERATURE =	400 degF
SCC OPERATING TEMPERATURE =	1,200 degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	800 degF
LOW TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	240 degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES	
PARTICULATE REMOVED AS ASH IN KILN =	0.0 %
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0 %
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0 %
PARTICULATE REMOVAL IN CYCLONE =	30.0 %
PARTICULATE REMOVAL IN BAGHOUSE =	95.0 %
STACK GAS CONDITIONS	
STACK GAS TEMPERATURE =	240 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	2.79 lbs/hr	
AUXILIARY FUEL FEED =	64.00 lbs/hr	172.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	923 lbs/hr	2,459 lbs/hr
COMBUSTION AIR REQUIRED =	2,532 scfm	556 scfm
	12,273 lbs/hr	2,694 lbs/hr
EXCESS AIR REQUIRED =	1,222 %	10 %
FLUE GAS PRODUCED =	12,338 lbs/hr	15,204 lbs/hr
	429 lbmol/hr	528 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	6.6 minutes	
MAX GAS RESIDENCE TIME =	1.36 seconds	1.84 seconds
FLUE GAS VELOCITY =	14.7 ft/sec	8.1 ft/sec

ENERGY BALANCES

HEAT RELEASE RATE =	1,246,324 Btu/hr	3,341,960 Btu/hr
OPERATING TEMPERATURE =	400 degF	1,200 degF
RADIATION HEAT LOSSES =	2,664 Btu/hr	11,135 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	2,804 Btu/hr	
AUX FUEL HEAT INPUT =	1,243,520 Btu/hr	3,341,960 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,243,660 Btu/hr	4,574,485 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,279 Btu/hr/ft3	14,018 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.04 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	0.65 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	1.44 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	2.14 lbs/hr

ENERGY BALANCES

HEAT REMOVED BY HI TEMP COOLER =	1,702,883 Btu/hr
COOLING AIR TEMP OUT =	103 degF
HEAT REMOVED BY LO TEMP COOLER =	2,384,036 Btu/hr
COOLING AIR TEMP OUT =	165 degF

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER	EMISSIONS AFTER HI TEMP GAS COOLER
FLUE GAS FLOWRATE :		
SCFM :	3,159 scfm	3,159 scfm
ACFM :	10,663 acfm	8,093 acfm
LBMOL/HR :	528 lbmol/hr	528 lbmol/hr
LBS/HR :	15,204 lbs/hr	15,204 lbs/hr
LBS/HR DRY :	14,861 lbs/hr	14,861 lbs/hr
FLUE GAS TEMPERATURE :	1,200 degF	800 degF
FLUE GAS SATURATION TEMP :	161 degF	148 degF
FLUE GAS ACTUAL HUMIDITY :	0.0231	0.0231
FLUE GAS SAT HUMIDITY :	0.3028	0.1921
FLUE GAS COMPOSITION :		
N2 :	77.26 %	77.26 %
O2 :	15.87 %	15.87 %
CO2 :	3.25 %	3.25 %
H2O :	3.61 %	3.61 %
EXCESS AIR :	340 %	340 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.05 lbs/hr	0.05 lbs/hr
CONCENTRATION :	2 ppmv	2 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.51 lbs/hr	1.51 lbs/hr
CONCENTRATION :	45 ppmv	45 ppmv
NOx :		
FLOWRATE :	1.33 lbs/hr	1.33 lbs/hr
CONCENTRATION :	55 ppmv	55 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	21 ppmv	21 ppmv
PARTICULATES :		
FLOWRATE :	2.21 lbs/hr	2.17 lbs/hr
CONCENTRATION :	0.08 gr/dscf	0.08 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	3,159 scfm	3,159 scfm
ACFM :	4,496 acfm	4,496 acfm
LBMOL/HR :	528 lbmol/hr	528 lbmol/hr
LBS/HR :	15,204 lbs/hr	15,204 lbs/hr
LBS/HR DRY :	14,861 lbs/hr	14,861 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	108 degF	108 degF
FLUE GAS ACTUAL HUMIDITY :	0.0231	0.0231
FLUE GAS SAT HUMIDITY :	0.0551	0.0551
FLUE GAS COMPOSITION :		
N2 :	77.26 %	77.26 %
O2 :	15.87 %	15.87 %
CO2 :	3.25 %	3.25 %
H2O :	3.61 %	3.61 %
EXCESS AIR :	340 %	340 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.05 lbs/hr	0.05 lbs/hr
CONCENTRATION :	2 ppmv	2 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.51 lbs/hr	1.51 lbs/hr
CONCENTRATION :	45 ppmv	45 ppmv
NOx :		
FLOWRATE :	1.33 lbs/hr	1.33 lbs/hr
CONCENTRATION :	55 ppmv	55 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	21 ppmv	21 ppmv
PARTICULATES :		
FLOWRATE :	2.17 lbs/hr	1.52 lbs/hr
CONCENTRATION :	0.0832 gr/dscf	0.0582 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,159 scfm	3,159 scfm
ACFM :	4,496 acfm	4,496 acfm
LBMOL/HR :	528 lbmol/hr	528 lbmol/hr
LBS/HR :	15,202 lbs/hr	15,202 lbs/hr
LBS/HR DRY :	14,861 lbs/hr	14,861 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	108 degF	108 degF
FLUE GAS ACTUAL HUMIDITY :	0.0231	0.0231
FLUE GAS SAT HUMIDITY :	0.0551	0.0551
FLUE GAS COMPOSITION :		
N2 :	77.26 %	77.26 %
O2 :	15.87 %	15.87 %
CO2 :	3.25 %	3.25 %
H2O :	3.61 %	3.61 %
EXCESS AIR :	340 %	340 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.05 lbs/hr	0.05 lbs/hr
CONCENTRATION :	2 ppmv	2 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	1.51 lbs/hr	1.51 lbs/hr
CONCENTRATION :	45 ppmv	45 ppmv
NOx :		
FLOWRATE :	1.33 lbs/hr	1.33 lbs/hr
CONCENTRATION :	55 ppmv	55 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	21 ppmv	21 ppmv
PARTICULATES :		
FLOWRATE :	0.08 lbs/hr	0.08 lbs/hr
CONCENTRATION :	0.0029 gr/dscf	0.0029 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0090 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 7
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	78 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	343 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	343 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	2.21 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	2.21 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	2.21 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	2.21 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		14 degF
CRITICAL HUMIDITY =		0.0022 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00009 X T 0.00090

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 8
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	6.54 %
WEIGHT % H =	12.50 %	0.34 %
WEIGHT % O =	0.00 %	11.71 %
WEIGHT % N =	0.00 %	23.74 %
WEIGHT % S =	0.30 %	1.44 %
WEIGHT % Cl =	0.00 %	1.25 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	4,552 ppm
PPM Na K B Ca Mg =	0 ppm	13,753 ppm
PPM HEAVY METALS =	0 ppm	531,429 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	1,881 Btu/lb
FEED RATE TO KILN =	61.00 lbs/hr	33.31 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	173.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 8
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS	
KILN INSIDE DIAMETER =	2.54 feet
KILN LENGTH =	20.00 feet
KILN INSIDE VOLUME =	102 ft3
KILN SLOPE =	0.188 ft/ft
KILN ROTATIONAL VELOCITY =	1.70 rpm
SCC LENGTH =	4.67 feet
SCC WIDTH =	4.67 feet
SCC HEIGHT =	15.00 feet
SCC INSIDE VOLUME =	327 ft3
COMBUSTION INLET AIR CONDITIONS	
PRIMARY AIR TEMPERATURE =	50 degF
PRIMARY AIR REL HUMIDITY =	70 %
SECONDARY AIR TEMPERATURE =	50 degF
SECONDARY AIR REL HUMIDITY =	70 %
INCINERATOR OPERATING CONDITIONS	
WASTE FEED TEMPERATURES =	50 degF
KILN OPERATING TEMPERATURE =	400 degF
SCC OPERATING TEMPERATURE =	1,200 degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	800 degF
LOW TEMP GAS COOLER OPERATING CONDITIONS	
FLUE GAS OUTLET TEMPERATURE =	240 degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES	
PARTICULATE REMOVED AS ASH IN KILN =	0.0 %
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0 %
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0 %
PARTICULATE REMOVAL IN CYCLONE =	30.0 %
PARTICULATE REMOVAL IN BAGHOUSE =	95.0 %
STACK GAS CONDITIONS	
STACK GAS TEMPERATURE =	240 degF

CLIENT : SENECA ARMY DEPOT
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ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	33.31 lbs/hr	
AUXILIARY FUEL FEED =	61.00 lbs/hr	173.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	984 lbs/hr	2,473 lbs/hr
COMBUSTION AIR REQUIRED =	2,536 scfm	565 scfm
	12,290 lbs/hr	2,738 lbs/hr
EXCESS AIR REQUIRED =	1,142 %	11 %
FLUE GAS PRODUCED =	12,368 lbs/hr	15,278 lbs/hr
	429 lbmol/hr	530 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	4.7 minutes	
MAX GAS RESIDENCE TIME =	1.35 seconds	1.83 seconds
FLUE GAS VELOCITY =	14.8 ft/sec	8.2 ft/sec

ENERGY BALANCES		
HEAT RELEASE RATE =	1,247,886 Btu/hr	3,361,390 Btu/hr
OPERATING TEMPERATURE =	400 degF	1,200 degF
RADIATION HEAT LOSSES =	2,664 Btu/hr	11,135 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	62,656 Btu/hr	
AUX FUEL HEAT INPUT =	1,185,230 Btu/hr	3,361,390 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,245,222 Btu/hr	4,595,478 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,294 Btu/hr/ft3	14,082 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.40 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	5.90 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	13.08 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	19.39 lbs/hr

ENERGY BALANCES	
HEAT REMOVED BY HI TEMP COOLER =	1,711,122 Btu/hr
COOLING AIR TEMP OUT =	103 degF
HEAT REMOVED BY LO TEMP COOLER =	2,395,570 Btu/hr
COOLING AIR TEMP OUT =	166 degF

CLIENT : SENECA ARMY DEPOT
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PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER	EMISSIONS AFTER HI TEMP GAS COOLER
FLUE GAS FLOWRATE :		
SCFM :	3,169 scfm	3,169 scfm
ACFM :	10,712 acfm	8,130 acfm
LBMOL/HR :	530 lbmol/hr	530 lbmol/hr
LBS/HR :	15,278 lbs/hr	15,278 lbs/hr
LBS/HR DRY :	14,936 lbs/hr	14,936 lbs/hr
FLUE GAS TEMPERATURE :	1,200 degF	800 degF
FLUE GAS SATURATION TEMP :	161 degF	148 degF
FLUE GAS ACTUAL HUMIDITY :	0.0229	0.0229
FLUE GAS SAT HUMIDITY :	0.3027	0.1921
FLUE GAS COMPOSITION :		
N2 :	77.31 %	77.31 %
O2 :	15.79 %	15.79 %
CO2 :	3.25 %	3.25 %
H2O :	3.59 %	3.59 %
EXCESS AIR :	332 %	332 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.36 lbs/hr	2.36 lbs/hr
CONCENTRATION :	70 ppmv	70 ppmv
NOx :		
FLOWRATE :	13.32 lbs/hr	13.32 lbs/hr
CONCENTRATION :	547 ppmv	547 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
PARTICULATES :		
FLOWRATE :	20.08 lbs/hr	19.67 lbs/hr
CONCENTRATION :	0.77 gr/dscf	0.75 gr/dscf

CLIENT : SENECA ARMY DEPOT
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DATE : 02-11-1993

PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	3,169 scfm	3,169 scfm
ACFM :	4,517 acfm	4,515 acfm
LBMOL/HR :	530 lbmol/hr	530 lbmol/hr
LBS/HR :	15,278 lbs/hr	15,272 lbs/hr
LBS/HR DRY :	14,936 lbs/hr	14,936 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	108 degF	108 degF
FLUE GAS ACTUAL HUMIDITY :	0.0229	0.0229
FLUE GAS SAT HUMIDITY :	0.0549	0.0549
FLUE GAS COMPOSITION :		
N2 :	77.31 %	77.31 %
O2 :	15.79 %	15.79 %
CO2 :	3.25 %	3.25 %
H2O :	3.59 %	3.59 %
EXCESS AIR :	332 %	332 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.36 lbs/hr	2.36 lbs/hr
CONCENTRATION :	70 ppmv	70 ppmv
NOx :		
FLOWRATE :	13.32 lbs/hr	13.32 lbs/hr
CONCENTRATION :	547 ppmv	547 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
PARTICULATES :		
FLOWRATE :	19.67 lbs/hr	13.77 lbs/hr
CONCENTRATION :	0.7512 gr/dscf	0.5258 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
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PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	3,169 scfm	3,169 scfm
ACFM :	4,511 acfm	4,511 acfm
LBMOL/HR :	530 lbmol/hr	530 lbmol/hr
LBS/HR :	15,259 lbs/hr	15,259 lbs/hr
LBS/HR DRY :	14,936 lbs/hr	14,936 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	108 degF	108 degF
FLUE GAS ACTUAL HUMIDITY :	0.0229	0.0229
FLUE GAS SAT HUMIDITY :	0.0549	0.0549
FLUE GAS COMPOSITION :		
N2 :	77.31 %	77.31 %
O2 :	15.79 %	15.79 %
CO2 :	3.25 %	3.25 %
H2O :	3.59 %	3.59 %
EXCESS AIR :	332 %	332 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	0.43 lbs/hr	0.43 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.36 lbs/hr	2.36 lbs/hr
CONCENTRATION :	70 ppmv	70 ppmv
NOx :		
FLOWRATE :	13.32 lbs/hr	13.32 lbs/hr
CONCENTRATION :	547 ppmv	547 ppmv
CO :		
FLOWRATE :	0.32 lbs/hr	0.32 lbs/hr
CONCENTRATION :	22 ppmv	22 ppmv
PARTICULATES :		
FLOWRATE :	0.69 lbs/hr	0.69 lbs/hr
CONCENTRATION :	0.0263 gr/dscf	0.0263 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0797 gr/dscf

CLIENT : SENECA ARMY DEPOT
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ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	78 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	342 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	342 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	20.08 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	20.08 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	20.08 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	20.08 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		14 degF
CRITICAL HUMIDITY =		0.0022 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00009 X T 0.00092

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 9
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

FEED COMPOSITION TO KILN	AUX FUEL	SOLID WASTE
WEIGHT % C =	87.20 %	25.03 %
WEIGHT % H =	12.50 %	2.52 %
WEIGHT % O =	0.00 %	51.98 %
WEIGHT % N =	0.00 %	12.50 %
WEIGHT % S =	0.30 %	0.15 %
WEIGHT % Cl =	0.00 %	1.23 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	35,568 ppm
PPM HEAVY METALS =	0 ppm	30,465 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	100.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	5,112 Btu/lb
FEED RATE TO KILN =	11.00 lbs/hr	204.08 lbs/hr

FEED COMPOSITION TO SCC	AUX FUEL	LIQUID WASTE
WEIGHT % C =	87.20 %	0.00 %
WEIGHT % H =	12.50 %	0.00 %
WEIGHT % O =	0.00 %	0.00 %
WEIGHT % N =	0.00 %	0.00 %
WEIGHT % S =	0.30 %	0.00 %
WEIGHT % Cl =	0.00 %	0.00 %
WEIGHT % F =	0.00 %	0.00 %
WEIGHT % Br =	0.00 %	0.00 %
WEIGHT % P =	0.00 %	0.00 %
PPM Si =	0 ppm	0 ppm
PPM Na K B Ca Mg =	0 ppm	0 ppm
PPM HEAVY METALS =	0 ppm	0 ppm
WEIGHT % COMBUSTIBLES =	100.00 %	0.00 %
WEIGHT % WATER =	0.00 %	0.00 %
WEIGHT % INERTS =	0.00 %	0.00 %
HIGHER HEATING VALUE =	19,430 Btu/lb	0 Btu/lb
FEED RATE TO SCC =	353.00 lbs/hr	0.00 lbs/hr

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 9
DATE : 02-11-1993

ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

DESIGN CRITERIA

INCINERATOR DIMENSIONS		
KILN INSIDE DIAMETER =	2.54	feet
KILN LENGTH =	20.00	feet
KILN INSIDE VOLUME =	102	ft3
KILN SLOPE =	0.188	ft/ft
KILN ROTATIONAL VELOCITY =	1.50	rpm
SCC LENGTH =	4.67	feet
SCC WIDTH =	4.67	feet
SCC HEIGHT =	15.00	feet
SCC INSIDE VOLUME =	327	ft3
COMBUSTION INLET AIR CONDITIONS		
PRIMARY AIR TEMPERATURE =	50	degF
PRIMARY AIR REL HUMIDITY =	70	%
SECONDARY AIR TEMPERATURE =	50	degF
SECONDARY AIR REL HUMIDITY =	70	%
INCINERATOR OPERATING CONDITIONS		
WASTE FEED TEMPERATURES =	50	degF
KILN OPERATING TEMPERATURE =	350	degF
SCC OPERATING TEMPERATURE =	1,600	degF
HIGH TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	800	degF
LOW TEMP GAS COOLER OPERATING CONDITIONS		
FLUE GAS OUTLET TEMPERATURE =	240	degF
AIR POLLUTION CONTROL REMOVAL EFFICIENCIES		
PARTICULATE REMOVED AS ASH IN KILN =	0.0	%
PARTICULATE REMOVAL IN HIGH TEMP COOLER =	2.0	%
PARTICULATE REMOVAL IN LOW TEMP COOLER =	0.0	%
PARTICULATE REMOVAL IN CYCLONE =	30.0	%
PARTICULATE REMOVAL IN BAGHOUSE =	95.0	%
STACK GAS CONDITIONS		
STACK GAS TEMPERATURE =	240	degF

CLIENT : SENECA ARMY DEPOT
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ROTARY KILN INCINERATOR
MATERIAL AND ENERGY BALANCES
100 % LOAD

MATERIAL BALANCES	KILN	SCC
LIQUID WASTE FEED =		0.00 lbs/hr
SOLID WASTE FEED =	204.08 lbs/hr	
AUXILIARY FUEL FEED =	11.00 lbs/hr	353.00 lbs/hr
STOICHIOMETRIC AIR REQ'D =	732 lbs/hr	5,047 lbs/hr
COMBUSTION AIR REQUIRED =	2,883 scfm	1,142 scfm
	13,974 lbs/hr	5,533 lbs/hr
EXCESS AIR REQUIRED =	1,798 %	10 %
FLUE GAS PRODUCED =	14,148 lbs/hr	20,034 lbs/hr
	490 lbmol/hr	693 lbmol/hr
BOTTOM ASH REMOVED =	0 lbs/hr	
KILN SOLIDS RESIDENCE TIME =	5.3 minutes	
MAX GAS RESIDENCE TIME =	1.26 seconds	1.13 seconds
FLUE GAS VELOCITY =	15.9 ft/sec	13.3 ft/sec

ENERGY BALANCES		
HEAT RELEASE RATE =	1,256,987 Btu/hr	6,858,790 Btu/hr
OPERATING TEMPERATURE =	350 degF	1,600 degF
RADIATION HEAT LOSSES =	2,526 Btu/hr	15,377 Btu/hr
ASH REMOVAL HEAT LOSS =	0 Btu/hr	
SOLID WASTE HEAT INPUT =	1,043,257 Btu/hr	
AUX FUEL HEAT INPUT =	213,730 Btu/hr	6,858,790 Btu/hr
LIQUID WASTE HEAT INPUT =		0 Btu/hr
FLUE GAS ENTHALPY =	1,254,461 Btu/hr	8,097,874 Btu/hr
VOLUMETRIC HEAT RELEASE =	12,384 Btu/hr/ft3	24,801 Btu/hr/ft3

MATERIAL BALANCES	OFF-GAS CLEANING SYSTEM
COOLING AIR FOR HI TEMP GAS COOLER =	118,764 lbs/hr
=	23,639 scfm
COOLING AIR FOR LO TEMP GAS COOLER =	76,682 lbs/hr
=	15,263 scfm
DRY SOLIDS FROM HI TEMP COOLER =	0.38 lbs/hr
DRY SOLIDS FROM LO TEMP COOLER =	0.00 lbs/hr
DRY SOLIDS REMOVED IN CYCLONE =	5.60 lbs/hr
DRY SOLIDS REMOVED IN BAGHOUSE =	12.42 lbs/hr
TOTAL DRY SOLIDS REMOVED IN APCS =	18.41 lbs/hr

ENERGY BALANCES	
HEAT REMOVED BY HI TEMP COOLER =	4,487,435 Btu/hr
COOLING AIR TEMP OUT =	190 degF
HEAT REMOVED BY LO TEMP COOLER =	3,141,205 Btu/hr
COOLING AIR TEMP OUT =	202 degF

CLIENT : SENECA ARMY DEPOT
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PARAMETER	EMISSIONS AFTER SEC. COMB. CHAMBER	EMISSIONS AFTER HI TEMP GAS COOLER
FLUE GAS FLOWRATE :		
SCFM :	4,149 scfm	4,149 scfm
ACFM :	17,395 acfm	10,640 acfm
LBMOL/HR :	693 lbmol/hr	693 lbmol/hr
LBS/HR :	20,034 lbs/hr	20,033 lbs/hr
LBS/HR DRY :	19,477 lbs/hr	19,477 lbs/hr
FLUE GAS TEMPERATURE :	1,600 degF	800 degF
FLUE GAS SATURATION TEMP :	171 degF	149 degF
FLUE GAS ACTUAL HUMIDITY :	0.0286	0.0286
FLUE GAS SAT HUMIDITY :	0.4314	0.1994
FLUE GAS COMPOSITION :		
N2 :	76.66 %	76.66 %
O2 :	14.30 %	14.30 %
CO2 :	4.43 %	4.43 %
H2O :	4.46 %	4.46 %
EXCESS AIR :	236 %	236 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	2.59 lbs/hr	2.59 lbs/hr
CONCENTRATION :	102 ppmv	102 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.79 lbs/hr	2.79 lbs/hr
CONCENTRATION :	63 ppmv	63 ppmv
NOx :		
FLOWRATE :	42.51 lbs/hr	42.51 lbs/hr
CONCENTRATION :	1,333 ppmv	1,333 ppmv
CO :		
FLOWRATE :	0.54 lbs/hr	0.54 lbs/hr
CONCENTRATION :	28 ppmv	28 ppmv
PARTICULATES :		
FLOWRATE :	19.06 lbs/hr	18.68 lbs/hr
CONCENTRATION :	0.56 gr/dscf	0.55 gr/dscf

CLIENT : SENECA ARMY DEPOT
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PARAMETER	EMISSIONS AFTER LO TEMP GAS COOLER	EMISSIONS AFTER CYCLONE
FLUE GAS FLOWRATE :		
SCFM :	4,149 scfm	4,149 scfm
ACFM :	5,911 acfm	5,909 acfm
LBMOL/HR :	693 lbmol/hr	693 lbmol/hr
LBS/HR :	20,033 lbs/hr	20,028 lbs/hr
LBS/HR DRY :	19,477 lbs/hr	19,477 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	111 degF	111 degF
FLUE GAS ACTUAL HUMIDITY :	0.0286	0.0286
FLUE GAS SAT HUMIDITY :	0.0603	0.0603
FLUE GAS COMPOSITION :		
N2 :	76.66 %	76.66 %
O2 :	14.30 %	14.30 %
CO2 :	4.43 %	4.43 %
H2O :	4.46 %	4.46 %
EXCESS AIR :	236 %	236 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	2.59 lbs/hr	2.59 lbs/hr
CONCENTRATION :	102 ppmv	102 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.79 lbs/hr	2.79 lbs/hr
CONCENTRATION :	63 ppmv	63 ppmv
NOx :		
FLOWRATE :	42.51 lbs/hr	42.51 lbs/hr
CONCENTRATION :	1,333 ppmv	1,333 ppmv
CO :		
FLOWRATE :	0.54 lbs/hr	0.54 lbs/hr
CONCENTRATION :	28 ppmv	28 ppmv
PARTICULATES :		
FLOWRATE :	18.68 lbs/hr	13.08 lbs/hr
CONCENTRATION :	0.5498 gr/dscf	0.3849 gr/dscf

CLIENT : SENECA ARMY DEPOT
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PARAMETER	EMISSIONS AFTER BAGHOUSE	EMISSIONS AT STACK
FLUE GAS FLOWRATE :		
SCFM :	4,149 scfm	4,149 scfm
ACFM :	5,906 acfm	5,906 acfm
LBMOL/HR :	693 lbmol/hr	693 lbmol/hr
LBS/HR :	20,015 lbs/hr	20,015 lbs/hr
LBS/HR DRY :	19,477 lbs/hr	19,477 lbs/hr
FLUE GAS TEMPERATURE :	240 degF	240 degF
FLUE GAS SATURATION TEMP :	111 degF	111 degF
FLUE GAS ACTUAL HUMIDITY :	0.0286	0.0286
FLUE GAS SAT HUMIDITY :	0.0603	0.0603
FLUE GAS COMPOSITION :		
N2 :	76.66 %	76.66 %
O2 :	14.30 %	14.30 %
CO2 :	4.43 %	4.43 %
H2O :	4.46 %	4.46 %
EXCESS AIR :	236 %	236 %
FLUE GAS CONTAMINANTS :		
HCl :		
FLOWRATE :	2.59 lbs/hr	2.59 lbs/hr
CONCENTRATION :	102 ppmv	102 ppmv
HF :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
Br2 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
P2O5 :		
FLOWRATE :	0.00 lbs/hr	0.00 lbs/hr
CONCENTRATION :	0 ppmv	0 ppmv
SO2 :		
FLOWRATE :	2.79 lbs/hr	2.79 lbs/hr
CONCENTRATION :	63 ppmv	63 ppmv
NOx :		
FLOWRATE :	42.51 lbs/hr	42.51 lbs/hr
CONCENTRATION :	1,333 ppmv	1,333 ppmv
CO :		
FLOWRATE :	0.54 lbs/hr	0.54 lbs/hr
CONCENTRATION :	28 ppmv	28 ppmv
PARTICULATES :		
FLOWRATE :	0.65 lbs/hr	0.65 lbs/hr
CONCENTRATION :	0.0192 gr/dscf	0.0192 gr/dscf

Note: Particulate concentration leaving stack, corrected to 7% oxygen on a dry basis in the stack gas is 0.0447 gr/dscf

CLIENT : SENECA ARMY DEPOT
JOB NO. : 720229-09000
SUBJECT : TRIAL BURN PLAN - TEST NO. 9
DATE : 02-11-1993

ADDITIONAL MATERIAL BALANCE INFORMATION

WATER ENTERING IN ALL FEED STREAMS =	0 lbs/hr
WATER ENTERING IN COMBUSTION AIR =	102 lbs/hr
WATER IN FLUE GAS LEAVING SCC =	556 lbs/hr
WATER IN FLUE GAS LEAVING STACK =	556 lbs/hr
INERTS ENTERING KILN IN WASTE FEEDS =	0.00 lbs/hr
ASH FORMED IN KILN =	19.06 lbs/hr
TOTAL PARTICULATES FORMED IN KILN =	19.06 lbs/hr
TOTAL ASH REMOVED FROM KILN =	0.00 lbs/hr
TOTAL PARTICULATE LEAVING KILN =	19.06 lbs/hr
TOTAL PARTICULATE FORMED IN SCC =	0.00 lbs/hr
TOTAL PARTICULATES LEAVING SCC =	19.06 lbs/hr

PLUME FORMATION CONDITIONS AT STACK

CRITICAL TEMPERATURE =		20 degF
CRITICAL HUMIDITY =		0.0028 lbs H2O/lb dry air
CRITICAL EQUATION =	H =	0.00012 X T 0.00045

APPENDIX E

STACK GAS SAMPLING METHODS

APPENDIX E

STACK GAS SAMPLING METHODS

This appendix contains the sampling methods for metals, volatile organic compounds, semivolatile organic compounds, and energetic compounds. The energetic compound sampling method is the AEHA STEM Method. The text for this sampling procedure was pulled from the QA/QC plan for the Tooele Army Depot.

All other source test methods to be used during this trial burn are EPA Reference Methods. The EPA Reference Methods are presented in full in 40 CFR Part 60, Appendix A.

E-1 PARTICULATE MATTER SAMPLING TRAIN

Methodology for the Determination of Metals Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes.

EPA Methods Manual for Compliance with the BIF Regulations EPA/530-SW-91-010.

Section 3.0

SAMPLING AND ANALYTICAL METHODS

3.1 Methodology for the Determination of Metals Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes

3.1.1 Applicability and Principle

3.1.1.1 Applicability. This method is being developed for the determination of total chromium (Cr), cadmium (Cd), arsenic (As), nickel (Ni), manganese (Mn), beryllium (Be), copper (Cu), zinc (Zn), lead (Pb), selenium (Se), phosphorus (P), thallium (Tl), silver (Ag), antimony (Sb), barium (Ba), and mercury (Hg) stack emissions from hazardous waste incinerators and similar combustion processes. This method may also be used for the determination of particulate emissions following the procedures and precautions described. Modifications to the sample recovery and analysis procedures described in this protocol for the purpose of determining particulate emissions may potentially impact the front-half mercury determination. Mercury emissions should be determined using EPA Method 101A given in 40 CFR Part 61.

3.1.1.2 Principle. The stack sample is withdrawn isokinetically from the source, with particulate emissions collected in the probe and on a heated filter and gaseous emissions collected in a series of chilled impingers containing an aqueous solution of dilute nitric acid combined with dilute hydrogen peroxide in each of two impingers, and acidic potassium permanganate solution in each of two impingers. Sampling train components are recovered and digested in separate front- and back-half fractions. Materials collected in the sampling train are digested with acid solutions to dissolve organics and to remove organic constituents that may create analytical interferences. Acid digestion is performed using conventional Parr® Bomb or microwave digestion techniques. The nitric acid and hydrogen peroxide impinger solution, the acidic potassium permanganate impinger solution, the HCl rinse solution, and the probe rinse and digested filter solutions are analyzed for mercury by cold vapor atomic absorption spectroscopy (CVAAS). The nitric acid

and hydrogen peroxide solution and the probe rinse and digested filter solutions of the train catches are analyzed for Cr, Cd, Ni, Mn, Be, Cu, Zn, Pb, Se, P, Tl, Ag, Sb, Ba, and As by inductively coupled argon plasma emission spectroscopy (ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of antimony, arsenic, cadmium, lead, selenium, and thallium, if these elements require greater analytical sensitivity than can be obtained by ICAP. Additionally, if desired, the tester may use AAS for analyses of all metals if the resulting in-stack method detection limits meet the goal of the testing program. For convenience, aliquots of each digested sample Fraction 1A plus Fraction 2A can be combined proportionally with respect to the original Fraction 1 (normally diluted to 300 ml following digestion and prior to analysis) Section 3.1.5.3.3; and concentrated Fraction 2A (normally diluted to 150 ml following digestion and prior to analysis) Section 3.1.5.3.4.1 or 3.1.5.3.4.2 for a single analytical determination. The efficiency of the analytical procedure is quantified by the analysis of spiked quality control samples containing each of the target metals and/or other quality assurance measures, as necessary, including actual sample matrix effects checks.

3.1.2 Range, Sensitivity, Precision, and Interferences

3.1.2.1 Range. For the analyses described in this methodology and for similar analyses, the ICAP response is linear over several orders of magnitude. Samples containing metal concentrations in the nanograms per milliliter (ng/ml) to micrograms per milliliter (μg/ml) range in the analytical finish solution can be analyzed using this technique. Samples containing greater than approximately 50 μg/ml of chromium, lead, or arsenic should be diluted to that level or lower for final analysis. Samples containing greater than approximately 20 μg/ml of cadmium should be diluted to that level before analysis.

3.1.2.2 Analytical Sensitivity. ICAP analytical detection limits for the sample solutions (based on SW-846, Method 6010) are approximately as follows: Sb (32 ng/ml), As (53 ng/ml), Ba (2 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Cu (6 ng/ml), Pb (42 ng/ml), Mn (2 ng/ml), Ni (15

ng/ml), P (75 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), Tl (40 ng/ml), and Zn (2 ng/ml). The actual method detection limits are sample dependent and may vary as the sample matrix may affect the limits. The analytical detection limits for analysis by direct aspiration AAS (based on SW-846, Method 7000 series) are approximately as follows: Sb (200 ng/ml), As (2 ng/ml), Ba (100 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Cu (20 ng/ml), Pb (100 ng/ml), Mn (10 ng/ml), Ni (40 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml), and Zn (5 ng/ml). The detection limit for mercury by CVAAS is approximately 0.2 ng/ml). The use of GFAAS can give added sensitivity compared to the use of direct aspiration AAS for the following metals: Sb (3 ng/ml), As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Pb (1 ng/ml), Se (2 ng/ml), and Tl (1 ng/ml).

Using (1) the procedures described in this method, (2) the analytical detection limits described in the previous paragraph, (3) a volume of 300 ml, Fraction 1, for the front half and 150 ml, Fraction 2A, for the back-half samples, and (4) a stack gas sample volume of 1.25 m^3, the corresponding in-stack method detection limits are presented in Table A-1 and calculated as shown:

$$\frac{A \times B}{C} = D$$

where: A = analytical detection limit, $\mu g/ml$.
B = volume of sample prior to aliquot for analysis, ml.
C = stack sample volume, dscm (dsm^3).
D = in-stack detection limit, $\mu g/m^3$.

Values in Table 3.1-1 are calculated for the front and back half and/or the total train.

To ensure optimum sensitivity in obtaining the measurements, the concentrations of target metals in the solutions are suggested to be at least ten times the analytical detection limits. Under certain conditions, and with greater care in the analytical procedure, this concentration can be as low as approximately three times the analytical detection limit. In all cases, on at

least one sample (run) in the source test and for each metal analyzed, repetitive analyses, method of standard additions (MSA), serial dilution, or matrix spike addition, etc., shall be used to establish the quality of the data.

Actual in-stack method detection limits will be determined based on actual source sampling parameters and analytical results as described above. If required, the method in-stack detection limits can be made more sensitive than those shown in Table A-1 for a specific test by using one or more of the following options:

- A 1-hour sampling run may collect a stack gas sampling volume of about 1.25 m^3. If the sampling time is increased and 5 m^3 are collected, the in-stack method detection limits would be one fourth of the values shown in Table A-1 (this means that with this change, the method is four times more sensitive than a 1-hour run. Larger sample volumes (longer runs) would make it even more sensitive).

- The in-stack detection limits assume that all of the sample is digested (with exception of the aliquot for mercury) and the final liquid volumes for analysis are 300 ml, Fraction 1 for the front half and 150 ml, Fraction 2A, for the back-half sample. If the front-half volume is reduced from 300 ml to 30 ml, the front-half in-stack detection limits would be one tenth of the values shown above (ten times more sensitive). If the back-half volume is reduced from 150 ml to 25 ml, the in-stack detection limits would be one sixth of the above values. Matrix effects checks are necessary on analyses of samples and typically are of greater significance for samples that have been concentrated to less than the normal original sample volume. Reduction to a volume of less than 25 ml may not allow redissolving of the residue and may increase interference by other compounds.

- When both of the above two improvements are used on one sample at the same time, the resultant improvements are multiplicative. For example, where stack gas volume is increased by a factor of five and the total liquid sample digested volume of both the front and back halves is reduced by a factor of six, the in-stack method detection limit is reduced by a factor of thirty (the method is thirty times more sensitive).

Table 3.1-1

IN-STACK METHOD DETECTION LIMITS (ug/m^3)
FOR TRAIN FRACTIONS USING ICAP AND AAS

Metal	Front-half Fraction 1 Probe and Filter	Back-half Fraction 2 Impingers 1-3	Back-half Fractions "Hg, only" Impingers 4-6	Total Train
Antimony	7.7 (0.7)*	3.8 (0.4)*		11.5 (1.1)*
Arsenic	12.7 (0.3)*	6.4 (0.1)*		19.1 (0.4)*
Barium	0.5	0.3		0.8
Beryllium	0.07 (0.05)*	0.04 (0.03)*		0.11 (0.08)*
Cadmium	1.0 (0.02)*	0.5 (0.01)*		1.5 (0.03)*
Chromium	1.7 (0.2)*	0.8 (0.1)*		2.5 (0.3)*
Copper	1.4	0.7		2.1
Lead	10.1 (0.2)*	5.0 (0.1)*		15.1 (0.3)*
Manganese	0.5 (0.2)*	0.2 (0.1)*		0.7 (0.3*)
Mercury	0.6**	3.0**	2.0**	5.6**
Nickel	3.6	1.8		5.4
Phosphorus	18	9		27
Selenium	18 (0.5)*	9 (0.3)*		27 (0.8)*
Silver	1.7	0.9		2.6
Thallium	9.6 (0.2)*	4.8 (0.1)*		14.4 (0.3)*
Zinc	0.5	0.3		0.8

()* Detection limit when analyzed by GFAAS.
** Detection limit when analyzed by CVAAS, estimated for Back Half and Total Train.
Note: Actual method in-stack detection limits will be determined based on actual source sampling parameters and analytical results as described earlier in this section.

- Conversely, reducing stack gas sample volume and increasing sample liquid volume will increase in-stack detection limits (the method would then be less sensitive). The front-half and back-half samples (Fractions 1A plus and 2A) can be combined proportionally (see Section 3.1.1.2 of this methodology) prior to analysis. The resultant liquid volume (excluding the mercury fractions, which must be analyzed separately) is recorded. Combining the sample as described does not allow determination (whether front or back half) of where in the train the sample was captured. The in-stack method detection limit then becomes a single value for all metals except mercury, for which the contribution of the mercury fractions must be considered.

- The above discussion assumes no blank correction. Blank corrections are discussed later in this method.

3.1.2.3 Precision. The precisions (relative standard deviation) for each metal detected in a method development test at a sewage sludge incinerator, are as follows: Sb (12.7%), As (13.5%), Ba (20.6%), Cd (11.5%), Cr (11.2%), Cu (11.5%), Pb (11.6%), P (14.6%), Se (15.3%), Tl (12.3%), and Zn (11.8%). The precision for nickel was 7.7% for another test conducted at a source simulator. Beryllium, manganese, and silver were not detected in the tests; however, based on the analytical sensitivity of the ICAP for these metals, it is assumed that their precisions should be similar to those for the other metals, when detected at similar levels.

3.1.2.4 Interferences. Iron can be a spectral interference during the analysis of arsenic, chromium, and cadmium by ICAP. Aluminum can be a spectral interference during the analysis of arsenic and lead by ICAP. Generally, these interferences can be reduced by diluting the sample, but this increases the method detection limit (in-stack detection limit). Refer to EPA Method 6010 (SW-846) or the other analytical methods used for details on potential interferences for this method. The analyst must eliminate or reduce interferences to acceptable levels. For all GFAAS analyses, matrix modifiers should be used to limit interferences, and standards should be matrix matched.

3.1.3 Apparatus

3.1.3.1 Sampling Train. A schematic of the sampling train is shown in Figure 3.1-1. It is similar to the 40 CFR Part 60, Appendix A Method 5 train. The sampling train consists of the following components:

3.1.3.1.1 Probe Nozzle (Probe Tip) and Borosilicate or Quartz Glass Probe Liner. Same as Method 5, Sections 2.1.1 and 2.1.2, except that glass nozzles are required unless an alternate probe tip prevents the possibility of contamination or interference of the sample with its materials of construction. If a probe tip other than glass is used, no correction (because of any effect on the sample by the probe tip) of the stack sample test results can be made.

3.1.3.1.2 Pitot Tube and Differential Pressure Gauge. Same as Method 2, Sections 2.1 and 2.2, respectively.

3.1.3.1.3 Filter Holder. Glass, same as Method 5, Section 2.1.5, except that a Teflon filter support or other non-metallic, non-contaminating support must be used to replace the glass frit.

3.1.3.1.4 Filter Heating System. Same as Method 5, Section 2.1.6.

3.1.3.1.5 Condenser. The following system shall be used for the condensation and collection of gaseous metals and for determining the moisture content of the stack gas. The condensing system should consist of four to seven impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first impinger is optional and is recommended as a moisture knockout trap for use during test conditions which require such a trap. The first impinger shall be appropriately-sized, if necessary, for an expected large moisture catch and generally constructed as described for the first impinger in Method 5, Paragraph 2.1.7. The second impinger (or the first HNO_3/H_2O_2 impinger) shall also be constructed as described for the first impinger in Method 5. The third impinger (or the second HNO_3/H_2O_2 impinger) shall be the same as the Greenburg Smith impinger

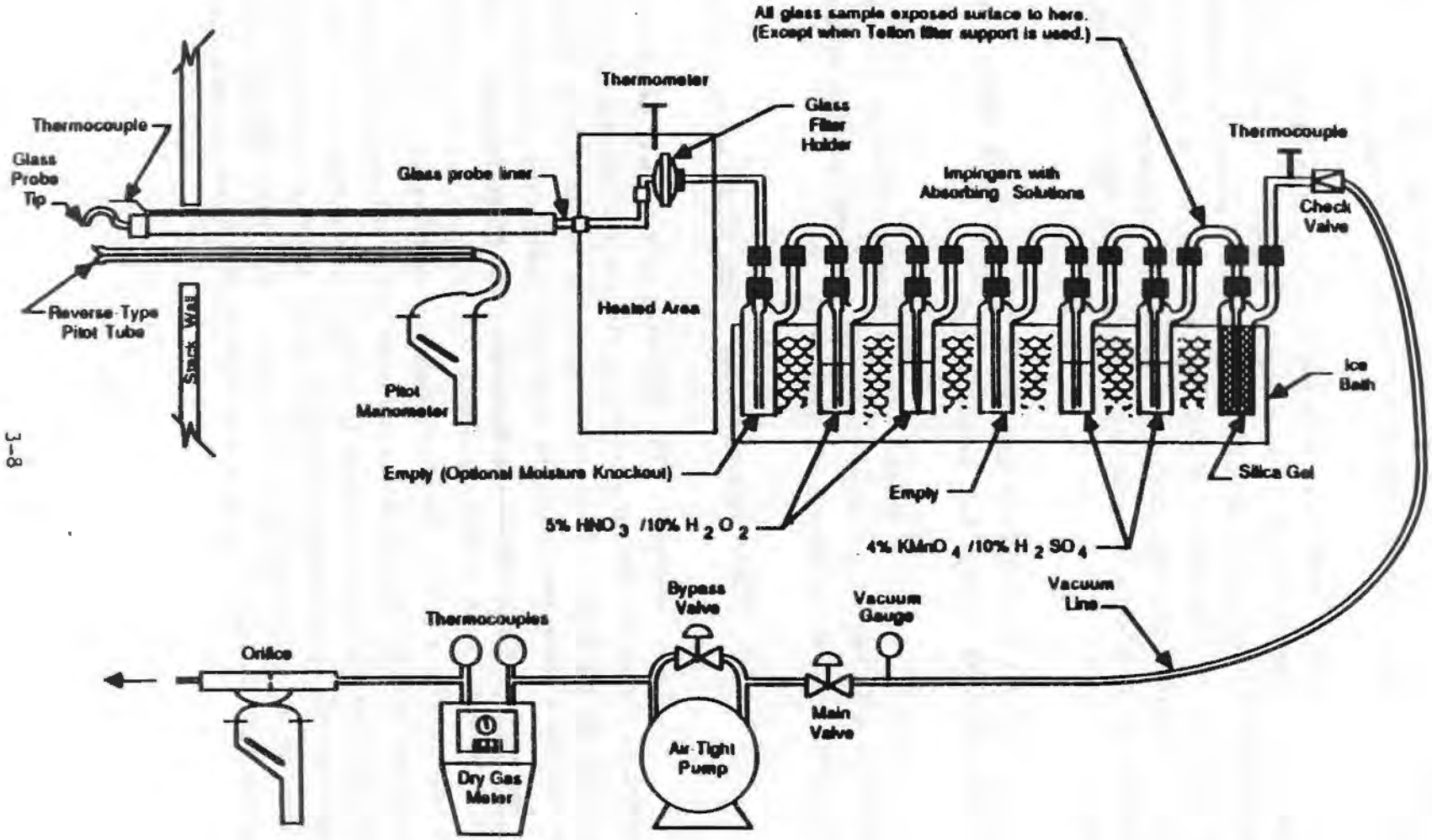

Figure 3.1-1 Schematic of multiple metals sampling train configuration.

with the standard tip described as the second impinger in Method 5, Paragraph 2.1.7. All other impingers used in the methods train are the same as the first HNO_3/H_2O_2 impinger described in this paragraph. In summary, the first impinger which may be optional as described in this methodology shall be empty, the second and third shall contain known quantities of a nitric acid/hydrogen peroxide solution (Section 3.1.4.2.1), the fourth shall be empty, the fifth and sixth shall contain a known quantity of acidic potassium permanganate solution (Section 3.1.4.2.2), and the last impinger shall contain a known quantity of silica gel. A thermometer capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger. When the moisture knockout impinger is not needed, it is removed from the train and the other impingers remain the same. If mercury analysis is not to be performed, the potassium permanganate impingers and the empty impinger preceding them are removed.

3.1.3.1.6 Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.8 through 2.1.10, respectively.

3.1.3.1.7 Teflon Tape. For capping openings and sealing connections, if necessary, on the sampling train.

3.1.3.2 Sample Recovery. Same as Method 5, Sections 2.2.1 through 2.2.8 (Nonmetallic Probe-Liner and Probe-Nozzle Brushes or Swabs, Wash Bottles, Sample Storage Containers, Petri Dishes, Glass Graduated Cylinder, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel), respectively, with the following exceptions and additions:

3.1.3.2.1 Nonmetallic Probe-Liner and Probe-Nozzle Brushes or Swabs. For quantitative recovery of materials collected in the front half of the sampling train. Description of acceptable all-Teflon component brushes or swabs is to be included in EPA's Emission Measurement Technical Information Center (EMTIC) files.

3.1.3.2.2 Sample Storage Containers. Glass bottles with Teflon-lined caps which are non-reactive to the oxidizing solutions, with a capacity

of 1000- and 500-ml, shall be used for $KMnO_4$-containing samples and blanks. Polyethylene bottles may be used for other sample types.

3.1.3.2.3 Graduated Cylinder. Glass or equivalent.

3.1.3.2.4 Funnel. Glass or equivalent.

3.1.3.2.5 Labels. For identification of samples.

3.1.3.2.6 Polypropylene Tweezers and/or Plastic Gloves. For recovery of the filter from the sampling train filter holder.

3.1.3.3 Sample Preparation and Analysis. For the analysis, the following equipment is needed:

3.1.3.3.1 Volumetric Flasks, 100-ml, 250-ml, and 1000-ml. For preparation of standards and sample dilution.

3.1.3.3.2 Graduated Cylinders. For preparation of reagents.

3.1.3.3.3 Parr® Bombs or Microwave Pressure Relief Vessels with Capping Station (CEM Corporation model or equivalent).

3.1.3.3.4 Beakers and Watchglasses. 250-ml beakers for sample digestion with watchglasses to cover the tops.

3.1.3.3.5 Ring Stands and Clamps. For securing equipment such as filtration apparatus.

3.1.3.3.6 Filter Funnels. For holding filter paper.

3.1.3.3.7 Whatman 541 Filter Paper (or equivalent). For filtration of digested samples.

3.1.3.3.8 Disposable Pasteur Pipets and Bulbs.

3.1.3.3.9 Volumetric Pipets.

3.1.3.3.10 Analytical Balance. Accurate to within 0.1 mg.

3.1.3.3.11 Microwave or Conventional Oven. For heating samples at fixed power levels or temperatures.

3.1.3.3.12 Hot Plates.

3.1.3.3.13 Atomic Absorption Spectrometer (AAS). Equipped with a background corrector.

3.1.3.3.13.1 Graphite Furnace Attachment. With antimony, arsenic, cadmium, lead, selenium, thallium hollow cathode lamps (HCLs) or electrodeless discharge lamps (EDLs). [Same as EPA SW-846 Methods 7041 (antimony), 7060 (arsenic), 7131 (cadmium), 7421 (lead), 7740 (selenium), and 7841 (thallium).]

3.1.3.3.13.2 Cold Vapor Mercury Attachment. With a mercury HCL or EDL. The equipment needed for the cold vapor mercury attachment includes an air recirculation pump, a quartz cell, an aerator apparatus, and a heat lamp or desiccator tube. The heat lamp should be capable of raising the ambient temperature at the quartz cell by 10°C such that no condensation forms on the wall of the quartz cell. (Same as EPA Method 7470.)

3.1.3.3.14 Inductively Coupled Argon Plasma Spectrometer. With either a direct or sequential reader and an alumina torch. (Same as EPA Method 6010.)

3.1.4 Reagents

The complexity of this methodology is such that to obtain reliable results, the testers (including analysts) should be experienced and knowledgeable in source sampling, in handling and preparing (including mixing) reagents as described, and using adequate safety procedures and protective equipment in performing this method, including sampling, mixing reagents, digestions, and

analyses. Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1.4.1 Sampling. The reagents used in sampling are as follows:

3.1.4.1.1 Filters. The filters shall contain less than 1.3 $\mu g/in^2$ of each of the metals to be measured. Analytical results provided by filter manufacturers are acceptable. However, if no such results are available, filter blanks must be analyzed for each target metal prior to emission testing. Quartz fiber or glass fiber (which meet the requirement of containing less than 1.3 $\mu g/in^2$ of each metal) filters without organic binders shall be used. The filters should exhibit at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2986-71 (incorporated by reference). For particulate determination in sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3, as described in EPA Method 5. Quartz fiber filters meeting these requirements are recommended for use in this method.

3.1.4.1.2 Water. To conform to ASTM Specification D1193.77, Type II (incorporated by reference). If necessary, analyze the water for all target metals prior to field use. All target metal concentrations should be less than 1 ng/ml.

3.1.4.1.3 Nitric Acid. Concentrated. Baker Instra-analyzed or equivalent.

3.1.4.1.4 Hydrochloric Acid. Concentrated. Baker Instra-analyzed or equivalent.

3.1.4.1.5 Hydrogen Peroxide, 30 Percent (V/V).

3.1.4.1.6 Potassium Permanganate.

3.1.4.1.7 Sulfuric Acid. Concentrated.

3.1.4.1.8 Silica Gel and Crushed Ice. Same as Method 5, Sections 3.1.2 and 3.1.4, respectively.

3.1.4.2 Pretest Preparation for Sampling Reagents.

3.1.4.2.1 Nitric Acid (HNO_3)/Hydrogen Peroxide (H_2O_2) Absorbing Solution, 5 Percent HNO_3/10 Percent H_2O_2. Carefully with stirring, add 50 ml of concentrated HNO_3 to a 1000-ml volumetric flask containing approximately 500 ml of water, and then, carefully with stirring, add 333 ml of 30 percent H_2O_2. Dilute to volume (1000 ml) with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.2.2 Acidic Potassium Permanganate ($KMnO_4$) Absorbing Solution, 4 Percent $KMnO_4$ (W/V), 10 Percent H_2SO_4 (V/V). Prepare fresh daily. Mix carefully, with stirring, 100 ml of concentrated H_2SO_4 into 800 ml of water, and add water with stirring to make a volume of 1 L: this solution is 10 percent H_2SO_4 (V/V). Dissolve, with stirring, 40 g of $KMnO_4$ into 10 percent H_2SO_4 (V/V) and add 10 percent H_2SO_4 (V/V) with stirring to make a volume of 1 L: this is the acidic potassium permanganate absorbing solution. Prepare and store in glass bottles to prevent degradation. The reagent shall contain less than 2 ng/ml of Hg.

> Precaution: To prevent autocatalytic decomposition of the permanganate solution, filter the solution through Whatman 541 filter paper. Also, due to the potential reaction of the potasaium permanganate with the acid, there may be pressure buildup in the sample storage bottle; these bottles shall not be fully filled and shall be vented both to relieve potential excess pressure and prevent explosion due to pressure buildup. Venting is required, but should not allow contamination of the sample; a No. 70-72 hole drilled in the container cap and Teflon liner has been used.

3.1.4.2.3 Nitric Acid, 0.1 N. With stirring, add 6.3 ml of concentrated HNO_3 (70 percent) to a flask containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.2.4 Hydrochloric Acid (HCl), 8 N. Make the desired volume of 8 N HCl in the following proportions. Carefully with stirring, add 690 ml of concentrated HCl to a flask containing 250 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of Hg.

3.1.4.3 Glassware Cleaning Reagents.

3.1.4.3.1 Nitric Acid, Concentrated. Fisher ACS grade or equivalent.

3.1.4.3.2 Water. To conform to ASTM Specifications D1193-77, Type II.

3.1.4.3.3 Nitric Acid, 10 Percent (V/V). With stirring, add 500 ml of concentrated HNO_3 to a flask containing approximately 4000 ml of water. Dilute to 5000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.4 Sample Digestion and Analysis Reagents.

3.1.4.4.1 Hydrochloric Acid, Concentrated.

3.1.4.4.2 Hydrofluoric Acid, Concentrated.

3.1.4.4.3 Nitric Acid, Concentrated. Baker Instra-analyzed or equivalent.

3.1.4.4.4 Nitric Acid, 50 Percent (V/V). With stirring, add 125 ml of concentrated HNO_3 to 100 ml of water. Dilute to 250 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.4.5 Nitric Acid, 5 Percent (V/V). With stirring, add 50 ml of concentrated HNO_3 to 800 ml of water. Dilute to 1000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

3.1.4.4.6 Water. To conform to ASTM Specifications D1193-77, Type II.

3.1.4.4.7 Hydroxylamine Hydrochloride and Sodium Chloride Solution. See EPA Method 7470 for preparation.

3.1.4.4.8 Stannous Chloride. See Method 7470.

3.1.4.4.9 Potassium Permanganate, 5 Percent (W/V). See Method 7470.

3.1.4.4.10 Sulfuric Acid, Concentrated.

3.1.4.4.11 Nitric Acid, 50 Percent (V/V).

3.1.4.4.12 Potassium Persulfate, 5 Percent (W/V). See Method 7470.

3.1.4.4.13 Nickel Nitrate, $Ni(NO_3)_2 \cdot 6H_2O$.

3.1.4.4.14 Lanthanum, Oxide, La_2O_3.

3.1.4.4.15 AAS Grade Hg Standard, 1000 µg/ml.

3.1.4.4.16 AAS Grade Pb Standard, 1000 µg/ml.

3.1.4.4.17 AAS Grade As Standard, 1000 µg/ml.

3.1.4.4.18 AAS Grade Cd Standard, 1000 µg/ml.

3.1.4.4.19 AAS Grade Cr Standard, 1000 µg/ml.

3.1.4.4.20 AAS Grade Sb Standard, 1000 µg/ml.

3.1.4.4.21 AAS Grade Ba Standard, 1000 µg/ml.

3.1.4.4.22 AAS Grade Be Standard, 1000 µg/ml.

3.1.4.4.23 AAS Grade Cµ Standard, 1000 µg/ml.

3.1.4.4.24 AAS Grade Mn Standard, 1000 µg/ml.

3.1.4.4.25 AAS Grade Ni Standard, 1000 µg/ml.

3.1.4.4.26 AAS Grade P Standard, 1000 µg/ml.

3.1.4.4.27 AAS Grade Se Standard, 1000 µg/ml.

3.1.4.4.28 AAS Grade Ag Standard, 1000 µg/ml.

3.1.4.4.29 AAS Grade Tl Standard, 1000 µg/ml.

3.1.4.4.30 AAS Grade Zn Standard, 1000 µg/ml.

3.1.4.4.31 AAS Grade Al Standard, 1000 µg/ml.

3.1.4.4.32 AAS Grade Fe Standard, 1000 µg/ml.

3.1.4.4.33 The metals standards may also be made from solid chemicals as described in EPA Method 200.7. EPA SW-846 Method 7470 or Standard Methods for the Analysis of Water and Wastewater, 15th Edition, Method 303F should be referred to for additional information on mercury standards.

3.1.4.4.34 Mercury Standards and Quality Control Samples. Prepare fresh weekly a 10 µg/ml intermediate mercury standard by adding 5 ml of 1000 µg/ml mercury stock solution to a 500-ml volumetric flask; dilute with

stirring to 500 ml by first carefully adding 20 ml of 15 percent HNO_3 and then adding water to the 500-ml volume. Mix well. Prepare a 200 ng/ml working mercury standard solution fresh daily: add 5 ml of the 10 µg/ml intermediate standard to a 250-ml volumetric flask and dilute to 250 ml with 5 ml of 4 percent $KMnO_4$, 5 ml of 15 percent HNO_3, and then water. Mix well. At least six separate aliquots of the working mercury standard solution should be used to prepare the standard curve. These aliquots should contain 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the working standard solution containing 0, 200, 400, 600, 800, and 1000 ng mercury, respectively. Quality control samples should be prepared by making a separate 10 µg/ml standard and diluting until in the range of the calibration.

3.1.4.4.35 ICAP Standards and Quality Control Samples. Calibration standards for ICAP analysis can be combined into four different mixed standard solutions as shown below.

MIXED STANDARD SOLUTIONS FOR ICAP ANALYSIS

Solution	Elements
I	As, Be, Cd, Mn, Pb, Se, Zn
II	Ba, Cu, Fe
III	Al, Cr, Ni
IV	Ag, P, Sb, Tl

Prepare these standards by combining and diluting the appropriate volumes of the 1000 µg/ml solutions with 5 percent nitric acid. A minimum of one standard and a blank can be used to form each calibration curve. However, a separate quality control sample spiked with known amounts of the target metals in quantities in the midrange of the calibration curve should be prepared. Suggested standard levels are 25 µg/ml for Al, Cr, and Pb, 15 µg/ml for Fe, and 10 µg/ml for the remaining elements. Standards containing less than 1 µg/ml of metal should be prepared daily. Standards containing greater than 1 µg/ml of metal should be stable for a minimum of 1 to 2 weeks.

3.1.4.4.36 Graphite Furnace AAS Standards. Antimony, arsenic, cadmium, lead, selenium, and thallium. Prepare a 10 µg/ml standard by adding 1 ml of 1000 µg/ml standard to a 100-ml volumetric flask. Dilute with stirring to 100 ml with 10 percent nitric acid. For graphite furnace AAS, the

standards must be matrix matched. Prepare a 100 ng/ml standard by adding 1 ml of the 10 μg/ml standard to a 100-ml volumetric flask and dilute to 100 ml with the appropriate matrix solution. Other standards should be prepared by dilution of the 100 ng/ml standards. At least five standards should be used to make up the standard curve. Suggested levels are 0, 10, 50, 75, and 100 ng/ml. Quality control samples should be prepared by making a separate 10 μg/ml standard and diluting until it is in the range of the samples. Standards containing less than 1 μg/ml of metal should be prepared daily. Standards containing greater than 1 μg/ml of metal should be stable for a minimum of 1 to 2 weeks.

3.1.4.4.37 Matrix Modifiers.

3.1.4.4.37.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of $Ni(NO_3)_2 \cdot 6H_2O$ in approximately 50 ml of water in a 100-ml volumetric flask. Dilute to 100 ml with water.

3.1.4.4.37.2 Nickel Nitrate, 0.1 Percent (V/V). Dilute 10 ml of the 1 percent nickel nitrate solution from Section 4.4.37.1 above to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for As.

3.1.4.4.37.3 Lanthanum. Carefully dissolve 0.5864 g of La_2O_3 in 10 ml of concentrated HNO_3 and dilute the solution by adding it with stirring to approximately 50 ml of water, and then dilute to 100 ml with water. Mix well. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for Pb.

3.1.5 Procedure

3.1.5.1 Sampling. The complexity of this method is such that, to obtain reliable results, testers and analysts should be trained and experienced with the test procedures, including source sampling, reagent preparation and handling, sample handling, analytical calculations, reporting, and

descriptions specifically at the beginning of and throughout Section 3.1.4 and all other sections of this methodology.

3.1.5.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except that, unless particulate emissions are to be determined, the filter need not be desiccated or weighed. All sampling train glassware should first be rinsed with hot tap water and then washed in hot soapy water. Next, glassware should be rinsed three times with tap water, followed by three additional rinses with water. All glassware should then be soaked in a 10 percent (V/V) nitric acid solution for a minimum of 4 hours, rinsed three times with water, rinsed a final time with acetone, and allowed to air dry. All glassware openings where contamination can occur should be covered until the sampling train is assembled for sampling.

3.1.5.1.2 Preliminary Determinations. Same as Method 5, Section 4.1.2.

3.1.5.1.3 Preparation of Sampling Train. Follow the same general procedures given in Method 5, Section 4.1.3, except place 100 ml of the nitric acid/hydrogen peroxide solution (Section 3.1.4.2.1) in each of the two HNO_3/H_2O_2 impingers as shown in Figure 3.1-1 (normally the second and third impingers), place 100 ml of the acidic potassium permanganate absorbing solution (Section 3.1.4.2.2) in each of the two permanganate impingers as shown in Figure A-1, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the last impinger. Alternatively, the silica gel may be weighed directly in the impinger just prior to train assembly.

Several options are available to the tester based on the sampling requirements and conditions. The use of an empty first impinger can be eliminated if the moisture to be collected in the impingers will be less than approximately 100 ml. If necessary, use as applicable to this methodology the procedure described in Section 7.1.1 of EPA Method 101A, 40 CFR Part 61, Appendix B, to maintain the desired color in the last permanganate impinger.

Retain for reagent blanks volumes of the nitric acid/hydrogen peroxide solution per Section 3.1.5.2.9 of this method and of the acidic potassium permanganate solution per Section 3.1.5.2.10. These reagent blanks should be labeled and analyzed as described in Section 3.1.7. Set up the sampling train as shown in Figure 3.1-1, or if mercury analysis is not to be performed in the train, then it should be modified by removing the two permanganate impingers and the impinger preceding the permanganate impingers. If necessary to ensure leak-free sampling train connections and prevent contamination Teflon tape or other non-contaminating material should be used instead of silicone grease.

> Precaution: Extreme care should be taken to prevent contamination within the train. Prevent the mercury collection reagent (acidic potassium permanganate) from contacting any glassware of the train which is washed and analyzed for Mn. Prevent hydrogen peroxide from mixing with the acidic potassium permanganate.

Mercury emissions can be measured, alternatively, in a separate train which measures only mercury emissions by using EPA Method 101A with the modifications described below (and with the further modification that the permanganate containers shall be processed as described in the Precaution in Section 3.1.4.2.2 and the Note in Section 3.1.5.2.5 of this methodology). This alternative method is applicable for measurement of mercury emissions, and it may be of special interest to sources which must measure both mercury and manganese emissions.

> [Section 7.2.1 of Method 101A shall be modified as follows after the 250 to 400-ml $KMnO_4$ rinse:
>
> > To remove any precipitated material and any residual brown deposits on the glassware following the permanganate rinse, rinse with approximately 100 ml of deionized distilled water, and add this water rinse carefully assuring transfer

of all loose precipitated materials from the three permanganate impingers into the permanganate Container No. 1. If no visible deposits remain after this water rinse, do not rinse with 8 N HCl. However, if deposits do remain on the glassware after this water rinse, wash the impinger surfaces with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled Container No. 1.A. containing 200 ml of water as follows. Place 200 ml of water in a sample container labeled Container No. 1.A. Wash the impinger walls and stem with the HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing all permanganate impingers combined. Rinse the first impinger, then pour the actual rinse used for the first impinger into the second impinger for its rinse, etc. Finally, pour the 25 ml of 8 N HCl rinse carefully with stirring into Container No. 1.A. Analyze the HCl rinse separately by carefully diluting with stirring the contents of Container No. 1.A. to 500 ml with deionized distilled water. Filter (if necessary) through Whatman 40 filter paper, and then analyze for mercury according to Section 7.4, except limit the aliquot size to a maximum of 10 ml. Prepare and analyze a water diluted blank 8 N HCl sample by using the same procedure as that used by Container No. 1.A., except add 5 ml of 8 N HCl with stirring to 40 ml of water, and then dilute to 100 ml with water. Then analyze as instructed for the sample from Container No. 1.A. Because the previous separate permanganate solution rinse (Section 7.2.1) and

water rinse (as modified in these guidelines) have the capability to recover a very high percentage of the mercury from the permanganate impingers, the amount of mercury in the HCl rinse in Container No. 1.A. may be very small, possibly even insignificantly small. However, add the total of any mercury analyzed and calculated for the HCl rinse sample Container No. 1.A. to that calculated from the mercury sample from Section 7.3.2 which contains the separate permanganate rinse (and water rinse as modified herein) for calculation of the total sample mercury concentration.

3.1.5.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Section 4.1.4.1 (Pretest Leak-Check), Section 4.1.4.2 (Leak-Checks During the Sample Run), and Section 4.1.4.3 (Post-Test Leak-Checks).

3.1.5.1.5 Sampling Train Operation. Follow the procedures given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5.

3.1.5.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

3.1.5.2 Sample Recovery. Begin cleanup procedures as soon as the probe is removed from the stack at the end of a sampling period.

The probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-contaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling. This normally causes a

vacuum to form in the filter holder, thus causing the undesired result of drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train and cap the open outlet. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger and cap the impinger. Cap off the filter holder outlet and impinger inlet. Use non-contaminating caps, whether ground-glass stoppers, plastic caps, serum caps, or Teflon tape to close these openings.

Alternatively, the train can be disassembled before the probe and filter holder/oven are completely cooled, if this procedure is followed: Initially disconnect the filter holder outlet/impinger inlet and loosely cap the open ends. Then disconnect the probe from the filter holder or cyclone inlet and loosely cap the open ends. Cap the probe tip and remove the umbilical cord as previously described.

Transfer the probe and filter-impinger assembly to a cleanup area that is clean and protected from the wind and other potential causes of contamination or loss of sample. Inspect the train before and during disassembly and note any abnormal conditions. The sample is recovered and treated as follows (see schematic in Figure 3.1-2). Ensure that all items necessary for recovery of the sample do not contaminate it.

3.1.5.2.1 Container No. 1 (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. Acid-washed polypropylene or Teflon coated tweezers or clean, disposable surgical gloves rinsed with water and dried should be used to handle the filters. If it is necessary to fold the filter, make certain the particulate cake is inside the fold. Carefully transfer the filter and any particulate matter or filter fibers that adhere to the filter holder gasket to the petri dish by using a dry (acid-cleaned) nylon bristle brush. Do not use any metal-containing materials when recovering this train. Seal the labeled petri dish.

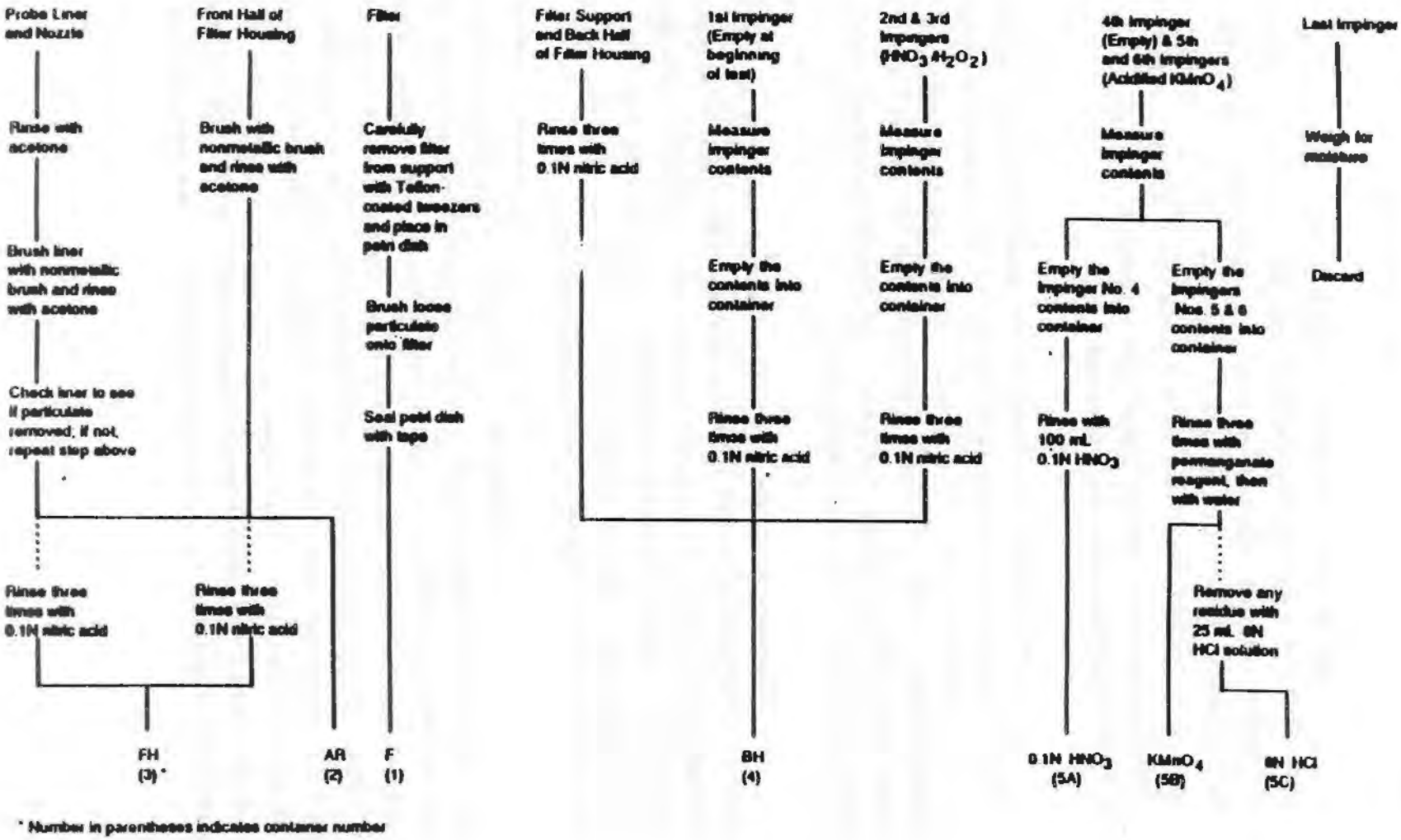

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Figure 3.1-2 Sample recovery scheme.

3.1.5.2.2 Container No. 2 (Acetone Rinse). NOTE: Perform Section 3.1.5.2.2 only if determination of particulate emissions are desired in addition to metals emissions. If only metals emissions are desired, skip Section 3.1.5.2.2 and go to Section 3.1.5.2.3. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter and any condensate from the probe nozzle, probe fitting (plastic such as Teflon, polypropylene, etc. fittings are recommended to prevent contamination by metal fittings; further, if desired, a single glass piece consisting of a combined probe tip and probe liner may be used, but such a single glass piece is not a requirement of this methodology), probe liner, and front half of the filter holder by washing these components with 100 ml of acetone and placing the wash in a glass container. Note: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nonmetallic brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the sample-exposed, inside parts of the fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Allow the acetone to drain from the lower end into the sample container. A funnel may be used to aid in transferring liquid washings to the container. Follow the acetone rinse with a nonmetallic probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe three times or more until none remains in the probe liner on visual inspection. Rinse the

brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Clean the inside of the front half of the filter holder by rubbing the surfaces with a nonmetallic nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container clearly to identify its contents.

3.1.5.2.3 Container No. 3 (Probe Rinse). Keep the probe assembly clean and free from contamination as described in Section 3.1.5.2.2 of this method during the 0.1 N nitric acid rinse described below. Rinse the probe nozzle and fitting probe liner, and front half of the filter holder thoroughly with 100 ml of 0.1 N nitric acid and place the wash into a sample storage container. Note: The use of exactly 100 ml is necessary for the subsequent blank correction procedures. Perform the rinses as applicable and generally as described in Method 12, Section 5.2.2. Record the volume of the combined rinse. Mark the height of the fluid level on the outside of the storage container and use this mark to determine if leakage occurs during transport. Seal the container and clearly label the contents. Finally, rinse the nozzle, probe liner, and front half of the filter holder with water followed by acetone and discard these rinses.

3.1.5.2.4 Container No. 4 (Impingers 1 through 3, HNO_3/H_2O_2 Impingers and Moisture Knockout Impinger, when used, Contents and Rinses). Due to the potentially large quantity of liquid involved, the tester may place

the impinger solutions from impingers 1 through 3 in more than one container. Measure the liquid in the first three impingers volumetrically to within 0.5 ml using a graduated cylinder. Record the volume of liquid present. This information is required to calculate the moisture content of the sampled flue gas. Clean each of the first three impingers, the filter support, the back half of the filter housing, and connecting glassware by thoroughly rinsing with 100 ml of 0.1 N nitric acid using the procedure as applicable and generally as described in Method 12, Section 5.2.4. Note: The use of exactly 100 ml of 0.1 N nitric acid rinse is necessary for the subsequent blank correction procedures. Combine the rinses and impinger solutions, measure and record the volume. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport. Seal the container and clearly label the contents.

3.1.5.2.5 Container Nos. 5A, 5B, and 5C. 5A (0.1 N HNO_3), 5B ($KMnO_4$/ H_2SO_4 absorbing solution), and 5C (8 N HCl rinse and dilution). (As described previously at the end of Section 3.1.3.1.5 of this method, if mercury is not being measured in this train, then impingers 4, 5, and 6, as shown in Figure A-1, are not necessary and may be eliminated.) Pour all the liquid, if any, from the impinger which was empty at the start of the run and which immediately precedes the two permanganate impingers (normally impinger No. 4) into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place the liquid in Sample Container No. 5A. Rinse the impinger (No. 4) with 100 ml of 0.1 N HNO_3 and place this into Container No. 5A.

Pour all the liquid from the two permanganate impingers into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Place this $KMnO_4$ absorbing solution stack sample from the two permanganate impingers into Container No. 5B. Using 100 ml total of fresh acidified potassium permanganate solution, rinse the two permanganate impingers and connecting glass pieces a minimum of three times and place the rinses into Container No. 5B, carefully ensuring transfer of all loose precipitated materials from the two impingers into Container No. 5B. Using 100 ml total of water, rinse the

permanganate impingers and connecting glass pieces a minimum of three times, and place the rinses into Container 5B, carefully ensuring transfer of all loose precipitated material, if any, from the two impingers into Container No. 5B. Mark the height of the fluid level on the outside of the bottle to determine if leakage occurs during transport. See the following note and the Precaution in Paragraph 3.1.4.2.2 and properly prepare the bottle and clearly label the contents.

> Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles shall not be completely filled and shall be vented to relieve potential excess pressure. Venting is required. A No. 70-72 hole drilled in the container cap and Teflon liner has been used.

If no visible deposits remain after the above described water rinse, do not rinse with 8 N HCl. However, if deposits do remain on the glassware after this water rinse, wash the impinger surfaces with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled Container No. 5C containing 200 ml of water as follows: Place 200 ml of water in a sample container labeled Container No. 5C. Wash the impinger walls and stem with the HCl by turning the impinger on its side and rotating it so that the HCl contacts all inside surfaces. Use a total of only 25 ml of 8 N HCl for rinsing both permanganate impingers combined. Rinse the first impinger, then pour the actual rinse used for the first impinger into the second impinger for its rinse. Finally, pour the 25 ml of 8 N HCl rinse carefully with stirring into Container No. 5C. Mark the height of the fluid level on the outside of the bottle to determine if leakage occurs during transport.

3.1.5.2.6 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger.

The small amount of particles that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel impinger is used for moisture calculations. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

3.1.5.2.7 Container No. 7 (Acetone Blank). If particulate emissions are to be determined, at least once during each field test, place a 100-ml portion of the acetone used in the sample recovery process into a labeled container for use in the front-half field reagent blank. Seal the container.

3.1.5.2.8 Container No. 8A (0.1 N Nitric Acid Blank). At least once during each field test, place 300 ml of the 0.1 N nitric acid solution used in the sample recovery process into a labeled container for use in the front-half and back-half field reagent blanks. Seal the container. Container No. 8B (water blank). At least once during each field test, place 100 ml of the water used in the sample recovery process into a labeled Container No. 8B. Seal the container.

3.1.5.2.9 Container No. 9 (5% Nitric Acid/10% Hydrogen Peroxide Blank). At least once during each field test, place 200 ml of the 5% nitric acid/10% hydrogen peroxide solution used as the nitric acid impinger reagent into a labeled container for use in the back-half field reagent blank. Seal the container.

3.1.5.2.10 Container No. 10 (Acidified Potassium Permanganate Blank). At least once during each field test, place 100 ml of the acidified potassium permanganate solution used as the impinger solution and in the sample recovery process into a labeled container for use in the back-half field reagent blank for mercury analysis. Prepare the container as described in Section 3.1.5.2.5.

> Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles shall not be

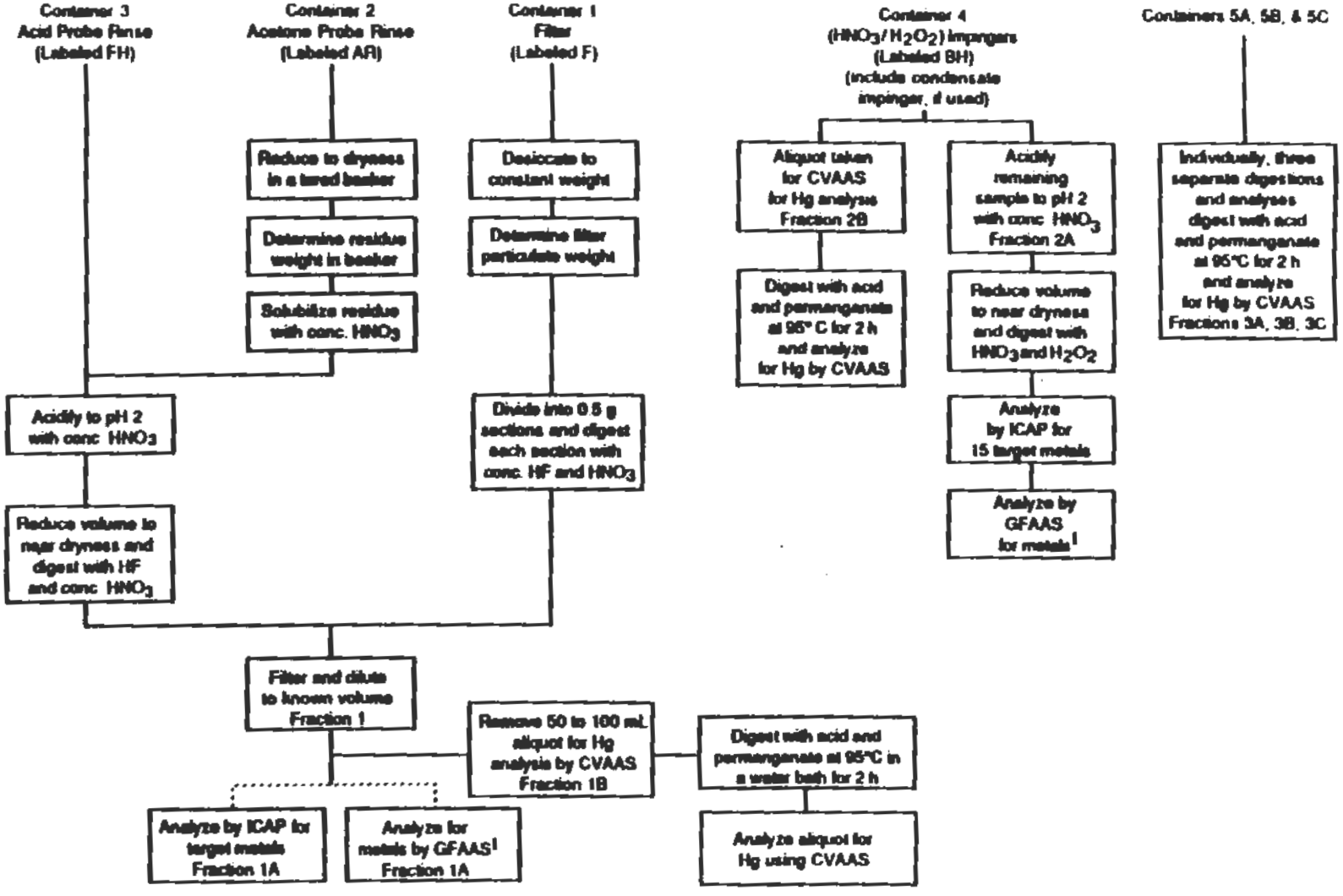
completely filled and shall be vented to relieve potential excess pressure. Venting is required. A No. 70-72 hole drilled in the container cap and Teflon liner has been used.

3.1.5.2.11 Container No. 11 (8 N HCl Blank). At least once during each field test, perform both of the following: Place 200 ml of water into a sample container. Pour 25 ml of 8 N HCl carefully with stirring into the 200 ml of water in the container. Mix well and seal the container.

3.1.5.2.12 Container No. 12 (Filter Blank). Once during each field test, place three unused blank filters from the same lot as the sampling filters in a labeled petri dish. Seal the petri dish. These will be used in the front-half field reagent blank.

3.1.5.3 Sample Preparation. Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram illustrating sample preparation and analysis procedures for each of the sample train components is shown in Figure 3.1-3.

3.1.5.3.1 Container No. 1 (Filter). If particulate emissions are being determined, then desiccate the filter and filter catch without added heat and weigh to a constant weight as described in Section 4.3 of Method 5. For analysis of metals, divide the filter with its filter catch into portions containing approximately 0.5 g each and place into the analyst's choice of either individual microwave pressure relief vessels or Parr^{R} Bombs. Add 6 ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid to each vessel. For microwave heating, microwave the sample vessels for approximately 12-15 minutes in intervals of 1 to 2 minutes at 600 Watts. For conventional heating, heat the Parr Bombs at 140°C (285°F) for 6 hours. Cool the samples to room temperature and combine with the acid digested probe rinse as required in Section 3.1.5.3.3, below.



[1] Analysis by AAS for metals found at less than 2 ug/mL in digestate solution, if desired. Or analyze for each metal by AAS, if desired.

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Figure 3.1-3 Sample preparation and analysis scheme.

Notes: 1. Suggested microwave heating times are approximate and are dependent upon the number of samples being digested. Twelve to 15 minute heating times have been found to be acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by sorbent reflux within the vessel.

2. If the sampling train uses an optional cyclone, the cyclone catch should be prepared and digested using the same procedures described for the filters and combined with the digested filter samples.

3.1.5.3.2 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to an acid-cleaned, tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. If particulate emissions are being determined, desiccate for 24 hours without added heat, weigh to a constant weight according to the procedures described in Section 4.3 of Method 5, and report the results to the nearest 0.1 mg. Redissolve the residue with 10 ml of concentrated nitric acid and, carefully with stirring, quantitatively combine the resultant sample including all liquid and any particulate matter with Container No. 3 prior to beginning the following Section 3.1.5.3.3.

3.1.5.3.3 Container No. 3 (Probe Rinse). The pH of this sample shall be 2 or lower. If the pH is higher, the sample should be acidified to pH 2 by the careful addition with stirring of concentrated nitric acid. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Digest the sample in microwave vessels or Parr^{R} Bombs by quantitatively transferring the sample to the vessel or bomb, by carefully adding the 6 ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid

and then continuing to follow the procedures described in Section 3.1.5.3.1; then combine the resultant sample directly with the acid digested portions of the filter prepared previously in Section 3.1.5.3.1. The resultant combined sample is referred to as Fraction 1 precursor. Filter the combined solution of the acid digested filter and probe rinse samples using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. This dilution is Fraction 1. Measure and record the volume of the Fraction 1 solution to within 0.1 ml. Quantitatively remove a 50-ml aliquot and label as Fraction 1B. Label the remaining 250-ml portion as Fraction 1A. Fraction 1A is used for ICAP or AAS analysis. Fraction 1B is used for the determination of front-half mercury.

3.1.5.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample (Fraction 2) to within 0.5 ml. Remove a 75- to 100-ml aliquot for mercury analysis and label as Fraction 2B. Label the remaining portion of Container No. 4 as aliquot Fraction 2A. Aliquot Fraction 2A defines the volume of 2A <u>prior</u> to digestion. All of the aliquot Fraction 2A is digested to produce concentrated Fraction 2A. Concentrated Fraction 2A defines the volume of 2A <u>after</u> digestion which is normally 150 ml. Only concentrated Fraction 2A is analyzed for metals (except that it is not analyzed for mercury). The Fraction 2B aliquot should be prepared and analyzed for mercury as described in Section 3.1.5.4.3. Aliquot Fraction 2A shall be pH 2 or lower. If necessary, use concentrated nitric acid, by careful addition and stirring, to lower aliquot Fraction 2A to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Next follow either the conventional or microwave digestion procedures described in Sections 3.1.5.3.4.1 and 3.1.5.3.4.2, below.

3.1.5.3.4.1 Conventional Digestion Procedure. Add 30 ml of 50 percent nitric acid and heat for 30 minutes on a hot plate to just below boiling. Add 10 ml of 3 percent hydrogen peroxide and heat for 20 more minutes. Add 50 ml of hot water and heat the sample for an additional 20 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate

volume for the expected metals concentrations) with water. This dilution is concentrated Fraction 2A. Measure and record the volume of the Fraction 2A solution to within 0.1 ml.

3.1.5.3.4.2 Microwave Digestion Procedure. Add 10 ml of 50 percent nitric acid and heat for 6 minutes in intervals of 1 to 2 minutes at 600 Watts. Allow the sample to cool. Add 10 ml of 3 percent hydrogen peroxide and heat for 2 more minutes. Add 50 ml of hot water and heat for an additional 5 minutes. Cool, filter the sample, and dilute to 150 ml (or the appropriate volume for the expected metals concentrations) with water. This dilution is concentrated Fraction 2A. Measure and record the volume of the Fraction 2A solution to within 0.1 ml.

> Note: All microwave heating times given are approximate and are dependent upon the number of samples being digested at a time. Heating times as given above have been found acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by solvent reflux within the vessel.

3.1.5.3.5 Container Nos. 5A, 5B, and 5C (Impingers 4, 5, and 6). Keep these samples separate from each other and measure and record the volumes of 5A and 5B separately to within 0.5 ml. Dilute sample 5C to 500 ml with water. These samples 5A, 5B, and 5C are referred to respectively as Fractions 3A, 3B, and 3C. Follow the analysis procedures described in Section 3.1.5.4.3.

Because the permanganate rinse and water rinse have the capability to recover a high percentage of the mercury from the permanganate impingers, the amount of mercury in the HCl rinse (Fraction 3C) may be very small, possibly even insignificantly small. However, as instructed in this method, add the total of any mercury measured in and calculated for the HCl rinse (Fraction 3C) to that for Fractions 1B, 2B, 3A, and 3B for calculation of the total sample mercury concentration.

3.1.5.3.6 Container No. 6 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

3.1.5.4 Sample Analysis. For each sampling train, seven individual samples are generated for analysis. A schematic identifying each sample and the prescribed sample preparation and analysis scheme is shown in Figure 3.1-3. The first two samples, labeled Fractions 1A and 1B, consist of the digested samples from the front half of the train. Fraction 1A is for ICAP or AAS analysis as described in Sections 3.1.5.4.1 and/or 3.1.5.4.2. Fraction 1B is for determination of front-half mercury as described in Section 3.1.5.4.3.

The back half of the train was used to prepare the third through seventh samples. The third and fourth samples, labeled Fractions 2A and 2B, contain the digested samples from the moisture knockout, if used, and HNO_3/H_2O_2 Impingers 1 through 3. Fraction 2A is for ICAP or AAS analysis. Fraction 2B will be analyzed for mercury.

The fifth through seventh samples, labeled Fractions 3A, 3B, and 3C, consist of the impinger contents and rinses from the empty and permanganate impingers 4, 5, and 6. These samples are analyzed for mercury as described in Section 3.1.5.4.3. The total back-half mercury catch is determined from the sum of Fraction 2B and Fractions 3A, 3B, and 3C.

3.1.5.4.1 ICAP Analysis. Fraction 1A and Fraction 2A are analyzed by ICAP using EPA SW-846 Method 6010 or Method 200.7 (40 CFR 136, Appendix C). Calibrate the ICAP, and set up an analysis program as described in Method 6010 or Method 200.7. The quality control procedures described in Section 3.1.7.3.1 of this method shall be followed. Recommended wavelengths for use in the analysis are listed below:

Element	Wavelength (nm)
Aluminum	308.215
Antimony	206.833
Arsenic	193.696
Barium	455.403
Beryllium	313.042
Cadmium	226.502
Chromium	267.716
Copper	324.754
Iron	259.940
Lead	220.353
Manganese	257.610
Nickel	231.604
Phosphorus	214.914
Selenium	196.026
Silver	328.068
Thallium	190.864
Zinc	213.856

The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

Initially, analyze all samples for the desired target metals (except mercury) plus iron and aluminum. If iron and aluminum are present in the sample, the sample may have to be diluted so that each of these elements is at a concentration of less than 50 ppm to reduce their spectral interferences on arsenic, cadmium, chromium, and lead.

> Note: When analyzing samples in a hydrofluoric acid matrix, an alumina torch should be used; since all front-half samples will contain hydrofluoric acid, use an alumina torch.

3.1.5.4.2 AAS by Direct Aspiration and/or Graphite Furnace. If analysis of metals in Fraction 1A and Fraction 2A using graphite furnace or direct aspiration AAS is desired, Table 3.1-2 should be used to determine

Table 3.1-2

APPLICABLE TECHNIQUES, METHODS, AND MINIMIZATION OF INTERFERENCE FOR AAS ANALYSIS

Metal	Technique	SW-846 Method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Sb	Aspiration	7040	217.6	1000 mg/mL Pb Ni, Cu, or acid	Use secondary wavelength of 231.1 nm; match sample & standards' acid concentration or use nitrous oxide/acetylene flame
Sb	Furnace	7041	217.6	High Pb	Secondary wavelength or Zeeman correction
As	Furnace	7060	193.7	Arsenic volatization	Spiked samples and add nickel nitrate solution to digestates prior to analysis
				Aluminum	Use Zeeman background correction
Ba	Aspiration	7080	553.6	Calcium	High hollow cathode current and narrow band set
				Barium ionization	2 mL of KCl per 100 mL of sample
Be	Aspiration	7090	234.9	500 ppm Al High Mg and Si	Add 0.1% fluoride Use method of standard additions
Be	Furnace	7091	234.9	Be in optical path	Optimize parameters to minimize effects
Cd	Aspiration	7130	228.8	Absorption and light scattering	Background correction is required
Cd	Furnace	7131	228.8	As above Excess chloride	As above Ammonium phosphate used as a matrix modifier
				Pipet tips	Use cadmium-free tips
Cr	Aspiration	7190	357.9	Alkali metal	KCl ionization suppressant in samples and standards
				Absorption and scatter	Consult manufacturer's literature
Cr	Furnace	7191	357.9	200 mg/L Ca and P	All calcium nitrate for a known constant effect and to eliminate effect of phosphate
Cu	Aspiration	7210	324.7	Absorption and scatter	Consult manufacturer's manual

(continued)

Table 3.1-2

APPLICABLE TECHNIQUES, METHODS, AND MINIMIZATION OF INTERFERENCE FOR AAS ANALYSIS

Metal	Technique	SW-846 Method No.	Wavelength (nm)	Interferences	
				Cause	Minimization
Fe	Aspiration	7380	248.3	Contamination	Great care taken to avoid contamination
Pb	Aspiration	7420	283.3	217.0 nm alternate	Background correction required
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 uL of phosphorus acid to 1 mL of prepared sample in sampler cup
Mn	Aspiration	7460	279.5	403.1 nm alternate	Background correction required
Ni	Aspiration	7520	232.0	352.4 nm alternate	Background correction required
				Fe, Co, and Cr	Matrix matching or nitrous-oxide/ acetylene flame
				Nonlinear response	Sample dilution or use 352.3 nm line
Se	Furnace	7740	196.0	Volatility	Spike samples and reference materials and add nickel nitrate to minimize volatilization
				Adsorption & scatter	Background correction is required and Zeeman background correction can be useful
Ag	Aspiration	7760	328.1	Adsorption & scatter	Background correction is required
				AgCl insolube	Avoid hydrochloric acid unless silver is in solution as a chloride complex
				Viscosity	Sample and standards monitored for aspiration rate
Tl	Aspiration	7840	276.8		Background correction is required
					Hydrochloric acid should not be used
Tl	Furnace	7841	276.8	Hydrochloric acid or chloride	Background correction is required
					Verify that losses are not occurring for volatization by spiked samples or standard addition; Palladium is a suitable matrix modifier
Zn	Aspiration	7950	213.9	High Si, Cu, & P	Strontium removes Cu and phosphate
				Contamination	Great care taken to avoid contamination

which techniques and methods should be applied for each target metal. Table 3.1-2 should also be consulted to determine possible interferences and techniques to be followed for their minimization. Calibrate the instrument according to Section 3.1.6.3 and follow the quality control procedures specified in Section 3.1.7.3.2.

3.1.5.4.3 Cold Vapor AAS Mercury Analysis. Fraction 1B, Fraction 2B, and Fractions 3A, 3B, and 3C should be analyzed separately for mercury using cold vapor atomic absorption spectroscopy following the method outlined in EPA SW-846 Method 7470 or in Standard Methods for Water and Wastewater Analysis, 15th Edition, Method 303F. Set up the calibration curve (zero to 1000 ng) as described in SW-846 Method 7470 or similar to Method 303F, using 300-ml BOD bottles instead of Erlenmeyers. Dilute separately, as described below, a 1 ml to 10 ml aliquot of each original sample to 100 ml with water. Record the amount of the aliquot used for dilution to 100 ml. If no prior knowledge exists of the expected amount of mercury in the sample, a 5-ml aliquot is suggested for the first dilution to 100 ml and analysis. To determine the stack emission value for mercury, the amount of the aliquot of the sample used for dilution and analysis is dependent on the amount of mercury in the aliquot: the total amount of mercury in the aliquot used for analysis shall be less than 1 μg, and within the range (zero to 1000 ng) of the calibration curve. Place each sample aliquot into a separate 300-ml BOD bottle and add enough Type II water to make a total volume of 100 ml. Then analyze the 100 ml for mercury by adding to it sequentially the sample preparation solutions and performing the sample preparation and analysis as described in the procedures of SW-846 Method 7470 or Method 303F. If, during the described analysis, the reading maximum(s) are off-scale (because the aliquot of the original sample analyzed contained more mercury than the maximum of the calibration range) including the analysis of the 100-ml dilution of the 1-ml aliquot of the original sample causing a reading maximum which is off-scale, then perform the following: dilute the original sample (or a portion of it) with 0.15% HNO_3 in water (1.5 ml concentrated HNO_3 per liter aqueous solution) so that when a 1-ml to 10-ml aliquot of the dilution of the original sample is then further diluted to 100 ml in the BOD bottle,

and analyzed by the procedures described above, it will yield an analysis within the range of the calibration curve.

3.1.6 Calibration

Maintain a laboratory log of all calibrations.

3.1.6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

3.1.6.2 Inductively Coupled Argon Plasma Spectrometer Calibration. Prepare standards as outlined in Section 3.1.4.4. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures using the above standards. The instrument calibration should be checked once per hour. If the instrument does not reproduce the concentrations of the standard within 10 percent, the complete calibration procedures should be performed.

3.1.6.3 Atomic Absorption Spectrometer - Direct Aspiration, Graphite Furnace and Cold Vapor Mercury Analyses. Prepare the standards as outlined in Section 3.1.4.4. Calibrate the spectrometer using these prepared standards. Calibration procedures are also outlined in the EPA methods referred to in Table 3.1-2 and in SW-846 Method 7470 or Standard Methods for Water and Wastewater, 15th Edition, Method 303F (for mercury). Each standard curve should be run in duplicate and the mean values used to calculate the calibration line. The instrument should be recalibrated approximately once every 10 to 12 samples.

3.1.7 Quality Control

3.1.7.1 Sampling. Field Reagent Blanks. When analyzed, the blank samples in Container Numbers 7 through 12 produced previously in Sections

3.1.5.2.7 through 3.1.5.2.12, respectively, shall be processed, digested, and analyzed as follows: Digest and process one of the filters from Container No. 12 per Section 3.1.5.3.1, 100 ml from Container No. 7 per Section 3.1.5.3.2, and 100 ml from Container No. 8A per Section 3.1.5.3.3. This produces Fraction Blank 1A and Fraction Blank 1B from Fraction Blank 1. [If desired, the other two filters may be digested separately according to Section 3.1.5.3.1, diluted separately to 300 ml each, and analyzed separately to produce a blank value for each of the two additional filters. If these analyses are performed, they will produce two additional values for each of Fraction Blank 1A and Fraction Blank 1B. The three Fraction Blank 1A values will be calculated as three values of M_{fnb} in Equation 3 of Section 3.1.8.4.3, and then the three values shall be totalled and divided by 3 to become the value M_{fnb} to be used in the computation of M_t by Equation 3. Similarly, the three Fraction Blank 1B values will be calculated separately as three values, totalled, averaged, and used as the value for Hg_{fnb} in Equation 8 of Section 3.1.8.5.3. The analyses of the two extra filters are optional and are <u>not</u> a requirement of this method, but if the analyses are performed, the results must be considered as described above.] Combine 100 ml of Container No. 8A with 200 ml of the contents of Container No. 9 and digest and process the resultant volume per Section 3.1.5.3.4. This produces concentrated Fraction Blank 2A and Fraction Blank 2B from Fraction Blank 2. A 100-ml portion of Container No. 8A is Fraction Blank 3A. Combine 100 ml of the contents of Container No. 10 with 33 ml of the contents of Container No. 8B. This produces Fraction Blank 3B (use 400 ml as the volume of Fraction Blank 3B when calculating the blank value. Use the actual volumes when calculating all the other blank values). Dilute 225 ml of the contents of Container No. 11 to 500 ml with water. This produces Fraction Blank 3C. Analyze Fraction Blank 1A and Fraction Blank 2A per Section 3.1.5.4.1 and/or 3.1.5.4.2. Analyze Fraction Blank 1B, Fraction Blank 2B, and Fraction Blanks 3A, 3B, and 3C per Section 3.1.5.4.3. The analysis of Fraction Blank 1A produces the front-half reagent blank correction values for the metals except mercury; the analysis of Fraction Blank 1B produces the front-half reagent blank correction value for mercury. The analysis of concentrated Fraction Blank 2A produces the back-half reagent blank correction values for the metals except mercury, while

separate analysis of Fraction Blanks 2B, 3A, 3B, and 3C produce the back-half reagent blank correction value for mercury.

3.1.7.2 An attempt may be made to determine if the laboratory reagents used in Section 3.1.5.3 caused contamination. They should be analyzed by the procedures in Section 3.1.5.4. The Administrator will determine whether the laboratory blank reagent values can be used in the calculation of the stationary source test results.

3.1.7.3 Quality Control Samples. The following quality control samples should be analyzed.

3.1.7.3.1 ICAP Analysis. Follow the quality control shown in Section 8 of Method 6010. For the purposes of a three-run test series, these requirements have been modified to include the following: two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (must be within 25% or analyze by the method of standard additions), one quality control sample to check the accuracy of the calibration standards (must be within 25% of calibration), and one duplicate analysis (must be within 10% of average or repeat all analyses).

3.1.7.3.2 Direct Aspiration and/or Graphite Furnace AAS Analysis for antimony, arsenic, barium, beryllium, cadmium, copper, chromium, lead, nickel, manganese, mercury, phosphorus, selenium, silver, thallium, and zinc. All samples should be analyzed in duplicate. Perform a matrix spike on at least one front-half sample and one back-half sample or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the method of standard additions. A quality control sample should be analyzed to check the accuracy of the calibration standards. The results must be within 10% or the calibration repeated.

3.1.7.3.3 Cold Vapor AAS Analysis for Mercury. All samples should be analyzed in duplicate. A quality control sample should be analyzed to check the accuracy of the calibration standards (within 15% or repeat

calibration). Perform a matrix spike on one sample from the nitric impinger portion (must be within 25% or samples must be analyzed by the method of standard additions). Additional information on quality control can be obtained from EPA SW-846 Method 7470 or in Standard Methods for the Examination of Water and Wastewater, 15th Edition, Method 303F.

3.1.8 Calculations

3.1.8.1 Dry Gas Volume. Using the data from this test, calculate $V_{m(std)}$, the dry gas sample volume at standard conditions as outlined in Section 6.3 of Method 5.

3.1.8.2 Volume of Water Vapor and Moisture Content. Using the data obtained from this test, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_{ws} of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

3.1.8.3 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity.

3.1.8.4 Metals (Except Mercury) in Source Sample.

3.1.8.4.1 Fraction 1A, Front Half, Metals (except Hg). Calculate separately the amount of each metal collected in Fraction 1 of the sampling train using the following equation:

$$M_{fh} = C_{a1} F_d V_{soln,1} \qquad \text{Eq. 1}^*$$

where:

M_{fh} = total mass of each metal (except Hg) collected in the front half of the sampling train (Fraction 1), µg.
C_{a1} = concentration of metal in sample Fraction 1A as read from the standard curve (µg/ml).

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*If Fractions 1A and 2A are combined, proportional aliquots must be used. Appropriate changes must be made in Equations 1-3 to reflect this approach.

F_d = dilution factor (F_d = the inverse of the fractional portion of the concentrated sample in the solution actually used in the instrument to produce the reading C_{a1}. For example, when 2 ml of Fraction 1A are diluted to 10 ml, F_d = 5).

$V_{soln,1}$ = total volume of digested sample solution (Fraction 1), ml.

3.1.8.4.2 Fraction 2A, Back Half, Metals (except Hg). Calculate separately the amount of each metal collected in Fraction 2 of the sampling train using the following equation:

$$M_{bh} = C_{a2} F_a V_a \qquad \text{Eq. 2*}$$

where:

M_{bh} = total mass of each metal (except Hg) collected in the back half of the sampling train (Fraction 2), μg.

C_{a2} = concentration of metal in sample concentrated Fraction 2A, as read from the standard curve (μg/ml).

F_a = aliquot factor, volume of Fraction 2 divided by volume of aliquot Fraction 2A (see Section 3.1.5.3.4).

V_a = total volume of digested sample solution (concentrated Fraction 2A), ml (see Section 3.1.5.3.4.1 or 3.1.5.3.4.2, as applicable).

3.1.8.4.3 Total Train, Metals (except Hg). Calculate the total amount of each of the quantified metals collected in the sampling train as follows:

$$M_t = (M_{fh} - M_{fhb}) + (M_{bh} - M_{bhb}) \qquad \text{Eq. 3*}$$

where:

M_t = total mass of each metal (separately stated for each metal) collected in the sampling train, μg.

M_{fhb} = blank correction value for mass of metal detected in front-half field reagent blank, μg.

M_{bhb} = blank correction value for mass of metal detected in back-half field reagent blank, μg.

Note: If the measured blank value for the front half (m_{fhb}) is in the range 0.0 to A μg [where A μg equals the value determined by multiplying 1.4 μg per square inch (1.4 μg/in^2) times the actual area in square inches (in^2)

of the filter used in the emission sample], m_{fhb} may be used to correct the emission sample value (m_{fh}); if m_{fhb} exceeds A μg, the greater of the two following values (either I. or II.) may be used:

I. A μg, or

II. the lesser of (a) m_{fhb}, or (b) 5 percent of m_{fh}.

If the measured blank value for the back half (m_{bhb}) is in the range of 0.0 to 1 μg, m_{bhb} may be used to correct the emission sample value (m_{bh}); if m_{bhb} exceeds 1 μg, the greater of the two following values may be used: 1 μg or 5 percent of m_{bh}.

3.1.8.5 Mercury in Source Sample.

3.1.8.5.1 Fraction 1B, Front Half, Hg. Calculate the amount of mercury collected in the front half, Fraction 1, of the sampling train using the following equation:

$$Hg_{fh} = \frac{Q_{fh}}{V_{f1B}} \times V_{soln,1} \qquad \text{Eq. 4}$$

where:

Hg_{fh} = total mass of mercury collected in the front half of the sampling train (Fraction 1), μg.
Q_{fh} = quantity of mercury in analyzed sample, μg.
$V_{soln,1}$ = total volume of digested sample solution (Fraction 1), ml.
V_{f1B} = volume of Fraction 1B analyzed, ml. See the following notice.

Note: V_{f1B} is the actual amount of Fraction 1B analyzed. For example, if 1 ml of Fraction 1B were diluted to 100 ml to bring it into the proper analytical range, and 1 ml of the 100-ml dilution were analyzed, V_{f1B} would be 0.01 ml.

3.1.8.5.2 Fraction 2B and Fractions 3A, 3B, and 3C, Back Half, Hg. Calculate the amount of mercury collected in Fractions 2 using Equation 5 and in Fractions 3A, 3B, and 3C using Equation 6. Calculate the total amount of mercury collected in the back half of the sampling train using Equation 7.

$$Hg_{bh2} = \frac{Q_{bh2}}{V_{f2B}} \times V_{soln,2} \qquad \text{Eq. 5}$$

where:

Hg_{bh2} = total mass of mercury collected in Fraction 2, μg.
Q_{bh2} = quantity of mercury in analyzed sample, μg.
$V_{soln,2}$ = total volume of Fraction 2, ml.
V_{f2B} = volume of Fraction 2B analyzed, ml (see the following note).

Note: V_{f2B} is the actual amount of Fraction 2B analyzed. For example, if 1 ml of Fraction 2B were diluted to 10 ml to bring it into the proper analytical range, and 5 ml of the 10-ml dilution was analyzed, V_{f2B} would be 0.5.

Use Equation 6 to calculate separately the back-half mercury for Fractions 3A, then 3B, then 3C.

$$Hg_{bh3(A,B,C)} = \frac{Q_{bh3(A,B,C)}}{V_{f3(A,B,C)}} \times V_{soln,3(A,B,C)} \qquad \text{Eq. 6}$$

where:

$Hg_{bh3(A,B,C)}$ = total mass of mercury collected separately in Fraction 3A, 3B, or 3C, μg.
$Q_{bh3(A,B,C)}$ = quantity of mercury in separately analyzed samples, μg.
$V_{f3(A,B,C)}$ = volume of Fraction 3A, 3B, or 3C analyzed, ml (see Note in Sections 3.1.8.5.1 and 3.1.8.5.2, and calculate similarly).
$V_{soln,3(A,B,C)}$ = total volume of Fraction 3A, 3B, or 3C, ml.

$$Hg_{bh} = Hg_{bh2} + Hg_{bh3A} + Hg_{bh3B} + Hg_{bh3C} \qquad \text{Eq. 7}$$

where:

Hg_{bh} = total mass of mercury collected in the back half of the sampling train, μg.

3.1.8.5.3 Total Train Mercury Catch. Calculate the total amount of mercury collected in the sampling train using Equation 8.

$$Hg_t = (Hg_{fh} - Hg_{fhb}) + (Hg_{bh} - Hg_{bhb}) \quad \text{Eq. 8}$$

where:

Hg_t = total mass of mercury collected in the sampling train, μg.
Hg_{fhb} = blank correction value for mass of mercury detected in front-half field reagent blank, μg.
Hg_{bhb} = blank correction value for mass of mercury detected in back-half field reagent blanks, μg.

Note: If the total of the measured blank values (Hg_{fhb} + Hg_{bhb}) is in the range of 0 to 6 μg, then the total may be used to correct the emission sample value (Hg_{fh} + Hg_{bh}); if it exceeds 6 μg, the greater of the following two values may be used; 6 μg or 5 percent of the emission sample value (Hg_{fh} + Hg_{bh}).

3.1.8.6 Metal Concentration of Stack Gas. Calculate each metal separately for the cadmium, total chromium, arsenic, nickel, manganese, beryllium, copper, lead, phosphorus, thallium, silver, barium, zinc, selenium, antimony, and mercury concentrations in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_s = K_4(M_t/V_{m(std)}) \quad \text{Eq. 9}$$

where:

C_s = concentration of each metal in the stack gas, mg/dscm.
K_4 = 10^{-3} mg/μg.
M_t = total mass of each metal collected in the sampling train, μg; (substitute Hg_t for M_t for the mercury calculation).
$V_{m(std)}$ = volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

3.1.8.7 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

3.1.9 Bibliography

3.1.9.1 Method 303F in Standard Methods for the Examination of Water and Wastewater, 15th Edition, 1980. Available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.

3.1.9.2 EPA Methods 6010, 7000, 7041, 7060, 7131, 7421, 7470, 7740, and 7841, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846, Third Edition. September 1988. Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, D.C. 20460.

3.1.9.3 EPA Method 200.7, Code of Federal Regulations, Title 40, Part 136, Appendix C. July 1, 1987.

3.1.9.4 EPA Methods 1 through 5, and 12 Code of Federal Regulations, Title 40, Part 60, Appendix A, July 1, 1987.

E-2 SW846-METHOD 0030

Volatile Organic Sampling Train (VOST)

METHOD 0030

VOLATILE ORGANIC SAMPLING TRAIN

1.0 PRINCIPLE AND APPLICATION

1.1 Principle

1.1.1 This method describes the collection of volatile principal organic hazardous constituents (POHCs) from the stack gas effluents of hazardous waste incinerators. For the purpose of definition, volatile POHCs are those POHCs with boiling points less than 100°C. If the boiling point of a POHC of interest is less than 30°C, the POHC may break through the sorbent under the conditions of the sample collection procedure.

1.1.2 Field application for POHCs of this type should be supported by laboratory data which demonstrate the efficiency of a volatile organic sampling train (VOST) to collect POHCs with boiling points less than 30°C. This may require using reduced sample volumes collected at flow rates between 250 and 500 mL/min. Many compounds which boil above 100°C (e.g., chlorobenzene) may also be efficiently collected and analyzed using this method. VOST collection efficiency for these compounds should be demonstrated, where necessary, by laboratory data of the type described above.

1.1.3 This method employs a 20-liter sample of effluent gas containing volatile POHCs which is withdrawn from a gaseous effluent source at a flow rate of 1 L/min, using a glass-lined probe and a volatile organic sampling train (VOST). (Operation of the VOST under these conditions has been called FAST-VOST.) The gas stream is cooled to 20°C by passage through a water-cooled condenser and volatile POHCs are collected on a pair of sorbent resin traps. Liquid condensate is collected in an impinger placed between the two resin traps. The first resin trap (front trap) contains approximately 1.6 g Tenax and the second trap (back trap) contains approximately 1 g each of Tenax and petroleum-based charcoal (SKC Lot 104 or equivalent), 3:1 by volume. A total of six pairs of sorbent traps may be used to collect volatile POHCs from the effluent gas stream.

1.1.4 An alternative set of conditions for sample collection has been used. This method involves collecting sample volume of 20 liters or less at reduced flow rate. (Operation of the VOST under these conditions has been referred to as SLO-VOST.) This method has been used to collect 5 liters of sample (0.25 L/min for 20 min) or 20 liters of sample (0.5 L/min for 40 min) on each pair of sorbent cartridges. Smaller sample volumes collected at lower flow rates should be considered when the boiling points of the POHCs of interest are below 35°C. A total of six pairs of sorbent traps may be used to collect volatile POHCs from the effluent gas stream.

1.1.5 Analysis of the traps is carried out by thermal desorption purge-and-trap by gas chromatography/mass spectrometry (see Method 5040). The VOST is designed to be operated at 1 L/min with traps being replaced every 20 min for a total sampling time of 2 hr. Traps may be analyzed separately or combined onto one trap to improve detection limit. However, additional flow rates and sampling times are acceptable. Recent experience has shown that when less than maximum detection ability is required, it is acceptable and probably preferable to operate the VOST at 0.5 L/min for a total of three 40-min periods. This preserves the 2-hr sampling period, but reduces the number of cartridge changes in the field as well as the number of analyses required.

1.2 Application

1.2.1 This method is applicable to the determination of volatile POHCs in the stack gas effluent of hazardous waste incinerators. This method is designed for use in calculating destruction and removal efficiency (DRE) for the volatile POHCs and to enable a determination that DRE values for removal of the volatile POHCs are equal to or greater than 99.99%.

1.2.2 The sensitivity of this method is dependent upon the level of interferences in the sample and the presence of detectable levels of volatile POHCs in blanks. The target detection limit of this method is 0.1 ug/m^3 (ng/L) of flue gas, to permit calculation of a DRE equal to or greater than 99.99% for volatile POHCs which may be present in the waste stream at 100 ppm. The upper end of the range of applicability of this method is limited by breakthrough of the volatile POHCs on the sorbent traps used to collect the sample. Laboratory development data have demonstrated a range of 0.1 to 100 ug/m^3 (ng/L) for selected volatile POHCs collected on a pair of sorbent traps using a total sample volume of 20 liters or less (see Paragraph 1.1.4).

1.2.3 This method is recommended for use only by experienced sampling personnel and analytical chemists or under close supervision by such qualified persons.

1.2.4 Interferences arise primarily from background contamination of sorbent traps prior to or after use in sample collection. Many potential interferences can be due to exposure of the sorbent materials to solvent vapors prior to assembly and exposure to significant concentrations of volatile POHCs in the ambient air at hazardous waste incinerator sites.

1.2.5 To avoid or minimize the low-level contamination of train components with volatile POHCs, care should be taken to avoid contact of all interior surface or train components with synthetic organic materials (e.g., organic solvents, lubricating and sealing greases), and train components should be carefully cleaned and conditioned according to the procedures described in this protocol.

2.0 APPARATUS

2.1 Volatile Organic Sampling Train: A schematic diagram of the principal components of the VOST is shown in Figure 1 and a diagram of one acceptable version of the VOST is shown in Figure 2. The VOST consists of a glass-lined probe followed by an isolation valve, a water-cooled glass condenser, a sorbent cartridge containing Tenax (1.6 g), an empty impinger for condensate removal, a second water-cooled glass condenser, a second sorbent cartridge containing Tenax and petroleum-based charcoal (3:1 by volume; approximately 1 g of each), a silica gel drying tube, a calibrated rotameter, a sampling pump, and a dry gas meter. The gas pressure during sampling and for leak-checking is monitored by pressure gauges which are in line and downstream of the silica gel drying tube. The components of the sampling train are described below.

2.1.1 **Probe:** The probe should be made of stainless steel with a borosilicate or quartz glass liner. The temperature of the probe is to be maintained above 130°C but low enough to ensure a resin temperature of 20°C. A water-cooled probe may be required at elevated stack temperatures to protect the probe and meet the above requirements. Isokinetic sample collection is not a requirement for the use of VOST since the compounds of interest are in the vapor phase at the point of sample collection.

2.1.2 **Isolation valve:** The isolation valve should be a greaseless stopcock with a glass bore and sliding Teflon plug with Teflon wipers (Ace 8193 or equivalent).

2.1.3 **Condensers:** The condensers (Ace 5979-14 or equivalent) should be of sufficient capacity to cool the gas stream to 20°C or less prior to passage through the first sorbent cartridge. The top connection of the condenser should be able to form a leak-free, vacuum-tight seal without using sealing greases.

2.1.4 **Sorbent cartridges:**

2.1.4.1 The sorbent cartridges used for the VOST may be used in either of two configurations: the inside-outside (I/O) configuration in which the cartridge is held within an outer glass tube and in a metal carrier, and the inside-inside (I/I) configuration in which only a single glass tube is used, with or without a metal carrier. In either case, the sorbent packing will be the same.

2.1.4.1.1 The first of a pair of sorbent cartridges shall be packed with approximately 1.6 g Tenax GC resin and the second cartridge of a pair shall be packed with Tenax GC and petroleum-based charcoal (3:1 by volume; approximately 1 g of each).

2.1.4.1.2 The second sorbent cartridge shall be packed so that the sample gas stream passes through the Tenax layer first and then through the charcoal layer.

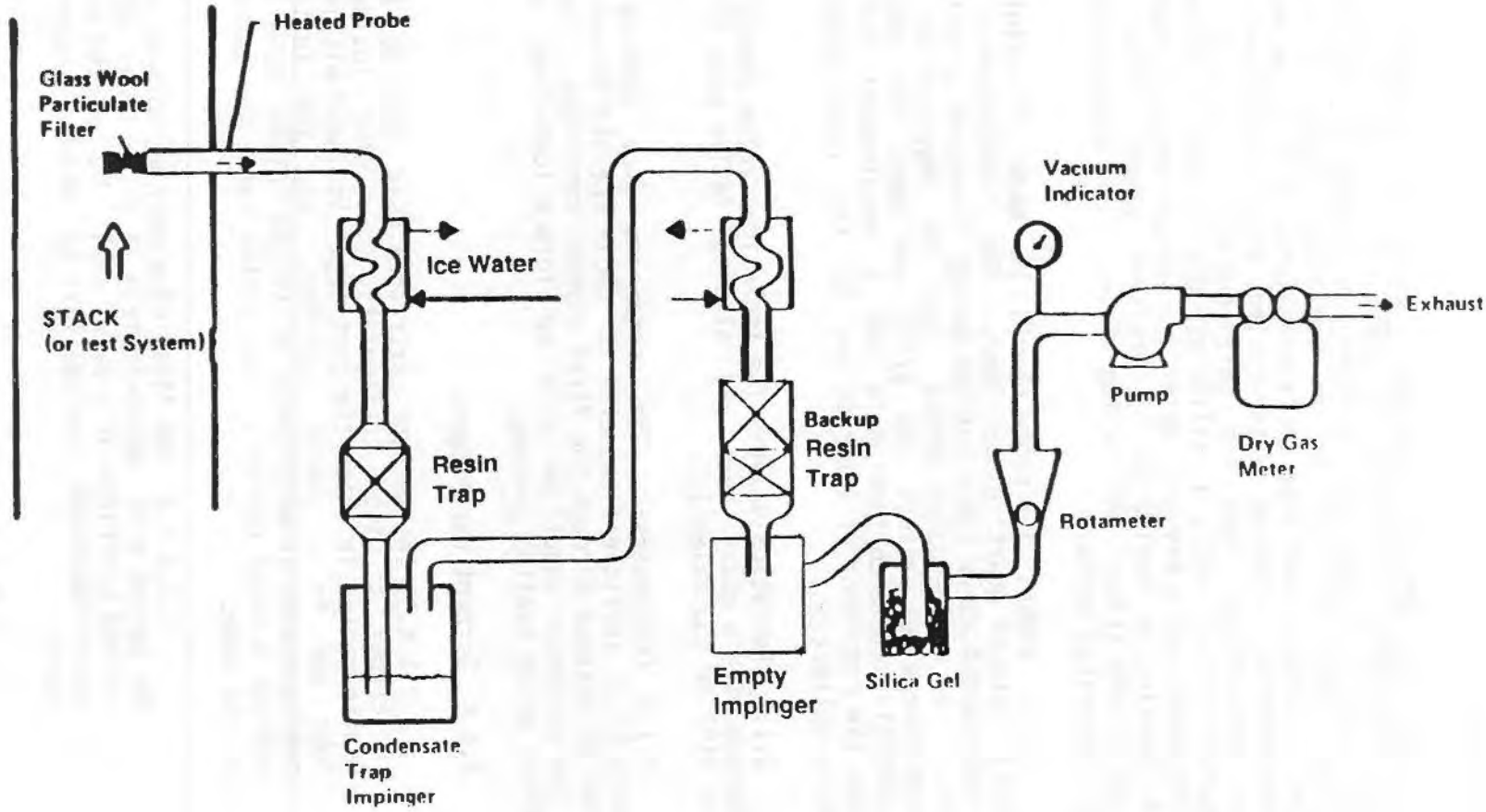


Figure 1. Schematic of Volatile Organic Sampling Train (VOST).

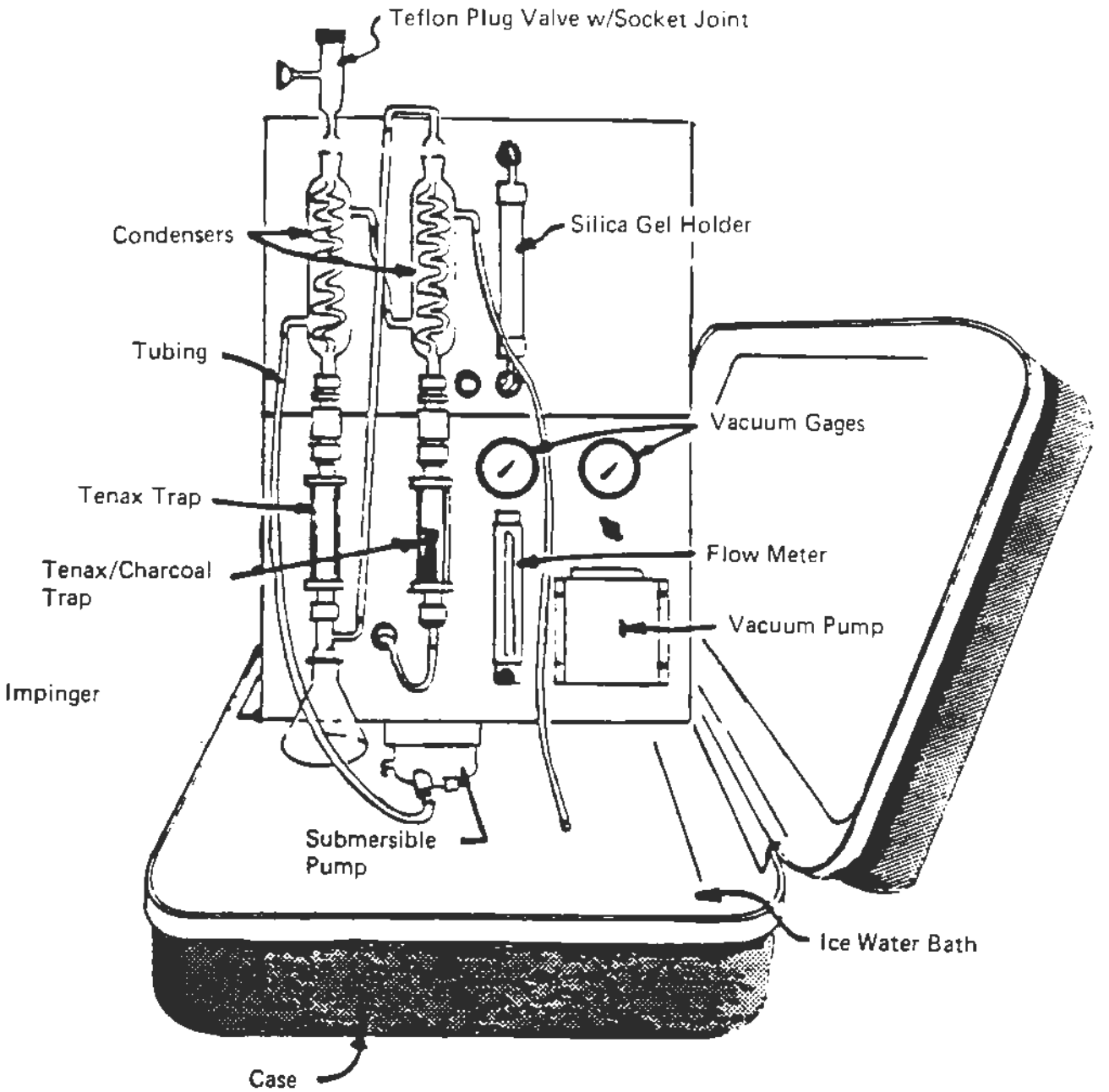


Figure 2. Volatile Organic Sampling Train (VOST).

2.1.4.2 The sorbent cartridges shall be glass tubes with approximate dimensions of 10 cm by 1.6 cm I.D. The two acceptable designs (I/O, I/I) for the sorbent cartridge are described in further detail below.

2.1.4.2.1 **Inside/Inside sorbent cartridge:** A diagram of an I/I sorbent cartridge is shown in Figure 3. This cartridge is a single glass tube (10 cm by 1.6 cm I.D.) which has the ends reduced in size to accommodate a 1/4- or 3/8-in. Swagelok or Cajon gas fitting. The resin is held in place by glass wool at each end of the resin layer. The amounts of each type of sorbent material used in the I/I design are the same as for the I/O design. Threaded end caps are placed on the sorbent cartridge after packing with sorbent to protect the sorbent from contamination during storage and transport.

2.1.4.2.2 **Inside/Outside type sorbent cartridge:** A diagram of an I/O sorbent cartridge is shown in Figure 4. In this design the sorbent materials are held in the glass tube with a fine mesh stainless steel screen and a C-clip. The glass tube is then placed within a larger diameter glass tube and held in place using Viton O-rings. The purpose of the outer glass tube is to protect the exterior of the resin-containing tube from contamination. The two glass tubes are held in a stainless steel cartridge holder, where the ends of the glass tubes are held in place by Viton O-rings placed in machine grooves in each metal end piece. The three cylindrical rods are secured in one of the metal end pieces and fastened to the other end piece using knurled nuts, thus sealing the glass tubes into the cartridge holder. The end pieces are fitted with a threaded nut onto which a threaded end cap is fitted with a Viton O-ring seal, to protect the resin from contamination during transport and storage.

2.1.5 **Metering system:** The metering system for VOST shall consist of vacuum gauges, a leak-free pump (Thomas Model 107 or equivalent, Thomas Industries, Sheboygan, Wisconsin), a calibrated rotameter (Linde Model 150, Linde Division of Union Carbide, Keasbey, New Jersey) for monitoring the gas flow rate, a dry gas meter with 2% accuracy at the required sampling rate, and related valves and equipment. Provisions should be made for monitoring the temperature of the sample gas stream between the first condenser and first sorbent cartridge. This can be done by placing a thermocouple on the exterior glass surface of the outlet from the first condenser. The temperature at that point should be less than 20°C. If it is not, an alternative condenser providing the required cooling capacity must be used.

2.1.6 **Sample transfer lines:** All sample transfer lines to connect the probe to the VOST shall be less than 5 ft in length, and shall be heat-traced Teflon with connecting fittings which are capable of forming leak-free, vacuum-tight connections without the use of sealing grease.

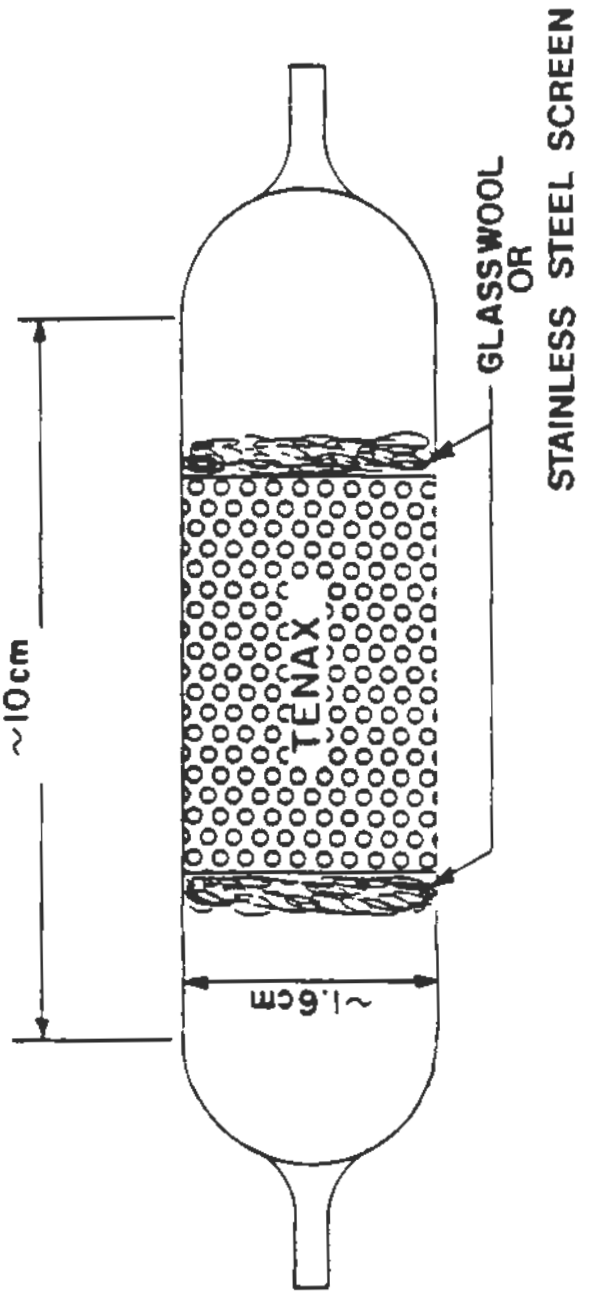
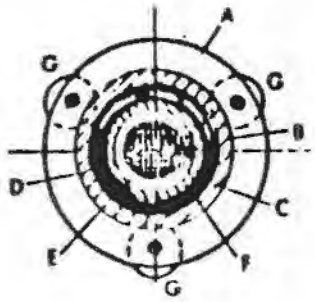


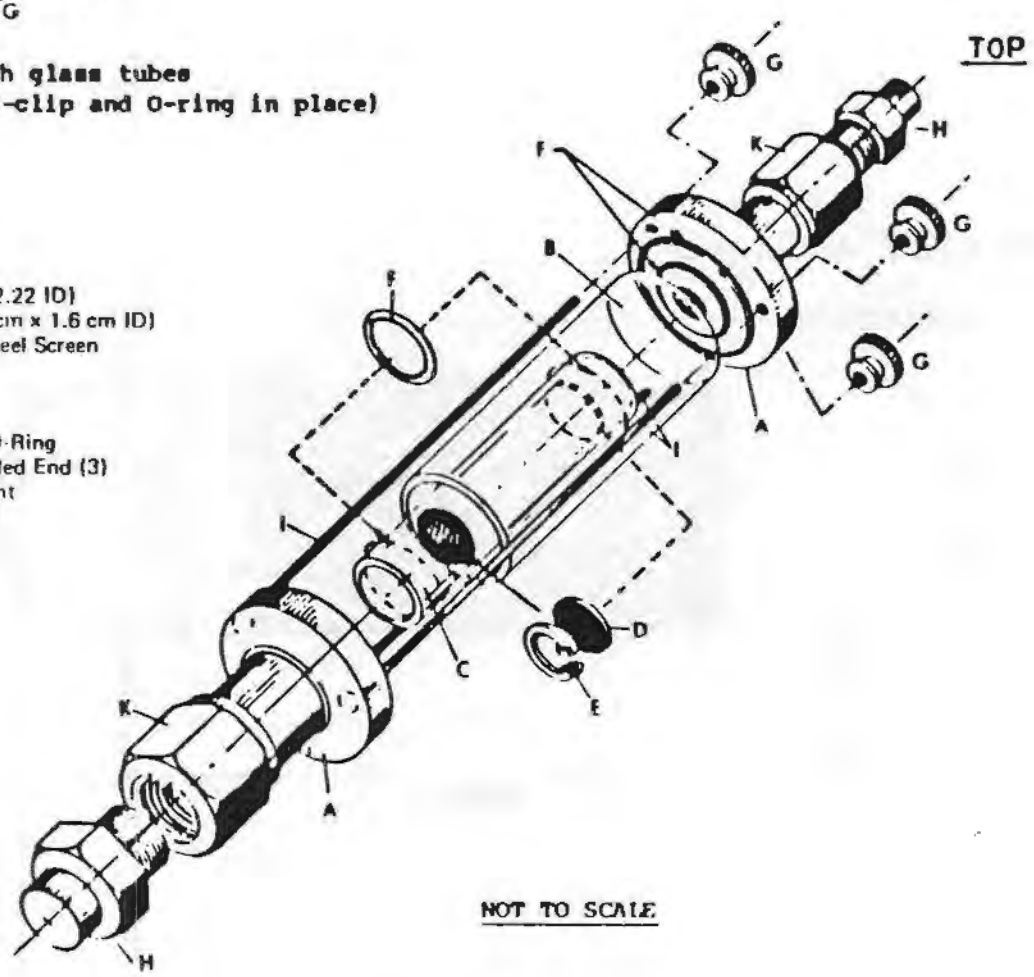
Figure 3. Inside-inside vost cartridge



Section cut through glass tubes
(showing screen, C-clip and O-ring in place)

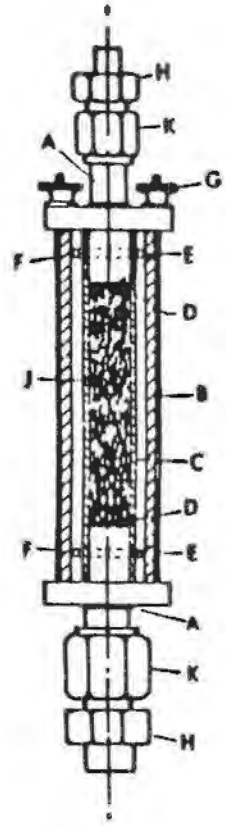
LEGEND

A - Stainless Steel Carrier
B - Glass Tube (9.84 L x 2.22 ID)
C - Small Glass Tube (10 cm x 1.6 cm ID)
D - Fine Mesh Stainless Steel Screen
E - Stainless Steel C-Clip
F - O-Ring (Viton)
G - Nuts (4)
H - End Cap with Viton O-Ring
I - Metal Rod with Threaded End (3)
J - Tenax/Charcoal Sorbent
K - Cajon Fitting



BOTTOM

NOT TO SCALE



Assembled Trap
NTS

Figure 4. Sorbent Trap Assembly (I/O)
Volatile Organic Sampling Train (VOST)

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Date September 1986

All other sample transfer lines used with the VOST shall be Teflon with connecting fittings that are capable of forming leak-free, vacuum-tight connections without the use of sealing grease.

3.0 REAGENTS AND MATERIALS

3.1 2,6-Diphenylene oxide polymer (Tenax, 35/60 mesh):

3.1.1 The new Tenax is Soxhlet extracted for 24 hr with methanol (Burdick & Jackson, pesticide grade or equivalent). The Tenax is dried for 6 hr in a vacuum oven at 50°C before use. Users of I/O and I/I sorbent cartridges have used slightly different thermal conditioning procedures. I/O sorbent cartridges packed with Tenax are thermally conditioned by flowing organic-free nitrogen (30 mL/min) through the resin while heating to 190°C. Some users have extracted new Tenax and charcoal with pentane to remove nonpolar impurities. However, these users have experienced problems with residual pentane in the sorbents during analysis.

3.1.2 If very high concentrations of volatile POHCs have been collected on the resin (e.g., micrograms of analytes), the sorbent may require Soxhlet extraction as described above. Previously used Tenax cartridges are thermally reconditioned by the method described above.

3.2 Charcoal (SKC petroleum-base or equivalent): New charcoal is prepared and charcoal is reconditioned as described in Paragraph 4.4. New charcoal does not require treatment prior to assembly into sorbent cartridges. Users of VOST have restricted the types of charcoal used in sorbent cartridges to only petroleum-based types. Criteria for other types of charcoal are acceptable if recovery of POHC in laboratory evaluations meet the criteria of 50 to 150%.

3.3 Viton-O-Ring: All O-rings used in VOST shall be Viton. Prior to use, these O-rings should be thermally conditioned at 200°C for 48 hr. O-rings should be stored in clean, screw-capped glass containers prior to use.

3.4 Glass tubes/Condensers: The glass resin tubes and condensers should be cleaned with a nonionic detergent in an ultrasonic bath, rinsed well with organic-free water, and dried at 110°C. Resin tubes of the I/O design should be assembled prior to storage as described in Paragraph 4.1. Resin tubes of the I/I design can be stored in glass culture tube containers with cotton cushioning and Teflon-lined screw caps. Condensers can be capped with appropriate end caps prior to use.

3.5 Metal parts: The stainless steel carriers, C-clips, end plugs, and screens used in the I/O VOST design are cleaned by ultrasonication in a warm nonionic detergent solution, rinsed with distilled water, air-dried, and heated in a muffle furnace for 2 hr at 400°C. Resin tubes of the I/I design require Swagelok or equivalent end caps with Supelco M-1 ferrules. These should be heated at 190°C along with the assembled cartridges.

3.6 Silica gel (Indicating type, 6-16 mesh): New silica gel may be used as received. Silica gel which has been previously used should be dried for 2 hr at 175°C (350°F).

3.7 Cold packs: Any commercially available reusable liquids or gels that can be repeatedly frozen are acceptable. They are typically sold in plastic containers as "Blue Ice" or "Ice-Packs." Enough should be used to keep cartridges at or near 4°C.

3.8 Water: Water used for cooling train components in the field may be tap water; and water used for rinsing glassware should be organic-free.

3.9 Glass wool: Glass wool should be Soxhlet extracted for 8 to 16 hr, using methanol, and oven dried at 110°C before use.

4.0 SAMPLE HANDLING AND PROCEDURE

4.1 Assembly:

4.1.1 The assembly and packing of the sorbent cartridges should be carried out in an area free of volatile organic material, preferably a laboratory in which no organic solvents are handled or stored and in which the laboratory air is charcoal filtered. Alternatively, the assembly procedures can be conducted in a glove box which can be purged with organic-free nitrogen.

4.2 Tenax cartridges:

4.2.1 The Tenax, glass tubes, and metal cartridge parts are cleaned and stored (see Section 3.0). Approximately 1.6 g of Tenax is weighed and packed into the sorbent tube which has a stainless steel screen and C-clip (I/O design) or glass wool (I/I design) in the downstream end. The Tenax is held in place by inserting a stainless steel screen and C-clips in the upstream end (I/O design) or glass wool (I/I design). Each cartridge should be marked, using an engraving tool, with an arrow to indicate the direction of sample flow, and a serial number.

4.2.2 Conditioned resin tubes of the I/O design are then assembled into the metal carriers according to the previously described inside/inside or inside/outside procedures (with end caps) and are placed on cold packs for storage and transport. Conditioned resin tubes of the I/I design are capped and placed on cold packs for storage and transport.

4.3 Tenax/Charcoal tubes

4.3.1 The Tenax, charcoal, and metal cartridge parts are cleaned and stored as previously described (see Section 3.0). The tubes are packed with approximately a 3:1 volume ratio of Tenax and charcoal (approximately 1 g each). The Tenax and charcoal are held in place by the stainless steel screens and C-clips (I/O design) or by glass wool (I/I design). The glass tubes containing the Tenax and charcoal are then

conditioned as described below (see Paragraph 4.4). Place the I/O glass tubes in the metal carriers (see Paragraph 2.1.4.2.2), put end caps on the assembled cartridges, mark direction of sample flow and serial number, and place the assembled cartridges on cold packs for storage and transport.

4.3.2 Glass tubes of the I/I design are conditioned, and stored in the same manner as the I/O tubes.

4.4 Trap Conditioning - QC

4.4.I Following assembly and leak-checking, the traps are connected in reverse direction to sampling to a source of organic-free nitrogen, and nitrogen is passed through each trap at a flow rate of 40 mL/min, while the traps are heated to 190°C for I2-28 hr. The actual conditioning period may be determined based on adequacy of the resulting blank checks.

4.4.2 The following procedure is used to blank check each set of sampling cartridges prior to sampling to ensure cleanliness. The procedure provides semi-quantitative data for organic compounds with boiling points below 110°C on Tenax and Tenax/Charcoal cartridges. It is not intended as a substitute for Method 5040.

4.4.2.1 The procedure is based on thermal desorption of each set of two cartridges, cryofocusing with liquid nitrogen onto a trap packed with glass beads, followed by thermal desorption from the trap and analysis by GC/FID.

4.4.2.2 The detection limit is based on the analysis of Tenax cartridges spiked with benzene and toluene and is around 2 ng for each compound.

4.4.2.3 The results of analyzing spiked cartridges on a daily basis should not vary by more than 20 percent. If the results are outside this range, the analytical system must be evaluated for the probable cause and a second spiked cartridge analyzed.

4.4.2.4 The GC operating conditions are as follows:

GC Operating Conditions

Column: Packed column 6 ft x 1/8" stainless steel 1.0 percent SP-1000 on Carbopack B 60/80, or equivalent.
Temperature program: 50°C for 5 min, 20°C/min increase to 190°C, hold 13 min.
Injector: 200°C.
Detector: F.I.D. 250°C.
Carrier Gas: Helium at 25 mL/min.
Sample valve: Valco 6-port with 40" x 1/16" stainless steel trap packed with 60/80 mesh glass beads.
Cryogen: Liquid nitrogen.
Trap heater: Boiling water, hot oil, or electrically heated.

Desorption heater: Supelco "clam shell" (high capacity carrier gas purifier) heater and Variac, adjusted to 180°C to 200°C.

4.4.2.5 Calibration is accomplished by preparing a spiked Tenax cartridge with benzene and toluene and analyzing according to the standard operating procedure. A standard of benzene, toluene and bromofluorobenzene (BFB) is prepared by injecting 2.0 uL of benzene and toluene and 1.0 uL of BFB into 10 mL of methanol. The concentration of this stock is 175 ng/uL of benzene and toluene, and 150 ng/uL BFB. One microliter of the stock standard is injected onto a Tenax cartridge through a heated injection port set at 150°C. A GC oven can be used for this with the oven at room temperature. Helium carrier gas is set at 50 mL/min. The solvent flush technique should be used. After two min, remove the Tenax cartridge and place in the desorption heater for analysis. BFB is also used as an internal standard spike for GC/MS analysis which provides a good comparison between GC/FID and GC/MS. The results of this spike analysis should not vary more than 20 percent day to day. Initially and then periodically this spiked Tenax should be reanalyzed a second time to verify that the 10 min desorption time and 180-200°C temperature are adequate to remove all of the spiked components. It should be noted that only one spiked Tenax cartridge need be prepared and analyzed daily unless otherwise needed to ensure proper instrument operation.

An acceptable blank level is left to the discretion of the method analyst. An acceptable level is one that allows adequate determination of expected components emitted from the waste being burned.

4.4.3 After conditioning, traps are sealed and placed on cold packs until sampling is accomplished. Conditioned traps should be held for a minimum amount of time to prevent the possibility of contamination.

4.4.4 It may be useful to spike the Tenax and Tenax/charcoal traps with the compounds of interest to ensure that they can be thermally desorbed under laboratory conditions. After spiked traps are analyzed they may be reconditioned and packed for sampling.

4.5 Pretest preparation:

4.5.1 All train components shall be cleaned and assembled as previously described. A dry gas meter shall have been calibrated within 30 days prior to use, using an EPA-supplied standard orifice.

4.5.2 The VOST is assembled according to the schematic diagram in Figure 1. The cartridges should be positioned so that sample flow is

through the Tenax first and then the Tenax/charcoal. Cooling water should be circulated to the condensers and the temperature of the cooling water should be maintained near 0°C. The end caps of the sorbent cartridges should be placed in a clean screw-capped glass container during sample collection.

4.6 Leak-checking:

4.6.1 The train is leak-checked by closing the valve at the inlet to the first condenser and pulling a vacuum of 250 mm (10 in. Hg) above the normal operating pressure. The traps and condensers are isolated from the pump and the leak rate noted. The leak rate should be less than 2.5 mm Hg after 1 min. The train is then returned to atmospheric pressure by attaching a charcoal-filled tube to the train inlet and admitting ambient air filtered through the charcoal. This procedure will minimize contamination of the VOST components by excessive exposure to the fugitive emissions at hazardous waste incinerator sites.

4.7 Sample Collection

4.7.1 After leak-checking, sample collection is accomplished by opening the valve at the inlet to the first condenser, turning on the pump, and sampling at a rate of 1 liter/min for 20 min. The volume of sample for any pair of traps should not exceed 20 liters.

4.7.2 Following collection of 20 liters of sample, the train is leak-checked a second time at the highest pressure drop encountered during the run to minimize the chance of vacuum desorption of organics from the Tenax. The train is returned to atmospheric pressure, using the method discussed in Paragraph 4.1 and the two sorbent cartridges are removed. The end caps are replaced and the cartridges shall be placed in a suitable environment for storage and transport until analysis. The sample is considered invalid if the leak test does not meet specification.

4.7.3 A new pair of cartridges is placed in the VOST, the VOST leak-checked, and the sample collection process repeated as described above. Sample collection continues until six pairs of traps have been used.

4.7.4 All sample cartridges should be kept on cold packs until they are ready for analysis.

4.8 Blanks

4.8.1 **Field blanks/trip blanks:** Blank Tenax and Tenax/charcoal cartridges are taken to the sampling site and the end caps removed for the period of time required to exchange two pairs of traps on VOST. After the two VOST traps have been exchanged, the end caps are replaced on the blank Tenax and Tenax/charcoal tubes and these are returned to the cold packs and analyzed with the sample traps. At least one pair of field blanks (one Tenax, one Tenax/charcoal) shall be included with each

six pairs of sample cartridges collected (or for each field trial using VOST to collect volatile POHCs).

4.8.2 **Trip blanks:** At least one pair of blank cartridges (one Tenax, one Tenax/charcoal) shall be included with shipment of cartridges to a hazardous waste incinerator site. These "field blanks" will be treated like any other cartridges except that the end caps will not be removed during storage at the site. This pair of traps will be analyzed to monitor potential contamination which may occur during storage and shipment.

4.8.3 **Laboratory blanks:** One pair of blank cartridges (one Tenax, one Tenax/charcoal) will remain in the laboratory using the method of storage which is used for field samples. If the field and trip blanks contain high concentrations of contaminants (e.g., greater than 2 ng of a particular POHC), the laboratory blank shall be analyzed in order to identify the source of contamination.

5.0 CALCULATIONS (for sample volume)

5.1 The following nomenclature are used in the calculation of sample volume:

P_{bar} = Barometric pressure at the exit orifice of the dry gas meter, mm (in.) Hg.

P_{std} = Standard absolute pressure, 760 mm (29.92 in.) Hg.

T_m = Dry gas meter average absolute temperature, K (°R).

T_{std} = Standard absolute temperature, 293K (528°R).

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).

γ = Dry gas meter calibration factor.

5.2 The volume of gas sampled is calculated as follows:

$$V_{m(std)} = V_m \gamma \frac{T_{std} P_{bar}}{T_m P_{std}} = K_1 \gamma \frac{V_m P_{bar}}{T_m}$$

where:

K_1 = 0.3858 K/mm Hg for metric units, or

K_1 = 17.64 °R/in. Hg for English units.

6.0 ANALYTICAL PROCEDURE

See Method 5040.

7.0 PRECISION AND ACCURACY REQUIREMENTS

7.1 Method Performance Check

Prior to field operation of the VOST at a hazardous waste incinerator, a method performance check should be conducted using either selected volatile POHCs of interest or two or more of the volatile PDHCs for which data are available. This check may be conducted on the entire system (VOST/GC/MS) by analysis of a gas cylinder containing POHCs of interest or on only the analytical system by spiking of the POHCs onto the traps. The results of this check for replicate pairs of traps should demonstrate that recovery of the analytes fall within 50% to 150% of the expected values.

7.2 Performance Audit

During a trial burn a performance audit must be completed. The audit results should agree within 50% to 150% of the expected value for each specific target compound. This audit consists of collecting a gas sample containing one or more POHCs in the VOST from an EPA ppb gas cylinder. Collection of the audit sample in the VOST may be conducted either in the laboratory or at the trial burn site. Anaysis of the VOST audit sample must be by the same person, at the same time, and with the same analytical procedure as used for the regular VOST trail burn samples. EPA ppb gas cylinders currently available for VOST Audit are shown in Table 1 below.

The audit procedure, audit equipment and audit cylinder may be obtained by writing:

Audit Cylinder Gas Coordinator (MD-77B)
Quality Assurance Division
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

or by calling the Audit Cylinder Gas Coordinator at (919) 541-4531.

The request for the audit must be made at least 30 days prior to the scheduled trial burn. If a POHC is selected for which EPA does not have an audit cylinder, this audit is not required.

8.0 REFERENCES

1. Protocol for the Collection and Analysis of Volatile POHCs Using VOST. EPA/600/8-84/007, March 1984.

2. Sykes, A.L., Standard Operating Procedure for Blanking Tenax and Tenax/Charcoal Sampling Cartridges for Volatile Organic Sampling Train (VOST), Radion Corporation, P.O. Box 13000, Research Triangle Park, NC 27709.

3. Validation of the Volatile Organic Sampling Train (VOST) Protocol, Vols. I and II, EPA/600/4-86/014a, January 1986.

TABLE 1: Organic Gases in the ppb Audit Repository

Group I	Ranges of cylinders currently available:
5 Organics in N_2:	7 - 90 ppb
Carbon tetrachloride	90 - 430 ppb
Chloroform	430 - 10,000 ppb
Perchloroethylene	
Vinyl chloride	
Benzene	

Group II	Ranges of cylinders currently available:
9 Organics in N_2	7 - 90 ppb
Trichloroethylene	90 - 430 ppb
1,2-Dichloroethane	
1,2-Dibromoethane	
F-12	
F-11	
Bromomethane	
Methyl ethyl ketone	
1,1,1-Trichloroethane	
Acetronitrile	

TABLE 1: Organic Gases in the ppb Audit Repository (Continued)

Group III	Ranges of cylinders currently available:
7 Organics in N_2:	7 - 90 ppb
Vinylidene chloride	90 - 430 ppb
F-113	
F-114	
Acetone	
1,4-Dioxane	
Toluene	
Chlorobenzene	

Group IV	Ranges of cylinders currently available:
6 Organics in N_2:	7 - 90 ppb
Acrylonitrile	430 - 10,000
1,3-Butadiene	
Ethylene oxide	
Methylene chloride	
Propylene oxide	
Ortho-xylene	

E-3 SW846-METHOD 0010

Modified Method 5 Sampling Train

METHOD 0010

MODIFIED METHOD 5 SAMPLING TRAIN

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of semivolatile Principal Organic Hazardous Compounds (POHCs) from incineration systems (PHS, 1967). This method also may be used to determine particulate emission rates from stationary sources as per EPA Method 5 (see References at end of this method).

2.0 SUMMARY OF METHOD

2.1 Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. Principal components of the train include a high-efficiency glass- or quartz-fiber filter and a packed bed of porous polymeric adsorbent resin. The filter is used to collect organic-laden particulate materials and the porous polymeric resin to adsorb semivolatile organic species. Semivolatile species are defined as compounds with boiling points >100°C.

2.2 Comprehensive chemical analyses of the collected sample are conducted to determine the concentration and identity of the organic materials.

3.0 INTERFERENCES

3.1 Oxides of nitrogen (NO_x) are possible interferents in the determination of certain water-soluble compounds such as dioxane, phenol, and urethane; reaction of these compounds with NO_x in the presence of moisture will reduce their concentration. Other possibilities that could result in positive or negative bias are (1) stability of the compounds in methylene chloride, (2) the formation of water-soluble organic salts on the resin in the presence of moisture, and (3) the solvent extraction efficiency of water-soluble compounds from aqueous media. Use of two or more ions per compound for qualitative and quantitative analysis can overcome interference at one mass. These concerns should be addressed on a compound-by-compound basis before using this method.

4.0 APPARATUS AND MATERIALS

4.1 Sampling train:

4.1.1 A schematic of the sampling train used in this method is shown in Figure 1. This sampling train configuration is adapted from EPA Method 5 procedures, and, as such, the majority of the required equipment

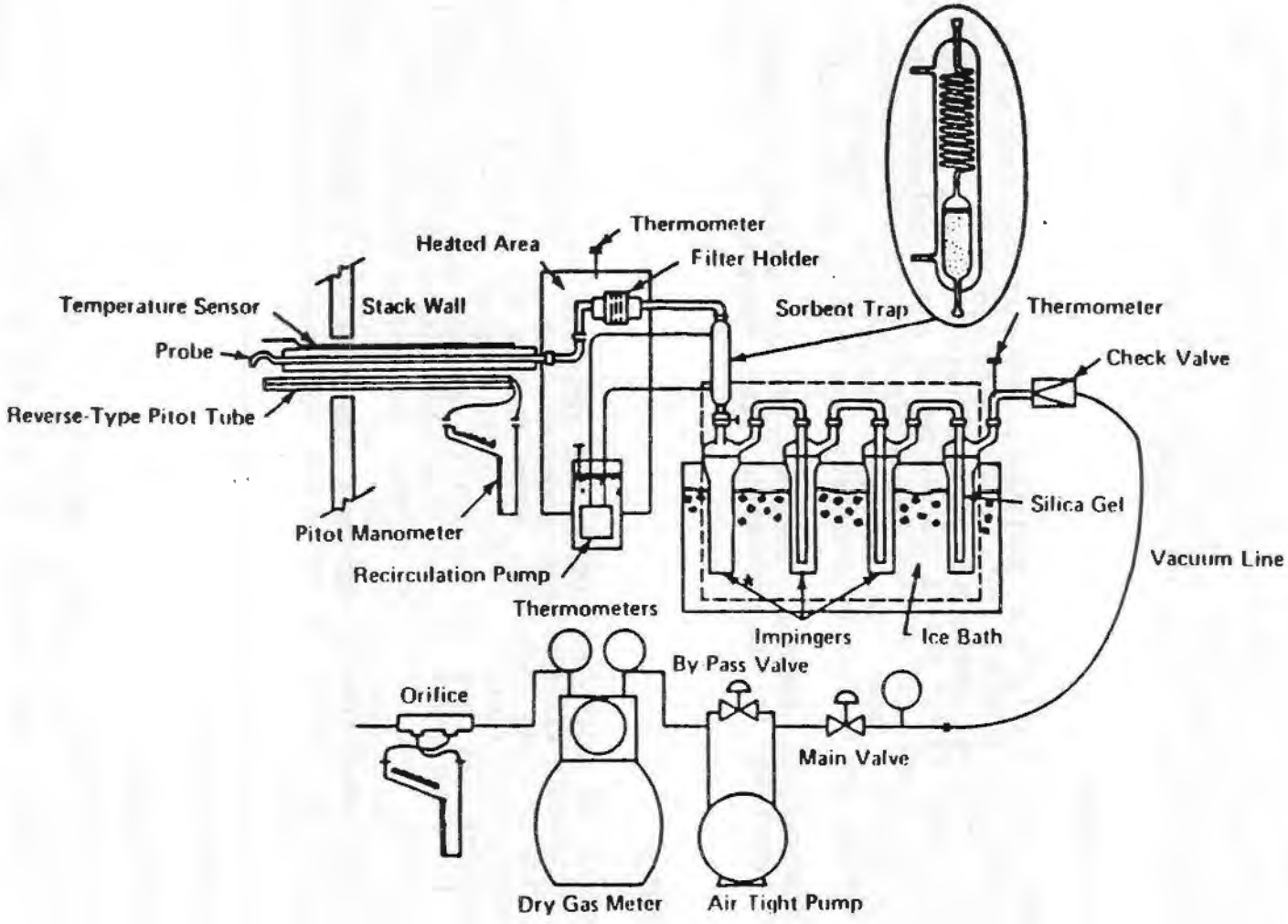

Figure 1. Modified Method 5 Sampling Train.

Revision 0
Date September 1986

is identical to that used in EPA Method 5 determinations. The new components required are a condenser coil and a sorbent module, which are used to collect semivolatile organic materials that pass through the glass- or quartz-fiber filter in the gas phase.

4.1.2 Construction details for the basic train components are given in APTD-0581 (see Martin, 1971, in Section 13.0, References); commercial models of this equipment are also available. Specifications for the sorbent module are provided in the following subsections. Additionally, the following subsections list changes to APTD-0581 and identify allowable train configuration modifications.

4.1.3 Basic operating and maintenance procedures for the sampling train are described in APTD-0576 (see Rom, 1972, in Section 13.0, References). As correct usage is important in obtaining valid results, all users should refer to APTD-0576 and adopt the operating and maintenance procedures outlined therein unless otherwise specified. The sampling train consists of the components detailed below.

4.1.3.1 Probe nozzle: Stainless steel (316) or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant I.D. The nozzle shall be buttonhook or elbow design and constructed from seamless tubing (if made of stainless steel). Other construction materials may be considered for particular applications. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.16 cm (1/16 in.), e.g., 0.32-1.27 cm (1/8-1/2 in.), or larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Paragraph 9.1.

4.1.3.2 Probe liner: Borosilicate or quartz-glass tubing with a heating system capable of maintaining a gas temperature of 120 ± 14°C (248 ± 25°F) at the exit end during sampling. (The tester may opt to operate the equipment at a temperature lower than that specified.) Because the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) are considered acceptable. Either borosilicate or quartz-glass probe liners may be used for stack temperatures up to about 480°C (900°F). Quartz liners shall be used for temperatures between 480 and 900°C (900 and 1650°F). (The softening temperature for borosilicate is 820°C (1508°F), and for quartz 1500°C (2732°F).) Water-cooling of the stainless steel sheath will be necessary at temperatures approaching and exceeding 500°C.

4.1.3.3 Pitot tube: Type S, as described in Section 2.1 of EPA Method 2, or other appropriate devices (Vollaro, 1976). The pitot tube shall be attached to the probe to allow constant monitoring of the stack-gas velocity. The impact (high-pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see EPA Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of EPA Method 2.

4.1.3.4 Differential pressure gauge: Inclined manometer or equivalent device as described in Section 2.2 of EPA Method 2. One manometer shall be used for velocity-head (ΔP) readings and the other for orifice differential pressure (ΔH) readings.

4.1.3.5 Filter holder: Borosilicate glass, with a glass frit filter support and a sealing gasket. The sealing gasket should be made of materials that will not introduce organic material into the gas stream at the temperature at which the filter holder will be maintained. The gasket shall be constructed of Teflon or materials of equal or better characteristics. The holder design shall provide a positive seal against leakage at any point along the filter circumference. The holder shall be attached immediately to the outlet of the cyclone or cyclone bypass.

4.1.3.6 Filter heating system: Any heating system capable of maintaining a temperature of 120 $\pm$ 14°C (248 $\pm$ 25°F) around the filter holder during sampling. Other temperatures may be appropriate for particular applications. Alternatively, the tester may opt to operate the equipment at temperatures other than that specified. A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

4.1.3.7 Organic sampling module: This unit consists of three sections, including a gas-conditioning section, a sorbent trap, and a condensate knockout trap. The gas-conditioning system shall be capable of conditioning the gas leaving the back half of the filter holder to a temperature not exceeding 20°C (68°F). The sorbent trap shall be sized to contain approximately 20 g of porous polymeric resin (Rohm and Haas XAD-2 or equivalent) and shall be jacketed to maintain the internal gas temperature at 17 $\pm$ 3°C (62.5 $\pm$ 5.4°F). The most commonly used coolant is ice water from the impinger ice-water bath, constantly circulated through the outer jacket, using rubber or plastic tubing and a peristaltic pump. The sorbent trap should be outfitted with a glass well or depression, appropriately sized to accommodate a small thermocouple in the trap for monitoring the gas entry temperature. The condensate knockout trap shall be of sufficient size to collect the condensate following gas conditioning. The organic module components shall be oriented to direct the flow of condensate formed vertically downward from the conditioning section, through the adsorbent media, and into the condensate knockout trap. The knockout trap is usually similar in appearance to an empty impinger directly underneath the sorbent module; it may be oversized but should have a shortened center stem (at a minimum, one-half the length of the normal impinger stems) to collect a large volume of condensate without bubbling and overflowing into the impinger train. All surfaces of the organic module wetted by the gas sample shall be fabricated of borosilicate glass, Teflon, or other inert materials. Commercial versions of the

complete organic module are not currently available, but may be assembled from commercially available laboratory glassware and a custom-fabricated sorbent trap. Details of two acceptable designs are shown in Figures 2 and 3 (the thermocouple well is shown in Figure 2).

4.1.3.8 Impinger train: To determine the stack-gas moisture content, four 500-mL impingers, connected in series with leak-free ground-glass joints, follow the knockout trap. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm (1/2-in.) I.D. glass tube extending about 1.3 cm (1/2 in.) from the bottom of the outer cylinder. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of water or appropriate trapping solution. The third shall be empty or charged with a caustic solution, should the stack gas contain hydrochloric acid (HCl). The fourth shall contain a known weight of silica gel or equivalent desiccant.

4.1.3.9 Metering system: The necessary components are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment, as shown in Figure 1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry-gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems capable of maintaining sampling rates within 10% of isokineticity and of determining sample volumes to within 2% may be used. The metering system must be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates. Sampling trains using metering systems designed for flow rates higher than those described in APTD-0581 and APTD-0576 may be used, provided that the specifications of this method are met.

4.1.3.10 Barometer: Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30-m (100 ft) elevation increase (vice versa for elevation decrease).

4.1.3.11 Gas density determination equipment: Temperature sensor and pressure gauge (as described in Sections 2.3 and 2.4 of EPA Method 2), and gas analyzer, if necessary (as described in EPA Method 3). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal.

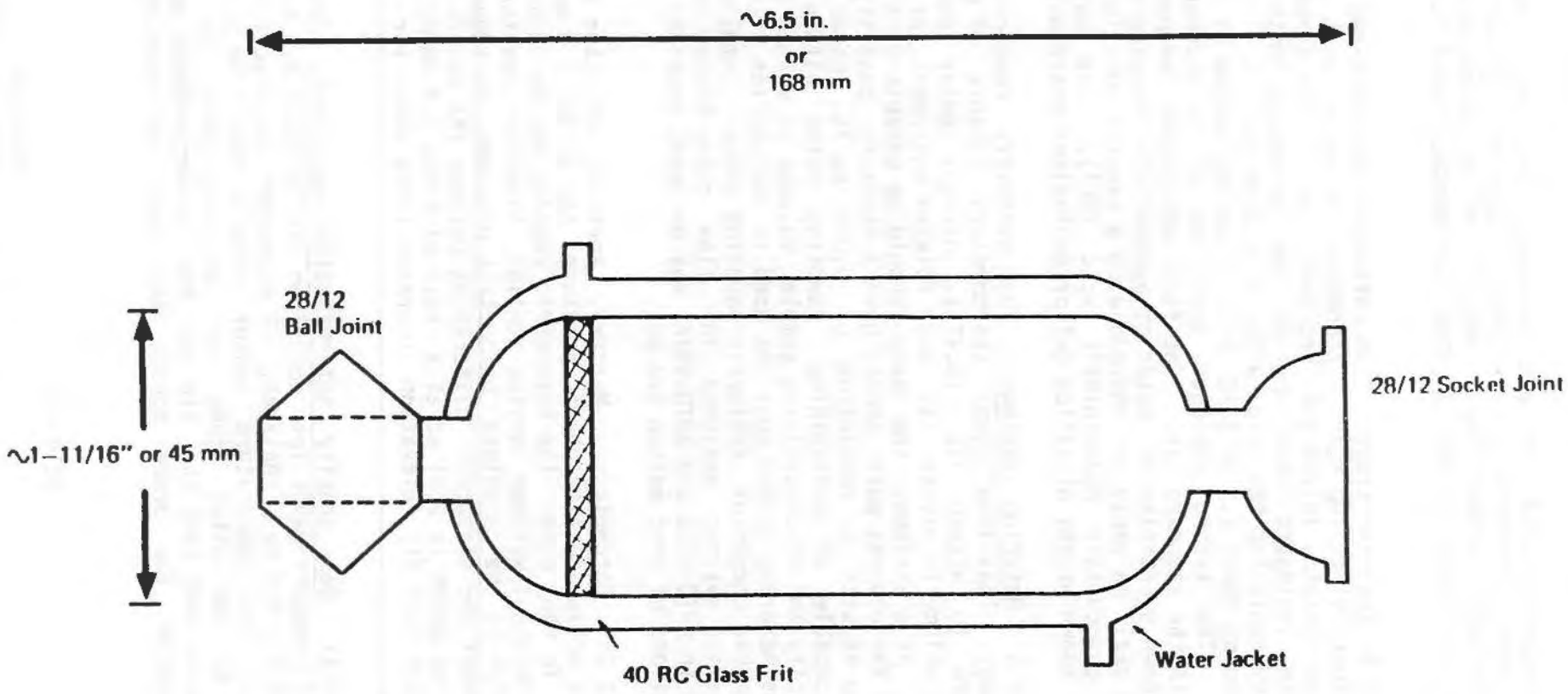

Figure 2. Adsorbent Sampling System.

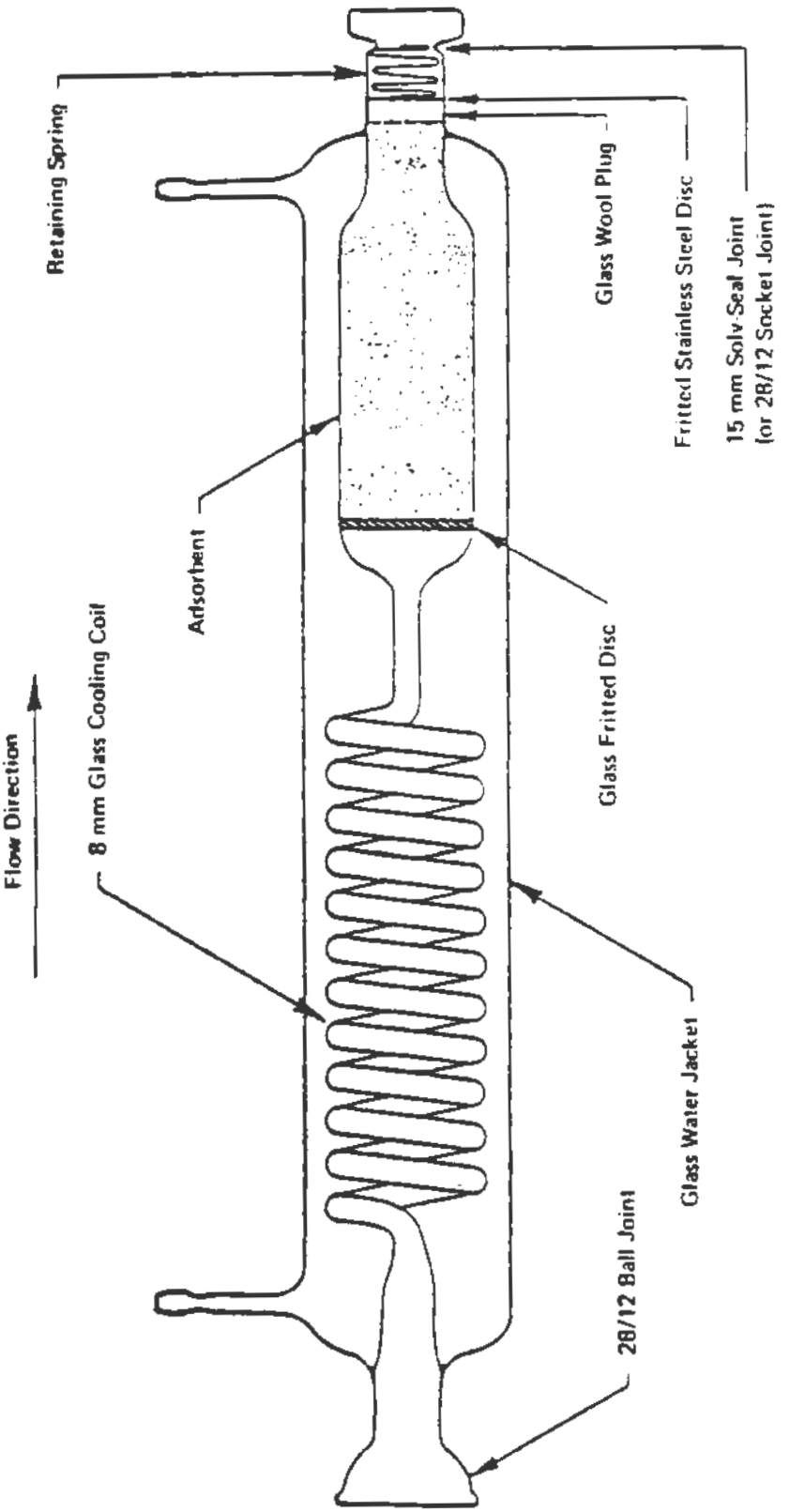

Figure 3. Adsorbent Sampling System.

Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see EPA Method 2, Figure 2-7). As a second alternative, if a difference of no more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube.

4.1.3.12 Calibration/field-preparation record: A permanently bound laboratory notebook, in which duplicate copies of data may be made as they are being recorded, is required for documenting and recording calibrations and preparation procedures (i.e., filter and silica gel tare weights, clean XAD-2, quality assurance/quality control check results, dry-gas meter, and thermocouple calibrations, etc.). The duplicate copies should be detachable and should be stored separately in the test program archives.

4.2 Sample Recovery:

4.2.1 **Probe liner:** Probe nozzle and organic module conditioning section brushes; nylon bristle brushes with stainless steel wire handles are required. The probe brush shall have extensions of stainless steel, Teflon, or inert material at least as long as the probe. The brushes shall be properly sized and shaped to brush out the probe liner, the probe nozzle, and the organic module conditioning section.

4.2.2 **Wash bottles:** Three. Teflon or glass wash bottles are recommended; polyethylene wash bottles should not be used because organic contaminants may be extracted by exposure to organic solvents used for sample recovery.

4.2.3 **Glass sample storage containers:** Chemically resistant, borosilicate amber and clear glass bottles, 500-mL or 1,000-mL. Bottles should be tinted to prevent action of light on sample. Screw-cap liners shall be either Teflon or constructed so as to be leak-free and resistant to chemical attack by organic recovery solvents. Narrow-mouth glass bottles have been found to exhibit less tendency toward leakage.

4.2.4 **Petri dishes:** Glass, sealed around the circumference with wide (1-in.) Teflon tape, for storage and transport of filter samples.

4.2.5 **Graduated cylinder and/or balances:** To measure condensed water to the nearest 1 mL or 1 g. Graduated cylinders shall have subdivisions not >2 mL. Laboratory triple-beam balances capable of weighing to ± 0.5 g or better are required.

4.2.6 **Plastic storage containers:** Screw-cap polypropylene or polyethylene containers to store silica gel.

4.2.7 **Funnel and rubber policeman:** To aid in transfer of silica gel to container (not necessary if silica gel is weighed in field).

4.2.8 **Funnels:** Glass, to aid in sample recovery.

4.3 Filters: Glass- or quartz-fiber filters, without organic binder, exhibiting at least 99.95% efficiency (<0.05% penetration) on 0.3-um dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3. Reeve Angel 934 AH or Schleicher and Schwell #3 filters work well under these conditions.

4.4 Crushed ice: Quantities ranging from 10-50 lb may be necessary during a sampling run, depending on ambient air temperature.

4.5 Stopcock grease: Solvent-insoluble, heat-stable silicone grease. Use of silicone grease upstream of the module is not permitted, and amounts used on components located downstream of the organic module shall be minimized. Silicone grease usage is not necessary if screw-on connectors and Teflon sleeves or ground-glass joints are used.

4.6 Glass wool: Used to plug the unfritted end of the sorbent module. The glass-wool fiber should be solvent-extracted with methylene chloride in a Soxhlet extractor for 12 hr and air-dried prior to use.

5.0 REAGENTS

5.1 Adsorbent resin: Porous polymeric resin (XAD-2 or equivalent) is recommended. These resins shall be cleaned prior to their use for sample collection. Appendix A of this method should be consulted to determine appropriate precleaning procedure. For best results, resin used should not exhibit a blank of higher than 4 mg/kg of total chromatographable organics (TCO) (see Appendix B) prior to use. Once cleaned, resin should be stored in an airtight, wide-mouth amber glass container with a Teflon-lined cap or placed in one of the glass sorbent modules tightly sealed with Teflon film and elastic bands. The resin should be used within 4 wk of the preparation.

5.2 Silica gel: Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hr before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

5.3 Impinger solutions: Distilled organic-free water (Type II) shall be used, unless sampling is intended to quantify a particular inorganic gaseous species. If sampling is intended to quantify the concentration of additional species, the impinger solution of choice shall be subject to Administrator approval. This water should be prescreened for any compounds of interest. One hundred mL will be added to the specified impinger; the third impinger in the train may be charged with a basic solution (1 N sodium hydroxide or sodium acetate) to protect the sampling pump from acidic gases. Sodium acetate should be used when large sample volumes are anticipated because sodium hydroxide will react with carbon dioxide in aqueous media to form sodium carbonate, which may possibly plug the impinger.

5.4 Sample recovery reagents:

5.4.1 Methylene chloride: Distilled-in-glass grade is required for sample recovery and cleanup (see Note to 5.4.2 below).

5.4.2 Methyl alcohol: Distilled-in-glass grade is required for sample recovery and cleanup.

NOTE: Organic solvents from metal containers may have a high residue blank and should not be used. Sometimes suppliers transfer solvents from metal to glass bottles; thus blanks shall be run prior to field use and only solvents with low blank value (<0.001%) shall be used.

5.4.3 Water: Water (Type II) shall be used for rinsing the organic module and condenser component.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Because of complexity of this method, field personnel should be trained in and experienced with the test procedures in order to obtain reliable results.

6.2 Laboratory preparation:

6.2.1 All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified.

6.2.2 Weigh several 200- to 300-g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus containers. As an alternative to preweighing the silica gel, it may instead be weighed directly in the impinger or sampling holder just prior to train assembly.

6.2.3 Check filters visually against light for irregularities and flaws or pinhole leaks. Label the shipping containers (glass Petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

6.2.4 Desiccate the filters at 20 + 5.6°C (68 + 10°F) and ambient pressure for at least 24 hr, and weigh at intervals of at least 6 hr to a constant weight (i.e., <0.5-mg change from previous weighing), recording results to the nearest 0.1 mg. During each weighing the filter must not be exposed for more than a 2-min period to the laboratory atmosphere and relative humidity above 50%. Alternatively (unless otherwise specified by the Administrator), the filters may be oven-dried at 105°C (220°F) for 2-3 hr, desiccated for 2 hr, and weighed.

6.3 Preliminary field determinations:

6.3.1 Select the sampling site and the minimum number of sampling points according to EPA Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and range of velocity heads using EPA Method 2. It is recommended that a leak-check of the pitot lines (see EPA Method 2, Section 3.1) be performed. Determine the stack-gas moisture content using EPA Approximation Method 4 or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack-gas dry molecular weight, as described in EPA Method 2, Section 3.6. If integrated EPA Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

6.3.2 Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of EPA Method 2).

6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

6.3.4 A minimum of 3 dscm (105.9 dscf) of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of POHCs from incineration systems. Additional sample volume shall be collected as necessitated by analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Section 10.0.

6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by EPA Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus one-half min.

6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-sample volumes. In these cases, the Administrator's approval must first be obtained.

6.4 Preparation of collection train:

6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon film or aluminum foil until just prior to assembly or until sampling is about to begin.

6.4.2 Fill the sorbent trap section of the organic module with approximately 20 g of clean adsorbent resin. While filling, ensure that the trap packs uniformly, to eliminate the possibility of channeling. When freshly cleaned, many adsorbent resins carry a static charge, which will cause clinging to trap walls. This may be minimized by filling the trap in the presence of an antistatic device. Commercial antistatic devices include Model-204 and Model-210 manufactured by the 3M Company, St. Paul, Minnesota.

6.4.3 If an impinger train is used to collect moisture, place 100 mL of water in each of the first two impingers, leave the third impinger empty (or charge with caustic solution, as necessary), and transfer approximately 200-300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

6.4.4 Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

6.4.5 When glass liners are used, install the selected nozzle using a Viton-A O-ring when stack temperatures are <260°C (500°F) and a woven glass-fiber gasket when temperatures are higher. See APTD-0576 (Rom, 1972) for details. Other connecting systems utilizing either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above, or by a leak-free direct mechanical connection. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

6.4.6 Set up the train as in Figure 1. During assembly, do not use any silicone grease on ground-glass joints that are located upstream of the organic module. A very light coating of silicone grease may be used on all ground-glass joints that are located downstream of the organic module, but it should be limited to the outer portion (see APTD-0576) of the ground-glass joints to minimize silicone-grease contamination. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and the filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack. The organic module condenser must be maintained at a temperature of 17 $\pm$ 3°C. Connect all temperature sensors to an appropriate potentiometer/display unit. Check all temperature sensors at ambient temperature.

6.4.7 Place crushed ice around the impingers and the organic module condensate knockout.

6.4.8 Turn on the sorbent module and condenser coil coolant recirculating pump and begin monitoring the sorbent module gas entry temperature. Ensure proper sorbent module gas entry temperature before proceeding and again before any sampling is initiated. It is extremely important that the XAD-2 resin temperature never exceed 50°C (122°F), because thermal decomposition will occur. During testing, the XAD-2 temperature must not exceed 20°C (68°F) for efficient capture of the semivolatile species of interest.

6.4.9 Turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize.

6.5 Leak-check procedures

6.5.1 Pre-test leak-check:

6.5.1.1 Because the number of additional intercomponent connections in the Semi-VOST train (over the M5 Train) increases the possibility of leakage, a pre-test leak-check is required.

6.5.1.2 After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 381-mm Hg (15-in. Hg) vacuum.
(NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.)

6.5.1.3 If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first attaching a carbon-filled leak-check impinger (shown in Figure 4) to the inlet of the filter holder (cyclone, if applicable) and then plugging the inlet and pulling a 381-mm Hg (15-in. Hg) vacuum. (Again, a lower vacuum may be used, provided that it is not exceeded during the test.) Then, connect the probe to the train and leak-check at about 25-mm Hg (1-in. Hg) vacuum; alternatively, leak-check the probe with the rest of the sampling train in one step at 381-mm Hg (15-in. Hg) vacuum. Leakage rates in excess of 4% of the average sampling rate or >0.00057 m^3/min (0.02 cfm), whichever is less, are unacceptable.

6.5.1.4 The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with fine-adjust valve fully open and coarse-adjust valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do not reverse direction of the fine-adjust valve; this will cause water to back up into the organic module. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check, as shown below, and start over.

CROSS SECTIONAL VIEW

Leak Testing Apparatus

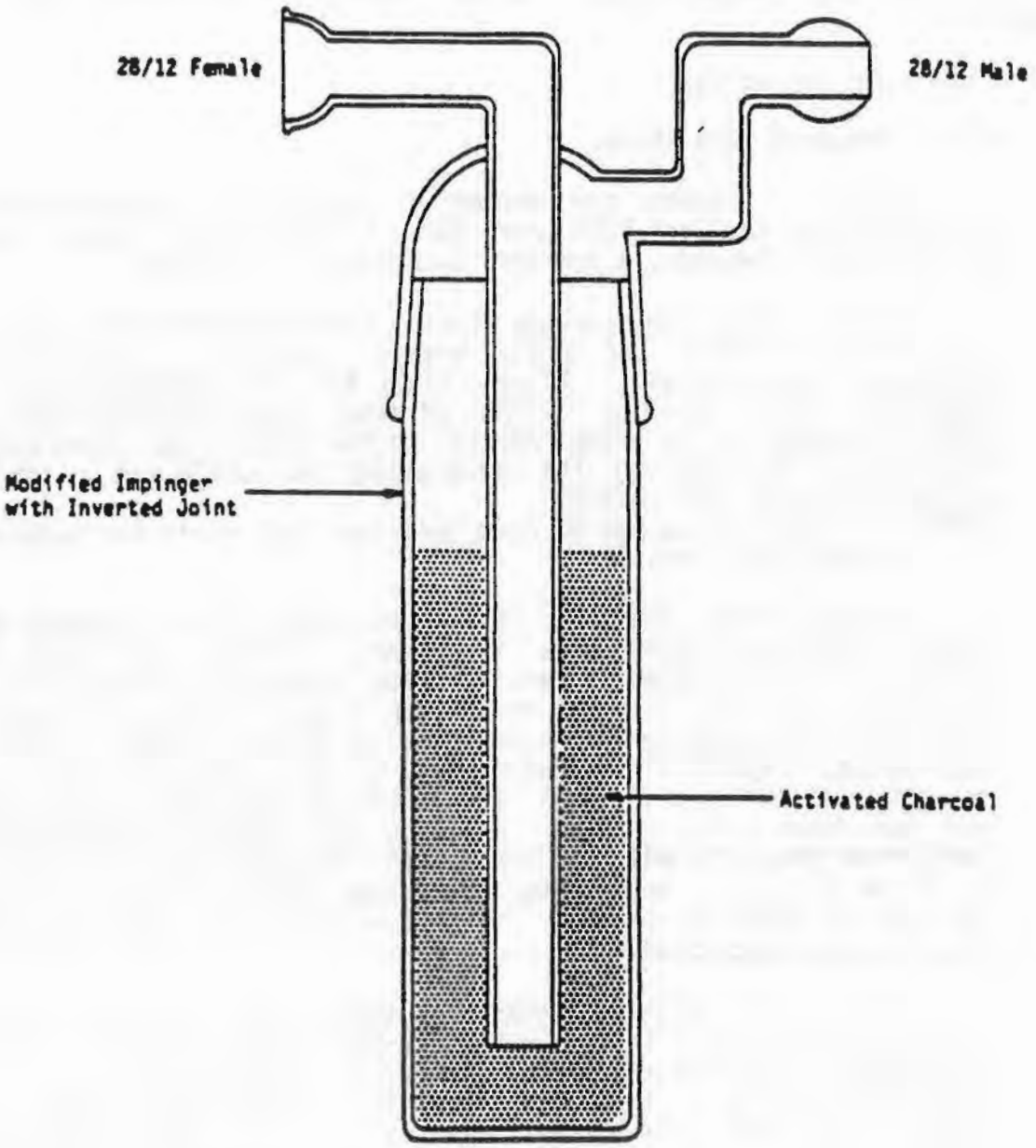

Figure 4. Leak-check impinger.

6.5.1.5 When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable). When the vacuum drops to 127 mm (5 in.) Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed. This prevents the water in the impingers from being forced backward into the organic module and silica gel from being entrained backward into the third impinger.

6.5.2 Leak-checks during sampling run:

6.5.2.1 If, during the sampling run, a component (e.g., filter assembly, impinger, or sorbent trap) change becomes necessary, a leak-check shall be conducted immediately after the interruption of sampling and before the change is made. The leak-check shall be done according to the procedure outlined in Paragraph 6.5.1, except that it shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If a higher leakage rate is obtained, the tester shall void the sampling run. (It should be noted that any "correction" of the sample volume by calculation by calculation reduces the integrity of the pollutant concentrations data generated and must be avoided.)

6.5.2.2 Immediately after a component change, and before sampling is reinitiated, a leak-check similar to a pre-test leak-check must also be conducted.

6.5.3 Post-test leak-check:

6.5.3.1 A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done with the same procedures as those with the pre-test leak-check, except that it shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate, correct the sample volume (as shown in the calculation section of this method), and consider the data obtained of questionable reliability, or void the sampling run.

6.6 Sampling-train operation:

6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, unless otherwise specified by the Administrator. Maintain a temperature around the filter of 120 ± 14°C (248 ± 25°F) and a gas temperature entering the sorbent trap at a maximum of 20°C (68°F).

6.6.2 For each run, record the data required on a data sheet such as the one shown in Figure 5. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made before and after each leak-check, and when sampling is halted. Take other readings required by Figure 5 at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity-head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

6.6.3 Clean the stack access ports prior to the test run to eliminate the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are at the specified temperature, and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point, with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations, are available. These nomographs are designed for use when the Type S pitot-tube coefficient is 0.84 $\pm$ 0.02 and the stack-gas equivalent density (dry molecular weight) is equal to 29 $\pm$ 4. APTD-0576 details the procedure for using the nomographs. If the stack-gas molecular weight and the pitot-tube coefficient are outside the above ranges, do not use the nomographs unless appropriate steps (Shigehara, 1974) are taken to compensate for the deviations.

6.6.4 When the stack is under significant negative pressure (equivalent to the height of the impinger stem), take care to close the coarse-adjust valve before inserting the probe into the stack, to prevent water from backing into the organic module. If necessary, the pump may be turned on with the coarse-adjust valve closed.

6.6.5 When the probe is in position, block off the openings around the probe and stack access port to prevent unrepresentative dilution of the gas stream.

6.6.6 Traverse the stack cross section, as required by EPA Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the access port, in order to minimize the chance of extracting deposited material.

6.6.7 During the test run, make periodic adjustments to keep the temperature around the filter holder and the organic module at the proper levels; add more ice and, if necessary, salt to maintain a temperature of <20°C (68°F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

Plant _____

Location _____

Operator _____

Date _____

Run No. _____

Sample Box No. _____

Meter Box No. _____

Meter $H@$ _____

C Factor _____

Pitot Tube Coefficient C_p _____

Schematic of Stack Cross Section

Ambient Temperature _____

Barometric Pressure _____

Assumed Moisture % _____

Probe Length, m (ft) _____

Nozzle Identification No. _____

Average Calibrated Nozzle Diameter, cm (in) _____

Probe Heater Setting _____

Leak Rate, m^3/min, (cfm) _____

Probe Liner Material _____

Static Pressure, mm Hg (in. Hg) _____

Filter No. _____

Traverse Point Number	Sampling Time (θ) min.	Vacuum mm Hg (in. Hg)	Stack Temperature (T_s) °C(°F)	Velocity Head ($\sqrt{P_s}$) mm (in) H_2O	Pressure Differential Across Orifice Meter mm (H_2O) (in H_2O)	Gas Sample Volume m_3 (ft_3)	Gas Sample Temp. at Dry Gas Meter Inlet °C(°F)	Gas Sample Temp. at Dry Gas Meter Outlet °C(F°)	Filter Holder Temperature °C(F°)	Temperature of Gas Entering Sorbet Trap °C(F°)	Temperature of Gas Leaving Condenser or Last Impinger
Total							Avg.	Avg.			
Average											

Figure 5. Particulate field data.

6.6.8 If the pressure drop across the filter or sorbent trap becomes too high, making isokinetic sampling difficult to maintain, the filter/sorbent trap may be replaced in the midst of a sample run. Using another complete filter holder/sorbent trap assembly is recommended, rather than attempting to change the filter and resin themselves. After a new filter/sorbent trap assembly is installed, conduct a leak-check. The total particulate weight shall include the summation of all filter assembly catches.

6.6.9 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

6.6.10 Note that when two or more trains are used, separate analysis of the front-half (if applicable) organic-module and impinger (if applicable) catches from each train shall be performed, unless identical nozzle sizes were used on all trains. In that case, the front-half catches from the individual trains may be combined (as may the impinger catches), and one analysis of front-half catch and one analysis of impinger catch may be performed.

6.6.11 At the end of the sample run, turn off the coarse-adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry-gas meter reading, and conduct a post-test leak-check. Also, leak-check the pitot lines as described in EPA Method 2. The lines must pass this leak-check in order to validate the velocity-head data.

6.6.12 Calculate percent isokineticity (see Section 10.8) to determine whether the run was valid or another test run should be made.

7.0 SAMPLE RECOVERY

7.1 Preparation:

7.1.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the tip to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling down because this will create a vacuum in the filter holder, drawing water from the impingers into the sorbent module.

7.1.2 Before moving the sample train to the cleanup site, remove the probe from the sample train and cap the open outlet, being careful not to lose any condensate that might be present. Cap the filter inlet.

Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the organic module and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the organic module.

7.1.3 Cap the filter-holder outlet and the inlet to the organic module. Separate the sorbent trap section of the organic module from the condensate knockout trap and the gas-conditioning section. Cap all organic module openings. Disconnect the organic-module knockout trap from the impinger train inlet and cap both of these openings. Ground-glass stoppers, Teflon caps, or caps of other inert materials may be used to seal all openings.

7.1.4 Transfer the probe, the filter, the organic-module components, and the impinger/condenser assembly to the cleanup area. This area should be clean and protected from the weather to minimize sample contamination or loss.

7.1.5 Save a portion of all washing solutions (methanol/methylene chloride, Type II water) used for cleanup as a blank. Transfer 200 mL of each solution directly from the wash bottle being used and place each in a separate, prelabeled glass sample container.

7.1.6 Inspect the train prior to and during disassembly and note any abnormal conditions.

7.2 Sample containers:

7.2.1 **Container no. 1:** Carefully remove the filter from the filter holder and place it in its identified Petri dish container. Use a pair or pairs of tweezers to handle the filter. If it is necessary to fold the filter, ensure that the particulate cake is inside the fold. Carefully transfer to the Petri dish any particulate matter or filter fibers that adhere to the filter-holder gasket, using a dry nylon bristle brush or sharp-edged blade, or both. Label the container and seal with 1-in.-wide Teflon tape around the circumference of the lid.

7.2.2 **Container no. 2:** Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components first with methanol/methylene chloride (1:1 v/v) into a glass container. Distilled water may also be used. Retain a water and solvent blank and analyze in the same manner as with the samples. Perform rinses as follows:

7.2.2.1 Carefully remove the probe nozzle and clean the inside surface by rinsing with the solvent mixture (1:1 v/v methanol/-methylene chloride) from a wash bottle and brushing with a nylon bristle brush. Brush until the rinse shows no visible particles; then make a final rinse of the inside surface with the solvent mix. Brush and rinse the inside parts of the Swagelok fitting with the solvent mix in a similar way until no visible particles remain.

7.2.2.2 Have two people rinse the probe liner with the solvent mix by tilting and rotating the probe while squirting solvent into its upper end so that all inside surfaces will be wetted with solvent. Let the solvent drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container.

7.2.2.3 Follow the solvent rinse with a probe brush. Hold the probe in an inclined position and squirt solvent into the upper end while pushing the probe brush through the probe with a twisting action; place a sample container underneath the lower end of the probe and catch any solvent and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the solvent or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above-prescribed manner at least six times (metal probes have small crevices in which particulate matter can be entrapped). Rinse the brush with solvent and quantitatively collect these washings in the sample container. After the brushing, make a final solvent rinse of the probe as described above.

7.2.2.4 It is recommended that two people work together to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

7.2.2.5 Clean the inside of the front half of the filter holder and cyclone/cyclone flask, if used, by rubbing the surfaces with a nylon bristle brush and rinsing with methanol/methylene chloride (1:1 v/v) mixture. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone and cyclone flask (if applicable). Brush and rinse any particulate material adhering to the inner surfaces of these components into the front-half rinse sample. After all solvent washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that solvent will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents.

7.2.3 **Container no. 3:** The sorbent trap section of the organic module may be used as a sample transport container, or the spent resin may be transferred to a separate glass bottle for shipment. If the sorbent trap itself is used as the transport container, both ends should be sealed with tightly fitting caps or plugs. Ground-glass stoppers or Teflon caps may be used. The sorbent trap should then be labeled, covered with aluminum foil, and packaged on ice for transport to the laboratory. If a separate bottle is used, the spent resin should be quantitatively transferred from the trap into the clean bottle. Resin that adheres to the walls of the trap should be recovered using a rubber policeman or spatula and added to this bottle.

7.2.4 **Container no. 4:** Measure the volume of condensate collected in the condensate knockout section of the organic module to within ±1 mL by using a graduated cylinder or by weighing to within ±0.5 g using a triple-beam balance. Record the volume or weight of liquid present and note any discoloration or film in the liquid catch. Transfer this liquid to a prelabeled glass sample container. Inspect the back half of the filter housing and the gas-conditioning section of the organic module. If condensate is observed, transfer it to a graduated or weighing bottle and measure the volume, as described above. Add this material to the condensate knockout-trap catch.

7.2.5 **Container no. 5:** All sampling train components located between the high-efficiency glass- or quartz-fiber filter and the first wet impinger or the final condenser system (including the heated Teflon line connecting the filter outlet to the condenser) should be thoroughly rinsed with methanol/methylene chloride (1:1 v/v) and the rinsings combined. This rinse shall be separated from the condensate. If the spent resin is transferred from the sorbent trap to a separate sample container for transport, the sorbent trap shall be thoroughly rinsed until all sample-wetted surfaces appear clean. Visible films should be removed by brushing. Whenever train components are brushed, the brush should be subsequently rinsed with solvent mixture and the rinsings added to this container.

7.2.6 **Container no. 6:** Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere strongly to the impinger wall. Because the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the container and its contents to 0.5 g or better.

7.3 Impinger water:

7.3.1 Make a notation of any color or film in the liquid catch. Measure the liquid in the first three impingers to within ±1 mL by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

7.3.2 Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Paragraph 4.1.3.7). Amber glass containers should be used for storage of impinger catch, if required.

7.3.3 If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

7.4 Sample preparation for shipment: Prior to shipment, recheck all sample containers to ensure that the caps are well secured. Seal the lids of all containers around the circumference with Teflon tape. Ship all liquid samples upright on ice and all particulate filters with the particulate catch facing upward. The particulate filters should be shipped unrefrigerated.

8.0 ANALYSIS

8.1 Sample preparation:

8.1.1 General: The preparation steps for all samples will result in a finite volume of concentrated solvent. The final sample volume (usually in the 1- to 10-mL range) is then subjected to analysis by GC/MS. All samples should be inspected and the appearance documented. All samples are to be spiked with surrogate standards as received from the field prior to any sample manipulations. The spike should be at a level equivalent to 10 times the MDL when the solvent is reduced in volume to the desired level (i.e., 10 mL). The spiking compounds should be the stable isotopically labeled analog of the compounds of interest or a compound that would exhibit properties similar to the compounds of interest, be easily chromatographed, and not interfere with the analysis of the compounds of interest. Suggested surrogate spiking compounds are: deuterated naphthalene, chrysene, phenol, nitrobenzene, chlorobenzene, toluene, and carbon-13-labeled pentachlorophenol.

8.1.2 Condensate: The "condensate" is the moisture collected in the first impinger following the XAD-2 module. Spike the condensate with the surrogate standards. The volume is measured and recorded and then transferred to a separatory funnel. The pH is to be adjusted to pH 2 with 6 N sulfuric acid, if necessary. The sample container and graduated cylinder are sequentially rinsed with three successive 10-mL aliquots of the extraction solvent and added to the separatory funnel. The ratio of solvent to aqueous sample should be maintained at 1:3. Extract the sample by vigorously shaking the separatory funnel for 5 min. After complete separation of the phases, remove the solvent and transfer to a Kuderna-Danish concentrator (K-D), filtering through a bed of precleaned, dry sodium sulfate. Repeat the extraction step two additional times. Adjust the pH to 11 with 6 N sodium hydroxide and reextract combining the acid and base extracts. Rinse the sodium sulfate into the K-D with fresh solvent and discard the desiccant. Add Teflon boiling chips and concentrate to 10 mL by reducing the volume to slightly less than 10 mL and then bringing to volume with fresh solvent. In order to achieve the necessary detection limit, the sample volume can be further reduced to 1 mL by using a micro column K-D or nitrogen blow-down. Should the sample start to exhibit precipitation, the concentration step should be stopped and the sample redissolved with fresh solvent taking the volume to some finite amount. After adding a standard (for the purpose of quantitation by GC/MS), the sample is ready for analysis, as discussed in Paragraph 8.2.

8.1.3 **Impinger:** Spike the sample with the surrogate standards; measure and record the volume and transfer to a separatory funnel. Proceed as described in Paragraph 8.1.2.

8.1.4 **XAD-2:** Spike the resin directly with the surrogate standards. Transfer the resin to the all-glass thimbles by the following procedure (care should be taken so as not to contaminate the thimble by touching it wi[illegible] anything other than tweezers or other solvent-rinsed mechanical holding devices). Suspend the XAD-2 module directly over the thimble. The glass frit of the module (see Figure 2) should be in the up position. The thimble is contained in a clean beaker, which will serve to catch the solvent rinses. Using a Teflon squeeze bottle, flush the XAD-2 into the thimble. Thoroughly rinse the glass module with solvent into the beaker containing the thimble. Add the XAD-2 glass-wool plug to the thimble. Cover the XAD-2 in the thimble with a precleaned glass-wool plug sufficient to prevent the resin from floating into the solvent reservoir of the extractor. If the resin is wet, effective extraction can be accomplished by loosely packing the resin in the thimble. If a question arises concerning the completeness of the extraction, a second extraction, without a spike, is advised. The thimble is placed in the extractor and the rinse solvent contained in the beaker is added to the solvent reservoir. Additional solvent is added to make the reservoir approximately two-thirds full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle 5-6 times per hr. Extract the resin for 16 hr. Transfer the solvent and three 10-mL rinses of the reservoir to a K-D and concentrate as described in Paragraph 8.1.2.

8.1.5 **Particulate filter (and cyclone catch):** If particulate loading is to be determined, weigh the filter (and cyclone catch, if appl[illegible]). The particulate filter (and cyclone catch, if applicable) is t[illegible]red to the glass thimble and extracted simultaneously with the XAD-2 resin.

8.1.6 **Train solvent rinses:** All train rinses (i.e., probe, impinger, filter housing) using the extraction solvent and methanol are returned to the laboratory as a single sample. If the rinses are contained in more than one container, the intended spike is divided equally among the containers proportioned from a single syringe volume. Transfer the rinse to a separatory funnel and add a sufficient amount of organic-free water so that the methylene chloride becomes immiscible and its volume no longer increases with the addition of more water. The extraction and concentration steps are then performed as described in Paragraph 8.1.2.

8.2 Sample analysis:

8.2.1 The primary analytical tool for the measurement of emissions from hazardous waste incinerators is GC/MS using fused-silica capillary GC columns, as described in Method 8270 in Chapter Four of this manual. Because of the nature of GC/MS instrumentation and the cost associated

with sample analysis, prescreening of the sample extracts by gas chromatography/flame ionization detection (GC/FID) or with electron capture (GC/ECD) is encouraged. Information regarding the complexity and concentration level of a sample prior to GC/MS analysis can be of enormous help. This information can be obtained by using either capillary columns or less expensive packed columns. However, the FID screen should be performed with a column similar to that used with the GC/MS. Keep in mind that GC/FID has a slightly lower detection limit than GC/MS and, therefore, that the concentration of the sample can be adjusted either up or down prior to analysis by GC/MS.

8.2.2 The mass spectrometer will be operated in a full scan (40-450) mode for most of the analyses. The range for which data are acquired in a GC/MS run will be sufficiently broad to encompass the major ions, as listed in Chapter Four, Method 8270, for each of the designated POHCs in an incinerator effluent analysis.

8.2.3 For most purposes, electron ionization (EI) spectra will be collected because a majority of the POHCs give reasonable EI spectra. Also, EI spectra are compatible with the NBS Library of Mass Spectra and other mass spectral references, which aid in the identification process for other components in the incinerator process streams.

8.2.4 To clarify some identifications, chemical ionization (CI) spectra using either positive ions or negative ions will be used to elucidate molecular-weight information and simplify the fragmentation patterns of some compounds. In no case, however, should CI spectra alone be used for compound identification. Refer to Chapter Four, Method 8270, for complete descriptions of GC conditions, MS conditions, and quantitative and quantitative identification.

9.0 CALIBRATION

9.1 Probe nozzle: Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make measurements at three separate places across the diameter and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

9.2 Pitot tube: The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of EPA Method 2, or assigned a nominal coefficient of 0.84 if it is not visibly nicked, dented, or corroded and if it meets design and intercomponent spacing specifications.

9.3 Metering system:

9.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: Make a 10-min calibration run at 0.00057 m^3/min (0.02 cfm); at the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

9.3.2 After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). The vacuum shall be set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

9.3.3 Leak-check of metering system: That portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 6): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13-18 cm (5-7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks, if present, must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by >5%, either the test series shall be voided or calculations for test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

9.4 Probe heater: The probe-heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

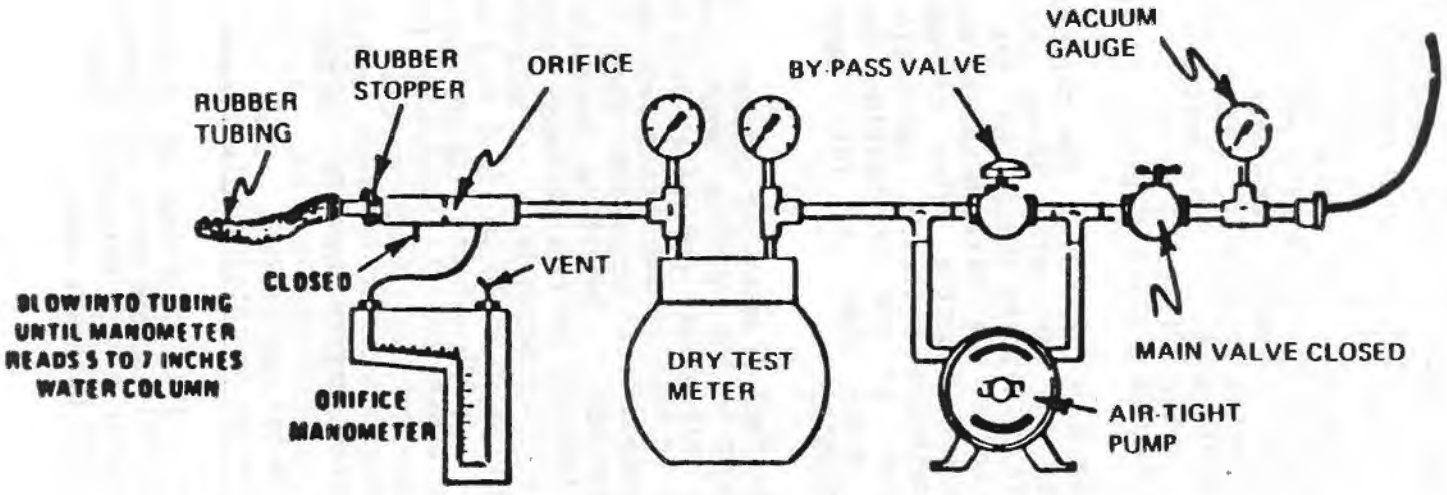

Figure 6. Leak-check of meter box.

9.5 Temperature gauges: Each thermocouple must be permanently and uniquely marked on the casting; all mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at ambient air temperatures, with and without the extension lead, must be noted and recorded. Correction is necessary if the use of an extension lead produces a change >1.5%.

9.5.1 Impinger, organic module, and dry-gas meter thermocouples: For the thermocouples used to measure the temperature of the gas leaving the impinger train and the XAD-2 resin bed, three-point calibration at ice-water, room-air, and boiling-water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to ±2°C (3.6°F) with those of the absolute value of the reference thermometer.

9.5.2 Probe and stack thermocouple: For the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice-water, boiling-water, and hot-oil-bath temperatures must be performed; it is recommended that room-air temperature be added, and that the thermometer and the thermocouple agree to within 1.5% at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

9.6 Barometer: Adjust the barometer initially and before each test series to agree to within ±25 mm Hg (0.1 in. Hg) of the mercury barometer or the corrected barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

Triple-beam balance: Calibrate the triple-beam balance before each test series, using Class-S standard weights; the weights must be within ±0.5% of the standards, or the balance must be adjusted to meet these limits.

10.0 CALCULATIONS

10.1 Carry out calculations. Round off figures after the final calculation to the correct number of significant figures.

10.2 Nomenclature:

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_d = Type S pitot tube coefficient (nominally 0.84 ± 0.02), dimensionless.

I = Percent of isokinetic sampling.

L_a = Maximum acceptable leakage rate for a leak-check, either pre-test or following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4% of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak-check conducted prior to the "i^{th}" component change (i = 1, 2, 3...n) m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).

M_d = Stack-gas dry molecular weight, g/g-mole (lb/lb-mole).

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s = Absolute stack-gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m^3/K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole).

T_m = Absolute average dry-gas meter temperature (see Figure 6), K (°R).

T_s = Absolute average stack-gas temperature (see Figure 6), K (°R).

T_{std} = Standard absolute temperature, 293K (528°R).

V_{lc} = Total volume of liquid collected in the organic module condensate knockout trap, the impingers, and silica gel, mL.

V_m = Volume of gas sample as measured by dry-gas meter, dscm (dscf).

$V_{m(std)}$ = Volume of gas sample measured by the dry-gas meter, corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

V_s = Stack-gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).

W_a = Weight of residue in acetone wash, mg.

γ = Dry-gas-meter calibration factor, dimensionless.

ΔH = Average pressure differential across the orifice meter (see Figure 2), mm H_2O (in. H_2O).

ρ_w = Density of water, 0.9982 g/mL (0.002201 lb/mL).

θ = Total sampling time, min.

θ_1 = Sampling time interval from the beginning of a run until the first component change, min.

θ_i = Sampling time interval between two successive component changes, beginning with the interval between the first and second changes, min.

θ_p = Sampling time interval from the final (n^{th}) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

10.3 Average dry-gas-meter temperature and average orifice pressure drop: See data sheet (Figure 5, above).

10.4 Dry-gas volume: Correct the sample measured by the dry-gas meter to standard conditions (20°C, 760 mm Hg [68°F, 29.92 in. Hg]) by using Equation 1:

$$V_{m(std)} = V_m \gamma \frac{T_{std}}{T_m} \frac{P_{bar} + \Delta H/13.6}{P_{std}} = K_1 V_m \gamma \frac{P_{bar} + \Delta H/13.6}{T_m} \qquad (1)$$

where:

K_1 = 0.3858 K/mm Hg for metric units, or
K_1 = 17.64°R/in. Hg for English units.

It should be noted that Equation 1 can be used as written, unless the leakage rate observed during any of the mandatory leak-checks (i.e., the post-test leak-check or leak-checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 1 must be modified as follows:

a. Case I (no component changes made during sampling run): Replace V_m in Equation 1 with the expression:

$$V_m - (L_p - L_a)$$

b. Case II (one or more component changes made during the sampling run): Replace V_m in Equation 1 by the expression:

$$V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\theta_1 - (L_p - L_a)\theta_p$$

and substitute only for those leakage rates (L_1 or L_p) that exceed L_a.

10.5 Volume of water vapor:

$$V_{w(std)} = V_{lc} \frac{P_w}{M_w} \frac{RT_{std}}{P_{std}} = K_2 V_{lc} \qquad (2)$$

where:

K_2 = 0.001333 m^3/mL for metric units, or
K_2 = 0.04707 ft^3/mL for English units.

10.6 Moisture content:

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad (3)$$

NOTE: In saturated or water-droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 3) and a second from the assumption of saturated conditions. The lower of the two values of B_w shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note to Section 1.2 of Method 4. For the purposes of this method, the average stack-gas temperature from Figure 6 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ±1°C (2°F).

10.7 Conversion factors:

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.31

10.8 Isokinetic variation:

10.8.1 Calculation from raw data:

$$I = \frac{100\ T_s[K_3 F_{lc} + (V_m/T_m)\ (P_{bar} + \Delta H/13.6)]}{608 V_s P_s A_n} \qquad (4)$$

where:

K_3 = 0.003454 mm Hg-m^3/mL-K for metric units, or
K_3 = 0.002669 in. Hg-ft^3/mL-°R for English units.

10.8.2 Calculation for intermediate values:

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} V_s \theta A_n P_s 60(1-B_{ws})} \qquad (5)$$

$$= K_4 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta (1-B_{ws})}$$

where:

K_4 = 4.320 for metric units, or
K_4 = 0.09450 for English units.

10.8.3 Acceptable results: If 90% $\leq$ I $\leq$ 110%, the results are acceptable. If the results are low in comparison with the standard and I is beyond the acceptable range, or if I is less than 90%, the Administrator may opt to accept the results.

10.9 To determine the minimum sample volume that shall be collected, the following sequence of calculations shall be used.

10.9.1 From prior analysis of the waste feed, the concentration of POHCs introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the maximum amount of POHC allowed to be present in the effluent. This may be expressed as:

$$\frac{(WF)\ (POHC_1\ conc)}{100} \quad \frac{(100-\%DRE)}{100} = \text{Max } POHC_1 \text{ Mass} \qquad (6)$$

where:

WF = mass flow rate of waste feed per hr, g/hr (lb/hr).

$POHC_1$ = concentration of Principal Organic Hazardous Compound (wt %) introduced into the combustion process.

DRE = percent Destruction and Removal Efficiency required.

Max POHC = mass flow rate (g/hr [lb/hr]) of POHC emitted from the combustion source.

10.9.2 The average discharge concentration of the POHC in the effluent gas is determined by comparing the Max POHC with the volumetric flow rate being exhausted from the source. Volumetric flow rate data are available as a result of preliminary Method 1-4 determinations:

$$\frac{\text{Max POHC}_1 \text{ Mass}}{DV_{eff(std)}} = \text{Max POHC}_1 \text{ conc} \qquad (7)$$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf).

$POHC_1$ conc = anticipated concentration of the POHC in the exhaust gas stream, g/dscm (lb/dscf).

10.9.3 In making this calculation, it is recommended that a safety margin of at least ten be included:

$$\frac{LDL_{POHC} \times 10}{POHC_1 \text{ conc}} = V_{TBC} \qquad (8)$$

where:

LDL_{POHC} = detectable amount of POHC in entire sampling train.
NOTE: The whole extract from an XAD-2 cartridge is seldom if ever, injected at once. Therefore, if aliquoting factors are involved, the LDL_{POHC} is not the same as the analytical (or column) detection limit.

V_{TBC} = minimum dry standard volume to be collected at dry-gas meter.

10.10 Concentration of any given POHC in the gaseous emissions of a combustion process:

1) Multiply the concentration of the POHC as determined in Method 8270 by the final concentration volume, typically 10 mL.

C_{POHC} (ug/mL) x sample volume (mL) = amount (ug) of POHC in sample (9)

where:

C_{POHC} = concentration of POHC as analyzed by Method 8270.

2) Sum the amount of POHC found in all samples associated with a single train.

Total (ug) = XAD-2 (ug) + condensate (ug) + rinses (ug) + impinger (ug) (10)

3) Divide the total ug found by the volume of stack gas sampled (m^3).

(Total ug)/(train sample volume) = concentration of POHC (ug/m^3) (11)

11.0 QUALITY CONTROL

11.1 Sampling: See EPA Manual 600/4-77-027b for Method 5 quality control.

11.2 Analysis: The quality assurance program required for this study includes the analysis of field and method blanks, procedure validations, incorporation of stable labeled surrogate compounds, quantitation versus stable labeled internal standards, capillary column performance checks, and external performance tests. The surrogate spiking compounds selected for a particular analysis are used as primary indicators of the quality of the analytical data for a wide range of compounds and a variety of sample matrices. The assessment of combustion data, positive identification, and quantitation of the selected compounds are dependent on the integrity of the samples received and the precision and accuracy of the analytical methods employed. The quality assurance procedures for this method are designed to monitor the performance of the analytical method and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

11.2.1 **Field Blanks:** Field blanks must be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters, and resin cartridges. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling area, and leak-checked at the beginning and end of the testing (or for the same total number of times as the actual test train). The filter housing and probe of the blank train will be heated during the sample test. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the sampling train.

11.2.2 **Method blanks:** A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

11.2.3 Refer to Method 8270 for additional quality control considerations.

12.0 METHOD PERFORMANCE

12.1 Method performance evaluation: Evaluation of analytical procedures for a selected series of compounds must include the sample-preparation procedures and each associated analytical determination. The analytical procedures should be challenged by the test compounds spiked at appropriate levels and carried through the procedures.

12.2 Method detection limit: The overall method detection limits (lower and upper) must be determined on a compound-by-compound basis because different compounds may exhibit different collection, retention, and extraction efficiencies as well as instrumental minimum detection limit (MDL). The method detection limit must be quoted relative to a given sample volume. The upper limits for the method must be determined relative to compound retention volumes (breakthrough).

12.3 Method precision and bias: The overall method precision and bias must be determined on a compound-by-compound basis at a given concentration level. The method precision value would include a combined variability due to sampling, sample preparation, and instrumental analysis. The method bias would be dependent upon the collection, retention, and extraction efficiency of the train components. From evaluation studies to date using a dynamic spiking system, method biases of -13% and -16% have been determined for toluene and 1,1,2,2-tetrachloroethane, respectively. A precision of 19.9% was calculated from a field test data set representing seven degrees of freedom which resulted from a series of paired, unspiked Semivolatile Organic Sampling trains (Semi-VOST) sampling emissions from a hazardous waste incinerator.

13.0 REFERENCES

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METHOD 0010, APPENDIX A

PREPARATION OF XAD-2 SORBENT RESIN

1.0 SCOPE AND APPLICATION

1.1 XAD-2 resin as supplied by the manufacturer is impregnated with a bicarbonate solution to inhibit microbial growth during storage. Both the salt solution and any residual extractable monomer and polymer species must be removed before use. The resin is prepared by a series of water and organic extractions, followed by careful drying.

2.0 EXTRACTION

2.1 Method 1: The procedure may be carried out in a giant Soxhlet extractor. An all-glass thimble containing an extra-coarse frit is used for extraction of XAD-2. The frit is recessed 10-15 mm above a crenellated ring at the bottom of the thimble to facilitate drainage. The resin must be carefully retained in the extractor cup with a glass-wool plug and stainless steel screen because it floats on methylene chloride. This process involves sequential extraction in the following order.

Solvent	Procedure
Water	Initial rinse: Place resin in a beaker, rinse once with Type II water, and discard. Fill with water a second time, let stand overnight, and discard.
Water	Extract with H_2O for 8 hr.
Methyl alcohol	Extract for 22 hr.
Methylene chloride	Extract for 22 hr.
Methylene chloride (fresh)	Extract for 22 hr.

2.2 Method 2:

2.2.1 As an alternative to Soxhlet extraction, a continuous extractor has been fabricated for the extraction sequence. This extractor has been found to be acceptable. The particular canister used for the apparatus shown in Figure A-1 contains about 500 g of finished XAD-2. Any size may be constructed; the choice is dependent on the needs of the sampling programs. The XAD-2 is held under light spring tension between a pair of coarse and fine screens. Spacers under the bottom screen allow for even distribution of clean solvent. The three-necked flask should be of sufficient size (3-liter in this case) to hold solvent

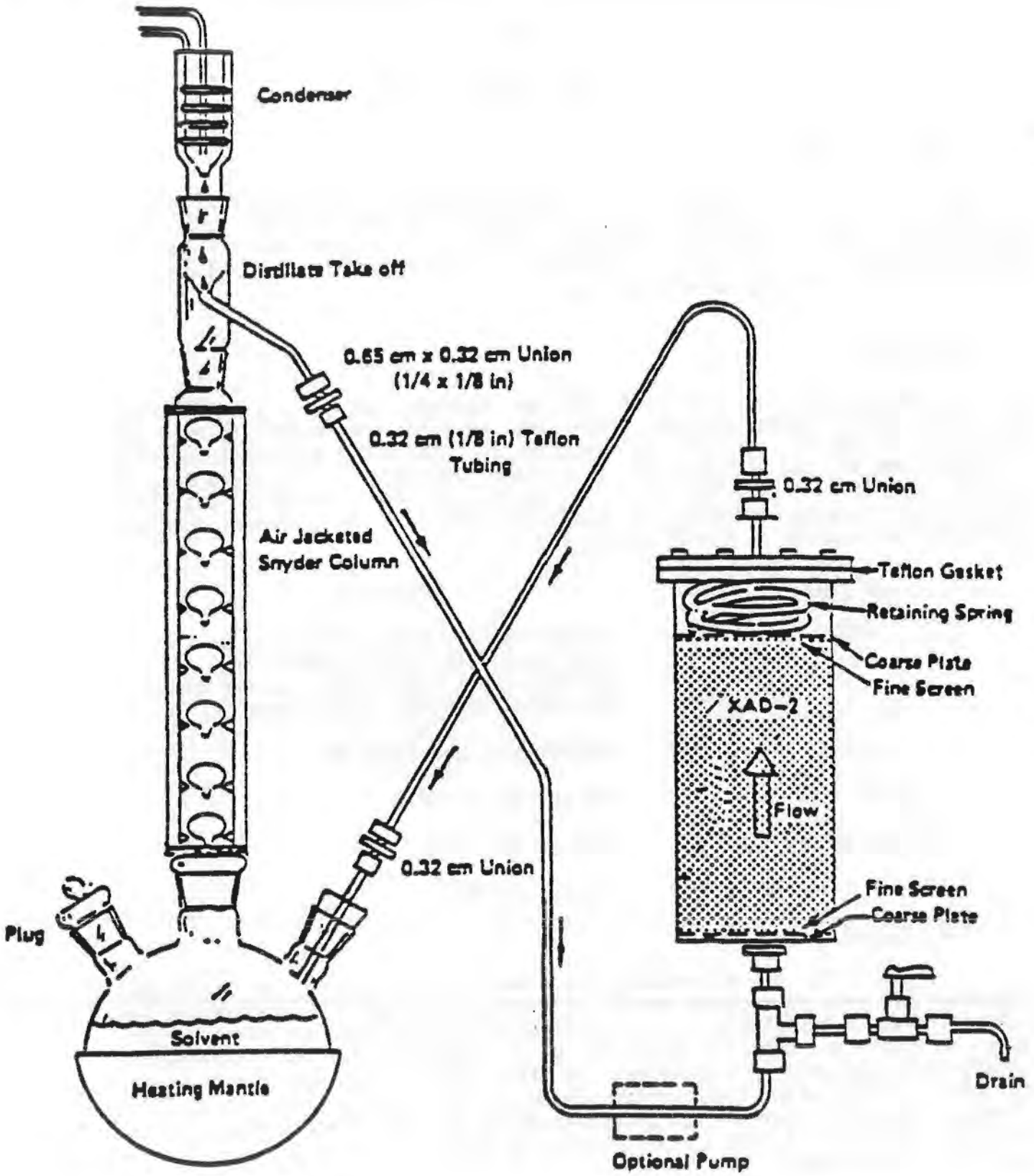

Figure A-1. XAD-2 cleanup extraction apparatus.

equal to twice the dead volume of the XAD-2 canister. Solvent is refluxed through the Snyder column, and the distillate is continuously cycled up through the XAD-2 for extraction and returned to the flask. The flow is maintained upward through the XAD-2 to allow maximum solvent contact and prevent channeling. A valve at the bottom of the canister allows removal of solvent from the canister between changes.

2.2.2 Experience has shown that it is very difficult to cycle sufficient water in this mode. Therefore the aqueous rinse is accomplished by simply flushing the canister with about 20 liters of distilled water. A small pump may be useful for pumping the water through the canister. The water extraction should be carried out at the rate of about 20-40 mL/min.

2.2.3 After draining the water, subsequent methyl alcohol and methylene chloride extractions are carried out using the refluxing apparatus. An overnight or 10- to 20-hr period is normally sufficient for each extraction.

2.2.4 [illegible] terials of construction are glass, Teflon, or stainless steel. Pump [illegible]ld not contain extractable materials. Pumps are not used with methanol [illegible] thylene chloride.

3.0 DRYING

3.1 After evaluation of several methods of removing residual solvent, a fluidized-bed technique has proved to be the fastest and most reliable drying method.

3.2 A simple column with suitable retainers, as shown in Figure A-2, will serve as a satisfactory column. A 10.2-cm (4-in.) Pyrex pipe 0.6 m (2 ft) long will hold all of the XAD-2 from the extractor shown in Figure A-1 or the Soxhlet extractor, with sufficient space for fluidizing the bed while generating a minimum resin load at the exit of the column.

3.3 Method 1: The gas used to remove the solvent is the key to preserving the cleanliness of the XAD-2. Liquid nitrogen from a standard commercial liquid nitr[illegible] cylinder has routinely proved to be a reliable source of large volumes [illegible] gas free from organic contaminants. The liquid nitrogen cylinder is connected to the column by a length of precleaned 0.95-cm (3/8-in.) copper tubing, coiled to pass through a heat source. As nitrogen is bled from the cylinder, it is vaporized in the heat source and passes through the column. A convenient [illegible]t source is a water bath heated from a steam line. The final nitroge[illegible]erature should only be warm to the touch and not over 40°C. Experience has shown that about 500 g of XAD-2 may be dried overnight by consuming a full 160-liter cylinder of liquid nitrogen.

3.4 Method 2: As a second choice, high-purity tank nitrogen may be used to dry the XAD-2. The high-purity nitrogen must first be passed through a bed

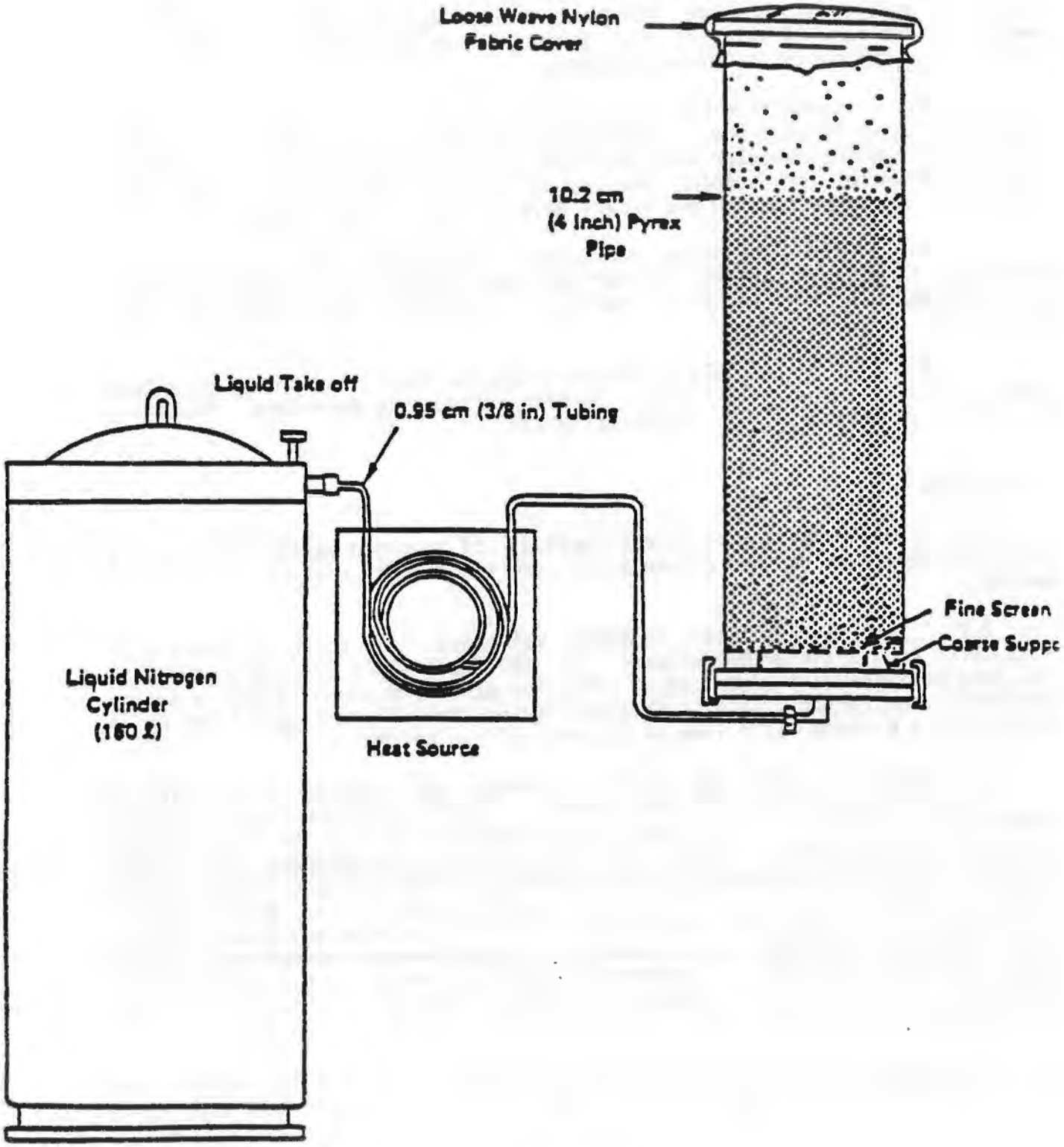

Figure A-2. XAD-2 fluidized-bed drying apparatus.

of activated charcoal approximately 150 mL in volume. With either type of drying method, the rate of flow should gently agitate the bed. Excessive fluidization may cause the particles to break up.

4.0 QUALITY CONTROL PROCEDURES

4.1 For both Methods 1 and 2, the quality control results must be reported for the batch. The batch must be reextracted if the residual extractable organics are >20 ug/mL by TCO analysis or the gravimetric residue is >0.5 mg/20 g XAD-2 extracted. (See also section 5.1, Method 0010.)

4.2 Four control procedures are used with the final XAD-2 to check for (1) residual methylene chloride, (2) extractable organics (TCO), (3) specific compounds of interest as determined by GC/MS, as described in Section 4.5 below, and (4) residue (GRAV).

4.3 Procedure for residual methylene chloride:

4.3.1 Description: A 1±0.1-g sample of dried resin is weighed into a small vial, 3 mL of toluene are added, and the vial is capped and well shaken. Five uL of toluene (now containing extracted methylene chloride) are injected into a gas chromatograph, and the resulting integrated area is compared with a reference standard. The reference solution consists of 2.5 uL of methylene chloride in 100 mL of toluene, simulating 100 ug of residual methylene chloride on the resin. The acceptable maximum content is 1,000 ug/g resin.

4.3.2 Experimental: The gas chromatograph conditions are as follows:

6-ft x 1/8-in. stainless steel column containing 10% OV-101 on 100/120 Supelcoport;

Helium carrier at 30 mL/min;

FID operated on 4 x 10^{-11} A/mV;

Injection port temperature: 250°C;

Detector temperature: 305°C;

Program: 30°C(4 min) 40°C/min 250°C (hold); and

Program terminated at 1,000 sec.

4.4 Procedure for residual extractable organics:

4.4.1 Description: A 20±0.1-g sample of cleaned, dried resin is weighed into a precleaned alundum or cellulose thimble which is plugged with cleaned glass wool. (Note that 20 g of resin will fill a thimble, and the

resin will float out unless well plugged.) The thimble containing the resin is extracted for 24 hr with 200-mL of pesticide- grade methylene chloride (Burdick and Jackson pesticide-grade or equivalent purity). The 200-mL extract is reduced in volume to 10-mL using a Kuderna-Danish concentrator and/or a nitrogen evaporation stream. Five uL of that solution are analyzed by gas chromatography using the TCO analysis procedure. The concentrated solution should not contain >20 ug/mL of TCO extracted from the XAD-2. This is equivalent to 10 ug/g of TCO in the XAD-2 and would correspond to 1.3 mg of TCO in the extract of the 130-g XAD-2 module. Care should be taken to correct the TCO data for a solvent blank prepared (200 mL reduced to 10 mL) in a similar manner.

4.4.2 **Experimental:** Use the TCO analysis conditions described in the revised Level 1 manual (EPA 600/7-78-201).

4.5 GC/MS Screen: The extract, as prepared in paragraph 4.4.1, is subjected to GC/MS analysis for each of the individual compounds of interest. The GC/MS procedure is described in Chapter Four, Method 8270. The extract is screened at the MDL of each compound. The presence of any compound at a concentration >25 ug/mL in the concentrated extract will require the XAD-2 to be recleaned by repeating the methylene chloride step.

4.6 Methodology for residual gravimetric determination: After the TCO value and GC/MS data are obtained for the resin batch by the above procedures, dry the remainder of the extract in a tared vessel. There must be <0.5 mg residue registered or the batch of resin will have to be extracted with fresh methylene chloride again until it meets this criterion. This level corresponds to 25 ug/g in the XAD-2, or about 3.25 mg in a resin charge of 130 g.

METHOD 0010, APPENDIX B

TOTAL CHROMATOGRAPHABLE ORGANIC MATERIAL ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 In this procedure, gas chromatography is used to determine the quantity of lower boiling hydrocarbons (boiling points between 90° and 300°C) in the concentrates of all organic solvent rinses, XAD-2 resin and LC fractions - when Method 1 is used (see References, Method 0010) - encountered in Level 1 environmental sample analyses. Data obtained using this procedure serve a twofold purpose. First, the total quantity of the lower boiling hydrocarbons in the sample is determined. Then whenever the hydrocarbon concentrations in the original concentrates exceed 75 ug/m^3, the chromatography results are reexamined to determine the amounts of individual species.

The extent of compound identification is limited to representing all materials as normal alkanes based upon comparison of boiling points. Thus the method is not qualitative. In a similar manner, the analysis is semiquantitative; calibrations are prepared using only one hydrocarbon. They are replicated but samples routinely are not.

.2 Application: This procedure applies solely to the Level 1 C7-C16 gas chromatographic analysis of concentrates of organic extracts, neat liquids, and of LC fractions. Throughout the procedure, it is assumed the analyst has been given a properly prepared sample.

1.3 Sensitivity: The sensitivity of this procedure, defined as the slope of a plot of response versus concentration, is dependent on the instrument and must be verified regularly. TRW experience indicates the nominal range is of the order of 77 uV·V·sec·uL/ng of n-heptane and 79 uV·sec·ul/ng of n-hexadecane. The instrument is capable of perhaps one hundredfold greater sensitivity. The level specified here is sufficient for Level 1 analysis.

1.4 Detection limit: The detection limit of this procedure as written is 1.3 ng/uL for a 1 uL injection of n-decane. This limit is arbitrarily based on defining the minimum detectable response as 100 uv·sec. This is an easier operational definition than defining the minimum detection limit to be that amount of material which yields a signal twice the noise level.

1.5 Range: The range of the procedure will be concentrations of 1.3 ng/uL and greater.

1.6 Limitations

1.6.1 Reporting limitations: It should be noted that a typical environmental sample will contain compounds which: (a) will not elute in the specified boiling ranges and thus will not be reported, and/or (b)

will not elute from the column at all and thus will not be reported. Consequently, the organic content of the sample as reported is a lower bound and should be regarded as such.

1.6.2 **Calibration limitations:** Quantitation is based on calibration with n-decane. Data should therefore be reported as, e.g., mg C8/m^3 as n-decane. Since response varies linearly with carbon number (over a wide range the assumption may involve a 20% error), it is clear that heptane (C7) detected in a sample and quantitated as decane will be overestimated. Likewise, hexadecane (C16) quantitated as decane will be underestimated. From previous data, it is estimated the error involved is on the order of 6-7%.

1.6.3 **Detection limitations:** The sensitivity of the flame ionization detector varies from compound to compound. However, n-alkanes have a greater response than other classes. Consequently, using an n-alkane as a calibrant and assuming equal responses of all other compounds tends to give low reported values.

2.0 SUMMARY OF METHOD

2.1 A mL aliquot of all 10-mL concentrates is disbursed for GC-TCO analysis. With boiling point-retention time and response-amount calibration curves, the data (peak retention times and peak areas) are interpreted by first summing peak areas in the ranges obtained from the boiling point-retention time calibration. Then, with the response-amount calibration curve, the area sums are converted to amounts of material in the reported boiling point ranges.

2.2 After the instrument is set up, the boiling point-retention time calibration is effected by injecting a mixture of n-C7 through n-C16 hydrocarbons and operating the standard temperature program. Response-quantity calibrations are accomplished by injecting n-decane in n-pentane standards and performing the standard temperature program.

2.3 Definitions

2.3.1 **GC:** Gas chromatography or gas chromatograph.

2.3.2 **C7-C16 n-alkanes:** Heptane through hexadecane.

2.3.3 **GCA temperature program:** 4 min isothermal at 60°C, 10°C/min from 60° to 220°C.

2.3.4 **TRW temperature program:** 5 min isothermal at room temperature, then program from 30°C to 250°C at 15°C/min.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: This procedure is intended for use on a Varian 1860 gas chromatograph, equipped with dual flame ionization detectors and a linear temperature programmer. Any equivalent instrument can be used provided that electrometer settings, etc., be changed appropriately.

4.2 Gases:

4.2.1 Helium: Minimum quality is reactor grade. A 4A or 13X molecular sieve drying tube is required. A filter must be placed between the trap and the instrument. The trap should be recharged after every third tank of helium.

4.2.2 Air: Zero grade is satisfactory.

4.2.3 Hydrogen: Zero grade.

4.3 Syringe: Syringes are Hamilton 701N, 10 uL, or equivalent.

4.4 Septa: Septa will be of such quality as to produce very low bleed during the temperature program. An appropriate septum is Supelco Microsep 138, which is Teflon-backed. If septum bleed cannot be reduced to a negligible level, it will be necessary to install septum swingers on the instrument.

4.5 Recorder: The recorder of this procedure must be capable of not less than 1 mV full-scale display, a 1-sec time constant and 0.5 in. per min chart rate.

4.6 Integrator: An integrator is required. Peak area measurement by hand is satisfactory but too time-consuming. If manual integration is required, the method of "height times width at half height" is used.

4.7 Columns:

4.7.1 Preferred column: 6 ft x 1/8 in. O.D. stainless steel column of 10% OV-101 on 100/120 mesh Supelcoport.

4.7.2 Alternate column: 6 ft x 1/8 in. O.D. stainless steel column of 10% OV-1 (or other silicon phase) on 100/120 mesh Supelcoport.

4.8 Syringe cleaner: Hamilton syringe cleaner or equivalent connected to a suitable vacuum source.

5.0 REAGENTS

5.1 Pentane: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for standards and for syringe cleaning.

5.2 Methylene chloride: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for syringe cleaning.

6.0 SAMPLING HANDLING AND PRESERVATION

6.1 The extracts are concentrated in a Kuderna-Danish evaporator to a volume less than 10 mL. The concentrate is then quantitatively transferred to a 10-mL volumetric flask and diluted to volume. A 1-mL aliquot is taken for both this analysis and possible subsequent GC/MS analysis and set aside in the sample bank. For each GC-TCO analysis, obtain the sample sufficiently in advance to allow it to warm to room temperature. For example, after one analysis is started, return that sample to the sample bank and take the next sample.

7.0 PROCEDURES

7.1 Setup and checkout: Each day, the operator will verify the following:

7.1.1 That supplies of carrier gas, air and hydrogen are sufficient, i.e., that each tank contains > 100 psig.

7.1.2 That, after replacement of any gas cylinder, all connections leading to the chromatograph have been leak-checked.

7.1.3 That the carrier gas flow rate is 30 ± 2 mL/min, the hydrogen flow rate is 30 ± 2 mL/min, and the air flow rate is 300 ± 20 mL/min.

7.1.4 That the electrometer is functioning properly.

7.1.5 That the recorder and integrator are functioning properly.

7.1.6 That the septa have been leak-checked (leak-checking is effected by placing the soap bubble flow meter inlet tube over the injection port adaptors), and that no septum will be used for more than 20 injections.

7.1.7 That the list of samples to be run is ready.

7.2 Retention time calibration:

7.2.1 To obtain the temperature ranges for reporting the results of the analyses, the chromatograph is given a normal boiling point-retention time calibration. The n-alkanes, their boiling points, and data reporting ranges are given in the table below:

	NBP,°C	Reporting Range,°C	Report As
n-heptane	98	90-110	C7
n-octane	126	110-140	C8
n-nonane	151	140-160	C9
n-decane	174	160-180	C10
n-undecane	194	180-200	C11
n-dodecane	214	200-220	C12
n-tridecane	234	220-240	C13
n-tetradecane	252	240-260	C14
n-pentadecane	270	260-280	C15
n-hexadecane	288	280-300	C16

7.2.2 **Preparation of standards:** Preparing a mixture of the C7-C16 alkanes is required. There are two approaches: (1) use of a standards kit (e.g., Polyscience Kit) containing bottles of mixtures of selected n-alkanes which may be combined to produce a C7-C16 standard; or (2) use of bottles of the individual C7-C16 alkanes from which accurately known volumes may be taken and combined to give a C7-C16 mixture.

7.2.3 **Procedure for retention time calibration:** This calibration is performed at the start of an analytical program; the mixture is chromatographed at the start of each day. To attain the required retention time precision, both the carrier gas flow rate and the temperature program specifications must be observed. Details of the procedure depend on the instrument being used. The general procedure is as follows:

7.2.3.1 Set the programmer upper limit at 250°C. If this setting does not produce a column temperature of 250°C, find the correct setting.

7.2.3.2 Set the programmer lower limit at 30°C.

7.2.3.3 Verify that the instrument and samples are at room temperature.

7.2.3.4 Inject 1 uL of the n-alkane mixture.

7.2.3.5 Start the integrator and recorder.

7.2.3.6 Allow the instrument to run isothermally at room temperature for five min.

7.2.3.7 Shut the oven door.

7.2.3.8 Change the mode to Automatic and start the temperature program.

7.2.3.9 Repeat Steps 1-9 a sufficient number of times so that the relative standard deviation of the retention times for each peak is <5%.

7.3 Response calibration:

7.3.1 For the purposes of a Level 1 analysis, response-quantity calibration with n-decane is adequate. A 10-uL volume of n-decane is injected into a tared 10 mL volumetric flask. The weight injected is obtained and the flask is diluted to the mark with n-pentane. This standard contains about 730 ng n-decane per uL n-pentane. The exact concentration depends on temperature, so that a weight is required. Two serial tenfold dilutions are made from this standard, giving standards at about 730, 73, and 7.3 ng n-decane per uL n-pentane, respectively.

7.3.2 Procedure for response calibration: This calibration is performed at the start of an analytical program and monthly thereafter. The most concentrated standard is injected once each day. Any change in calibration necessitates a full calibration with new standards. Standards are stored in the refrigerator locker and are made up monthly.

7.3.2.1 Verify that the instrument is set up properly.

7.3.2.2 Set electrometer at 1×10^{-10} A/mV.

7.3.2.3 Inject 1 uL of the highest concentration standard.

7.3.2.4 Run standard temperature program as specified above.

7.3.2.5 Clean syringe.

7.3.2.6 Make repeated injections of all three standards until the relative standard deviations of the areas of each standard are ≤5%.

7.4 Sample analysis procedure:

7.4.1 The following apparatus is required:

7.4.1.1 Gas chromatograph set up and working.

7.4.1.2 Recorder, integrator working.

7.4.1.3 Syringe and syringe cleaning apparatus.

7.4.1.4 Parameters: Electrometer setting is 1×10^{-10} A/mV; recorder is set at 0.5 in./min and 1 mV full-scale.

7.4.2 Steps in the procedure are:

7.4.2.1 Label chromatogram with the data, sample number, etc.

7.4.2.2 Inject sample.

7.4.2.3 Start integrator and recorder.

7.4.2.4 After isothermal operation for 5 min, begin temperature program.

7.4.2.5 Clean syringe.

7.4.2.6 Return sample; obtain new sample.

7.4.2.7 When analysis is finished, allow instrument to cool. Turn chromatogram and integrator output and data sheet over to data analyst.

7.5 Syringe cleaning procedure:

7.5.1 Remove plunger from syringe.

7.5.2 Insert syringe into cleaner; turn on aspirator.

7.5.3 Fill pipet with pentane; run pentane through syringe.

7.5.4 Repeat with methylene chloride from a separate pipet.

7.5.5 Flush plunger with pentane followed by methylene chloride.

7.5.6 Repeat with methylene chloride.

7.6 Sample analysis decision criterion: The data from the TCO analyses of organic extract and rinse concentrates are first used to calculate the total concentration of C7-C16 hydrocarbon-equivalents (Paragraph 7.7.3) in the sample with respect to the volume of air actually sampled, i.e., ug/m^3. On this basis, a decision is made both on whether to calculate the quantity of each n-alkane equivalent present and on which analytical procedural pathway will be followed. If the total organic content is great enough to warrant continuing the analysis -- >500 ug/m^3 -- a TCO of less than 75 ug/m^3 will require only LC fractionation and gravimetric determinations and IR spectra to be obtained on each fraction. If the TCO is greater than 75 ug/m^3, then the first seven LC fractions of each sample will be reanalyzed using this same gas chromatographic technique.

7.7 Calculations:

7.7.1 **Boiling Point - Retention Time Calibration:** The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.1.1 Average the retention times and calculate relative standard deviations for each n-hydrocarbon.

7.7.1.2 Plot average retention times as abscissae versus normal boiling points as ordinates.

7.7.1.3 Draw in calibration curve.

7.7.1.4 Locate and record retention times corresonding to boiling ranges 90-100, 110-140, 140-160, 160-180, 180-200, 200-220, 220-240, 240-260, 260-280, 280-300°C.

7.7.2 **Response-amount calibration:** The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.2.1 Average the area responses of each standard and calculate relative standard deviations.

7.7.2.2 Plot response (uv·sec) as ordinate versus ng/uL as abscissa.

7.7.2.3 Draw in the curve. Perform least squares regression and obtain slope (uV·sec·uL/ng).

7.7.3 **Total C7-C16 hydrocarbons analysis:** The required data for this calculation are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.3.1 Sum the areas of all peaks within the retention time range of interest.

7.7.3.2 Convert this area (uV·sec) to ng/uL by dividing by the weight response for n-decane (uV·sec.uL/ng).

7.7.3.3 Multiply this weight by the total concentrate volume (10 mL) to get the weight of the C7-C16 hydrocarbons in the sample.

7.7.3.4 Using the volume of gas sampled or the total weight of sample acquired, convert the result of Step 7.7.3.3 above to ug/m^3.

7.7.3.5 If the value of total C7-C16 hydrocarbons from Step 7.7.3.4 above exceeds 75 ug/m^3, calculate individual hydrocarbon concentrations in accordance with the instructions in Paragraph 7.7.5.5 below.

7.7.4 **Individual C7-C16 n-Alkane Equivalent Analysis:** The required data from the analyses are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.4.1 Sum the areas of peaks in the proper retention time ranges.

7.7.4.2 Convert areas (uV·sec) to ng/uL by dividing by the proper weight response (uV·sec·uL/ng).

7.7.4.3 Multiply each weight by total concentrate volume (10 mL) to get weight of species in each range of the sample.

7.7.4.4 Using the volume of gas sampled on the total weight of sample acquired, convert the result of Step 7.7.4.3 above to ug/m^3.

8.0 QUALITY CONTROL

8.1 Appropriate QC is found in the pertinent procedures throughout the method.

9.0 METHOD PERFORMANCE

9.1 Even relatively comprehensive error propagation analysis is beyond the scope of this procedure. With reasonable care, peak area reproducibility of a standard should be of the order of 1% RSD. The relative standard deviation of the sum of all peaks in a fairly complex waste might be of the order of 5-10%. Accuracy is more difficult to assess. With good analytical technique, accuracy and precision should be of the order of 10-20%.

10.0 REFERENCES

1. Emissions Assessment of Conventional Stationary Combustion Systems: Methods and Procedure Manual for Sampling and Analysis, Interagency Energy/Environmental R&D Program, Industrial Environmental Research Laboratory, Research Triangle Park, NC 27711, EPA-600/7-79-029a, January 1979.

E-4 AEHA STEM METHOD

SECTION 6. SAMPLING PROCEDURES

6.1. Introduction. The POHC emissions are to be sampled in the exhaust gas stream with a modification of the EPA Method 5 sampling train. As with standard methodology, isokinetic sampling is conducted with the train.

6.2. USAEHA Sampling Train for Energetic Materials.

6.2.1. Components. The sampling train to be used for emission testing is shown in Figure 6.1. The components of this modification of the EPA Method 5 sampling train from inlet to outlet are as follows:

- Nozzle
- Pyrex®-lined probe
- Cyclone eliminator (optional)
- 4-inch glass-fiber filter
- 90-degree connector
- Impinger No. 1, dry
- 180-degree connector
- Impinger No. 2, dry
- 180-degree connector
- Impinger No. 3, dry
- 180-degree connector
- XAD-2 resin tube (vertical orientation)
- 180-degree connector
- Straight glass tube
- 180-degree connector
- Impinger No. 4, dry
- 180-degree connector
- Impinger No. 5, silica gel

6.2.2 Special Considerations.

6.2.2.1 The probe and filter housing will be assembled in a normal EPA Method 5 sampling box to allow heating of both the probe and the filter to 248 °F ± 25 °F. The impingers will be packed in an ice bath to provide the necessary cooling of the gas sample.

6.2.2.2 The three impingers placed prior to the resin tube are used to cool the gas to an acceptable temperature (<68 °F) and to remove moisture from the gas sample. The cooler temperature is necessary to ensure that the resin will function properly as a POHC collection medium. Depending upon the temperature and the moisture content of the stack gases, additional impingers may be added prior to the resin module. This decision will be made by the project engineer based upon preliminary measurements of or calculation of stack conditions. Recovery of the impingers before the resin module does not depend upon the number of impingers.

®Pyrex is a registered trademark of Corning Glass Works, Houghton Park, Corning, New York.

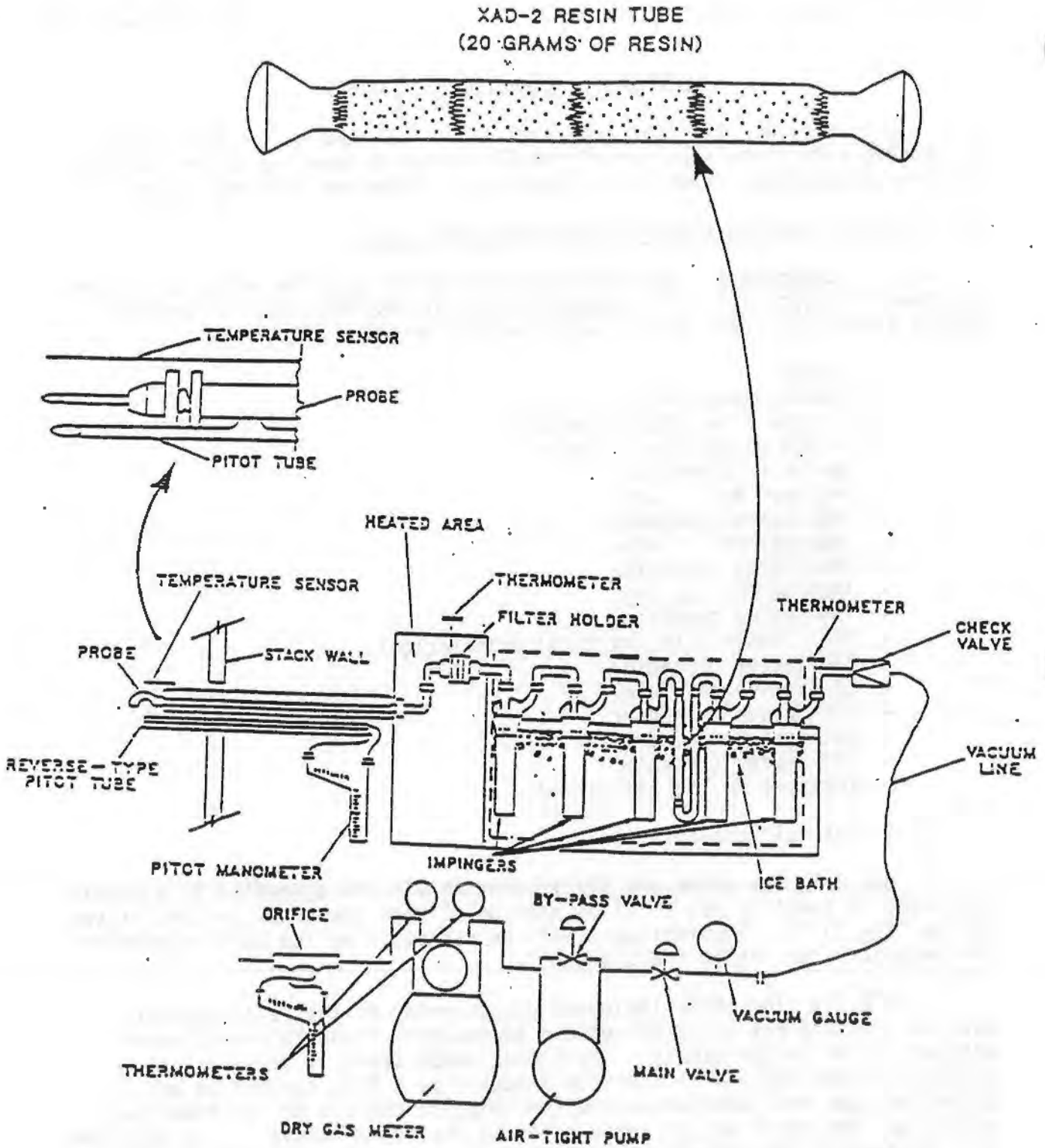

FIGURE 6-1. MODIFIED EPA METHOD 5 SAMPLING TRAIN

6.2.3. Sampling Preparations.

6.2.3.1. Glassware Cleanup (Field/Laboratory). All glassware to be used for sampling or sample recovery are considered clean for use once it has undergone a detergent wash in a sonic bath, a distilled water rinse, and then an acetone (reagent grade) rinse. Exceptions to this rule include the resin tubes and the separatory funnels. The resin tubes may receive the standard glass cleaning procedures, time permitting, but there is a limited number of tubes available, and there is a high potential for their breakage. The separatory funnels are so large that they would seriously slow down the glassware turnaround time through the sonic bath. Both the tubes and the separatory funnels may be cleaned by an abbreviated procedure. This procedure calls for one or two rinses with toluene followed by several acetone rinses. Acetone not only provides an additional solvent rinse to remove contaminating organic compounds, but helps remove the film that toluene creates on glass. Because of that film, any glassware that was rinsed with toluene or came in contact with toluene, and is scheduled to go into the sonic bath, should be rinsed with acetone first to help extend the useful life of the sonic bath solution. Additional glassware that may receive the abbreviated cleaning procedures could include other high use, limited number items such as graduated cylinders.

6.2.3.2. Resin Cleanup. XAD-2 resin is purchased by the USAEHA in a purified form (previously cleaned by soxhlet extraction). A portion of this resin (5 grams) will be extracted as per the procedure discussed in Section 8 of this plan to ensure no interference compounds exist on the resin which may impede the analysis at a later date. The resin is not reused, but cleanup may be required if the analysis so indicates. If the need for cleaning the resin ever arises, the procedures recommended in Appendix B of reference 5 will be utilized.

6.2.4. Sampling Train Assembly. Glass fiber filters [99.95 percent efficiency ($\leq$0.05-percent penetration) for 0.3 micron dioctyl phthalate smoke particles] for the USAEHA train configuration do not need to be preweighed, since they will undergo organic sample recovery immediately after sampling. Each impinger will be labeled with the run number and impinger number. Additionally, the other key components of the train (resin tube, filter housing, and probe wash recovery bottles) will also be labeled with the run number (paragraph 7.2.1). The resin tube will be packed with four 5.0 gram sections of XAD-2 resin with glass wool on both sides of each section. Following this packing, the sample flow direction will be marked by an arrow on the tube wall. Once the train components are assembled in the field laboratory, the impingers and resin tube will be weighted so the amount of moisture collected in the train during sampling may be determined at the end of the run. If the moisture gain in the resin tube frequently shows a small negative number, then that moisture value will be disregarded. Connecting glassware behind the resin section should be temporarily marked (e.g., with masking tape) to differentiate it from the glassware in front of the resin.

6.2.5. Sampling Train Spiking. A solution for a field surrogate spike of the sampling train will be prepared by the analytical laboratory in accordance with the procedures in Appendix B. Table 6.1 contains a list of surrogate compounds to be utilized for varying POHC's. In the event of multiple POHC's, the sampling train should be spiked with a surrogate for each of the specified POHC's. The project engineer will determine the level of this spike and supply that value to the laboratory. To calculate the spike level, the project engineer must determine the quantity of the target compound captu[illegible] by the sampling train in the sampling period for the case of the lowes[illegible]ntity captured (e.g., highest DRE legitimately possible for the incine[illegible]r). The spike level will be at least four times this quantity. For example, assume for the case of an incinerator, the quantity of the target compound captured in a 1-hour period at 99.999 percent DRE is 50 µg. The minimum spike level should be 200 µg, 4 times the 50 µg level. When the sampling train is assembled, 50 mL of deionized water will be added to the first impinger to support a field spike. Half of the calculated spike level should be on the first resin section, placed there by direct injection, while the other half of the spike level will be in the 50 mL of water in the first impinger. The spike level for the entire train should not exceed 1000 µg.

TABLE 6.1. LIST OF POHC's AND SURROGATE COMPOUNDS

Principal Organic Hazardous Constituent	Surrogate Compound
Nitroglycerin	Ethylene glycol dinitrate
2,4-Dinitrotoluene	3,4-Dinitrotoluene
2,6-Dinitrotoluene	3,4-Dinitrotoluene
2,4,6-Trinitrotoluene	2,4,5-Trinitrotoluene
Cyclotrimethylene-trinitramine (RDX)	

6.2.6. Sampling Procedures. The actual sampling operation is conducted using the standard EPA Method 5 sampling procedure as described in references 1 and 4. The Method 5 procedures are utilized for pretest and post-test leak checks, isokinetic sampling rate, and filter changes.

SECTION 8. SAMPLE RECOVERY AND ANALYTICAL PROCEDURES

8.1. Introduction. The sampling train has been described in detail in Section 6 of this QA plan and for the purposes of sample recovery procedures has three major sections. The first section is the filter and all the components preceding it. The second major section starts with the back half of the filter housing and includes all the following glassware (mainly the three impingers) up to the resin module. The third section is the resin module itself. While there are two impingers that follow the resin module, they are not major features of the POHC sampling train since their only function is moisture removal.

8.2. POHC Sampling Train Recovery Procedures.

8.2.1 The initial sample recovery starts outside the field laboratory as the train is disassembled. The probe is removed from the sampling train and capped. The first and last impingers are also capped. The sample box containing the impingers and the resin module and the sampling probe are brought into the field laboratory for sample recovery. In the field laboratory, the nozzle, probe liner, and the front half of the filter housing are rinsed with the recovery solvent (see Table 8.1), the solvent volume is measured, and the liquid is placed in a sample container. The filter is removed from its housing and immersed in the recovery solvent in the same container as the rinse. An additional rinse of the probe liner/nozzle section with acetone is performed and is placed in a separate container. This rinse will provide a QA check on the rinse/recovery effectiveness. Figure 8-1 diagrams the recovery of the front section of the sampling train.

TABLE 8-1. EXTRACTION SOLVENTS UTILIZED FOR VARIOUS POHC's

Principal Hazardous Organic Constituent	Surrogate Compound	Extraction Solvent
Nitroglycerin	Ethylene glycol dinitrate	Toluene
2,4-Dinitrotoluene	3,4-Dinitrotoluene	Toluene
2,6-Dinitrotoluene	3,4-Dinitrotoluene	Toluene
2,4,6-Trinitrotoluene	2,4,5-Trinitrotoluene	Toluene
Cyclotrimethylene-trinitramine (RDX)		Toluene/ Iso-amylacetate (5:95 by volume)

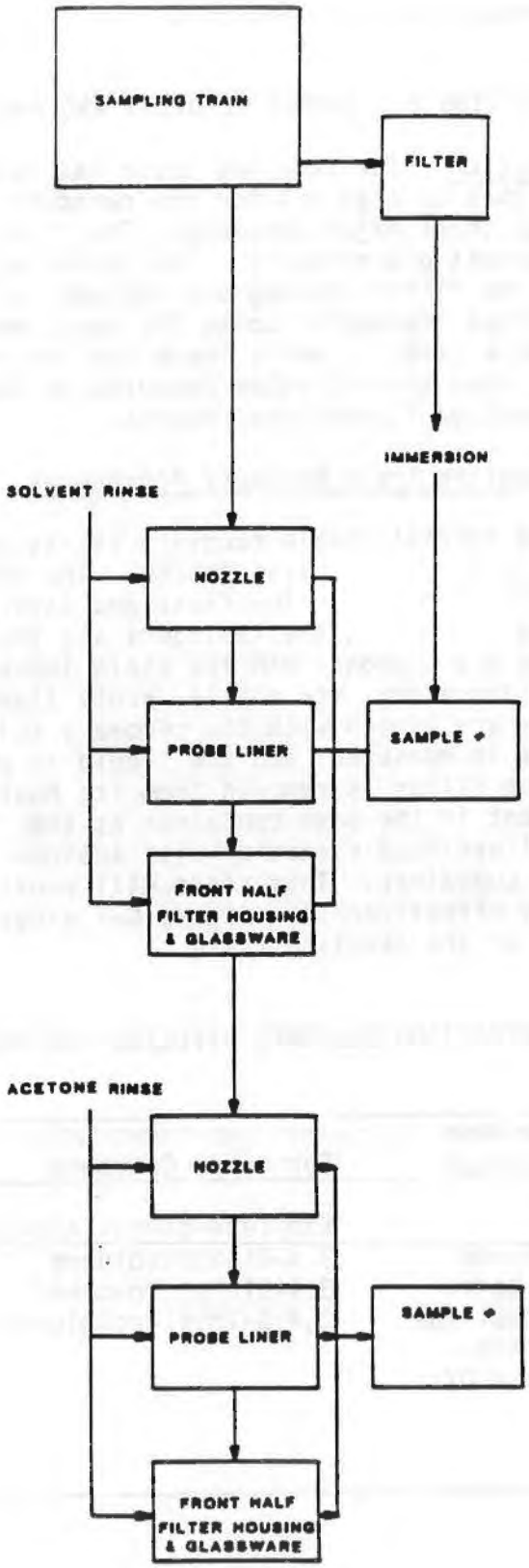

FIGURE 8-1. SAMPLING TRAIN FRONT SECTION RECOVERY

8.2.2 Each of the impingers are weighed prior to further organic recovery steps in order to determine the exhaust gas moisture content. Once completed, the condensate is collected and each of the first three impingers, and their associated connections are rinsed with distilled and deionized water. The rinse and condensate are combined and extracted three times. These extractions are completed in separatory funnels using shakeout techniques. Solvent volumes are based on total water volume to be extracted at a ratio of 4 to 1 (water to solvent). The extraction procedure consists of shaking the separatory funnel and its contents for three minutes, venting frequently. The organic and water layers should be allowed to separate to some degree, followed by 3 more minutes of shaking. This settling/shaking should be conducted one additional time and then the two layers allowed to separate to a narrow interfacial band if possible. The organic sample is obtained following this third shaking/settling. The first two extracts are completed at neutral pH conditions, while the water is acidified for the third extraction by adding concentrated sulfuric acid. The impingers and associated glassware are then rinsed with acetone to provide a QA check on the water rinse/recovery procedure. Impinger recovery is shown schematically in Figure 8.2.

8.2.3 The resin module is recovered in four sections and each section generates three samples. The extraction solvent (30 mL) is added to each resin section and glass wool plug, and the samples are then subjected to mechanical shakeout procedures (30 minutes). A portion of the solvent solution is removed to become the first sample (10 mL: sample extract number 1) and additional solvent is added to the resin section (10 mL). The resin sample then receives another mechanical shakeout followed by removal of the extract (10 mL: sample extract number 2). Additional solvent (10 mL) is then added to the resin. Following further shakeout, the third sample extraction is completed. The solvent and the resin are left combined for additional extraction (contact) time. The third extract is separated from the solvent/resin mixture at the USAEHA laboratories. This third extraction is completed just to verify the recovery completion. Resin module recovery is shown schematically in Figure 8-3.

8.3. Analytical Procedures.

8.3.1. In general terms, the methodology for analyzing the various samples generated by the recovery of the stack gas sampling train will utilize gas chromatography. The GC's chromatographs will be equipped with either an electron capture or a nitrogen/phosphorus detector and will utilize capillary column chromatography. Individual samples will be analyzed from each portion of the sampling train. No sample combinations or volume reductions will be utilized for samples showing positive analyses. Volume reduction procedures may be utilized for samples that have no detectable or quantifiable analyte. After all the samples have been analyzed via gas chromatography, a portion of these samples will be submitted for confirmational analysis on a GC/MS. This analysis has the confirmation of the GC peak as its primary objective, but in those cases where the concentration of the target analytes (POHC and surrogate) is large enough to overcome the sensitivity constraints of the GC/MS, the analysis will be utilized for the confirmation of the quantitation. The analytical procedures are discussed in detail in Appendix B of this plan.

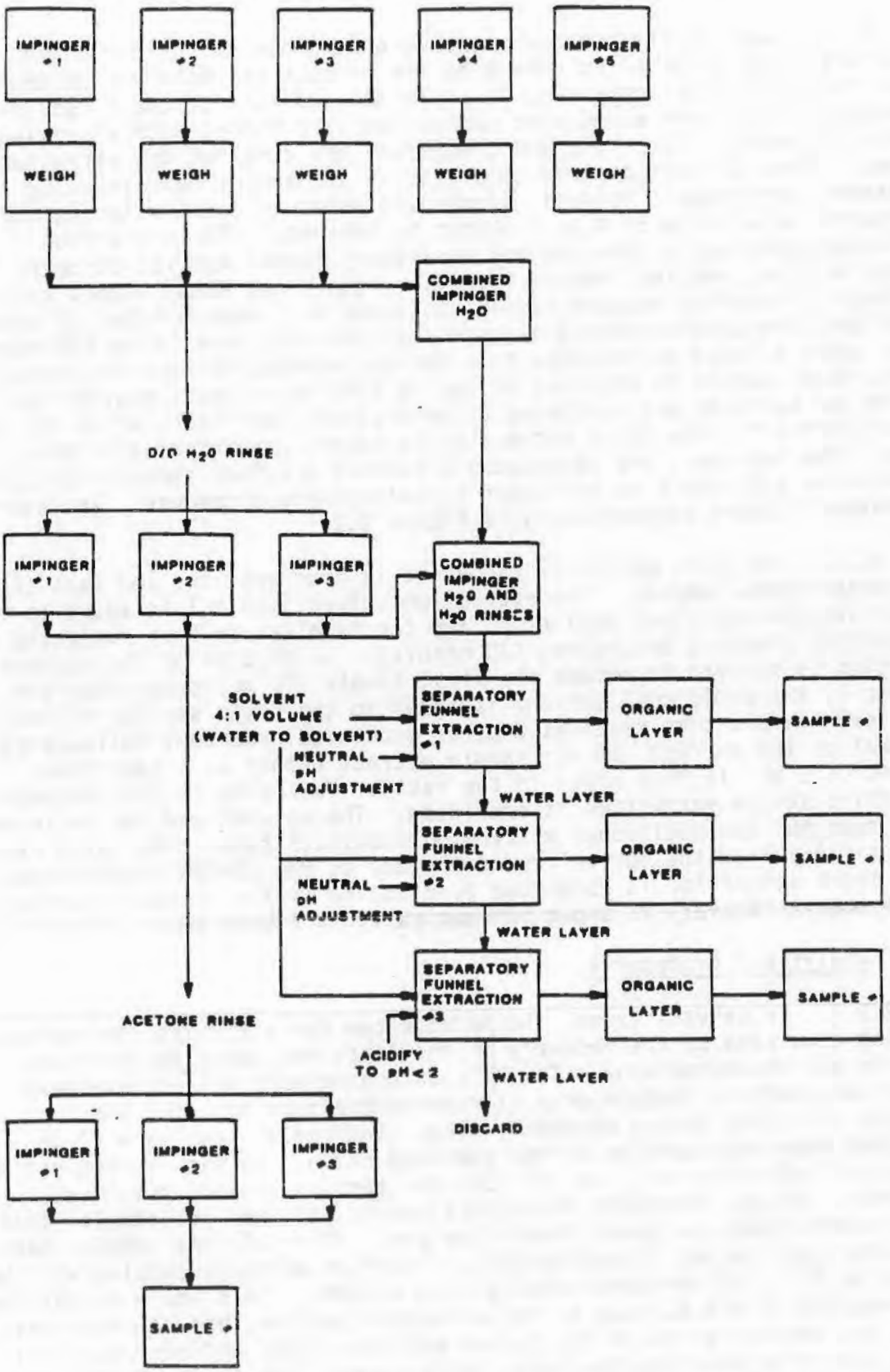

FIGURE 8-2. SAMPLING TRAIN IMPINGER SECTION RECOVERY

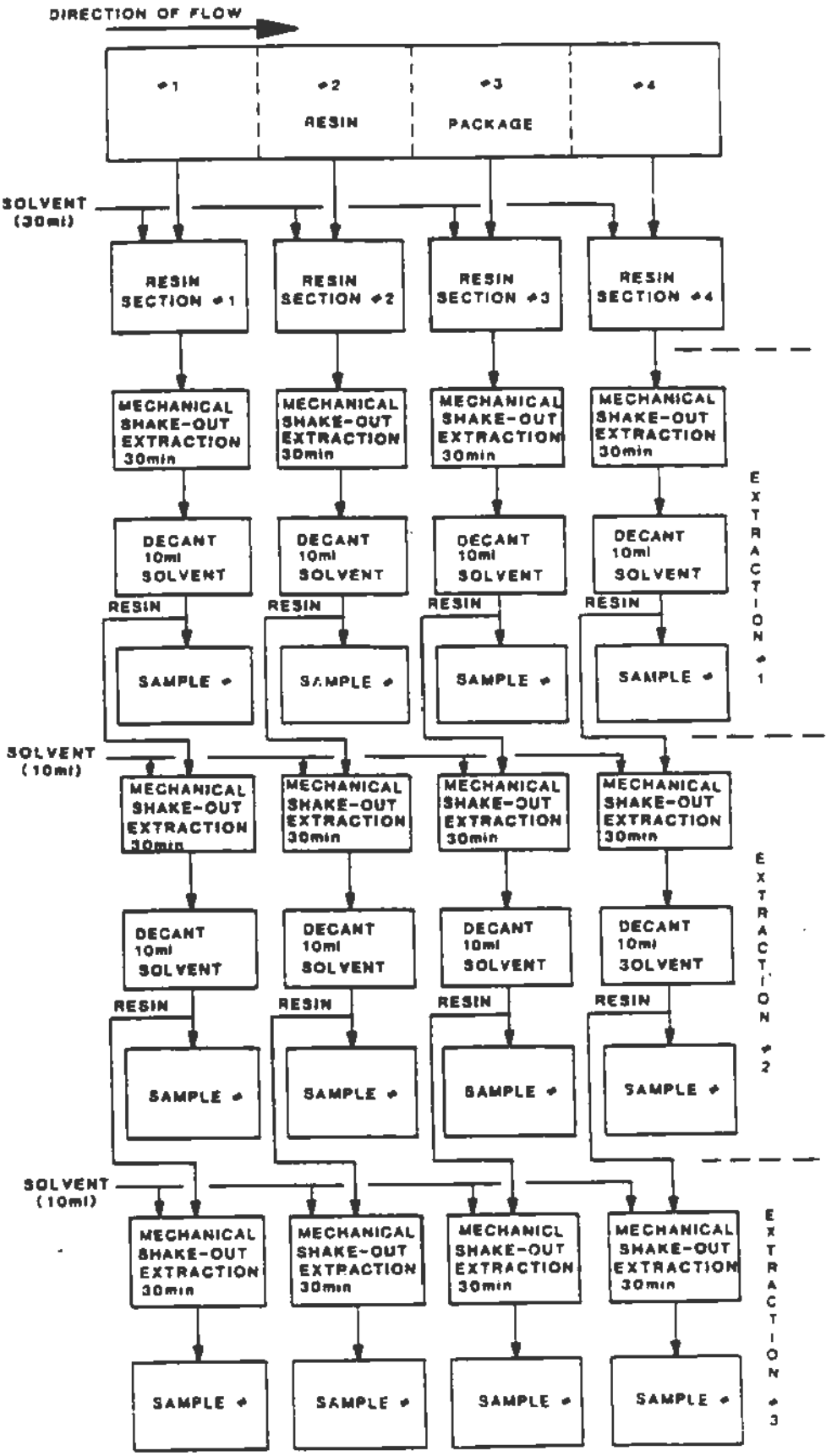

FIGURE 8-3. SAMPLING TRAIN RESIN MODULE RECOVERY

8.3.2. In order to determine a material balance on the energetic compounds, all residues from the process must be collected in addition to the stack gas sampling. Residues will be either shakeout extracted (paragraph 8.2.3) or liquid-liquid extracted (paragraph 8.2.2) depending upon their matrix, and the resulting solution will be analyzed by gas chromatography. The same analytical procedures will be utilized as with the sampling train samples.

APPENDIX F

AEHA ANALYTICAL PROCEDURE FOR COMPOUNDS IN STACK GAS SAMPLES

APPENDIX F

This Appendix contains the analytical procedures for determining the concentrations of energetic materials in stack gas. The text has been taken from a previous QA/QC plan for the APE 1236 at the Tooele Army Depot. Also contained in this Appendix is the Data Validation Method and the EPA approval memo for the AEHA STEM Methodology.

APPENDIX A

METHOD VALIDATION DATA

1. Analytical Procedures.

1.1. POHC Compounds.

1.1.1. The detection limit of the analytical procedure for NG, 2,4-dinitrotoluene (2,4-DNT), and 2,6-dinitrotoluene (2,6-DNT) in toluene is 0.025 µg/mL of solution in laboratory samples of only the POHC compound and toluene. Contamination of the solution by combustion byproducts in all probability will decrease the detection limit (increase the minimum concentration). The 0.025 µg/mL detection limit is based on the amount of POHC needed to produce gas chromatographic peaks whose heights are five times the height of the baseline noise. Taking matrix effects into consideration and without preconcentration of the actual samples, the overall quantitation limit for NG, 2,4-DNT, and 2,6-DNT is approximately 2.0 to 3.0 µg per train component.

1.1.2. Multiple injections of analytical standards of the 2,4-DNT, 2,6-DNT, and NG of 0.05 to 2.00 µg/mL were analyzed. The linearities that were achieved for the concentration range represent the calibration curve for the POHC compound standard solutions. The correlation coefficients obtained from the least squares analysis of the data were 0.996 for NG, 0.998 for 2,6-DNT, and 0.997 for 2,4-DNT (refer to Figures A-1 through A-3). Standard solutions in the concentration range of 1 to 100 µg of these POHC compound per 20 mL of toluene were analyzed in triplicate (refer to Table A-1 for these results). The average coefficient of variation for NG obtained from the replicate analysis of the standards was 2.3 percent. By similar analysis, the average coefficient of variation was 10.51 percent for 2,6-DNT and 8.64 percent for 2,4-DNT. Blind controls submitted with actual samples were analyzed 30 days following control solution preparation. Results for the accuracy of these audit samples are presented in Table A-2.

1.1.3. Similar analyses were performed on solutions of 2,4,6-trinitrotoluene (2,4,6-TNT) in toluene and RDX in a mixture of iso-amylacetate and toluene. The compounds, RDX and 2,4,6-TNT, are listed here as POHC's even though these compounds do not fit into the strict regulatory definition of a POHC. The calibration curves for RDX and 2,4,6-TNT (Figures A-4 and A-5), as with the curve for those compounds listed in paragraph 1.1.2., are linear with correlation coefficients of 0.987 and 0.992, respectively. Triplicate analysis was performed on standard solutions of these compounds in the concentration range of 1 to 100 µg per 20 mL of solvent. The relative standard deviations for these analyses are also contained in Table A-1. The average coefficient of variation for 2,4,6-TNT was 16.63 percent, while the average for RDX was 13.85 percent. Results from the accuracy of audit samples containing these compounds are contained in Table A-2.

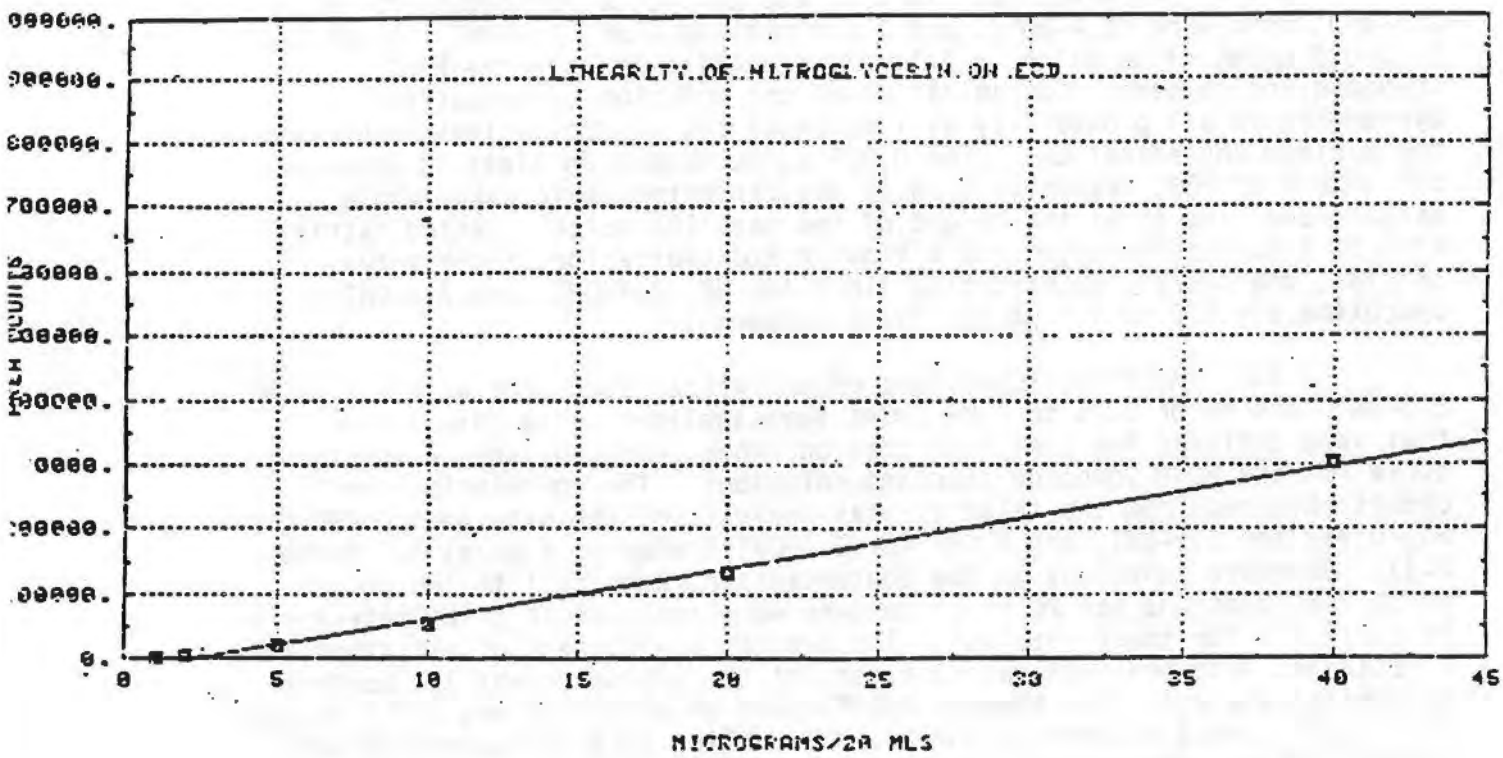

FIGURE A-1. LINEARITY OF NITROGLYCERIN ON ECD

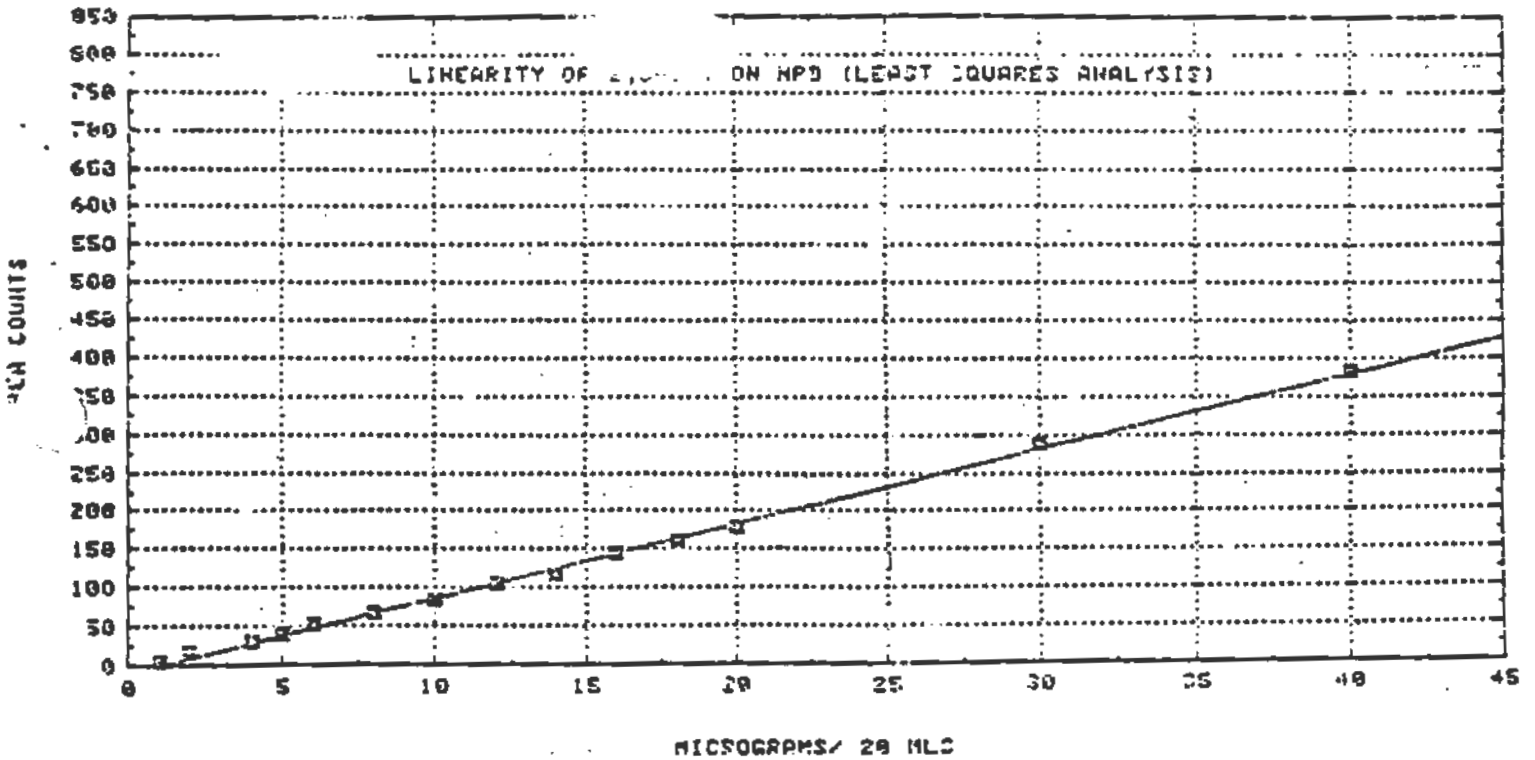

FIGURE A-2. LINEARITY OF 2,6-DNT ON NPD

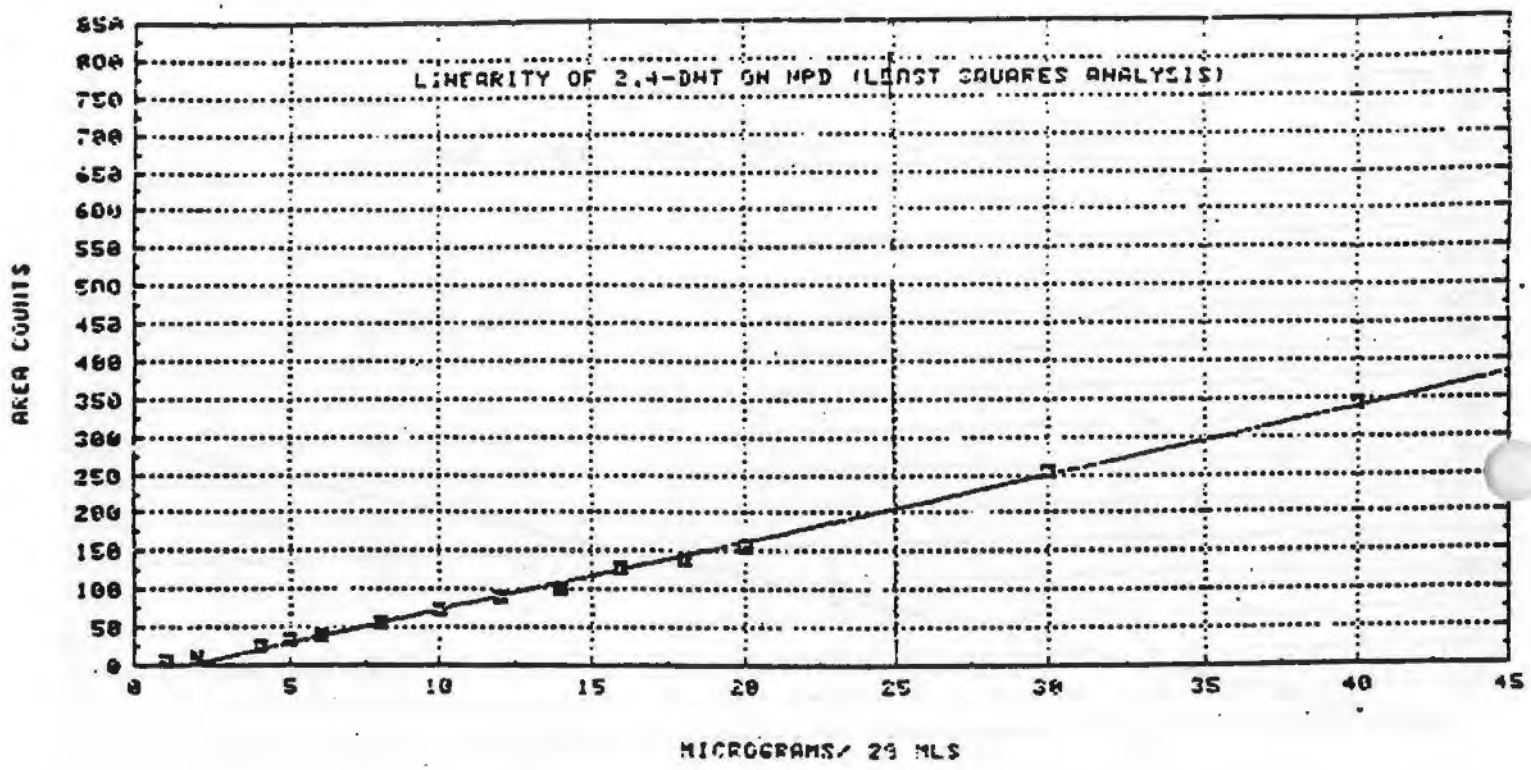

FIGURE A-3. LINEARITY OF 2,4-DN

TABLE A-1. PRECISION ANALYSIS FOR STANDARD SOLUTIONS OF POHC COMPOUNDS

Concentration (µg per 20 mL solvent*)	Compound	Relative Standard Deviation (%)
1.0	2,6-DNT	4.416
	4-DNT	0.565
	NG	3.100
	RDX	21.680
	2,4,6-TNT	32.901
2.0	2,6-DNT	- -
	2,4-DNT	- -
	NG	2.900
	RDX	- -
	2,4,6-TNT	- -
5.0	2,6-DNT	19.973
	2,4-DNT	20.736
	NG	2.800
	RDX	21.403
	2,4,6-TNT	24.794
10.0	2,6-DNT	- -
	2,4-DNT	- -
	NG	1.800
	RDX	- -
	2,4,6-TNT	- -
20.0	2,6-DNT	13.805
	2,4-DNT	4.941
	NG	2.500
	RDX	10.379
	2,4,6-TNT	13.137
40.0	2,6-DNT	4.335
	2,4-DNT	5.376
	NG	0.900
	RDX	14.643
	2,4,6-TNT	6.539
80.0	2,6-DNT	6.696
	2,4-DNT	1.882
	NG	- -
	RDX	7.376
	2,4,6-TNT	1.639
100.0	2,6-DNT	6.147
	2,4-DNT	4.213
	NG	- -
	RDX	4.040
	2,4,6-TNT	2.057

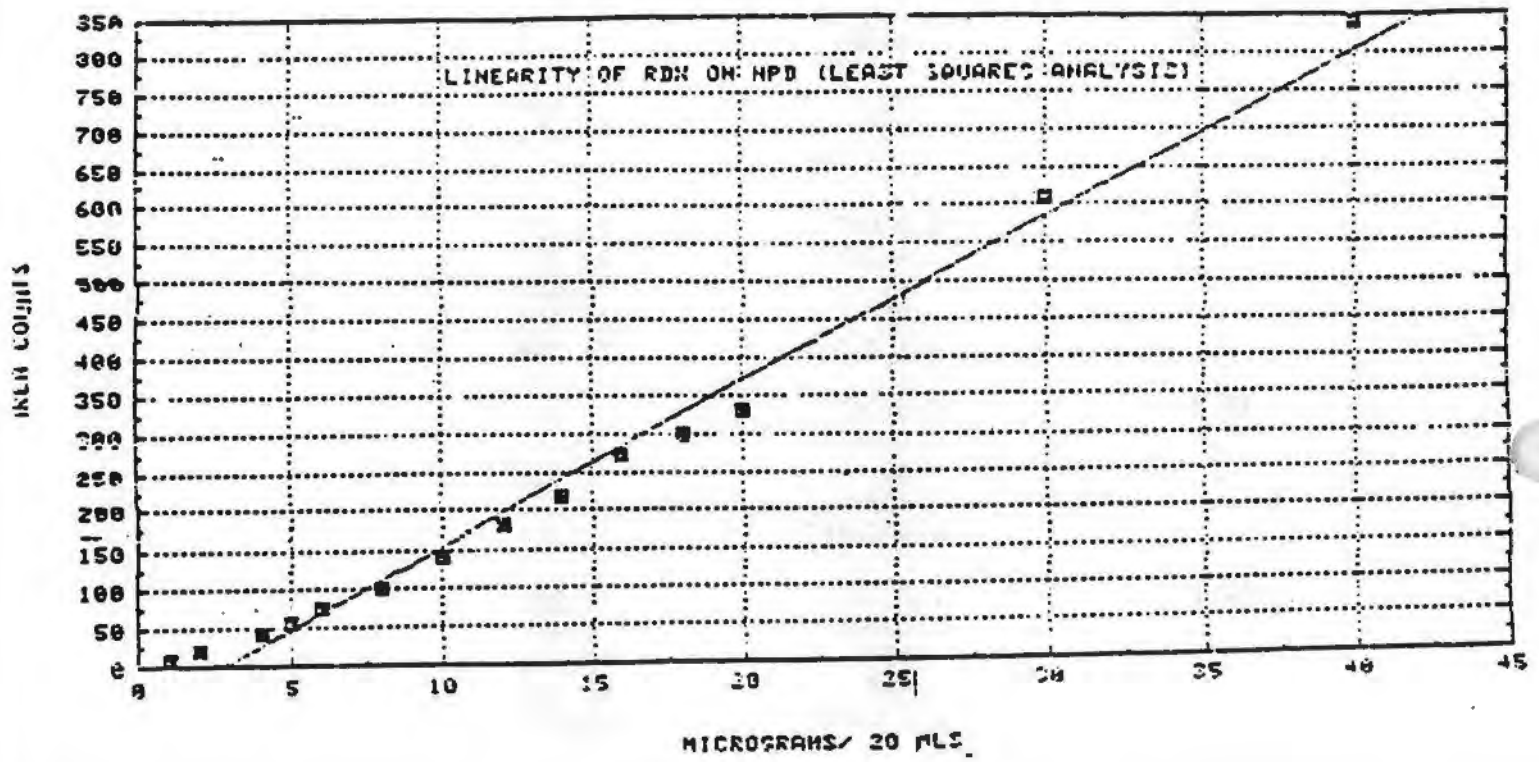

FIGURE A-4. LINEARITY OF RDX ON NPD

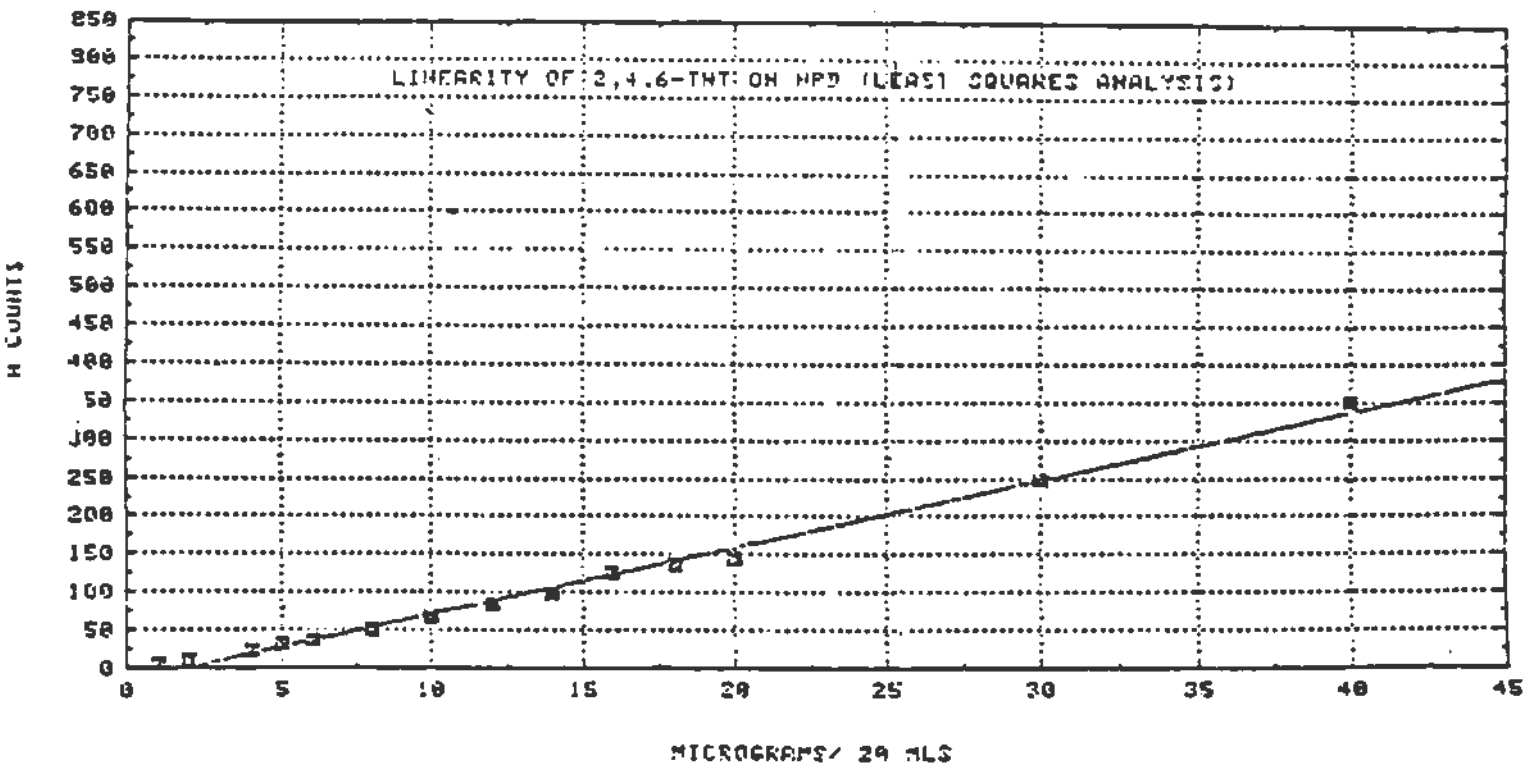

FIGURE A-5. LINEARITY OF 2,4,6-TNT ON NPD

TABLE A-2. RESULTS FROM THE ANALYSIS OF AUDIT SAMPLES

Energetic Compound	No. of Samples	Average Recovery (%)	Recovery Std Dev (%)	Average Dev (%)	Ave. Abs. Dev (%)
NG	10	99.8	1.9	0.2	1.6
2,4-DNT	10	104.4	5.4	-4.5	5.9
2,4,6-TNT	6	99.0	2.8	-1.0	2.4
RDX	6	98.7	1.3	-1.3	1.5

1.2. Surrogate Compounds.

1.2.1. These compounds are spiked onto the sampling train before use in order to monitor the recovery efficiency of the POHC and to ensure sample degradation is not occurring. Precision and accuracy data has been obtained for these compounds to ensure that their recoveries are on the same order as those for the POHC compounds and that they serve as reliable surrogates for the target POHC's.

1.2.2. Replicate analysis of analytical standards of 3,4-dinitrotoluene (3,4-DNT), 2,4,5-trinitrotoluene (2,4,5-TNT), and EGDN in toluene over a concentration range of 0.05 to 2.0 µg/mL were performed in order to generate calibration curves for these compounds. The calibration curves are illustrated in Figures A-6 through A-9. The correlation coefficients for the curves are 0.996 for ethylene glycol dinitrate, 0.998 for 3,4-DNT, and 0.992 for 2,4,5-TNT. A precision analysis was also performed on standard solutions of the surrogate compounds. The results from this analysis are contained in Table A-3. The average coefficients of variation were 1.61 for EGDN, 13.79 for 3,4-DNT, and 22.15 for 2,4,5-TNT.

2. Extraction Procedures.

2.1. Nitroglycerin Stability in Water. According to the Army's Technical Manual on Military Explosives (TM9-1300-214, reference 12), NG is hydrolyzed by water. The rate of this hydrolysis reaction was not determined. Since the greatest amount of work appears to be in sampling for NG, there was concern over sample degradation due to NG remaining in the impinger water during shipment and the contacting of water and NG on the resin bed. Therefore, the shakeout extraction was explored, since it would provide a relatively simple and effective method for field extractions of the exhaust gas samples. (Bench scale testing of the shakeout extraction method is discussed in paragraphs 2.2 and 2.3 of this Appendix.

2.1.1. Following discussions with EPA in 1986 (reference 13), a study was conducted to establish the stability of NG in water and to determine the rate of decay if it did exist. Distilled/deionized water after pH adjustment to 7.0 was spiked with NG and brought to a known volume (Rate of hydrolysis is pH dependent also). The water was shaken periodically and six 25 mL aliquots were obtained on a periodic basis for 3 weeks. Each of these aliquots was extracted with toluene for 30 minutes, and samples were obtained from the toluene fraction of the resulting 2-phase mixture.

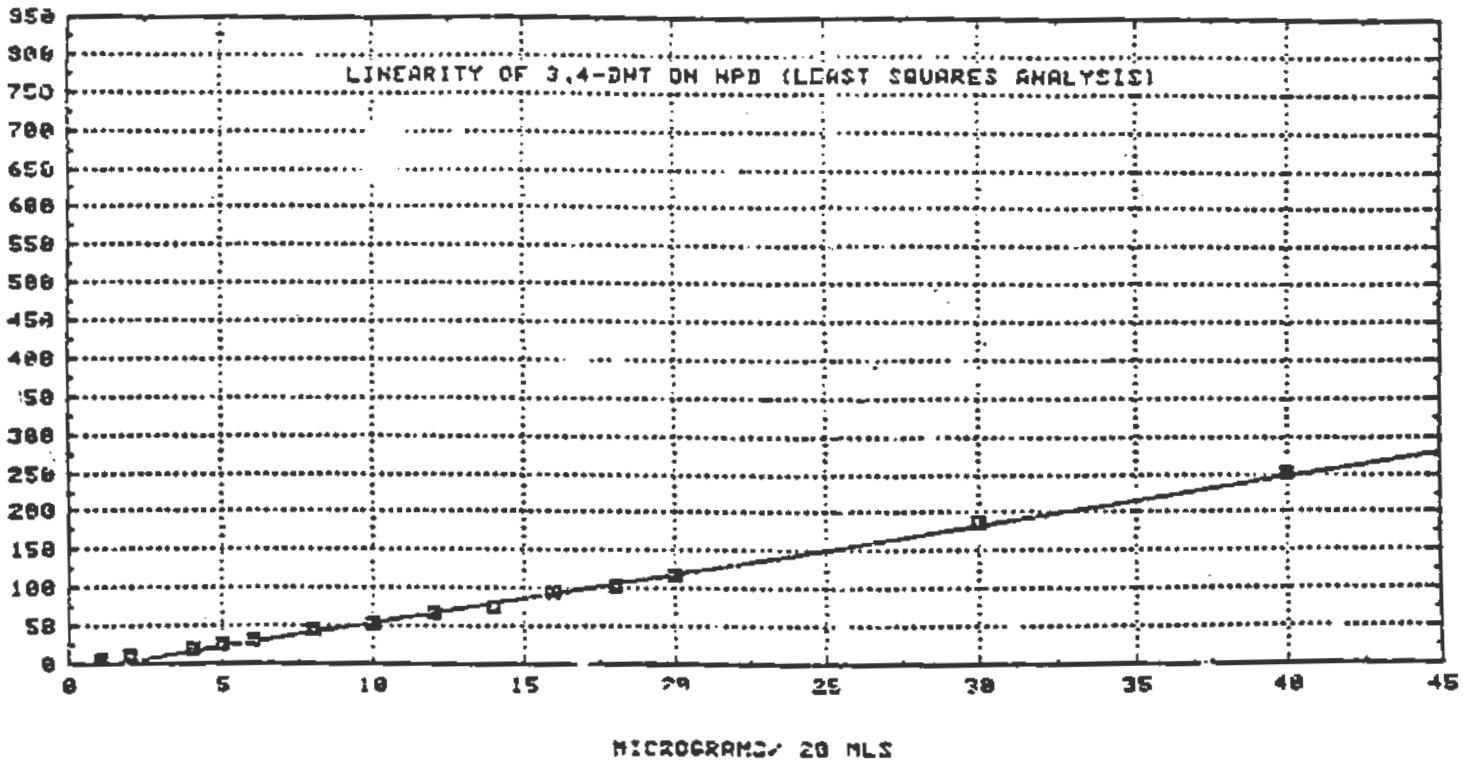

FIGURE A-6. LINEARITY OF 3,4-DNT ON NPD (LEAST SQUARES ANALYSIS)

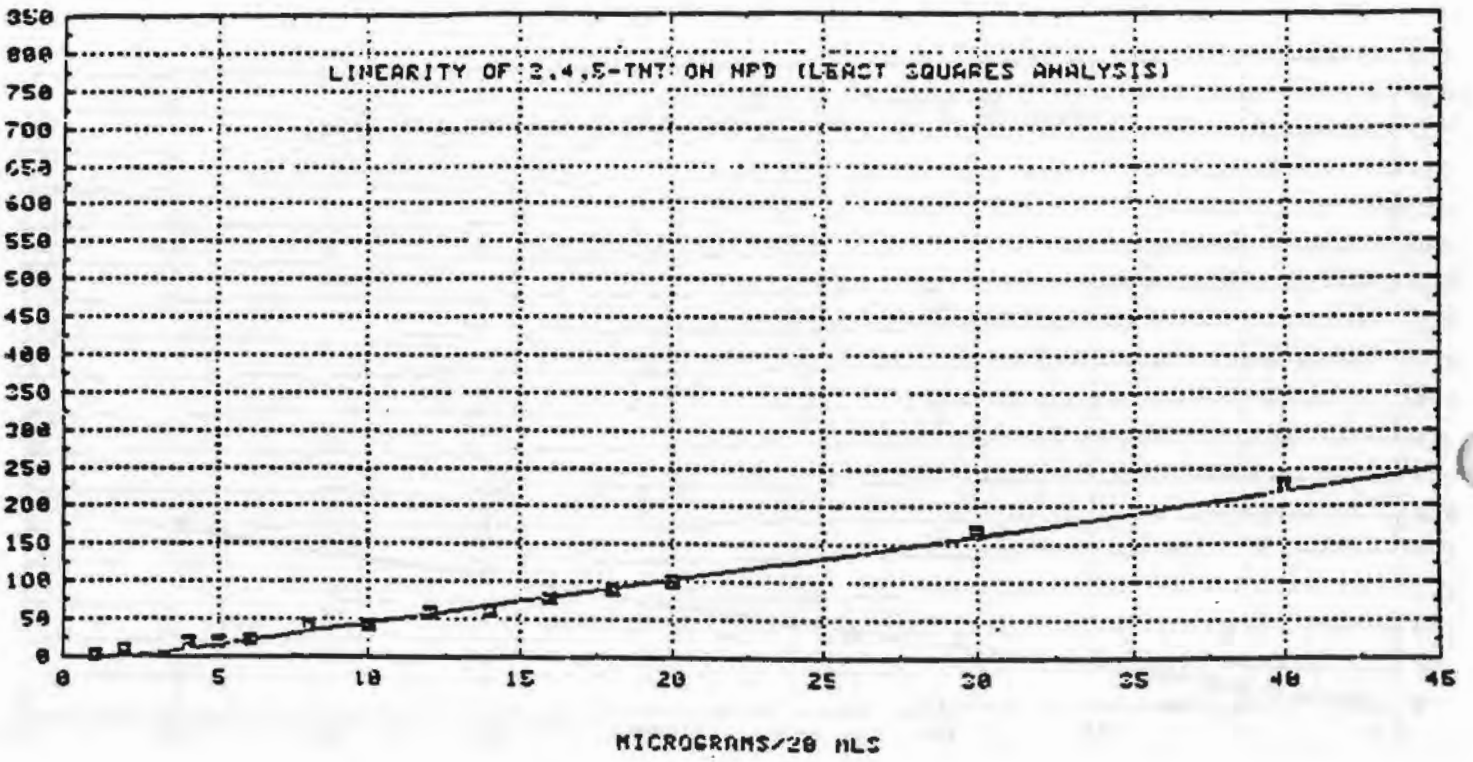

FIGURE A-7. LINEARITY OF 2,4,5-TNT ON NPD (LEAST SQUARES ANALYSIS)

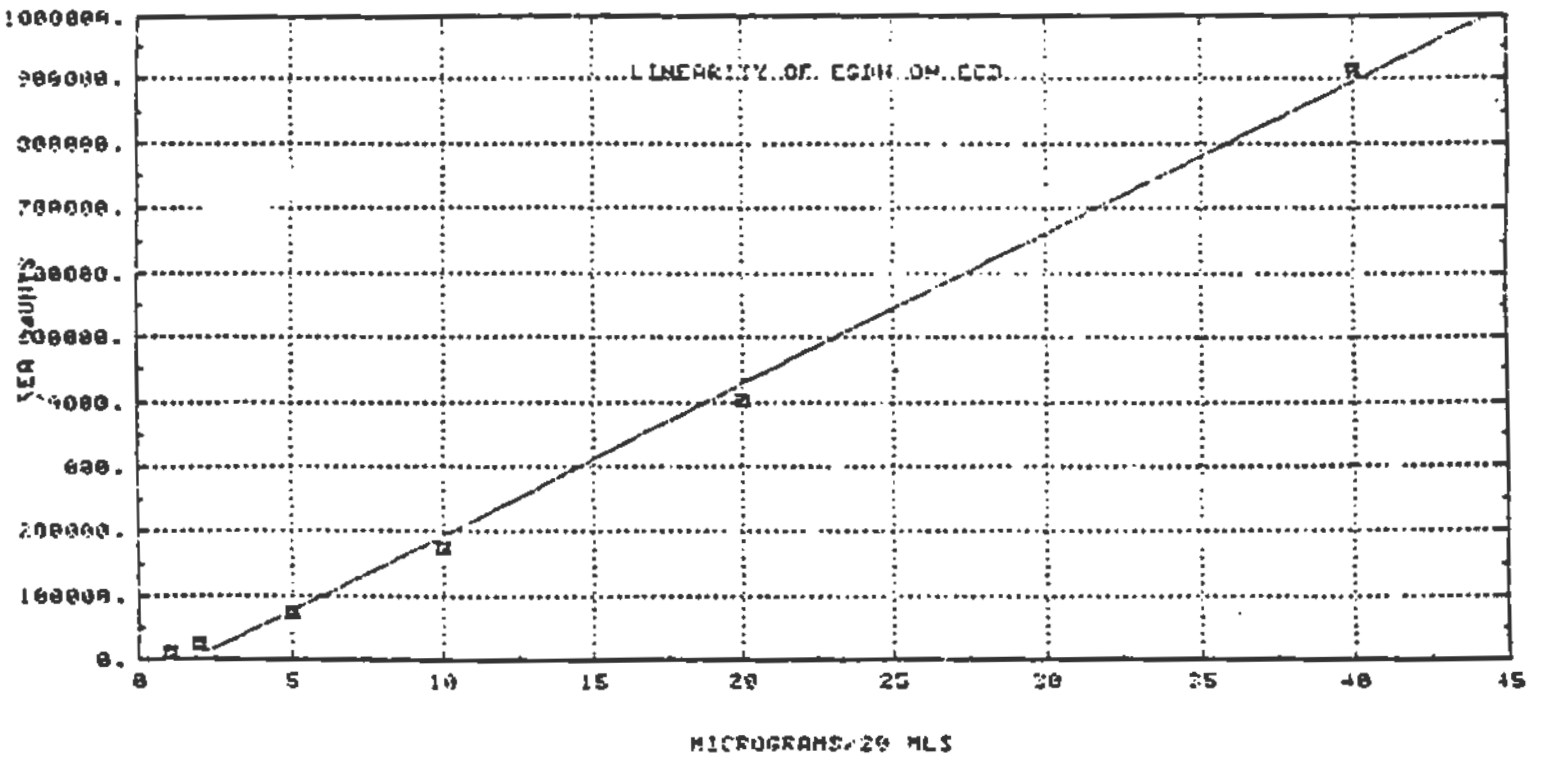

FIGURE A-8. LINEARITY OF EGDN ON ECD

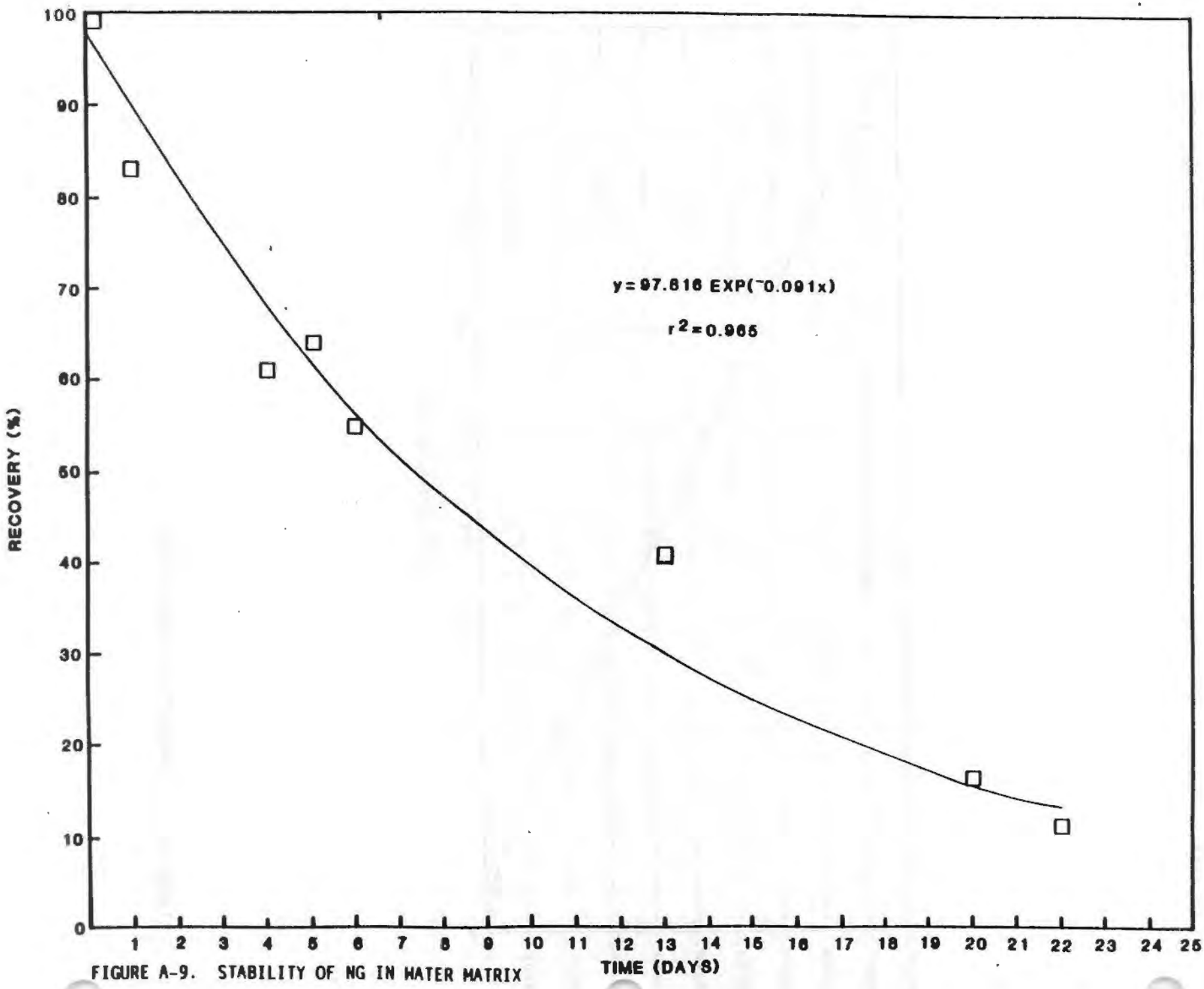

FIGURE A-9. STABILITY OF NG IN WATER MATRIX

TABLE A-3. PRECISION ANALYSIS FOR STANDARD SOLUTIONS OF SURROGATE COMPOUNDS

Conce on (µg per 20 mL solvent)	Compound	Relative Standard Deviation (%)
1.0	3,4-DNT	24.871
	2,4,5-TNT	41.204
	EGDN	0.431
2.0	3,4-DNT	- -
	2,4,5-TNT	- -
	EGDN	1.620
5.0	3,4-DNT	19.058
	2,4,5-TNT	19.864
	EGDN	2.300
	HMX	
10.0	3,4-DNT	- -
	2,4,5-TNT	- -
	EGDN	2.500
20.0	3,4-DNT	11.209
	2,4,5-TNT	13.698
	EGDN	1.700
40.0	3,4-DNT	7.471
	2,4,5-TNT	30.038
	EGDN	1.100
80.0	3,4-DNT	10.419
	2,4,5-TNT	12.065
	EGDN	- -
100.0	3,4-DNT	3.398
	2,4,5-TNT	8.542
	EGDN	- -

After the 3-week time period was over, the flask which contained the spiked water was rinsed with toluene followed by acetone. All samples were analyzed for NG using the methodologies discussed in Appendix B of this QA/QC plan.

2.1.2. Results of this stability study are illustrated in Figure A-9. The mean concentration for NG in the day zero sample was 188 µg/mL, which is 12 percent greater than the theoretical concentration of 167 µg/mL. Therefore, all recoveries are based on the originally observed value of 188 µg/mL versus the theoretical value of 167 µg/mL. Exponential regression analysis was performed on the analytical data and the curve for the percent loss versus time has a correlation coefficient of 0.965. According to the resulting curve, 50 percent of the original quantity of NG in a sample would have degraded in less than 8 days and losses in the first couple of days can also be appreciable. Each chromatogram was closely reviewed in an attempt to determine the degradation product, but neither the GC or subsequent GC/MS analysis could provide conclusive identification of the degradation compound.

2.2. Shakeout Extractions.

2.2.1. An extraction efficiency study using shakeout procedures was conducted by bench scale testing to determine if the extraction procedure with toluene would be effective for NG, 2,4-DNT, 2,6-DNT, 2,4,6-TNT and RDX. Toluene was demonstrated to be an efficient solvent for the extraction of NG and TNT and DNT isomers from water. However, for low concentrations of RDX/HMX in water (50 ppb), recoveries of the target compounds were very low using toluene. Figure A-10 illustrates the variability of the extraction efficiency with various toluene/iso-amylacetate concentrations as a binary solvent mixture. For this reason, when RDX is a target analyte, the extraction solvent is a mixture of iso-amylacetate and toluene (95:5 volume percent). Similar behavior was not evident at higher concentrations in water or at any concentration in the resin matrix. The methodology required evaluation of a low spike level and a high spike level condition for both the water and resin extractions. The resin extractions matched previous sampling train procedures (2.5 grams of XAD-2 resin with 20 mL of toluene) and the water extractions consisted of using a 4:1 water-to-toluene ratio. Results from these extraction studies are summarized in Table A-4. An additional extraction study was conducted to determine if subsequent extractions were needed to improve the efficiency of NG recovery. The results of this testing are summarized in Table A-5. A zero percent recovery indicates the extract had less than the detectable level of the POHC compound.

2.2.2. Recoveries demonstrated for NG (refer to Table A-4) are all in excess of 90 percent. Recoveries for 2,4-DNT and 2,6-DNT are slightly in excess of 100 percent. The additional extraction study (refer to Table A-5) demonstrates that a single extraction should be sufficient to obtain efficient recoveries of the POHC compounds from the sample train.

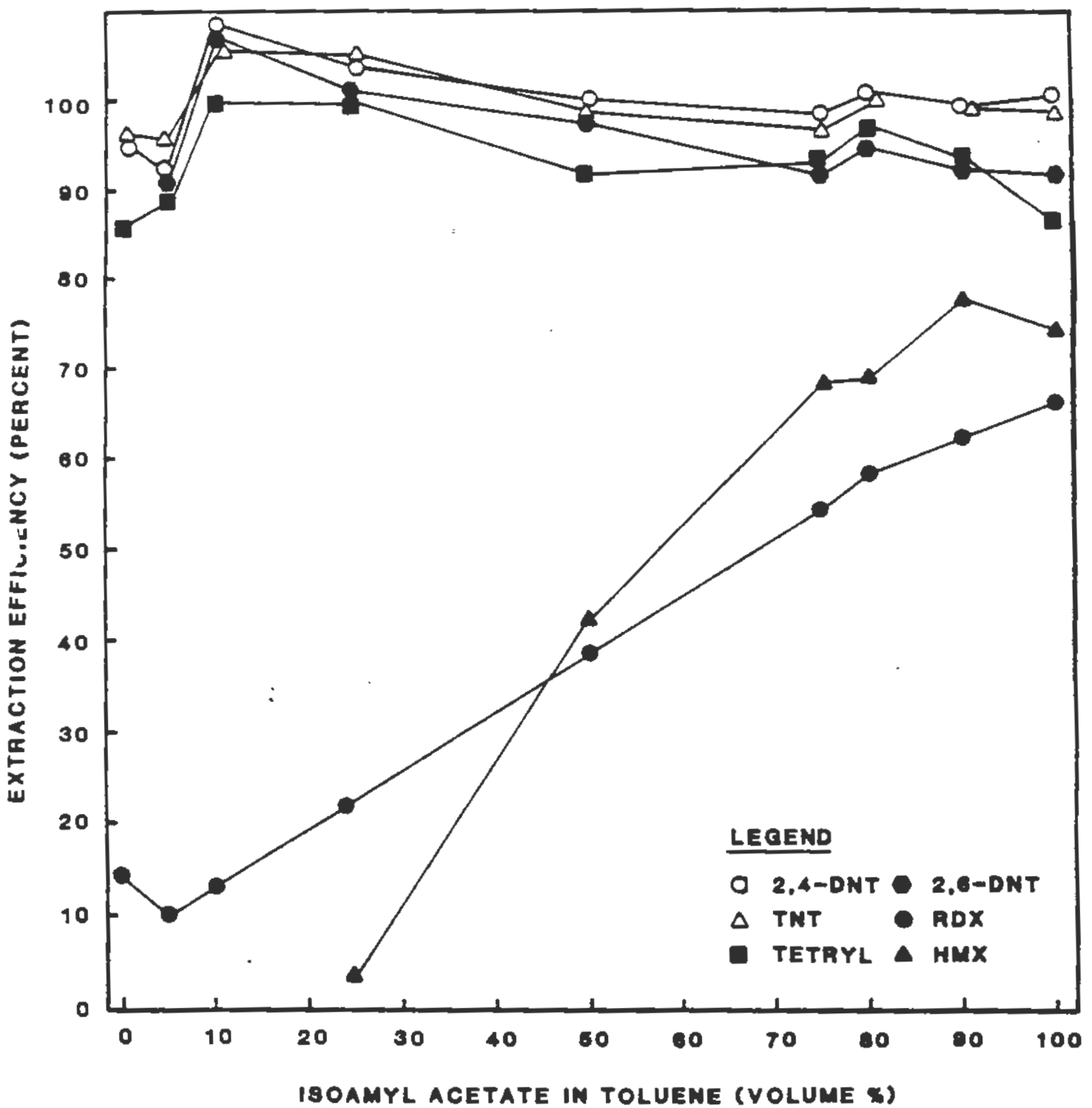

FIGURE A-10. EFFECT OF VARIABILITY IN CONCENTRATION OF TOLUENE/ISO-AMYLACETATE EXTRACTION SOLVENT ON EXTRACTION EFFICIENCY OF 50 ppb ENERGETIC MATERIAL IN WATER

TABLE A-4. EXTRACTION EFFICIENCY STUDY

Matrix	Compound	Spike Condition	Spike Level (µg)	Average Recovery (%)	Standard Deviation (%)
Resin	NG	Low	10	101	8
		High	100	131	9
	2,4-DNT	Low	20	118	7
		High	200	100	4
	2,6-DNT	Low	20	124	9
		High	200	116	3
	RDX	Low	100	100	5
		High	1000	67	3
	TNT	Low	20	106	6
		High	200	83	4
Water	NG	Low	5	91	10
		High	50	98	4
	2,4-DNT	Low	10	121	11
		High	100	122	3
	2,6-DNT	Low	10	105	7
		High	100	110	3
	RDX	Low	50	111	18
		High	500	94	5
	TNT	Low	10	125	10
		High	100	118	3

TABLE A-5. EFFECT OF SUBSEQUENT EXTRACTION ON OVERALL EXTRACTION EFFICIENCY

Spike Level	Sample Matrix/Extraction	Average Recovery (%) NG	2,4-DNT	2,6-DNT	RDX	TNT
1 µg each	Water/first	109	125	122	11	103
	Water/second	0	0	0	0	0
	Resin/first	0	96	100	0	69
	Resin/second	0	0	0	0	0
2 µg each	Water/first	83	123	121	72	113
	Water/second	0	0	0	0	0
	Resin/first	91	121	109	104	100
	Resin/second	0	0	0	0	0
10 µg each	Water/first	96	99	100	61	93
	Water/second	0	0	0	20	0
	Resin/first	60	110	115	39	78
	Resin/second	0	0	0	6	7
100 µg each	Water/first	89	103	97	52	91
	Water/second	3	4	4	25	5
	Resin/first	93	103	98	64	72
	Resin/second	0	0	0	13	10

2.3. Shakeout and Soxhlet Extractions. Also as a result of the meeting held with EPA in 1986, a bench scale test was conducted to evaluate the completeness of recovery of the target compounds from the resin using shakeout techniques. Furthermore, the size of each resin section was increased from 2.5 to 5.0 grams, but the volume of solvent remained unchanged (20 mL of toluene). The results for these multiple extractions are summarized in Table A-6. For the compounds of interest, the majority of the recovery occurs in the first extraction and under good solvent/resin mixing the recoveries are greater than 80 percent. The spiked resins following the third extraction were soxhlet extracted to ensure complete recovery was being achieved. Seven solvents were evaluated for their efficiencies towards extracting these energetic compounds in a soxhlet apparatus. These solvents were: acetone, methylene chloride, isooctane, toluene, pentane, acetonitrile, and methanol. A summary of the results for determining a suitable solvent are contained in Table A-7.

TABLE A-6. RECOVERY OF ANALYTES FROM MULTIPLE EXTRACTIONS OF 5 GRAM RESIN SECTIONS USING SHAKEOUT TECHNIQUES

Spike Level	Number	NG	2,6-DNT	2,4-DNT	3,4-DNT	2,4,6-TNT	2,4,5-TNT
2 µg	First	65.0	75.0	80.0	80.0	80.0	70.0
	Second	20.0	12.5	9.0	6.0	2.5	9.0
	Third	0.0	5.0	0.0	0.0	1.5	0.0
	Total	85.0	92.5	89.0	86.0	84.0	79.0
10 µg	First	97.0	90.0	88.0	89.0	94.0	99.0
	Second	0.0	7.0	8.0	6.0	2.0	0.0
	Third	0.0	0.0	0.0	0.0	0.0	0.0
	Total	97.0	97.0	96.0	95.0	96.0	99.0
50 µg	First	91.6	92.0	95.4	91.2	92.4	96.0
	Second	0.0	0.0	0.0	0.0	1.2	0.0
	Third	1.4	2.2	0.4	0.6	0.0	0.0
	Total	93.0	94.2	95.8	91.8	93.6	96.0
100 µg	First	101.0	77.3	72.4	73.0	72.2	74.8
	Second	0.0	11.8	14.7	14.9	14.1	7.9
	Third	1.1	0.0	0.0	0.0	0.5	0.0
	Total	102.1	89.1	87.1	87.9	86.8	82.7

TABLE A-7. RECOVERY OF ANALYTES FROM SPIKED RESIN USING SOXHLET EXTRACTION - SPIKE LEVEL OF 150 µg

	Average Recovery (%)					
Solvent	NG	2,4-DNT	2,6-DNT	3,4-DNT	2,4,6-TNT	2,4,5-TNT
Acetone	106	101	96	96	84	79
Methylene Chloride	115	103	97	94	85	83
Isooctane	63	56	36	39	24	14
Toluene	101	92	92	91	90	91
Pentane*	49	47	27	29	16	6
Acetonitrile	92	85	82	83	79	78
Methanol	85	70	59	69	60	51

* - Based on single sample analysis

Because methylene chloride demonstrated the greatest average efficiency for all six compounds and because EPA methodology currently uses methylene chloride for the soxhlet methodology, methylene chloride was used to evaluate the effectiveness of the shakeout procedure. However, methylene chloride does present problems in the analysis via GC with an electron capture detector. Each resin section when placed in the soxhlet apparatus still contained interstitial solvent (toluene). Therefore, the methylene chloride was evaporated from the resulting sample using sample "sweat-down" techniques until the toluene was the major concentration solvent (>95 percent). Due to the labile nature of the target compounds, standard solutions of the six target compounds in methylene chloride were first subjected to the sweat-down and then redissolved in toluene. The recoveries obtained are summarized in Table A-8.

TABLE A-8. RECOVERY OF TARGET COMPOUNDS FROM KUDERNA-DANISH CONCENTRATION OF METHYLENE CHLORIDE SOLUTIONS

	Average Recovery (%)					
Spike Level	NG	2,4-DNT	2,6-DNT	3,4-DNT	2,4,6-TNT	2,4,5-TNT
2 µg	83	92	92	97	57	36
10 µg	30	97	90	101	74	71
50 µg	69	129	133	139	122	104
100 µg	81	108	114	118	109	84

The sweat-down of these samples even under mild conditions is not desirable. The recoveries demonstrate that while some recoveries are high, the possibility still exists for loss of the POHC or its surrogate. The spiked resins referred to above were soxhlet extracted with methylene chloride and after correcting for the residual analyte from the third extraction, the concentration of the analytes in the solutions from the third extract was nondetectable at the 0.05 µg/mL level.

3. Sampling Procedure.

3.1. The first phase of the field validation involved the sampling of incinerator burner exhaust by sampling trains spiked with NG, 2,4-DNT, and 2,6-DNT and the matching of the spike recoveries with those from identical trains which received no sampling exposure. The spiking locations were the first resin section and the first impinger prior to the sampling exposure, and the filter following the sampling exposure. In order to facilitate spiking the first impinger, 50 mL of distilled/deionized water was added,

even though this differs from the configuration of the train as discussed in Section 6 of this QA/QC plan. Since this impinger has a short stem, no impinging action took place. Three different spike levels were used and for each spike level a second train was spiked, but it received no sampling exposure. These second trains are referred to as the QA trains. The recovery results are summarized in Table A-9. Variations in the results for NG recovery during Trial 1 testing prompted a second trial with spiked trains with improved analytical techniques (refer to Table A-10). Results with the QA trains demonstrated that the sampling exposure did not affect spike recovery, and therefore, these trains were not repeated in the second trials. The NG recoveries obtained during the second trial were greater than 90 percent and more consistent results were encountered.

3.2. The second phase consisted of actual field testing [during the incineration of propellent, explosive, pyrotechnic (PEP) materials] of the sampling train and comparison of the POHC collection performance between the simultaneous operation of this train and the EPA sampling train configuration. The collection efficiency for the USAEHA sampling train configuration are summarized in Tables A-10 through A-13. Based on individual runs, the two sampling train configurations may be considered equivalent.

3.3. An additional concern with the sampling train configuration is the ability to cool high temperature stack gases to less than 68 °F prior to entering the resin cartridge. A bench scale test was conducted in June 1987 to obtain temperature profiles of the exhaust gas at different locations throughout the sampling train (reference 14). For ΔH of 1.0, the temperature of the exhaust gas entering the resin cartridge and exiting the last impinger was below 60 °F even though the temperature of the flue gas entering the train ranged from 1,746 to 1,852 °F. For ΔH of 2.5, the exhaust gas temperature entering the resin cartridge was below 68 °F and the temperature of the gas exiting the last impinger was below 68 °F. The temperature range of the gas entering the sampling train was 1,728 to 1,802 °F. The sampling train, therefore, is capable of lowering the temperature of gas sampled from high temperature stacks to meet the temperature requirement for the resin (<68 °F) with three impingers before the resin cartridge and an ice bath.

TABLE A-9. SAMPLING TRAIN SPIKE RECOVERY (INITIAL)

Condition*	Spike Location	Average Recovery (%)/ Standard Deviation (%) NG	2,6-DNT	2,6-DNT
1X STD Train	Filter	103/6	108/6	109/7
	Impinger	73/18	109/7	106/9
	Resin	156/30	98/3	94/8
1X QA Train	Filter	102/11	112/3	110/2
	Impinger	89/1	109/4	108/5
	Resin	69/†	104/†	106/†
2X STD Train	Filter	104/25	100/11	96/10
	Impinger	83/44	113/1	109/3
	Resin	109/5†	†	95/9
2X QA Train	Filter	84/†	67/1	62/1
	Impinger	134/45	113/1	106/1
	Resin	76/2	121/2	116/1
10X STD Train	Filter	96/16	119/2	118/1
	Impinger	138/28	115/2	110/2
	Resin	135/41	108/4	100/5
10X QA Train	Filter†	95/21	124/5	122/7
	Impinger	127/4	119/3	119/3
	Resin	92/15	122/3	122/4

* The baseline level (1X) from spiked analytes are as follows: 10 µg NG, 5 µg 2,4-DNT, and 5 µg 2,6-DNT. Each of the spike levels are a multiple of this baseline.
† This note indicates either the data are limited or affected by a lost sample, an analytical problem, or an original spiking error.

TABLE A-10. COMPARISON OF SAMPLING TRAIN SPIKE RECOVERY FOR NITROGLYCERIN

Train Type	Spike Level (µg)	Spike Location	Trial 1		Trial 2	
			Average Recovery (%)	Standard Deviation (%)	Average Recovery (%)	Standard Deviation (%)
Standard	10 µg	Filter	103	6		
		Impinger	73	18		
		Resin	156	30		
QA	10 µg	Filter	102	11		
		Impinger	89	1		
		Resin	69	-		
Standard	20 µg	Filter	104	25	99	1
		Impinger	83	44	103	4
		Resin	109	5	92	5
QA	20 µg	Filter	84	-		
		Impinger	134	45		
		Resin	76	2		
Standard	100 µg	Filter	96	16	100	3
		Impinger	138	28	105	2
		Resin	135	41	99	3
QA	100 µg	Filter	95	21		
		Impinger	127	4		
		Resin	92	15		

TABLE A-11. NITROGLYCERIN COLLECTION EFFICIENCY

Run Number Train Configuration	3-4 USAEHA	3-4 EPA	5-6 USAEHA	5-6 EPA	7-8 USAEHA	7-8 EPA
Gas Volume Sampling (std. cubic feet, dry)'	61.9	57.8	64.0	63.1	69.6	66.3
Total POHC Collected (µg)	19.7	14.5	33.1	35.6	19.7	24.1
POHC Distribution (% of total)"						
Impinger/Condenser						
1st extraction	81.7	14.5	68.9	11.0	76.1	0
2d extraction	0	0	6.6	0	0	0
3d extraction	0	0	0	0	0	0
Resin						
1st section	18.3	85.5	24.5	59.6	23.9	76.3
2d section	0	0	0	9.8	0	0
3d section	0	0	0	0.0	0	0
4th section	0	0	0	10.1	0	0
Condensate Trap						
1st extraction	-	0	-	9.6	-	14.9
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

See footnote on page A-24.

Run Number Train Configuration	9-10 USAEHA	9-10 EPA	11-12 USAEHA	11-12 EPA	13-14 USAEHA	13-14 EPA
Gas Volume Sampling (std. cubic feet, dry)	36.8	37.0	42.0	39.9	40.4	38.1
Total POHC Collected (µg)	16.7	16.3	12.2	14.6	15.4	14.0
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	64.1	9.2	36.1	11.0	58.4	33.6
2d extraction	0	0	0	0	0	0
3d extraction	0	0	0	0	0	0
Resin						
1st section	35.9	62.0	63.9	77.4	41.6	66.4
2d section	0	13.5	0	11.6	0	0
3d section	0	7.4	0	0	0	0
4th section	0	8.0	0	0	0	0
Condensate Trap						
1st extraction	-	0	-	0	-	0
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

* A zero percentage of the total NG collected indicates that none was detected in that sample. The quantitation limits for NG ranged from 0.8 µg to 2.0 µg per sample and were a function of the chromatogram complexity for each sample.

TABLE A-12. 2,4 DINITROTOLUENE COLLECTION EFFICIENCY

Run Number Train Configuration	9-10 USAEHA	9-10 EPA	11-12 USAEHA	11-12 EPA	13-14 USAEHA	13-14 EPA
Gas Volume Sampling (std. cubic feet, dry)	36 8	37.0	42.0	39.9	40.4	38.1
Total POHC Collected (µg)	127.2	127.8	141.5	138.5	158.2	145.1
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	52.9	11.8	49.1	25.3	57.8	25.3
2d extraction	4.6	0	3.0	1.0	4.6	3.1
3d extraction	0	0	0	0	2.0	0
Resin						
1st section	41.1	66.1	47.9	69.1	35.7	58.4
2d section	1.0	11.7	0	4.3	0	9.0
3d section	0	4.3	0	0.0	0	0
4th section	0	6.0	0	0	0	0
Condensate Trap						
1st extraction	-	0	-	0	-	0
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

See Footnote on page A-26.

Run Number Train Configuration	15-16 USAEHA	15-16 EPA	17-18 USAEHA	17-18 EPA	19-20 USAEHA	19-20 EPA
Gas Volume Sampling (std. cubic feet, dry)	81.3	77.7	69.8	65.4	72.9	72.0
Total POHC Collected (µg)	1303	1295	1924	1928	1792	1498
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	47.0	4.3	50.2	4.6	56.2	4.5
2d extraction	3.2	0.3	7.9	0.8	3.9	0.5
3d extraction	1.8	0.2	1.8	0.3	1.5	0.2
Resin						
1st section	47.7	65.0	40.1	61.4	38.2	77.0
2d section	0.3	14.2	0	15.8	0.3	11.3
3d section	0	6.3	0	5.5	0.0	3.7
4th section	0	3.7	0	3.7	0	1.7
Condensate Trap						
1st extraction	-	4.7	-	5.7	-	1.0
2d extraction	-	1.0	-	1.8	-	0
3d extraction	-	0.4	-	0.5	-	0

* A zero percentage of the total 2,4 DNT collected indicates that none was detected in that sample. The quantitation limits for 2,4 DNT ranged from 1.0 µg to 3.0 µg per sample and were a function of the chromatogram complexity for each sample.

TABLE A-13. 2,6 DINITROTOLUENE COLLECTION EFFICIENCY

Run Number Train Configuration	15-16 USAEHA	15-16 EPA	17-18 USAEHA	17-18 EPA	19-20 USAEHA	19-20 EPA
Gas Volume Sampling (std. cubic feet, dry)	81.3	77.7	69.8	65.4	72.9	72.0
Total POHC Collected (µg)	104.8	89.4	124.5	99.8	119.2	88.1
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	38.9	0	42.4	0	38.2	0
2d extraction	0	0	6.7	0	3.9	0
3d extraction	0	0	3.5	0	0	0
Resin						
1st section	61.1	72.1	47.4	71.6	57.9	91.4
2d section	0	16.8	0	12.7	0	8.6
3d section	0	7.0	0	5.6	0	0
4th section	0	4.0	0	3.2	0	0
Condensate Trap						
1st extraction	-	0	-	6.8	-	0
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

* A zero percentage of the total 2,6 DNT collected indicates that none was detected in that sample. The quantitation limits for 2,6 DNT ranged from 1.0 µg to 3.0 µg per sample and were a function of the chromatogram complexity for each sample.

F-2 EPA APPROVAL MEMO FOR USING AEHA STEM METHOD FOR SAMPLING NITROGLYCERIN AND DINITROTOLUENE IN STACK GAS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
AIR AND ENERGY ENGINEERING RESEARCH LABORATORY
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27711

MEMORANDUM

DATE: August 18, 1986

SUBJECT: Current Recommendation USAEHA Sampling Train, Based on Meeting at Research Triangle Park

FROM: Larry D. Johnson
Technical Support Office (MD-62)

TO: Betty Willis, Chief, KN/TN Unit
Waste Engineering Section, Region IV

As a result of my May 19, 1986, memo to you in which I recommended against use of the sampling train proposed by US Army Environmental Hygiene Agency (USAEHA), I was contacted by William K. Emily of Crane Naval Weapons Support Center in Crane, Indiana. Mr. Emily has been involved in preparation of the permit application for the Naval Weapons Station in Charleston, SC which also proposed the use of the USAEHA train. He felt that further information could be provided by USAEHA and asked to meet with us at RTP. The meeting was held 8/7/86 and was attended by the following people:

William K. Emily, Chemist, Crane Naval Weapons Support Center, Crane, IN
David L. Dangherill, Chief, Source Surveillance Board, USAEHA Aberdeen Proving Ground, MD
Captain Charles J. Mort, Air Pollution Engineering Division, USAEHA Aberdeen Proving Ground, MD
Larry D. Johnson, Research Chemist, EPA/AEERL, RTP
Merrill D. Jackson, Research Chemist, EPA/AEERL, RTP
John H. Margeson, Research Chemist, EPA/EMSL, RTP

During the meeting, new information was provided and was used to clarify some of the reasons behind the USAEHA procedures. The most important of this information was the fact that nitroglycerin hydrolyzes readily and presents sampling and analysis problems because of this, especially if heated during recovery. USAEHA was concerned with the Soxhlet extraction step in our procedures because of this, and also wanted to perform extraction in the field in order to remove the nitroglycerin from contact with water as quickly as possible.

After discussing this problem and other details with the group, we arrived at a series of modifications and requirements which we feel would make the USAEHA train and associated procedures acceptable for nitroglycerin and dinitrotoluene (I don't think it would be reasonable to let them use the USAEHA train on nitroglycerin and then require MM5 on dinitrotoluene).

The requirements we believe necessary are:

1. The USAEHA train must be loaded with at least 20g of sorbent instead of 10g. This can be in two sequential sorbent tubes, but one is preferable.

2. The sections of sorbent must be analyzed separately (they do this already), and if more than 10% of the total sorbent catch is found on the last section, then the sample run is void.

3. The extractions must be performed in the field, and must consist of three sequential shake extractions.

4. On the first project to use the USAEHA train, the extracted resin and filter must also be shipped back to the lab for Soxhlet extraction. The Soxhlet extracts will be analyzed and any POHC found will be added to the appropriate total for calculations of DRE. In the event that significant amounts of POHC are found in the Soxhlet extracts, USAEHA should be required to perform this QC extraction on subsequent trial burns. If no significant amount of POHC is found, then they should not be required to perform the Soxhlet extraction on subsequent burns. The choice of solvent for the Soxhlet extraction is still somewhat open. USAEHA is concerned that toluene will require excessive temperatures. Hexane, pentane, or cyclohexane are acceptable to us (as is toluene), but they need to decide which is preferable from their standpoint. We'll be glad to review what they propose.

5. The sorbent tube or tubes must be vertical rather than horizontal, and the gas flow should be downward through the resin bed. We realize this arrangement is more difficult for the field crew, but feel that it is necessary.

I do want to stress that we've only agreed to accept the USAEHA train for nitroglycerin and dinitrotoluene. Use of the train for other materials would have to be negotiated separately, and all of the EPA personnel present feel that we should require MM5 except in special cases.

I believe I've covered all the key points, but if I've missed anything, please let me know. I haven't included all the details and reasoning discussed, since we met for a whole day. I'll be glad to discuss any of this material or the subject in general.

cc: Beth Graham, Region IV
Caron Falconer, Region IV
Bob Reimer, Region IV
Gary Gross, Region III
Robin Anderson, OSW, WH-563
Sonya Stalmack, OSW, WH-563
M. D. Jackson, MD-62
J. H. Margeson, MD-77A
W. K. Emily, Crane Naval Weapons Ctr
D. L. Daugherill, USAEHA
Cpt. C. J. Hart, USAEHA

APPENDIX B

ANALYTICAL PROCEDURE
ENERGETIC COMPOUNDS IN STACK GAS SAMPLES

1. Scope and Application.

1.1. This method provides procedures for the detection and quantitation of energetic compounds from stack gas samples. The procedure is organized into a general section, which covers those aspects of the method which are independent of the target analyte, and specific sections for each compound validated on the sampling train.

1.2. Samples are collected in a modification of the EPA Method 5 sampling train where a 20-gram XAD-2 resin module has been placed downstream of three impingers. Therefore, the target compounds are captured in both the resin and water matrices. The sampling rate is on the order of 1-cubic meter of stack gas per hour.

1.3. This method is designed for use by analysts who are experienced in the use of a GC and a GC/MS.

2. Summary of Method. This method provides gas chromatographic operating conditions for the analysis of energetic compounds in a suitable extraction solvent (toluene or a mixture of toluene and iso-amylacetate). These samples are obtained from the solvent extraction of stack sampling train media - filter, impinger water, and resin. The target compounds are separated in the GC by temperature programming of a capillary column. These compounds are than detected by either a nitrogen/phosphorus (NP) or an electron capture detector (ECD). Positive results are confirmed on a GC/MS instrument if concentrations are adequate for GC/MS instrument detection.

3. Interferences. Any compound which has the same general retention time as the target analyte and gives a detector response is a potential interference to the analytical instruments. Laboratory reagent/solvent blanks and extracts from the XAD-2 resin must be analyzed to demonstrate the level of contamination that would interfere with the measurement. Modification of the gas chromatographic parameters may be utilized to circumvent interferences.

4. Safety. The following safety precautions are presented as guidelines only since safety procedures should already be in place for the analytical laboratories.

4.1. Protective Equipment. Throw-away plastic gloves, apron or laboratory coat, and safety glasses should be worn at all times in the laboratory areas. Skin contact with the energetic compounds and solvents should be avoided.

4.2. Personal Hygiene. As a backup to protective equipment, hands and lower arms should be washed thoroughly before any breaks (coffee, lunch, end of shift) with any mild soap.

4.3. Ventilation. The manipulation of samples and the use of reagents/solvents should be restricted to laboratory hoods. The electron capture detector on the gas chromatographic instruments must be properly vented.

4.4. Waste. Proper waste disposal techniques must be utilized and every effort should be made to minimize the generation of contaminated wastes.

5. Apparatus and Equipment.

5.1. Gas Chromatograph. Hewlett-Packard (HP) 5880 GC's capable of temperature programming and which are equipped with HP 7671 autoinjectors and capillary column systems operating in the splitless mode will be utilized. Depending upon the target analyte, the GC will be equipped with either an electron capture or nitrogen-phosphorus detector (see compound-specific sections). A Hewlett-Packard HP3357 Data System is interfaced with the GC for integrating peak areas and recording chromatograms.

5.2. Gas Chromatograph/Mass Spectrometer. The Finnigan Model 9610 system with temperature programming and splitless mode capillary column capabilities will be utilized. The Model 9610 Gas Chromatograph will have a direct interface with a Finnigan Model 4021 mass spectrometer. The mass spectral data are obtained with electron impact ionization at a nominal electron energy of 70 eV. A computerized data system is utilized to acquire, store, reduce and output the mass spectral data.

5.3. Chromatographic Columns. Refer to the compound specific sections.

5.4. Glassware.

5.4.1. Vials. Wheaton, screw-capped, septum vials with a volume of 40 mL and Teflon®-lined caps are used to extract resin sections. Additionally, 2 mL auto-sampler vials supplied by Hewlett-Packard are used.

5.4.2. Jars. Glass jars with Teflon-lined caps capable of holding 16 fluid ounces are used for the transportation of the extracts of impinger water, the samples of feed material and/or process residues (e.g., ash), and the particulate filter with the rinses of the probe liner and train glassware.

5.4.3. Separatory Funnels. Glass separatory funnels of 500 mL volume will be used in the extraction of aqueous samples.

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APPENDIX

ANALYTICAL PROCEDURE
ENERGETIC COMPOUNDS IN STACK GAS SAMPLES

1. Scope and Application.

1.1. This method provides procedures for the detection and quantitation of energetic compounds from stack gas samples. The procedure is organized into a general section, which covers those aspects of the method which are independent of the target analyte, and specific sections for each compound validated on the sampling train.

1.2. Samples are collected in a modification of the EPA Method 5 sampling train where a 20-gram XAD-2 resin module has been placed downstream of three impingers. Therefore, the target compounds are captured in both the resin and water matrices. The sampling rate is on the order of 1-cubic meter of stack gas per hour.

1.3. This method is designed for use by analysts who are experienced in the use of a GC and a GC/MS.

2. Summary of Method. This method provides gas chromatographic operating conditions for the analysis of energetic compounds in a suitable extraction solvent (toluene or a mixture of toluene and iso-amylacetate). These samples are obtained from the solvent extraction of stack sampling train media - filter, impinger water, and resin. The target compounds are separated in the GC by temperature programming of a capillary column. These compounds are than detected by either a nitrogen/phosphorus (NP) or an electron capture detector (ECD). Positive results are confirmed on a GC/MS instrument if concentrations are adequate for GC/MS instrument detection.

3. Interferences. Any compound which has the same general retention time as the target analyte and gives a detector response is a potential interference to the analytical instruments. Laboratory reagent/solvent blanks and extracts from the XAD-2 resin must be analyzed to demonstrate the level of contamination that would interfere with the measurement. Modification of the gas chromatographic parameters may be utilized to circumvent interferences.

4. Safety. The following safety precautions are presented as guidelines only since safety procedures should already be in place for the analytical laboratories.

4.1. Protective Equipment. Throw-away plastic gloves, apron or laboratory coat, and safety glasses should be worn at all times in the laboratory areas. Skin contact with the energetic compounds and solvents should be avoided.

4.2. Personal Hygiene. As a backup to protective equipment, hands and lower arms should be washed thoroughly before any breaks (coffee, lunch, end of shift) with any mild soap.

4.3. Ventilation. The manipulation of samples and the use of reagents/solvents should be restricted to laboratory hoods. The electron capture detector on the gas chromatographic instruments must be properly vented.

4.4. Waste. Proper waste disposal techniques must be utilized and every effort should be made to minimize the generation of contaminated wastes.

5. Apparatus and Equipment.

5.1. Gas Chromatograph. Hewlett-Packard (HP) 5880 GC's capable of temperature programming and which are equipped with HP 7671 autoinjectors and capillary column systems operating in the splitless mode will be utilized. Depending upon the target analyte, the GC will be equipped with either an electron capture or nitrogen-phosphorus detector (see compound-specific sections). A Hewlett-Packard HP3357 Data System is interfaced with the GC for integrating peak areas and recording chromatograms.

5.2. Gas Chromatograph/Mass Spectrometer. The Finnigan Model 9610 system with temperature programming and splitless mode capillary column capabilities will be utilized. The Model 9610 Gas Chromatograph will have a direct interface with a Finnigan Model 4021 mass spectrometer. The mass spectral data are obtained with electron impact ionization at a nominal electron energy of 70 eV. A computerized data system is utilized to acquire, store, reduce and output the mass spectral data.

5.3. Chromatographic Columns. Refer to the compound specific sections.

5.4. Glassware.

5.4.1. Vials. Wheaton, screw-capped, septum vials with a volume of 40 mL and Teflon®-lined caps are used to extract resin sections. Additionally, 2 mL auto-sampler vials supplied by Hewlett-Packard are used.

5.4.2. Jars. Glass jars with Teflon-lined caps capable of holding 16 fluid ounces are used for the transportation of the extracts of impinger water, the samples of feed material and/or process residues (e.g., ash), and the particulate filter with the rinses of the probe liner and train glassware.

5.4.3. Separatory Funnels. Glass separatory funnels of 500 mL volume will be used in the extraction of aqueous samples.

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5.4.4. Miscellaneous. Volumetric flasks with ground glass stoppers, disposable pipets, graduated cylinders, and micro-syringes are used for the preparation of standards, performance of dilutions, and conducting injections to the gas chromatograph instrument.

5.5. Additional Equipment.

5.5.1. Mechanical Shaker. A roto-rack capable of 20 rotations per minute will be used for the extraction of resin samples.

5.5.2. Miscellaneous. Stainless steel spatulas or spoons may be used in preparing the samples for analysis.

6. Reagents and Solvents.

6.1. Solvents. ACS grade: toluene, acetone, iso-amylacetate.

6.2. Reagents.

6.2.1. 99.6 percent or greater purity of 2,4-DNT, 2,6-DNT, 3,4-DNT, 2,4,6-TNT, 2,4,5-TNT, NG, and RDX (SRM's from Picatinny Arsenal).

6.2.2. ACS grade anhydrous sodium sulfate.

6.3. Miscellaneous.

6.3.1. Distilled/deionized water.

6.3.2. Ultra high purity gas as required for GC analysis

7. Preparation of Standard Solutions.

7.1. Stock Solutions.

7.1.1. The stock standard solutions are prepared by dissolving 0.050 grams of each potential analyte in 50 mL of acetone. Larger volumes may be used at the discretion of the analyst. If the compound purity is certified at 96 percent or greater, the analyte weight can be used without correction to calculate the concentration of the stock solution.

7.1.2. These stock standards should be transferred to Teflon-sealed screw-cap bottles, stored at approximately 40 °F, and protected from the light by using amber colored containers. Due to the nature of the analytes and the acetone solvent, the stock standards should be monitored frequently for signs of degradation and/or evaporation, especially prior to preparing any intermediate standards.

7.1.3. Stock standards should be replaced after 12 months or sooner if a problem is indicated.

7.2. Intermediate Standards.

7.2.1. Intermediate standard solutions should be prepared using known volumes of the stock standard and diluting the solution with toluene. The intermediate standards should contain 100 µg/mL of each of the target analytes.

7.2.2. Storage and preservation of the intermediate standard solutions are the same as those for the stock solutions.

7.2.3. Intermediate standard solutions should be replaced after 8 weeks or sooner if a problem is indicated.

7.3. External Calibration Standards.

7.3.1. Calibration standards should be prepared at a minimum of three concentration levels but preferably at five concentration levels by adding volumes of one or more of the stock solutions to a volumetric flask and diluting to volume with toluene. One of these concentrations should be at a concentration near but above the method detection limit, and the other concentrations should correspond to the expected range of concentrations found in the field samples.

7.3.2. These solutions should also be preserved according to the general procedures in paragraph 7.1.2. and should be replaced after 15 days.

7.4. Working Standards. Fresh working range standards should be prepared on a daily basis by diluting known volumes of the intermediate standard solutions in toluene. These standards can then be utilized as performance standards for the GC instrument.

7.5. Field Surrogate Spike Solution. A acetone/acetonitrile solution containing one or more surrogate compounds should be prepared at a concentration to be determined by the project engineer (see Section 6 of the QA/QC plan, paragraph 6.2.5) and coordinated with the analyst.

8. Instrument Calibration.

8.1. Gas chromatographic operating parameters should be set at those listed in the compound specific sections of this section so that the retention times are equivalent. The instrument will be calibrated using an external standard calibration technique on a daily basis.

8.2. For the target compounds, calibration standards will be prepared utilizing the procedure in paragraph 7.3 of Appendix B. Each calibration standard should be injected into the GC system using the technique that will be used to introduce the actual samples into the instrument [method (syringe, autoinjector) and volume]. Peak area and corresponding sample concentration/mass should be tabulated. These results will be utilized to prepare a calibration curve for each of the target compounds (POHC's and

surrogates) in terms of detector response versus analyte concentration. In addition to calculating the slope and intercept of the line, the correlation coefficient should be calculated to assess linearity. These calibration curves should be verified throughout the daily sample groups by the analysis of a calibration standard solution. A drift in response for any compound which exceeds 20-relative percent difference will initiate a recalibration of the instrument for that particular compound. For the GC/MS instrument, standards and procedures specified by the manufacturer should be used to tune and calibrate the instrument. A single standard solution should then be analyzed by the GC/MS instrument to determine the response factor for the target compounds, to include the internal standard (anthracene-d_{10}).

9. Retention Time Window Determination.

9.1. Prior to determining retention time windows, the GC instrument should be operated for approximately 48 hours to allow for chromatographic system stabilization.

9.2. Three to five injections of the mixture of target compounds should be analyzed. From this analysis, the mean and standard deviation of the retention times for each compound will be calculated and utilized in defining the retention time windows for the analysis. The retention time window will be defined as $\pm$ two standard deviations ($\pm 2\sigma$) of the mean retention time. For those cases where the standard deviation of the retention time is zero, a value of $\pm$ 0.05 minutes of the retention time will be used as the window. Retention time windows for each target compound must be calculated for each chromatographic column to be utilized. The analyst may find it necessary to modify these windows depending upon the quantity and nature of other detectable compounds in the field samples (i.e., matrix effects). The calibration standards that have been dispersed among the field samples can be utilized as performance checks with regards to retention time windows. If any of the compound peaks fall outside the retention time window established, the chromatographic system should be considered out of control and corrective action should be initiated.

10. Sample Analysis.

10.1. An aliquot of the extracts from all sections of the sampling train will be placed in autosampler vials. The volume of the extract should be known and recorded prior to removal of the aliquot of the sample.

10.2. Prior to analyzing the group of samples for that day, the instrument calibration should have been performed and the retention time windows confirmed. This data should be recorded in the project data file on a daily basis. Each sample is analyzed in triplicate with one of the multilevel standards and a solvent blank placed after every sixth sample. The group of samples should also be bracketed with calibration standards.

10.3. Since linearity has been demonstrated for energetic compound chromatographic response (refer to Appendix A), chromatograms should be manipulated so that all peaks are on-scale. Integration methodologies should be compared with the chromatogram to ensure the methodology is appropriate for the peak shape. If all chromatographic peaks can not be brought on-scale electronically, the samples should be diluted and reanalyzed until all peaks are on-scale. By bringing all peaks on-scale, chromatographic resolution in the vicinity of the peaks corresponding to the target compounds can be examined. If peak resolution is acceptable, the undiluted sample may be used for quantitation.

10.4. Analytical results are obtained by use of the calibration plot prepared for each of the compounds. The integrator has been programmed to report the results in µg/mL.

10.5. No second gas chromatographic column will be utilized for confirmational analysis. Depending upon the compound and the equipment availability, the analyst at his discretion may perform secondary analysis using another detector type. However, 10 percent of the samples positive for either the POHC or the surrogates will be analyzed by GC/MS for confirmation of the identification provided by GC/ECD or GC/NPD analysis. If the concentration of the target compound is sufficient for GC/MS instrument semiquantitative analysis, then the GC/MS should be utilized for confirmation of the quantitation as well as the identification.

10.6. The chromatographic conditions for the analysis of compounds are contained in the sections following this appendix. For those analytical methods which utilize either NP or EC detectors, the selection of which detectors would depend on the type of interferences present in the samples. The selection would also depend on whether NG would also be a target analyte. Calculation methodology is covered in Appendix D.

SECTION B-1

ANALYSIS OF SAMPLES/STANDARDS FOR 2,4,5- AND 2,4,6-TNT

1. Applicability. This methodology is for the analysis of samples for 2,4,6-TNT as the only POHC of interest. The isomer 2,4,5-TNT has been spiked onto the sampling train as a recovery surrogate.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 170 °C
Detector Temperature: 200 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 125 °C for 5 minutes; ramp 2 °C per minute to 200 °C; hold 200 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 240 °C

3. Chromatograms. Example chromatograms are illustrated in Figures B-1.1 and B-1.2 for a standard solution and a field sample containing the TNT isomers.

MULTIPLIER = 20

Ihp3 5880A SAMPLER INJECTION @ 19:09 SEP 14, 1984
SAMPLE # : ID CODE :
5 40.8 UG STD
ANALYSIS OF RESIN EXTRACT FOR TRINITROTOLUENE USING ECD
ESTD

RT	AREA	TYPE	CAL	AMOUNT	NAME
2.36	76854.70	PV	1	10.401	UG 2,6-DNT
2.82	133659.00	VV	2	10.201	UG 2,5-DNT
3.23	50730.10	VV	3	10.608	UG 2,4-DNT
4.70	103683.00	VB	4	10.715	UG 3,4-DNT
8.05	310396.00	BB	5	40.810	UG 2,4,6-TNT

FIGURE B-1.1: STANDARD - 2,4,6-TNT

MULTIPLIER = 20

EXP3 5880M SAMPLER INJECTION @ 00:56 SEP 15, 1984
SAMPLE # : ID CODE :
12 10-33-1
ANALYSIS OF RESIN EXTRACT FOR TRINITROTOLUENE USING ECD
ESTD

RT	AREA	TYPE	CAL	AMOUNT	NAME
2.33	8464.17	HH	1	1.014	UG 2,6-DNT
2.61	12592.30	HH	2	0.858	UG 2,5-DNT
2.99	12114.00	HH	3	2.006	UG 2,4-DNT
4.76	1990.98	HH	4	0.203	UG 3,4-DNT
8.04	483054.00	HH	5	32.705	UG 2,4,6-TNT

FIGURE B-1.2: SAMPLE - 2,4,6-TNT

SECTION B-2

ANALYSIS OF SAMPLES/STANDARDS FOR 2,4-, 2,6-, and 3,4-DNT

1. Applicability. This methodology is for the analysis of samples for 2,4- and 2,6-DNT as the POHC's of interest. The isomer 3,4-DNT has been spiked onto the sampling train as a recovery surrogate.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 170 °C
Detector Temperature: 200 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 90 °C for 5 minutes; ramp 2 °C per minute to 200 °C; hold 200 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 240 °C

3. Chromatograms. Example chromatograms are illustrated in Figures B-2.1 and B-2.2 for a standard solution and a field sample containing the DNT isomers.

MULTIPLIER = 20

START AUTO SEQ 9, 9

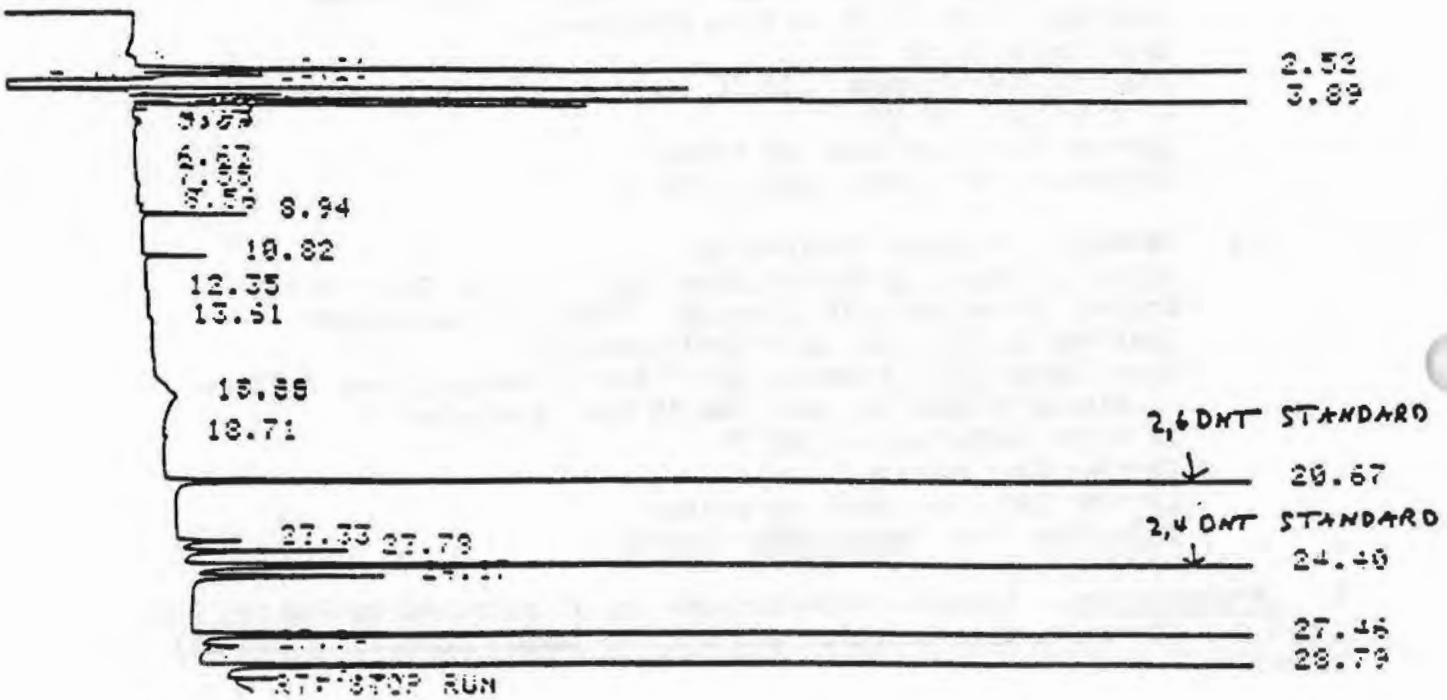


FIGURE B-2.1: STANDARD - 2,4- AND 2,6-DNT

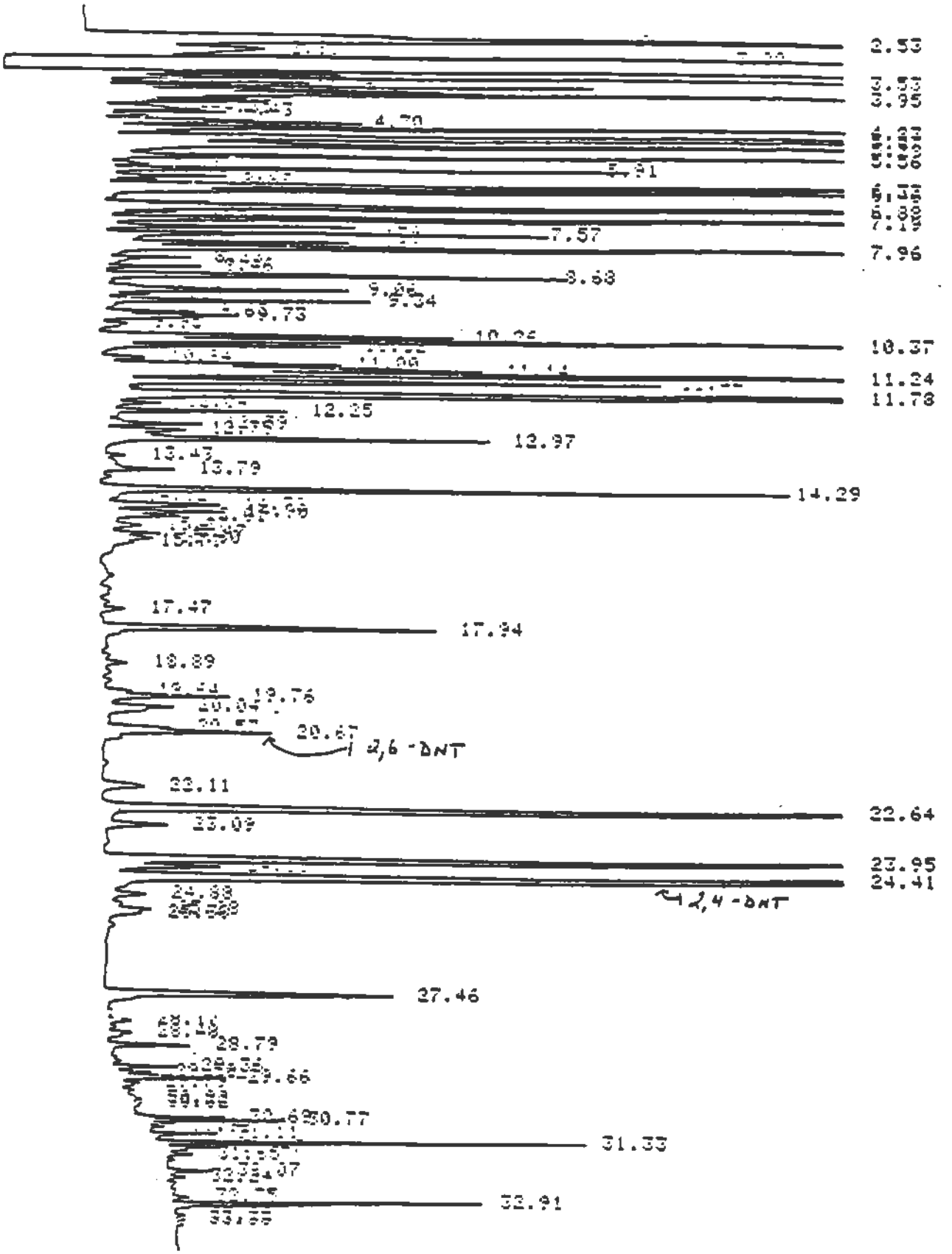


FIGURE B-2.2: SAMPLE - 2,4- AND 2,6-DNT

SECTION B-3

ANALYSIS OF SAMPLES/STANDARDS FOR NITROGLYCERIN

1. Applicability. This methodology is for the analysis of samples for NG as the only POHC of interest. The compound EGDN has been spiked onto the sampling train as a recovery surrogate.

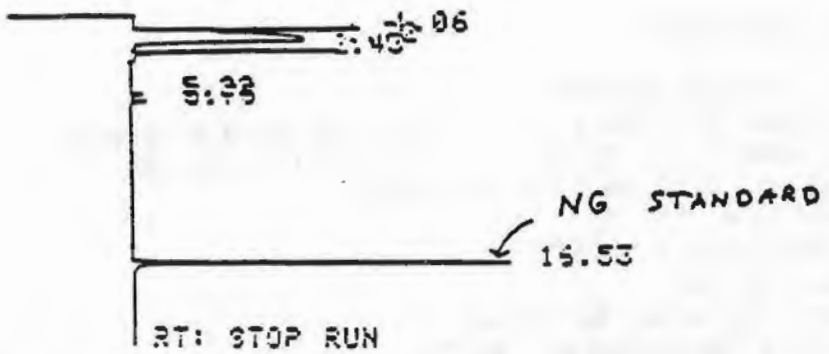
2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless [illegible]ent time of 0.5 minutes
Column: 30 meters x 0.32 mm [illegible]sed silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 120 °C
Detector Temperature: 140 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 40 cc/sec
Injection Port Temperature: 140 °C

2.2. Detector: Nitrogen/Phosphorus
Nitrogen/phosphorus detector may not be utilized for NG analysis.

3. Chromatograms. Example chromatograms are illustrated in Figures B-3.1 and B-3.2 for a standard solution and a field sample containing nitroglycerin and ethylene glycol dinitrate.

START AUTO SEQ 26, 26



[hp] 5880A SAMPLER INJECTION @ 08:00 DEC 17, 1984
SAMPLE # : ID CODE :
26
METHOD ABORTED
AREA %

RT	AREA	TYPE	AREA %
1.06	28605.80	BV	730.921
1.72	80385.30	VP	2053.970
2.43	10973.30	PV	280.385
5.22	874.16	BP	22.336
5.75	849.33	VP	21.702
16.53	34859.50	BB	890.688

FIGURE B-3.1: STANDARD - NG

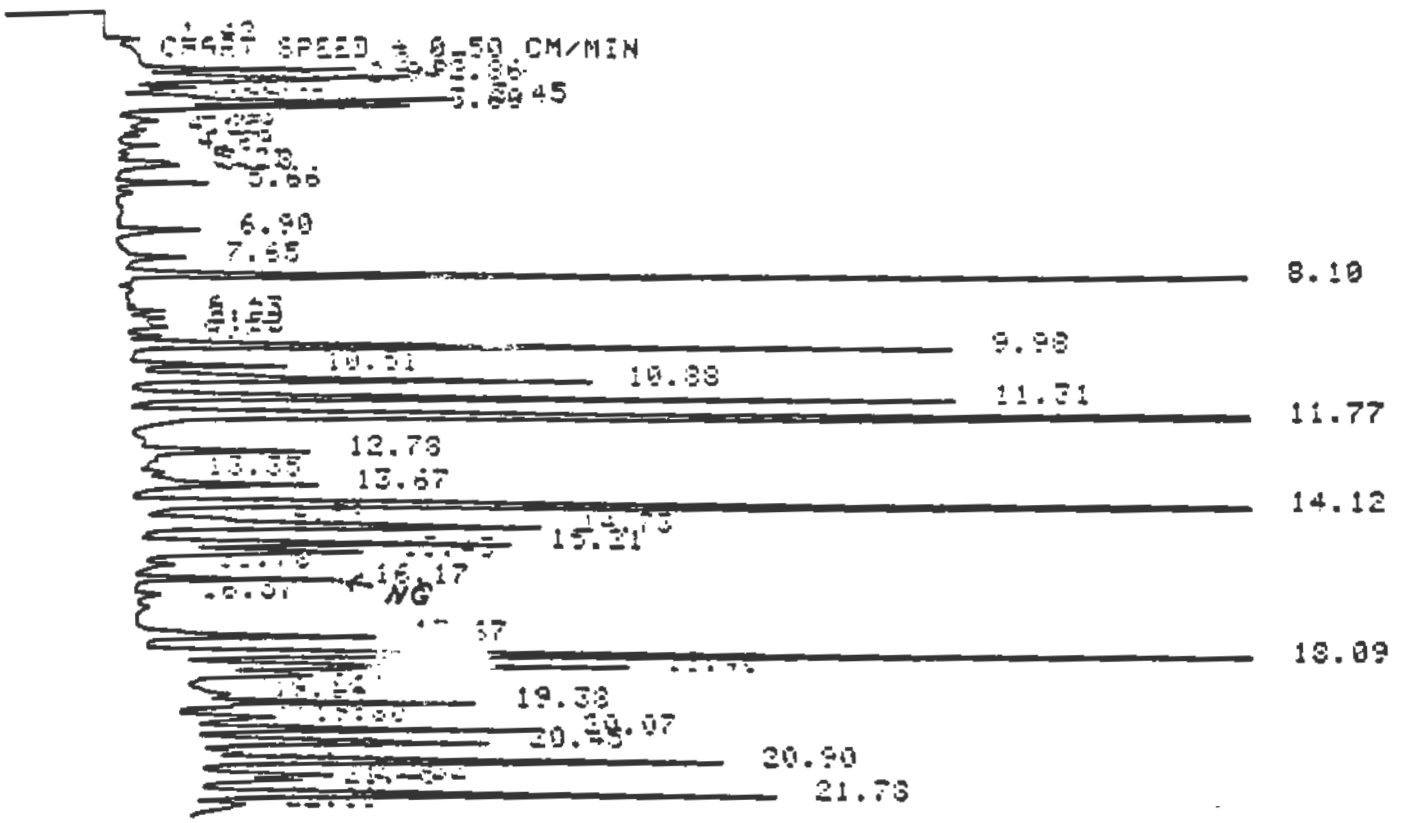


FIGURE B-3.2: SAMPLE - NG

SECTION B-4

ANALYSIS OF SAMPLES/STANDARDS FOR RDX

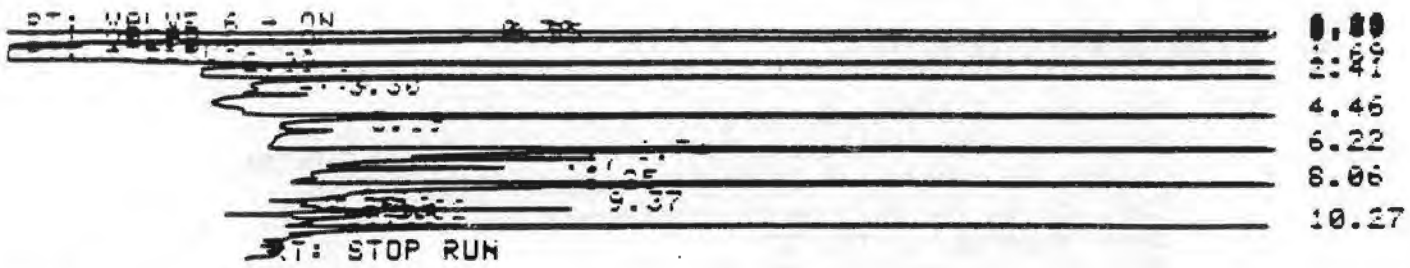
1. Applicability. This methodology is for the analysis of samples for RDX as the only POHC of interest.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica Column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 170 °C
Detector Temperature: 200 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica Column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 125 °C for 5 minutes; ramp 2 °C per minute to 200 °C; hold 200 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 240 °C

3. Chromatograms. Example chromatograms are illustrated in Figure B-4.1 for a standard solution containing RDX.



[hp] 5880A SAMPLER INJECTION @ 19:35 NOV 9, 1987
SAMPLE # : ID CODE :
8 20PPB-STD
H20 FOR EXPLOSIVES
ESTD COMPENSATED ANALYSIS

RT	AREA	TYPE	CAL	AMOUNT	NAME
1.69	116070.00	BB	1	21.496	2, 6-DNT
2.41	69532.80	BB	2	21.438	2, 4-DNT
4.46	115873.00	BB	3	21.249	2, 4, 6-TNT
6.22	103789.00	BB	4	23.248	RDX
8.06	35432.20	BB	5	22.146	TETRYL
10.27	16750.30	BB	6	30.567	HMX

MULTIPLIER = 1

FIGURE B-4.1: STANDARD SOLUTION CONTAINING RDX

SECTION B-5

ANALYSIS OF SAMPLES/STANDARDS FOR RDX AND DNT/TNT ISOMERS

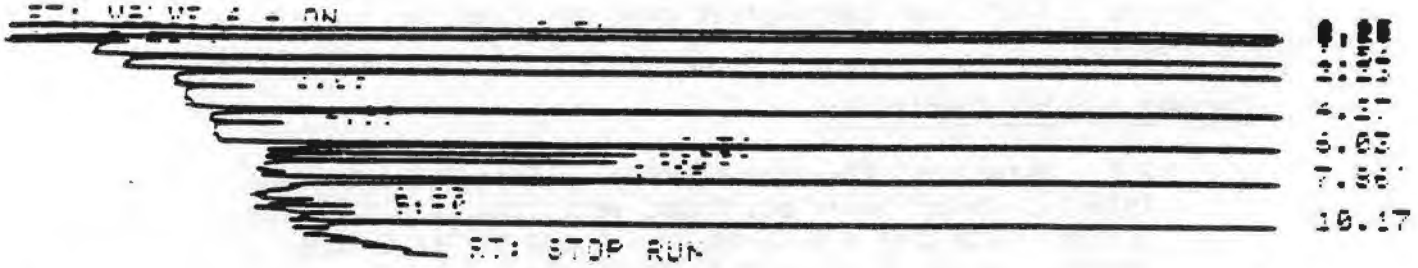
1. Applicability. This methodology is for the analysis of samples for 2,4,6-TNT, 2,4-DNT, 2,6-DNT and RDX as the POHC's of interest. The isomers 2,4,5-TNT and 3,4-DNT have been spiked onto the sampling train as a recovery surrogate.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture (Thin Phase Column)
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 10 meters x 0.32 mm ID - fused silica Column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 100 °C for 1 minute; ramp 25 °C per minute up to 225 °C; hold 225 °C for 2 minutes
Detector Temperature: 275 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 1 mL/min
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus (Wide Bore Column)
Injection Mode: Direct on-column; 2 µL
Column: 20 meters x 0.53 mm ID -fused silica Column
Coating: DB-5, 1.50 µm film thickness
Oven Temperature Program: 100 °C for 3 minutes; ramp 10 °C per minute to 225 °C; hold 225 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 5 mL/min
Carrier Make-up Flow Rate: 25 mL/min
Injection Port Temperature: 130 °C

3. Chromatograms. Example chromatograms are illustrated in Figures B-5.1 for a standard solution containing the target compounds.




```
[n:] 59604  SAMPLER INJECTION @ 15:01  NOV 10, 1987
  SAMPLE # :  ID TEXT  :
           6    20PPB-STD
                  -30   FOR   EXPLOSIVES
ESTD COMPENSATED ANALYSIS

  RT          AREA    TYPE CAL    AMOUNT  NAME

  1.54       88804.50   BB    1   20.034  3, 5-DNT
  2.13       53145.10   BB    2   19.989  2, 4-DNT
  4.27       89397.50   BB    3   19.778  2, 4, 6-TNT
  6.03       87452.50   BB    4   19.851  RDX
  7.66       39214.10   BB    5   19.938  TETRYL
 10.17       13042.50   BB    6   25.129  HMX

MULTIPLIER = 1
```

FIGURE B-5.1: STANDARD SOLUTION

SECTION B-6

CONFIRMATIONAL ANALYSIS BY GC/MS

1. Applicability. This me[illegible]s for the confirmational analysis performed on the samples in [illegible]ovide confirmation of the quantitation obtained by GC/ECD [illegible]n if possible. Otherwise, the identification of the target compo[illegible]onfirmed.

2. Chromatographic Conditions.

Injection Mode: Splitless for 1.2 minutes Injection
Volume: 2 - 3 µL
Column: 30 meters x 0.32 mm ID - fused silica Column
Coating: DB-5 (permanent bonded SE54), 0.25 µm film thickness
Oven Temperature Program: 150 °C for 1 minute; ramp 8 °C per minute up to 285 °C; hold 285 °C for 20 minutes
Carrier Gas: Helium
Carrier Gas Flow Rate: 10 psi at inlet
Injection Port Temperature: 150 °C
Internal Standard: Anthracene - D_{10}

3. Mass Spectrometric Conditions.

Interface: Direct (fused silica column inserted directly into source chamber)
Ionization Mode: Electron Impact
Electron Energy: 70 eV
Scan Rate: 1 scan per second
Scan Mass Range: 35-350

4. Chromatograms/Spectra. Reconstructed ion chromatograms and mass spectra of the analytes are shown in Figures B-6.1 through B-6.6.

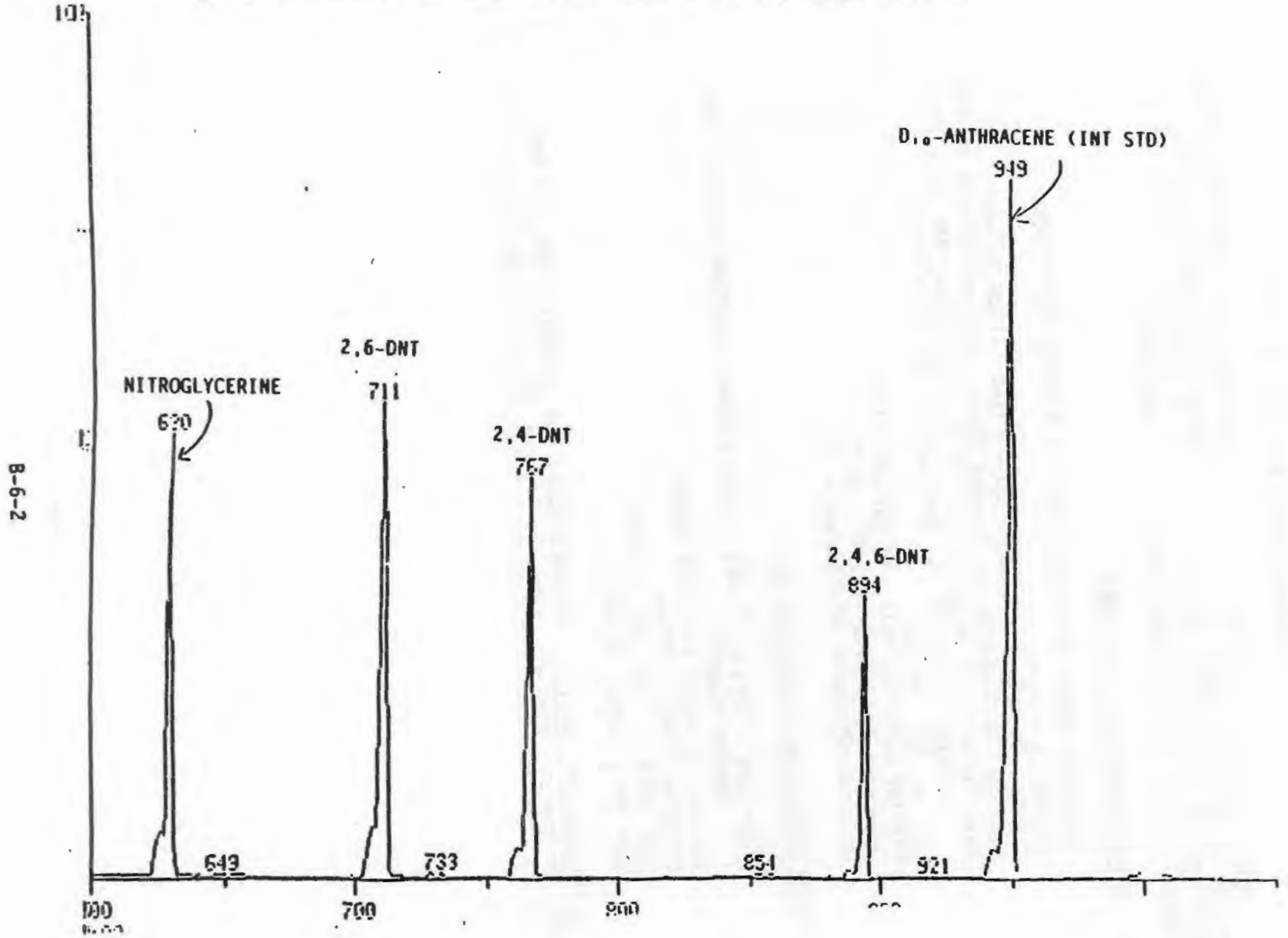
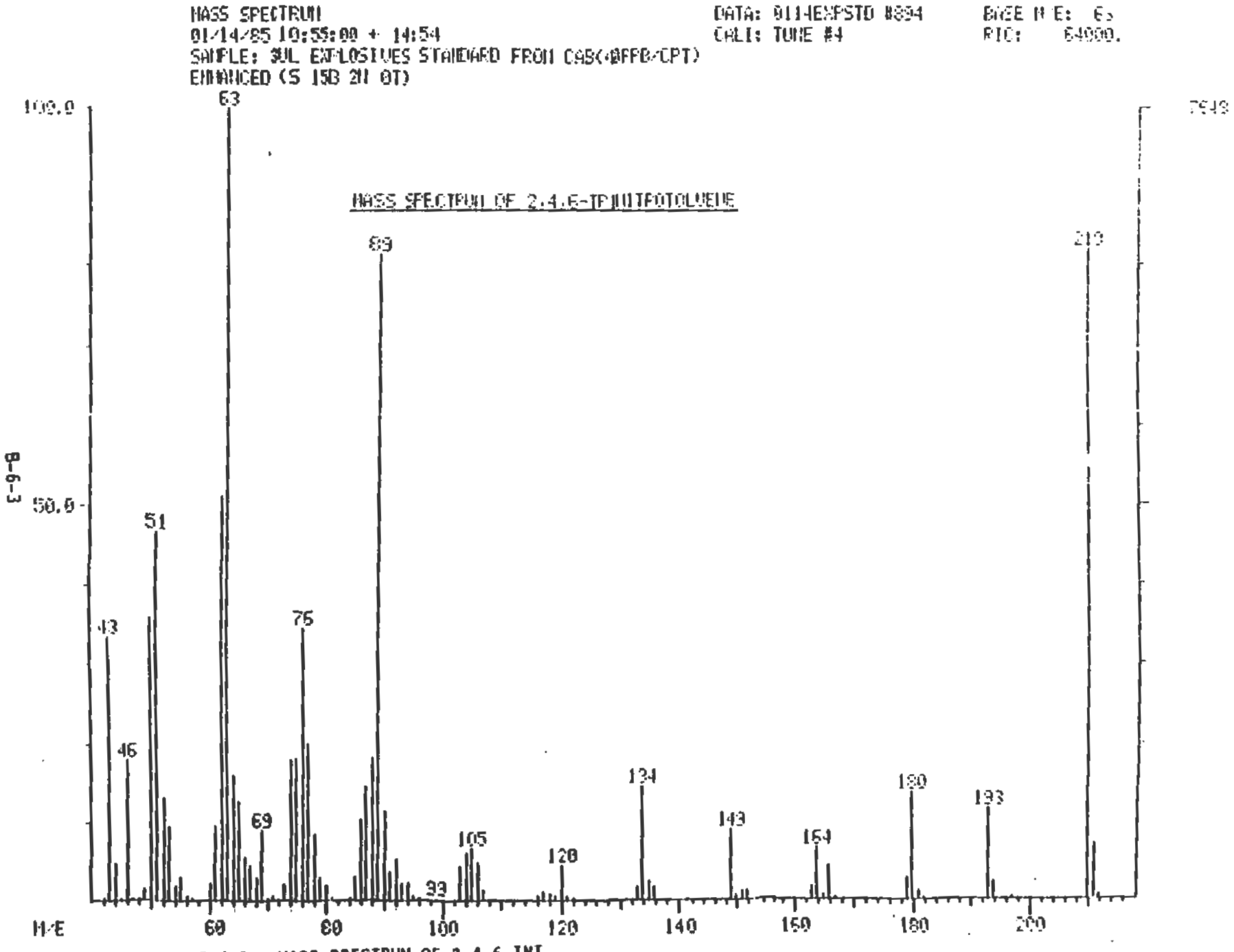


FIGURE B-6.1: GC/MS ANALYSIS OF EXPLOSIVE COMPOUNDS STANDAR.



B-6-3

FIGURE B-6.2: MASS SPECTRUM OF 2,4,6-TNT

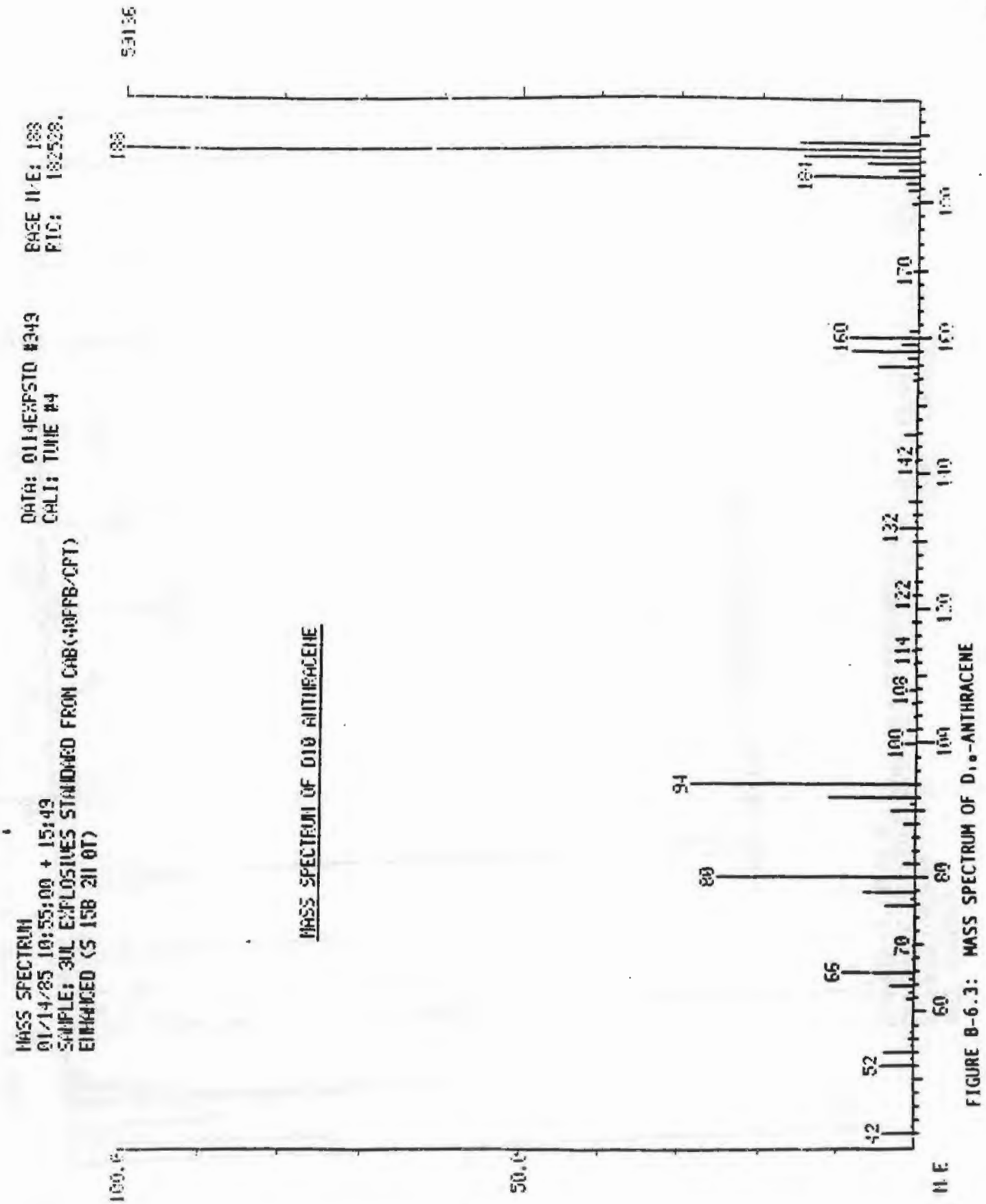


FIGURE 8-6.3: MASS SPECTRUM OF D_{10}-ANTHRACENE

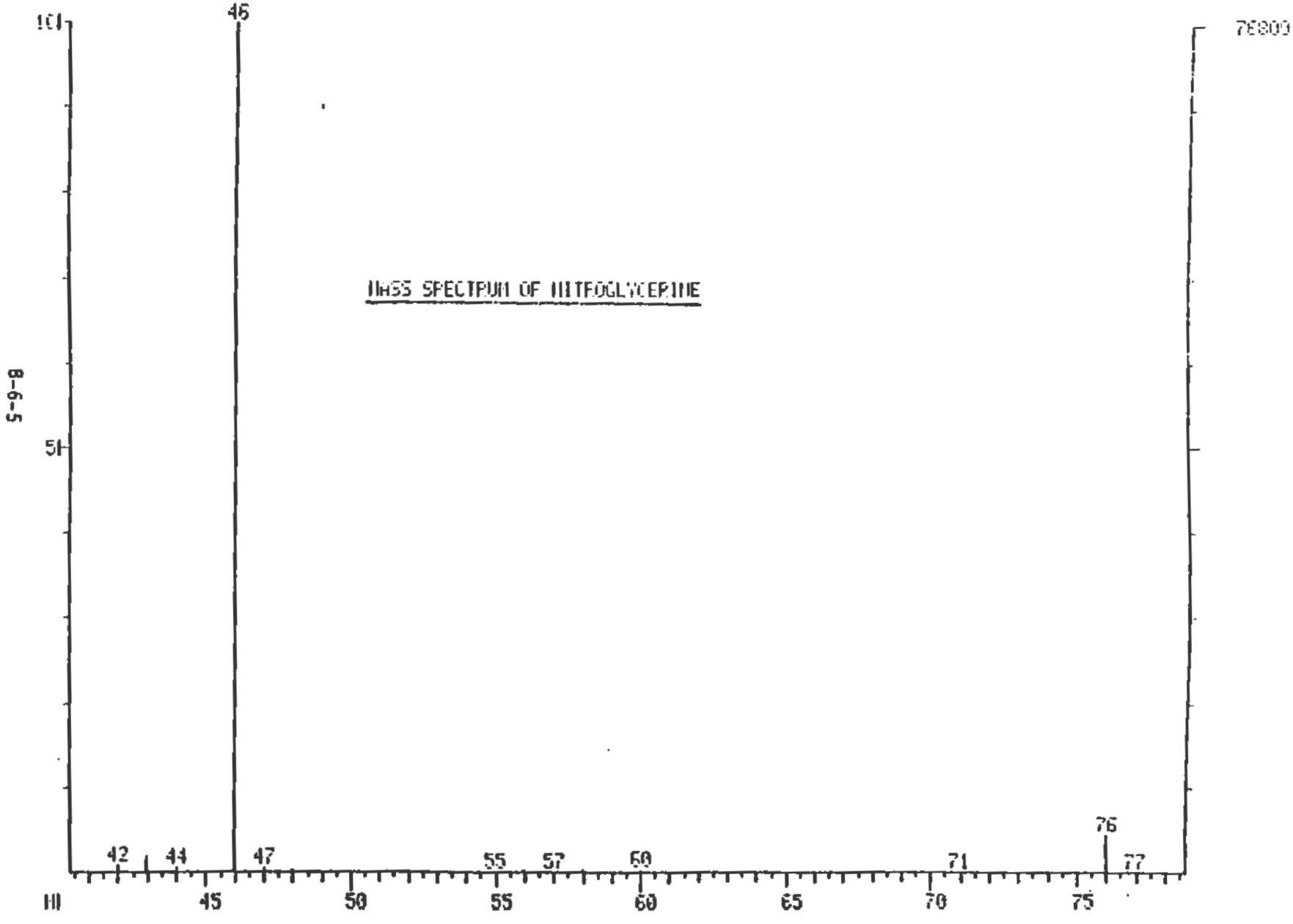


FIGURE B-6.4: MASS SPECTRUM OF NG

MASS SPECTRUM
01/14/85 10:55:00 + 11:51
SAMPLE: 3UL EXPLOSIVES STANDARD FROM CAB(40PPB/CPT)
ENHANCED (S 15B 2N 0T)

DATA: 0114EXPSTD #711
CAL: TUNE #4

BASE M/E: 63
RIC: 117760.

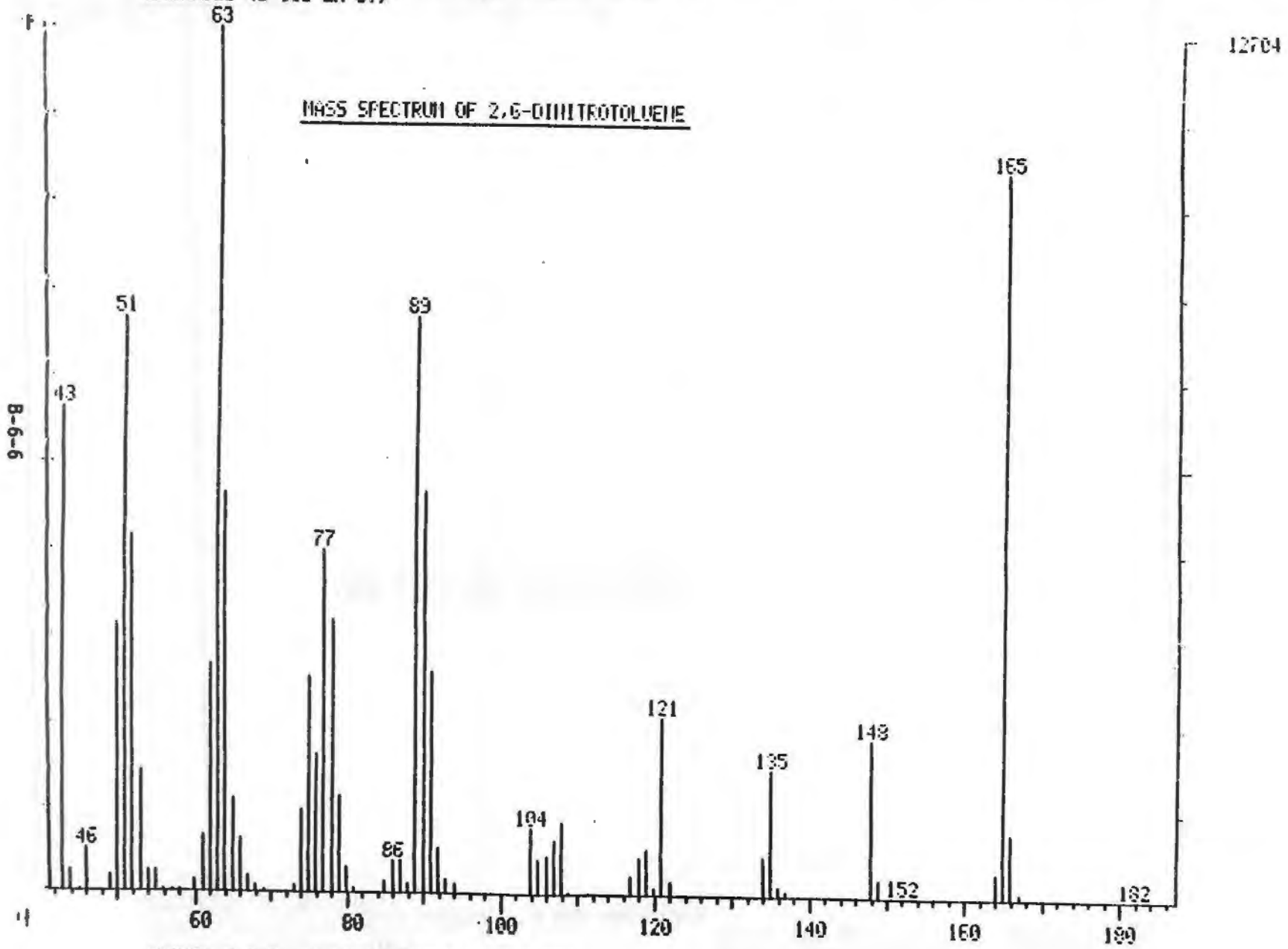


FIGURE 8-6.5: MASS SPECTRUM OF 2,6-DNT

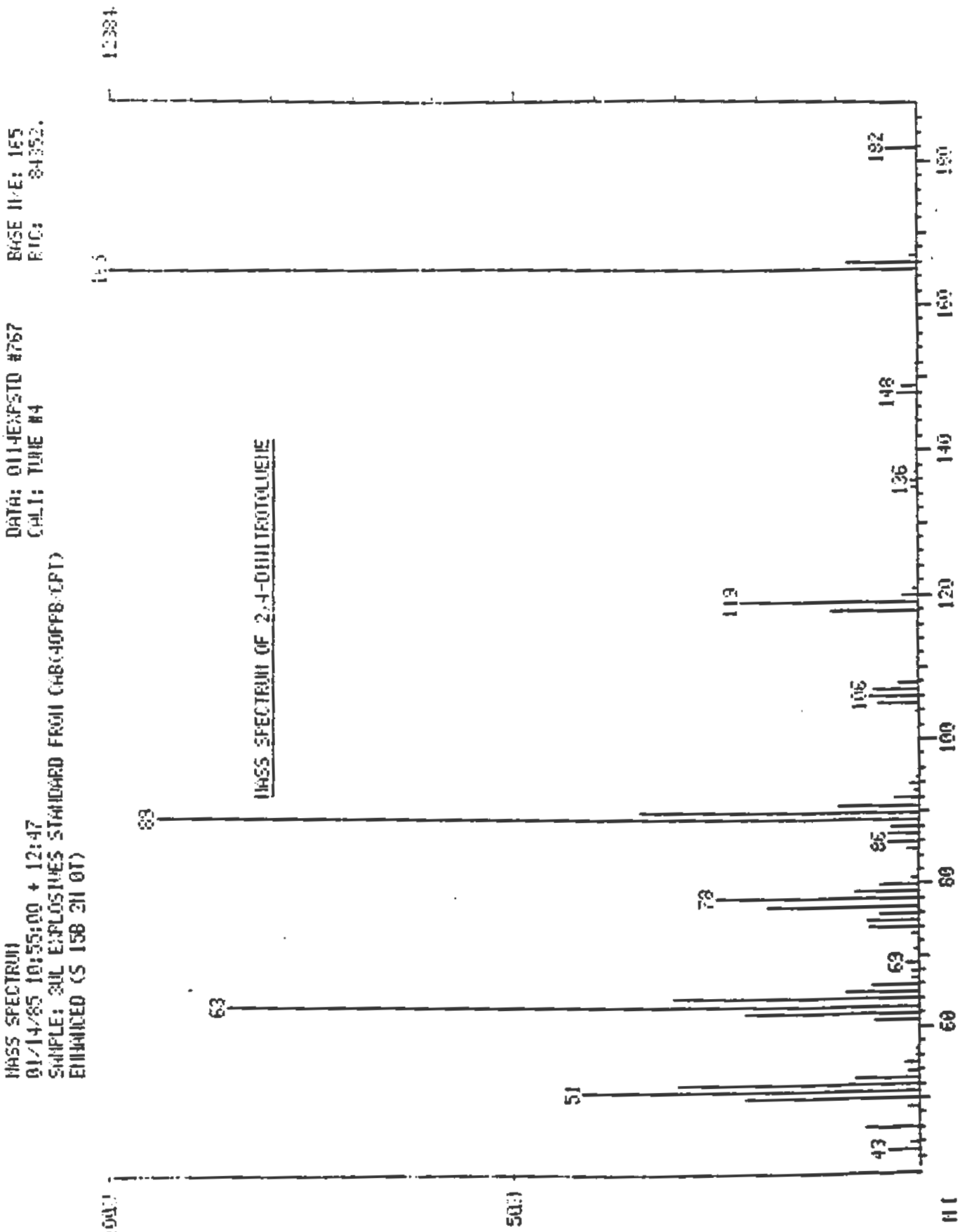


FIGURE B-6.6: MASS SPECTRUM OF 2,4-DNT

SEARCH AND QUANTITATION MASS CONDITIONS

Compound	Scan Range	Search Masses	Quantitation Mass
2,6-DNT	690-740	63;89;165	165
2,4-DNT	740-790	63;89;165	165
3,4-DNT	790-840	63;89;182	182
NG	600-650	46;76	46
RDX	980-1030	46;75;120	46
2,4,6-TNT	870-920	63;89;210	210
2,4,5-TNT	930-980	63;118;210	210
Anthracene-d10 (internal standard)	930-970	188	188

APPENDIX G

QUALITY ASSURANCE AND QUALITY CONTROL PLAN

QUALITY ASSURANCE AND QUALITY CONTROL PLAN

DEACTIVATION FURNACE TRIAL BURN

SENECA ARMY DEPOT
ROMULUS, NEW YORK

Prepared for: U.S. Army
Corps of Engineers
Huntsville, Alabama

Prepared by: Engineering-Science, Inc.
Fairfax, Virginia

November 1992
FB505

QUALITY ASSURANCE AND QUALITY CONTROL PLAN

DEACTIVATION FURNACE TRIAL BURN

Seneca Army Depot
Romulus, New York

Michael Duchesneau
Project Manager, Engineering-Science, Inc.

Trial Burn Plan
For The
Seneca Army Depot
Contents of Appendix G
Quality Assurance and Quality Control Plan

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SECTION 1

PROJECT DESCRIPTION

The objective of this project is to conduct a trial burn test of a deactivation furnace **(Figure G.1)** located at the Seneca Army Depot in Romulus, New York. The primary objective of the trial burn is to determine the fate of selected principal organic hazardous compounds (POHCs). Other important objectives are to determine the concentration of POHCs, selected metals, particulate matter, carbon monoxide (CO), oxygen (O_2) total hydrocarbon (THC), nitrogen oxides (NO_x), Dioxins and Furans in the stack gas. Waste feed characteristics (heat content, density, viscosity, ash content, halogen content, etc.) will be based on military specifications for the ammunition being deactivated. This data will be used together with furnace process data and source test data to determine if the furnace can comply with EPA regulations regarding the destruction of hazardous waste.

This project will involve evaluating the furnace as it operates under nine different test conditions. Each test condition represents a different "worst-case" feed scenario. For example, test condition 1 will be the worst case condition for the POHC, hexachloro benzene (HCB). The planned testing for this test condition will focus on the collection of data that will enable a determination to be made as to the ability of the furnace to satisfactorily destroy the POHC, HCB. The waste feed for each test condition will be a munition item or a bulk explosive. During test condition 1, HCB will be spiked into the waste stream. Feed rates will be monitored with an automatic waste feed system and waste characteristics will be based on military specifications for manufacture. (Sampling the waste feed stream is too dangerous.) Furnace stack gas during each test condition will be evaluated by using 40 CFR Part 60 Appendix A reference methods, SW-846 Sampling and Analysis methods, Boiler and Industrial Furnace Methods Manual methods and the Sampling Train for Energetic Materials (STEM) method from the Army Environmental Hygiene Agency. This data will be combined with munition feed data to calculate the destruction and removal efficiency of the furnace.

Table G-1 summarizes the overall test program and identifies the waste feed, feed rate, sampling method, analytical method, frequency of collection and the parameter to be measured.

FIGURE G.1

APE 1236 - ISOMETRIC VIEW

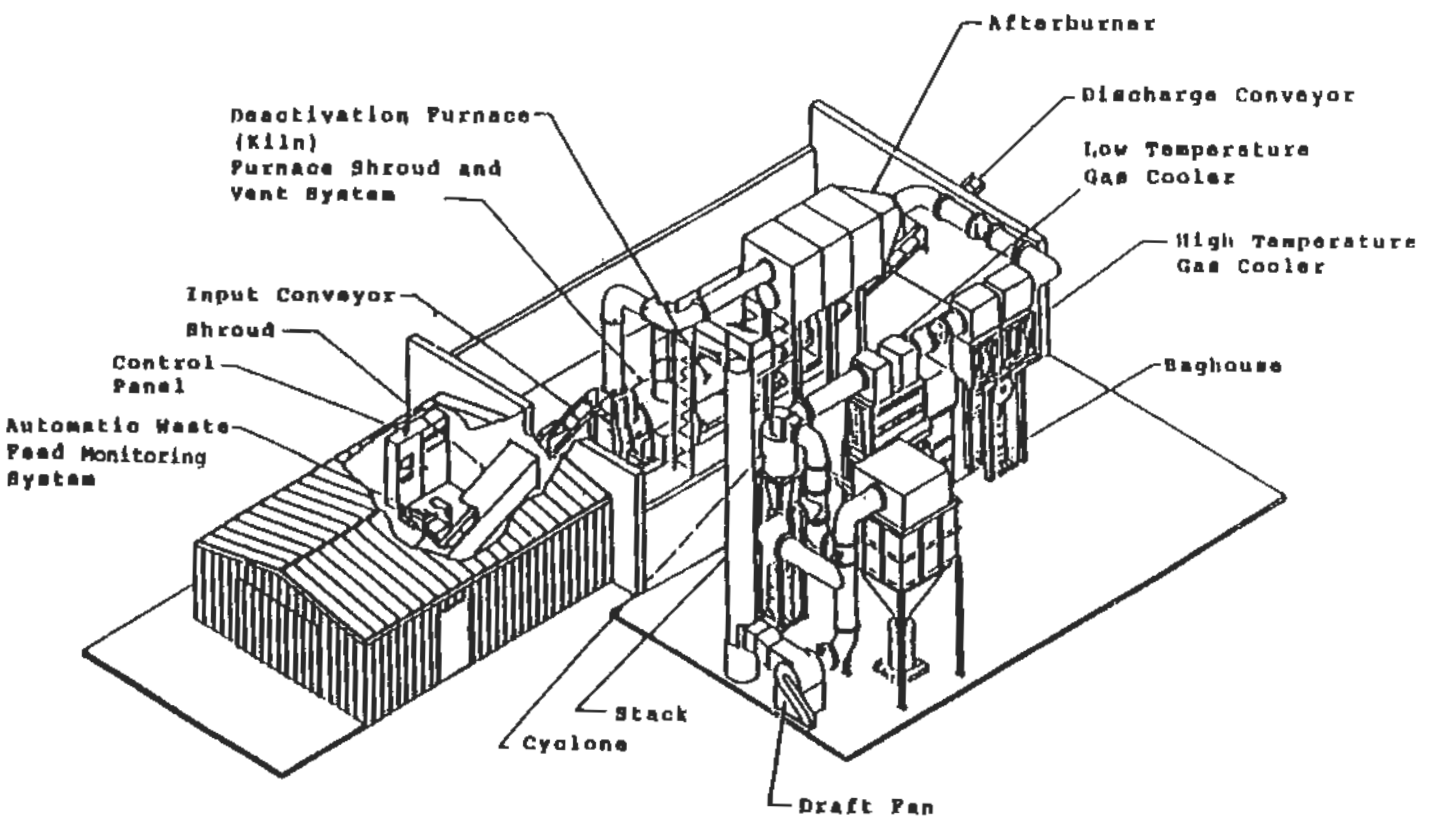


TABLE G-1

OVERALL TEST PROGRAM SUMMARY

Test	Munition Item No.	Item Feed Rate (Item/Hr)	Parameter	Parameter Feed Rate (Lb/HR)	Sampling Method	Analytical Method	Frequency of Collection
1	182		HCB		SW-846 Method 0010	SW-846 8120	3 Runs/Test
2	182		TCE		SW-846 Method 0030	SW-846 Method 8010	3 Runs/Test
3	143	19,146	NG	44.39	AEHA STEM	AEHA STEM	3 Runs/Test
4	49	8,000	DNT	26.86	AEHA STEM	AEHA STEM	3 Runs/Test
5	57	6,377	Particles Barium	98.17 22.21	EPA RM 5 BIF Metals Method	SW-846 8010	3 Runs/Test 3 Runs/Test
6	120	375,601	Antimony	5.02	BIF Metals Method	SW-846 8010	3 Runs/Test
7	127	3,163	Chromium	0.04	BIF Metals Method	SW-846 8010	3 Runs/Test
8	200	220	Lead	16.07	BIF Metals Method	SW-846 8010	3 Runs/Test
9	23	22,500	Dioxin/Furan Precursor Max Waste (DEP)	3.09 204	EPA RM 23	EPA M23	3 Runs/Test

During each test condition, DF performance data will also be gathered to evaluate CO, O_2, THC, and NO_x stack gas concentrations. Combustion chamber temperatures and pressures, combustion air flowrates, auxiliary fuel feed rates and other significant process data will be gathered during each test condition. **Table G-2** summarizes the process data collection program; it outlines the parameters being monitored and establishes the frequency for this monitoring.

TABLE G-2

OVERALL PROCESS MONITORING SUMMARY

Parameter	Monitoring Frequency
Kiln Outlet Temperature (°F)	Continuous
Afterburner Outlet Temperature (°F)	Continuous
Stack Gas Velocity (fps)	Continuous
Kiln Pressure (in H_2O)	Continuous
Kiln Rotation (rpm)	Once per Test
PEP Waste Feedrate (lb/hr)	Continuous
Waste Feedrate (items/hr)	Continual Counting
Baghouse Pressure Drop (in H_2O)	Continuous
Cyclone Pressure Drop (in H_2O)	Continuous
Baghouse Outlet Temperature (°F)	Continuous
CO Level (ppm)	Continuous
O_2 Level (%)	Continuous
Fuel Usage (gph)	Continuous
HTHE[1] Exit Temperature (°F)	Continuous
LTHE[2] Exit Temperature (°F)	Continuous

[1] High Temperature Heat Exchanger
[2] Low Temperature Heat Exchanger

SECTION 2
PROJECT ORGANIZATION AND RESPONSIBILITY

The project organizational chart shown in Figure G.2 illustrates the relationships among management, staffing, and quality assurance for the state agency, SEAD, and Engineering-Science (ES).

2.1 PROGRAM MANAGEMENT

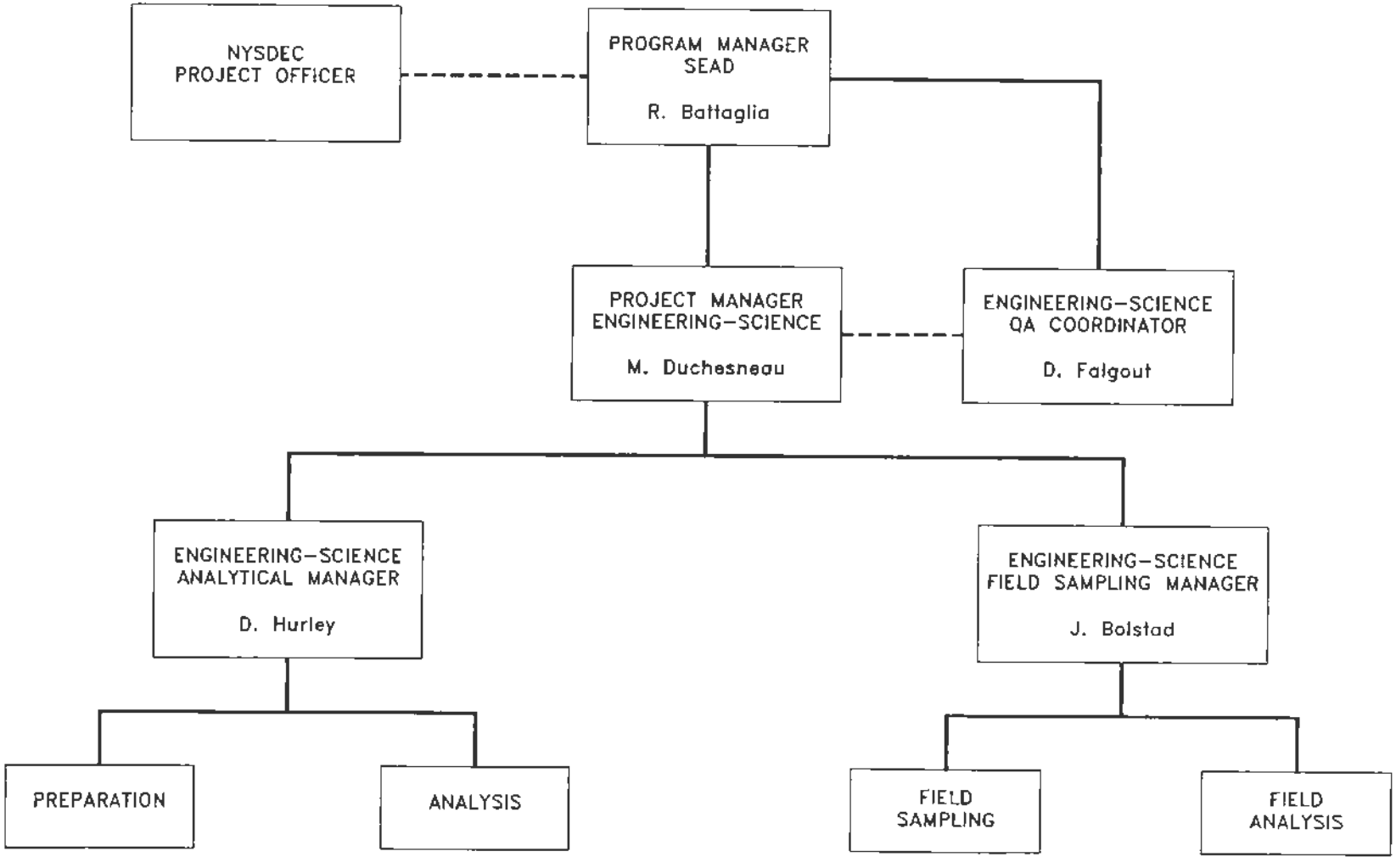
The facility-designated signatory and program manager at SEAD is Mr. Randy Battaglia. The ES project manager is Mr. Michael Duchesneau. Mr. Duchesneau will ensure that the sampling/analysis teams meet their assigned project scope, scheduling and cost requirements. The ES project manager (PM) will report to the SEAD program manager. The ES PM will coordinate the daily activities of the field sampling and the analytical teams, ensuring that they understand their respective roles, and that they deliver all samples or results according to project requirements. The ES PM will provide a direct line of communication to the State Agency's project officer and will ensure that all data is reported to the agency in an acceptable format and will work closely with the ES Quality Assurance Coordinator (QAC) to maintain the data quality of this QA plan. The ES QAC for this project is Dr. Dennis Falgout. The responsibilities of the QAC are detailed in the following sub-section.

Reporting to the ES PM will be the ES field sampling manager (Mr. Jon Bolstad) and the ES analytical manager (Ms. Donna Hurley). Mr. Bolstad's responsibility will be to manage the field sampling activities and to ensure that the necessary sampling and monitoring activities have been carried out correctly and in conformance with the Trial Burn Plan and this QA/QC plan. Ms. Hurley will be responsihle for supervising and coordinating recovery and analytical activities in the field and in the laboratory. Resumes of the ES personnel are provided in **Attachment G-1**.

2.2 RESPONSIBILITY AND AUTHORITY OF THE QA COORDINATOR

The QAC will coordinate or conduct field and laboratory audits to ensure that the quality assurance/quality control activities that are presented in this QA/QC Plan are implemented. Independent of all personnel involved in the technical program, the QAC will submit audit reports to the program manager. Specific duties include:

FIGURE G.2
QA/QC ORGANIZATION CHART



- Assist in the development and review of the trial burn protocol and test plan;
- Monitor the quality of data collected;
- Report QA results and information and suggest/document corrective actions when required;
- Maintain current version of all measurement procedures to be used; and
- A written independent assessment of overall data quality to be submitted with the trial burn report.

The QAC has the authority to stop or change sampling and analytical activities, if necessary, to ensure data quality goals. Dr. Falgout is the ES Eastern Region Quality Assurance Officer. In this position, Dr. Falgout regularly provides independent reviews of ES projects for conformance to established QA/QC criteria.

2.3 RESPONSIBILITY AND AUTHORITY OF SAMPLING AND ANALYSIS GROUPS

Sampling and field analysis activities will be performed by ES personnel under the direction of the field team leader. Field sampling, process monitoring, and quality control functions, will be conducted under the field team leader's direction.

The analytical manager will supervise the sample recovery and field extractions during this project. This person will also be responsible for the general coordination of laboratory activities with the field sampling team and with the QAC and project manager.

Laboratory sample recovery and analytical work will be supervised by the analytical laboratory manager. The analytical laboratory will have a designated project coordinator who is responsible for preparation of sorbents and other collection media, and for the coordination of source test sample preparation and analysis.

SECTION 3
QUALITY ASSURANCE AND QUALITY CONTROL OBJECTIVES

The precision objectives for measurements are shown in **Table G-3**. These are based on the relative percent difference of duplicate analyses, or split samples. Accuracy objectives shown in **Table G-4** are based on the percent bias in the analyzed value of standards or spikes of known quantity and composition of the total number of samples collected and analyzed.

Representative sampling of stack gases will be achieved by using standard EPA procedures. Representative sampling of fly ash from the heat exchangers, cyclone, and baghouse will accomplished by collecting all the ash at a location at the end of each run and thoroughly mixing it prior to sampling. Laboratory analysis of the ash is expected to require 500 to 1,000 grams. A visual inspection of each ash hopper will determine if sufficient ash is present for collection. If there is not a sufficient amount of ash at the end of each run, then ash will be collected at the end of each test condition. During the miniburn, more information will be gathered regarding the accumulation rate of fly ash.

Completeness objectives for sampling and analysis of each parameter have been established as 100%. Completeness is defined as the amount of valid data collected for a measurement system compared to the planned amount of data to be collected during normal operating conditions. During the trial burn, each test condition requires three valid sample runs for the selected parameter(s) to meet the 100% completion objective.

During each sample run, field blanks, trip blanks and reagent blanks will be collected according to the method being performed. A field blank for the MM5-style sampling trains will consist of a complete train that is carried to the source test location and leak checked. After the leak check, the train is capped and then set aside for the duration of the sampling event. At the conclusion of the sampling event the field blank train is leak checked again and then recovered and submitted for analysis as if it were a sample.

TABLE G-3
QA OBJECTIVES FOR PRECISION AND COMPLETENESS OF EACH MAJOR MEASUREMENT

Measurement Parameter	No. of Samples	No. of Field Blanks[a]	No. of Trip Blanks[b]	Precision Goals	S&A Completeness Goals (%)
POHC (MM5)	6	4	4	NA	100
POHC (VOST)					
POHC (STEM)	3	1	1	NA	100
Particulate Matter (M5)	3	NA	1	NA	100
Metals (BIF Metals)	12	NA	4	NA	100
Dioxins and Furans (M23)	3	3	3	NA	100
Stack Gas Volumetric Flowrate (M2)	32	NA	NA	NA	100
CO_2, O_2 (M3)	32	NA	NA	0.2% O_2, CO_2 [c]	100
CO, O_2 (CEM)	Continuous	NA	NA	5%[d]	100[d]
Moisture (M4)	32	NA	NA	NA	100
NO_x (M7E)	Continuous	NA	NA	5%	100
THC (M25A)	Continuous	NA	NA	5%	100[e]

NA = Not applicable
[a] One field blank per day of source testing will be collected
[b] Reagent blanks, filter blanks, and sorbent trap blanks
[c] Absolute
[d] Based on high-range span gas at full-scale
[e] Completeness is based on 40 CFR Part 60, Appendix F requirements for continuous emission monitors.

TABLE G-4
QA OBJECTIVES FOR ACCURACY OF EACH MAJOR MEASUREMENT

Measurement Parameter	No. of Samples	No. of Spikes	Accuracy Goals Spikes % Recovery	No. of Reference Standards	Accuracy Goals Reference Standards
POHC (MM5)	6	6 surrogate 1 POHC	50-150% 50-150%	5 CAL 1 ICC 1 CCC	<30% RSD of RRF Avg. 70%-130% Theoretical ±30% of RRF
POHC (VOST)					
POHC (STEM)	3	1 blind 3 surrogate	70-130% 75-125%	5 CAL 1 CCC	CORR = 0.99 30% DIFF from CAL RF
Particulate Matter (M5)	3	NA	NA	NA	NA
Metals (BIF Metals)	12	at least 2	70-130%	2 to 5 CAL 2 CCC 1 ICC	CORR >0.995 90-110% (flame) 90-110%
Dioxins and Furans (M23)	3	3 surrogate	70-130%	5 CAL 1 CCC 1 Audit	M23 Section 6.1.1 M23 Section 6.1.2.1 M23 Section 8
Stack Gas Volumetric Flowrate (M2)	32	NA	NA	NA	NA
CO_2, O_2 (M3)	32	NA	NA	1	0.2 or 0.3%[a]
CO, O_2 (CEM)	Continuous	NA	NA	1	The greater of 10% of PTM or 10 ppm[b]
Moisture (M4)	32	NA	NA	NA	NA
NO_x (M7E)	Continuous	NA	NA	1	20%[c]
THC (M25A)	Continuous	NA	NA	1	20%[d]

NA = Not applicable
CAL = Calibration Standard
RSD = Relative Standard Deviation
CCC = Continuing Calibration Check
PTM = Performance Test Method
ICC = Initial Calibration Check
RRF = Relative Response Factor
CORR = Correlations Coefficient

[a] The accuracy goal is dependent on the stack gas concentrations of CO_2 and O_2 as per Method 3.
[b] This is a relative accuracy (RA) that applies to the CO monitor as described in the *Methods Manual for Compliance with BIF Regulations*. As per the BIF Methods Manual, there is not an RA for the O_2 analyzer. It is incorporated into the CO RA calculation.
[c] 40 CFR Part 60, Appendix F.
[d] 40 CFR Part 60, Appendix A, Method 25A.

SECTION 4
SAMPLING AND MONITORING PROCEDURES

The primary sampling site is in the exhaust from the APE1236. Sampling at this location will permit the calculation of the destruction and removal efficiency (DRE) of the POHCs in the waste fuel. Other sampling streams that are important are the fly ashes generated at the two heat exchangers, the cyclone and the bag house. Composition of the APE 1236 waste feed (munitions) will be determined from munition specification data.

This section describes the proposed sampling sites and sampling and analytical methods. The methods specified in this plan will be those described in 40 CFR Part 60 Appendix A; SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition; and the EPA Methods Manual for Compliance with the BIF Regulations. The exception to this will be the sampling and analytical methods used to sample energetic compounds in stack gas and fly ash. These methods have been established by the United States Army Environmental Hygiene Agency (AEHA) and are presented in Attachment B. Generally, the sampling and analysis methods that have been selected are EPA methods or EPA-approved methods. **Table G-5** summarizes the sampling and analysis plan.

4.1 SAMPLING PLAN

The sampling plan has been developed by following the approach described below:

I. Assemble available data
- process diagrams
- equipment drawings
- production data (schedule, frequency, rate, etc.)
- flow data
- existing sampling points
- chemical characterization data of stream to be sampled
- process variables to be measured routinely

TABLE G-5
TRIAL BURN TESTING SUMMARY

Analysis Parameters	Sampling Method	Collection Frequency	Test Series Number	Sample Preparation	Analytical Method
STACK GAS					
Particulate Matter	Reference Method 5	3 runs/test series	4	NA(1)	Reference Method 5
Stack Gas Volumetric Flowrate	Reference Method 2	3 runs/test series	all	NA	NA
Temperature	Reference Method 2	3 runs/test series	all	NA	NA
Moisture	Reference Method 4	3 runs/test series	all	NA	NA
CO_2, O_2	Reference Method 3 and CEMs	3 runs/test series and Continuous	all	NA	Reference Method 3 and CEMs
NO_x	Reference Method 7E	Continuous	all	NA	Reference Methods 7E
CO	CEM	Continuous	all	NA	NDIR
THC	Reference Method 25A	Continuous	all	NA	Reference Method 25A
POHCs (HCB) (TCE) (NG) (DNT)	 SW-846 Method 0010 SW-846 Method 0030 AEHA STEM Method (2) AEHA STEM Method	 3 runs/test series 3 runs/test series 3 runs/test series 3 runs/test series	 1 2 3 4	 SW-846 Method 0010 SW-846 Methods 5040 AEHA STEM Method AEHA STEM Method	 SW-846 Method 8120A SW-846 Method 5040(8240) AEHA STEM Method AEHA STEM Method
Barium	BIF Metals Method(3)	3 runs/test series	5	BIF Metals Method	SW-846 Method 6010A
Antimony	BIF Metals Method	3 runs/test series	6	BIF Metals Method	SW-846 Method 6010A
Chromium	BIF Metals Method	3 runs/test series	7	BIF Metals Method	SW-846 Method 6010A
Lead	BIF Metals Method	3 runs/test series	8	BIF Metals Method	SW-846 Method 6010A
Dioxins/Furans	Reference Method 23	3 runs/test series	9	Reference Method 23	Reference Method 23
FLY ASH					
HCB	Trowel Method (S007)	3 samples/series	1	SW-846 Method 3540	SW-846 Method 8120A
TCE	Trowel Method (S007)	3 samples/series	2	SW-846 Method 5030	SW-846 Method 8010
NG	Trowel Method (S007)	3 samples/series	3	SW-846 Method 3540	AEHA STEM Method
DNT	Trowel Method (S007)	3 samples/series	4	SW-846 Method 3540	AEHA STEM
13 RCRA Metals	Trowel Method (S007)	3 samples/series	All	SW-846 Method 1311 SW-846 Method 3050	SW-846 Method 6010A SW-84 Method 7471
Dioxins/Furans	Trowel Method (S007)	3 samples/series	9	SW-846 Method 8280	SW-846 Method 8280

(1) Not Applicable
(2) Army Environmental Hygiene Agency Sampling Train for Energetic Materials
(3) Boiler and Industrial Furnace Metals Method
(4) Arthur D. Little, "Sampling and Analysis Methods for Hazardous Waste Combustion" EPA-600/8-84-002, PB84-155845, February 1984

II. Conduct presampling site evaluation:

- complete data acquisition (flow rates, temperature, production schedule, verify normal operation, identify type and frequency of process upsets and or equipment malfunctions)
- locate all sample streams and sampling points
- identify ease of access to sampling locations (make or recommend modifications when necessary)
- determine appropriate protective sampling gear
- set-up means of obtaining relevant process data

III. Prepare site-specific sampling plan:

- prepare a detailed test plan based on information gathered in I and II (collection method for each type of sample, numbers of samples to be collected, total number of samples to be analyzed, sample handling procedures, and QA/QC measures)
- identify process data and method of collection
- estimate scheduling for sampling and analysis
- submit test plan for State Agency approval and make modifications as required

IV. Conduct Miniburn

V. Conduct sampling:

- make presampling preparation (clean and pack sample containers, necessary protective equipment, appropriate sampling equipment, record books, chain of custody forms, and sample identification labels)
- conduct sampling according to the test plan
- obtain data on plant operating conditions during sampling
- provide for field duplicates, blanks, splits, and spikes
- preserve and ship samples
- deliver samples for chemical analysis
- clean-up of sampling gear

VI. Prepare report on sampling rationale, methods, analytical results, processes description, and process parameters:
- evaluate data
- provide summary and conclusions
- submit draft report for review

The miniburn described in this sampling approach will consist of three test conditions. Conditions 1, 4, and 5 will be tested during the miniburn. These conditions were selected since they will provide an initial evaluation of the furnace performance for the POHCs with the highest incinerability ranking (HCB), the lowest heat of combustion (NG), and the munition feed with the highest ash and barium content. Results from the miniburn will be used to fine-tune the sampling plan to be certain that the trial burn data will be acceptable and appropriate for evaluating the furnace performance.

4.2 WASTE FEEDS (W_{IN} FOR POHCS)

Waste feed to the APE 1236 is monitored and controlled by the Automatic Waste Feed System (AWFS). After a munition has been selected, its descriptive name is entered into a computer that controls the AWFS. The computer checks a programmed database and sets the waste feed rate for the AWFS. When the munition is placed on the AWFS scale, the computer evaluates the munition weight and determines if the feedrate will exceed the pre-programmed limit. The weight of the munition load is recorded by the computer and saved to a database for future access. These data will be used to calculate the DRE of the furnace.

4.3 POHCs (W_{OUT})

4.3.1 Modified Method 5 Sampling Train

Summary of Method

Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in multicomponent sampling train. Particles that condense at or above a temperature of 120 $\pm$ 14° C (248 $\pm$ 25° F) are collected on a heated glass

fiber filter, and gases are sorbed onto a packed column of XAD-2 resin. Uncombined moisture is collected in a series of chilled glass impingers. The sample fractions are recovered and analyzed using high resolution chromatography to determine the amount of target analytes collected. The method is applicable for the collection of semi-volatile organic compounds with boiling points between 100° C (212° F) and 350° C.

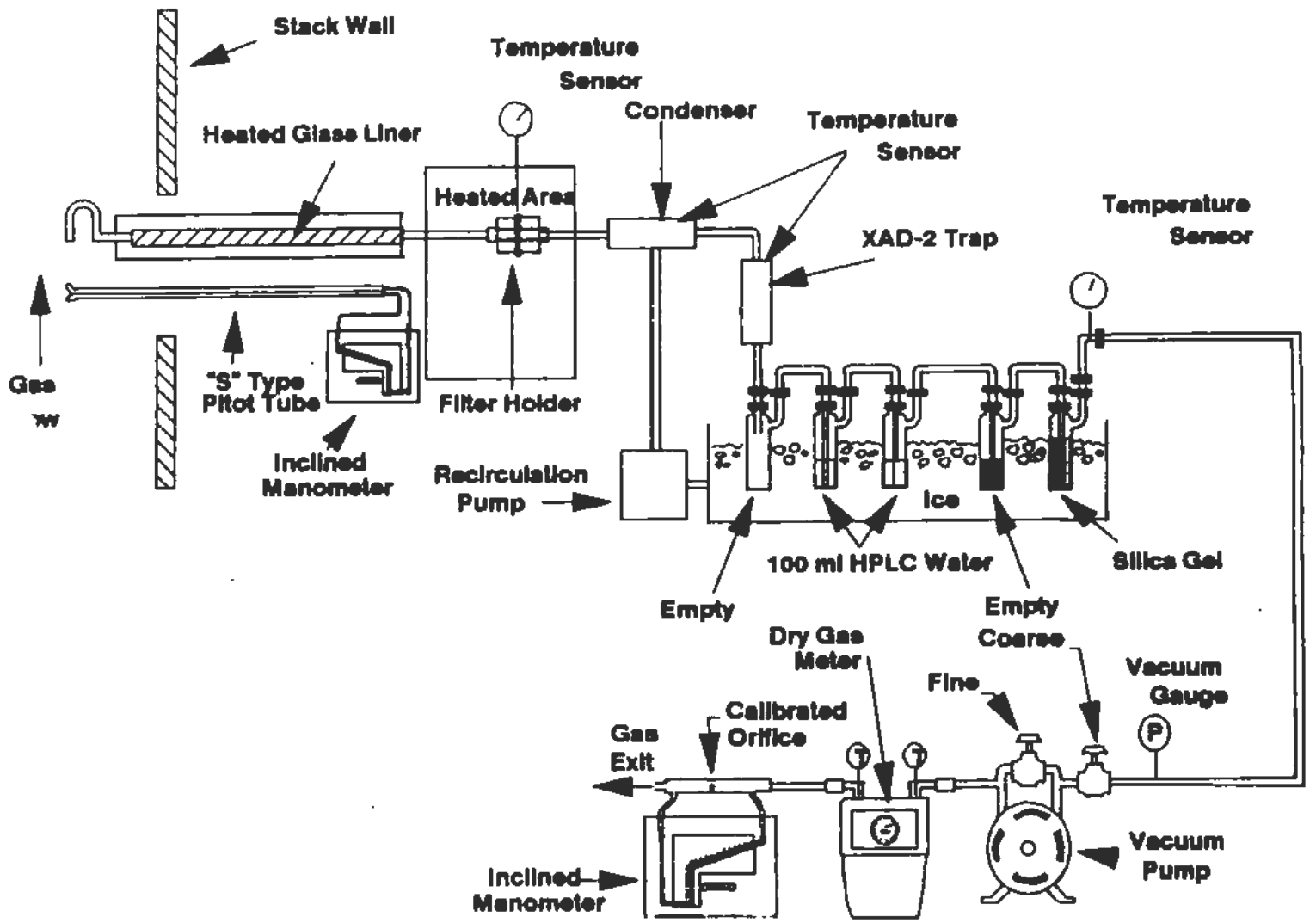
Description of Sampling Equipment

The Modified Method 5 (MM5) sampling train is based on the EPA Method 5 sampling train, but includes added components for the capture of semi-volatile organic species **(Figure G.3)** The sampling train consists of a stainless steel or glass nozzle; a glass probe liner with a heating system capable of maintaining the sample gas temperature at 248 ± 25° F; a type-S Pitot tube/dual manometer system to measure stack gas velocity pressure; an in-stack thermocouple capable of measuring the stack temperature to within 1.5 percent of the minimum absolute stack temperature; a glass filter holder with a glass filter frit support housed in a filter heating system capable of maintaining a temperature of 248 ± 25° F; an organic sampling module; a moisture condenser; and a pump and metering system.

The organic sampling module consists of three components: a water-cooled gas conditioner, a sorbent trap, and a moisture knock-out trap. The gas conditioner consists of a condenser coil which is surrounded by a water jacket through which ice water is continuously circulated. The condenser is designed to ensure that the temperature of the sample gas leaving the conditioner does not exceed 20° C (68° F). The second component of the organic sampling module is a sorbent trap that contains approximately 20 g of the porous polymeric resin. The sorbent trap is outfitted with a water jacket through which ice water is continuously circulated, such that the internal gas temperature is maintained at a temperature of 17 ± 3° C (62.5 ± 5.4° F). The sorbent module is outfitted with a thermocouple well into which a thermocouple can be inserted to monitor the internal gas temperature. The last section of the organic module consists of a moisture knock-out trap, which is simply an impinger with a shortened stem, so that the sample gas cannot bubble through the collected condensate during sampling.

FIGURE G.3

METHOD 23 AND SW-846 METHOD 0010 SAMPLING TRAIN



The moisture condenser ensures that the volume measured by the dry gas meter is free of moisture and also allows for the calculation of the stack gas moisture content via Method 4. The condenser consists of four impingers connected in series with leak-free ground glass fittings. The first impinger is a modified Greenburg-Smith impinger which is charged with 100 ml of high performance liquid chromatography (HPLC)-grade water, and the second impinger is a standard Greenburg-Smith which is also charged with 100 ml of HPLC-grade water. The third and fourth impingers are modified Greenburg-Smith impingers. The third impinger is empty, and the fourth contains a known mass of silica gel as a final water trap and to protect the sample pump and meter. The metering system consists of a vacuum gauge, leak free pump, a dry gas meter capable of measuring volume to within 2%, and thermocouples capable of measuring the dry gas meter temperature to within 5.4° F.

Description of Operation

After the sampling location and minimum number of sampling points have been determined, the stack pressure and temperature and the range of velocity heads are measured, and the moisture content is determined or estimated based on knowledge of the process. A nozzle size is selected based on the range of velocity heads such that it is not necessary to change nozzles during a run to maintain isokinetic sampling rates, and the differential pressure gauge is checked to ensure that it is capable of measuring the range of velocity heads.

A total sampling time is selected such that the sampling time per point is not less than two minutes, and that the sample volume collected meets or exceeds the minimum required volume. This volume is based on the expected concentration of the semi-volatile species present in the exhaust stream and the detection limit of the analytical method that will be used. A sample volume of at least three dry standard cubic meters (105.9 dscf) will be collected. During this trial burn, each MM5 sampling run will last for three hours. Prior to starting the sampling run, the portholes are cleaned to minimize the chance of sampling deposited material.

Because of the number of additional intercomponent connections in the Semi-VOST train which increase the possibility of leakage, a pre-test leak check is required. After the sampling train has been assembled, the filter and probe heating system are activated the allowed to stabilize at the desired operating temperatures. The sampling train is plugged off at the nozzle and a vacuum of fifteen inches Hg is applied to the train. Leakage rates in excess of

four percent of the average sampling rate or 0.02 cfm, whichever is less, are unacceptable. Silicone stopcock grease, which may be used on a Method 5 sampling train, is not allowed on the Modified Method 5 sampling train due to the possibility of analytical interference with the target analytes. If it is necessary to stop a sampling run during a test to replace a component of the sampling train, a leak check is conducted at the highest vacuum observed up to that point in the test before the sampling train is disassembled. Before resumption of the sampling, a leak check similar to the pre-test leak check is conducted.

At the beginning of each run, operational and run specific data, including the initial dry gas meter reading, are recorded at the top of the Particulate Field Data Sheet **(Figure G.4.)** The readings required on the data sheet are recorded at the end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted for any reason. The Pitot lines and manometer level are checked periodically to ensure accurate measurements of velocity head and sample flow rate.

To begin sampling, the probe nozzle cap is removed and the operating temperatures are verified. The probe is inserted into the stack to the first sampling point and the pump is immediately started. The sampling rate is adjusted rapidly to isokinetic conditions, and sampling is conducted for the previously determined time at each traverse point. During the sampling run the sampling rate is maintained within $\pm$ 10% of the true isokinetic sampling rate and the temperature of the probe and filter holder are maintained at 120 $\pm$ 14° C (248 $\pm$ 25° F). If the pressure drop across the filter becomes too large, making isokinetic sampling difficult to maintain, the filter may be changed. A leak check is performed before and after such changes.

At the end of the sampling run, the course adjust valve is closed and the probe is removed from the stack. The pump is turned off, a final dry gas meter reading is taken and a post-test leak check is performed. The post-test leak check is conducted in the same manner as the pre-test leak check except that the vacuum placed on the system is equal to, or slightly greater than the maximum vacuum observed during the sampling run. The maximum leakage rate allowed is 0.02 cfm. If the post-test leakage rate exceeds 0.02 cfm, the run is invalidated.

FIELD DATA

Plant ______
Date ______
Sampling Location ______
Sample Type ______
Run Number ______
Operator ______
Ambient Temperature ______
Barometric Pressure ______
Static Pressure (P_s) ______
Filter Number(s) ______
Pretest Leak Rate = ____ cfm @ ____ in. Hg
Pretest Pitot Leak Check ______
Pretest Orsat Leak Check ______
Read and Record all Data Every ___ Minutes

Schematic of Traverse Point Layout

Probe Length and Type ______
Pitot Tube I.D. No. ______
Nozzle I.D. ______
Assumed Moisture, % ______
Temp. Readout S/N ______
Meter Box Number ______
Meter $\Delta H_@$ ______
C Factor ______
Meter Gamma ______
Heater Box Setting ______
Reference Δp ______
Post Test Leak Rate = ____ cfm @ ____ in. Hg
Post Test Pitot Leak Check ______
Post Test Orsat Leak Check ______

Traverse Point Number	Sampling Time, (min) / Clock Time (24-hour clock)	Gas Meter Reading (V_m) ft^3	Velocity Head (ΔP_s) in. H_2O	Orifice Pres. Differential (ΔH) in. H_2O		Stack Temp. (T_s) °F	Dry Gas Meter Temp.		Pump Vacuum in. Hg	Sample Box Temp. Filter Temp. °F	Impinger Temp. °F
				Desired	Actual		Inlet ($T_{m_{in}}$) °F	Outlet ($T_{m_{out}}$) °F			
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FIGURE G.4
PARTICULATE FIELD DATA SHEET

Sample Recovery

When the probe is removed from the stack, it is allowed to cool. The nozzle is cleaned of all external particulate matter and capped to prevent contamination or loss of sample. After the final leak check, the sample train is disassembled and all openings are capped. The probe, filter assembly, organic module, and the impingers are removed to the cleanup area. At the sample clean-up area, the contents of the sampling train components are quantitatively recovered into glass storage containers and logged into the field sample log.

Container No. 1 consists of the glass fiber filter/petri dish set. The exposed filter is removed from the filter holder using tweezers and carefully transferred to its original petri dish. Any loose particles that are present in the front-half of the filter holder are carefully brushed into the petri dish, as is filter material that may be attached to the filter frit. The interior surfaces of the nozzle and probe are washed with a 1:1 solution of pesticide-grade methanol/methylene chloride into a pre-cleaned glass storage bottle, which comprises Container No. 2. The probe liner is brushed with a nylon brush until no visible particles appear in the wash, and the liner is rinsed a final time with the solvent mix. The cyclone bypass (if used), and front half of the filter holder are also rinsed with the solvent mix and collected with the probe rinse. A sample label is filled out an attached to the outside of the bottle, the liquid level is marked, and the lid is wrapped with Teflon® tape. Container No. 3 consists of the sorbent trap section of the organic sampling module. The sorbent trap is removed from the sampling train and tightly capped using ground glass fittings and impinger clamps. The trap is then wrapped in aluminum foil, labeled, and placed inside of a leak-proof bag. The trap is then stored on ice for shipment to the analytical laboratory.

The weight of the condensate in the knock-out impinger is measured by weighing the impinger and condensate to the nearest milligram. The condensate is then transferred to a pre-cleaned glass sample bottle, which comprises container No. 4. The back-half of the filter holder and the gas condenser are visually examined to determine the presence of condensate. If any is present, the volume is added to the knock-out impinger and weighed and recovered with that impinger. A sample label is filled out and attached to the outside of the sample bottle, the liquid level is marked and the cap is wrapped in Teflon® tape. Container No. 5 consists of a methanol/methylene chloride rinse of the back-half of the filter holder, the gas condenser and the condensate knockout impinger. The rinses from these components are collected in a pre-cleaned glass bottle a label is filled out and attached to the outside of the

bottle, the liquid level is marked, and the cap is wrapped with Teflon® tape. If brushing of any of these components is required, the brush is rinsed with the solvent solution, and the rinse is collected into Container No. 5.

The three impingers behind the condensate knockout are individually weighed and then the contents of these impingers are recovered for analysis. The contents are transferred into a pre-cleaned glass bottle, and a label is filled out and attached to the outside of the bottle. The liquid level is marked and the cap is wrapped in Teflon® tape. The silica gel impinger and its contents are weighed together and subtracted from the initial weight of the impinger and its contents. A note indicating the color and condition of the silica gel is made on the sample recovery data sheet to record whether moisture break-through has occurred.

Sample Train Pre-test Preparations
Glassware Cleaning Protocol

1. Acetone rinse with reagent-grade acetone.
2. Four-hour hot detergent soak using Alconox or other suitable glassware cleaning detergent.
3. Hot tap water rinse.
4. Bake for 12 hours at 250° C.
5. Four-hour chromic acid soak. Alternatively, a two percent v/v solution of ChemSolv concentrate from Malinkrodt may be used.
6. Hot tap water rinse.
7. Distilled, deionized water rinse.
8. Acetone rinse with pesticide-grade acetone.
9. Methylene chloride/methanol rinse with a 1:1 solution of pesticide-grade methylene chloride and methanol.

Filter Preparation Protocol

Petri dishes are purchased new and are washed with distilled, deionized water followed by acetone and are dried at 105° C for 1 hour. The petri dishes are allowed to cool, and are marked with a unique identifying code. One filter is assigned to each numbered petri dish, and the combination is thereafter treated as a single unit.

XAD-2 Resin Preparation Protocol

XAD-2 adsorbing resin used in the sorbent trap must be carefully extracted and dried prior to its use in the field, whether the resin used has been purchased new or is being recycled. The resin preparation involves repeated extractions with Type II distilled, deionized water, methyl alcohol, and methylene chloride. Extractions may be conducted using a giant Soxhlet extractor or a continuous extractor which has been constructed according to specifications outlined in "Test Methods for Evaluating Solid Waste, Physical Chemical Methods, Field Manual, Volume II SW-846", Part III, Chapter 10, Method 0010, Appendix A, "Preparation of XAD-2 Sorbent Resin." Initially, the resin is rinsed in a beaker with Type II water, then the resin is extracted for 8 hours with Type II water. This is followed by a 22-hour extraction with methyl alcohol, a 22-hour extraction with methylene chloride, and a final 22-hour extraction with fresh methylene chloride. After the extractions, the cleaned XAD-2 resin is dried using the fluidized bed technique by gently passing a stream of nitrogen through a bed of XAD-2 resin. Before its use in the field, the prepared XAD-2 resin is subjected to three quality assurance checks to evaluate it acceptability for use. The quality control results must be reported for each batch of resin extracted. The control procedures consist of analyzing the prepared resin to determine: 1) the amount of residual methylene chloride on the resin, which must be shown to be less that 1,000 μg/g of resin; 2) the amount of residual extractable organic, which must be less than 20 μg/ml of extract; 3) the presence of any of the target analytes in the resin extract in concentration greater that 25 μg/ml, and 4) the amount of residue remaining after the extract is dried, which must be less than 0.5 mg. The QA/QC procedures are conducted on a 20 g sample of the prepared resin. If any of the results of the QA/QC checks exceed the acceptable limits, the resin is re-extracted.

Description of Sample Analysis

The sample train fractions are spiked with surrogate standards, extracted and concentrated to volumes of approximately 1.0 ml prior to analysis by Gas Chromatography/Mass Spectrometry (GC/MS). GC/MS with fused-silica capillary columns is the primary analytical tool for the measurement of emissions from hazardous waste incinerators. Prescreening of the sample extracts is often conducted using GC/FID (flame ionization detector) or GC/ECD (electron capture detector), to determine the analytes present in the sample and their approximate concentrations in the sample extract.

4.3.2 Energetic Materials Sampling Train

Summary of Method

Particulate matter and organic materials are withdrawn isokinetically from the source and collected on a glass fiber filter, in a series of three chilled impingers, and in a chilled resin tube which contains approximately 20 grams of porous polymeric adsorbing material (XAD-2 resin). The fractions are recovered, extracted, and analyzed using GC/ECD, GC/NPD, and GC/MS.

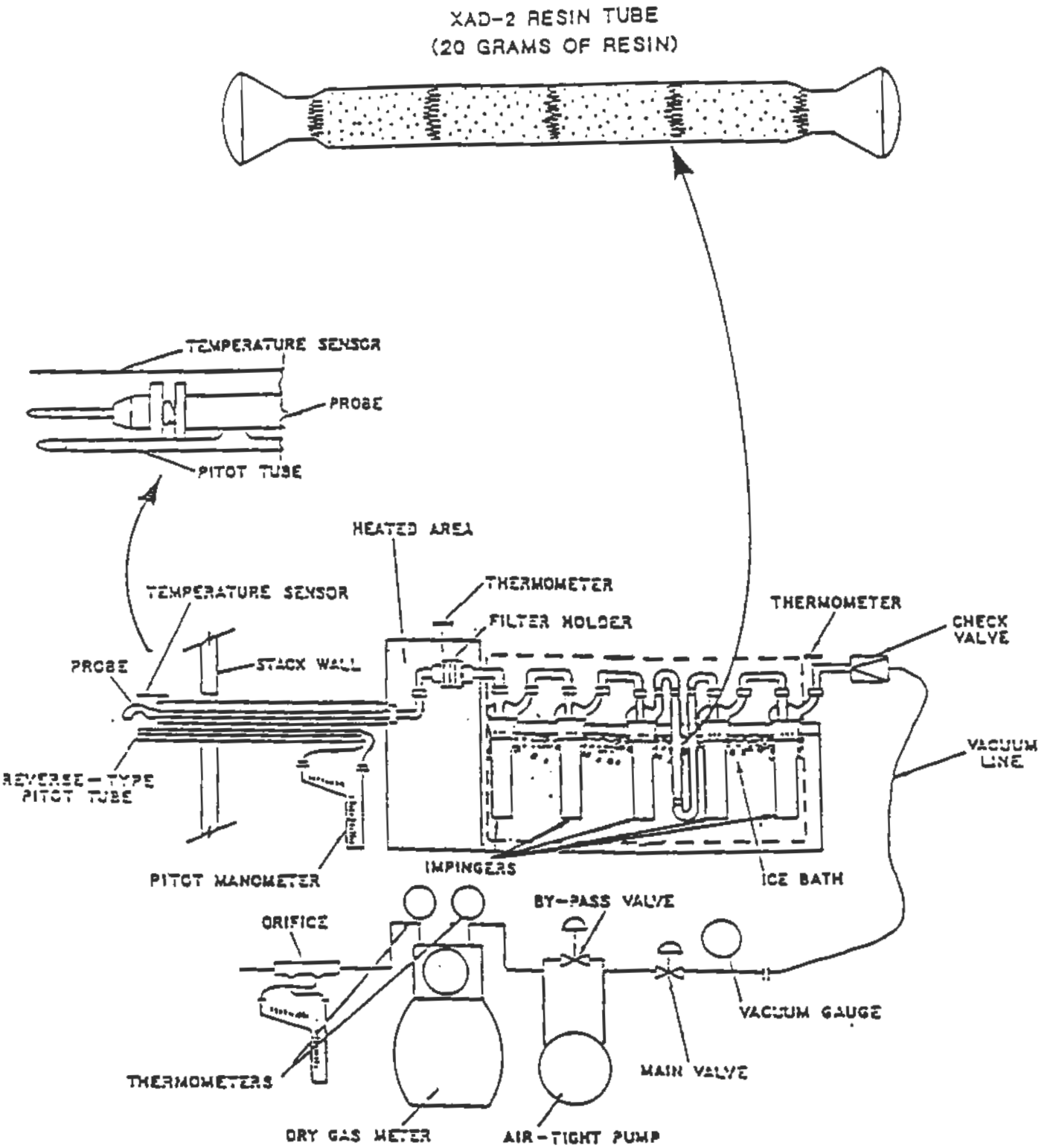
Description of Sampling Equipment

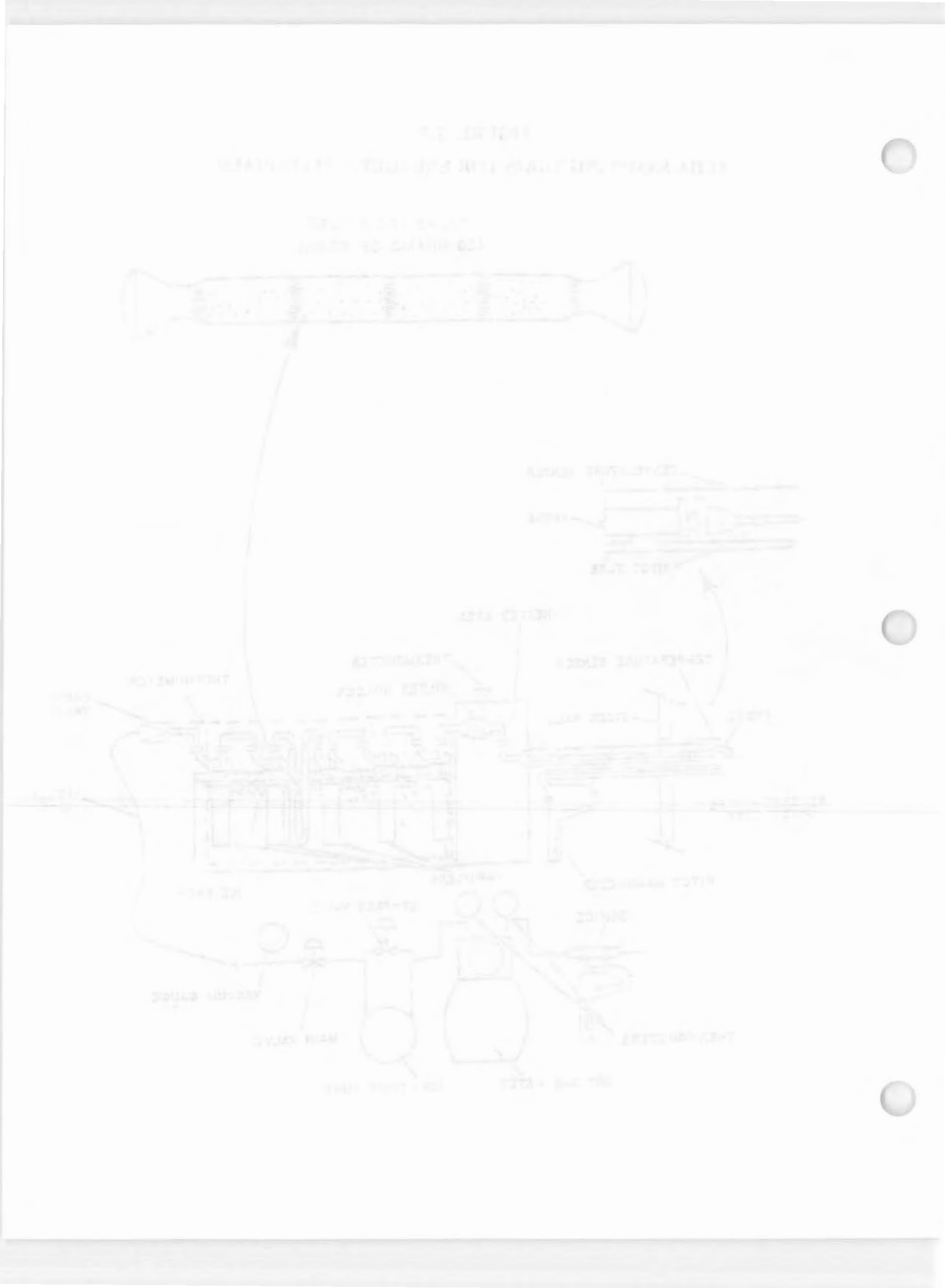
The energetic materials sampling train has been developed and used by the US Army Environmental Hygiene Agency (USAEHA). The sampling method is based on the US EPA Modified Method 5 sampling train, and is designated AEHA MM5. The sampling train is similar to the Method 5 train, and consists of a stainless steel or glass nozzle; a glass probe liner with a heating system capable of maintaining the sample gas temperature at 248 $\pm$ 25° F; a type-S Pitot tube/dual manometer system to measure stack gas velocity pressure; an in-stack thermocouple capable of measuring the stack temperature to within 1.5 percent of the minimum absolute stack gas temperature; a glass filter holder with a glass filter frit support housed in a filter heating system capable of maintaining a temperature of 248 $\pm$ 25° F; a moisture condenser/sorbent module system; and a pump and metering system **(Figure G.5)**.

The condenser/sorbent module system provides additional media for the capture of the analytes of interest, condenses moisture in the stack gas to allow calculation of the stack gas moisture content, and protects the pump and metering system. The condenser/sorbent module system consists of five impingers and a sorbent module which contains approximately 20 grams of XAD-2 resin connected in series with leak-free ground glass fittings. The first impinger is a modified Greenburg-Smith impinger with a shortened stem which extends about half-way into the impinger bottle. The impinger is charged with 50 ml of HPLC-grade water which is used as a spiking medium. The second impinger is a modified Greenburg-Smith impinger with a shortened stem that extends about three-fourths of the way into the impinger bottle, and is left dry. The third impinger is a standard modified Greenburg-Smith impinger, which is also left dry. The sorbent resin tube is connected to the outlet of the third impinger.

FIGURE G.5

AEHA SAMPLING TRAIN FOR ENERGETIC MATERIALS





The tube is charged with 20 g of XAD-2 resin, packed in 5-g sections. The sections are separated with glass wool, and glass wool is also packed on the ends of the tube to prevent the loss of resin during sampling. The sorbent tube is situated vertically and is connected to the outlet of the third impinger with a U-tube. The outlet of the sorbent tube is connected to a straight glass tube with another U-tube and the tube is connected to the inlet of the fourth impinger with a third U-tube. The fourth and fifth impingers are both modified Greenburg-Smith impingers. The fourth impinger is left dry and the fifth impinger is charged with a known mass of silica gel desiccant.

The metering system consists of a vacuum gauge, leak free pump, a dry gas meter capable of measuring volume to within 2%, and thermocouples capable of measuring the dry gas meter temperature to within 5.4° F.

Description of Operation

After the sampling location and minimum number of sampling points have been determined, the stack pressure and temperature and the range of velocity heads are measured, and the moisture content is determined or estimated based on knowledge of the process. A nozzle size is selected based on the range of velocity heads such that it is not necessary to change nozzles during a run to maintain isokinetic sampling rates, and the differential pressure gauge is checked to ensure that it is capable of measuring the range of velocity heads.

A total sampling time is selected such that the sampling time per point is not less than two minutes, and that the sample volume collected meets or exceeds the minimum required volume. During this trial burn, STEM sampling runs will last one-hour and collect approximately 45 scf. The portholes are cleaned to minimize the chance of sampling deposited material.

Prior to the start of a sampling run a pre-test leak check is conducted on the sampling train to ensure that ambient air will not dilute the stack gas sample during the sampling run. The sampling probe is capped off at the nozzle, the pump is activated, and the sampling train is evacuated to approximately fifteen inches of mercury vacuum. The sample train leakage rate is read from the dry gas meter and recorded. If the leakage rate is less than 0.02 cubic feet per minute, the sample train is considered acceptable for use.

At the beginning of each run, operational and run specific data, including the initial dry gas meter reading, are recorded at the top of the Particulate Field Data Sheet. The readings required on the data sheet are recorded at the end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted for any reason. The Pitot lines and manometer level are checked periodically to ensure accurate measurements of velocity head and sample flow rate.

To begin sampling, the probe nozzle cap is removed and the operating temperatures are verified. The probe is inserted into the stack to the first sampling point and the pump is immediately started. The sampling rate is adjusted rapidly to isokinetic conditions, and sampling is conducted for the previously determined time at each traverse point. During the sampling run the sampling rate is maintained within $\pm$ 10% of the true isokinetic sampling rate and the temperature of the probe and filter holder are maintained at 120 $\pm$ 14° C (248 $\pm$ 25° F). If the pressure drop across the filter becomes too large, making isokinetic sampling difficult to maintain, the filter may be changed. A leak check is performed before and after such changes.

At the end of the sampling run, the course adjust valve is closed and the probe is removed from the stack. The pump is turned off, a final dry gas meter reading is taken and a post-test leak check is performed. The post-test leak check is conducted in the same manner as the pre-test leak check except that the vacuum place on the system is equal to a slightly greater than the maximum vacuum observed during the sampling run. The maximum leakage rate allowed is 0.02 cfm. If the post-test leakage rate exceeds 0.02 cfm, the run is either invalidated, or a correction is made to the sample volume based on the recorded leakage rate.

Sample Train Pre-test Preparations
Glassware Cleaning Protocol

All glass components upstream and including the adsorbent module as well as glass petri dishes for filter storage and transport are cleaned based on the protocol described in Section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." When cleaning glassware, special care is given to the removal of grease sealant from the components. The glassware cleaning procedure consists of eight steps outlined below:

1. Acetone rinse with reagent-grade acetone.
2. Four-hour hot detergent soak using Alconox or other suitable glassware cleaning detergent.
3. Hot tap water rinse.
4. Four-hour chromic acid soak. Alternatively, a 2 percent v/v solution of ChemSolve® (Malinkrodt).
5. Hot tap water rinse.
6. Distilled, deionized water rinse.
7. Acetone rinse with pesticide-grade acetone.
8. Methylene chloride rinse with pesticide-grade methylene chloride.

After the glassware has been allowed to air dry, all openings are sealed with heavy-duty aluminum foil that has been previously rinsed with pesticide-grade hexane.

Filter Preparation Protocol

Glass fiber filters without organic binder, are capable of capturing at least 99.95 percent when subjected to 0.3-micron dioctyl phthalate smoke particles. Filters are examined for pinhole leaks by holding them up to the light. Filters are cleaned before use by extraction in a Soxhlet extraction apparatus once with toluene for three hours and repeated with toluene for 16 hours. The filters are removed from the apparatus and allowed to cool, and are dried under a clean stream of nitrogen. The filters are then stored in cleaned petri dishes and sealed with Teflon® tape.

XAD-2 Resin Preparation Protocol

XAD-2 adsorbing resin used in the sorbent trap must be carefully extracted and dried prior to its use in the field, whether the resin used has been purchased new or is being recycled. The resin preparation involved repeated extractions with Type II distilled, deionized water, methyl alcohol, methylene chloride, and toluene. Extractions may be conducted using a giant Soxhlet extractor or a continuous extractor which has been constructed according to specifications outlined in "Test Methods for Evaluating Solid Waste, Physical Chemical Methods, Field Manual, Volume II SW-846", Part III, Chapter 10, Method 0010, Appendix A, "Preparation of XAD-2 Sorbent Resin." Initially, the resin is rinsed in a beaker with Type II water, then the resin is extracted for 8 hours with Type II water. This is followed by a

22-hour extraction with methyl alcohol, a 22-hour extraction with methylene chloride, and a 22-hour extraction with toluene. After the extractions, the cleaned XAD-2 resin is dried using the fluidized bed technique by gently passing a stream of nitrogen through a bed of XAD-2 resin.

Before its use in the field, the prepared XAD-2 resin is subjected to a quality assurance check to evaluate it acceptability for use. The quality control results must be reported for each batch of resin extracted. A 1.0 mg sample of the dried resin is weighed out and transferred to a small vial, and 3 ml of toluene is added. The vial is capped and shaken. The sample resin is extracted and the a 2 μl sample of the extract injected into a gas chromatograph for analysis. The results of the analysis are compared against the results of a reference solution which consists of a mixture of 2.5 μl of methylene chloride and 100 ml of toluene. The maximum concentration of methylene chloride allowed is 1000 μg per gram of resin. If this limit is exceeded the resin must be further dried until the solvent concentration is acceptable. The cleaned resin has a shelf-life of four weeks when stored in an amber glass container with a Teflon®-lined cap.

During the preparation and assembly of the sampling train, all openings where contamination may occur are kept covered until just prior to assembly or sampling. Fifty milliliters of distilled water are placed in the first impinger and the contents of a tared silica gel bottle are transferred into the fourth impinger. Leak tight connections are made and secured between the impingers. Using tweezers or clean disposable surgical gloves, a tared filter is removed from its packaging and placed in the filter holder such that it is properly centered and the gasket prevents the gas stream from circumventing the filter. The nozzle is connected to the probe by a leak-free fitting and the probe is marked to indicate the proper distance into the stack for each sampling point. The train is then assembled taking care to ensure leak-tight connections. Ice is packed around the impingers.

Sample Recovery

When the probe is removed from the stack, it is allowed to cool. The nozzle is cleaned of all external particulate matter and capped to prevent contamination or loss of sample. After the final leak check, the sample train is disassembled and all openings are capped. The probe, filter assembly, organic module, and the impingers are removed to the cleanup area.

At the sample clean-up area, the contents of the sampling train components are quantitatively recovered into glass storage containers and logged into the field sample log.

Container No. 1 consists of the front-half toluene rinse. The probe nozzle, probe liner, and cyclone by-pass are rinsed with toluene and the rinses are collected in a pre-cleaned glass bottle. The probe nozzle and liner is brushed at least three times, and the brush is rinsed with toluene into the sample bottle. The filter is carefully removed from the filter holder and placed into the bottle containing the probe rinse. Any filter material present on the filter support frit is carefully scraped off and transferred to the bottle. The front-half of the filter holder and the cyclone bypass which connects the probe liner to the filter holder are rinsed with toluene and then brushed three times each, and the rinse is added to Container No. 1. A sample label is filled out attached to the outside of the bottle, the liquid level in the bottle is marked and the cap is sealed with Teflon® tape. The bottle is refrigerated and stored for shipment to the laboratory.

Container No. 2 consists of an acetone rinse of the probe nozzle, probe liner, cyclone bypass, and the front-half of the filter holder. Each of these components is rinsed three times and the rinses collected in a pre-cleaned glass bottle. A sample label is filled out and attached to the outside of the bottle, the liquid level is marked, and the bottle is seal with Teflon® tape. The bottle is then refrigerated and stored for shipment to the laboratory.

The three impingers upstream of the sorbent module are weighed to determine the weight gain due to moisture collected during the sampling run. The condensate in these impingers is then collected into a pre-cleaned glass sample bottle. Each impinger is then rinsed and shaken with distilled, deionized water, and the water added to the condensate. The condensate/rinse is then extracted three times with toluene. The extraction solvent volume for the first extraction is approximately 4 to 1, water to toluene, and the solvent volume for the second extraction is conducted with 20 ml of toluene, as is the third extraction. The first two extractions are conducted at the resultant pH of the combined condensate and rinse solutions, but the third extraction is conducted at a pH of 4 or less. The condensate is acidified using sulfuric acid. The extract from the condensate and rinses is collected in a pre-cleaned glass bottle and sealed. This bottle comprises container No. 3. A sample label is filled out and attached to the outside of the sample bottle, and the liquid level is marked. The cap is wrapped with Teflon® tape, and the sample is refrigerated and stored for shipment to the laboratory.

Each section of the sorbent module is recovered separately and extracted three times, with each extraction generating a separate sample. The sorbent module therefore generates twelve samples for analysis. These extracts comprise Container Nos. 4A-C, 5A-C, 6A-C, and 7A-C. The first resin section, along with the glass wool plug between the first and second section, is carefully removed from the sorbent module, extracted with 30 ml of toluene, and shaken for 30 minutes. The extract is transferred to a pre-cleaned glass sample bottle, sealed, labeled, marked and refrigerated. The resin is extracted again with 10 ml of toluene and shaken for thirty minutes, collected into a pre-cleaned sample bottle, sealed, labeled, marked and refrigerated. The resin is then extracted a third time with 10 ml toluene and shaken for 30 minutes. The extract from the third extraction and the resin are both transferred to a pre-cleaned glass sample bottle, sealed, labeled, marked, and refrigerated for shipment. The recovery of the succeeding three resin sections and glass wool plugs is conducted repeating the procedures described above.

The impingers downstream of the sorbent module are recovered and analyzed for moisture only. The fourth and fifth impingers are weighed to determine the net weight gain of the silica gel due to moisture. The contents of both the fourth and the fifth impingers are discarded.

Calibration Procedures

The analytical balance used in the analysis is serviced and calibrated every 6 months by a factory service representative. Prior to each weighing session, the balance is calibrated with Class S weights at either 60 g, for filter weighings, or 100 g for beaker weighings.

Meter boxes are subjected to a five point calibration when they are initially received, and annually thereafter. At the conclusion of a field test, a calibration check is performed to ensure that the meter correction factor has not changed. The calibration check is conducted at one point equivalent to the average orifice pressure and highest system vacuum attained during the test. After the meter box is leak checked, the barometric pressure and temperature at the wet test meter and at the inlet and outlet of the dry gas meter are recorded on a calibration form and the meter box is allowed to warm-up. The initial dry gas meter and wet test meter readings are taken. Sample times are chosen to give 5-10 cubic feet total volume, and measured with a stop watch. Final readings are taken and recorded on the calibration sheet.

4.3.3 Volatile Organic Sampling Train (VOST)

Summary of Method

This method is used to collect Volatile Principal Organic Hazardous Constituents (POHCs) from stack gas effluents of hazardous waste incinerators. A 20-liter sample of effluent gas is withdrawn from the stack and the POHCs are collected on a pair of Tenax and Tenax/Charcoal sorbent tubes. The resin is desorbed and analyzed using a GC/MS.

Description of Sampling Equipment

The VOST consists of a condenser, a sorbent cartridge containing Texax (1.6 g), an empty impinger for condensate removal, a second water-cooled glass condenser, a second sorbent cartridge containing Tenax and petroleum-based charcoal (3:1 by volume; approximately 1g of each), a silica gel drying tube, a calibrated rotameter, a sampling pump, and a dry gas meter. The gas pressure during sampling and for leak-checking is monitored by pressure gauges which are in line and downstream of the silica gel drying tube.

Description of Operation

The VOST system is designed to draw effluent gas through a probe into a water cooled condenser and two sorbent traps. The condenser is utilized to assure that the temperature of the gas entering the first sorbent trap is 21°F (70°F) or less. Water vapor condensed by cooling the sampled gas is allowed to percolate through the first sorbent trap.

The gas then passes through a second sorbent trap connected in series. The first sorbent trap is packed with approximately 1.6 grams of clean Tenax-GC resin, while the second trap contains both Tenax-GC and activated charcoal. The second trap is packed and oriented such that the sampled gas passes through roughly 1.0 gram of Tenax-GC first, followed by 1.0 gram of charcoal. During a sampling period of 20 minutes, a total volume of 20 liters will be drawn through each pair of traps. A VOST run will consist of the collection of three independent and sequential 20-L samples of the stack gas through a pair of sorbent cartridges. The intent of collecting three independent samples is to allow intitial characterization with one pair of the cartridges to assess the level of POHCs present. Then the remaining pair (if necessary) of cartridges are desorbed to meet detection limit requirements in the stack gas. The

condensate is analyzed separately as an aqueous sample.

Sample Train Pre-Test Preparations

Prior to shipment of the VOST sorbent traps for sampling, all traps will be conditioned by baking them at 200 deg C for 18 hours while purging them with helium or nitrogen at a rate of about 30 mL/min. One pair of traps from each batch will be chosen at random and checked for contamination using the thermal desorption GC/MS procedure. If the sorbent trap pair taken is not contaminated, the remaining tubes in the batch will be packed for shipment. Otherwise, the batch will be reconditioned and rechecked. Sealed, conditioned traps will be shipped to the field by placing them in clean, glass screw cap vials, which will be placed into gallon metal cans, packed with frozen blue ice. The can will then be sealed and packed on ice.

In the laboratory, pairs of VOST sorbent traps will be prepared and analyzed as follows. Prior to the analysis, each pair of Tenax/Tenax/Charcoal tubes will be joined together using a stainless steel Swagelok union such that the direction of gas flow through the traps will match that during sampling. The inlet end of the trap pair will be attached to the exit (downstream) side of a gas chromatograph injector port. The internal standard and surrogates will be flash vaporized onto each trap pair.

The VOST pair of sorbent traps will then be detached from the injection port and connected to the thermal desorption purge-and-trap unit. The direction of purge gas flow will be counter to that used during sampling and during spiking. The samples will then be analyzed by thermal desorption purge-and-trap GC/MS.

Sample Recovery

Calibration Procedures

A minimum of four calibration standards (traps containing the target analyte, surrogate, and internal standard) will be prepared and analyzed for initial calibration. At least 1 pair of lab blank traps (trap containing the surrogate and internal standard) will be analyzed prior to field samples and a pair of lab blank traps will be analyzed each day field samples are analyzed. One pair of spiked duplicate traps (containing target analyte, surrogate and internal

standard) will also be analyzed daily. One pair of audit sample traps (from EPA) will also be analyzed prior to field sampling.

The concentration of the target analyte (POHC), TCE will be quantified. In addition other compounds, present in excess of 10% of the response for the internal standard in the samples will be tentatively identified. The tentaive identifications will be generated by means of an on-line search against the NBS library of mass spectra. The computer-generated identifications will be reviewed by a mass spectral interpretation specialist. "Tentatively identified compound" assignments will be reported if the identifications from the computer search seemed reasonable in the specialist's professional judgement, considering the similarities and differences between sample and reference mass spectra and the retention time of the non-versus the internal standard, assuming a relative response factor of 1.0.

4.4 Determination of CO_2 and O_2 Concentration by USEPA Method 3

Summary of Method

The composition of the exhaust gas from combustion sources and many other process exhausts may be measured by the Orsat™ analyzer. The method measures the concentrations of oxygen and carbon dioxide in the dry gas. It is assumed that the balance of the dry gas is nitrogen and the molecular weight of the dry gas is calculated. The result is accurate if no other compounds (other than carbon monoxide) are present at significant concentration. The presence of carbon monoxide does not introduce errors since its molecular weight is the same as that of molecular nitrogen.

Description of Sampling Equipment

The sampling train consists of a probe, a moisture removal device, a pump, a rate meter and an inert-plastic bag. In addition, in cases where USEPA Method 5 is being used to measure

particulate matter, the Method 3 sampling probe is usually attached to the Method 5 probe so as to obtain a traversed sample.

Description of Operation

particulate matter, the Method 3 sampling probe is usually attached to the Method 5 probe so as to obtain a traversed sample.

Description of Operation

A Method 3 run will be conducted at the start of each day of testing and during each sampling run.

A sample of gas is withdrawn from a single point in the stack, dried and pumped into an inert plastic bag at approximately one liter per minute for a minimum of 30-minutes.

At the conclusion of the sampling period a sample of the gas from the bag is caused to displace 100 cc of slightly acidic water in a burette. This gas is then flushed back and forth through a gas washing bottle that contains a strong caustic solution which absorbs carbon dioxide. The volume of gas remaining is then measured in the burette. The gas is then flushed through a second gas washing bottle that contains alkaline paragallol which absorbs oxygen.

After each absorption step the volume of gas remaining is measured and recorded. The gas remaining is assigned a molecular weight of 28 amu, which is the molecular weight of both nitrogen (N_2) and carbon monoxide (CO). The presence of other gases at typically encountered concentrations (i.e., SO_2, NO_x) does not affect the accuracy of the measurement significantly; in fact, argon which is present at 0.9 percent in air, is usually ignored.

Description of Leak Check Procedures

The procedure for leak checking of the sampling train consists of inspection of all connections and fittings to ensure that they are tight. The procedure for leak checking of the Orsat™ analyzer consists of filling all the gas washing bottles and the burette to their zero marks, placing the reservoir bottle at the level of the bottom of the burette and observing the device for four minutes. If the burette level changes no more than 0.2 ml, and the level in the gas masking bottle does not fall below the bottom of the capillary, the unit is deemed leak-free.

The flexible bag is leak-checked by pressurizing it to 2"-4"water above atmospheric pressure. The pressurized bag is then connected to a manometer and allowed to stand for 10 minutes. A leak-free bag will hold the pressure constant.

Description of Sample Volumes and Detection Limits

The sample is collected at a rate of 0.25 to 1.0 lpm. The total volume collected ranges from 30 to 60 liters depending upon the length of the sampling run. A sample collected in conjunction with a particle test run will be of longer duration than one collected in conjunction with a moisture (USEPA Method 4) run.

The limit of detection of the procedure is approximately 0.2 percent by volume of the gases measured.

Calibration Procedures

Calibration consists of ensuring that the absorbing solutions are not so weak that they will not effectively absorb the CO_2 and O_2. The O_2 solution can be checked conveniently by analyzing ambient air and, because its total capacity is far less than the CO_2 absorbing reagent, this is usually adequate.

4.5 STACK GAS MOISTURE

Moisture Concentration by USEPA Reference Method 4

Summary of Method

Water vapor is a major component of many gas streams and the concentration must be measured in order to calculate the dry molecular weight of the stack gas. The stack gas molecular weight is in turn used in the calculation of velocity and volumetric flow. In addition, moisture concentration must be determined to allow calculation of pollutant concentrations on both dry and wet bases for tests when Continuous Emission Monitors (CEMS) are used. Method 4 is a procedure whereby moisture is removed from a metered volume of stack gas and the quantity of water collected is measured. Since one gram of water occupies 0.0472 ft^3 when in the vapor phase at standard temperature and pressure, the

volume of water vapor can be determined from the amount of water collected, and the moisture proportion by volume calculated if the volume of dry gas sampled is known.

Description of Sampling Equipment

The sampling equipment consists of a heated probe, an in-stack or heated out-of-stack filter, moisture trap, vacuum pump, and a dry gas meter. The moisture trap configuration used by ES consists of a train of four glass impingers. The impinger train consists of three Greenburg-Smith impingers and one modified Greenburg-Smith impinger. The impinger train is charged with known quantities of water and silica gel prior to each test. The pump and meter system is identical to that used in a Method 5 test.

Description of Operation

Prior to sampling, the impingers are charged with water and silica gel. The first impinger is a modified Greenburg-Smith impinger, and is charged with 100 ml of water, the second impinger, a standard Greenburg-Smith type, is charged with 100 ml of water, the third impinger (modified Greenburg-Smith) is left empty, and the last impinger (modified Greenburg-Smith) is charged with approximately 200 g of indicating silica gel.

Sample gas is withdrawn from the stack at a constant rate and the volume recorded by subtracting the final volume on the dry gas meter from the initial volume. The minimum sample volume required by the method is 21 dry standard cubic feet. The Method 4 sampling train is frequently combined with the Method 5, or any of a number of sampling trains (e.g., Multiple Metals or Modified Method 5 sampling trains) so that the target pollutant and moisture concentrations can be determined for identical test runs. When the M4/M5 combination is used, the sampling is almost always conducted at isokinetic sampling conditions.

Description of Leak Check Procedures

Prior to sampling, the probe tip of the sample train is plugged and a vacuum of 15 inches of mercury is placed on the system by the sample pump. The maximum leakage rate allowed is 0.02 cubic feet per minute. At the conclusion of sampling, a post-test leak check is conducted at the highest vacuum observed during the test run. Once again, the maximum

allowable leak rate is 0.02 cfm. Should the moisture train leak at the rate greater than the maximum allowed, the run is voided, or a correction to the sample volume is made which corrects for the observed post-test leak rate.

Description of Sample Volumes and Detection Limits

Method 4 specifies that the sample volume must be at least 21 cubic feet, corrected to standard conditions; however, in practice, the Method 4 train is often used in conjuction with a Method 5 test, which specifies a minimum corrected volume of 30 cubic feet. The detection limit is limited by the precision and accuracy of the graduated cylinder and analytical balance used to measure the volume and mass of the contents of the impinger train. A stack gas moisture concentration of approximately one percent may be detected by the method, but at low moisture concentrations (i.e., <3percent) precision is lost. In cases where the moisture is 3 percent or less, alternate procedures, such as wet/dry bulb psychrometry are generally more appropriate.

4.6 PARTICULATE MATTER

Determination of Particulate Emissions by USEPA Reference Method 5

Summary of Method

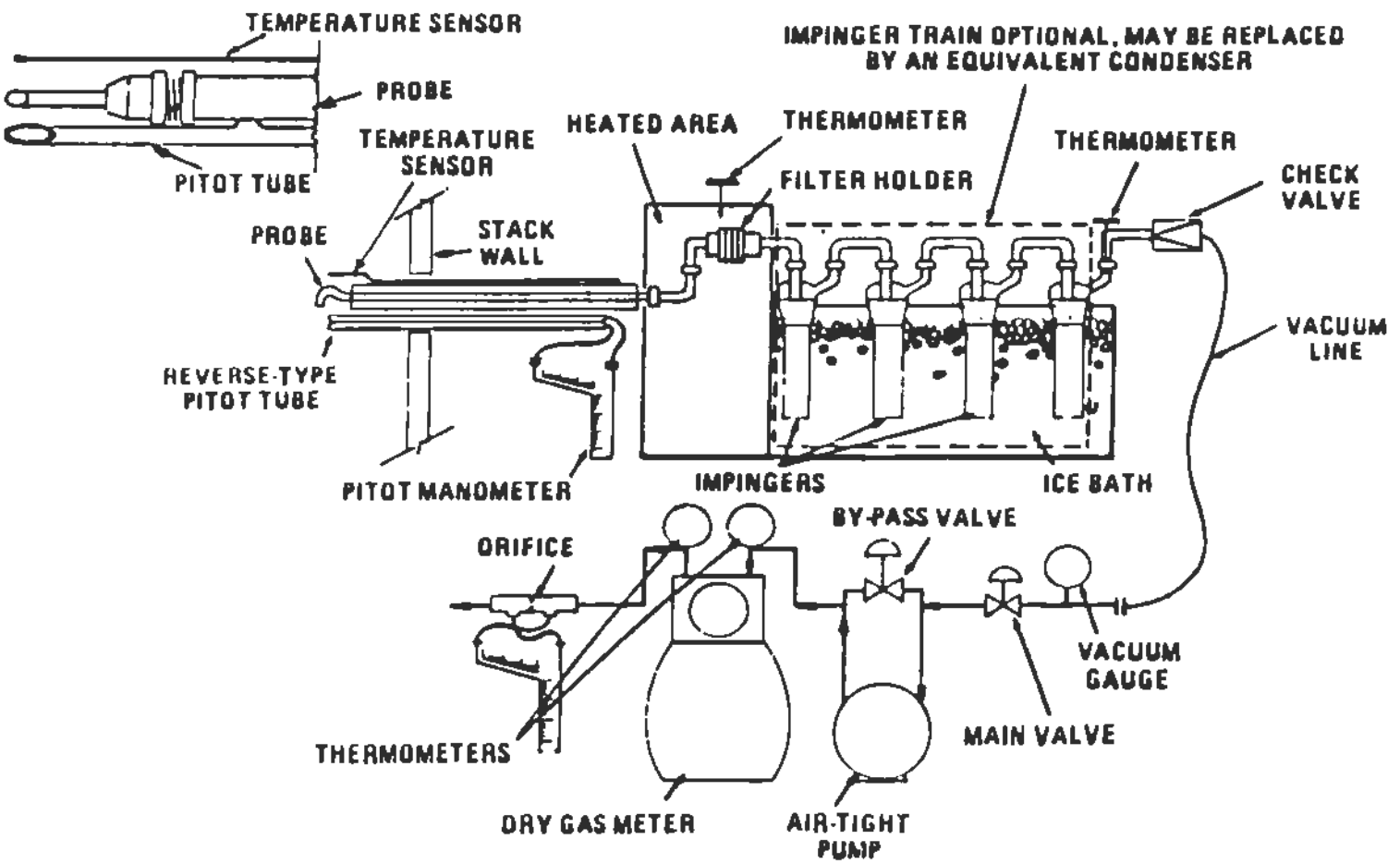
Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120 ± 14° C (248 ± 25° F). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

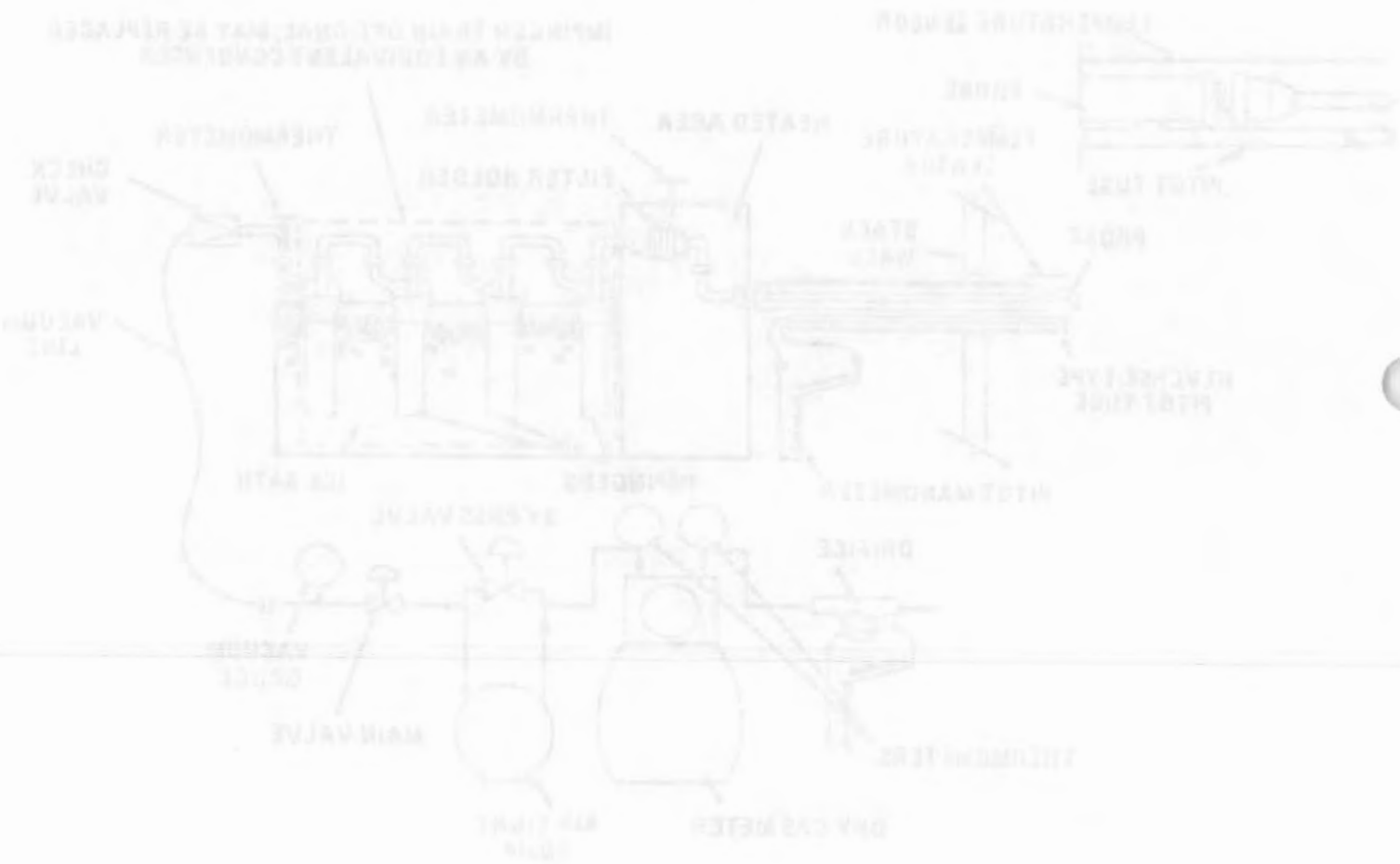
Description of Sampling Equipment

A schematic of the Method 5 sampling train is shown in **Figure G.6**. The sampling train consists of a stainless steel or glass nozzle; a glass probe liner with a heating system capable of maintaining the sample gas temperature at 248 ± 25° F; a type-S Pitot tube/dual manometer system measure stack gas velocity pressure; an in-stack thermocouple capable of measuring the stack temperature to within 3° F; a glass filter holder with a glass filter frit support housed in a filter heating system capable of maintaining a temperature of 248 ± 25° F; a moisture condenser; and a pump and metering system.

FIGURE G-6

METHOD 5 SAMPLING TRAIN





The condenser ensures that the volume measured by the dry gas meter is free of moisture and also allows for the calculation of the stack gas moisture content via Method 4. The condenser consists of four impingers connected in series with leak-free ground glass fittings.

The first impinger is a modified Greenburg-Smith impinger which is charged with 100 ml of water, and the second impinger is a standard Greenburg-Smith which is also charged with 100 ml of water. The third and forth impingers are modified Greenburg-Smith impingers. The third impinger is empty, and the fourth contains a known mass of silica gel as a final water trap and to protect the sample pump and meter. The metering system consists of a vacuum gauge, leak free pump, a dry gas meter capable of measuring volume to within 2%, and thermocouples capable of measuring the dry gas meter temperature to within 5.4° F.

Description of Operation

After the sampling location and minimum number of sampling points have been determined, the stack pressure and temperature and the range of velocity heads are measured, and the moisture content is determined or estimated based on knowledge of the process. A nozzle size is selected based on the range of velocity heads such that it is not necessary to change nozzles during a run to maintain isokinetic sampling rates, and the differential pressure gauge is checked to ensure that it is capable of measuring the range of velocity heads.

A total sampling time is selected such that the sampling time per point is not less than two minutes, and that the sample volume collected meets or exceeds the minimum required volume. During this trial burn, each M5 sampling run will be for approximately one-hour to collect a minimum volume of 30 ft^3. The portholes are cleaned to minimize the chance of sampling deposited material.

Prior to the start of a sampling run a pre-test leak check is conducted on the sampling train to ensure that ambient air will not dilute the stack gas sample during the sampling run. The sampling probe is capped off at the nozzle, the pump is activated, and the sampling train is evacuated to approximately fifteen inches of mercury vacuum. The sample train leakage rate is read from the dry gas meter and recorded. If the leakage rate is less than 0.02 cubic feet per minute, the sample train is considered acceptable for use.

At the beginning of each run, operational and run specific data, including the initial dry gas meter reading, are recorded at the top of the Particulate Field Data Sheet **(Figure G.4.)** The readings required on the data sheet are recorded at the end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted for any reason. The Pitot lines and manometer level are checked periodically to ensure accurate measurements of velocity head and sample flow rate.

To begin sampling, the probe nozzle cap is removed and the operating temperatures are verified. The probe is inserted into the stack to the first sampling point and the pump is immediately started. The sampling rate is adjusted rapidly to isokinetic conditions, and sampling is conducted for the previously determined time at each traverse point. During the sampling run the sampling rate is maintained within $\pm$ 10% of the true isokinetic sampling rate and the temperature of the probe and filter holder are maintained at 120 $\pm$ 14° C (248 $\pm$ 25° F). If the pressure drop across the filter becomes too large, making isokinetic sampling difficult to maintain, the filter may be changed. A leak check is performed before and after such changes.

At the end of the sampling run, the course adjust valve is closed and the probe is removed from the stack. The pump is turned off, a final dry gas meter reading is taken and a post-test leak check is performed. The post-test leak check is conducted in the same manner as the pre-test leak check except that the vacuum place on the system is equal to a slightly greater than the maximum vacuum observed during the sampling run. The maximum leakage rate allowed is 0.02 cfm. If the post-test leakage rate exceeds 0.02 cfm, the run is either invalidated, or a correction is made to the sample volume based on the recorded leakage rate.

Sample Train Pre-test Preparations

During the preparation and assembly of the sampling train, all openings where contamination may occur are kept covered until just prior to assembly or sampling. One hundred milliliters of distilled water are placed in each of the first two impingers and the contents of a tared silica gel bottle are transferred into the fourth impinger. Leak tight connections are made and secured between the impingers. Using tweezers or clean disposable surgical gloves, a tared filter is removed from its packaging and placed in the filter holder such that it is properly centered and the gasket prevents the gas stream from circumventing the filter. The nozzle is connected to the probe by a leak-free fitting and the probe is marked to indicate

the proper distance into the stack for each sampling point. The train is then assembled taking care to ensure leak-tight connections. Crushed ice is packed around the impingers.

Sample Recovery

When the probe is removed from the stack, it is allowed to cool. The nozzle is cleaned of all external particulate matter and capped to prevent contamination or loss of sample. After the final leak check, the sample train is disassembled and all openings are capped. The probe, filter assembly and impingers are removed to the cleanup area.

A 200 ml aliquot of the acetone used for cleanup is set aside as a blank. A 200 ml aliquot of the distilled water used in the impingers is also set aside as a blank if further analysis is required of the impinger catches.

The glass fiber filter is recovered from the filter holder by carefully removing the filter and returning it to its assigned petri dish. Any loose particle matter present in the front half of the filter holder is brushed onto the filter, and filter material that has adhered to the support frit is carefully scraped from the frit and placed into the petri dish. Stack particulate matter deposited in the sampling nozzle and probe during the run is quantitatively recovered by washing the interior of the liner and nozzle with acetone. The liner is brushed with a clean liner brush and the acetone rinse is collected into a labeled glass bottle. The front half of the filter holder is also washed with acetone and brushed, and the rinse is combined with the probe wash. The color of the indicating silica gel is examined to determine if it has been completely spent and a notation as to its condition made. The silica gel is weighed for moisture determination. A notation is made of any color or film in the liquid catch. The first three impingers and any condensate are weighed and recorded to within 1 mg using a top-loading scale. The impinger catch is then discarded unless further analysis is required.

Particle Weight Analysis

Beakers

Gloves are worn at all times when handling glassware. Glassware for residue analysis is purchased new and is rinsed with distilled, deionized water and oven dried at 105° C for 2 hours. The glassware is then placed in a desiccator to cool after which all beakers are

individually numbered, desiccated, and are weighed to a constant weight. Constant weight is defined as a difference of 0.5 mg or less between consecutive weighings made not less than 6 hours. apart. This weight is recorded as the Beaker Tare Weight. The beakers are stored in a closed cabinet in a constant humidity environment until use.

After the probe and filter holder rinses have been collected and returned to the laboratory, the samples are logged in the Sample Log Book. One tared beaker is used for each front half sample collected. The sample bottle and its contents are weighed on a Triple Beam Balance, and the mass recorded as Initial Bottle Weight. The wash is then poured into a tared beaker and the bottle rinsed with a known volume of acetone (usually 25 ml) from the same lot number as was used for the sample recovery in the field. If 25 ml is not sufficient, additional portions are used until all visible particulate material has been removed. The total lab wash volume (25 ml or more) is recorded. The sample bottle is reweighed and the result recorded as Final Bottle Weight. The Final Bottle Weight is subtracted from the Initial Bottle Weight to give the Difference, and the mass of acetone is determined by dividing the Difference by the density of the acetone.

The beaker containing the front half wash and bottle rinse is dried down by placing the beaker into a fume hood and allowing the acetone to completely evaporate. The dry beaker is placed into a desiccator for 24 hours and is then weighed to a constant weight. This weight is recorded in the Beaker sample log book as the Final Weight. The amount of particulate residue is calculated by subtracting Beaker Tare Weight from the Final Weight. A field solvent blank is also dried down to determine the particulate matter concentration in the acetone, and the sample weights are corrected based on the blank concentration.

The temperature and relative humidity in the weighing room are recorded during each weighing and controlled such that the temperature is 20 $\pm$ 5.6° C and relative humidity is less than 50 percent.

Filters

Petri dishes are purchased new and are washed with distilled, deionized water followed by acetone and are dried at 105° C for 1 hour. The petri dishes are allowed to cool, and are marked with a unique identifying code, to match the codes on the filters. One numbered filter is assigned to each numbered petri dish, and the combination is thereafter treated as a single unit.

The petri dish/filter units are desiccated for at least 24 hours, and are then weighed to constant weight (± 0.5 mg difference) at not less than 6 hour intervals. This weight is recorded as the petri dish/filter tare weight. The units are stored in a petri dish box which has the petri dish numbers clearly marked on the outside. After sampling, the units are returned to the laboratory, entered in the Filter sample log book and desiccated for at least 24 hours. The units are then weighed to constant weight at intervals of at least 6 hours. This is the unit final weight. Sample weight is calculated by subtraction of the unit weight from the unit final weight.

If no additional analyses are to be performed on the filters, the filter-petri dish set is returned to the petri dish box and stored for at least one year.

4.7 Continuous Gas Analyzers

4.7.1 O2 Concentration by Process Monitor

The O_2 continuous monitor has a range of 0 to 25 percent. The monitoring system has the capability to monitor O_2 and provide a record of the acquired data. O_2 analyzer output is also processed by a math function and used to correct CO concentration to 7 percent. The O_2 analyzer is a Beckman Industrial Model 755 O_2 analyzer.

The O_2 monitoring system is automatically calibrated once during each 24-hour period. For the trial burn, the system will be calibrated at the beginning of the day. A sampling system bias will be performed at the end of each sampling run.

4.7.2 NO_x Concentration by USEPA Method 7E

Summary of Method

Oxides of nitrogen compounds (NO_x) in a stack gas stream are measured continuously through the use of a Continuous Emission Monitor. The CEM principle of operation is based on the chemiluminescent reaction between nitric oxide (NO) and ozone (O_3). To measure NO concentrations, the sample gas is blended with O_3 in a heated flow reactor. Nitric oxide is oxidized to nitrous oxide (NO_2) by the following reaction:

$$NO + O_3 > NO_2 + O_2 + h\nu$$

Nitrous oxide produced by the reaction is in an excited state, and as the molecules return to ground state, light is emitted. The light emitted is monitored through an optical filter by a high-sensitivity photomultiplier tube positioned at one end of the reactor. The filter/photomultiplier responds to a narrow wavelength band of light, which reduces the effects of interfering compounds. The output of the photomultiplier tube is linearly proportional to the amount of NO present in the sample. NO_x concentrations are measured by passing the stack gas sample through a catalytic reduction chamber which reduces NO_x compounds to NO. The concentration of NO_x can be determined as the difference between the NO and NO_x response when the sample gas is passing through the converter.

Description of Sampling Equipment

The analyzer and sampling system used for this analysis meets the specifications outlined in 40 CFR Appendix A, Method 7E. Specifically, the analyzer calibration error is demonstrated to be less than two percent of the operating range for the zero, mid-range, and high range calibration gases; the sampling system bias is demonstrated to be within five percent of the analyzer calibration response to the zero and mid- or high-range calibration gases; and the zero and calibration drift are less than three percent of the operating range over the period of each sample run.

The NO_x analyzer used by Engineering-Science is a Thermo Electron Model 10AR Oxides of Nitrogen Analyzer. The instrument has normal operating ranges of 0-2.5, 10, 25, 100, 250, 1,000, 2,500, and 10,000 ppm. Calibration standards are chosen to be approximately fifty to sixty percent of span for the mid-range gas, and eighty to ninety percent for the high-range gas. Pre-purified air or nitrogen is used as a zero gas.

The sample extraction system consists of a heated stainless steel sampling probe which is outfitted with an in-stack sintered filter. Gas is extracted through the filter and probe assembly into a heated control box which contains the valves and connections to a heated sample line and an unheated calibration gas line. A three-way valve directs the flow of sample gas or calibration gas into the heated line. The heated line consists of an insulated and heated Teflon® tube through which sample gas is transported to the gas conditioner. The line is a self-limiting type which maintains in internal temperature of 250° F plus the ambient temperature. The gas conditioner consists of a PVC condensate trap and a condensing coil of Teflon® tubing immersed in an ice bath. The gas conditioner is connected to a leak-free

sample pump, and the outlet of the pump is connected to a vented manifold, from which gas is directed to the NO_x and other analyzers.

Instrument responses are read and recorded by a computer-driven data acquisition system. The system consists of an analog-to-digital signal converter, a personal computer, and a dot-matrix printer. The signal converter translates the analog instrument response to a digital signal, which the computer translates to the appropriate engineering units. The converted instrument responses are recorded on the computer's hard disk drive and on a paper printout generated by the printer.

Description of Operation

Prior to operation, a NO_x converter efficiency check is performed. After converter efficiency is established, the CEM system is connected to the effluent source and energized and allowed to stabilize, which takes approximately two hours. When the analyzer and sampling system have been determined to be ready for use, the analyzer is calibrated by introducing the zero, high-, and mid-range calibration standards, one at a time. The zero standard and the upscale standard closest to the stack pollutant concentration are then introduced into the sampling system at the heated control box via the calibration gas line to determine the sampling system bias. If all calibrations are within the specifications described above, sampling may begin. A sampling run lasts for the duration of other sampling methods being conducted during each test condition (i.e., a one-hour M5 sample run). At the conclusion of the sampling run, the zero and upscale calibration standards are again introduced into the sampling system to determine the magnitude of the calibration drift during the run. If the magnitude of the calibration drift exceeds three percent, the analyzer and sampling system are re-calibrated before additional runs are conducted. If the instrument response at the end of the run exceeds the bias specification of five percent, the run is invalidated, and the system is recalibrated before repeating the sampling run. During each day of testing, a sampling system bias will be conducted at the conclusion of each POHC, particle, or metals sampling run.

Description of Detection Limits

The detection limit of the measurement system is typically two percent of the instrument span range. For an operating range of 250 ppm, the instrument detection limit is approximately 5 ppm.

Calibration Standards

Calibration standards are chosen such that the instrument can be calibrated on the operating range which most closely corresponds to the average stack gas concentration. Nitric oxide of varying concentrations in nitrogen is used as the calibrant gas, and is prepared by the manufacturer according to EPA Revised Traceability Protocol-1. The zero gas consists of zero grade air or nitrogen.

4.7.3 Carbon Monoxide Concentration by Process Monitor

The CO continuous monitor is a dual range monitor with a low range of 0-200 ppm and a high range of 0-3000 ppm. The CO monitoring system will provide a record of the data. CO concentrations will be corrected to 7 percent O_2. The CO analyzer is a Beckman Industrial Model 880 Nondispersive Infrared (NDIR) Analyzer.

The CO monitoring system is automatically calibrated once during every 24-hour period. During the trial burn, the system will be calibrated at the beginning of each day. Sampling system bias checks will be conducted at the conclusion of each extractive sampling run.

4.7.4 Total Hydrocarbon Concentration by USEPA Method 25A

Summary of Method

Total hydrocarbons in a stack gas stream are measured continuously through the use of a Continuous Emission Monitor. The CEM contains a flame ionization detector, which measures the concentration of the hydrocarbons present in the stack sample. A portion of the stack gas sample is burned in a hydrogen flame which ionizes the hydrocarbons present in the sample. The flame is situated between two high-voltage plates which generate an electric field across the flame. As the hydrocarbon ions are generated, they are attracted to the appropriately charged plate, which results in the generation of a current across the electrode gap. The magnitude of the current is directly proportional to the concentration of the sample introduced to the FID.

Description of Sampling Equipment

The analyzer and sampling system used for this analysis meets the specifications outlined in 40 CFR Appendix A, Method 25A. Specifically, the analyzer calibration error is demonstrated to be less than five percent of the respective calibration gas value for the zero, low-, mid-, and high-range calibration gases; and the zero and calibration drift are less than three percent of the operating range over the period of each sample run.

The hydrocarbon analyzer used by Engineering-Science is the J.U.M. Total Hydrocarbon Gas Analyzer which uses a heated Flame Ionization Detector (FID). The J.U.M. has ranges full-scale operating ranges of 0 - 10, 100, 1,000, 10,000, and 100,000 ppm. The analyzer is typically calibrated with propane, so the results are reported on a propane-equivalent basis. Calibration standards are chosen to be approximately twenty-five to thirty-five percent of the operating range for the low-range gas, forty-five to fifty-five percent for the mid-range gas, and eighty to ninety percent for the high-range gas. Pre-purified air or nitrogen is used as a zero gas.

The sample extraction system consists of a heated stainless steel sampling probe which is outfitted with an in-stack sintered filter. Gas is extracted through the filter and probe assembly into a heated control box which contains the valves and connections to a heated sample line and an unheated calibration gas line. A three-way valve directs the flow of sample gas or calibration gas into the heated line. The heated line consists of an insulated and heated Teflon® tube through which sample gas is transported directly to the hydrocarbon analyzer. The line is a self-limiting type which maintains in internal temperature of 250° F plus the ambient temperature. A small heated-head pump located in the analyzer draws the sample from the stack, through the sample line and into the analyzer. A portion of the stack gas sample is introduced into the FID chamber by the use of a back-pressure regulator.

Instrument responses are read and recorded by a computer-driven data acquisition system. The system consists of an analog-to-digital signal converter, a personal computer, and a dot-matrix printer. The signal converter translates the analog instrument response to a digital signal, which the computer translates to the appropriate engineering units. The converted instrument responses are recorded on the computer's hard disk drive and on a paper printout generated by the printer.

Description of Operation

The CEM system is connected to the effluent source and energized and allowed to stabilize, which takes approximately two hours. When the analyzer and sampling system have been determined to be ready for use, the sampling system is calibrated by introducing the zero-, high-, mid-, and low-range calibration standards into the sampling system at the probe, one at a time. If all calibrations are within the specifications described above, sampling may begin. A sampling run generally lasts for one hour, but may be longer if conducted in conjunction with other testing. At the conclusion of the sampling run, the zero and mid-range calibration standards are again introduced into the sampling system to determine the magnitude of the calibration drift during the run. If the magnitude of the calibration drift exceeds three percent, the sampling system is re-calibrated before additional runs are conducted. The run is either invalidated, or the results of the run are corrected based on the calibration data observed after the run, and both sets of results are reported.

Description of Detection Limits

The analyzer manufacturer reports a analytical sensitivity of 1 ppm total hydrocarbons. When the sample extraction system is attached to the analyzer the detection limit rises to approximately two percent of the analyzer operating range. This corresponds to a detection limit of approximately 2 ppm when the instrument is operated on the 0 - 100 ppm range.

4.8 METHOD 23

Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans by USEPA Method 23.

Summary of Method

Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in multicomponent sampling train. Particles that condense at or above a temperature of 120 ± 14° C (248 ± 25° F) are collected on a heated glass fiber filter, and particles and gases not collected on the filter are trapped onto a packed column of XAD-2 resin. Uncombined moisture is collected in a series of chilled glass impingers. The sample fractions are recovered and analyzed using high resolution

chromatography and high resolution mass spectrometry to determine the concentrations of target analytes collected. The method is applicable for the collection of polychlorinated dibenzo-p-dioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's) from stationary sources.

Description of Sampling Equipment

The Method 23 sampling train is based on the EPA Method 5 sampling train, but includes added components for the capture of PCDD's and PCDF's **(Figure G.3)**. The sampling train consists of a nickel, nickel-plated stainless steel or glass nozzle; a glass probe liner with a heating system capable of maintaining the sample gas temperature at 248 ± 25° F; a type-S Pitot tube/dual manometer system to measure stack gas velocity pressure; an in-stack thermocouple capable of measuring the stack temperature to within 3° F; a glass filter holder with a glass filter frit support housed in a filter heating system capable of maintaining a temperature of 248 ± 25° F; an organic sampling module; a moisture condenser; and a pump and metering system.

The organic sampling module consists of three components, the first of which is a water-cooled gas conditioner. The gas conditioner consists of a condenser coil which is surrounded by a water jacket through which ice water is continuously circulated. The condenser is designed to ensure that the temperature of the sample gas leaving the conditioner does not exceed 20° C (68° F). The second component of the organic sampling module is a sorbent trap which is sized to contain approximately 20 g of the porous polymeric resin. The sorbent trap is outfitted with a water jacket through which ice water is continuously circulated, such that the internal gas temperature is maintained at a temperature of 17 ± 3° C (62.5 ± 5.4° F). The sorbent module is outfitted with a thermocouple well into which a thermocouple is inserted to monitor the internal gas temperature. The last section of the organic module consists of a moisture knock-out trap, which is simply an impinger with a shortened stem, so that the sample gas cannot bubble through the collected condensate during sampling.

The moisture condenser ensures that the volume measured by the dry gas meter is free of moisture and also allows for the calculation of the stack gas moisture content via Method 4. The condenser consists of four impingers connected in series with leak-free ground glass fittings. The first impinger is a modified Greenburg-Smith impinger which charged with 100

ml of HPLC water, and the second impinger is a standard Greenburg-Smith which is also charged with 100 ml of HPLC water. The third and forth impingers are modified Greenburg-Smith impingers. The third impinger is empty, and the fourth contains a known mass of silica gel as a final water trap and to protect the sample pump and meter. The metering system consists of a vacuum gauge, leak free pump, a dry gas meter accurate to within 2%, and thermocouples capable of measuring the dry gas meter temperature to within 5.4° F.

Description of Operation

After the sampling location and minimum number of sampling points have been determined, the stack pressure and temperature and the range of velocity heads are measured, and the moisture content is determined or estimated based on knowledge of the process. A nozzle size is selected based on the range of velocity heads such that it is not necessary to change nozzles during a run to maintain isokinetic sampling rates, and the differential pressure gauge is checked to ensure that it is capable of measuring the range of velocity heads.

A total sampling time is selected such that the sampling time per point is not less than two minutes, and that the sample volume collected meets or exceeds the minimum required volume. This volume is based on the expected concentration of the semi-volatile species present in the exhaust stream and the detection limit of the analytical method that will be used. A sample volume of three dry standard cubic meters (105.9 dscf) is typical. The portholes are cleaned to minimize the chance of sampling deposited material.

After the sampling train has been assembled, the filter and probe heating system are activated the allowed to stabilized at the desired operating temperatures. The sampling train is plugged off at the nozzle and a vacuum of fifteen inches Hg is pulled on the train. Leakage rates in excess of four percent of the average sampling rate or 0.02 cfm, whichever is less, are unacceptable. Silicone stopcock grease, which may be used on a Method 5 sampling train, is not allowed on the Modified Method 5 due to the possibility of analytical interference with the target analytes. If it is necessary to stop a sampling run during a test to replace a component of the sampling train, a leak check is conducted at the highest system vacuum observed up to that point in the test before the sampling train is disassembled. Before resumption of the sampling, a leak check similar to the pre-test leak check is conducted.

At the beginning of each run, operational and run specific data, including the initial dry gas meter reading, are recorded at the top of the Field Data Sheet. The readings required on the data sheet are recorded at the end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted for any reason. The Pitot lines and manometer level are checked periodically to ensure accurate measurements of velocity head and sample flow rate.

To begin sampling, the probe nozzle cap is removed and the operating temperatures are verified. The probe is inserted into the stack to the first sampling point and the pump is immediately started. The sampling rate is adjusted rapidly to isokinetic conditions, and sampling is conducted for the previously determined time at each traverse point. During the sampling run the sampling rate is maintained within $\pm$ 10% of the true isokinetic sampling rate and the temperature of the probe and filter holder are maintained at 120 $\pm$ 14° C (248 $\pm$ 25° F). If the pressure drop across the filter becomes too large, making isokinetic sampling difficult to maintain, the filter may be changed. A leak check is performed before and after such changes. At the end of the sampling run, the course adjust valve is closed and the probe is removed from the stack. The pump is turned off, a final dry gas meter reading is taken and a post-test leak check is performed. The post-test leak check is conducted in the same manner as the pre-test leak check except that the vacuum place on the system is equal to a slightly greater than the maximum vacuum observed during the sampling run. The maximum leakage rate allowed is 0.02 cfm. If the post-test leakage rate exceeds 0.02 cfm, the run is invalidated.

Sample Recovery

When the probe is removed from the stack, it is allowed to cool. The nozzle is cleaned of all external particulate matter and capped to prevent contamination or loss of sample. After the final leak check, the sample train is disassembled and all openings are capped. The probe, filter assembly, organic module, and the impingers are removed to the cleanup area.

At the sample clean-up area, the contents of the sampling train components are quantitatively recovered into glass storage containers and logged into the field sample log.

Container No. 1 consists of the glass fiber filter/petri dish set. The exposed filter is removed from the filter holder using tweezers and carefully transferred to its original petri dish. Any

loose particles that are present in the front-half of the filter holder are carefully brushed into the petri dish, as is filter material that may be attached to the filter frit. The petri dish is then sealed by wrapping with Teflon® tape.

The interior surfaces of the nozzle, probe, cyclone (or cyclone bypass) and front half of the filter holder are washed three times with acetone into a pre-cleaned glass storage bottle, which comprises Container No. 2. The probe liner is brushed with a nylon brush and rinsed three time with acetone until no visible particles appear in the wash, then the liner is rinsed three times with the methylene chloride. The rinses are collected on Container No. 2. The back-half of the filter holder and the gas condenser are each rinsed three times with acetone, and the rinses added to Container No. 2. The back-half of the filter holder and the gas condenser are then each soaked three times each with separate portions of methylene chloride. The duration of each soak is five minutes. The methylene chloride is added to the contents of Container No. 2. A sample label is filled out and attached to the outside of the bottle, the liquid level is marked, and the lid is wrapped with Teflon® tape.

Container No. 3 consists of a soak of the back-half of the filter holder and the gas condenser with toluene. The back-half of the filter holder and the gas condenser are each soaked for five minutes three times with separated portions of toluene, and the rinses are collected in a pre-cleaned glass bottle. A sample label is filled out, attached to the outside of the bottle and the liquid level is marked. The bottle cap is then wrapped with Teflon® tape.

Container No. 4 consists of the sorbent trap section of the organic sampling module. The sorbent trap is removed from the sampling train and tightly capped using ground glass fittings and impinger clamps. The trap is then wrapped in aluminum foil, labeled, and placed inside of a leak-proof bag. The trap is then stored on ice for shipment to the analytical laboratory. The condensate and the knock-out impinger is weighed using a top-loading balance accurate to the nearest milligram. The weight is recorded and the condensate is then transferred to a pre-cleaned glass sample bottle, which comprises Container No. 5. A sample label is filled out and attached to the outside of the sample bottle, the liquid level is marked and the cap is wrapped in Teflon® tape.

The three impingers (and their contents) behind the condensate knockout are weighed separately and the weights are recorded. The impinger catch is for the determination of moisture content in the sample gas stream only, and is discarded after the liquid volumes are

measured. The silica gel impinger and its contents are weighed together and subtracted from the initial weight of the impinger and its contents. A note indicating the color and condition of the silica gel is made on the sample recovery data sheet to record whether moisture break-through has occurred.

Sample Train Pre-test Preparations
Glassware Cleaning Protocol

All glass components upstream and including the adsorbent module as well as glass petri dishes for filter storage and transport are cleaned based on the protocol described in Section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." When cleaning glassware, special care is given to the removal of grease sealant from the components. The glassware cleaning procedure consists of eight steps outlined below:

1. Acetone rinse with reagent-grade acetone.
2. Four-hour hot detergent soak using Alconox or other suitable glassware cleaning detergent.
3. Hot tap water rinse.
4. Four-hour chromic acid soak. Alternatively, a 2 percent v/v solution of FL 70 concentrate from Fisher Scientific can be used.
5. Hot tap water rinse.
6. Distilled, deionized water rinse.
7. Acetone rinse with pesticide-grade acetone.
8. Methylene chloride rinse with pesticide-grade methylene chloride.

After the glassware has been allowed to air dry, all openings are sealed with heavy-duty aluminum foil that has been previously rinsed with pesticide-grade hexane.

Filter Preparation Protocol

Glass fiber filters without organic binder which demonstrate a capture efficiency of at least 99.95 percent of 0.3-micron dioctyl phthalate smoke particles, will be used. Filters are examined for pinhole leaks by holding them up to the light. Filters are cleaned before use by extraction in a Soxhlet extraction apparatus once with pesticide grade toluene for three

hours and repeated with toluene for 16 hours. The filters are removed from the apparatus and allowed to cool, and are dried under a clean stream of nitrogen. The filters are then stored in cleaned petri dishes and sealed with Teflon® tape.

XAD-2 Resin Preparation Protocol

XAD-2 adsorbing resin used in the sorbent trap must be carefully extracted and dried prior to its use in the field, whether the resin used has been purchased new or is being recycled. The resin preparation involves repeated extractions with Type II distilled, deionized water, and pesticide grade methyl alcohol, methylene chloride, and toluene. Extractions may be conducted using a giant Soxhlet extractor or a continuous extractor which has been constructed according to specifications outlined in "Test Methods for Evaluating Solid Waste, Physical Chemical Methods, Field Manual, Volume II SW-846", Part III, Chapter 10, Method 0010, Appendix A, "Preparation of XAD-2 Sorbent Resin." Initially, the resin is rinsed in a beaker with Type II water, then the resin is extracted for 8 hours with Type II water. This is followed by a 22-hour extraction with methyl alcohol, a 22-hour extraction with methylene chloride, and a 22-hour extraction with toluene. After the extractions, the cleaned XAD-2 resin is dried using the fluidized bed technique hy gently passing a stream of nitrogen through a bed of XAD-2 resin.

Before its use in the field, the prepared XAD-2 resin is subjected to a quality assurance check to evaluate its acceptability for use. The quality control results must be reported for each bath of resin extracted. A 1.0 mg sample of the dried resin is weighed out and transferred to a small vial, and 3 ml of toluene is added. The vial is capped and shaken. The sample resin is extracted and then a 2 μl sample of the extract injected into a gas chromatograph for analysis. The results of the analysis are compared against the results of a reference solution which consists of a mixture of 2.5 μl of methylene chloride and 100 ml of toluene. The maximum concentration of methylene chloride allowed is 1000 μg per gram of resin. If this limit is exceeded the resin must be further dried until the solvent concentration is acceptable. The cleaned resin has a shelf-life of four weeks when stored in an amber glass container with a Teflon®-lined cap.

Description of Sample Analysis

The sample train fractions are extracted, spiked with surrogate standards, and concentrated to volumes of approximately 1.0 ml prior to analysis by Gas Chromatography/Mass Spectrometry (GC/MS). GC/MS with fused-silica capillary columns is the primary analytical tool for the measurement of PCDD and PCDF emissions from hazardous waste incinerators. Prescreening of the sample extracts is often conducted using GC/FID (flame ionization detector) or GC/ECD (electron capture detector), to determine the analytes present in the sample and their approximate concentrations in the sample extract.

4.9 METALS

Determination of Metals Emissions by BIF Metals Method

Summary of Method

Particulate and gaseous metal emissions are withdrawn isokinetically from a source and collected on a heated filter, and in a series of chilled impingers containing a solution of dilute nitric acid combined with dilute hydrogen peroxide in each of two impingers. Sampling train components are recovered and digested in separated front- and back-half fractions. Materials collected in the sampling train are digested with acid solutions to dissolve inorganics and to remove organic constituents that may create analytical interferences.

Description of Sampling Equipment

The multi-metals sampling train is similar to the Method 5 train with a few exceptions. A schematic of the sampling train is shown in **Figure G.6** (same as Method 5 train). The sampling train consists of a stainless steel or glass nozzle; a glass probe liner with a heating system capable of maintaining the sample gas temperature at 248 $\pm$ 25° F; a type-S Pitot tube to determine velocity pressure in order to calculate stack gas velocity and volumetric flow; a dual inclined manometer for measuring the velocity pressure and orifice differential pressure; a glass filter holder with a glass filter frit support to support the particle filter; a filter heating system capable of maintaining the sample gas temperature at 248 $\pm$ 25° F; and a temperature gauge capable of measuring the temperature to within 3° F. Also included in the sampling train is a moisture condenser which is necessary to determine the stack gas

moisture content. Four to seven impingers are connected in series with leak-free ground glass fittings. The first impinger may be excluded if the amount of moisture collected during the sampling run is expected to be less than 100 ml. This impinger is a short-stem modified Greenburg-Smith, which is used as a water knock-out. The second and third impingers contain acidified peroxide absorbing solution. The second impinger is a modified Greenburg-Smith and the third is a conventional Greenburg-Smith. The fourth impinger is an empty modified Greenburg-Smith impingers. Impinger five and six are both conventional Greenburg-Smith impingers and contain acidified permanganate solution for the absorption of mercury. These impingers are optional if mercury is not a target metal. Since mercury is not a target metal, these two impingers will not be used. The seventh and final impinger is a modified Greenburg-Smith impinger filled with approximately 200 grams of silica gel to provide additional moisture collection capacity and to protect the sample pump. The metering system consists of a vacuum gauge, leak free pump, thermometers capable of measuring temperatures to within 5.4° F, and a dry gas meter capable of measuring volume to within 2%.

The filters used in the determination of metals emissions are certified by the supplier to contain less than 1.25 μg of each of the metals to be measured. Quartz fiber or glass fiber filters without organic binders are used, and exhibit at least 99.95 percent efficiency (<0.05% penetration) on 0.3 micron dioctyl phthalate smoke particles. Pallflex type 2500 QAT-UP Ultra Pure Filters (or their equivalent) are used in the method.

Sample Train Preparation

All glassware to be used in the sampling program is initially rinsed with hot tap water and then washed in hot soapy water. Glassware is then be rinsed three times with tap water, followed by three rinses with distilled water. The glassware is soaked for a minimum of four hours in a solution of 10 percent (V/V) nitric acid, rinsed three times with deionized water, and a final time with acetone, and allowed to air dry. Prior to assembly, all openings where contamination can occur shall be covered.

During the preparation and assembly of the sampling train, all openings where contamination may enter are kept covered until just prior to assembly or until immediately before sampling. The first impinger, used as a water knockout, is left empty. The next two impingers are charged with 100 ml each of HNO_3/H_2O_2 absorbing solution each, the fourth impinger is left

empty, and the fifth and sixth impingers are each charged with 100 ml of acidic permanganate solution. The seventh impinger contains approximately 200 g of indicating type silica gel. Leak-tight connections are made and secured between the impingers. Using tweezers or clean disposable surgical gloves, a tared filter is removed from its petri dish and placed in the filter holder such that it is properly centered and the gasket prevents the gas stream from circumventing the filter. The nozzle is connected to the probe by a leak-free fitting and the probe is marked to indicate the proper distance into the stack for each sampling point. Ice is packed around the impingers at the start of sampling.

Pretest Preparation

After the sampling site and the minimum number of sampling points is determined, the stack pressure and temperature and the range of velocity heads are measured, and the moisture content and dry gas molecular weight are determined or estimated based on knowledge of the process. A nozzle is selected based on the range of velocity heads such that it is not necessary to change nozzles during a test to maintain isokinetic sampling rates, and the differential pressure gauge is checked to ensure that it is suitable for measuring the range of velocity heads.

The sampling time is selected in order to collect a sample volume of at least 30 dry standard cubic feet (dscf). Sample periods are approximately one hour but are chosen such that the sampling time per sample point is not less than two minutes.

Sample Train Operation

During the sampling runs, the sampling rate is maintained within $\pm$ 10% of the true isokinetic sampling rate and the temperature of the probe and filter holder are maintained at 248 $\pm$ 25° F.

At the beginning of each run, the Field Data Sheet (Figure G.4) is filled out and site and test specific data is recorded, including the initial dry gas meter reading. During testing, readings are recorded on the data sheet at the end of each sampling point, when changes in flow rates are made, before and after each leak-check, and when sampling is halted for any reason. The manometer lines are zeroed and are checked periodically to ensure proper measurement of the velocity head.

To begin sampling, the probe nozzle cap is removed and the operating temperatures are verified. The probe is inserted to the proper depth and the pump is immediately started. The flow rate is adjusted rapidly to isokinetic conditions. Sampling is then conducted for the pre- determined time at each traverse point. If the pressure drop across the filter becomes too large, making isokinetic sampling difficult to maintain, the filter may be changed. A leak check is performed before and after such changes.

At the end of the sampling run, the course adjust valve is closed and the probe is removed from the stack. The pump is turned off, a final dry gas meter reading is taken and a post-test leak check is performed.

Sample Recovery

When the probe is removed from the stack, it is allowed to cool until it may be safely handled, and the nozzle is cleaned of all external particulate matter and loosely capped to prevent contamination or loss of sample. After the post-test leak check, the sample train is disassembled and all openings are capped. The probe, filter assembly and impingers are removed to the cleanup area.

The filter is carefully removed from the filter holder with tweezers or clean, disposable surgical gloves, and returned to its original petri dish. Any particles observed in the front half of the filter holder are brushed onto the filter in the filter container, and filter material stuck to the filter frit is scraped off and placed into the petri dish along with the filter.
Taking care to prevent contamination, particulate matter, including condensates, is quantitatively recovered from the probe nozzle, glass liner and front half of the filter holder by washing these parts with acetone and placing the wash in a labeled glass container. This step is deleted if the determination of particle emissions is not required. Brushes are used to facilitate the washing process.

The sampling nozzle, probe liner, and front half of the probe liner is then rinsed with a 100 ml of 0.1 N nitric acid into a glass container. It is important to use exactly 100 ml of the nitric acid solution because of blank corrections which are made later during analysis. The nozzle, probe liner, and front-half of the filter holder are once again rinsed with acetone, which is discarded.

The first three impingers and their contents are weighed, and the weights are recorded in the sample clean-up data sheet. This information is required to calculate the moisture content of the sampled flue gas. The filter support, the back-half of the filter holder and the first three impingers are rinsed with exactly 100 ml of 0.1 N nitric acid. The rinses and collected impinger solutions are combined into a single sample bottle and the liquid level is marked. The container is sealed and a label which clearly describes the contents is attached.

The silica gel is weighed and the mass recorded on the clean-up sheet (Figure 6-6-3). The silica gel is transferred from its impinger to the original container and sealed.

Once during each field test, reagent blanks of the solutions used are collected by transferring 100 ml of each solution into labeled containers. The containers are sealed and treated as samples henceforth. An unused filter is also collected as a blank and returned to the laboratory. Blank samples are analyzed to determine if contamination occurred during the field testing.

Description of Leak Check Procedures

With the system completely assembled, the nozzle is capped and the vacuum pump turned on. The coarse adjust valve is opened and the fine adjust valve is set to yield 15 inches of mercury vacuum. The leak rate is read from the dry gas meter. The maximum acceptable leak rate is 0.020 cfm at fifteen inches of mercury vacuum. At the conclusion of the test, a leak check is again conducted at the maximum vacuum observed during the test. If the leakage rate exceeds 0.020 cfm, the run is invalidated.

4.10 DEACTIVATIONFURNACE FLY ASH

After each test run, a sample of the fly ash will be sampled from each heat exchanger, the cyclone, and the baghouse. Each ash hopper will be sampled with a trowel as per the Trowel Method S007 from SW-846. Fly ash will be collected at the end of each run, and the ash from three runs will be composited into one sample. The sample will be well mixed and then a representative sample will be collected for the test condition. The composited sample will be analyzed for the POHC or metal of interest using the methods shown on Table G-5. Also, the toxicity characteristic leaching procedure (TCLP) will be run on the fly ash (SW-846 Method 1311) for metals.

SECTION 5
HANDLING, TRACEABILITY, AND HOLDING TIMES

The primary objective of this procedure is to create an accurate written record that can be used to trace the possession of a sample from the moment it is collected through to the final report. A sample is in custody if it is in any one of the following states:

- In actual physical possession.
- In view, after being in physical possession.
- In physical possession and locked up so that no one can tamper with it.
- In a secured area, restricted except to authorized personnel.
- In transit.

5.1 GENERAL REQUIREMENTS

- Name of sample custodian(s).
- Laboratory tracing report sheets to be used which identify:
  a. Sample code number, reserve sample quantity, aliquot for each test, responsible person, date received, date completed,
  b. Storage facility for reserve samples,
  c. Method for using hardbound workbooks in conjunction with lab tracking report sheets to note unusual events,
  d. Quality control inspection results on incoming samples, and
  e. Method of identifying sample at any stage of testing, using existing laboratory practices.

5.2 FIELD CUSTODY

Appropriate personnel will receive copies of the test plan prior to testing. Pre-test briefings will be held to apprise the participants of test objectives, sample locations, and chain-of-custody procedures. After samples are collected, a debriefing is held in the field to verify adherence to the chain-of-custody procedures and determine whether additional samples are required.

The field test team will be familiar with the following rules:

- Involve a minimum number of persons in sample collection and handling.
- Establish procedure guidelines to be used for each type of sample collected, preserved, and handled.
- Obtain samples using the appropriate sampling techniques.
- Attach sample tag or label securely to the sample container at the time the sample is collected. Tags will be completed legibly in waterproof ink. The tag will contain the following items as a minimum: the serial number of the tag, the station number and location, the date and time, the type of sample, the sequence number (if any), the analyses required, and the name of the sample collector **(see Figure G.7)**
- Attach a custody seal to each sample to protect against tampering **(Figure G-8)**.
- Use bound sample logbooks to record sample identifications and other pertinent information necessary to reconstruct the sample collection processes. Store the sample logbooks in a safe place where they are protected and accounted for at all times.
- A field data sheet with a standard format to minimize field entries will be used. The sample location, date, time, run number, type of samples taken, volume of each sample, measurements, and any other pertinent information or observation will be included on each field data sheet. The Field Team Leader will be responsible for the safe keeping of all field data sheets. The entries should be signed by the sample collector and reviewed by the Field Team Leader.
- The sample custodian is responsible for the care and custody of the samples until they are properly dispatched to the receiving laboratory or given to an assigned custodian. The sample custodian will ensure that each container is in his/her physical possession or in his/her view at all times, or stored in a locked place where it cannot be tampered with.

In the transfer-of-custody procedures, each custodian or sampler will sign, record, and date the transfer. Sample transfer can be a sample-by-sample basis or on a bulk basis. The following protocol will be followed for all samples as they are collected and prepared for distribution.

- Samples will be accompanied by a chain-of-custody record and analysis request form **(Figure G.9)** that includes the name of the study, collectors' signatures, station number, station location, date, time, type of sample, sequence number, number of

FIGURE G.7
SAMPLING LABEL

ENGINEERING-SCIENCE SAMPLE IDENTIFICATION LABEL

ENGINEERING-SCIENCE SAMPLE I.D.

10521 Rosehaven Street, Fairfax, Virginia 22030

Sample No.: _____

Plant: _____

Run No.: _____ Date: _____

Recovered by: _____ Job No.: _____

Sample Description: _____

Vol.: _____

Analyze for: _____

Comments/Instructions: _____

FIGURE G.8
CHAIN OF CUSTODY SEAL EXAMPLE

FIGURE G.9

CHAIN OF CUSTODY RECORD

ENGINEERING-SCIENCE **CHAIN OF CUSTODY RECORD**

PROJECT NO.	PROJECT NAME	ANALYSES
SAMPLERS: *(Name/Signature)*		

FIELD NUMBER:	COLLECTION DATE	COLLECTION TIME	SAMPLE NAME	NO. OF BOTTLES							COMMENTS (TYPE OF CONTAINER, SPECIAL PRESERVATION, SPECIAL HANDLING, ETC.)

Relinquished by: *(Name/Signature)*	Date/Time	Received by: *(Name/Signature)*	Date/Time	Carrier: *(In person, Fed X, UPS, etc)*
Relinquished by: *(Name/Signature)*	Date/Time	Received by: *(Name/Signature)*	Date/Time	Carrier: *(In person, Fed X, UPS, etc)*
Relinquished by: *(Name/Signature)*	Date/Time	Received by: *(Name/Signature)*	Date/Time	Carrier: *(In person, Fed X, UPS, etc)*

General Comments:

Distribution: Original: Accompanies Samples (Return to Originator), Yellow Copy: Field Crew, Pink Copy: Laboratory Files

containers, and analyses required. When turning over possession of samples, the transferor and transferee will sign, date, and time the record sheet.

- The field custodian or field sampler has the responsibility of packaging and dispatching samples to the laboratory for analysis. The appropriate chain-of-custody record must be filled out, dated, signed, and included with the sample. A copy will remain with the custodian.
- Packages must be accompanied by the chain-of-custody record which identifies the contents. The original must accompany the shipment. The field custodian will retain a copy.
- To avoid breakage, samples will be carefully packed in shipment containers such as ice chests or D.O.T.-approved shipping containers. The shipping containers will be sealed for shipment to the receiving laboratory. Samples will be shipped on the day of collection, if possible. If not, they will be shipped as soon as possible thereafter.
- If sent by mail, the package will be registered mail, with return receipt requested. If sent by common carrier, a bill of lading will be obtained. Receipts from post offices and bills of lading will be retained as part of the permanent chain-of-custody documentation.
- If samples are delivered to the laboratory when appropriate personnel are not there to receive them, the samples will be locked in a designated area within the laboratory or must be placed in a secure area by the recipient. The recipient is responsible for unlocking the samples and delivering custody to the appropriate custodian.

5.3 LABORATORY CUSTODY

The protocol listed below will be followed for all samples received at the laboratories.

- The laboratory shall designate a sample custodian and an alternate custodian to act in his absence. In addition, the laboratory will set aside a sample storage security area.
- Samples will be handled by a minimum number of persons.
- Incoming samples will be received by the custodian or the alternate. The custodian will sign the chain-of-custody records and retain them as a permanent record. Couriers picking up the samples shall sign jointly with the laboratory custodian.
- Immediately upon receipt, the samples are cross-checked with the chain-of-custody record to ensure the proper number of samples were received and that they correspond to the sample descriptions. Samples are also checked for damage and/or leaks. All abnormalities are documented.

- The custodian will ensure that the samples are logged into the laboratory "master" sample log **(see Figure G.10)**.
- The custodian will distribute the samples to the personnel performing the tests.
- The analyst will record in his laboratory notebook or analytical worksheet information describing the sample, the procedures performed, and the test results. The notes will be signed and dated, and will include abnormalities that occurred during the testing procedure. The notes will be retained as permanent record in the laboratory.
- Laboratory personnel are responsible for the care and custody of a sample once it is handed to them. It should be in their possession or secured in the laboratory at all times. Sample preparation forms will be drafted for each sample and include:
  a. blank determinations for all reagents which become an integral part of the sample
  b. clean-up reagent blank determination
  c. glassware blank determination

The laboratory area shall be maintained as a secured area and shall be restricted to authorized personnel only.

- Once the analyses are completed, any unused portion of the sample, together with identifying labels will be returned to the custodian. The sample will be retained in the custody room until permission to destroy the sample is received.
- Samples will be destroyed only upon the order of the Program Manager or when the samples have deteriorated.
- **Figure G.11** presents the chain-of-custody flow for samples from initial sampling to the reporting of results.

5.4 SAMPLE PRESERVATION AND HOLDING TIMES

Table G-6 shows the sample preservation procedures and the applicable holding times for each sample collected. After each sample has been recovered and entered into the field sampling logbook, it is preserved according to the sampling method requirements. The analytical laboratory will prepare and analyze the samples within the time frames specified in **Table G-6.**

FIGURE G.10

MASTER SAMPLE LOG

Plant _____ Page _____ of _____

Location _____ Project Number _____

Sample Number	Run Code	Date	Sample Type	Container Type #/Size	Disposition

FIGURE G.11
CHAIN OF CUSTODY

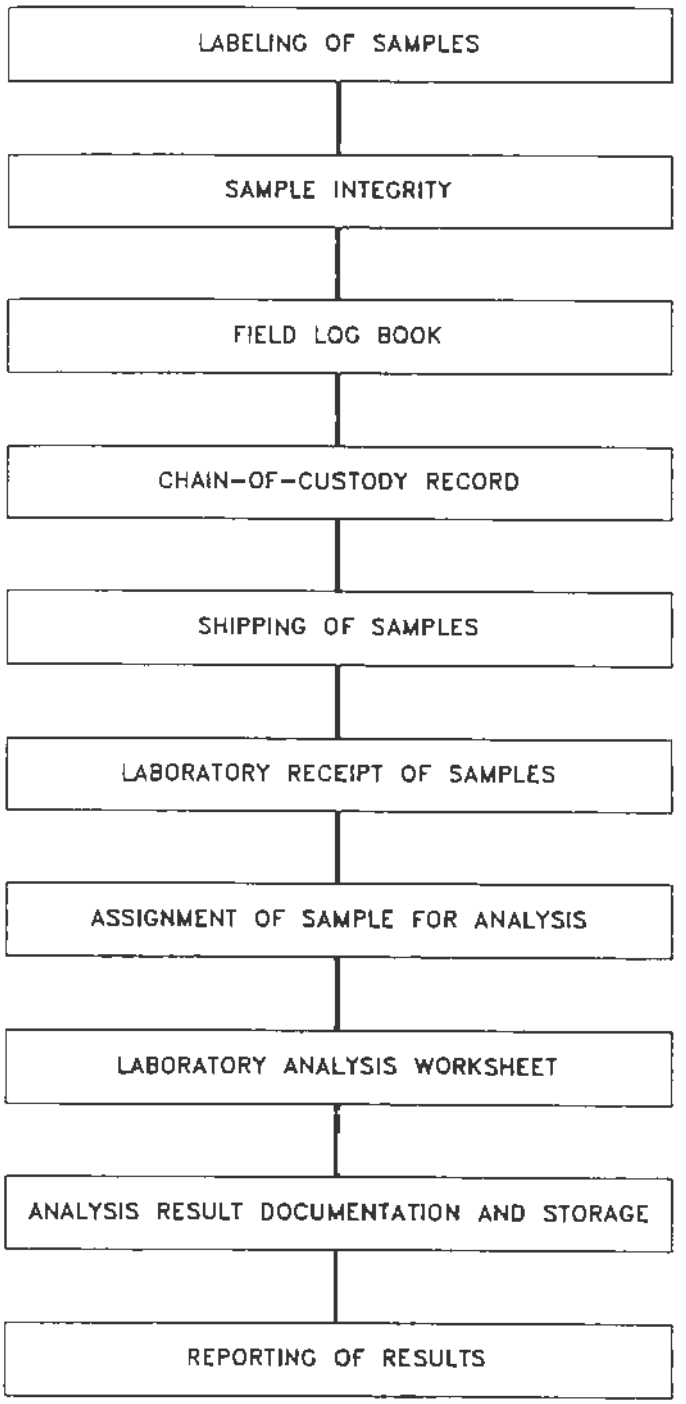


TABLE G-6

SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

Analysis Parameter	Sample Matrix	Container	Preservative	Max. Hold Time Before Prep (days)	Hold Time After Prep Until Analysis (days)
Semivolatile POHC	XAD-2	Cartridge	Chill with ice to 4° C	14	40
	Condensate	AG/TL	Chill with ice to 4° C	14	40
	Train rinses	AG/TL	Chill with ice to 4° C	14	40
	Filter	Petri dish	Chill with ice to 4° C	14	40
	Fly ash	AG/TL	Chill with ice to 4° C	14	40
	Auxiliary Fuel	AG/TL	Chill with ice to 4° C	14	40
Energetic POHC	Front half rinses	AG/TL	Chill with ice to 4° C	14	40
	Filter	Petri dish	Chill with ice to 4° C	14	40
	Condensate	AG/TL	Chill with ice to 4° C	14	40
	XAD-2	Cartridge	Chill with ice to 4° C	14	40
	Fly Ash	AG/TL	Chill with ice to 4° C	14	40
Dioxin and Furans	Front half rinses	AG/TL	Chill with ice to 4° C	14	40
	Filter	Petri dish	Chill with ice to 4° C	14	40
	Toluene Rinses	AG/TL	Chill with ice to 4° C	14	40
	XAD-2	Cartridge	Chill with ice to 4° C	14	40
Metals	Filter	Petri dish	None	14	40
	Front half rinses	AG/TL	Chill with ice to 4° C	14	40
	Fly ash	P, AG/TL	None		

TABLE G-6

SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

Analysis Parameter	Sample Matrix	Container	Preservative	Max. Hold Time Before Prep (days)	Hold Time After Prep Until Analysis (days)
Particulate Matter	Front half rinses Filter	AG/TL Petri dish	NA NA	NA NA	NA NA
Stack Gas Flow Rate	Stack emissions	NA	NA	NA	NA
CO_2, O_2 (Method 3)	Stack emissions	Plastic Bag	NA	NA	4 hours
Moisture	Stack emissions	NA	NA	NA	NA
NO_x (CEM)	Stack emissions	NA	NA	NA	NA
CO, O_2 (CEM)	Stack emissions	NA	NA	NA	NA
THC (CE)	Stack emissions	NA	NA	NA	NA

AG/TL = Amber glass container with Teflon® lined lid, P = polyethylene container

SECTION 6
CALIBRATION PROCEDURES AND FREQUENCY

6.1 SAMPLING EQUIPMENT

All stack sampling equipment to be used in this testing effort is periodically subjected to calibration and preventive maintenance procedures dictated by good practice and the EPA Quality Assurance Manual. No field equipment will be used which does not have valid calibration data prior to the test program, and calibrations of all equipment will be checked following the program.

6.1.1 Meter Boxes

Meter boxes used for source testing are subjected to multi-point calibrations once each year, or after repairs are made to the dry gas meter, orifice, or thermocouples. Meter boxes are assigned a unique ID number, and a calibration and maintenance notebook as they are received. Thereafter all calibrations and maintenance performed on meter boxes are recorded in the notebook. The dry gas meter and orifice are calibrated at five flow settings ranging from an orifice pressure of 0.5" WC to 4.0" WC. The meter box calibration factor (gamma) is calculated for each flow setting and checked to ensure that no individual gamma differs from the average by more than 0.02. Similarly the ΔH@ value of the orifice is calculated for each flow setting and checked to ensure that no individual value differs from the average by more than 0.15. Dry gas meter thermocouples are checked against a mercury-in-glass thermometer at three calibration points: Ice-point, ambient, and boiling H_2O point. Thermocouples are considered unacceptable and are repaired or replaced if they do not read within 5.4° F at each of the calibration points.

6.1.2 Pitot Tubes

Type-S pitot tubes are checked for conformance to the dimensional criteria described in Method 2 as they are received, and assigned a pitot tube coefficient, C_p, of 0.84. Since most pitot tube assemblies are permanently attached to stainless steel sampling probes, pitot tube calibration data is generally recorded in notebooks assigned to each sampling probe. The pitot tube assembly alignment is checked prior to and at the conclusion of each sampling program for which the pitot tube is used.

6.1.3 Thermocouples

Stack temperature thermocouples are permanently attached to the sampling probes and are checked as received for accuracy. The thermocouples are checked against a mercury-in-glass thermometer at three temperatures: ice-point, ambient, and boiling H_2O point. Calibration and maintenance data for each stack thermocouple are recorded in the appropriate sampling probe notebook. Probe liner, filter box, sample gas, and condenser thermocouples are checked for accuracy at three temperatures: ice-point, ambient, and boiling H_2O point. ES recognizes that the temperature of the probe liner, heated sample box, and sample gas temperature are generally maintained at temperatures of approximately 250° F. Since these temperatures are not used to calculate stack gas parameters or correct sample volumes, the calibration procedure is considered adequate when weighed against the danger of working with boiling oil. Each thermocouple is assigned a unique identification number and a notebook for recording calibration and maintenance data.

6.1.4 Sampling Nozzles

ES maintains a full range of sampling nozzles to conduct isokinetic sampling at a variety of exhaust gas velocities. Nozzles are stored in metal padded boxes to prevent damage during storage or transport. Nozzles diameters are determined prior to their use on a sampling train, after the nominal nozzle diameter to be used has been selected. The internal diameter of the nozzle is measured using a set of vernier calipers. The diameter used to calculate the nozzle area is determined from the average of three measurements of the nozzle in three different diameters. The nozzle is not used if an individual diameter differs from the average by more than 0.004 inch.

6.1.5 Orsat Analyzer

The Orsat analyzer is leak-checked and calibrated in accordance with EPA Reference Method 3.

6.1.6 Summary (Table)

A summary of sampling equipment with corresponding calibration procedures, frequencies, and acceptance criteria can be found in **Table G-7**.

TABLE G-7
CALIBRATION OF SAMPLING EQUIPMENT

Apparatus	Acceptable Limits	Frequency and Method of Measurements	Corrective Action
Wet Test Meter (64 ft^3/hr cap.)	$Y = 1.00 \pm 0.01$ for calibration range 7.5 to 65 ft^3/hr	Initially and annually by bell prover	Return to service center for corrective maintenance
Dry Gas Meter	Y tolerance for individual values ± 0.02 from average Y value	Calibration initially and annually against calibrated wet test meter at 0.50, 0.75, 1.0, 1.5, 2.0, and 4.0 in H_2O	Repair or replace as needed, recalibrate over full range of flow settings
	$Y_f = Y_i \pm 0.05Y_i$	Post-test calibration check after field use	Repair or replace as needed, recalibrate over full range of flow settings
	ΔH@ tolerance for individual values ± 0.20 from average ΔH@ value	Calibration initially and annually against calibrated wet test meter at 0.50, 0.75, 1.0, 1.5, 2.0, and 4.0 in. H_2O	Repair or replace as needed, recalibrate over full range of flow settings
Stack Thermocouple	1.5% of absolute temperature as indicated by ASTM mercury-in-glass thermometer	Initially and annually at ice-point, and boiling water point. Temperatures extrapolated to 1500° F	Adjust, determine calibration factor, or reject
Filter Heater Thermocouple	± 5.4° F as indicated by ASTM mercury-in-glass thermometer	Initially and annually at ice-point and boiling water point	Adjust, determine calibration factor, or reject
Condenser Outlet Thermocouple	± 2° F as indicated by ASTM mercury-in-glass thermometer	Initially and annually at ice-point and boiling water point	Adjust, determine calibration factor, or reject
Dry Gas Meter Thermocouples	± 5.4° F as indicated by ASTM mercury-in-glass thermometer	Initially and annually at ice-point and boiling water point	Adjust, determine calibration factor, or reject
S-type Pitot Tube Assemblies	$C_p = 0.84$	Initially and after field usage by geometric calibration procedures	Realign or replace
Probe Nozzle	Tolerance 0.004 in. for three measurements, 120° apart	Prior to each field test	Reshape and resharpen, then recalibrate
Analytical Balance	± 0.1 mg with Class S wts.	Annually serviced by field tech.	Adjust and repair as needed
		Adjusted prior to each use with Class S wts. (60.0000 g or 100.0000 g)	Adjust as needed to calibrate weight, call for factory service as needed

6.2 EQUIPMENT FOR INORGANIC ANALYSIS

6.2.1 Analytical Balances

Analytical balances are professionally cleaned and calibrated annually by certified balance technicians provided by the manufacturer. Following this professional calibration, a document is provided by the manufacturer, stating the model number, serial number and date of calibration. Additionally, a sticker noting the technician's name and the date of calibration is attached to the balance.

Each time the balance is used, a calibration check is performed using a set of Class S weights. The results of each calibration are recorded in a notebook which remains with the balance being used.

6.2.2 Atomic Absorption Spectrophotometer

Antimony, barium, chromium and lead will be determined using Atomic Absorption Spectrometry. The flame technique will be used unless the expected concentration of analyte in the samples is below these levels: antimony, 0.35 mg/L; barium, 0.5 mg/L; chromium, 0.2 mg/L; lead, 0.2 mg/L. Under these circumstances, the furnace technique will be used. Upon completion of the miniburn, additional data will be available to assist in the decision whether to use a flame or furnace technique.

Variations in atomic absorption instrument design complicate the delineation of specific calibration criteria. At a minimum, one standard and one blank should be used to generate a calibration line for flame AA analysis. For instruments capable of multipoint calibration, a minimum of three standards and a blank should be used. As a guideline, a linear correlation coefficient greater than 0.995 is suggested for curve acceptance.

Calibration curves for the furnace technique will be generated by preparing a blank and at least four standards. Standards and samples will be matrix matched, and necessary matrix modifications will be performed as prescribed by EPA SW-846.

All standards used will be either commercially obtained or prepared according to EPA SW-846. Standards having a concentration of less than 1 mg/L will be prepared daily. Standards having a concentration of 1 mg/L or higher will be prepared weekly.

After each initial calibration an accuracy check will be made with a mid-range standard prepared from a source independent of that used for the calibration standards. The result of this check should be within 90 to 110 percent of the theoretical value. If not, the source of the discrepancy must be determined and corrected before analysis can begin.

A continuing calibration check will be made to verify the accuracy of the calibration curve throughout sample analysis. A midrange standard will be run before and after each sample group, or for groups having more than 10 samples the check standard should be run after every tenth sample. The check will be considered acceptable if within 90 to 110 percent recovery for flame analysis or within 80 to 120 percent recovery for furnace analysis.

6.2.3 Summary (Table)

A summary of QA/QC Prodcedures for Metals Determination is provided in **Table G-8**. This table details calibration criteria.

6.3 EQUIPMENT FOR ORGANIC ANALYSIS

6.3.1 GC/MS

Analysis of liquid extracts resulting from sampling for polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and Principal Organic Hazardous Compounds (POHCs) will be performed with a gas chromatograph/mass spectrometer (GC/MS). An internal standard calibration technique will be employed.

The internal standard technique is a method whereby all the samples are spiked with known quantities of compounds which are analytically similar to the target compounds and which are not native to the samples of interest. Standards for initial calibration are prepared for all target compounds at a minimum of five concentration levels (including a blank) and each is spiked with a measured quantity of internal standard. The lowest calibration standard will have a concentration ten times lower than the concentration level corresponding to a 99.99

TABLE G-8
SUMMARY OF QA/QC PROCEDURES
FOR METALS DETERMINATION

Quality Parameter	Method of Determination	Frequency	Criteria
Calibration: Initial	Analysis of between two and five calibration standards	Before sample analysis	Instrument dependent, linear correlation coefficient ≥ 0.995
Calibration: Continuing Check	Analysis of midrange calibration standard	Before and after each sample group	90%-110% recovery for flame, 80%-120% for furnace
Accuracy: Initial calibration check	Analysis of standard independent of initial calibration standards	After each initial calibration	90%-110% of theoretical value
Accuracy: Spikes	Sample is divided, and one portion spiked with analyte at three times DL or two times sample level	One per sample matrix	70%-130% recovery
Precision: Duplicates	One sample divided and each portion prepared and analyzed separately	One per sample matrix	Range $<35\%$ if sample result above lowest standard[1]
Blank	Method blank carried through all sample preparation and analysis steps	One per sample batch	Below detection limit

[1]Range refers to the difference between two spike recoveries.

percent destruction removal efficiency (DRE). When these spiked calibration standards are analyzed, a response factor correlating response of the characteristic ions of each analyte with that of the internal standard may be calculated as follows:

RF = AsCis/AisCs

where:

RF = Response Factor

As = Characteristic ion area response of a target compound in the calibration standard

Ais = Characteristic ion area response of the internal standard

Cs = Amount (ng) of target compound in the calibration standard

Cis = Amount (ng) of internal standard

If the relative standard deviation of the average response factor is less than 30 percent, then the average RF can be used for analysis. Alternatively, the results can be used to plot a calibration curve of response ratios, As/Ais versus RF.

The working calibration curve or RF must be verified periodically by the measurement of one or more continuing calibration standards. If the response varies by an amount exceeding the criteria specified by each method, then new calibration standards must be prepared and a new curve generated.

6.3.1.1 Modified Method 5 (SW-846 Method 0010)

GC/MS calibrations for the analysis of naphthalene and 2,4-dinitrotoluene in samples resulting from the MM5 sampling train will be performed as described in Section 8.3.1 of this document and according to SW-846 Method 8270. Tuning of the hardware will be accomplished using 50 ng of decafluorotriphenylphosphine (DFTPP) as prescribed by Sections 7.3 and 7.4 of Method 8270. Hardware tuning will be performed every twelve hours. Internal standards used for this analysis will be acenaphthene-d_{10} and phenanthrene-d_{10}. These standards will be spiked into sample extracts so as to obtain a concentration of 40 ng/μl.

After the initial calibration is finished and before samples are analyzed an initial calibration check will be made by analyzing a standard which: 1) has been prepared from a different source material than the initial calibration standards; and 2) is prepared at a concentration level equivalent to 99.99 percent DRE. The average relative response factor obtained from

initial calibration will be considered valid if the response of this check standard is within 70 to 130 percent of its theoretical value. If this criterion is not met after two check runs, the nature of the discrepancy must be determined and corrected before analysis can begin.

Once sample analysis has begun, a continuing calibration standard will be measured every 12 hours and at the end of the day. This check will be made with one of the initial calibration standards and will be considered acceptable if the resulting response factor is within 30 percent of the average calibration relative response factor RRF. All samples must be bracketed by successful calibration checks; if a continuing calibration check fails after two attempts a new initial calibration must be performed with newly prepared standards, and all samples analyzed between the last successful calibration check and the failed calibration check must be re-analyzed.

6.3.1.2 Method 23

GC/MS calibration for analysis of PCDDs and PCDFs will be accomplished as described in EPA Method 23 and in Section 8.3.1 of this document. An initial five-level calibration will be performed using the compounds and concentrations listed in Table 2 of Method 23.

The internal standards will be added to every sample prior to extraction. There will be ten carbon-labeled PCDDs and PCDFs as internal standards representing the tetrathrough octachlorinated homologues. Each internal standard is used to quantify the indigenous PCDDs or PCDFs in its homologous series. (For example, the $^{13}C_{12}$-2,3,7,8-tetrachlorinated dibenzodioxin is used to calculate the concentrations of all other tetra-chlorinated isomers.) The relative standard deviation for the mean response factor of each standard will be calculated and considered acceptable if within the limits prescribed by Table 5 of the method. Sample analysis will then begin if the system operation requirements specified in Section 6.1.1 of Method 23 are met.

The continuing calibration check will be performed by analyzing solution #3 from Table 2 of the method and comparing the RRF for each compound in this standard to the corresponding RRF obtained during initial calibration. These response factors should agree within the limits prescribed in Table 5 of the method. If not, a new initial calibration must be performed with newly prepared standards, and all samples analyzed between the last successful calibration check and the failed calibration check must be re-analyzed.

6.3.1.3 Summary (of QAQC procedures for SVOST)

Summary tables of QA/QC procedures for SVOST analysis and M23 analysis are provided as **Tables G-9 and G-10**. Also, **Table G-11** presents the summary of QA/QC procedures for POHC determination in fly-ash.

6.3.2 GC/ECD

Analysis of nitroglycerin in samples resulting from the Sampling Train for Energetic Materials (STEM) will be performed using a gas chromatograph with an Electron Capture Detector (ECD) as prescribed by the USAEHA method.

A multipoint calibration curve will be generated by preparing and analyzing standards at five concentration levels, including a blank. The lowest standard above blank level will have a concentration equivalent to 10 times less than the 99.99 percent DRE level. The calibration curve generated by this method will be considered acceptable if it has a correlation coefficient of 0.99 or more.

The calibration curve will be verified every 12 hours and at the end of the day through analysis of a continuing calibration standard. This standard will be the lowest initial calibration standard and the response of this standard check must agree to within 30 percent of the average initial calibration response factor. If not, a new calibration curve must be generated from newly prepared standards and all samples analyzed since the last valid calibration check must be re-run.

6.3.2.1 Summary of QA/QC Procedures for STEM

A summary table of the AEHA STEM QA/QC procedures is provided as **Table G-12**.

6.4 CEM EQUIPMENT

6.4.1 CO Monitor

The calibration procedures and frequency for the CO process monitor are described in Section 4.6.3 of this appendix.

TABLE G-9
SUMMARY OF QA/QC PROCEDURES
FOR SVOST ANALYSIS

Quality Parameter	Method of Determination	Frequency	Criteria
Calibration: Initial	Five-level calibration curve for POHCs and internal standards. Lowest standard ten times lower than DRE critical level	At beginning of day	<30% RSD of average RRF
Calibration: Tuning	Hardware tuning using 50 ng DFTPP	Every 12 hours	Meet criteria of Metbod 8270 Section 7.3-7.4
Accuracy: Initial Calibration Check	Standard made at DRE critical concentration and prepared from a different source tban cal. standards	After initial calibration before sample analysis	70%-130% of theoretical value
Accuracy: Continuing calibration check	Analysis of a continuing calibration standard	Every 12 hours and at end of day	Within 30% of RRF from calibration
Accuracy: Surrogate Standard	Surrogate standards added to samples prior to extraction at ≤two times DRE critical level	Every SVOST component	50%-150% recovery
Precision: POHC Determination	Duplicate analysis of all SVOST components from the run of highest POHC concentrations	Once per trial burn	±50% RPD if concentration is above lowest cal. standard, otberwise ±100%
Blank: Metbod	Metbod blank for each SVOST component	One per sample batch	Blank value <two times DL. If greater, DL is changed to 1.5 times blank value
Blank: Field	Blank train carried through sample prep and analysis	One per test run series	Evaluated on a case-by-case basis

TABLE G-10
SUMMARY OF QA/QC PROCEDURES
FOR METHOD 23

Quality Parameter	Method of Determination	Frequency	Criteria
Calibration: Initial	Five standard calibration using compounds and concentrations listed in Table 2 of Method 23	At beginning of day	Conforming to criteria in Section 6.1.1 of Method 23
Accuracy: Continuing Calibration Check	Analysis of solution #3 from initial calibration	Every 12 hours and at end of day	Conforming to criteria in Section 6.1.12.1 of Method 23
Accuracy: Audit Sample (for compliance testing)	Analysis of audit sample containing tetra through octa isomers of PCDD and PCDF	With each set of compliance samples	Conforming to Section 8 of Method 23
Accuracy: Surrogate Standard	Surrogates added to the adsorbent cartridge of eacb train before sampling begins	Every train	70%-130% recovery
Accuracy: Internal Standard	Internal standards added to every sample prior to extraction	Every train	40%-130% for tetra- through hexachlorinated compounds and 25% to 130% for higber hepta- and octachlorinated homologues
Precision: Surrogate standard	Same surrogate spikes as for accuracy. Results from each run are compared	Every train	± 50 RPD
Blank: Method	Method blank for each M23 set of like samples	One per sample batch	Blank value < two times DL. If greater, DL is changed to 1.5 times blank value
Blank: Field	Field blank carried through sample prep and analysis	One per test run series	Evaluated on a case-by-case basis

TABLE G-11
SUMMARY OF QA/QC PROCEDURES
FOR POHC DETERMINATION IN FLY ASH

Quality Parameter	Method of Determination	Frequency	Criteria
Calibration: Initial	Five-level calibration curve for POHCs with internal standards. Samples bracketed by standards	At beginning of day	<30% RSD of average RRF
Calibration: Tuning	Hardware tuning using 50 ng DFTPP	Every 12 hours	Meet criteria of Method 8270 Section 7.3-7.4
Accuracy: Initial Calibration Check	Standard made at DRE critical concentration and prepared from a different source than cal. standards	After initial calibration before sample analysis	70%-130% of theoretical value
Accuracy: Continuing Calibration Check	Analysis of a continuing calibration standard	Every 12 hours and at end of day	Within 30% of RRF from calibration
Accuracy: Surrogate Standard	Surrogate standards spiked into sample at expected POHC level	Every sample	50%-130% recovery
Accuracy: Spikes (when GC/MS is not being used)	Sample divided in lab and spiked with POHCs at ≤ two times expected concentration	One per sample matrix	50%-130% recovery
Precision: Surrogate Standard	Same surrogates as for accuracy. Results from each run compared	One per test condition	Recoveries vary by <35%
Precision: POHC Determination	Duplicate preparation and analysis of sample	One per sample matrix	<35% range
Blank-Method	Method blank carried through all sample preparation steps	One per sample batch	<5% of sample levels

TABLE G-12
SUMMARY OF QA/QC PROCEDURES
FOR STEM ANALYSIS

Quality Parameter	Method of Determination	Frequency	Criteria
Calibration: Initial	Five-point calibration curve for nitroglycerin. Lowest standard at least 10X lower than DRE critical level	Daily	Correlation coefficient of at least 0.99
Calibration: Continuing Calibration Check	Analysis of a continuing calibration standard	Every 12 hours and at end of day	Within 30% of RF from initial calibration
Accuracy: Surrogate Spikes	Surrogate compound ethylene glycol dinitrate spiked into train prior to sampling at four times DRE concentration	Each sampling train	Mean of all results 75% to 125%
Accuracy: Blind Spikes	Independent lab QA coordinator to insert blanks spiked with POHC and surrogate into sample batch to be analyzed	10% of total number of samples	70%-130% recovery
Accuracy: Confirmational Analysis	Field samples testing positive for POHC and/or surrogate submitted for analysis by GC/MS	10% of positive samples	70%-130% recovery compared to GC result
Precision: Surrogate Spike	Same surrogate spikes as for accuracy. Results of each run compared	Each sampling train	±25% RPD
Precision: Blind Spikes	Same spikes as for accuracy	10% of total number of samples	±30% RPD
Precision: Duplicates	Sample extracts will be divided for replicate analysis	10% of field samples	±30% RPD
Blank: Method	Method blank for each STEM section	One per sample batch	Blank value < two times DL. If greater, DL is changed to 1.5 times bland value
Blank: Field	Blank train carried through sample prep and analysis	One per test run series	Evaluated on a case-by-case basis.

6.4.2 O_2 Monitor

The calibration procedures and frequency for the O_2 process monitor are described in **Section 4.6.1** of this appendix.

6.4.3 NO_x Monitor

The calibration procedures and frequency for the NOx monitor are described in **Section 4.6.2** of this appendix.

6.4.4 THC Monitor

The calibration procedures and frequency for the THC analyzer are described in **Section 4.6.4** of this appendix.

6.5 PROCESS INSTRUMENTATION

The sampling methods used for this trial burn will result in a variety of samples including impinger solutions, rinse solutions, filters, XAD cartridges, condensate and fly ash samples. Preparation and analysis of each sample will be accomplished through the use of appropriate EPA-approved methods. A summary of samples to be analyzed correlated with the preparatory and analytical methods to be used is given in **Table G-5**. No modifications will be made to the indicated methods. Internal standards for POHC quantification will be: Naphthalene-d8 for naphthalene; acenaphthene-d_{10} for 2,4-dinitrotoluene. Surrogate standards for POHC analysis will be: Acenaphthene or acenaphthylene for naphthalene; 3,4-dinitrotoluene for 2,4-dinitrotoluene; ethylene glycol dinitrate for nitroglycerin. Detailed information regarding the choice of spiking concentrations of standard is given in **Section 11** of this document.

Either flame or furnace atomic absorption spectrometry may be used for metals analysis, depending on the concentration of analyte in the samples. Criteria for choosing between AA methods are given in the 7000 methods of SW-846 and are stated in **Section 8** of this document.

SECTION 7
ANALYTICAL METHODS

The sampling methods used for this trial burn will result in a variety of samples including impinger solutions, rinse solutions, filters, XAD cartridges, condensate and fly ash samples. Preparation and analysis of each sample will be accomplished through the use of appropriate EPA-approved methods. A summary of samples to be analyzed correlated with the preparatory and analytical methods to be used is given in **Table G-5**. No modifications will be made to the indicated methods. Internal standards for POHC quantification will be: Naphthalene-d8 for naphthalene; acenaphthene-d_{10} for 2,4-dinitrotoluene. Surrogate standards for POHC analysis will be: Acenaphthene or acenaphthylene for naphthalene; 3,4-dinitrotoluene for 2,4-dinitrotoluene; ethylene glycol dinitrate for nitroglycerin. Detailed information regarding the choice of spiking concentrations of standard is given in Section 10 of this document.

Either flame or furnace atomic absorption spectrometry may be used for metals analysis, depending on the concentration of analyte in the samples. Criteria for choosing between AA methods are given in SW-846 and are stated in **Section 8** of this document.

Successful detection and quantification of each POHC will be demonstrated through the execution of a mini trial burn which will be conducted on the Seneca Army Depot Deactivation Furnace prior to full scale testing. Method performance data from the miniburn will be submitted to verify the effectiveness of the proposed methods in advance of regular emissions testing.

SECTION 8
INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Successful detection and quantification of each POHC will be demonstrated through the execution of a mini trial burn which will be conducted on the Seneca Army Depot Deactivation Furnace prior to full scale testing. Method performance data from the miniburn will be submitted to verify the effectiveness of the proposed methods in advance of regular emissions testing.

Proven methods of sampling and analysis must be retested and verified to ensure the reliability of the resulting data. The accuracy of analyte measurement can be checked in a number of ways: spiking samples with surrogate standards; splitting samples and spiking one portion of the split with analyte; and spiking blanks with analyte. In each of these cases a known quantity of material has been added to a sample and a percent recovery of that material can be calculated upon analysis to yield a measure of how accurately the method is determining actual quantities of analyte in samples. Similarly, from comparison of surrogate spikes from run to run, or from duplicate analysis of sample splits, relative percent differences or standard deviations can be calculated to indicate the reproducibility or precision of the data. Field blanks, reagent blanks, and method blanks give information about background interferences and possible sources of sample contamination. All of these techniques will be used to verify data quality as recommended by each reference method.

8.1 REAGENT QUALITY

Only spectrographic or reagent grade and HPLC grade solvents and reagents will be used. Blank solutions of reagents will be included with each sample batch in accordance with the appropriate reference method so that sources of contamination may be traced.

Use of high quality reagents, however, does not ensure high quality analytical results. This is due to variations in the quality of reagents from different suppliers and also differences occur in lots from the same source. In addition, impurities impact differently on different analyses. Constant attention is given to potential errors due to impurities and instability of the reagents, solvents and gases used for all analyses. The analyst must predetermine that all reagents used are of specified quality to ensure proper analytical results.

The procedures used to ensure reagent quality are:

- Select high quality reagents (analytical grade or better).
- Date and initial each reagent when received and when opened. Expiration dates are determined by the manufacturer, literature, or experience.
- Solutions prepared on-site are dated and initialed. The solution's identification, concentration, expiration date, and special storage conditions are indicated on the label.
- Use only reagents that have not expired.
- When an aliquot of a standard is taken, the information from standard label is included on the aliquot container.

The QA Coordinator is responsible for verifying that standards and reagents are correctly labeled.

8.2 ANALYTICAL QC CHECKS

8.2.1 Semivolatile POHC

8.2.1.1 Emissions Testing

Internal accuracy checks will be made of SVOST train component analysis in two ways. First, each SVOST component will be spiked with surrogate standards just prior to sample preparation for extraction. The surrogates will be added to result in a concentration of not more than two times the 99.99 percent DRE level. 3,4-dinitrotoluene will be used as the surrogate for 2,4-dinitrotoluene, acenaphthene or acenaphthylene will be used as surrogates for napthalene. Acceptable limits for recovery of these surrogates are between 50 and 150 percent. This check will be done on each sample run.

The second accuracy check will be to spike blank SVOST train components with the target analytes and their surrogates at no more than two times the 99.99 percent DRE level and have the components analyzed as samples. This check will be performed once per trial burn and will be considered acceptable if within 50 to 150 percent recovery.

Precision will be determined by tabulating the results of the sample surrogate spikes described above and comparing recoveries for each compound in like components from run to run. For three or fewer runs, a relative percent difference (RPD) of less than 40 percent is acceptable. For greater than three runs, a relative percent difference of less than 30 percent should be obtained.

Once per trial burn precision will also be measured by performing duplicate analysis of extracts from all components of the run having the highest POHC concentrations. If the concentration of this duplicate is higher than that of the lowest calibration standard, then ± 50 percent RPD is an acceptable result. If the duplicate concentration is lower than that of the lowest calibration standard a criterion of ± 100 percent must be met. Should there be no instrument response at all, then the calculation of an RPD is not possible and unnecessary.

To verify reagent quality and analytical technique a method blank will be prepared and analyzed for each SVOST component. A blank value of less than twice the detection limit is acceptable. Otherwise, the detection limit must be changed to 1.5 times the blank value. If this elevated blank value is not due to normal and unavoidable trace impurities then the source of contamination should be traced and corrected before any other samples are prepared.

Once per test run series a field blank will be analyzed. This blank is a train which has been prepared, carried to the field, exposed to the same procedures as sample trains but not used for sampling, and then analyzed as though it had been sampled. The result of the field blank will provide some information about possible contamination in train preparation and field handling, and will be evaluated on a case-by-case basis.

8.2.1.2 Ash Testing

The surrogate standards listed in **Section 8.2.1.1** will also be used in QC checks of ash analysis. Accuracy will be measured by spiking each ash sample with the surrogate compounds at ten times the minimum detection levels, the expected POHC concentration levels prior to sample preparation. Analysis of the samples should result in a surrogate recovery of 50 to 130 percent.

Precision will be determined in two ways: Sample surrogate spike recoveries will be compared to runs within the same test condition; and ash samples will be split and analyzed in duplicate for each sample matrix type or for five percent of ash samples, whichever is more frequent. The surrogate standard analysis should yield a percent difference of recovery of less than 35 percent. Duplicate analysis should yield less than a 35 percent difference over the range of samples analyzed.

A method blank will be prepared and carried through sample analysis. An acceptable blank will have a concentration of less than five percent sample levels.

8.2.2 Energetic POHC

8.2.2.1 Emissions Testing

Internal accuracy checks will be made of STEM sample analysis through surrogate spikes, blind spikes, and through confirmational analysis of samples testing positive for POHC. Prior to emissions testing, each sampling train will be spiked with the surrogate compound review recoveries ethylene glycol dinitrate at four times the 99.99 percent DRE level.

Emissions sampling and analysis will then progress normally. The mean of recoveries generated in this manner should fall between 75 and 125 percent.

Blind spikes will be inserted into each sample batch by an independent lab QA coordinator. These spikes will comprise 10 percent of the total number of samples and will be prepared by adding nitroglycerin and ethylene glycol dinitrate to a blank at four times the 99.99 percent DRE level. Spike recoveries should be 70 to 130 percent.

The third accuracy check will confirm the GC/ECD analytical results by having 10 percent of samples which tested positive for POHC or surrogate re-analyzed by GC/MS. In this confirmational analysis a recovery of 70 to 130 percent as compared to the GC/ECD analysis will be considered acceptable.

Precision of the analysis of energetic materials will also be determined in three ways: surrogate spike recoveries from the accuracy check will be compared from run to run with a QC goal of $\pm$ 25 percent RPD; blind spike recoveries from the accuracy check will be

compared from run to run with a QC goal of ± 30 percent RPD; 10 percent of all sample extracts will be divided for duplicate analysis with a QC goal of ± 30 percent RPD.

Method blanks will be run with each sample batch. The blank value thus obtained should be no more than two times the detection limit. Otherwise, the detection limit must be changed to 1.5 times the blank value.

A blank train will also be prepared, exposed to all field procedures except sampling and analyzed once for each test run series. The result of this check will be evaluated on a case-by-case basis.

8.2.2.2 Ash Testing

Precision and accuracy checks and objective for analysis of nitroglycerin in ash samples are identical to those outlined for SVOST analysis of ash samples described in **Section 10.2.1.2** of this document.

8.2.3 Dioxins and Furans (Method 23)

Accuracy for the analysis of PCDDs and PCDFs in stack gas will be determined through the use of surrogate standards, internal standards, and an audit sample.

Surrogate standards will be prepared in accordance with Method 23 and added to the adsorbent cartridge of each train before sampling begins. Following this, testing and train recovery will proceed normally and the samples will be sent to the laboratory. Prior to extraction each sample will then be spiked with the internal standards specified in Method 23. Sample extraction and analysis will then be completed and recoveries can be calculated for each added compound in each sample. The surrogate QC goal is 70 to 130 percent recovery. The internal standard QC goal is 40 to 130 percent for tetra- through hexachlorinated compounds and 25 to 130 percent for higher hepta- and octachlorinated homologues.

As an additional accuracy check, an audit sample containing tetra through octa isomers of PCDD and PCDF purchased from an independent source and analyzed with the samples. The QC criteria associated with this audit are specified in Section 6.1.2.1of Method 23.

The precision of this analysis will be determined by comparing the results of the sample surrogate spikes for each sample type from run to run. A relative percent difference in recovery of ± 50 percent is considered acceptable for this check.

A method blank will be run for each sample type in each sample batch. The resulting blank value should be no more than two times the detection limit. Otherwise the detection limit must be changed to 1.5 times the blank value.

A blank train will be carried through all field procedures except sampling and will then be analyzed. This field blank will be included in each test run series and the resulting data will be evaluated on a case-by-case basis.

8.2.4 METALS

8.2.4.1 Emissions Testing

Accuracy of the metals train analysis will be evaluated by splitting one sample of each type from each sample batch into two portions and spiking one portion of each sample with analyte. The spike will be added at two times the expected sample concentration unless sample level is near or below detection limit. In this case, the spike added will be three times the detection limit. The QC goal for this accuracy check is from 70 to 130 percent recovery.

Precision of metals analysis will be determined by dividing one sample of each type per batch into two portions and analyzing each portion as a separate sample. If the result of duplicate analysis is above the lowest calibration standard level, then a difference of less than 35 percent over the range of duplicates analyzed will be considered acceptable.

A method blank will be carried through all sample preparation and analysis for each sample type in each sample batch. The blank result should be below the detection limit.

8.2.4.2 Ash Testing

QA checks for metals testing of ash samples are identical to those outlined for stack emissions in **Section 10.2.4.1** of this document.

8.3 SUMMARY OF INTERNAL QC CHECKS

Information on QA check procedures is summarized in the following tables:

Semivolatiles in Stack Samples: **Table G-9**
Semivolatiles in Ash and Auxiliary Fuel Samples: **Table G-11**
Energetics in Stack Samples: **Table G-12**
PCDDs/PCDFs: **Table G-10**
Metals in Stack or Ash Samples: **Table G-8**

SECTION 9
DATA REDUCTION, VALIDATION AND REPORTING

Protocol to ensure proper recording, calculating and reporting of data has been established at every level of data handling.

9.1 DATA REDUCTION

9.1.1 Field Data

All field sampling data will be recorded on field data sheets during the sample runs. Recorded data include: Gas temperatures at the inlet and outlet of the dry gas meter and at the stack sample point; pressure differentials associated with the pitot tube and orifice; and sample volumes. Examples of field data sheets are given in **Section 6** of this appendix. Recorded data will then be transferred to a pre-made computer spreadsheet containing necessary formulae for calculation of stack gas characteristics including gas moisture content, velocity and molecular weight.

Continuous emissions analyzers will be interfaced with a computerized data system which scans instrument responses at user-defined intervals, converting those responses into relevant engineering units based on calibration data, and writing the results to both hard disk and printer. In this way instantaneous data will be recorded magnetically and on paper every 30 seconds. The data recorded will include time of sampling, testing parameter, and concentration of analyte detected. The sampling technician will enter the date into the computer system at the start of each sampling day to flag the beginning of a new set of data.

9.1.2 Analytical Data

Raw data for particulate analysis, raw data not collected and recorded by a computer data system, and any observations or notations of unusual circumstances will be recorded in a hardbound, prenumbered laboratory notebook. Each page of the notebook will contain spaces for the date and signature or initials of the analyst and all notations will be made in permanent ink. Any manually performed calculations will be recorded in the laboratory notebook.

Raw data from GC/ECD analysis will consist of retention time and peak area for each target and surrogate compound. Calibration data collected and stored by the GC integrating data system is used to generate a standard curve for the target compound through linear regression. As sample analysis is performed, sample peak retention times are compared to standard retention times by the data system for identification of analyte. Then peak areas of identified sample components are automatically interpolated along the standard curve to calculate the quantity of analyte detected and this result is stored on disk along with the assigned sample number.

Raw data from GC/MS analysis will consist of retention time, peak area, and mass spectral profiles of each standard and surrogate compound. Calibration data generated are used by the data system to calculate a relative response factor. As sample analysis is performed, sample peak retention time and spectral profile is automatically compared to calibration retention times and spectral profiles for identification of the analyte. Then identified sample components are quantified by the data system using the calibration relative response factor. The resulting quantified data are stored on disk along with the assigned sample number. Unedited printouts of GC and GC/MS data will be made and maintained in the laboratory's project work file. These file hard copies will include all raw data, converted data, chromatograms and mass spectra associated with samples, standards and surrogates.

If the atomic absorption spectrophotomer used for metals analysis has an associated data system, then this system will generate a linear regression from stored calibration data and sample data will automatically be interpolated along the calibration line to determine quantity of analyte detected. In this case, hard copies of all data shall be produced and maintained in the laboratory's project work file. Otherwise, all calibration and sample analysis data must be entered into a hardbound laboratory notebook as previously described.

9.2 DATA VALIDATION

9.2.1 Field Data

All field data sheets will be examined by a qualified individual other than the one who recorded the data. The data sheets will be checked for any obvious omissions of data or calculation errors. The data sheets will be kept in a project data file along with documentation of associated equipment calibrations. When the field data is transferred into

computer spreadsheets, another check will be made for accuracy of data entry and correctness of formulas used in calculations.

Computerized data from continuous emissions monitors will be examined to verify that they are correctly dated and that results have been correctly calculated by the data system. Printouts of this data will also be kept in the project data file.

9.2.2 Analytical Data

All data and calculations recorded in a laboratory notebook will be examined for completeness, correctness, and legibility by a qualified individual other than the one who originally recorded the data. After completion of this check the notebook should be signed or initialed by the examiner and dated.

GC and GC/MS data shall be examined to verify that hard copies of all data have been generated and filed, including all QC data. Additionally, 50 percent of the raw data shall be examined to confirm peak shape and resolution, to assure accurate integrator detection and identification of peaks, and to verify calculations.

Computer generated metals data will be checked to assure complete hard copy documentation of calibration and analytical results, that these are properly filed, and to verify calculations.

9.3 DATA REPORTING

All data resulting from sampling and analysis will be summarized in tables and presented in a report with explanatory narrative detailing results, interpretation and conclusions. Documentation of raw data and computations will be presented including copies of all field data sheets, computer calculation sheets of field data, laboratory notebook entries, chromatograms, mass spectra and any other computer generated records of analysis. All QA/QC data will be included and will be summarized in a table.

The project manager is responsible for data review and interpretation and oversees all aspects of report preparation and revision. The draft report is reviewed by the project manager and by senior technical personnel for completeness and accuracy before the final version is released.

SECTION 10
ROUTINE MAINTENANCE PROCEDURES AND SCHEDULE

10.1 DEACTIVATION FURNACE

The permit application describes the critical equipment necessary to maintain permit operating conditions and to demonstrate continuing compliance. Routine maintenance procedures and the schedule for those procedures are also described in the permit. The following is a list of the critical equipment required to ensure proper operating conditions are being maintained.

1. Fuel feed system
2. Waste feed system
3. Rotary kiln
4. Afterburner
5. High temperature heat exchanger
6. Low temperature heat exchanger
7. Cyclone
8. Baghouse
9. Induced draft fan
10. CO monitor
11. O_2 monitor
12. Stack gas velocity monitor
13. Thermocouples
14. Pressure gauges
15. Pressure transmitter

10.2 SAMPLING AND ANALYTICAL EQUIPMENT

A preventive maintenance schedule on all analytical instruments, balances, and equipment requiring maintenance will be followed. All maintenance, whether performed by the laboratory or other professional sources, is documented in permanent individual log books kept with each item. Entries are made each time maintenance is performed and include the

reason for maintenance, what was performed, by whom, and the dates and initials of the analyst in charge during the maintenance.

In the field sampling area, each piece of equipment has its own preventive maintenance schedule. The equipment supervisor and the sampling team leader have copies of these schedules. Under conditions or periods of heavy usage, certain pieces of equipment such as consoles are calibrated and maintained much more often than the schedule dictates to ensure data integrity.

Critical spare parts that should be on hand to minimize downtime in the field are a routine part of equipment checklists. For example, an extra Modified Method 5 console will be available on this project as well as redundant vacuum pumps, parts to repair vacuum pumps, and redundant glassware.

SECTION 11
PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

For each major measurement parameter, as shown in **Table G-3 and G-4**, the completeness, precision, and accuracy of the measurement data will be compiled and tabulated. Completeness is a simple measure of the number of acceptable samples or data points actually measured divided by the number which were planned expressed as a percent. Ways in which a sample can become incomplete or voided include not collecting the sample, sampling incorrectly, losing or breaking the sample in shipment, improper sample preservation, consuming the whole sample in a voided analysis, or outlier data point rejection.

Precision will be measured by the use of intralaboratory sample splits; accuracy will be measured by the use of sample spikes and EPA or NIST reference standards.

Precision is computed as percent relative difference, a measure of the difference between two samples assumed to be identical through dividing (splitting) an original sample, analyzing each portion, identifying the values of the first replicate (X_1) and that of the second replicate (X_2) and dividing the difference of X_1 and X_2 by the mean (X). This is called "Range Percent" in EPA/625/6-89/023 (Jan. 1990).

$$RPD = 100 \times \frac{x_1 - x_2}{X}$$

Accuracy is the difference between a measured value and the true value when the latter is known or assumed. The term accuracy is often used interchangeably with percent recovery and describes either recovery of a known amount of analyte (spike) added to a sample of known value, or recovery of an analyte of known value from a synthetic or environmental standard.

$$\% \text{ Recovery (spike)} = 100 \times \frac{\text{(concentration spike + sample) - sample}}{\text{concentration spike}}$$

$$\% \text{ Recovery (standard)} = 100 \times \frac{\text{observed value}}{\text{true value}}$$

Limit of detection is the lowest concentration of an analyte that the analytical process can reliably detect. The detection limit is estimated at three standard deviations above the measured average reagent blank. Procedures are available for evaluating and determining the true detection limit and are performed when economically and technically desirable. These procedures are beyond the scope of this program.

Concentration values below the limit of detection of the methods employed, result in the problem of determining how to report such values. This problem, should it occur, will be handled by reporting that the analysis was below the minimum detection limit and stating the minimum detection limit.

SECTION 12
AUDIT PROCEDURES, CORRECTIVE ACTION AND QA REPORTING

During the trial burn, the Quality Assurance Coordinator (or an appointed designee) will perform system and performance audits of the sampling and analytical programs. An overall audit of data quality will also be conducted. The results of these programs will be reported to the Program Manager and the necessary corrective actions will be implemented.

12.1 SYSTEMS AUDITS

The Quality Assurance Coordinator will perform a systems audit of the laboratories in accordance with ES' on-going quality assurance program. The results of the systems audit will be reported to the Project Manager. Corrective action will be taken in those areas identified as not meeting appropriate standards of quality.

Other systems audits to be performed include the following:

- Conduct periodic unannounced laboratory audits to evaluate laboratory performance.
- Verify and document procedures used in sample preparation and processing.
- Conduct unannounced inspections of sample custody, recordkeeping, and other data handling procedures.
- Review the analytical data and the statistical methodology used in the evaluation.

Unscheduled inspections may also be performed by and at the discretion of the NYSDEC Project Officer. They include inspections of QC data, maintenance/calibration records, or any specific element of the QA program.

12.2 PERFORMANCE AUDITS

The Quality Assurance Coordinator will provide the laboratories with samples to be analyzed as unknowns. Two or three of these samples will be analyzed initially. Additionally, one or two will be analyzed if requested by the Project Manager. The Quality Assurance Coordinator will determine acceptable levels of accuracy and precision. The kind and frequency of the performance audits will be left to the discretion of the Quality Assurance Coordinator.

12.3 CORRECTIVE ACTION

Whenever the precision and/or accuracy of QC check samples and/or field samples fail to meet the acceptance criteria in Table G-3, corrective action will be taken. Corrective action may be initiated by (1) the analyst/sampler, (2) the supervisor of the analyst/sampler, (3) the Project Manager, and (4) the Quality Assurance Coordinator. Periodic reviews of data and sampling/analytical activities by these individuals will ensure that problems requiring corrective action are identified and corrected. Reporting responsibilities are identical to those shown in the organizational chart (Figure G-2). The Quality Assurance Coordinator and the Project Manager will maintain a record of problems identified and documentation of their resolution.

If, during systems or performance audits, weaknesses or problems are uncovered, corrective action will be initiated immediately. Corrective action will include but not necessarily be limited to: recalibration of instruments using freshly prepared calibration standards, replacement of reagents that give unacceptable blank values, additional training of laboratory personnel, repair of instrumentation, and identification of source(s) of loss or contamination.

All appropriate information on corrective actions is forwarded to the requestor of the analysis. All actions and information obtained are systematically and fully documented in a timely manner. Every procedure requiring corrective action will be subject to additional periodic review to ensure that the data quality objectives are met.

12.4 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Written audit reports will be issued to the managers, team leaders, and QA coordinators for each audit conducted, and meetings will be held with these individuals to thoroughly discuss the findings. Formal review of any consistent deficiencies is noted during the next audit by the QA Coordinator.

Reports of systems and performance audits will be prepared by the Quality Assurance Coordinator. These will be presented to the Project Manager and will be available to the State Agency's Project Officer. These reports will be the basis of judgement about the quality of the project effort. They also will be the focal point of staff discussion of problems requiring corrective action. The overall audit of data quality will be reported in the trial burn report.

ATTACHMENT A
RESUMES OF THE KEY PERSONNEL

Biographical Data

JON N. BOLSTAD

Chemical Engineer

EXPERIENCE SUMMARY

Twenty years of experience in air pollution control and hazardous waste and combustion engineering with emphasis on control device design, design review, and air quality and emissions sampling and analysis including method development.

EXPERIENCE RECORD

1988-Date Engineering-Science. **Manager, Air Testing/Engineering** (1990-Date). Project manager for multi-million dollar ambient air monitoring project at Superfund remediation site, including development of an automated GC sampling, analysis, and data collection system and criteria pollutant samples. Technical Director/Project manager for many emission test projects including gas engines and turbines, VOC incinerators, municipal waste co-gen plants, coating facilities, industrial boilers, and other sources. Managed air testing for EPA SITE program process evaluation demonstration.

Supervising Engineer/Senior Project Manager (1988-1990). Project manager for design/build turnkey continuous emission monitoring systems for measurement of acetone and methylene chloride from military pyrotechnic facilities. QA officer for laboratory and emission testing activities. Conducted engineering analysis of impacts on air emissions of waste fuel burning in a sulfuric acid plant. Performed technical and economic feasibility study of VOC controls for a specialty aluminum coater. Managed a rapid-response project for air sampling and risk assessment of pesticide/herbicide disposal site at a major metropolitan airport. Consulting engineer to state government for review of RCRA permit application for bazardous waste incinerator.

1986-1988 Pacific Environmental Services, Reston, Virginia. **Senior Engineer.** Designed a dryer recirculation system for an 8-station rotogravure press, reducing exhaust flow from 80,000 SCFM to 20,000 SCFM and incinerating solvent vapors; tested pilot system on one of eight dryers to evaluate increases in solvent vapor concentration with respect to flammable limits, solvent retained in product, and dryer energy requirements.

Conducted design studies of press operations for a flexographic printer, including internal and external air flows, solvent vapor concentration and drying rates and temperatures for incineration system; specified a catalytic incinerator along with process modifications.

For an aluminum foil coater, he conducted engineering studies of several coating lines to evaluate solvent loss rates, effect of external recirculation and changes in air distribution internally in loop and tunnel dryers and slot and canopy hoods at the coating stations. He recommended modifications which improved capture efficiency from ~65% to ~80%.

Mr. Bolstad has also designed and conducted tests and reviewed tests conducted by others for the measurement of VOC capture and destruction efficiency at flexographic and rotogravure printers, coil coaters, flexible vinyl surface coaters, and foil coaters. Project manager for EPA project to design and build emission analyzer to measure benzene in gasoline vapors. A prototype UV unit was constructed and tested.

Served as technical consultant to local government for Part B permitting of hazardous waste incinerator.

1980-1986 Engineering-Science. **Senior Engineer.** Project Engineer/Field Test Leader for an IERL research project investigating the applicability of calciner kilns to destruction of hazardous wastes. Prepared sampling and analysis plans for POHCs, particulate matter, CO_2, NO_x, SO_2, CO, hydrocarbons, and HCl; collecting kiln operating data; and collecting and analyzing process, liquid waste, and solid waste samples. The effect of process conditions on hazardous waste destruction, fate of trace metals, and potential for emission of combustion-created compounds were evaluated.

For an organometallic chemical producer, conducted design studies and testing to modify a rotary kiln sludge incinerator to meet RCRA standards. Developed concept design for a vent gas/liquid organic/aqueous hazardous waste incinerator and air pollution control system for a pharmaceutical manufacturer. Managed trial burn tests at a plastics manufacturer's fluidized-bed incinerator.

Project Manager for an EPA New Source Performance Standard development project. Prepared sampling plans; developed sampling and analytical methods for measuring phenol, phenolic compounds, total organic carbon, formaldehyde, and particulate matter for evaluation of best available control technology; supervised the sampling, data reduction, and report preparation.

Project Manager for an emission sampling research program to generate particle size, particulate matter, SO_2, and NO_x emission data and collecting microscale fugitive particle emission data to calibrate a short-term dispersion model.

1971-1980 Montana Air Quality Bureau, Helena, Montana. **Engineering Section Supervisor** (1977-1980). Managed permit review program, including PSD review; developed new regulations and conducted engineering and economic evaluation of these new regulations. Supervised staff of regional engineers, staff engineers and planners, and chemist, as well as their local agencies. Performed important assessments on proposed state legislation.

Regional Engineer (1971-1977). Designed sampling and analysis plans, implemented sampling programs, and interpreted data on air emissions and effluent discharges for evaluations of: process modification and a "foam" scrubber for HF removal from an aluminum smelter; reduced sulfur emission control from a kraft pulp mill process and effluent; and from coal strip mine operations including overburden removal and storage, road construction, coal removal, and reclamation activities. These projects also required comparison of environmental baseline data with post construction data.

EDUCATION

B.S., Chemical Engineering, 1971, Montana State University, Bozeman, Montana

PROFESSIONAL AFFILIATIONS

American Institute of Chemical Engineers
Air Pollution Control Association

PUBLICATIONS AND PAPERS

"Design of two systems for VOC emission monitoring from solvent recovery/VOC control systems," AWMA Specialty Conference on Continuous Emission Monitoring, November, 1989.

"Determination of the Feasibility of Hazardous Waste Incineration in Calciner Kilns," APCA Paper 85-63.3, APCA National Meeting, June 1985 (coauthors J. Chehaske, W. Westbrook, M. Branscome, R. Mournigban).

"Environmental Impact Statement on the Expansion of the Horener-Waldorf Pulp Mill," Montana Department of Health and Environmental Sciences, 1975.

"Environmental Impact Statement on the Request for Variance by Anaconda Aluminum Company," Montana Department of Health and Environmental Sciences, 1974.

Biographical Data

DENNIS A. FALGOUT, PH.D., P.E.

Manager, Quality Assurance/Technical Direction

EXPERIENCE SUMMARY

Twenty-six years professional experience in environmental engineering especially in the areas of hazardous waste combustion permitting, air pollution control device evaluation and design, source emission testing and ambient air quality monitoring. Ten years as project manager and department manager at ES.

EXPERIENCE RECORD

1980-Date Engineering-Science. Responsible for quality assurance and technical management of hazardous waste and source monitoring studies including compliance determinations enforcement actions, control equipment evaluation and assistance with permit applications. Project manager of ambient monitoring and environmental impact assessment projects and preparation of environmental permitting applications.

From 1985 to 1990, **Manager** of the ES Air Engineering and Testing Department. The ES/SSCD Contract is managed by this department. The level of effort under this contract has averaged 20,000 man-hours per year. Dr. Falgout was the ES contract manager for the recent ES/OAQPS/EMB contract (68-02-4339), Stack Testing and Method Evaluation for Standard Setting. He managed the ES program of providing technical support to the EPA/OSW effort to develop regulations for boilers and industrial furnaces that burn hazardous waste materials.

Responsible for technical and administrative management of sampling and analysis of ambient air in vicinity of several hazardous waste sites during clean-up activities. These projects included solid sorbent sampling, automated gas chromatographic sampling/analysis (for light aromatic and chlorinated hydrocarbons), real time monitoring (for total hydrocarbons) and compound-specific manual methods (for PAH metals, H_2S and phenol). Threshold concentrations at which air emissions abatement actions were to be enacted were calculated from published data. On-site project managers were kept appraised of existing ambient concentrations.

Reviewed and assessed the QA/QC aspects of the MM5 and VOST procedures in conjunction with bis recent assessment of the test reports that comprise the emissions data base for the OSW program to develop regulations for industrial furnaces that co-fire hazardous waste. He developed acceptance criteria for the various data that are used to calculate DRE values and performed detailed technical reviews of the test reports. In addition, served in a QA advisory capacity on four, recent ES tests of the DRE of hazardous compounds; two of these tests were on cement kilns, one on a boiler, and one at a fluid bed incinerator.

Dr. Falgout was Technical Director on the recently completed revision of the RCRA Part B application for the central incineration complex (CIC) at Pine Bluff (Ark) Arsenal. This project included Trial Burn Test Plans and Trial Burn Tests of two incinerators (1-RKI; 1 FBI) plus engineering evaluations of two existing scrubbers. Additional work for the same client included turnkey installation of two continuous emissions monitors: one for methylene chloride and one for acetone.

He also was Technical Director for the top down analysis required by the New Jersey Department of Environmental Protection for selection of the air pollution control train for the sewage sludge incinerator that was being designed by ES for the Joint Meeting of Essex and Union Counties WWTP.

Dr. Falgout was Technical Director of a two-year study of odor generation in the Anne Arundel County Sewerage system. This study included evaluation of several candidate odor control techniques including installation of a pilot scrubber at a pump station.

Dr. Falgout was Technical Director of a study of the sources of odor emitted from the San Carlos (Tampa) waste water pump station and an engineering and cost analysis of potentially applicable air pollution control devices.

Dr. Falgout managed a project to assist the owner of an asphalt pavement plant in Philadelphia (PA) determine the rates of emission of total particles, volatile organic substances and toxics (including toxic metals and toxic organic compounds) and to improve both the process and the air pollution control device so as to reduce those emissions.

Prepared the Trial Burn Plan (Section D5b) for a RCRA/Part B application for a new hazardous waste incinerator being constructed in the State of Washington.

Dr. Falgout managed the design and implementation of a quality assurance program for the ES-Eastern Regional office.

1977-1980 Southern Research Institute, Birmingham, Alabama. **Senior Chemist and Program Manager**. Developed an automated gas chromatographic monitor for measurement of subparts per billion concentrations of toxic compounds in air. Managed a study of trace element emissions from a coal-fired power boiler and a study of sulfur dioxide conversion in the stack of an oil-fired power boiler.

1976-1977 Florida Department of Environmental Regulation, Tallahassee, Florida. **Senior Professional Engineer**. Prepared a rule regulating construction of complex sources of air pollution. Evaluated all applications for permits for state highway construction and prepared and presented expert testimony in several of these cases. Evaluated and recommended redistribution of the work load assignments of the technical staff. Evaluated the existing network of ambient air quality monitoring sites and recommended improved and new sites.

1975-1976 **Consulting Engineer**, Gainesville, Florida. Designed an ambient air quality monitoring and data acquisition system for a pulp mill, modeled the impact of point source emissions, and prepared applications to construct sources of air pollution. Prepared and presented short courses on ambient air monitoring and source emission testing for the staff of the Florida Department of Environmental Regulation.

1970-1972 University of Florida, Gainesville, Florida. **Graduate Student**. Researched photochemical and dark reactions of the oxidation of sulfur dioxide to sulfuric acid in the presence of nitrogen dioxide and water and measured psuedo-first order rate constants at various water vapor concentrations. Also measured rate constants for the oxidation of sulfur dioxide in the presence of nitrogen dioxide and organic compounds. Identified nitrous oxide (N_2O) and methyl nitrate as products of the reaction of the sulfur dioxide-nitrogen dioxide-acetone system, and measured the rates of their formation.

1967-1974 Environmental Science and Engineering, Inc., Gainesville, Florida. **Senior Engineer and Group Leader**. Managed ambient air quality monitoring and source testing projects,

prepared environmental impact statements. Developed a continuous in-stack fluoride monitor and a static balance probe for source testing and studied the sorption of sulfur dioxide on various filter media. As a part-time project manager while in graduate school, prepared a state-of-the-art report on source testing methods. set up an analytical laboratory at a municipal refuse composting plant, and prepared a feasibility study of various methods of refuse disposal for a large southern city.

1965-1967 Duval Air Improvement Authority, Jacksonville, Florida. **Acting Director**. Developed rules and administrative procedures, supervised the technical staff, prepared budget requests, and reported directly to the policy making board. As Chief Chemist, established an ambient air quality monitoring network, analytical procedures, and supervised the staff chemists and technicians.

EDUCATION

B.S., Chemistry, 1964, University of Florida, Gainesville, Florida.
M.S.E., Environmental Engineering, 1968, University of Florida, Gainesville, Florida.
Ph.D., Atmospheric Chemistry, 1972, University of Florida, Gainesville, Florida.

PROFESSIONAL AFFILIATIONS

Registered Professional Engineer (Civil) in the State of Florida (14574)
Air and Waste Management Association
Sigma Xi
American Association for the Advancement of Science

Biographical Data

DONNA A. HURLEY

Chemist

EXPERIENCE SUMMARY

Performance of a diversity of wet chemical and instrumental methods of analysis of varying sample types, especially environmental samples in both laboratory and field settings. Conducting of treatability studies of industrial wastes. Laboratory management responsibilities over a range of concerns including technical, health and safety, financial and human resources.

EXPERIENCE RECORD

1989-Date Engineering-Science. **Senior Scientist** (1991-Date). Office coordinator of field activities for a long-term, multi-faceted ambient air monitoring project involving review of field data, auditing of field analytical system operation, troubleshooting of field equipment problems and facilitation in personnel issues.

Responsible for operation of Air Quality Laboratory, including upgrading laboratory equipment and capabilities, providing analytical and consultative services for special in-house studies and for field projects and overseeing all laboratory activities.

Serve as Laboratory Health and Safety Representative, co-writing the E-S Fairfax Laboratory Health and Safety Plan, initiating a Chemical Disposal Plan to professionally eliminate hazardous chemicals, teaching annual Laboratory Safety Training classes, maintaining updated MSDS and chemical inventory files, and assuring that laboratory practices comply with OSHA and ES safety regulations.

Staff Scientist (1989-1991). Performed method development and on-site set-up of three fully automated thermal desorption/gas chromatographic systems to analyze ambient air around a USEPA SUPERFUND site continuously over a period of 18 months for a number of volatile organic compounds. Prepared a fourth "contingency backup" system as well. Held formal training classes on-site to instruct technicians in the operation and calibration of these systems, as well as in chromatographic theory and data interpretation.

Performed analysis of environmental samples in Air Quality Laboratory by gas chromatograhpy, atomic absorption, UV-VIS spectrometry and wet chemical methods. Participated in emissions monitoring of stationary sources, performing on-site, real time gas chromatographic analysis and other instrumental analyses of various industrial air emissions.

1988-1989 Environmental Options. Rocky Mount, Virginia. **Laboratory Director.** Supervised all aspects of operation of a commercial full service laboratory.

Designed, implemented, and oversaw a laboratory QA/QC program in which QC data was calculated, graphed, flagged (when necessary) and filed by computer, enabling ease of data review and prompt corrective action.

Performed analysis of air, soils, waters, petroleum products, and hazardous mixtures by gas chromatography (FID, ECD, PID), inductively coupled plasma, atomic absorption, UV-VIS spectrometry, and wet chemical methods.

1984-1988 Olver Incorporated. Blacksburg, Virginia. **Assistant Laboratory Director** (1986-1988). Responsible for assigning weekly activities schedules, providing technical assistance for new methods, training new hires and troubleshooting equipment/method problems.

Oversaw laboratory QA/QC program, maintaining updated quality control charts for precision and accuracy, taking corrective action when needed and designing and implementing a preventative equipment maintenance program. Served as laboratory safety officer, assuring compliance with current OSHA regulations.

Performed treatability studies on a variety of industrial wastes including metal plating wastes, cyanide wastes, organic wastes, oil-laden coal mine drain-off, steel mill baghouse dust, auto painting wastes, highly colored fabric dye wastewaters, etc. Also performed special studies such as aluminum removal from a plastic recycling process. Often conducted on-site, pilot-scale runs of successful treatments. Wrote or co-wrote resulting treatability reports.

Senior Laboratory Technician (1985-1988). Specialized in atomic absorption techniques (flame, furnace, cold vapor mercury and mercury hydride). Assisted in treatability studies, toxicity studies and macroinvertibrate studies. Assisted fellow technicians with technical problems and in training new technicians.

Laboratory Technician (1984-1985). Performed analysis of waters, industrial and municipal wastewaters and sludges and soils using atomic absorption, wet chemical and biological techniques.

EDUCATION

B.S., Chemistry, 1984, Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

AFFILIATIONS

Member of the American Chemical Society and the ACS Division of Environmental Chemistry.

ATTACHMENT B

AEHA STEM Method

UNITED STATES ARMY ENVIRONMENTAL HYGIENE AGENCY

ABERDEEN PROVING GROUND, MD 21010-5422

QUALITY ASSURANCE/QUALITY CONTROL
PROJECT PLAN

SAMPLING AND ANALYSIS OF STACK
EMISSIONS FOR ENERGETIC COMPOUNDS

U.S. ARMY ENVIRONMENTAL HYGIENE AGENCY
AIR POLLUTION ENGINEERING DIVISION
ABERDEEN PROVING GROUND, MARYLAND

SECTION 1. PROJECT TITLE AND PLAN APPROVALS

1.1. Project Title: Sampling and Analysis of Stack Emissions for Energetic Compounds

1.2. Approval Signatures:

Director of Environmental Quality — 11 Apr 88 — Date

Chief, Air Pollution Engineering Division — 8 June 88 — Date

Chief, Source Surveillance Branch — 2 June 88 — Date

Director of Laboratory Services — 26 Jul 88 — Date

Chief, Organic and Environmental Chemistry Division — 22 Jun 88 — Date

Chief, Analytical Quality Assurance Office — 24 June 88 — Date

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SECTION 3. PROJECT DESCRIPTION

3.1. Purpose. The purpose of this quality assurance plan is to delineate the sampling and analytical methodologies to be utilized in obtaining emission data for energetic materials (organic compounds) emanating from Army operations. The plan specifically describes the quality assurance/quality control procedures which are to be utilized in a sampling and analysis program to ensure a high degree of data quality.

3.2. Background. The U.S. Army conducts a number of operations which result in the potential for energetic compounds to be released to the atmosphere via stack emissions. Of these operations, the incineration of obsolete or off-specification munitions, propellants or explosives represents the greatest workload for sampling and analysis by USAEHA. Some of these explosives or energetic materials are classified as reactive hazardous wastes by regulations promulgated under the RCRA (40 CFR 261, reference 2). Therefore, the incinerators used for the disposal of these munitions and energetic materials are classified as hazardous waste incinerators and must comply with all appropriate RCRA regulations. In order to comply with these regulations, the U.S. Army will need to conduct trial burns on all those incinerators burning hazardous waste munitions and energetic material in order to demonstrate the performance standards of 40 CFR 264 (reference 3). Of these performance standards, the most critical standard to evaluate is the destruction and removal efficiency (DRE) of the designated Principal Organic Hazardous Constituent (POHC).

3.2.1. In the early 1980's, the USAEHA, in anticipation of this sampling requirement, initiated a project to evaluate and develop the sampling and analytical methodologies to complete the necessary trial burns on Army incinerators for the disposal of energetic material and munitions. This method evaluation and development was necessary since no standard reference method existed in order to conduct this type of sampling and subsequent analysis. Suggested methods, published by the EPA, were used as the reference point for the evaluation/development project. The project was directed at finding the optimum sampling and analytical methodologies for those potential POHC's found most frequently in energetic material feed items. The results of this validation are contained in Appendix A.

3.2.2. Work completed thus far has yielded sampling and analytical methodologies that are suitable for the following compounds:

- o nitroglycerin (NG)
- o 2,4-dinitrotoluene (2,4-DNT)
- o 2,6-dinitrotoluene (2,6-DNT)
- o 2,4,6-trinitrotoluene (2,4,6-TNT)
- o cyclotrimethylene trinitramine (RDX)

and for surrogates of the above compounds:

- o ethylene glycol dinitrate (EGDN)
- o 3,4-dinitrotoluene (3,4-DNT)
- o 2,4,5-trinitrotoluene (2,4,5-TNT)

3.3. Overview. Quality assurance/quality control procedures have a number of purposes:

3.3.1. Document the quality of the data.

3.3.2. Maintain the desired quality of data for the entire sampling and analysis project.

3.3.3. Provide guidelines for corrective actions when it becomes apparent that the procedures are "out of control."

The terms quality assurance and quality control are typically used together because of their complimentary nature, but they do represent different activities. Quality assurance is a systematic approach for ensuring that all the data and results compiled under a project are statistically valid and properly documented. These procedures include the delegation of responsibilities of the program, program audits, and data and documentation reviews. On the other hand, quality control is the mechanism through which QA achieves its goals. The maintenance performed on equipment, sample integrity, performance of specific sampling and analytical methods, and the tracking of data precision and accuracy are all QC procedures.

3.4 Document Organization. The main body of this plan discusses the procedures to be utilized in the sampling and analysis projects for quantifying the emission of energetic materials from sources. The Appendices provide detailed information that supports these procedures. Results of the sampling/analysis validation study are reported in Appendix A. Detailed analytical procedures documentation requirements and calculations are discussed in Appendices B, C, and D, respectively. To facilitate reading the QA/QC Project Plan, a Glossary of Terms and a List of Acronyms are provided in Appendices E and F. References are listed in Appendix G.

SECTION 4. PROJECT ORGANIZATION AND RESPONSIBILITIES

4.1. Introduction. QA/QC is the responsibility of each group involved in the project for their own respective work. No specific QA/QC coordinator will be appointed for the purposes of energetic material sampling and analysis projects. Each project officer retains the ultimate responsibility for the implementation of QA/QC procedures, and his/her signature on the final report attests to the review and assurances of the quality of the data. Coordinators, whose responsibility is QA/QC, currently exist within the analytical and sampling branches. Their responsibilities are not expanded by the implementation of this QA/QC plan for energetic material sampling and analysis. A schematic diagram of the relationships existing between the various functions involved in this program are illustrated in Figure 4.1.

4.2. Sampling Phase.

4.2.1. Program Manager. The program manager is responsible for the overall technical and administrative direction of the energetic material sampling/analysis project to include QA/QC. He is also responsible for ensuring adequate resources are available to comply with project scheduling and technical constraints. Furthermore, he is responsible for conducting performance and system audits of the sampling phase of the project.

4.2.2. Project Engineer. The project engineer reports to the program manager and is responsible for the implementation of the sampling program as outlined in the test protocol. Some of the project engineer's responsibilities include:

4.2.2.1. Managing the activities of the stack sampling team and the onsite analytical personnel.

4.2.2.2. Monitoring operations to assure that process conditions established for the test are maintained and that required operational data are properly recorded.

4.2.2.3. Obtaining from incinerator operators the appropriate operational data from the daily test activities.

4.2.2.4. Deciding if and when a sample run should be interrupted or terminated.

4.2.2.5. Monitoring to ensure field QA/QC procedures are being implemented.

4.2.2.6. Implementing the safety plan for the test activities.

4.2.2.7. Reporting of test activities, data, and operational/analytical results to the project manager.

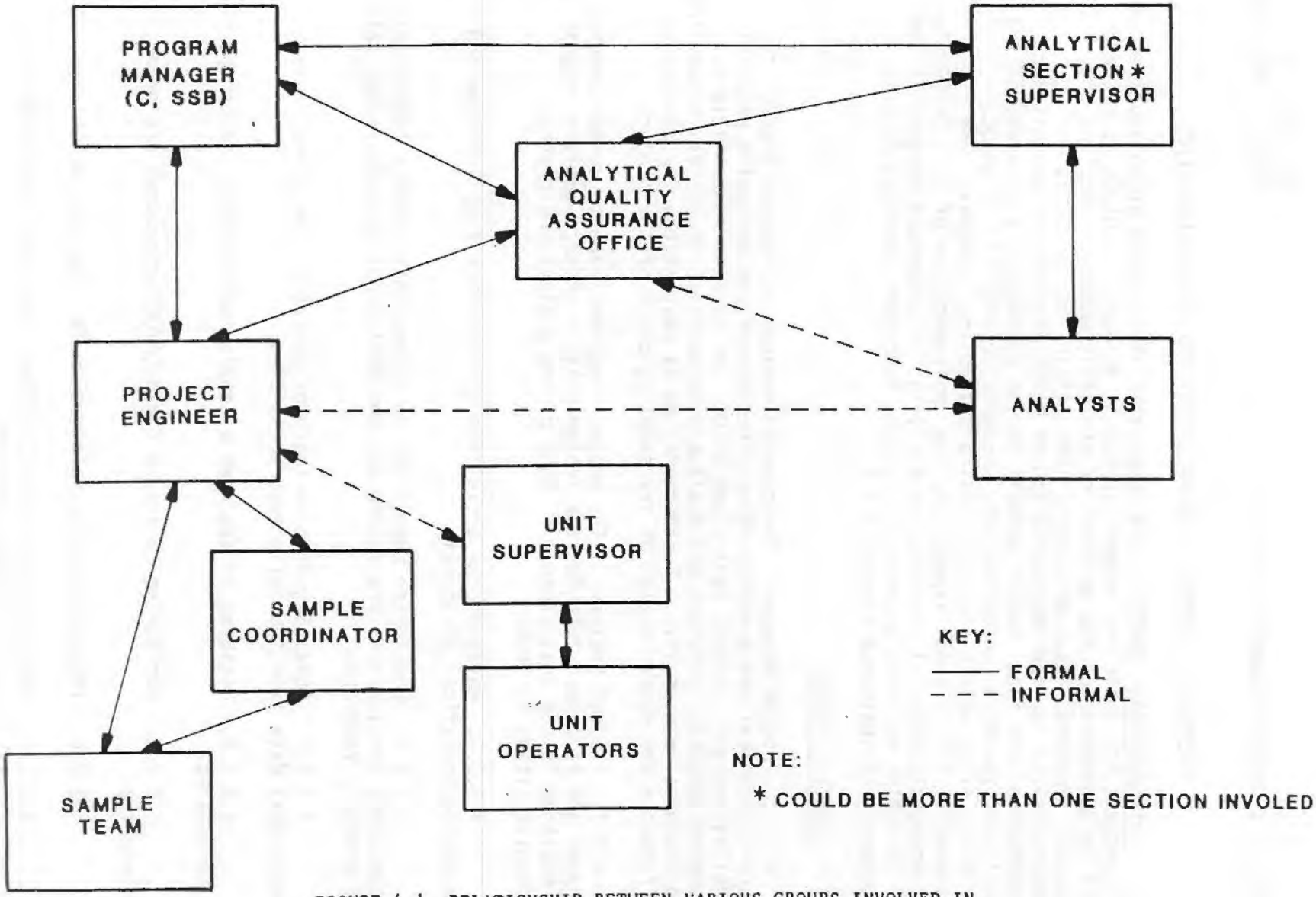

FIGURE 4.1 RELATIONSHIP BETWEEN VARIOUS GROUPS INVOLVED IN SAMPLING/ANALYSIS PROJECT

4.2.3. Sample Coordinator. The sample coordinator reports to the project engineer during field activities and has overall responsibility for the field activities related to sample handling, packaging, cataloging, and shipping. His primary responsibility is to assure that proper chain of custody of samples is maintained at all times. He will accomplish this by either maintaining custody of the samples himself or assuring the proper chain of custody documentation exists. Some of the additional responsibilities include:

4.2.3.1. Assigning and recording sample numbers.

4.2.3.2. Assuring only authorized personnel handle samples before, during, and after testing.

4.2.3.3. Overseeing sample preservation in the field.

4.2.3.4. Documenting sampling activities in a field log book along with sample container information.

4.2.3.5. Preparing samples for shipment.

4.2.3.6. Requesting sample analyses.

4.3. Analytical Phase.

4.3.1. Laboratory QA Officer. Sample custody in the laboratory will be maintained by the AQAO which receives and logs samples, inserts QA/QC samples, and distributes samples to the appropriate analytical section. This officer will also be responsible for validating all analytical data as it pertains to the inserted QA samples. Following a review of the entire analytical package for data completeness, the laboratory QA officer will transmit this package to the project engineer. Audits of the analytical and subsequent data reporting phases of the project will be conducted by AQAO.

4.3.2. Analytical Section Supervisor. Once a sample or group of samples is distributed to the appropriate analytical section, the supervisor of that section is responsible for sample custody. Samples will either be in his custody or the custody of his assigned analyst or secured under lock and key. The supervisor is also responsible for:

4.3.2.1. Directing the distribution of samples within his laboratory section.

4.3.2.2. Monitoring and verifying that the specified analytical and QA/QC procedures are being followed by the analyst.

4.3.2.3. Reviewing the analytical and QA/QC data and notifying the project engineer if data quality appears to warrant repeat analysis of any or all samples within his section.

4.3.2.4. Transmission of analytical and QA/QC data to the laboratory QA officer (AQAO).

4.3.2.5. Ensuring adequate resources are available to comply with project scheduling and technical constraints.

4.3.3. Project Chemist. The project chemist reports to the analytical section supervisor and is responsible for the analysis of all samples (both field and QA/QC samples). Some of the project chemist's responsibilities include:

4.3.3.1. Preparation of calibration and standard solutions and QC samples.

4.3.3.2. Calibration of the gas chromatographic instruments and preparation of calibration curves.

4.3.3.3. Preparation of field samples.

4.3.3.4. Analysis of samples.

4.3.3.5. Interpretation of chromatograms and analytical data, to include calculation of surrogate recoveries.

SECTION 5. QUALITY ASSURANCE OBJECTIVES

5.1. Introduction. In order to provide data of acceptable quality, quality assurance and quality control (QA/QC) procedures must be implemented throughout the project. The goals of the QA/QC procedures are to ensure the accuracy, precision, completeness, and the quality of the data are within accepted tolerance limits, and to ensure the integrity of the samples is not compromised. The objectives of this program are discussed in this section while the implementation of the procedures is discussed in Section 11.

5.2. Definitions.

5.2.1. Accuracy. In terms of data analysis, accuracy is defined as the degree of agreement of a measurement or an average of measurements of the same parameter, X, with an accepted reference or true value, T. Typically, accuracy is expressed as the difference between X and T as a percentage of the reference or true value.

$$\text{Accuracy} = [(X-T)/T]x100$$

5.2.2. Precision. Precision is the measure of the variability among individual measurements of the same parameter. An indicator of precision is the relative standard deviation [coefficient of variation (CV)] and is used for determining the precision of multiple measurements of the same parameter.

$$CV = [(\text{standard deviation})/(\text{sample mean})] \times 100$$

5.2.3. Data Completeness. Completeness is defined in the EPA guidelines for QA plans (reference 8) as a measure of the quantity of valid data obtained from a measurement system compared to the quantity that was expected to be obtained under normal conditions.

5.3. Assessment of Accuracy.

5.3.1. Reference Materials. All reference materials used in the preparation of calibration standards or as surrogate compounds will be of the highest purity commercially available. Extreme high purity for the organic solvents is not crucial given the nature of the types of compounds of interest.

5.3.2. Instrument Performance. A daily calibration curve will be used to assess the operation of the gas chromatographic systems.

5.3.3. Recovery of Surrogates. The recovery objectives will be: the mean of all measurements to be greater than or equal to 100 ± 25 percent and the standard deviation to be less than or equal to 25 percent. The spiking solution of surrogate compounds will be analyzed to verify its concentration.

5.3.4. Recovery of Blind Spikes. All samples will be processed through an independent QA Coordinator (AQAO) at the laboratory who will renumber the samples (to ensure sample number singularity within the laboratory) and insert samples spiked with both POHC and surrogate compounds. The recovery objectives for these blind spikes will be: the mean percentage recovery of all blind spikes to be greater than or equal to 100±30 percent and the standard deviation to be less than or equal to 30 percent.

5.3.5. Confirmational Analysis. Ten percent of all field samples positive for POHC and/or surrogate will be submitted for the confirmation of the gas chromatographic analysis by gas chromatograph/mass spectrometry analysis.

5.4. Assessment of Precision.

5.4.1. Instrument Calibration. As discussed in paragraph 5.3.1, a daily calibration curve will be established for each analyte. The objective is to obtain a correlation coefficient for this calibration curve greater than or equal to 0.99.

5.4.2. Analysis of Field Samples. Approximately 10 percent of the field samples (in extract form) will be split and analyzed to provide precision data on the analysis of surrogates and native POHC's in that group found to contain either of these items. All field samples will be analyzed in replicate to provide precision data on the analysis. The target precision will be a relative standard deviation of less than or equal to 30 percent.

5.5. Data Completeness. During the emission test program, the completeness objective will be to obtain analytical results for at least 95 percent of the samples collected.

5.6. Contamination Assessment. Laboratory, field and method blanks of all types of solvents, the resin extracts, the impinger water extracts, and the filter extracts (refer to Section 6) will be provided to the laboratory along with the samples for analysis. These samples will be used to assess the level of contamination due to the field exposure and the handling and preparation within the laboratory.

5.7. Analysis Timetable. For two reasons (sample degradation and regulatory requirements), an additional QA/QC objective will be to obtain analytical results within 30 days following sample arrival at the central laboratory.

5.8. Summary. A matrix of the QA/QC objectives discussed in this section is contained in the following Table 5.1.

ABLE. 5.1. MATRIX OF QUALITY ASSURANCE OBJECTIVES

Procedure	Type of Determination	Goal	Paragraph Reference
1. Analytical			
a. Use of reference materials (calibration standards, surrogates, etc)	Accuracy	Highest purity commercial available	5.3.1
b. Renumbering of trial burn samples	NA	Ensure singularity of sample numbers	5.3.4
c. Insert blind QA samples Quantity = 10% of total number of samples (POHC and surrogates)	Accuracy	Mean recovery ≥ 100±30% Recovery standard deviation ≤ 30%	5.3.4
d. Split samples for replicate analysis. Quantity = 10% of number samples containing surrogate spikes.	Precision	RSD ≤ 30%	5.4.1
e. Daily calibration of analytical instrument GC/HPD Multipoint; GC/MS - single point	Accuracy Precision	Ensure peak performance of instrument. Correlation coefficient for calibration curve ≥0.99.	5.4.2
f. Submission of positive analyses for GC/MS confirmation. [10% of positive field samples]	Accuracy Confirmation	POHC confirmation/ verification	5.3.5
g. Analysis of laboratory/ method blanks	Level of contamination	Minimize contamination	5.6
h. Performance check on GC instruments	Accuracy Precision	Peak performance	9.4.1
2. Extraction - field extraction	NA	Place POHC in more stable matrix	N/A
3. Sampling			
a. Equipment calibrations	NA	Meet EPA-600/4-77-027b guidelines	9.2
b. Background run - no waste feed	NA	Assess background levels of POHC or interferences .	N/A
c. Surrogate spike of XAD®-2 resin; analysis	Accuracy	Mean recovery ≥ 100±30% Recovery standard deviation ≤ 30%	5.3.3 6.2.5
d. Analysis of field blanks	Level of contamination	Minimize contamination	5.6

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SECTION 6. SAMPLING PROCEDURES

6.1. Introduction. The POHC emissions are to be sampled in the exhaust gas stream with a modification of the EPA Method 5 sampling train. As with standard methodology, isokinetic sampling is conducted with the train.

6.2. USAEHA Sampling Train for Energetic Materials.

6.2.1. Components. The sampling train to be used for emission testing is shown in Figure 6.1. The components of this modification of the EPA Method 5 sampling train from inlet to outlet are as follows:

- Nozzle
- Pyrex®-lined probe
- Cyclone eliminator (optional)
- 4-inch glass-fiber filter
- 90-degree connector
- Impinger No. 1, dry
- 180-degree connector
- Impinger No. 2, dry
- 180-degree connector
- Impinger No. 3, dry
- 180-degree connector
- XAD-2 resin tube (vertical orientation)
- 180-degree connector
- Straight glass tube
- 180-degree connector
- Impinger No. 4, dry
- 180-degree connector
- Impinger No. 5, silica gel

6.2.2 Special Considerations.

6.2.2.1 The probe and filter housing will be assembled in a normal EPA Method 5 sampling box to allow heating of both the probe and the filter to 248 °F ± 25 °F. The impingers will be packed in an ice bath to provide the necessary cooling of the gas sample.

6.2.2.2 The three impingers placed prior to the resin tube are used to cool the gas to an acceptable temperature (<68 °F) and to remove moisture from the gas sample. The cooler temperature is necessary to ensure that the resin will function properly as a POHC collection medium. Depending upon the temperature and the moisture content of the stack gases, additional impingers may be added prior to the resin module. This decision will be made by the project engineer based upon preliminary measurements of or calculation of stack conditions. Recovery of the impingers before the resin module does not depend upon the number of impingers.

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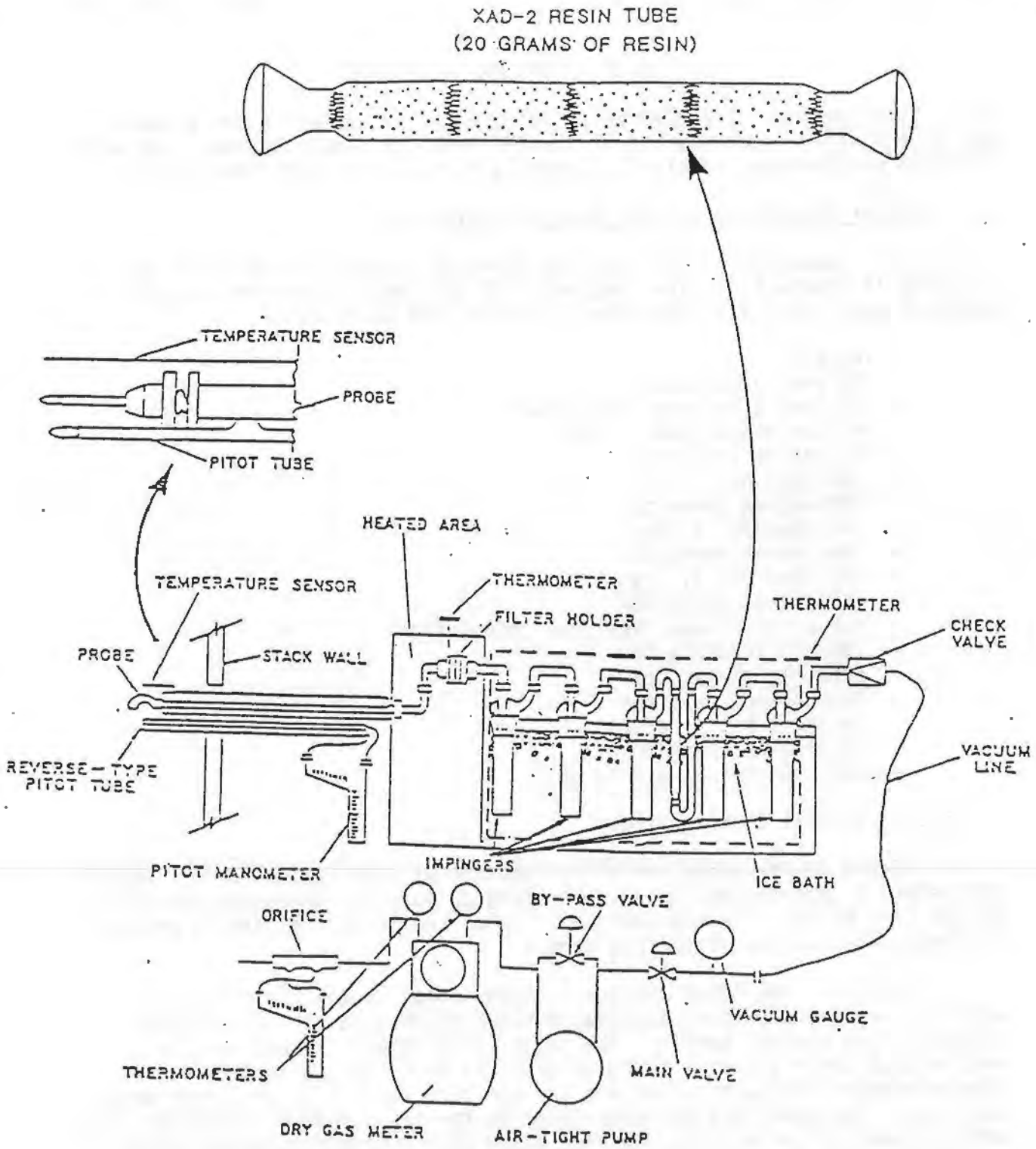

FIGURE 6-1. MODIFIED EPA METHOD 5 SAMPLING TRAIN

6.2.3. Sampling Preparations.

6.2.3.1. Glassware Cleanup (Field/Laboratory). All glassware to be used for sampling or sample recovery are considered clean for use once it has undergone a detergent wash in a sonic bath, a distilled water rinse, and then an acetone (reagent grade) rinse. Exceptions to this rule include the resin tubes and the separatory funnels. The resin tubes may receive the standard glass cleaning procedures, time permitting, but there is a limited number of tubes available, and there is a high potential for their breakage. The separatory funnels are so large that they would seriously slow down the glassware turnaround time through the sonic bath. Both the tubes and the separatory funnels may be cleaned by an abbreviated procedure. This procedure calls for one or two rinses with toluene followed by several acetone rinses. Acetone not only provides an additional solvent rinse to remove contaminating organic compounds, but helps remove the film that toluene creates on glass. Because of that film, any glassware that was rinsed with toluene or came in contact with toluene, and is scheduled to go into the sonic bath, should be rinsed with acetone first to help extend the useful life of the sonic bath solution. Additional glassware that may receive the abbreviated cleaning procedures could include other high use, limited number items such as graduated cylinders.

6.2.3.2. Resin Cleanup. XAD-2 resin is purchased by the USAEHA in a purified form (previously cleaned by soxhlet extraction). A portion of this resin (5 grams) will be extracted as per the procedure discussed in Section 8 of this plan to ensure no interference compounds exist on the resin which may impede the analysis at a later date. The resin is not reused, but cleanup may be required if the analysis so indicates. If the need for cleaning the resin ever arises, the procedures recommended in Appendix B of reference 5 will be utilized.

6.2.4. Sampling Train Assembly. Glass fiber filters [99.95 percent efficiency ($\leq$0.05-percent penetration) for 0.3 micron dioctyl phthalate smoke particles] for the USAEHA train configuration do not need to be preweighed, since they will undergo organic sample recovery immediately after sampling. Each impinger will be labeled with the run number and impinger number. Additionally, the other key components of the train (resin tube, filter housing, and probe wash recovery bottles) will also be labeled with the run number (paragraph 7.2.1). The resin tube will be packed with four 5.0 gram sections of XAD-2 resin with glass wool on both sides of each section. Following this packing, the sample flow direction will be marked by an arrow on the tube wall. Once the train components are assembled in the field laboratory, the impingers and resin tube will be weighted so the amount of moisture collected in the train during sampling may be determined at the end of the run. If the moisture gain in the resin tube frequently shows a small negative number, then that moisture value will be disregarded. Connecting glassware behind the resin section should be temporarily marked (e.g., with masking tape) to differentiate it from the glassware in front of the resin.

6.2.5. Sampling Train Spiking. A solution for a field surrogate spike of the sampling train will be prepared by the analytical laboratory in accordance with the procedures in Appendix B. Table 6.1 contains a list of surrogate compounds to be utilized for varying POHC's. In the event of multiple POHC's, the sampling train should be spiked with a surrogate for each of the specified POHC's. The project engineer will determine the level of this spike and supply that value to the laboratory. To calculate the spike level, the project engineer must determine the quantity of the target compound captured by the sampling train in the sampling period for the case of the lowest quantity captured (e.g., highest DRE legitimately possible for the incinerator). The spike level will be at least four times this quantity. For example, assume for the case of an incinerator, the quantity of the target compound captured in a 1-hour period at 99.999 percent DRE is 50 µg. The minimum spike level should be 200 µg, 4 times the 50 µg level. When the sampling train is assembled, 50 mL of deionized water will be added to the first impinger to support a field spike. Half of the calculated spike level should be on the first resin section, placed there by direct injection, while the other half of the spike level will be in the 50 mL of water in the first impinger. The spike level for the entire train should not exceed 1000 µg.

TABLE 6.1. LIST OF POHC's AND SURROGATE COMPOUNDS

Principal Organic Hazardous Constituent	Surrogate Compound
Nitroglycerin	Ethylene glycol dinitrate
2,4-Dinitrotoluene	3,4-Dinitrotoluene
2,6-Dinitrotoluene	3,4-Dinitrotoluene
2,4,6-Trinitrotoluene	2,4,5-Trinitrotoluene
Cyclotrimethylene-trinitramine (RDX)	

6.2.6. Sampling Procedures. The actual sampling operation is conducted using the standard EPA Method 5 sampling procedure as described in references 1 and 4. The Method 5 procedures are utilized for pretest and post-test leak checks, isokinetic sampling rate, and filter changes.

SECTION 7.0 SAMPLE CHAIN OF CUSTODY

7.1. Introduction. Sample custody is the responsibility of the sampling team during the transportation of the samples to and from the central laboratories and during the conduct of the trial burn sampling. At the central laboratories, the chain of custody will become the responsibility of the Quality Assurance organization within the central laboratory. In accordance with SW-846 (reference 6), a sample is considered to be under a person's custody if the sample is:

7.1.1. in that person's physical possession

7.1.2. in view of that person after acquiring possession

7.1.3. secured by that person so that no one can tamper with the sample

7.1.4. secured by that person in an area which is restricted to authorized personnel

These criteria will be used to define the meaning of "custody" and ensure the integrity of the trial burn samples from collection to data reporting. Limited access to the samples is an integral part of the chain of custody procedures.

7.2. Chain of Custody Documentation.

7.2.1. Labeling.

7.2.1.1. Sample Containers. Sample container labels will be affixed to sample containers prior to or at the time of sampling. The labels will be filled out at the time of the sample collection or sample recovery. Gummed labels or tags are adequate and will include the following information at a minimum: sample number; date and time of recovery; and installation. The sample will be annotated if any evidence exists which suggests that sample may be hazardous (such as extremely high explosive content).

7.2.1.2. Sampling Train Components. Impingers, XAD-2 resin modules, gas sample bags (Orsat), and glass fiber filter cartridges will be labeled with tape to indicate the run number prior to sampling. If more than one train is being utilized in simultaneous testing, these train components should also be labeled to indicate the type of train. Since samples will be recovered from each of these components immediately after the test run in the field laboratory, no additional labeling will be necessary.

7.2.2. Sample Seals. Sample seals will be placed over the top of the sample containers to detect unauthorized sample handling. Gummed labels or equivalent are acceptable for this purpose. The seal will include the following information: sample number; and the signature of person recovering the sample.

7.2.3. Field Log Notebook. A permanently bound field log notebook will be maintained by the Sample Coordinator. The Sample Coordinator will enter data from the chain of custody sheets. The following information will be included:

General Entries:
- Purpose of sampling (e.g., RCRA sampling)
- Installation
- Facility
- Project number
- Project officer
- Sample coordinator

Specific Entries Per Test Run:
- Date of run
- Type of sampling train
- Filter number
- Resin module number
- Run number
- Waste feed
- Orsat analytical data
- Moisture data
- Sample data from Field Sample Custody Sheets
  - Sample number
  - Component
  - Volume or weight
  - Remarks
  - Technician recovering sample
  - Date samples recovered
- Disposition of samples
  - Storage (location; locker; condition)
  - Shipment (means; person with custody)
  - Evidence that samples are or are not hazardous
- Signature of sample coordinator

All entries to the field log notebook will be made after sample recovery is complete and will provide a back-up to data and information contained on the looseleaf chain of custody sheets.

7.2.4. Chain of Custody Sheets. Custody of the samples will be documented using a series of chain of custody sheets which are described in paragraph 7.3. All chain of custody sheets will be retained by the Sample Coordinator.

7.3. Chain of Custody Procedures.

7.3.1. Presampling Procedures.

7.3.1.1. The chemical laboratory technician will collect precleaned sample bottles and containers for the following samples: probe washes, resin, impinger solutions, and miscellaneous source samples (e.g., ash) and additional sample bottles for QA/QC samples and blanks.

7.3.1.2. The chemical laboratory technician will prepare and pack glass fiber filters for the sampling. These will be placed in numbered petri dishes. Laboratory solutions (acetone, distilled/deionized water, and solvent) and resins will also be packed. The resin will also be placed in a numbered module and sealed. The sample numbers for the resin modules and filter containers, and the lot numbers for the various solvents will be recorded in the laboratory notebook.

7.3.1.3. Impingers, beakers for probe washes, separatory funnels, and other glassware will be cleaned and packed for shipment.

7.3.1.4. After cleaning, the above items will be stored in a locked laboratory in the custody of the senior chemical laboratory technician until the shipment to the test site.

7.3.1.5. Shipment of equipment will generally be accomplished in a mobile laboratory. The vehicle will be locked at all times when the driver is not in the vehicle. Once the mobile laboratory is onsite, all equipment and sample containers will be stored in a secure area until the Sample Coordinator assumes custody.

7.3.2. Onsite Sample Train Preparation.

7.3.2.1. The chemical laboratory technician will prepare the following for each particulate and POHC sampling train:

7.3.2.1.1. Impingers, each appropriately labeled [see paragraph 7.2.1.2.].

7.3.2.1.2. XAD-2 resin module for POHC sampling train, labeled.

7.3.2.1.3. Glass fiber filter, labeled.

7.3.2.1.4. Gas sampling bag (Orsat), labeled.

7.3.2.2. The chemical laboratory technician will enter the components of each train on the Sample Train Custody Sheet shown in Figure 7.1 and will sign in the appropriate space for each component prepared. Each component must be included along with any labeling information.

7.3.2.3. The engineering technician taking the sampling train to the sampling site will sign for each component received from the laboratory technician (Figure 7.1).

7.3.2.4. If, during the sampling run, any components are broken, plugged, etc., the person who signed for the sampling train will sign in the Remarks Section whether or not the component has been removed. The chemical laboratory technician will prepare and list a new component. The engineering technician receiving the new component will again sign for that new item.

7.3.3. Sample Recovery. The following sample recovery procedures are applicable to both the POHC and the particulate sampling trains:

7.3.3.1. When sample trains are returned from the sampling site, the chemical laboratory technician receiving the train will sign for each component on the Sampling Train Custody Sheet (Figure 7.1).

7.3.3.2. The sampling trains are then broken down and the various samples recovered. These samples are then recorded on the Field Sample Custody Sheet (Figure 7-2). Each chemical laboratory technician will maintain a separate data sheet for the samples that he recovers. As each sample is recovered, the technician will label and seal the samples as per paragraph 7.2 above.

7.3.3.3. After sample recovery is complete, custody of the samples will be transferred to the Sample Coordinator by executing the bottom portion of the Field Sample Custody Sheet (Figure 7-2).

7.3.3.4. The Sample Coordinator will consolidate the samples in the storage area of the mobile laboratory. The samples will either be in his view, in the view of other authorized test team members, or be under lock and key until these samples are shipped to the central laboratory.

7.3.3.5. All chain of custody sheets will be maintained by the Sample Coordinator.

7.3.3.6. After all the samples from an individual run are recovered and in the custody of the Sample Coordinator, he will enter the data into the Field Log Notebook as required by paragraph 7.2.

7.3.4. Sample Shipment. Samples will generally be returned to the USAEHA Laboratory (Aberdeen Proving Ground, Maryland) in the mobile laboratory. Prior to shipment, the Sample Coordinator will prepare a Consolidated Sample Custody Sheet (Figure 7-3) for each shipping container. Receipt of the samples will be acknowledged by the driver on this custody sheet. The custody sheet will be prepared in duplicate. A copy will be retained by the driver and the original will be retained by the Sample

SAMPLING TRAIN CUSTODY

INSTALLATION: _____ RUN NO. _____

PROJECT OFFICER: _____ PROJECT NO. _____

SAMPLING TRAIN COMPONENTS (List component and identification number)	REMARKS

Train Prepared By _____ Date/Time _____ / _____

Train Received By _____ Date/Time _____ / _____

Train Relinquished By _____ Date/Time _____ / _____

Train Received By _____ Date/Time _____ / _____

FIGURE 7-1. SAMPLING TRAIN CUSTODY SHEET

FIELD SAMPLE CUSTODY

INSTALLATION: _____ RUN NO. _____

PROJECT OFFICER: _____ PROJECT NO. _____

SAMPLE NO.	COMPONENT DESCRIPTION	VOL/WT	REMARKS

Samples Recovered By _____ Date Samples Recovered _____

Samples Received By _____ Date/Time _____/_____

FIGURE 7-2. FIELD SAMPLE CUSTODY SHEET

CONSOLIDATED SAMPLE CUSTODY

INSTALLATION: _____ PROJECT NO. _____

PROJECT OFFICER: _____

SAMPLE NO.	COMPONENT DESCRIPTION	VOL/WT	REMARKS

Relinquished By _____ Date/Time ____/____ Received By _____

Relinquished By _____ Date/Time ____/____ Received By _____

Relinquished By _____ Date/Time ____/____ Received By _____

Relinquished By _____ Date/Time ____/____ Received By _____

FIGURE 7-3. CONSOLIDATED SAMPLE CUSTODY SHEET

Coordinator. The samples will be locked in the mobile laboratory during shipment. Upon arrival at USAEHA, the samples will be returned to the Sample Coordinator for further distribution to the AQAO (POHC samples) or to the chemical laboratory technicians (particulate samples). The Consolidated Sample Custody Sheet will reflect all changes in custody (Sample Coordinator to driver; driver back to Sample Coordinator; Sample Coordinator to AQAO/lab technicians).

7.3.5. Laboratory Analysis (POHC Train Samples).

7.3.5.1. The POHC train samples will be transferred by the Sample Coordinator to the AQAO along with the Request for Chemical Analysis (Figure 7-4). The AQAO Office will log the samples into a central log book and assign laboratory sample numbers. These laboratory sample numbers will serve as a means of tracking samples through the laboratory analysis. Each sample number is unique and nonrecurring. The samples will be kept in a locked laboratory or cabinet pending transfer to the analytical laboratory.

7.3.5.2. Chain of custody from the QA Office to the analytical laboratory will be maintained on the laboratory chain of custody record (Figure 7-5). The samples, along with QA/QC samples, will be signed for by the supervisor of the analytical laboratory or his designated representative. The sample numbers are then logged into the laboratory computer along with the name of the assigned analyst. The samples are maintained in the custody of the analyst or under lock and key.

7.3.5.3. Laboratory analytical results are reported to the QA Office where the results will be analyzed, matched to initial sample numbers, and reported to the test project engineer.

7.3.6. Sample Inspection. Each individual receiving custody of the samples will inspect the samples for leakage and broken seals or any evidence of tampering. Discrepancies between information on sample labels and seals and chain of custody documentation should also be determined. All discrepancies should be reported to the Sample Coordinator. Analysis of samples should not be performed until all discrepancies have been resolved.

REQUEST FOR CHEMICAL ANALYSES				DATE	Page of
TO (Division)		FROM (Division)		POINT OF CONTACT	PHONE NO.
PROJECT NUMBER		SAMPLED INSTALLATION			ARLOC
REMARKS:					
REQUESTOR SAMPLE NUMBER *a*	NUMBER OF CONTAINERS SUBMITTED *b*	DATE AND HOUR COLLECTED *c*	SAMPLE POINT DESCRIPTION *d*		TYPE OF SAMPLE *e*

AERA Form 75, 1 Jun 81 (DISPOSAL)

FIGURE 7-4. REQUEST FOR CHEMICAL ANALYSES

US ARMY ENVIRONMENTAL HYGIENE AGENCY

CHAIN OF CUSTODY RECORD

INSTALLATION			COLLECTION DATE/TIME			TYPE OF SAMPLE
SITE IDENTIFICATION			ANALYTICAL QUALITY ASSURANCE OFFICE NUMBER			LABORATORY NUMBER
RELINQUISHED BY SIGNATURE	DATE	TIME	RECEIVED BY SIGNATURE	DATE	TIME	ANALYSES PERFORMED BY RECEIVER

AEHA Form 235, 1 Dec 83 (HSHB-OT)

FIGURE 7-5. LABORATORY CHAIN OF CUSTODY RECORD

SECTION 8. SAMPLE RECOVERY AND ANALYTICAL PROCEDURES

8.1. Introduction. The sampling train has been described in detail in Section 6 of this QA plan and for the purposes of sample recovery procedures has three major sections. The first section is the filter and all the components preceding it. The second major section starts with the back half of the filter housing and includes all the following glassware (mainly the three impingers) up to the resin module. The third section is the resin module itself. While there are two impingers that follow the resin module, they are not major features of the POHC sampling train since their only function is moisture removal.

8.2. POHC Sampling Train Recovery Procedures.

8.2.1 The initial sample recovery starts outside the field laboratory as the train is partially disassembled. The probe is removed from the sampling train and capped. The first and last impingers are also capped. The sample box containing the impingers and the resin module and the sampling probe are brought into the field laboratory for sample recovery. In the field laboratory, the nozzle, probe liner, and the front half of the filter housing are rinsed with the recovery solvent (see Table 8.1), the solvent volume is measured, and the liquid is placed in a sample container. The filter is removed from its housing and immersed in the recovery solvent in the same container as the rinse. An additional rinse of the probe liner/nozzle section with acetone is performed and is placed in a separate container. This rinse will provide a QA check on the rinse/recovery effectiveness. Figure 8-1 diagrams the recovery of the front section of the sampling train.

TABLE 8-1. EXTRACTION SOLVENTS UTILIZED FOR VARIOUS POHC's

Principal Hazardous Organic Constituent	Surrogate Compound	Extraction Solvent
Nitroglycerin	Ethylene glycol dinitrate	Toluene
2,4-Dinitrotoluene	3,4-Dinitrotoluene	Toluene
2,6-Dinitrotoluene	3,4-Dinitrotoluene	Toluene
2,4,6-Trinitrotoluene	2,4,5-Trinitrotoluene	Toluene
Cyclotrimethylene-trinitramine (RDX)		Toluene/ Iso-amylacetate (5:95 by volume)

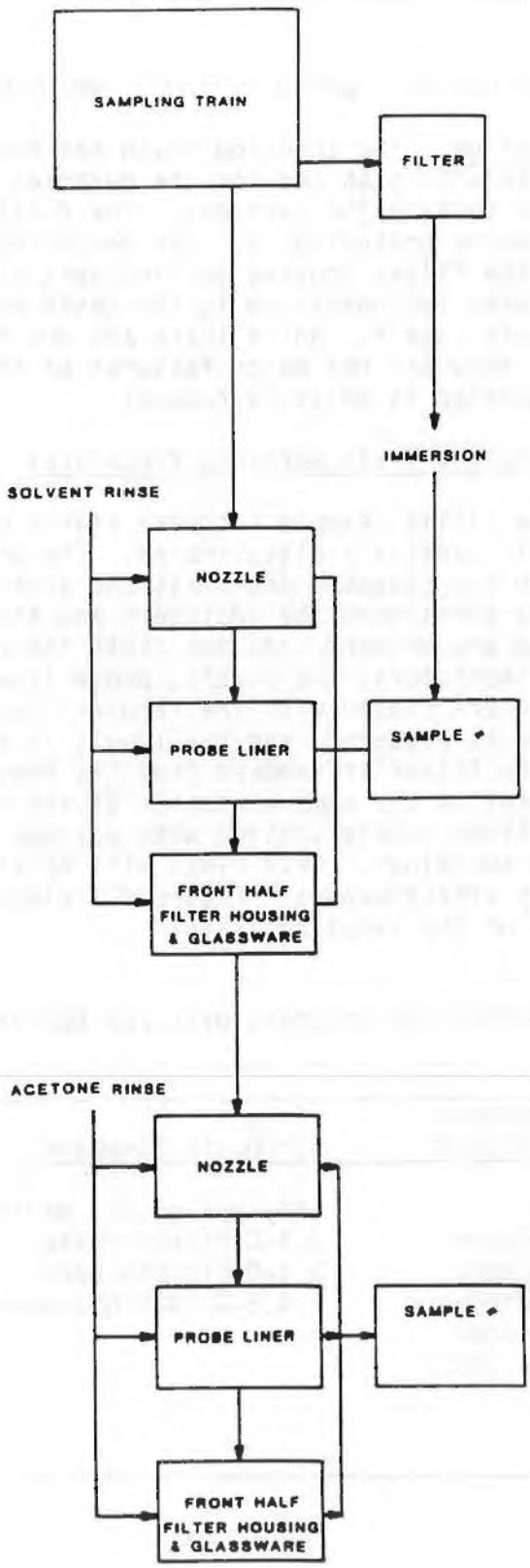

FIGURE 8-1. SAMPLING TRAIN FRONT SECTION RECOVERY

8.2.2 Each of the impingers are weighed prior to further organic recovery steps in order to determine the exhaust gas moisture content. Once completed, the condensate is collected and each of the first three impingers, and their associated connections are rinsed with distilled and deionized water. The rinse and condensate are combined and extracted three times. These extractions are completed in separatory funnels using shakeout techniques. Solvent volumes are based on total water volume to be extracted at a ratio of 4 to 1 (water to solvent). The extraction procedure consists of shaking the separatory funnel and its contents for three minutes, venting frequently. The organic and water layers should be allowed to separate to some degree, followed by 3 more minutes of shaking. This settling/shaking should be conducted one additional time and then the two layers allowed to separate to a narrow interfacial band if possible. The organic sample is obtained following this third shaking/settling. The first two extracts are completed at neutral pH conditions, while the water is acidified for the third extraction by adding concentrated sulfuric acid. The impingers and associated glassware are then rinsed with acetone to provide a QA check on the water rinse/recovery procedure. Impinger recovery is shown schematically in Figure 8.2.

8.2.3 The resin module is recovered in four sections and each section generates three samples. The extraction solvent (30 mL) is added to each resin section and glass wool plug, and the samples are then subjected to mechanical shakeout procedures (30 minutes). A portion of the solvent solution is removed to become the first sample (10 mL: sample extract number 1) and additional solvent is added to the resin section (10 mL). The resin sample then receives another mechanical shakeout followed by removal of the extract (10 mL: sample extract number 2). Additional solvent (10 mL) is then added to the resin. Following further shakeout, the third sample extraction is completed. The solvent and the resin are left combined for additional extraction (contact) time. The third extract is separated from the solvent/resin mixture at the USAEHA laboratories. This third extraction is completed just to verify the recovery completion. Resin module recovery is shown schematically in Figure 8-3.

8.3. Analytical Procedures.

8.3.1. In general terms, the methodology for analyzing the various samples generated by the recovery of the stack gas sampling train will utilize gas chromatography. The GC's chromatographs will be equipped with either an electron capture or a nitrogen/phosphorus detector and will utilize capillary column chromatography. Individual samples will be analyzed from each portion of the sampling train. No sample combinations or volume reductions will be utilized for samples showing positive analyses. Volume reduction procedures may be utilized for samples that have no detectable or quantifiable analyte. After all the samples have been analyzed via gas chromatography, a portion of these samples will be submitted for confirmational analysis on a GC/MS. This analysis has the confirmation of the GC peak as its primary objective, but in those cases where the concentration of the target analytes (POHC and surrogate) is large enough to overcome the sensitivity constraints of the GC/MS, the analysis will be utilized for the confirmation of the quantitation. The analytical procedures are discussed in detail in Appendix B of this plan.

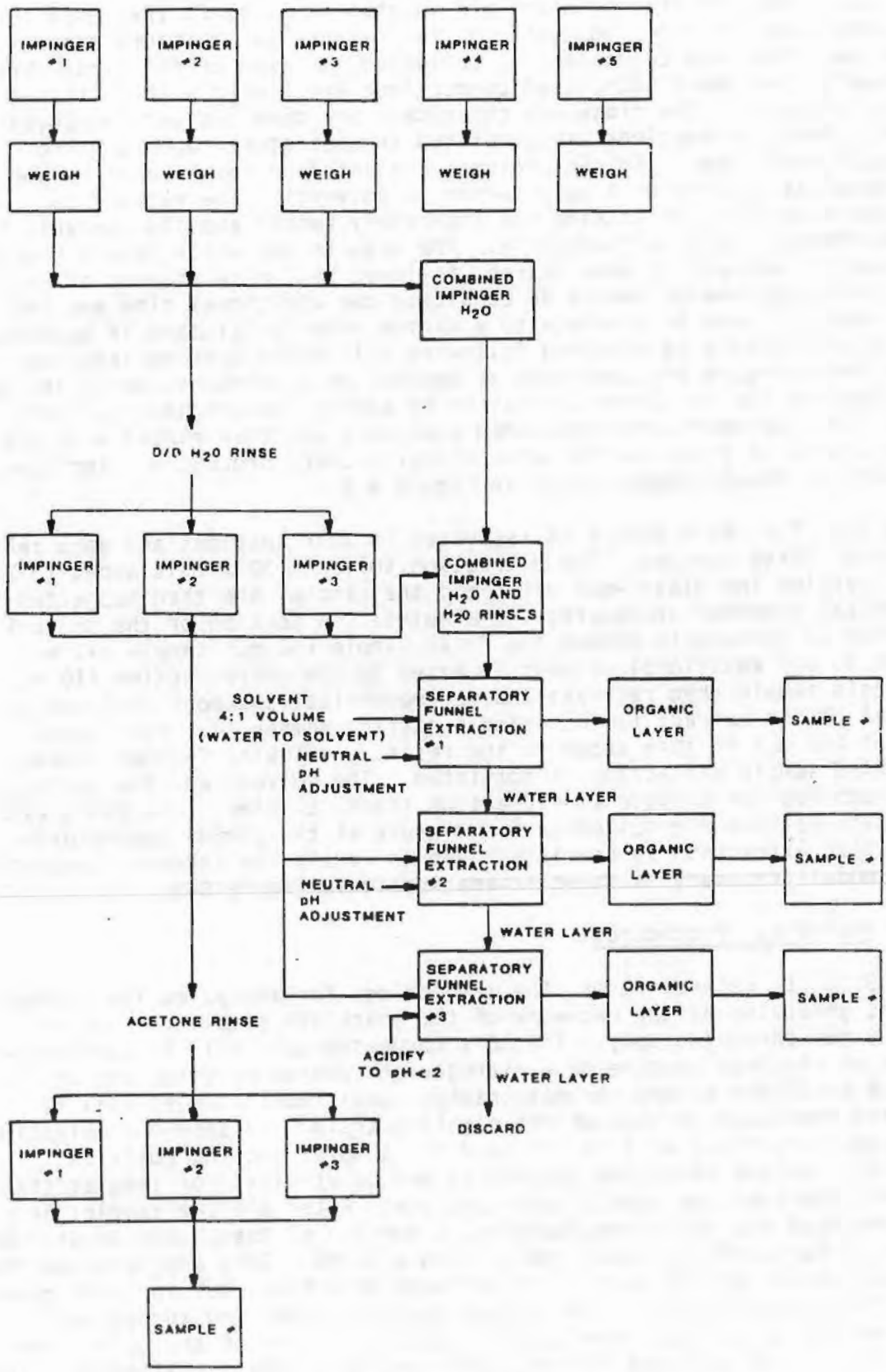

FIGURE 8-2. SAMPLING TRAIN IMPINGER SECTION RECOVERY

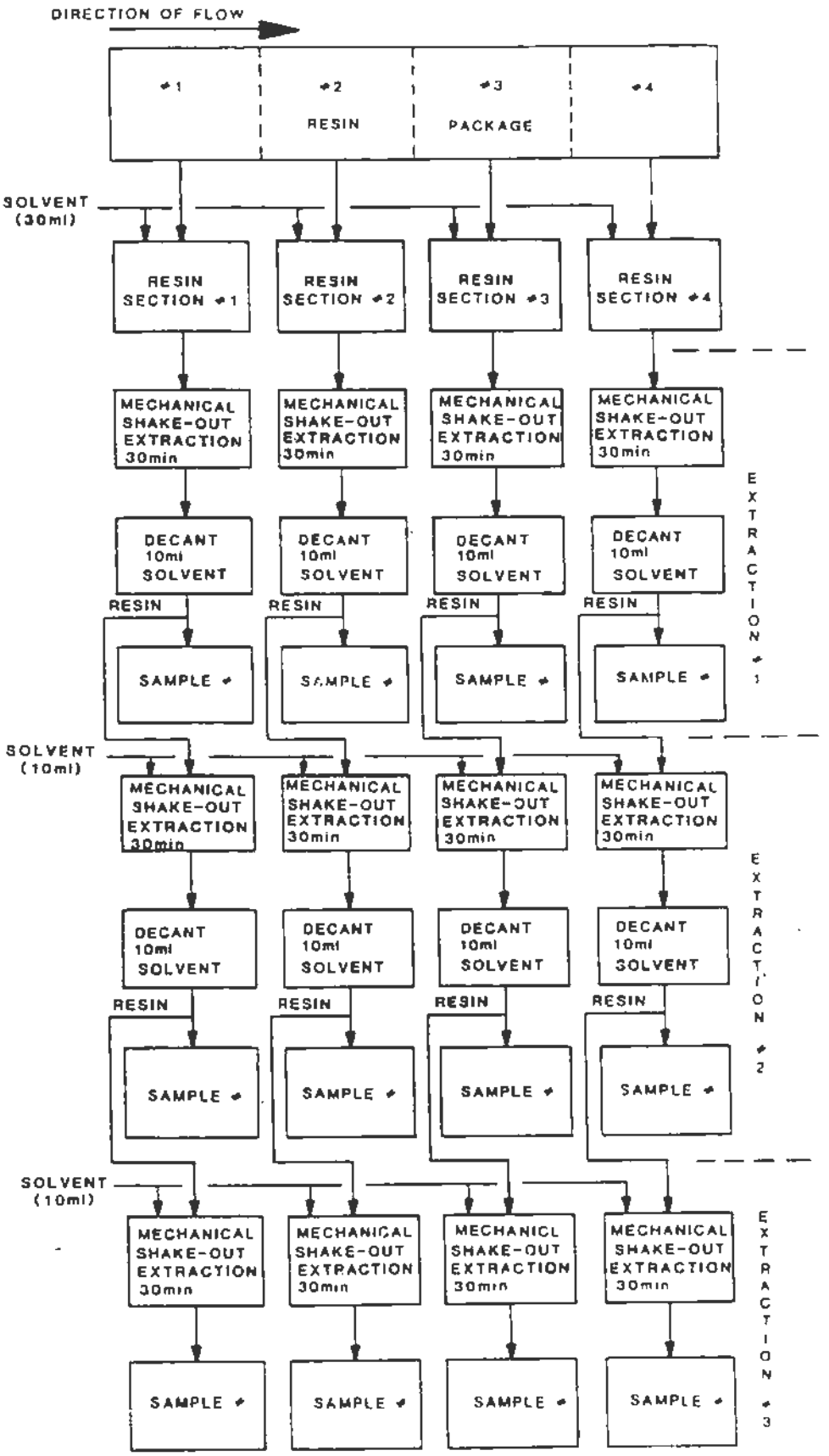

FIGURE 8-3. SAMPLING TRAIN RESIN MODULE RECOVERY

8.3.2. In order to determine a material balance on the energetic compounds, all residues from the process must be collected in addition to the stack gas sampling. Residues will be either shakeout extracted (paragraph 8.2.3) or liquid-liquid extracted (paragraph 8.2.2) depending upon their matrix, and the resulting solution will be analyzed by gas chromatography. The same analytical procedures will be utilized as with the sampling train samples.

SECTION 9. CALIBRATION PROCEDURES AND FREQUENCY

9.1. Introduction. The calibration procedures for the sampling and analytical instruments used in this project are extracted from the method documents referred to in this section of this plan and the sections describing the sampling and analysis.

9.2. Stack Sampling Equipment. The sampling equipment to be used for POHC determination is a modification of the Method 5 sampling train, with the only changes being in the impinger configuration, lack of water or solutions in the impingers, and the addition of the XAD-2 resin cartridge (see Section 6 for detailed discussion of sampling train). The actual hardware (such as pumps, dry gas meters, Pitot tubes, and manometers) to be used with both sampling trains is still subject to the requirements of the EPA as far as calibrations and QA procedures are concerned. This involves calibrations prior to and after testing according to the procedures outlined in 40 CFR 60 and the EPA Quality Assurance Handbook (references 1 and 4). This calibration includes the probe nozzle diameter measurements and pitot tube, dry gas meter, thermometer, and thermocouple/pyrometer system calibrations. Additionally, USAEHA routinely participates in the EPA national QA audit for dry gas meters.

9.2.1. Dry Gas Meter. The dry gas meter is calibrated prior to and following the emission testing using a wet test meter. For the post test calibration, three checks are made with the orifice set at its average value and with the maximum value of vacuum for all the runs.

9.2.2. Orifice. For the initial calibration, the orifice for the dry gas meter system is calibrated at manometer settings ranging from 0.3 to 5.0 inches of water.

9.2.3. Nozzle. The diameter of the particulate sampling nozzle is measured with a micrometer accurate to 0.001 inch. Three measurements are made at different locations around the perimeter with a maximum tolerance of 0.004 inch between the high and the low measurements.

9.2.4. Pitot Tube. The pitot tube located on the sampling probe assembly is calibrated using the geometric standard. If the pitot tube meets the geometric standard, a calibration coefficient of 0.84 is assigned.

9.2.5. Thermocouple/Pyrometer. Prior to emission testing, the thermocouple/pyrometer assembly is checked against a reference thermometer [ASTM mercury thermometer]. Following the assessment, the units are again checked at a temperature within 10 percent of the average absolute stack temperature with a reference thermometer/pyrometer assembly. For both the pre and post-test calibrations, the absolute temperatures measured with the thermocouple/pyrometer assembly should be within 1.5 percent of the value obtained by the reference thermocouple/pyrometer assembly.

9.2.6. Orsat Analyzer. The Orsat analyzer is leak- checked and calibrated in accordance with EPA Reference Method 3.

9.2.7. Summary. A summary of the procedures used for calibrating the stack sampling equipment is presented in Table 9-1.

9.3. Analytical Instruments.

9.3.1. Instrument Performance. At the beginning of the analytical phase of this project, a performance check will be performed on each instrument to demonstrate each GC's ability to meet the operating performance standards. For the purposes of this project, the operating performance standard for the instrument will include the analysis of at least one blank and one standard. The instruments response to the internal standard (if any) for GC/MS will be noted with the results for the group of actual samples analyzed.

9.3.2. Calibration Curve/Point. For the analysis of POHC containing samples by GC/ECD or GC/NPD, the instrument will be calibrated on a daily basis prior to the analysis of each group of samples by analyzing a set of solutions of energetic compounds in the extraction solvent at three to five concentration levels. The correlation coefficient for each calibration curve will be greater than 0.95. This daily calibration curve will also be used as an assessment of the instrument performance. Any tendency for the calibration curve or the instrument performance to drift will be monitored by reanalyzing at least one of the standard solutions daily. A new calibration curve will be generated if the response observed in the reanalysis of the standard varies by more than $\pm$ 25 percent from that predicted from the previous calibration curve. Since 10 percent of all the positive samples from the GC/ECD or GC/NPD analysis will be submitted for GC/MS confirmation, the GC/MS instrument will also be calibrated on a daily basis by a single point calibration standard. The purpose of the single point calibration is to determine the response factor of the analyte on the particular analytical instrument. If sufficient analyte is present in the sample, semi- quantitation can be performed utilizing an internal standard technique with anthracene - d10.

9.3.3. Summary. Table 9-2 summarizes the methods to be used for calibrating the analytical instruments.

TABLE 9.1. CALIBRATION OF SAMPLING EQUIPMENT

Apparatus	Acceptance Limits	Frequency and Method of Measurements	Actions If Requirements Are Not Met
Wet Test Meter (Capacity = 120 ft^3/hr)	Y_{wt} = 1.0±0.01 over calibration range for dry gas meter	Initial and annual by spirometer	Adjust until specifications are met, or return to manufacturer
Dry Gas Meter (Meter Box)	Y=1.0±0.01 Y_i=Y±0.01	Initial, annual, and when post-calibration Y exceeds annual Y ± 0.02 by wet test meter following procedures in EPA-600/4-77-027b	Repair or replace and then re-calibrate
	Y=Y±0.05Y	After field use (post-calibration	
Meter Box	$\Delta H_@$=1.84±0.25 $\Delta H_{@i}$=$\Delta H_@$±0.15	Initial and annual by wet test meter	Repair or replace and then recalibrate
Pyrometer and Thermocouple	±1.5% of abs. temperature	Initial, annual or when unit is repaired; calibrate with control pyrometer or ASTM mercury thermometer. 32 °F to 700 °F, extrapolated over range to 1800 °F	Adjust, determine a constant correction factor, or reject unit
	±1.5% of stack gas temperature ±0.5% of abs. temperature	After field use with control pyrometer	
Thermometers	Impinger thermometer ±2 °F	Initial by ASTM Mercury (Hg) thermometer in ice bath and at ambient	Adjust, determine a constant correction factor, or reject unit
	± 4 °F	After field use by ASTM Hg thermometer at ambient	

Apparatus	Acceptance Limits	Frequency and Method of Measurements	Actions If Requirements Are Not Met
Thermometers	Dry gas meter thermometer ±2 °F	Initial by ASTM Hg thermometer at ambient and 104-122 °F range	Adjust, determine a constant correction factor, or reject unit
	±4 °F	After field use by ASTM Hg thermometer at ambient	
	Filter box thermometer ±5 °F	Initial by ASTM Hg thermometer at 225 to 275 °F range	Adjust, determine a constant correction factor or reject unit
	±5 °F	After field use by ASTM Hg thermometer at ambient	
Probe Assembly with Type S Pitot Tube	C_p=0.84	Initial, after field use by geometric calibration	Adjust or repair
Probe Nozzle	Difference between high and low ≤0.004 in.; average of 3 ID measurements at 60 ° increments from each other	Initial and when reshaped or sharpened	Recalibrate, reshape, and sharpen when nozzle becomes nicked, dented or corroded
Analytical Balance	±1 mg of Class S weights	Check with Class S weights prior to use	Adjust or repair
Probe heating system	Capable of maintaining 248 °F ±25 °F	Calibrate component per reference 10 if constructed, per reference 11, or use published calibration curve	Repair or replace and then reverify calibration

TABLE 9.2. CALIBRATION OF ANALYTICAL EQUIPMENTS

Instrument	Standard Concentration	Standard Type	Actions if Requirements Are Not Met
Hewlett-Packard Model 5880 GC	1-40 µg	External Standard	Preparation of new external standards. Determination of new response factors.
Finnigan Model 4000 GC/MS		Internal Standard (Each sample)	Preparation of new internal and external standards. Determination of new response factors.
	40 µg*	External Standard	

*Single point calibration

SECTION 10. DATA REDUCTION, VALIDATION AND REPORTING

10.1. Data Reduction.

10.1.1. Sampling and Operational. Sampling data will be obtained in accordance with EPA Method 5 procedures. This will include: gas temperatures at the inlet and outlet of the dry gas meter and at the stack gas sample point; pressure differential associated with the Pitot tube and the orifice; and the sample volumes. Averages for these parameters during the sampling run, excluding the sample volume, will be calculated. Data for those pertinent operational parameters will be recorded during the sampling run and the average and standard deviation of these measurements will also be calculated. The purpose of the standard deviation is to assess the variability of the operational data over the sampling period.

10.1.2. Analytical. The raw data from the GC and GC/MS analysis of samples will consist of retention time, peak area counts, and, in the case of GC/MS, the mass spectra for both the surrogate and the target compounds. This raw peak area data from the GC will be converted to concentrations by using the calibration curve for the particular species as constructed through the analysis of standard solutions at various concentration levels. The peak area data is converted to concentration by the integrator connected to the GC instrument. Peak areas for the calibration standards are entered along with a retention time of the particular analyte and a linear regression is performed. As the analysis is conducted, the peak areas from the analysis of the unknowns are entered and the corresponding concentrations and the total mass of the analyte are computed from the calibration curve. A summary of the raw and converted data and the chromatograph trace will be printed and the original will be maintained in the laboratory's project work file. Detailed procedures for the construction of the project data file are contained in Appendix C.

10.2. Data Validation. In general terms, data validation involves the review of data and the acceptance or rejection of that data based upon specific criteria. The criteria are dependent upon the type and purpose of the data. The initial step in the data validation will, as a minimum, consist of a thorough examination of all calculations involved in the reduction of either the sampling or the analytical data. This examination should be conducted by a different person from the one who initially performed the calculations.

10.2.1. Sampling and Operation. The sampling and operational data will be validated by another project engineer performing at least one series of the calculations. All data obtained in the field will be checked by the project engineer to ensure its completeness and then placed in the project data file. The data file will also include all documentation associated with the calibration of the field equipment. Any redundant or

back-up data on particular operational parameters will be used to assist in the validation of the operational data. The feasibility of having redundant data from the stack sampling point is unlikely. If continuous emission analyzers are being utilized, the Orsat analysis from the stack sampling point will be used for confirmation. The two corresponding values must agree within + 10 percent in order for the data to be valid.

10.2.2. Analytical. The principal criteria to be used during the collection of the analytical data involve, on a weekly basis, the verification that a hard copy of all the data generated during the previous week has been stored in the laboratory project file. This further involves the review on a weekly basis at least 50 percent of the raw data (chromatograph traces and integrator outputs) to verify the adequacy of the documentation, confirm peak shape and resolution, and assure the integrator was sensing and interpreting the peaks correctly. The data file should also include the reports from the analysis of blanks, standards and any other QC data along with the results from the analysis on the submitted samples. Analytical data will be further reviewed to identify variations in the composition from the replicate analysis of samples and where variations appear significant, the calculations will be re-checked for errors. Additionally, the sample collection and analysis procedures will be reviewed to identify any potential causes for the inconsistencies.

10.3. Treatment of Outlying Data. All data that is a part of this assessment will be considered valid and reported unless any problems are identified which would further qualify the data. In the event of anomalous data or results, an investigation will be conducted in an attempt to locate the source of the problem whether it is sample collection, sample preparation, or analysis procedures. The anomalous results will be reported with the problems that were encountered and the documentation of these problems will serve to qualify the significance of the results. In those instances when the concentration of the analyte is undetectable, the detection limit for the sample will be reported along with the "less than" sign (<); if an emission level is calculated from the results on this sample, the emission level will be annotated with the "less than" sign. Detection limits will vary from sample to sample depending upon, among other things, the matrix type and the level of interferences.

10.4. Data Reporting. The data obtained from the field (sampling and operational) shall be summarized in tables and these tables will be found in the final report of the assessment. All required reports and documentation from the analytical laboratory, including chromatograms, mass spectra, calibration summaries, QC results, etc. (see Appendix D) shall be clearly labeled and placed in the project data file. Analytical results for both field samples and QA/QC inserts will also be summarized and included in the final report on the assessment. These data tables may also be utilized in interim reports depending upon the needs of management and the client.

SECTION 11. INTERNAL QUALITY CONTROL

11.1. Introduction. The effectiveness of the QA/QC program depends upon operation of the laboratory in accordance with procedures which systematically detect errors and prevent the recurrence of these errors and which measure the inherent errors in the sampling and analytical methodology. Two general steps in these QC checks is the daily calibration of the analytical instrument and the determination of glassware cleanliness. Additionally, QC samples will be prepared by the analytical section and interjected among the field samples at a frequency of 10 percent of the field samples submitted to the laboratory. The results from these QC procedures will be used to evaluate the validity of the entire data package through determining the precision and the accuracy of the analysis. The objectives for precision, accuracy and data completeness were contained in Section 5.

11.2. Instrument Calibration. Daily calibration curves (multipoint, three to five concentration levels) for the energetic materials (e.g., NG; isomers of DNT and TNT) will be prepared by the GC operator. This calibration curve will be used to assess the performance of the analytical instrument.

11.3. Blank Samples. These samples are analyzed along with the field samples in order to assess the level of contamination inherent in the field or laboratory handling of the samples. Field blanks (extraction solvents; extract of the field laboratory water; filter extract; and resin extract) are those blanks which are exposed to the field conditions and analyzed to assess the potential contamination to the field environment. Method blanks are prepared in the laboratory and are analyzed to assess the contamination due to sample preparation. Finally, reagent and solvent blanks are used to determine the background levels of the analytes or interferences in the reagents and solvents used in the analysis. Prior to conducting the emission sampling, one resin sample and one filter will be extracted and analyzed to ensure no contamination or interference compounds are present in these media before use. Furthermore, a background run consisting of only auxiliary fuel and no waste feed will be conducted prior to the introduction of the waste material in order to determine the background levels of the target compounds or any interferences. If a blank sample is contaminated, the cause and impact of the contamination is identified and assessed. Corrective action, as discussed in Section 15 of this plan, may be implemented.

11.4. Sample Analysis. The mainstay of the QC program is the replicate analysis of samples and the utilization of surrogate materials. Replicate analysis of samples is performed in order to assess the precision of the analytical results. Field samples are spiked with surrogate compounds prior to sampling. Laboratory method blanks are spiked with the same surrogate and target POHC compounds prior to extraction and analysis. The data on surrogate recoveries are used as an indicator of the accuracy of the sampling, sample preparation and sample analysis procedures.

11.4.1. Recovery of Surrogates. Each sampling train used for POHC sampling will be spiked prior to sampling with known quantities of the surrogate compounds at the first impinger and the first resin section. The recovery of these surrogates from the sampling train will provide an indication of the recovery of the POHC's. The recovery of the surrogate compounds added to a matrix (resin or impinger water) will be calculated by

$$\text{Recovery} = \frac{(\text{quantity of surrogate measured in matrix}) \times 100}{\text{quantity of surrogate added to matrix}}$$

The equation assumes that the surrogate is not present in the matrix at detectable quantities to begin with. The mean and the standard deviation of the surrogate recoveries will be calculated and compiled for the type of matrix (resin or impinger water) and for all recovery data.

11.4.2. Recovery of Blind Spikes. The QA Coordinator at the laboratory (AQAO) will insert a number of blind QA samples containing the target compounds (surrogates and/or POHC's) equal to 10 percent of the total number of samples submitted. The concentration range for these compounds will be calculated by the project engineer and submitted to the AQAO. The recovery of the POHC and surrogate compounds in these blind samples will be determined using the following formula:

$$\text{Recovery} = \frac{(\text{quantity of compound measured in sample}) \times 100}{\text{quantity of compound added to blind sample}}$$

As with the recovery of the surrogates in the field samples, the mean and standard deviation of the POHC and surrogate recoveries from the blind inserts will be determined.

11.4.3. Confirmational Analysis. Ten percent of all field samples positive for POHC and/or surrogate will be subjected to confirmational analysis by gas chromatography/mass spectrometry. The GC/MS instrument will be calibrated daily by the external standard method, as in the case of the GC instrument for quantitation. This calibration shall consist of analyzing a single concentration sample of the target compounds in the extraction solvent. Additionally, a second type of GC detector may also be used for confirmational analysis when the sample concentrations are less than the detection limit of the GC/MS methodology. Sensitivity differences between detectors (ECD versus NPD) may lead to conflicting concentrations for the same sample and therefore confirmational analysis with a second GC detector should be used cautiously.

11.5. Control Charts. As discussed in paragraph 11.1, the analytical laboratory will insert their own quality control samples and the accuracy and precision of the analysis on these samples will be used to construct control charts. Control charts, which are used for surveillance of repetitive analysis in a laboratory, plot the analytical data versus time in order to indicate the amount of variation about the target value that

can be expected to occur by chance. If the measured value is assumed to vary normally with a standard deviation of σ, then the control lines are centered on the target value T and are drawn at $T \pm 3\sigma$. This will cover 99.9 percent of the distribution. Control lines may also be drawn at $T \pm 2\sigma$ which covers 95 percent of the distribution. When results exceed the upper or lower control limits, the analytical system is declared "out of control" and corrective action is implemented. After the problem is resolved, the samples which were analyzed prior to the quality control sample are reanalyzed.

11.6. Analysis Timetable. Because of the nature of the target compounds for analysis (i.e., unstable for a variety of reasons), the recovery of these compounds are dependent upon the length of time between obtaining and analyzing the sample. While samples are field extracted in order to place the compounds in a more stable matrix (organic solvent), the extracts are still sensitive to degradation over time. Laboratory work has demonstrated that samples were stable up to 30 days. Furthermore, regulatory timetables are typically placed on projects of this type which require submission of final reports within 60 to 90 days following the completion of the field work. For these two reasons, analytical results should be obtained within 30 days following sample arrival at the laboratory. The Figure 11.1 illustrates a work plan for the movement and analysis of samples once they reach the laboratory.

PROJECT SCHEDULE WORKPLAN

QA/QC Plan: Sampling/Analysis for Energetic Compounds

	CURRENT ISSUE	SUPERCEDES
VERSION NUMBER	2	1
DATE	6 May 1988	16 February 1988

STEP	DESCRIPTION	RESPONSIBLE ORGANIZATION
1	Performance check/audit on all CC instruments	OECD
2	Samples arrive at laboratory - AQAO	APED
3	Sample processing - renumbering; insert addition (QA)	AQAO
4	Preparation of Standard Solutions and QC samples	OECD
5	Instrument calibrations/tuning	OECD
6	Sample delivery to analysis section	AQAO
7	Retention time window determination	OECD
8	Daily instrument calibration	OECD
9	Verify spiking solution concentration	OECD
10	Replicate analysis of samples	OECD
11	Recovery data calculated and compiled	OECD
12	Confirmational analysis by GC/MS	OECD
13	Documentation review -verification of hard copy -review raw analytical data -verify documentation	OECD/AQAO
14	Construct analyst control charts	OECD
15	Preparation of analytical section to project file	OECD
16	AQAO review of project file	AQAO
17	Transmit project file to APED	AQAO

FIGURE 11.1. PROJECT WORK PLAN

SECTION 12. PERFORMANCE AND SYSTEM AUDITS

12.1. Introduction. On a periodic basis, or if deemed necessary, an evaluation of the sampling and analytical functions for the emissions evaluation project will be performed to verify that all QC procedures are being properly performed. The evaluation process may consist of two types of audits: performance audits and system audits. The performance audit involves independent checks of the entire sampling and analysis system to evaluate the quality of the data being generated. A system audit involves a more direct inspection of the QC procedures being performed for the project. Key to the performance and system audits is the comparison of the written SOP's, checklists and this project plan versus the observations and evaluations of the ongoing work, records, laboratory data and instrument performance characteristics. If any discrepancies are identified in the course of the evaluation, recommendations concerning the resolution of the problem areas should be made and a report of the finding should be prepared and filed with the sampling program manager and the applicable analytical section manager.

12.2. Performance Audits. The performance audits on the sampling and analysis systems are in addition to any QC checks that may be performed by the operator or the analyst during the course of the project.

12.2.1. Sampling and Operational. A separate, unknown calibration gas can be utilized to check the continuous emission analyzer calibration, as well as the analysis of the stack gases by Orsat. The predominance of sampling and operation data originates from the reading and interpretation of gages and displays. An evaluation of the person recording this data is to be made by "over the shoulder" observation (i.e., an auditor looking over the shoulder of the person recording the data and reading/interpreting the data). Additional audits procedures will involve the use of field blank samples submitted to the laboratory for analysis. A series of calculations which were performed on the data should be reviewed in its entirety. Calculations which are performed by computer should be reviewed using a set of "dummy" raw data.

12.2.2. Analysis. Because of the difficulty in obtaining reference samples which would possess matrices similar to the ones being analyzed, the performance audit for the analytical system will rely on splitting actual field samples and the use of sample blanks. Additionally, XAD-2 resin and water matrix samples may be spiked with the target compounds, extracted as per the procedures in Section 8, and submitted for analysis with a group of field samples. Calculations performed on the raw analytical data should be reviewed using the techniques discussed above (paragraph 12.2.1).

12.3. System Audits. The system evaluation is performed to ensure that the required levels of equipment, instrumentation, and personnel are being

committed to the project. This audit process may also serve as a mechanism for identifying and resolving weaknesses that may have been identified in the performance audits or through routine data audits. The sequence of events and the areas of examination for system audits are listed below. A report of any system audit should be prepared and maintained with the emission evaluation program manager.

12.3.1. Meeting with Key Project Personnel. This meeting should provide the framework for the audit. The purpose of the audit should be discussed as well as any problem areas that may have been identified prior to the audit.

12.3.2. Review of Personnel Qualifications. As necessary, the qualifications of the personnel committed to the project should be reviewed to ensure their level of training and/or experience is appropriate for their role in the project.

12.3.3. Review of Equipment/Instrumentation. The instrumentation/equipment committed to the project should be reviewed, in particular, to ensure that there is sufficient redundancy to perform the required measurements in a timely manner.

12.3.4. Quality Control Procedures Review. The following areas should be reviewed:

12.3.4.1. Sample logging/tracking/receiving procedures.

12.3.4.2. Sample storage (field, transit, laboratory).

12.3.4.3. Procedures to prevent sample contamination.

12.3.4.4. Sample/laboratory security.

12.3.4.5. Safety procedures.

12.3.4.6. Conformance to written SOP's.

12.3.4.7. Recordkeeping procedures.

12.3.4.8. Glassware cleaning procedures.

12.3.4.9. Chain of custody procedures.

12.3.4.10. Data handling/analysis/reporting procedures.

12.3.4.11. Project file preparation.

12.3.4.12. Technical and managerial review of project operation.

12.3.5. Identification of Required Corrective Actions. At the end of the audit process, the actions needed to correct weakness areas should be discussed with the key personnel involved. All previously identified problems as well as any new problems identified should be discussed. The methodology and timetable should be established for the demonstration of improvements and for the documentation of these improvements.

SECTION 13. PREVENTIVE MAINTENANCE

13.1. Hardware/Instruments. The hardware associated with both the sampling and the analysis have regularly scheduled preventive maintenance. The purpose of this preventive maintenance is to ensure the sampling and analytical equipment is in serviceable condition and to detect minor problems with this equipment before these problems become major defects. Preventive maintenance will be performed by the personnel responsible for operating the equipment and in accordance with the guidelines established in the respective equipment literature of the vendor. For analytical equipment, the chromatographic carrier gas purification traps and injector septa should be replaced on a regular basis. Most maintenance on the analytical equipment, such as column replacement or detector cleaning, should be performed on an as needed basis when the performance of the instrument begins to degrade. The degradation may be evidenced by the lack of peak resolution, a shift in the calibration curve or a failure to meet the QC check criteria. For sampling equipment, an example of preventive maintenance would be the replacement of pump seals and other pump maintenance. Other examples of preventive maintenance would be: performance checks, the care and cleaning of equipment in general, and the replacement of expendable items such as fuses on the equipment.

13.2. Spare Parts. Because a large amount of preventive maintenance may be reactionary in nature, adequate supplies of spare items should be maintained so that they will be available when necessary. A preventive maintenance schedule will be implemented for those critical measurements systems (both analytical and sampling) of these project types.

SECTION 14. PROCEDURES FOR ASSESSING DATA ACCURACY, PRECISION AND COMPLETENESS

14.1. Calculation of Mean. The mean of a series of replicate measurements of a given parameter is calculated by

$$X = (1/n)\sum_{1} X_i$$

where: n = number of measurements
X_i = value of measurement

14.2. Assessment of Accuracy. The primary tool for determining the accuracy of the analytical method is the calculation of the degree of agreement of the measurement with a reference or theoretical value and the assessment of the recovery of surrogate compounds. Accuracy is defined as

$$\text{Accuracy} = [(X-T)/T]x100$$

Recovery of the surrogate compounds is calculated by

$$\text{Recovery} = \frac{\text{(quantity of surrogate in sample)x100}}{\text{quantity of surrogate added to sample}}$$

14.3. Assessment of Precision. The estimate of the precision of replicate measurements is expressed as the RSD [the coefficient of variation (CV)]:

$$CV = \sigma/X$$

where: σ = standard deviation

X = sample mean

Alternatively, for the duplicate measurement of a parameter, the precision is estimated by the RPD. Relative percent difference is calculated by

$$RPD = \frac{2(X_1 - X_2) \times 100}{(X_1 + X_2)}$$

14.4. Completeness. Completeness for determining compliance with the QA objective for the quantity of valid data is calculated by

$$\text{Completeness} = \frac{\text{(number of samples with valid results)}}{\text{(number of samples scheduled for analysis)}}$$

14.5. Assessment of Variances. In the event that analytical or calculated results demonstrate large variances or if a QA/QC objective has not been met, the use of analysis of variance (ANOVA) techniques may be necessary to identify the cause for and therefore, determine a means of decreasing these variances. This tool may be used by either the project engineer or the chemist at his own discretion. The sampling and analysis protocol should

be written so that it follows the generic scheme illustrated in Figure 14-1. The precision of the entire sampling and analysis, expressed as a variance, is equal to the sum of the variances for each separate activity in the project due to the rule of summation of variances.

$$S_t^2 = S_s^2 + S_p^2 + S_a^2$$

The replicate analysis of a single solution (sample) from the preparation phase yields an estimate for the variance of the analysis S_a^2. By splitting one of the sampling run's into three or more samples and then analyzing, an estimate of the variance for the combined sample preparation and analysis steps is obtained $S_p^2 + S_a^2$. As long as the replicate analysis discussed above was preformed on a solution in this group, the variance of the analysis can be subtracted, yielding the variance of the sample preparation step. Finally, the variance attributed to the three sampling runs represents the sum of the variances from the sampling, sample preparation and sample analysis steps. Since the variances of the sample analysis and sample preparation steps have already been determined, the variance of the sampling procedure is calculated by the difference. Figure 14-2 illustrates the generic sampling and analysis protocol with the variances generated from the steps. These variances can then be examined to determine if and what adjustments may be made to methodologies to correct sources of imprecision.

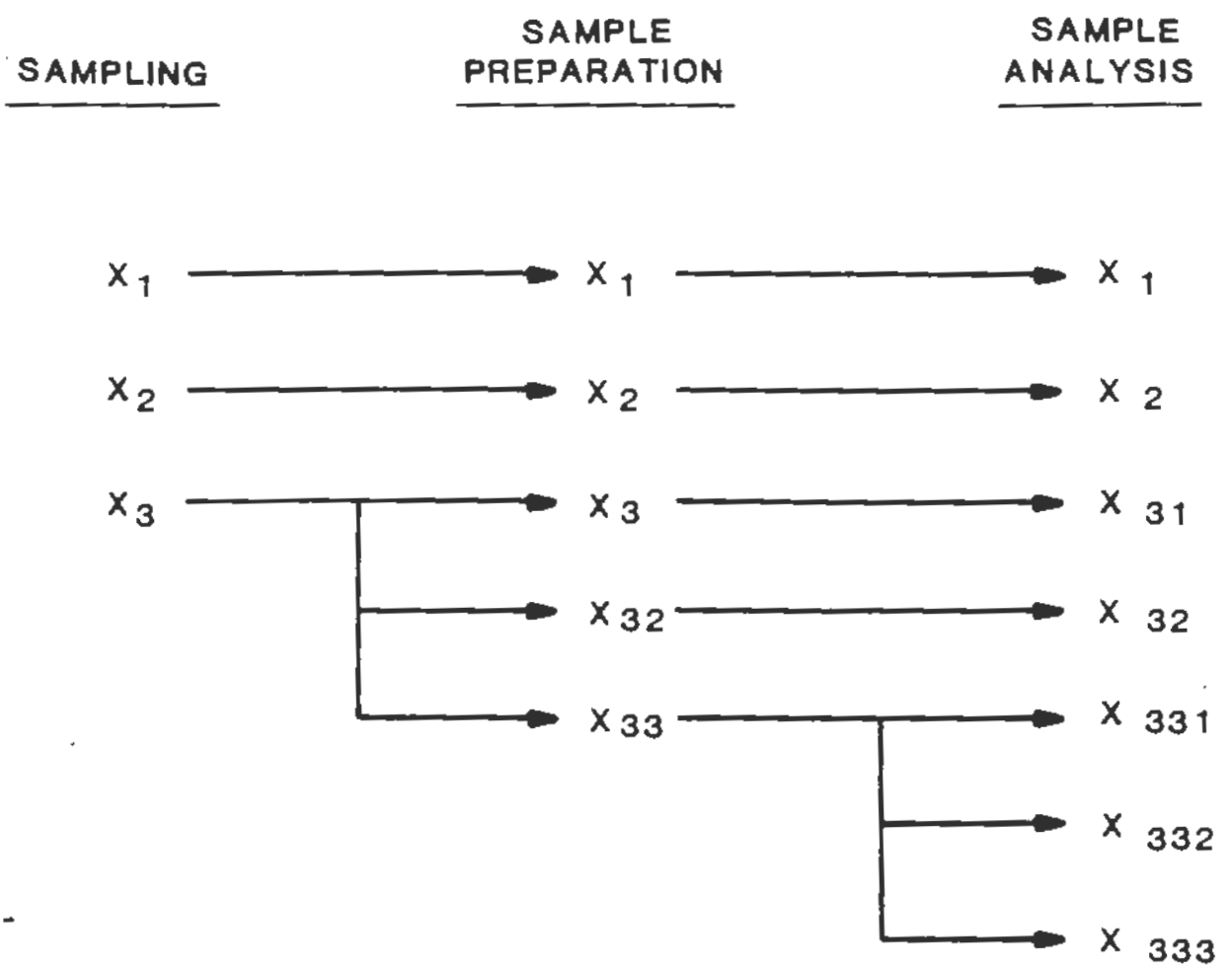

FIGURE 14-1. SCHEMATIC DIAGRAM OF A GENERIC SAMPLING AND ANALYSIS PROTOCOL

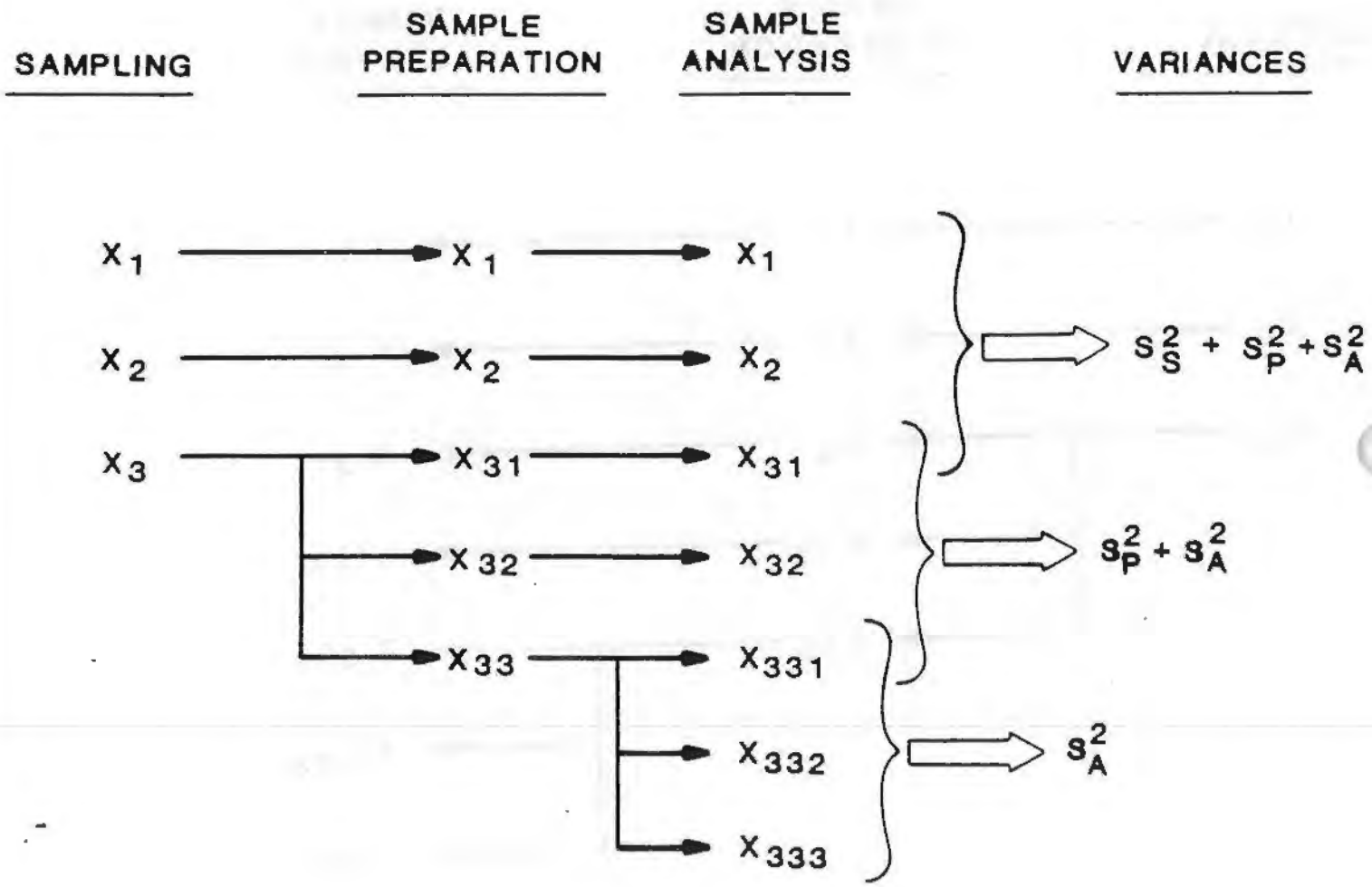

FIGURE 14-2. DIAGRAM OF VARIANCES DUE TO SAMPLING, PREPARATION AND ANALYSIS SCHEME

SECTION 15. CORRECTIVE ACTION

15.1 Introduction. The field sampling team and the analytical personnel are the persons in the best position to recognize problems which would affect data quality as they occur. Thus, these personnel have the responsibility of detecting minor problems, correcting these problems before a major malfunction can occur, and minimizing data loss through immediate response. When corrective action is performed by these individuals, they are responsible for notifying the project engineer or analysis coordinator who will document the action and who may, at his discretion, intensify the quality assurance surveillance of the sampling or analytical methodology.

15.2. Sampling Phase. The establishment of specific reaction limits for the sampling procedures beyond which corrective action is triggered is not practical, nor is it feasible. The burden of maintaining data quality in the field relies heavily on the personnel operating the sampling equipment. Each individual must fully understand the program objectives and the data quality required to meet those objectives so that problems may be identified and corrected.

15.3. Analytical Phase.

15.3.1. For the analytical method being employed, the standard deviation of replicates, the mean recovery of the surrogates from field samples, and the relative standard deviation of the surrogates will be computed and tracked. These data will be segregated according to the matrix being analyzed. The statistics will be updated as additional analysis are performed. When either the relative standard deviation of the replicate analysis or the relative standard deviation of surrogates exceeds twice the most recent value for these statistics (95 percent level for normal distribution), corrective action will be taken before any additional analysis are performed. Similar corrective action may be required if the mean recovery of the surrogates is less than the QA/QC objectives. If during either system or performance audits, problems are identified, the corrective action will be implemented immediately.

15.3.2. In the event corrective action is warranted, short-term responses may include, but are not necessarily limited to:

15.3.2.1. Recalibration of analytical instruments with newly prepared calibration standards.

15.3.2.2. Analysis of standard solution to assess instrument drift.

15.3.2.3. Instrument performance evaluation.

15.3.2.4. Replacement of solvents or reagents which may be yielding unacceptable blank levels.

15.3.2.5. Degradation of instrument performance may be due to injection port or front section of chromatography column fouling. The glass injection port inserts may be cleaned or repaired and then deactivated as per the instrument manufacturers instructions. Additional action may require the removal of a section of the capillary column near the injection port.

15.3.2.6. Retraining or additional training of analyst and/or laboratory personnel in sample preparation and analysis.

15.4. All Phases. If a more long-term corrective action is required, typical problem-solving techniques will be used to eliminate the source of nonconformance. These techniques are designed to define the problem, investigate and determine the cause of the problem, and finally eliminate the problem through the proper corrective action.

SECTION 16. QUALITY ASSURANCE REPORTS TO MANAGEMENT

16.1. Reports Generated During Sampling Phase. No reports will be specifically prepared during the sampling phase of the project. However, following the completion of the sampling phase, the project engineer will prepare for the project manager a report on all significant activities associated with the project. This report will address any sampling or operational problems experienced, the results of site-specific QC procedures (e.g., sampling equipment leak-checks, isokinetics), and the potential impact on the data quality. The report will also contain a schedule of milestones for the analysis phase of the project as well as for writing the report for the assessment. Specific requirements for reporting and archiving sampling and analytical data are presented in detail in Appendix D.

16.2. Reports Generated During Analysis Phase. Reports generated during the analytical phase will be in the form of QA reports to the laboratory management.

16.2.1. The AQAO and the Analysis Section Supervisor shall meet on a regular basis to assure that all QA/QC practices associated with this project are being implemented and to review possible/potential or existing problem areas. Notes will be kept during the meeting to document those discussions which are deemed pertinent.

16.2.2. Any data anomalies must be investigated to assure that they are not a result of operator or instrument deviation but rather a function of the methodology or the sample matrix.

16.2.3. Results of blind spikes and other performance standards will be logged and made a part of the project data file.

16.3. Quality Assurance Reports to Project Engineer. The AQAO will make a progress report of this project, through channels, to the project engineer within 5 working days of the receipt of the project file from the analytical lab. This report will include the following information:

16.3.1. Report of performance on analysis of blinds inserted by the QA section.

16.3.2. Report of all major problems encountered during the analysis of the samples as well as corrective actions taken to resolve the problem.

16.3.3. Summary of all system audits conducted to include findings and recommendations.

16.3.4. Miscellaneous information identified as pertinent.

APPENDIX A

METHOD VALIDATION DATA

1. Analytical Procedures.

1.1. POHC Compounds.

1.1.1. The detection limit of the analytical procedure for NG, 2,4-dinitrotoluene (2,4-DNT), and 2,6-dinitrotoluene (2,6-DNT) in toluene is 0.025 µg/mL of solution in laboratory samples of only the POHC compound and toluene. Contamination of the solution by combustion byproducts in all probability will decrease the detection limit (increase the minimum concentration). The 0.025 µg/mL detection limit is based on the amount of POHC needed to produce gas chromatographic peaks whose heights are five times the height of the baseline noise. Taking matrix effects into consideration and without preconcentration of the actual samples, the overall quantitation limit for NG, 2,4-DNT, and 2,6-DNT is approximately 2.0 to 3.0 µg per train component.

1.1.2. Multiple injections of analytical standards of the 2,4-DNT, 2,6-DNT, and NG of 0.05 to 2.00 µg/mL were analyzed. The linearities that were achieved for the concentration range represent the calibration curve for the POHC compound standard solutions. The correlation coefficients obtained from the least squares analysis of the data were 0.996 for NG, 0.998 for 2,6-DNT, and 0.997 for 2,4-DNT (refer to Figures A-1 through A-3). Standard solutions in the concentration range of 1 to 100 µg of these POHC compound per 20 mL of toluene were analyzed in triplicate (refer to Table A-1 for these results). The average coefficient of variation for NG obtained from the replicate analysis of the standards was 2.3 percent. By similar analysis, the average coefficient of variation was 10.51 percent for 2,6-DNT and 8.64 percent for 2,4-DNT. Blind controls submitted with actual samples were analyzed 30 days following control solution preparation. Results for the accuracy of these audit samples are presented in Table A-2.

1.1.3. Similar analyses were performed on solutions of 2,4,6-trinitrotoluene (2,4,6-TNT) in toluene and RDX in a mixture of iso-amylacetate and toluene. The compounds, RDX and 2,4,6-TNT, are listed here as POHC's even though these compounds do not fit into the strict regulatory definition of a POHC. The calibration curves for RDX and 2,4,6-TNT (Figures A-4 and A-5), as with the curve for those compounds listed in paragraph 1.1.2., are linear with correlation coefficients of 0.987 and 0.992, respectively. Triplicate analysis was performed on standard solutions of these compounds in the concentration range of 1 to 100 µg per 20 mL of solvent. The relative standard deviations for these analyses are also contained in Table A-1. The average coefficient of variation for 2,4,6-TNT was 16.63 percent, while the average for RDX was 13.85 percent. Results from the accuracy of audit samples containing these compounds are contained in Table A-2.

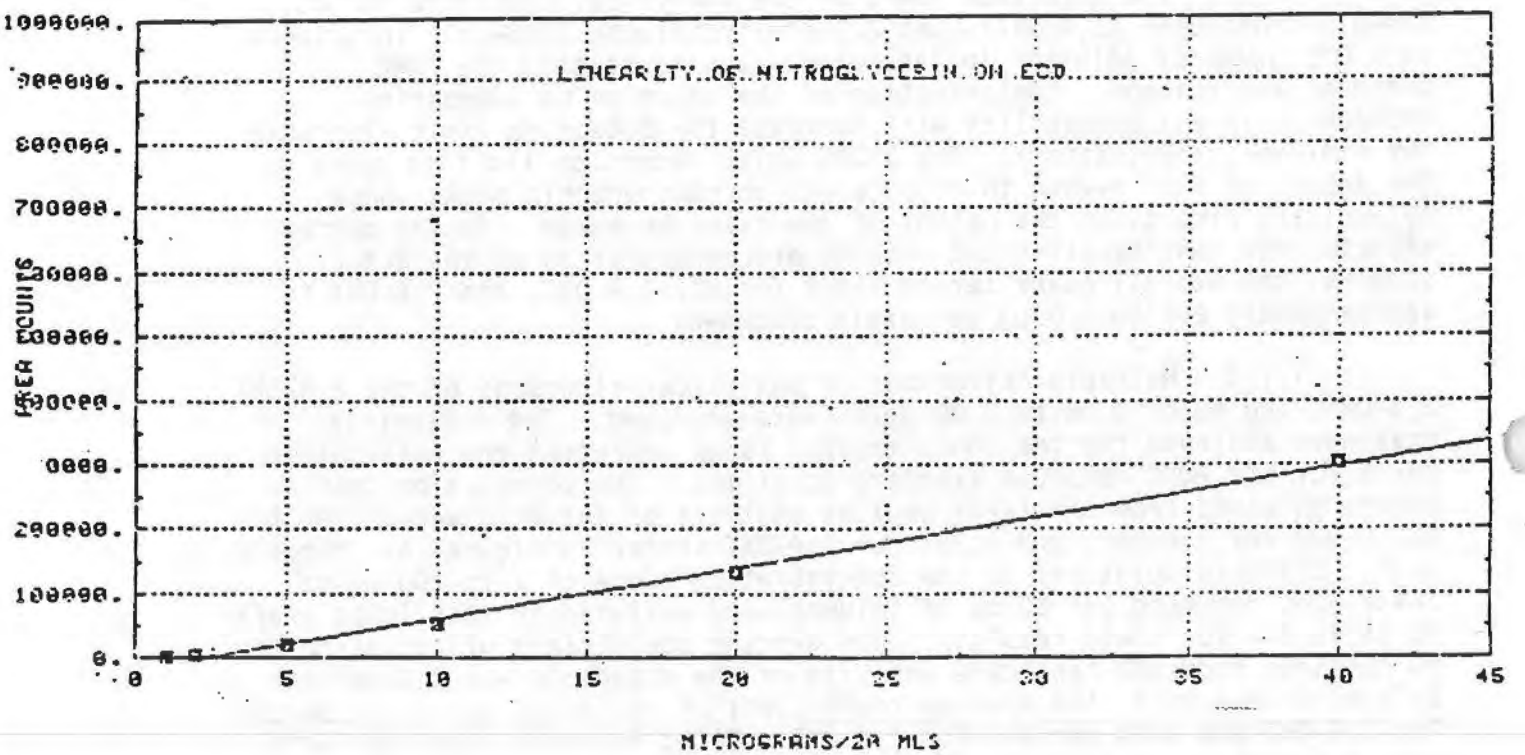

FIGURE A-1. LINEARITY OF NITROGLYCERIN ON ECD

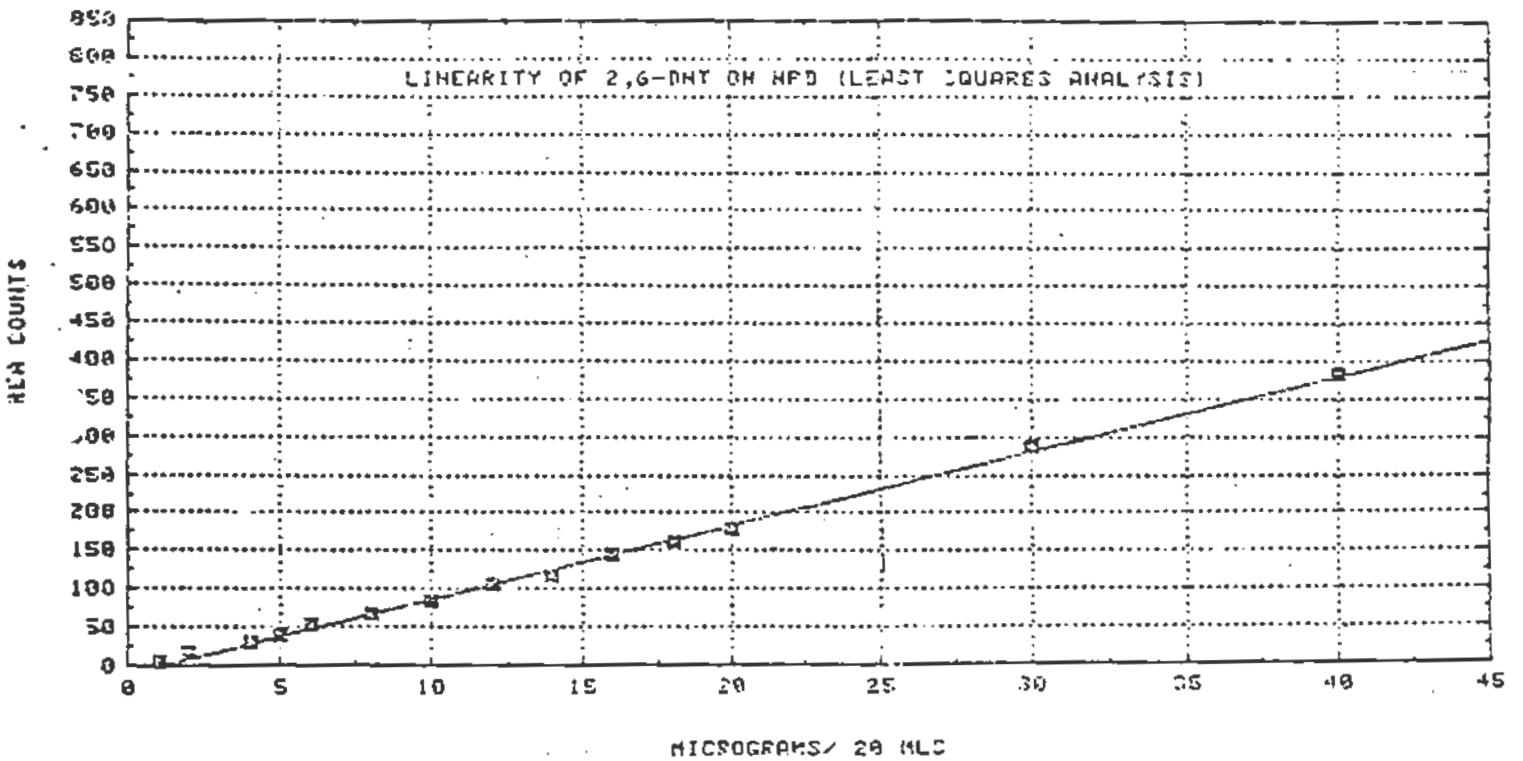

FIGURE A-2. LINEARITY OF 2,6-DNT ON NPD

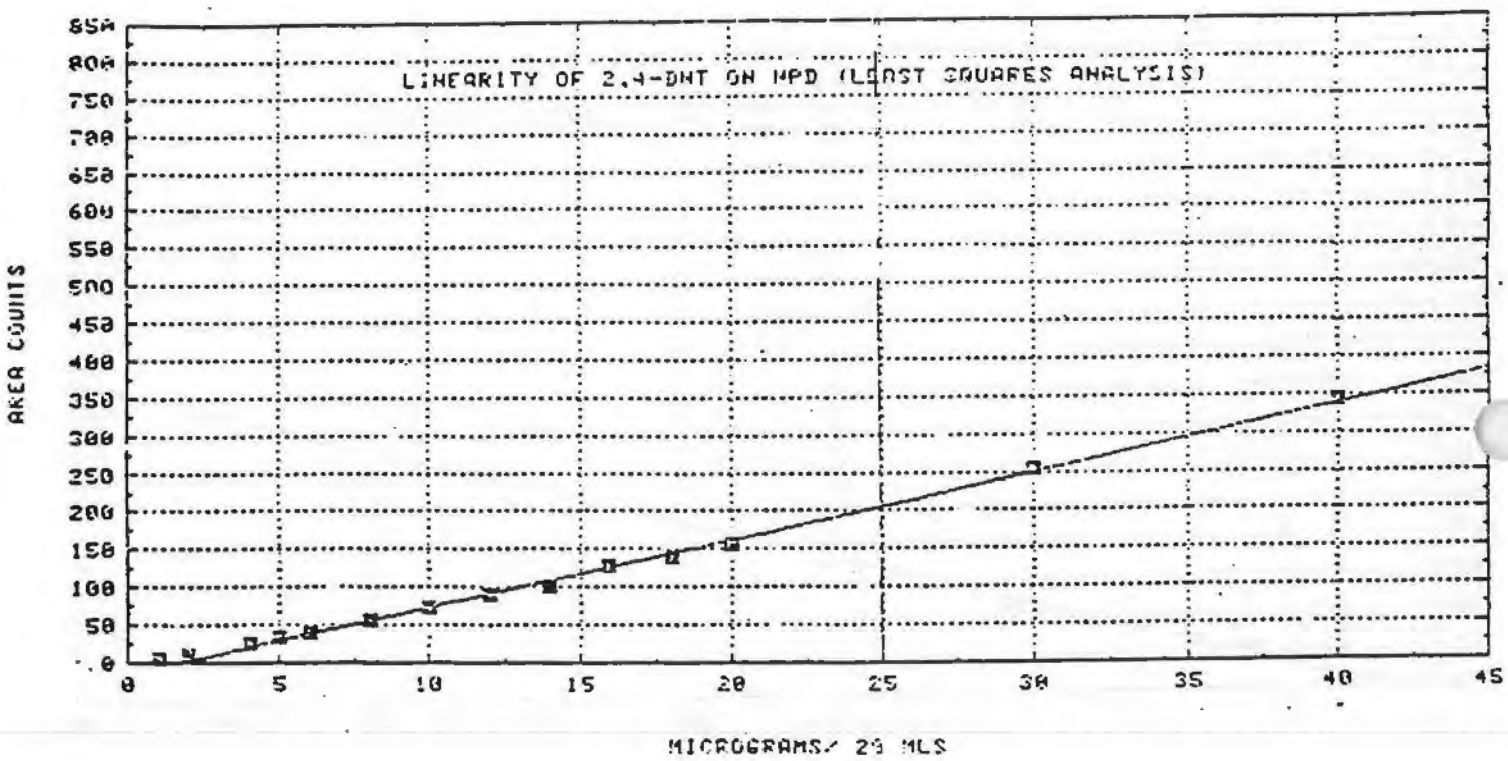

FIGURE A-3. LINEARITY OF 2,4-DN

TABLE A-1. PRECISION ANALYSIS FOR STANDARD SOLUTIONS OF POHC COMPOUNDS

Concentration (µg per 20 mL solvent*)	Compound	Relative Standard Deviation (%)
1.0	2,6-DNT	4.416
	2,4-DNT	0.565
	NG	3.100
	RDX	21.680
	2,4,6-TNT	32.901
2.0	2,6-DNT	- -
	2,4-DNT	- -
	NG	2.900
	RDX	- -
	2,4,6-TNT	- -
5.0	2,6-DNT	19.973
	2,4-DNT	20.736
	NG	2.800
	RDX	21.403
	2,4,6-TNT	24.794
10.0	2,6-DNT	- -
	2,4-DNT	- -
	NG	1.800
	RDX	- -
	2,4,6-TNT	- -
20.0	2,6-DNT	13.805
	2,4-DNT	4.941
	NG	2.500
	RDX	10.379
	2,4,6-TNT	13.137
40.0	2,6-DNT	4.335
	2,4-DNT	5.376
	NG	0.900
	RDX	14.643
	2,4,6-TNT	6.539
80.0	2,6-DNT	6.696
	2,4-DNT	1.882
	NG	- -
	RDX	7.376
	2,4,6-TNT	1.639
100.0	2,6-DNT	6.147
	2,4-DNT	4.213
	NG	- -
	RDX	4.040
	2,4,6-TNT	2.057

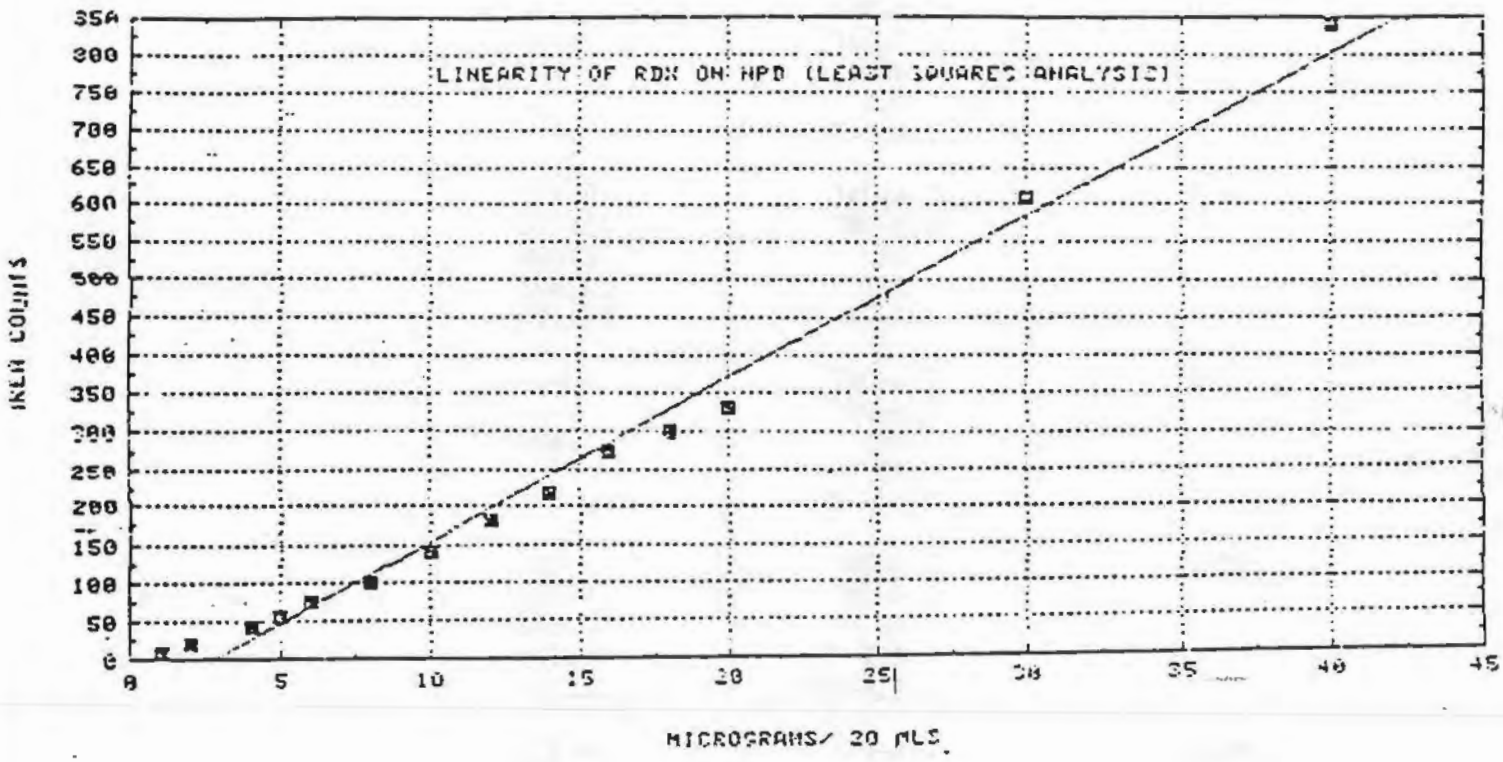

FIGURE A-4. LINEARITY OF RDX ON NPD

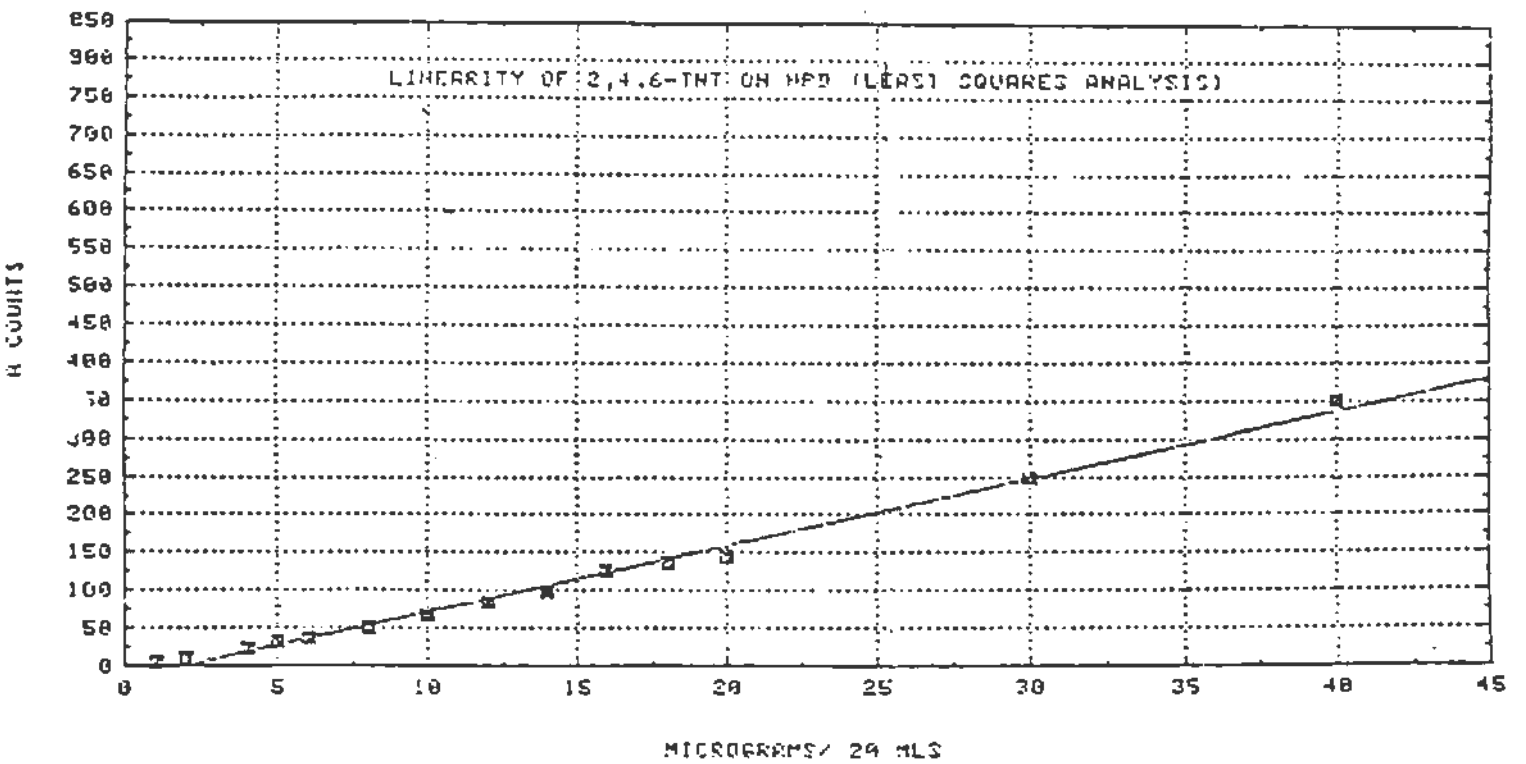

FIGURE A-5. LINEARITY OF 2,4,6-TNT ON NPD

TABLE A-2. RESULTS FROM THE ANALYSIS OF AUDIT SAMPLES

Energetic Compound	No. of Samples	Average Recovery (%)	Recovery Std Dev (%)	Average Dev (%)	Ave. Abs. Dev (%)
NG	10	99.8	1.9	0.2	1.6
2,4-DNT	10	104.4	5.4	-4.5	5.9
2,4,6-TNT	6	99.0	2.8	-1.0	2.4
RDX	6	98.7	1.3	-1.3	1.5

1.2. Surrogate Compounds.

1.2.1. These compounds are spiked onto the sampling train before use in order to monitor the recovery efficiency of the POHC and to ensure sample degradation is not occurring. Precision and accuracy data has been obtained for these compounds to ensure that their recoveries are on the same order as those for the POHC compounds and that they serve as reliable surrogates for the target POHC's.

1.2.2. Replicate analysis of analytical standards of 3,4-dinitrotoluene (3,4-DNT), 2,4,5-trinitrotoluene (2,4,5-TNT), and EGDN in toluene over a concentration range of 0.05 to 2.0 µg/mL were performed in order to generate calibration curves for these compounds. The calibration curves are illustrated in Figures A-6 through A-9. The correlation coefficients for the curves are 0.996 for ethylene glycol dinitrate, 0.998 for 3,4-DNT, and 0.992 for 2,4,5-TNT. A precision analysis was also performed on standard solutions of the surrogate compounds. The results from this analysis are contained in Table A-3. The average coefficients of variation were 1.61 for EGDN, 13.79 for 3,4-DNT, and 22.15 for 2,4,5-TNT.

2. Extraction Procedures.

2.1. Nitroglycerin Stability in Water. According to the Army's Technical Manual on Military Explosives (TM9-1300-214, reference 12), NG is hydrolyzed by water. The rate of this hydrolysis reaction was not determined. Since the greatest amount of work appears to be in sampling for NG, there was concern over sample degradation due to NG remaining in the impinger water during shipment and the contacting of water and NG on the resin bed. Therefore, the shakeout extraction was explored, since it would provide a relatively simple and effective method for field extractions of the exhaust gas samples. (Bench scale testing of the shakeout extraction method is discussed in paragraphs 2.2 and 2.3 of this Appendix.

2.1.1. Following discussions with EPA in 1986 (reference 13), a study was conducted to establish the stability of NG in water and to determine the rate of decay if it did exist. Distilled/deionized water after pH adjustment to 7.0 was spiked with NG and brought to a known volume (Rate of hydrolysis is pH dependent also). The water was shaken periodically and six 25 mL aliquots were obtained on a periodic basis for 3 weeks. Each of these aliquots was extracted with toluene for 30 minutes, and samples were obtained from the toluene fraction of the resulting 2-phase mixture.

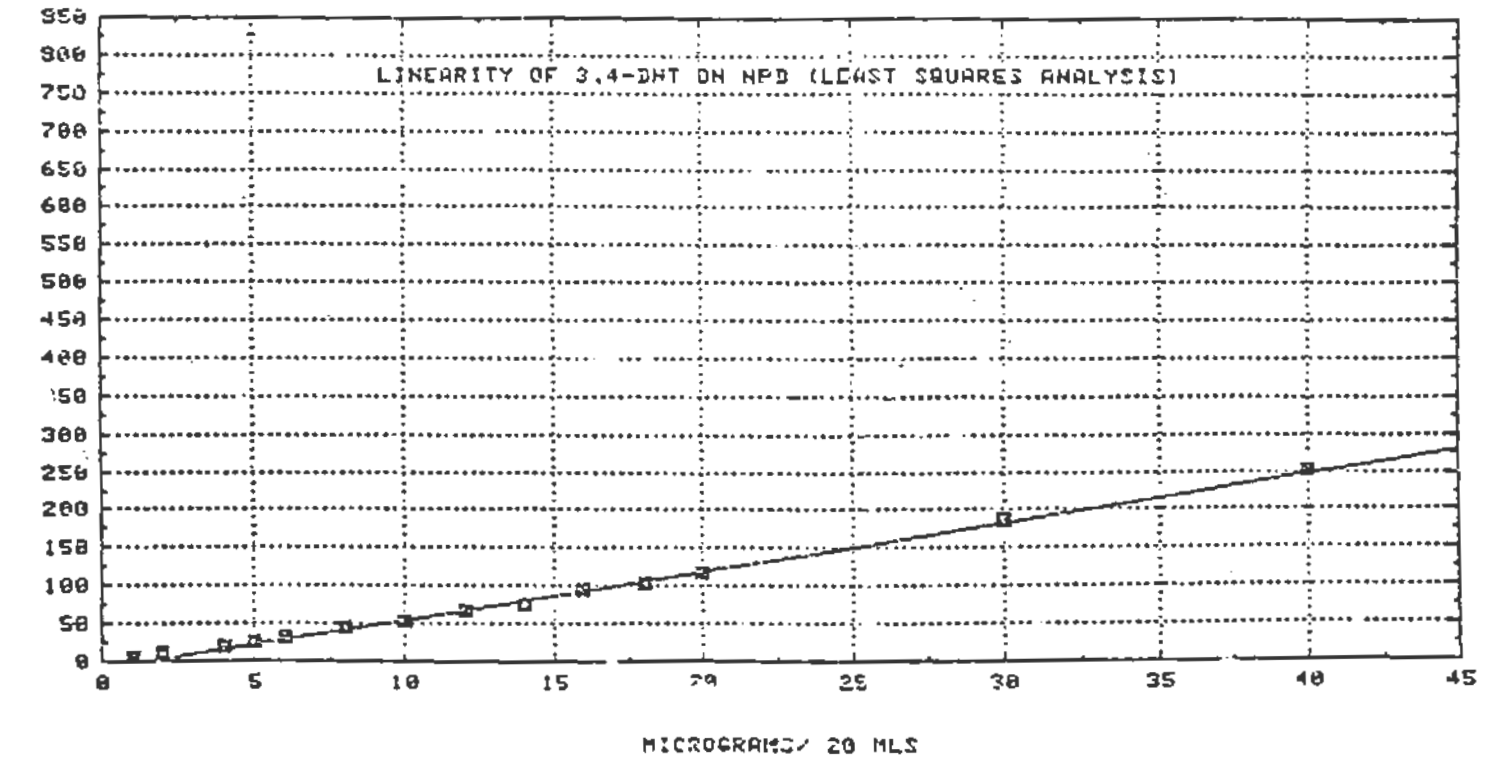

FIGURE A-6. LINEARITY OF 3,4-DNT ON NPD (LEAST SQUARES ANALYSIS)

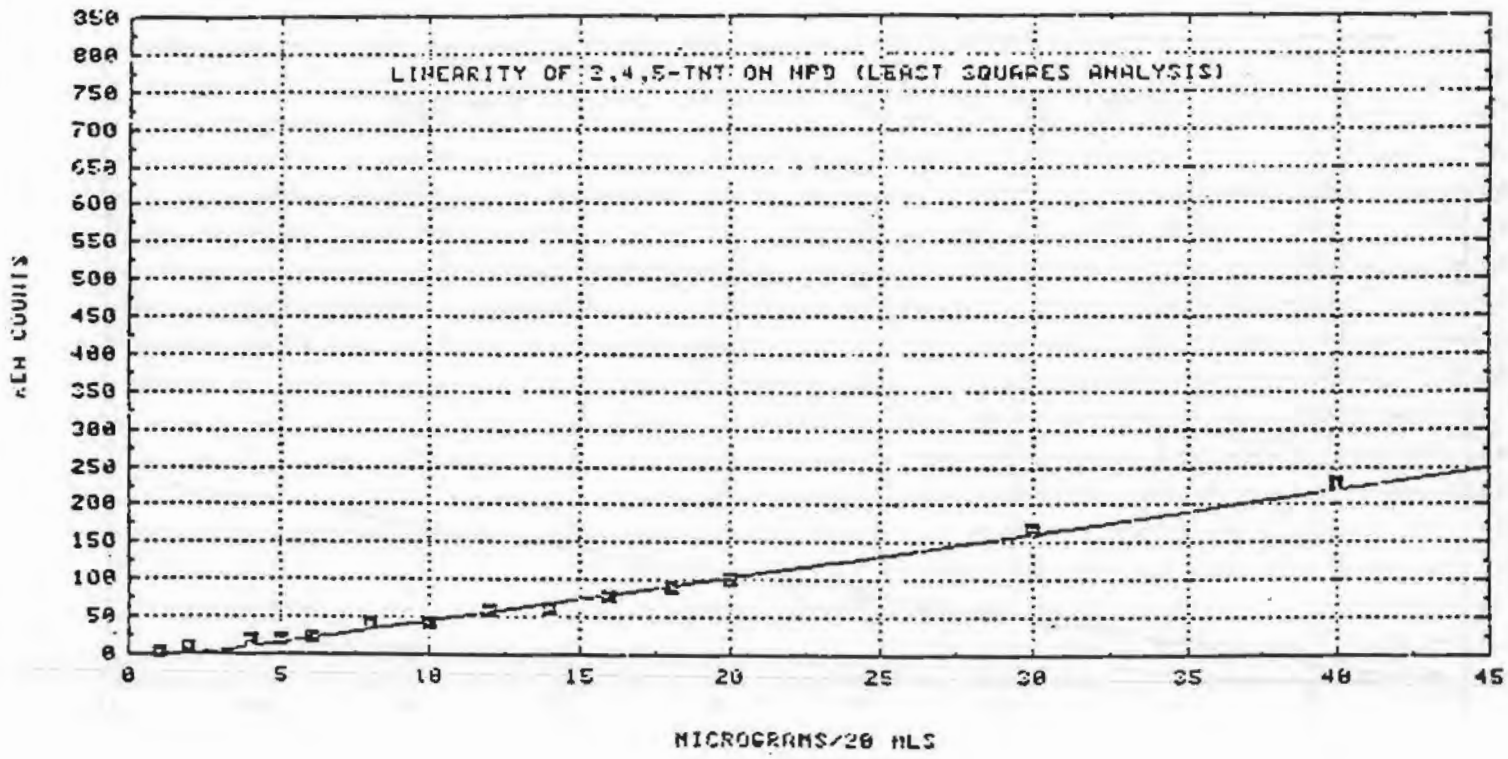

FIGURE A-7. LINEARITY OF 2,4,5-TNT ON NPD (LEAST SQUARES ANALYSIS)

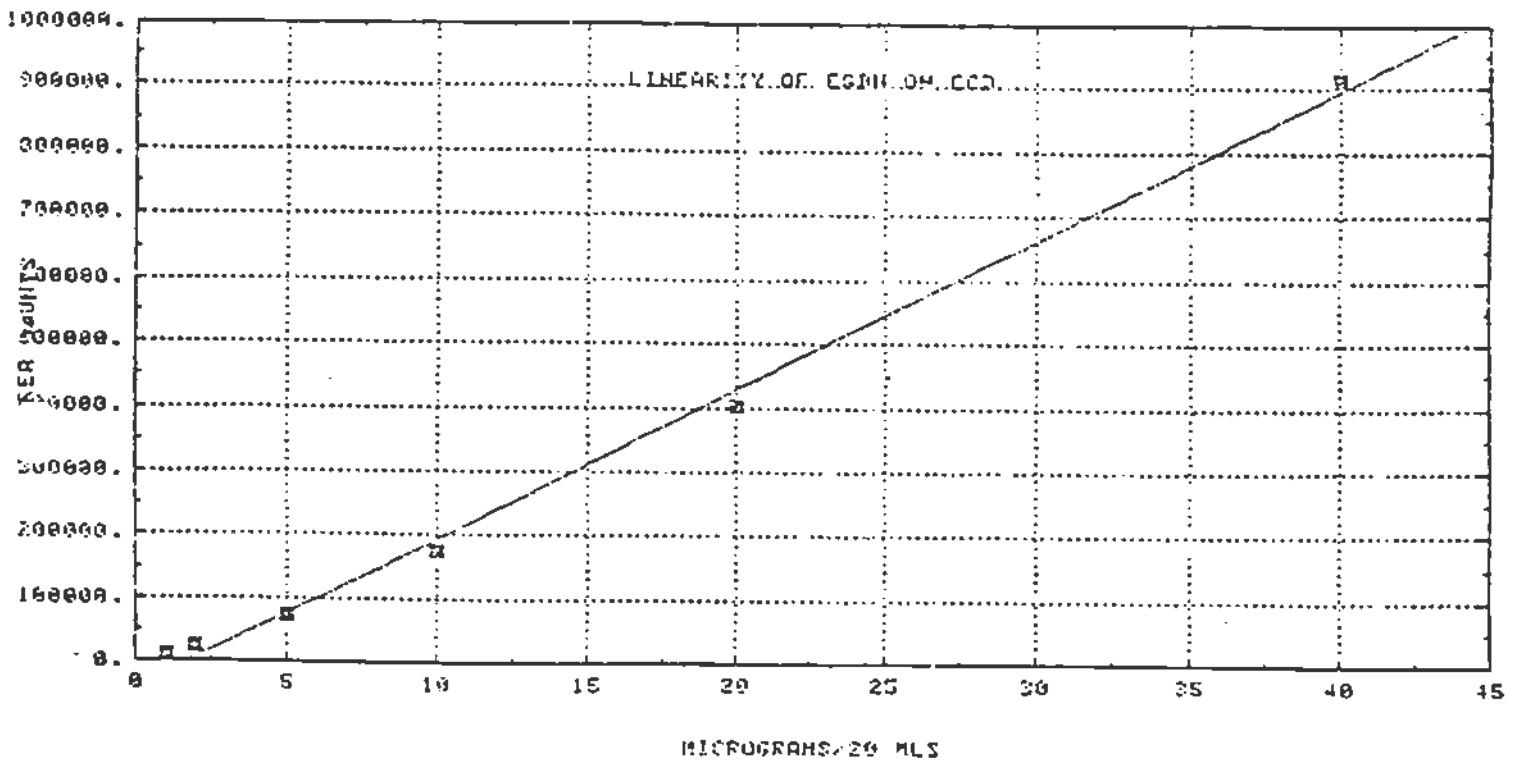

FIGURE A-8. LINEARITY OF EGDN ON ECD

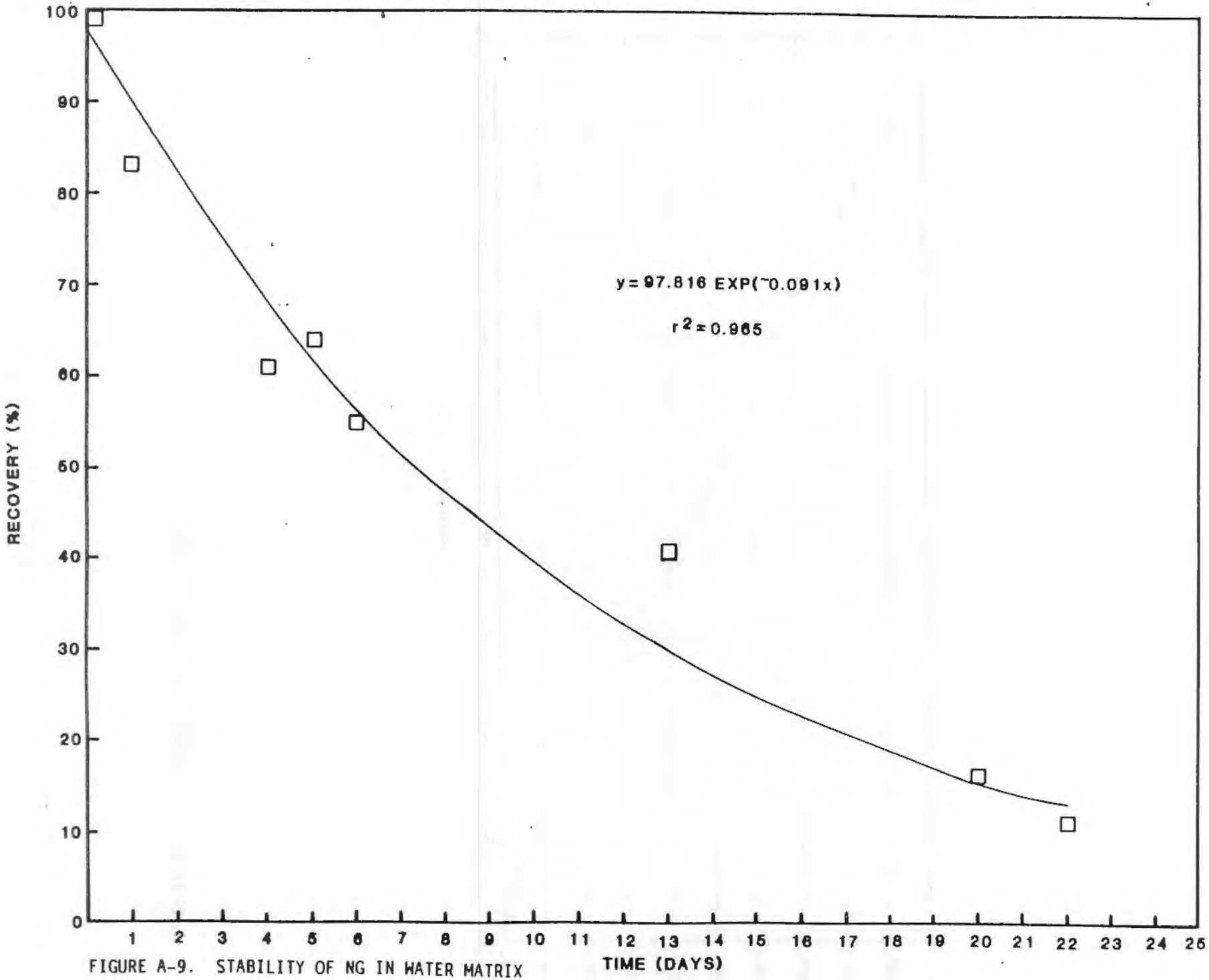

FIGURE A-9. STABILITY OF NG IN WATER MATRIX

TABLE A-3. PRECISION ANALYSIS FOR STANDARD SOLUTIONS OF SURROGATE COMPOUNDS

Concentration (µg per 20 mL solvent)	Compound	Relative Standard Deviation (%)
1.0	3,4-DNT	24.871
	2,4,5-TNT	41.204
	EGDN	0.431
2.0	3,4-DNT	- -
	2,4,5-TNT	- -
	EGDN	1.620
5.0	3,4-DNT	19.058
	2,4,5-TNT	19.864
	EGDN	2.300
	HMX	
10.0	3,4-DNT	- -
	2,4,5-TNT	- -
	EGDN	2.500
20.0	3,4-DNT	11.209
	2,4,5-TNT	13.698
	EGDN	1.700
40.0	3,4-DNT	7.471
	2,4,5-TNT	30.038
	EGDN	1.100
80.0	3,4-DNT	10.419
	2,4,5-TNT	12.065
	EGDN	- -
100.0	3,4-DNT	3.398
	2,4,5-TNT	8.542
	EGDN	- -

After the 3-week time period was over, the flask which contained the spiked water was rinsed with toluene followed by acetone. All samples were analyzed for NG using the methodologies discussed in Appendix B of this QA/QC plan.

2.1.2. Results of this stability study are illustrated in Figure A-9. The mean concentration for NG in the day zero sample was 188 µg/mL, which is 12 percent greater than the theoretical concentration of 167 µg/mL. Therefore, all recoveries are based on the originally observed value of 188 µg/mL versus the theoretical value of 167 µg/mL. Exponential regression analysis was performed on the analytical data and the curve for the percent loss versus time has a correlation coefficient of 0.965. According to the resulting curve, 50 percent of the original quantity of NG in a sample would have degraded in less than 8 days and losses in the first couple of days can also be appreciable. Each chromatogram was closely reviewed in an attempt to determine the degradation product, but neither the GC or subsequent GC/MS analysis could provide conclusive identification of the degradation compound.

2.2. Shakeout Extractions.

2.2.1. An extraction efficiency study using shakeout procedures was conducted by bench scale testing to determine if the extraction procedure with toluene would be effective for NG, 2,4-DNT, 2,6-DNT, 2,4,6-TNT and RDX. Toluene was demonstrated to be an efficient solvent for the extraction of NG and TNT and DNT isomers from water. However, for low concentrations of RDX/HMX in water (50 ppb), recoveries of the target compounds were very low using toluene. Figure A-10 illustrates the variability of the extraction efficiency with various toluene/iso-amylacetate concentrations as a binary solvent mixture. For this reason, when RDX is a target analyte, the extraction solvent is a mixture of iso-amylacetate and toluene (95:5 volume percent). Similar behavior was not evident at higher concentrations in water or at any concentration in the resin matrix. The methodology required evaluation of a low spike level and a high spike level condition for both the water and resin extractions. The resin extractions matched previous sampling train procedures (2.5 grams of XAD-2 resin with 20 mL of toluene) and the water extractions consisted of using a 4:1 water-to-toluene ratio. Results from these extraction studies are summarized in Table A-4. An additional extraction study was conducted to determine if subsequent extractions were needed to improve the efficiency of NG recovery. The results of this testing are summarized in Table A-5. A zero percent recovery indicates the extract had less than the detectable level of the POHC compound.

2.2.2. Recoveries demonstrated for NG (refer to Table A-4) are all in excess of 90 percent. Recoveries for 2,4-DNT and 2,6-DNT are slightly in excess of 100 percent. The additional extraction study (refer to Table A-5) demonstrates that a single extraction should be sufficient to obtain efficient recoveries of the POHC compounds from the sample train.

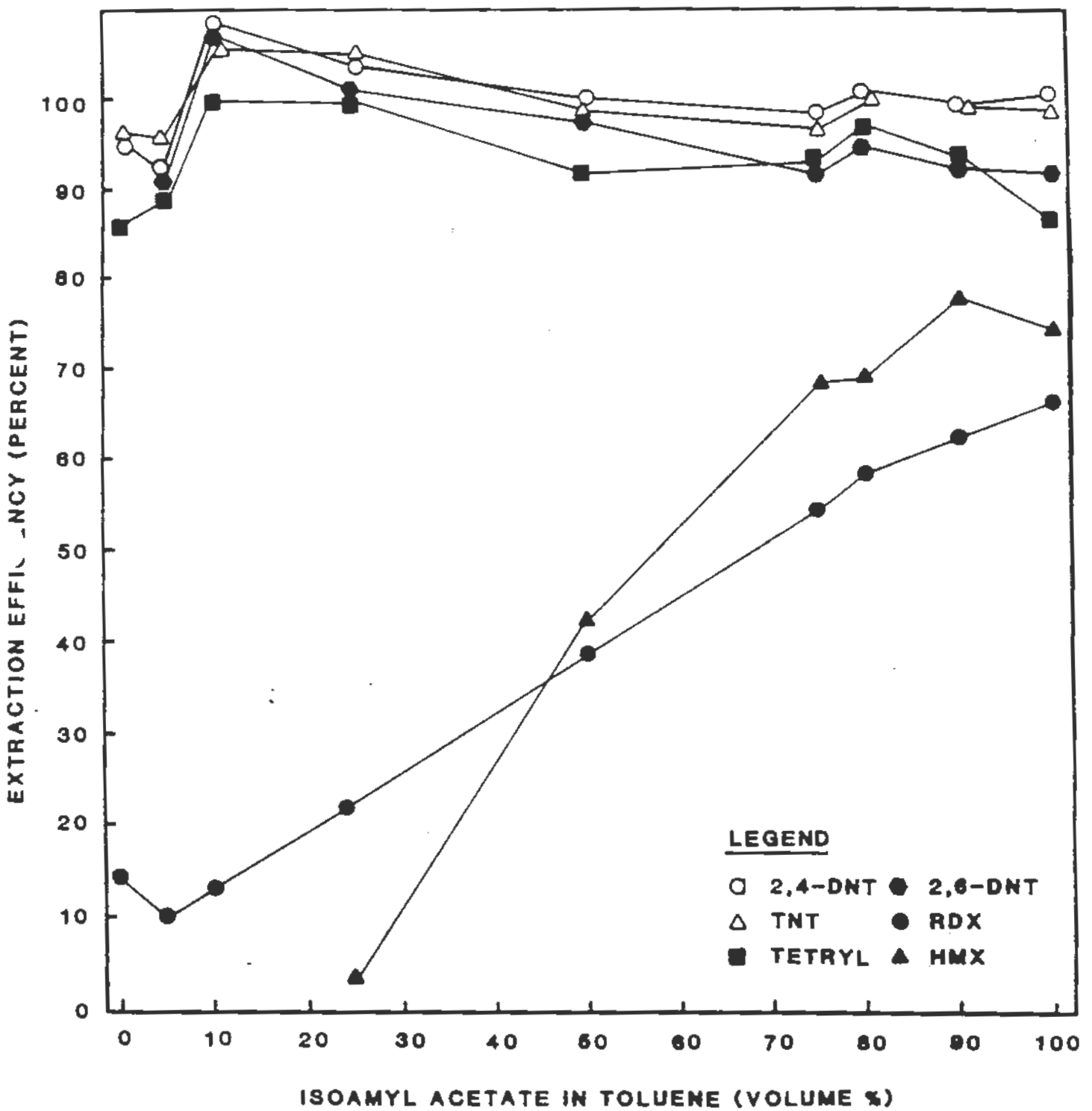

FIGURE A-10. EFFECT OF VARIABILITY IN CONCENTRATION OF TOLUENE/ISO-AMYLACETATE EXTRACTION SOLVENT ON EXTRACTION EFFICIENCY OF 50 ppb ENERGETIC MATERIAL IN WATER

TABLE A-4. EXTRACTION EFFICIENCY STUDY

Matrix	Compound	Spike Condition	Spike Level (µg)	Average Recovery (%)	Standard Deviation (%)
Resin	NG	Low	10	101	8
		High	100	131	9
	2,4-DNT	Low	20	118	7
		High	200	100	4
	2,6-DNT	Low	20	124	9
		High	200	116	3
	RDX	Low	100	100	5
		High	1000	67	3
	TNT	Low	20	106	6
		High	200	83	4
Water	NG	Low	5	91	10
		High	50	98	4
	2,4-DNT	Low	10	121	11
		High	100	122	3
	2,6-DNT	Low	10	105	7
		High	100	110	3
	RDX	Low	50	111	18
		High	500	94	5
	TNT	Low	10	125	10
		High	100	118	3

TABLE A-5. EFFECT OF SUBSEQUENT EXTRACTION ON OVERALL EXTRACTION EFFICIENCY

Spike Level	Sample Matrix/Extraction	Average Recovery (%) NG	2,4-DNT	2,6-DNT	RDX	TNT
1 µg each	Water/first	109	125	122	11	103
	Water/second	0	0	0	D	0
	Resin/first	0	96	100	0	69
	Resin/second	0	0	0	0	0
2 µg each	Water/first	83	123	121	72	113
	Water/second	0	0	0	0	0
	Resin/first	91	121	109	104	100
	Resin/second	0	0	0	0	0
10 µg each	Water/first	96	99	100	61	93
	Water/second	0	0	0	20	0
	Resin/first	60	110	115	39	78
	Resin/second	0	0	0	6	7
100 µg each	Water/first	89	103	97	52	91
	Water/second	3	4	4	25	5
	Resin/first	93	103	98	64	72
	Resin/second	0	0	0	13	10

2.3. Shakeout and Soxhlet Extractions. Also as a result of the meeting held with EPA in 1986, a bench scale test was conducted to evaluate the completeness of recovery of the target compounds from the resin using shakeout techniques. Furthermore, the size of each resin section was increased from 2.5 to 5.0 grams, but the volume of solvent remained unchanged (20 mL of toluene). The results for these multiple extractions are summarized in Table A-6. For the compounds of interest, the majority of the recovery occurs in the first extraction and under good solvent/resin mixing the recoveries are greater than 80 percent. The spiked resins following the third extraction were soxhlet extracted to ensure complete recovery was being achieved. Seven solvents were evaluated for their efficiencies towards extracting these energetic compounds in a soxhlet apparatus. These solvents were: acetone, methylene chloride, isooctane, toluene, pentane, acetonitrile, and methanol. A summary of the results for determining a suitable solvent are contained in Table A-7.

TABLE A-6. RECOVERY OF ANALYTES FROM MULTIPLE EXTRACTIONS OF 5 GRAM RESIN SECTIONS USING SHAKEOUT TECHNIQUES

Spike Level	Number	NG	2,6-DNT	2,4-DNT	3,4-DNT	2,4,6-TNT	2,4,5-TNT
2 µg	First	65.0	75.0	80.0	80.0	80.0	70.0
	Second	20.0	12.5	9.0	6.0	2.5	9.0
	Third	0.0	5.0	0.0	0.0	1.5	0.0
	Total	85.0	92.5	89.0	86.0	84.0	79.0
10 µg	First	97.0	90.0	88.0	89.0	94.0	99.0
	Second	0.0	7.0	8.0	6.0	2.0	0.0
	Third	0.0	0.0	0.0	0.0	0.0	0.0
	Total	97.0	97.0	96.0	95.0	96.0	99.0
50 µg	First	91.6	92.0	95.4	91.2	92.4	96.0
	Second	0.0	0.0	0.0	0.0	1.2	0.0
	Third	1.4	2.2	0.4	0.6	0.0	0.0
	Total	93.0	94.2	95.8	91.8	93.6	96.0
100 µg	First	101.0	77.3	72.4	73.0	72.2	74.8
	Second	0.0	11.8	14.7	14.9	14.1	7.9
	Third	1.1	0.0	0.0	0.0	0.5	0.0
	Total	102.1	89.1	87.1	87.9	86.8	82.7

TABLE A-7. RECOVERY OF ANALYTES FROM SPIKED RESIN USING SOXHLET EXTRACTION - SPIKE LEVEL OF 150 µg

	Average Recovery (%)					
Solvent	NG	2,4-DNT	2,6-DNT	3,4-DNT	2,4,6-TNT	2,4,5-TNT
Acetone	106	101	96	96	84	79
Methylene Chloride	115	103	97	94	85	83
Isooctane	63	56	36	39	24	14
Toluene	101	92	92	91	90	91
Pentane*	49	47	27	29	16	6
Acetonitrile	92	85	82	83	79	78
Methanol	85	70	59	69	60	51

* - Based on single sample analysis

Because methylene chloride demonstrated the greatest average efficiency for all six compounds and because EPA methodology currently uses methylene chloride for the soxhlet methodology, methylene chloride was used to evaluate the effectiveness of the shakeout procedure. However, methylene chloride does present problems in the analysis via GC with an electron capture detector. Each resin section when placed in the soxhlet apparatus still contained interstitial solvent (toluene). Therefore, the methylene chloride was evaporated from the resulting sample using sample "sweat-down" techniques until the toluene was the major concentration solvent (>95 percent). Due to the labile nature of the target compounds, standard solutions of the six target compounds in methylene chloride were first subjected to the sweat-down and then redissolved in toluene. The recoveries obtained are summarized in Table A-8.

TABLE A-8. RECOVERY OF TARGET COMPOUNDS FROM KUDERNA-DANISH CONCENTRATION OF METHYLENE CHLORIDE SOLUTIONS

		Average Recovery (%)				
Spike Level	NG	2,4-DNT	2,6-DNT	3,4-DNT	2,4,6-TNT	2,4,5-TNT
2 µg	83	92	92	97	57	36
10 µg	30	97	90	101	74	71
50 µg	69	129	133	139	122	104
100 µg	81	108	114	118	109	84

The sweat-down of these samples even under mild conditions is not desirable. The recoveries demonstrate that while some recoveries are high, the possibility still exists for loss of the POHC or its surrogate. The spiked resins referred to above were soxhlet extracted with methylene chloride and after correcting for the residual analyte from the third extraction, the concentration of the analytes in the solutions from the third extract was nondetectable at the 0.05 µg/mL level.

3. Sampling Procedure.

3.1. The first phase of the field validation involved the sampling of incinerator burner exhaust by sampling trains spiked with NG, 2,4-DNT, and 2,6-DNT and the matching of the spike recoveries with those from identical trains which received no sampling exposure. The spiking locations were the first resin section and the first impinger prior to the sampling exposure, and the filter following the sampling exposure. In order to facilitate spiking the first impinger, 50 mL of distilled/deionized water was added,

even though this differs from the configuration of the train as discussed in Section 6 of this QA/QC plan. Since this impinger has a short stem, no impinging action took place. Three different spike levels were used and for each spike level a second train was spiked, but it received no sampling exposure. These second trains are referred to as the QA trains. The recovery results are summarized in Table A-9. Variations in the results for NG recovery during Trial 1 testing prompted a second trial with spiked trains with improved analytical techniques (refer to Table A-10). Results with the QA trains demonstrated that the sampling exposure did not affect spike recovery, and therefore, these trains were not repeated in the second trials. The NG recoveries obtained during the second trial were greater than 90 percent and more consistent results were encountered.

3.2. The second phase consisted of actual field testing [during the incineration of propellent, explosive, pyrotechnic (PEP) materials] of the sampling train and comparison of the POHC collection performance between the simultaneous operation of this train and the EPA sampling train configuration. The collection efficiency for the USAEHA sampling train configuration are summarized in Tables A-10 through A-13. Based on individual runs, the two sampling train configurations may be considered equivalent.

3.3. An additional concern with the sampling train configuration is the ability to cool high temperature stack gases to less than 68 °F prior to entering the resin cartridge. A bench scale test was conducted in June 1987 to obtain temperature profiles of the exhaust gas at different locations throughout the sampling train (reference 14). For ΔH of 1.0, the temperature of the exhaust gas entering the resin cartridge and exiting the last impinger was below 60 °F even though the temperature of the flue gas entering the train ranged from 1,746 to 1,852 °F. For ΔH of 2.5, the exhaust gas temperature entering the resin cartridge was below 68 °F and the temperature of the gas exiting the last impinger was below 68 °F. The temperature range of the gas entering the sampling train was 1,728 to 1,802 °F. The sampling train, therefore, is capable of lowering the temperature of gas sampled from high temperature stacks to meet the temperature requirement for the resin (<68 °F) with three impingers before the resin cartridge and an ice bath.

TABLE A-9. SAMPLING TRAIN SPIKE RECOVERY (INITIAL)

Condition*	Spike Location	Average Recovery (%)/ Standard Deviation (%) NG	2,6-DNT	2,6-DNT
1X STD Train	Filter	103/6	108/6	109/7
	Impinger	73/18	109/7	106/9
	Resin	156/30	98/3	94/8
1X QA Train	Filter	102/11	112/3	110/2
	Impinger	89/1	109/4	108/5
	Resin	69/†	104/†	106/†
2X STD Train	Filter	104/25	100/11	96/10
	Impinger	83/44	113/1	109/3
	Resin	109/5†	†	95/9
2X QA Train	Filter	84/†	67/1	62/1
	Impinger	134/45	113/1	106/1
	Resin	76/2	121/2	116/1
10X STD Train	Filter	96/16	119/2	118/1
	Impinger	138/28	115/2	110/2
	Resin	135/41	108/4	100/5
10X QA Train	Filter†	95/21	124/5	122/7
	Impinger	127/4	119/3	119/3
	Resin	92/15	122/3	122/4

* The baseline level (1X) from spiked analytes are as follows: 10 µg NG, 5 µg 2,4-DNT, and 5 µg 2,6-DNT. Each of the spike levels are a multiple of this baseline.
† This note indicates either the data are limited or affected by a lost sample, an analytical problem, or an original spiking error.

TABLE A-10. COMPARISON OF SAMPLING TRAIN SPIKE RECOVERY FOR NITROGLYCERIN

Train Type	Spike Level (µg)	Spike Location	Trial 1		Trial 2	
			Average Recovery (%)	Standard Deviation (%)	Average Recovery (%)	Standard Deviation (%)
Standard	10 µg	Filter	103	6		
		Impinger	73	18		
		Resin	156	30		
QA	10 µg	Filter	102	11		
		Impinger	89	1		
		Resin	69	-		
Standard	20 µg	Filter	104	25	99	1
		Impinger	83	44	103	4
		Resin	109	5	92	5
QA	20 µg	Filter	84	-		
		Impinger	134	45		
		Resin	76	2		
Standard	100 µg	Filter	96	16	100	3
		Impinger	138	28	105	2
		Resin	135	41	99	3
QA	100 µg	Filter	95	21		
		Impinger	127	4		
		Resin	92	15		

TABLE A-11. NITROGLYCERIN COLLECTION EFFICIENCY

Run Number Train Configuration	3-4 USAEHA	3-4 EPA	5-6 USAEHA	5-6 EPA	7-8 USAEHA	7-8 EPA
Gas Volume Sampling (std. cubic feet, dry)'	61.9	57.8	64.0	63.1	69.6	66.3
Total POHC Collected (µg)	19.7	14.5	33.1	35.6	19.7	24.1
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	81.7	14.5	68.9	11.0	76.1	0
2d extraction	0	0	6.6	0	0	0
3d extraction	0	0	0	0	0	0
Resin						
1st section	18.3	85.5	24.5	59.6	23.9	76.3
2d section	0	0	0	9.8	0	0
3d section	0	0	0	0.0	0	0
4th section	0	0	0	10.1	0	0
Condensate Trap						
1st extraction	-	0	-	9.6	-	14.9
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

See footnote on page A-24.

Run Number Train Configuration	9-10 USAEHA	9-10 EPA	11-12 USAEHA	11-12 EPA	13-14 USAEHA	13-14 EPA
Gas Volume Sampling (std. cubic feet, dry)	36.8	37.0	42.0	39.9	40.4	38.1
Total POHC Collected (µg)	16.7	16.3	12.2	14.6	15.4	14.0
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	64.1	9.2	36.1	11.0	58.4	33.6
2d extraction	0	0	0	0	0	0
3d extraction	0	0	0	0	0	0
Resin						
1st section	35.9	62.0	63.9	77.4	41.6	66.4
2d section	0	13.5	0	11.6	0	0
3d section	0	7.4	0	0	0	0
4th section	0	8.0	0	0	0	0
Condensate Trap						
1st extraction	-	0	-	0	-	0
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

* A zero percentage of the total NG collected indicates that none was detected in that sample. The quantitation limits for NG ranged from 0.8 µg to 2.0 µg per sample and were a function of the chromatogram complexity for each sample.

TABLE A-12. 2,4 DINITROTOLUENE COLLECTION EFFICIENCY

Run Number Train Configuration	9-10 USAEHA	9-10 EPA	11-12 USAEHA	11-12 EPA	13-14 USAEHA	13-14 EPA
Gas Volume Sampling (std. cubic feet, dry)	36.8	37.0	42.0	39.9	40.4	38.1
Total POHC Collected (µg)	127.2	127.8	141.5	138.5	158.2	145.1
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	52.9	11.8	49.1	25.3	57.8	25.3
2d extraction	4.6	0	3.0	1.0	4.6	3.1
3d extraction	0	0	0	0	2.0	0
Resin						
1st section	41.1	66.1	47.9	69.1	35.7	58.4
2d section	1.0	11.7	0	4.3	0	9.0
3d section	0	4.3	0	0.0	0	0
4th section	0	6.0	0	0	0	0
Condensate Trap						
1st extraction	-	0	-	0	-	0
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

See footnote on page A-26.

Run Number Train Configuration	15-16 USAEHA	15-16 EPA	17-18 USAEHA	17-18 EPA	19-20 USAEHA	19-20 EPA
Gas Volume Sampling (std. cubic feet, dry)	81.3	77.7	69.8	65.4	72.9	72.0
Total POHC Collected (µg)	1303	1295	1924	1928	1792	1498
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	47.0	4.3	50.2	4.6	56.2	4.5
2d extraction	3.2	0.3	7.9	0.8	3.9	0.5
3d extraction	1.8	0.2	1.8	0.3	1.5	0.2
Resin						
1st section	47.7	65.0	40.1	61.4	38.2	77.0
2d section	0.3	14.2	0	15.8	0.3	11.3
3d section	0	6.3	0	5.5	0.0	3.7
4th section	0	3.7	0	3.7	0	1.7
Condensate Trap						
1st extraction	-	4.7	-	5.7	-	1.0
2d extraction	-	1.0	-	1.8	-	0
3d extraction	-	0.4	-	0.5	-	0

* A zero percentage of the total 2,4 DNT collected indicates that none was detected in that sample. The quantitation limits for 2,4 DNT ranged from 1.0 µg to 3.0 µg per sample and were a function of the chromatogram complexity for each sample.

TABLE A-13. 2,6 DINITROTOLUENE COLLECTION EFFICIENCY

Run Number Train Configuration	15-16 USAEHA	15-16 EPA	17-18 USAEHA	17-18 EPA	19-20 USAEHA	19-20 EPA
Gas Volume Sampling (std. cubic feet, dry)	81.3	77.7	69.8	65.4	72.9	72.0
Total POHC Collected (µg)	104.8	89.4	124.5	99.8	119.2	88.1
POHC Distribution (% of total)*						
Impinger/Condenser						
1st extraction	38.9	0	42.4	0	38.2	0
2d extraction	0	0	6.7	0	3.9	0
3d extraction	0	0	3.5	0	0	0
Resin						
1st section	61.1	72.1	47.4	71.6	57.9	91.4
2d section	0	16.8	0	12.7	0	8.6
3d section	0	7.0	0	5.6	0	0
4th section	0	4.0	0	3.2	0	0
Condensate Trap						
1st extraction	-	0	-	6.8	-	0
2d extraction	-	0	-	0	-	0
3d extraction	-	0	-	0	-	0

* A zero percentage of the total 2,6 DNT collected indicates that none was detected in that sample. The quantitation limits for 2,6 DNT ranged from 1.0 µg to 3.0 µg per sample and were a function of the chromatogram complexity for each sample.

APPENDIX B

ANALYTICAL PROCEDURE
ENERGETIC COMPOUNDS IN STACK GAS SAMPLES

1. Scope and Application.

1.1. This method provides procedures for the detection and quantitation of energetic compounds from stack gas samples. The procedure is organized into a general section, which covers those aspects of the method which are independent of the target analyte, and specific sections for each compound validated on the sampling train.

1.2. Samples are collected in a modification of the EPA Method 5 sampling train where a 20-gram XAD-2 resin module has been placed downstream of three impingers. Therefore, the target compounds are captured in both the resin and water matrices. The sampling rate is on the order of 1-cubic meter of stack gas per hour.

1.3. This method is designed for use by analysts who are experienced in the use of a GC and a GC/MS.

2. Summary of Method. This method provides gas chromatographic operating conditions for the analysis of energetic compounds in a suitable extraction solvent (toluene or a mixture of toluene and iso-amylacetate). These samples are obtained from the solvent extraction of stack sampling train media - filter, impinger water, and resin. The target compounds are separated in the GC by temperature programming of a capillary column. These compounds are than detected by either a nitrogen/phosphorus (NP) or an electron capture detector (ECD). Positive results are confirmed on a GC/MS instrument if concentrations are adequate for GC/MS instrument detection.

3. Interferences. Any compound which has the same general retention time as the target analyte and gives a detector response is a potential interference to the analytical instruments. Laboratory reagent/solvent blanks and extracts from the XAD-2 resin must be analyzed to demonstrate the level of contamination that would interfere with the measurement. Modification of the gas chromatographic parameters may be utilized to circumvent interferences.

4. Safety. The following safety precautions are presented as guidelines only since safety procedures should already be in place for the analytical laboratories.

4.1. Protective Equipment. Throw-away plastic gloves, apron or laboratory coat, and safety glasses should be worn at all times in the laboratory areas. Skin contact with the energetic compounds and solvents should be avoided.

4.2. Personal Hygiene. As a backup to protective equipment, hands and lower arms should be washed thoroughly before any breaks (coffee, lunch, end of shift) with any mild soap.

4.3. Ventilation. The manipulation of samples and the use of reagents/solvents should be restricted to laboratory hoods. The electron capture detector on the gas chromatographic instruments must be properly vented.

4.4. Waste. Proper waste disposal techniques must be utilized and every effort should be made to minimize the generation of contaminated wastes.

5. Apparatus and Equipment.

5.1. Gas Chromatograph. Hewlett-Packard (HP) 5880 GC's capable of temperature programming and which are equipped with HP 7671 autoinjectors and capillary column systems operating in the splitless mode will be utilized. Depending upon the target analyte, the GC will be equipped with either an electron capture or nitrogen-phosphorus detector (see compound-specific sections). A Hewlett-Packard HP3357 Data System is interfaced with the GC for integrating peak areas and recording chromatograms.

5.2. Gas Chromatograph/Mass Spectrometer. The Finnigan Model 9610 system with temperature programming and splitless mode capillary column capabilities will be utilized. The Model 9610 Gas Chromatograph will have a direct interface with a Finnigan Model 4021 mass spectrometer. The mass spectral data are obtained with electron impact ionization at a nominal electron energy of 70 eV. A computerized data system is utilized to acquire, store, reduce and output the mass spectral data.

5.3. Chromatographic Columns. Refer to the compound specific sections.

5.4. Glassware.

5.4.1. Vials. Wheaton, screw-capped, septum vials with a volume of 40 mL and Teflon®-lined caps are used to extract resin sections. Additionally, 2 mL auto-sampler vials supplied by Hewlett-Packard are used.

5.4.2. Jars. Glass jars with Teflon-lined caps capable of holding 16 fluid ounces are used for the transportation of the extracts of impinger water, the samples of feed material and/or process residues (e.g., ash), and the particulate filter with the rinses of the probe liner and train glassware.

5.4.3. Separatory Funnels. Glass separatory funnels of 500 mL volume will be used in the extraction of aqueous samples.

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5.4.4. Miscellaneous. Volumetric flasks with ground glass stoppers, disposable pipets, graduated cylinders, and micro-syringes are used for the preparation of standards, performance of dilutions, and conducting injections to the gas chromatograph instrument.

5.5. Additional Equipment.

5.5.1. Mechanical Shaker. A roto-rack capable of 20 rotations per minute will be used for the extraction of resin samples.

5.5.2. Miscellaneous. Stainless steel spatulas or spoons may be used in preparing the samples for analysis.

6. Reagents and Solvents.

6.1. Solvents. ACS grade: toluene, acetone, iso-amylacetate.

6.2. Reagents.

6.2.1. 99.6 percent or greater purity of 2,4-DNT, 2,6-DNT, 3,4-DNT, 2,4,6-TNT, 2,4,5-TNT, NG, and RDX (SRM's from Picatinny Arsenal).

6.2.2. ACS grade anhydrous sodium sulfate.

6.3. Miscellaneous.

6.3.1. Distilled/deionized water.

6.3.2. Ultra high purity gas as required for GC analysis

7. Preparation of Standard Solutions.

7.1. Stock Solutions.

7.1.1. The stock standard solutions are prepared by dissolving 0.050 grams of each potential analyte in 50 mL of acetone. Larger volumes may be used at the discretion of the analyst. If the compound purity is certified at 96 percent or greater, the analyte weight can be used without correction to calculate the concentration of the stock solution.

7.1.2. These stock standards should be transferred to Teflon-sealed screw-cap bottles, stored at approximately 40 °F, and protected from the light by using amber colored containers. Due to the nature of the analytes and the acetone solvent, the stock standards should be monitored frequently for signs of degradation and/or evaporation, especially prior to preparing any intermediate standards.

7.1.3. Stock standards should be replaced after 12 months or sooner if a problem is indicated.

7.2. Intermediate Standards.

7.2.1. Intermediate standard solutions should be prepared using known volumes of the stock standard and diluting the solution with toluene. The intermediate standards should contain 100 µg/mL of each of the target analytes.

7.2.2. Storage and preservation of the intermediate standard solutions are the same as those for the stock solutions.

7.2.3. Intermediate standard solutions should be replaced after 8 weeks or sooner if a problem is indicated.

7.3. External Calibration Standards.

7.3.1. Calibration standards should be prepared at a minimum of three concentration levels but preferably at five concentration levels by adding volumes of one or more of the stock solutions to a volumetric flask and diluting to volume with toluene. One of these concentrations should be at a concentration near but above the method detection limit, and the other concentrations should correspond to the expected range of concentrations found in the field samples.

7.3.2. These solutions should also be preserved according to the general procedures in paragraph 7.1.2. and should be replaced after 15 days.

7.4. Working Standards. Fresh working range standards should be prepared on a daily basis by diluting known volumes of the intermediate standard solutions in toluene. These standards can then be utilized as performance standards for the GC instrument.

7.5. Field Surrogate Spike Solution. A acetone/acetonitrile solution containing one or more surrogate compounds should be prepared at a concentration to be determined by the project engineer (see Section 6 of the QA/QC plan, paragraph 6.2.5) and coordinated with the analyst.

8. Instrument Calibration.

8.1. Gas chromatographic operating parameters should be set at those listed in the compound specific sections of this section so that the retention times are equivalent. The instrument will be calibrated using an external standard calibration technique on a daily basis.

8.2. For the target compounds, calibration standards will be prepared utilizing the procedure in paragraph 7.3 of Appendix B. Each calibration standard should be injected into the GC system using the technique that will be used to introduce the actual samples into the instrument [method (syringe, autoinjector) and volume]. Peak area and corresponding sample concentration/mass should be tabulated. These results will be utilized to prepare a calibration curve for each of the target compounds (POHC's and

surrogates) in terms of detector response versus analyte concentration. In addition to calculating the slope and intercept of the line, the correlation coefficient should be calculated to assess linearity. These calibration curves should be verified throughout the daily sample groups by the analysis of a calibration standard solution. A drift in response for any compound which exceeds 20-relative percent difference will initiate a recalibration of the instrument for that particular compound. For the GC/MS instrument, standards and procedures specified by the manufacturer should be used to tune and calibrate the instrument. A single standard solution should then be analyzed by the GC/MS instrument to determine the response factor for the target compounds, to include the internal standard (anthracene-d_{10}).

9. Retention Time Window Determination.

9.1. Prior to determining retention time windows, the GC instrument should be operated for approximately 48 hours to allow for chromatographic system stabilization.

9.2. Three to five injections of the mixture of target compounds should be analyzed. From this analysis, the mean and standard deviation of the retention times for each compound will be calculated and utilized in defining the retention time windows for the analysis. The retention time window will be defined as $\pm$ two standard deviations ($\pm 2\sigma$) of the mean retention time. For those cases where the standard deviation of the retention time is zero, a value of $\pm$ 0.05 minutes of the retention time will be used as the window. Retention time windows for each target compound must be calculated for each chromatographic column to be utilized. The analyst may find it necessary to modify these windows depending upon the quantity and nature of other detectable compounds in the field samples (i.e., matrix effects). The calibration standards that have been dispersed among the field samples can be utilized as performance checks with regards to retention time windows. If any of the compound peaks fall outside the retention time window established, the chromatographic system should be considered out of control and corrective action should be initiated.

10. Sample Analysis.

10.1. An aliquot of the extracts from all sections of the sampling train will be placed in autosampler vials. The volume of the extract should be known and recorded prior to removal of the aliquot of the sample.

10.2. Prior to analyzing the group of samples for that day, the instrument calibration should have been performed and the retention time windows confirmed. This data should be recorded in the project data file on a daily basis. Each sample is analyzed in triplicate with one of the multilevel standards and a solvent blank placed after every sixth sample. The group of samples should also be bracketed with calibration standards.

10.3. Since linearity has been demonstrated for energetic compound chromatographic response (refer to Appendix A), chromatograms should be manipulated so that all peaks are on-scale. Integration methodologies should be compared with the chromatogram to ensure the methodology is appropriate for the peak shape. If all chromatographic peaks can not be brought on-scale electronically, the samples should be diluted and reanalyzed until all peaks are on-scale. By bringing all peaks on-scale, chromatographic resolution in the vicinity of the peaks corresponding to the target compounds can be examined. If peak resolution is acceptable, the undiluted sample may be used for quantitation.

10.4. Analytical results are obtained by use of the calibration plot prepared for each of the compounds. The integrator has been programmed to report the results in µg/mL.

10.5. No second gas chromatographic column will be utilized for confirmational analysis. Depending upon the compound and the equipment availability, the analyst at his discretion may perform secondary analysis using another detector type. However, 10 percent of the samples positive for either the POHC or the surrogates will be analyzed by GC/MS for confirmation of the identification provided by GC/ECD or GC/NPD analysis. If the concentration of the target compound is sufficient for GC/MS instrument semiquantitative analysis, then the GC/MS should be utilized for confirmation of the quantitation as well as the identification.

10.6. The chromatographic conditions for the analysis of compounds are contained in the sections following this appendix. For those analytical methods which utilize either NP or EC detectors, the selection of which detectors would depend on the type of interferences present in the samples. The selection would also depend on whether NG would also be a target analyte. Calculation methodology is covered in Appendix D.

SECTION B-1

ANALYSIS OF SAMPLES/STANDARDS FOR 2,4,5- AND 2,4,6-TNT

1. Applicability. This methodology is for the analysis of samples for 2,4,6-TNT as the only POHC of interest. The isomer 2,4,5-TNT has been spiked onto the sampling train as a recovery surrogate.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 170 °C
Detector Temperature: 200 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 125 °C for 5 minutes; ramp 2 °C per minute to 200 °C; hold 200 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 240 °C

3. Chromatograms. Example chromatograms are illustrated in Figures B-1.1 and B-1.2 for a standard solution and a field sample containing the TNT isomers.

MULTIPLIER = 20

[hp] 5880A SAMPLER INJECTION @ 19:09 SEP 14, 1984
SAMPLE # : ID CODE :
5 40.8 UG STD
ANALYSIS OF RESIN EXTRACT FOR TRINITROTOLUENE USING ECD
ESTD

RT	AREA	TYPE	CAL	AMOUNT	NAME
2.35	76854.70	PV	1	10.401	UG 2,6-DNT
2.82	135659.00	VV	2	10.381	UG 2,5-DNT
3.28	50730.10	VV	3	10.608	UG 2,4-DNT
4.30	103683.00	VB	4	10.715	UG 3,4-DNT
8.05	310396.00	BB	5	40.810	UG 2,4,6-TNT

FIGURE B-1.1: STANDARD - 2,4,6-TNT

MULTIPLIER = 20

RT: VALUE ALLOTED 0.33

RT: 3.04

RT: SELECTION ON

2,4,6 TNT

[illegible] 5880A SAMPLER INJECTION @ 00:56 SEP 15, 1988
SAMPLE # : ID CODE :
12 10-33-1
ANALYSIS OF RESIN EXTRACT FOR TRINITROTOLUENE USING ECD
STD

RT	AREA	TYPE	CAL	AMOUNT	NAME
2.35	8464.17	HH	1	1.014	UG 2,6-DNT
2.81	12782.30	HH	2	0.858	UG 2,5-DNT
3.29	12114.00	HH	3	2.206	UG 2,4-DNT
4.36	1990.98	HH	4	0.203	UG 3,4-DNT
8.04	403854.00	HH	5	22.335	UG 2,4,6-TNT

FIGURE B-1.2: SAMPLE - 2,4,6-TNT

SECTION 8-2

ANALYSIS OF SAMPLES/STANDARDS FOR 2,4-, 2,6-, and 3,4-DNT

1. Applicability. This methodology is for the analysis of samples for 2,4- and 2,6-DNT as the POHC's of interest. The isomer 3,4-DNT has been spiked onto the sampling train as a recovery surrogate.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 170 °C
Detector Temperature: 200 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 90 °C for 5 minutes; ramp 2 °C per minute to 200 °C; hold 200 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 240 °C

3. Chromatograms. Example chromatograms are illustrated in Figures B-2.1 and B-2.2 for a standard solution and a field sample containing the DNT isomers.

MULTIPLIER = 20

START AUTO SEQ 9: 9

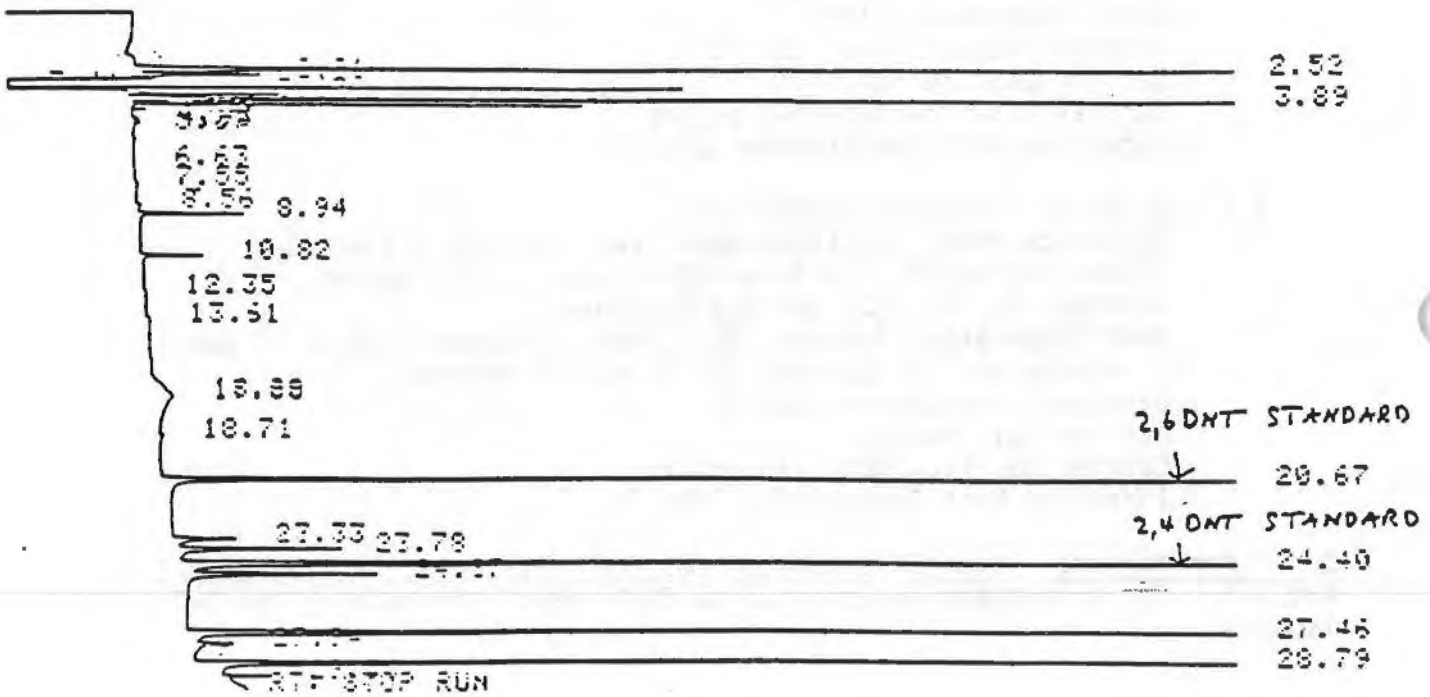


FIGURE B-2.1: STANDARD - 2,4- AND 2,6-DNT

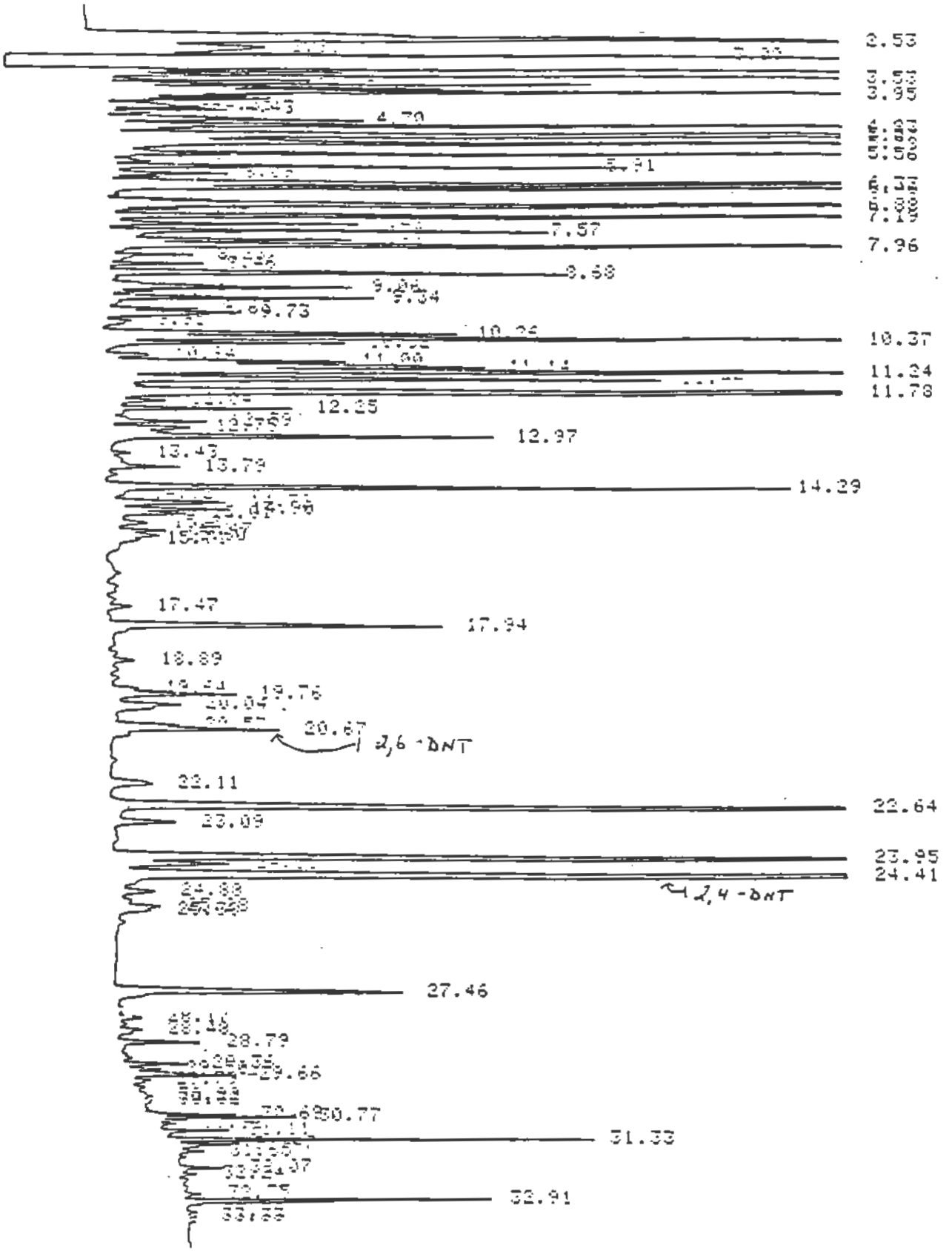


FIGURE B-2.2: SAMPLE - 2,4- AND 2,6-DNT

SECTION 8-3

ANALYSIS OF SAMPLES/STANDARDS FOR NITROGLYCERIN

1. Applicability. This methodology is for the analysis of samples for NG as the only POHC of interest. The compound EGDN has been spiked onto the sampling train as a recovery surrogate.

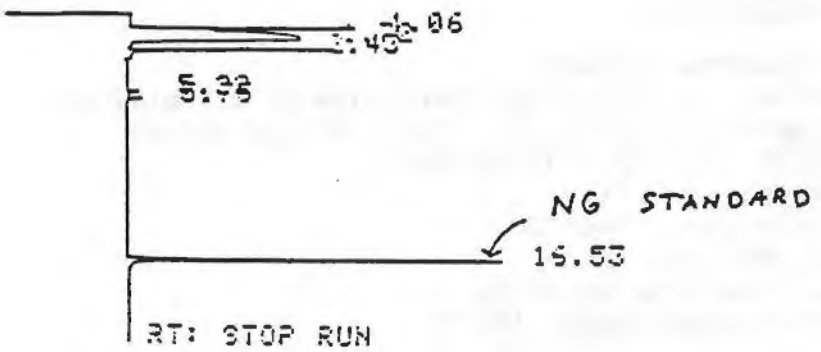
2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 30 meters x 0.32 mm ID - fused silica column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 120 °C
Detector Temperature: 140 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 40 cc/sec
Injection Port Temperature: 140 °C

2.2. Detector: Nitrogen/Phosphorus
Nitrogen/phosphorus detector may not be utilized for NG analysis.

3. Chromatograms. Example chromatograms are illustrated in Figures 8-3.1 and 8-3.2 for a standard solution and a field sample containing nitroglycerin and ethylene glycol dinitrate.

START AUTO SEQ 26, 26



[HP] 5880A SAMPLER INJECTION @ 08:00 DEC 17, 1984
SAMPLE # : ID CODE :
26
METHOD ABORTED
AREA %

RT	AREA	TYPE	AREA %
1.06	28605.80	BV	730.921
1.72	80385.30	VP	2053.970
2.43	10973.30	PV	280.385
5.22	874.16	BP	22.336
5.79	849.33	VP	21.702
16.53	34858.50	BB	890.688

FIGURE B-3.1: STANDARD - NG

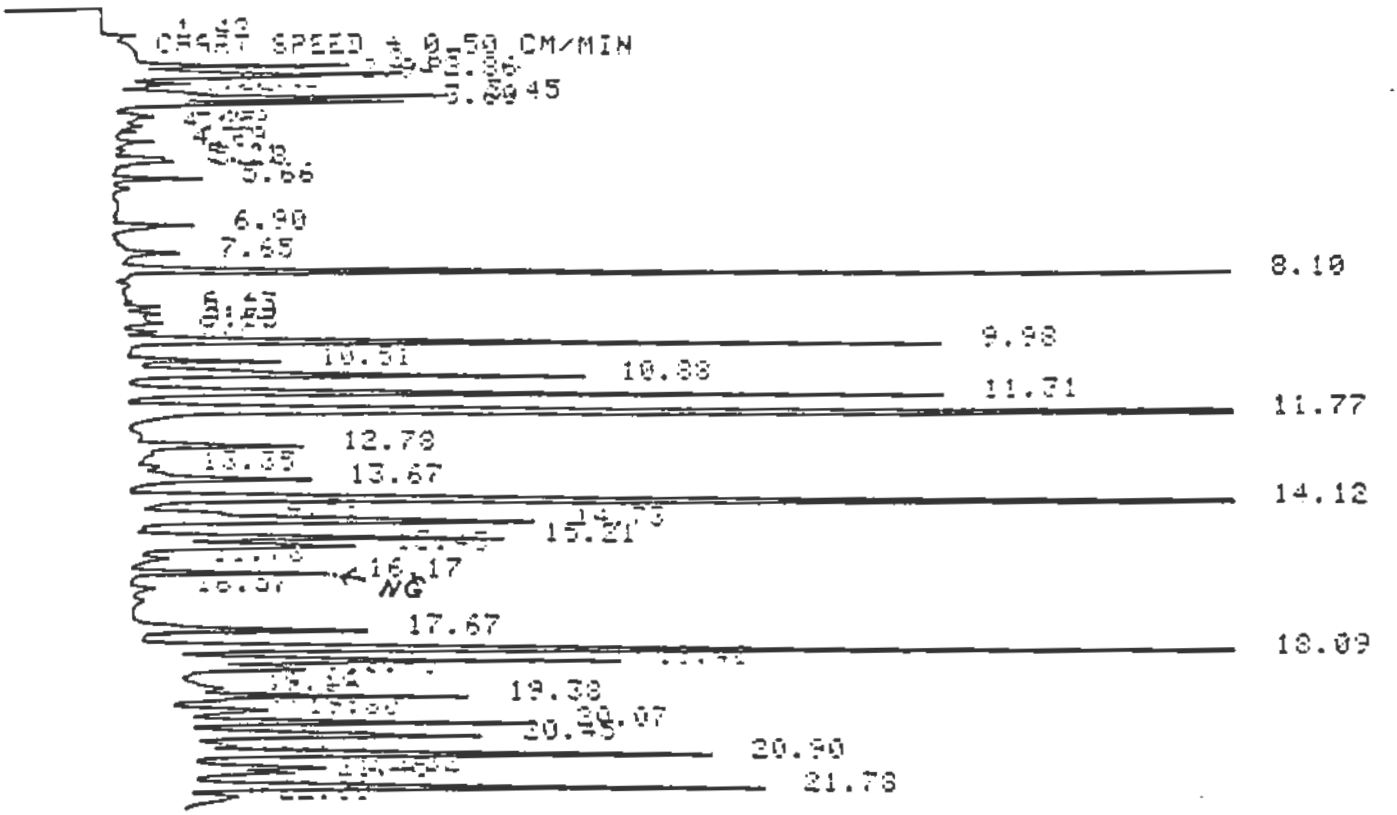
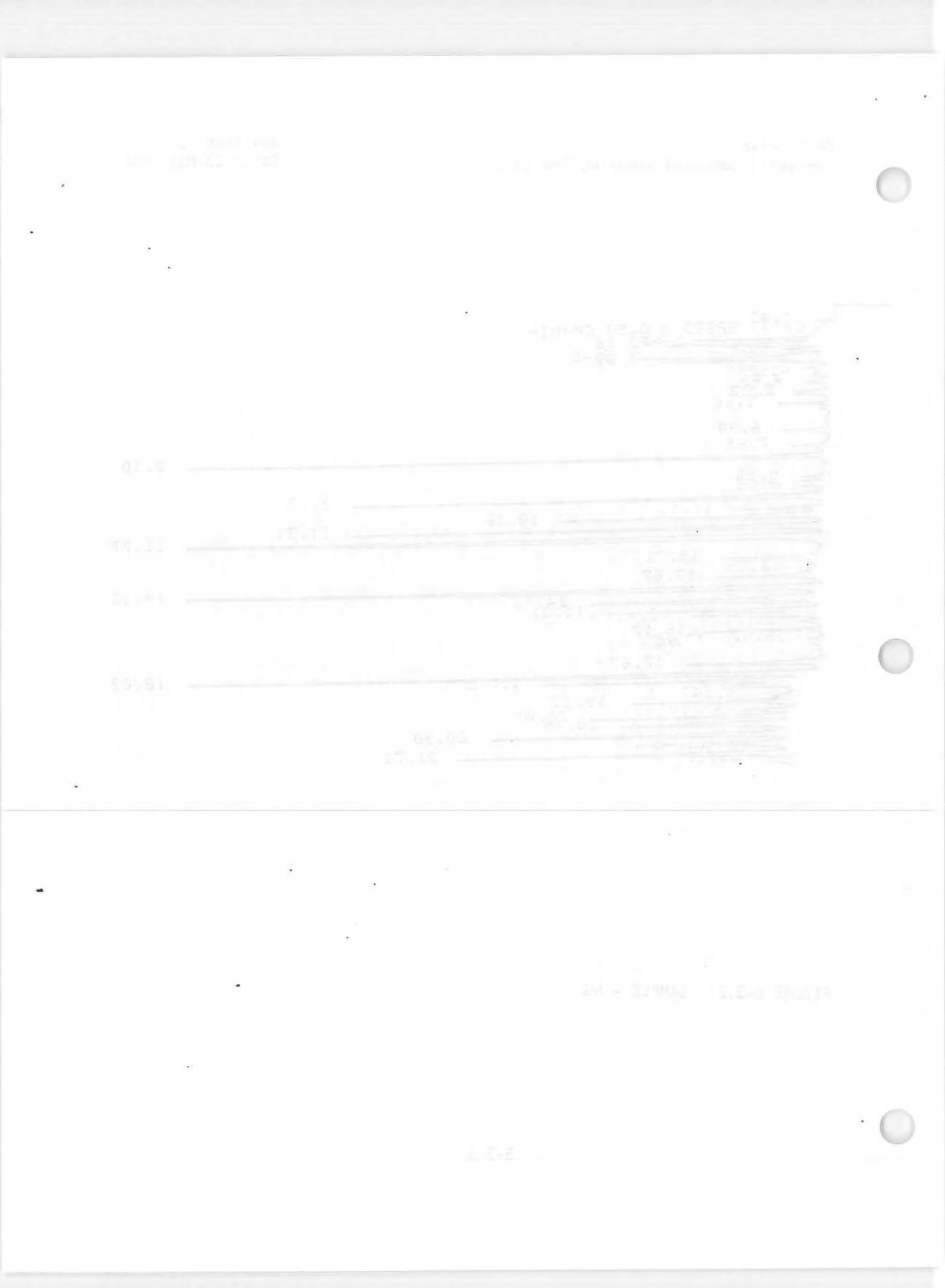


FIGURE B-3.2: SAMPLE - NG



SECTION B-4

ANALYSIS OF SAMPLES/STANDARDS FOR RDX

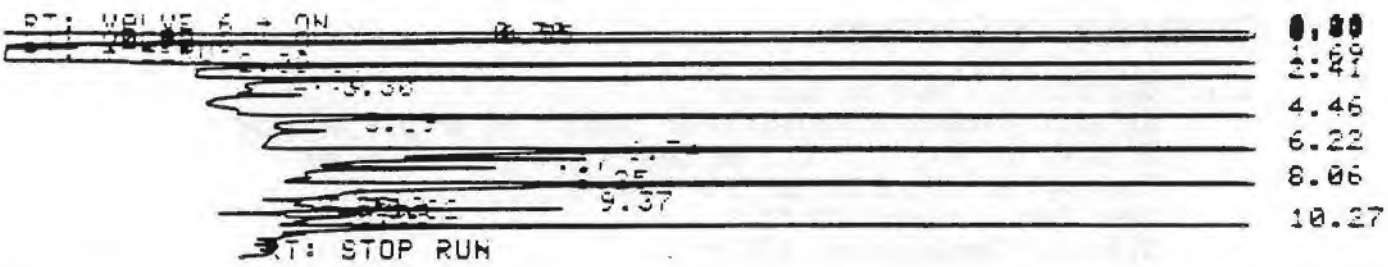
1. Applicability. This methodology is for the analysis of samples for RDX as the only POHC of interest.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica Column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature: 170 °C
Detector Temperature: 200 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 200 °C

2.2. Oetector: Nitrogen/Phosphorus
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 60 meters x 0.32 mm ID - fused silica Column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 125 °C for 5 minutes; ramp 2 °C per minute to 200 °C; hold 200 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 45 cc/sec
Injection Port Temperature: 240 °C

3. Chromatograms. Example chromatograms are illustrated in Figure B-4.1 for a standard solution containing RDX.



[hp] 5880A SAMPLER INJECTION @ 19:35 NOV 9, 1987
SAMPLE # : ID CODE :
8 20PPB-STD
H20 FOR EXPLOSIVES
ESTD COMPENSATED ANALYSIS

RT	AREA	TYPE	CAL	AMOUNT	NAME
1.69	116070.00	BB	1	21.496	2, 6-DNT
2.41	69532.80	BB	2	21.438	2, 4-DNT
4.46	115873.00	BB	3	21.249	2, 4, 6-TNT
6.22	103789.00	BB	4	23.248	RDX
8.06	35432.20	BB	5	22.146	TETRYL
10.27	16750.30	BB	6	30.567	HMX

MULTIPLIER = 1

FIGURE B-4.1: STANDARD SOLUTION CONTAINING RDX

SECTION B-5

ANALYSIS OF SAMPLES/STANDARDS FOR RDX AND DNT/TNT ISOMERS

1. Applicability. This methodology is for the analysis of samples for 2,4,6-TNT, 2,4-DNT, 2,6-DNT and RDX as the POHC's of interest. The isomers 2,4,5-TNT and 3,4-DNT have been spiked onto the sampling train as a recovery surrogate.

2. Chromatographic Conditions.

2.1. Detector: Electron Capture (Thin Phase Column)
Injection Mode: Splitless Mode; Vent time of 0.5 minutes
Column: 10 meters x 0.32 mm ID - fused silica Column
Coating: SE 30, 0.25 µm film thickness
Oven Temperature Program: 100 °C for 1 minute; ramp 25 °C per minute up to 225 °C; hold 225 °C for 2 minutes
Detector Temperature: 275 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 1 mL/min
Injection Port Temperature: 200 °C

2.2. Detector: Nitrogen/Phosphorus (Wide Bore Column)
Injection Mode: Direct on-column; 2 µL
Column: 20 meters x 0.53 mm ID -fused silica Column
Coating: DB-5, 1.50 µm film thickness
Oven Temperature Program: 100 °C for 3 minutes; ramp 10 °C per minute to 225 °C; hold 225 °C for 10 minutes
Detector Temperature: 300 °C
Carrier Gas: Helium
Carrier Gas Flow Rate: 5 mL/min
Carrier Make-up Flow Rate: 25 mL/min
Injection Port Temperature: 130 °C

3. Chromatograms. Example chromatograms are illustrated in Figures B-5.1 for a standard solution containing the target compounds.

[n:] 9960A SAMPLER INJECTION @ 15:01 NOV 10- 1987
SAMPLE # : ID TIDE :
E 20PPB-STD
420 FOR EXPLOSIVES
ESTD COMPENSATED ANALYSIS

RT	AREA	TYPE	CRL	AMOUNT	NAME
1.54	98804.50	BB	1	20.034	2, 6-DNT
3.33	53145.10	BB	2	19.989	2, 4-DNT
4.27	89397.50	BB	3	19.778	2, 4, 6-TNT
6.03	87452.50	BB	4	19.851	RDX
7.66	99214.10	BB	5	19.938	TETRYL
10.17	130462.50	BB	6	25.129	HMX

MULTIPLIER = 1

FIGURE B-5.1: STANDARD SOLUTION

SECTION B-6

CONFIRMATIONAL ANALYSIS BY GC/MS

1. Applicability. This methodology is for the confirmational analysis performed on the samples in order to provide confirmation of the quantitation obtained by GC/ECD or GC/NPD if possible. Otherwise, the identification of the target compound is confirmed.

2. Chromatographic Conditions.

Injection Mode: Splitless for 1.2 minutes Injection
Volume: 2 - 3 µL
Column: 30 meters x 0.32 mm ID - fused silica Column
Coating: DB-5 (permanent bonded SE54), 0.25 µm film thickness
Oven Temperature Program: 150 °C for 1 minute; ramp 8 °C per minute up to 285 °C; hold 285 °C for 20 minutes
Carrier Gas: Helium
Carrier Gas Flow Rate: 10 psi at inlet
Injection Port Temperature: 150 °C
Internal Standard: Anthracene - D_{10}

3. Mass Spectrometric Conditions.

Interface: Direct (fused silica column inserted directly into source chamber)
Ionization Mode: Electron Impact
Electron Energy: 70 eV
Scan Rate: 1 scan per second
Scan Mass Range: 35-350

4. Chromatograms/Spectra. Reconstructed ion chromatograms and mass spectra of the analytes are shown in Figures B-6.1 through B-6.6.

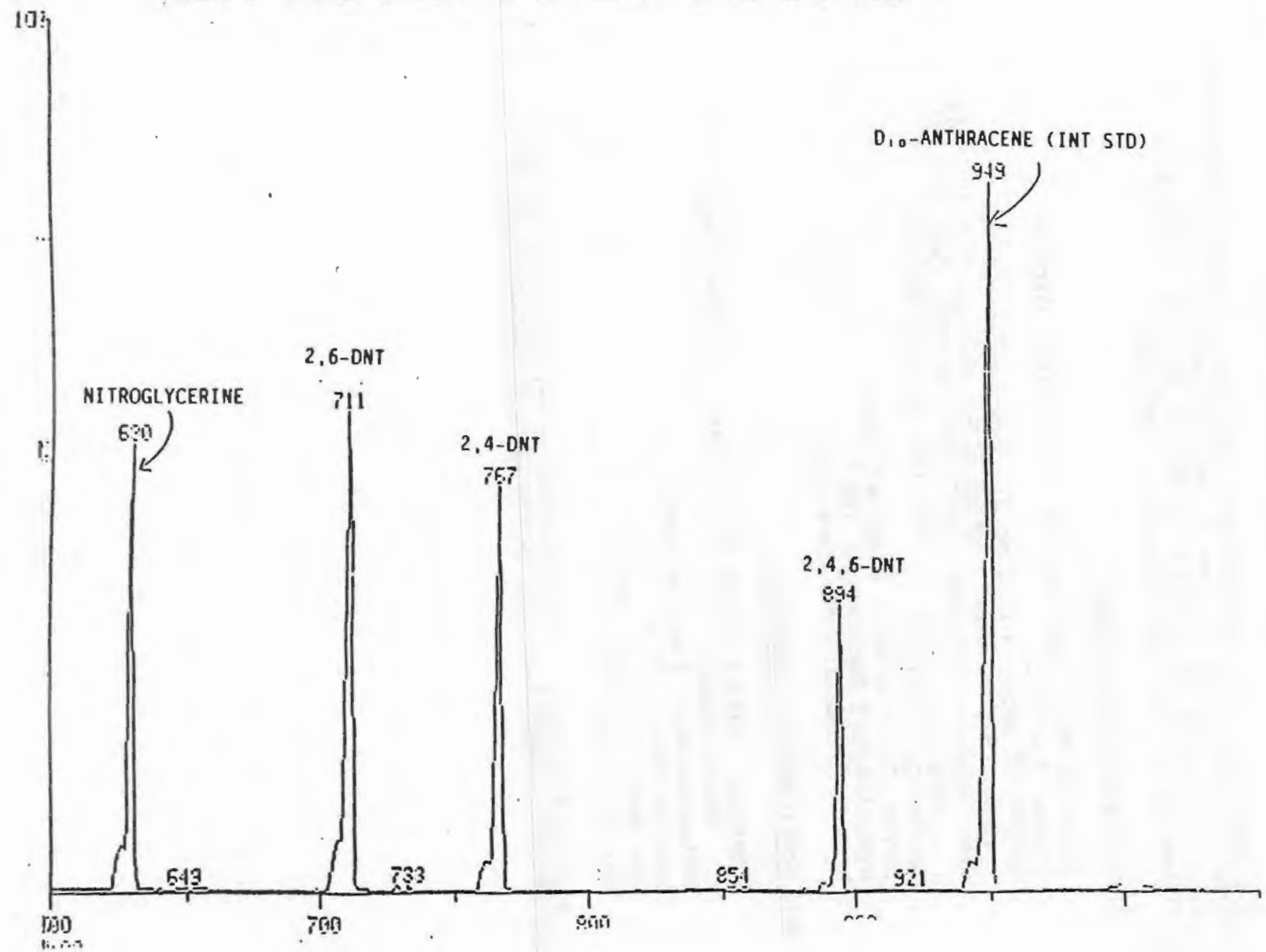


FIGURE B-6.1: GC/MS ANALYSIS OF EXPLOSIVE COMPOUNDS STANDAk.

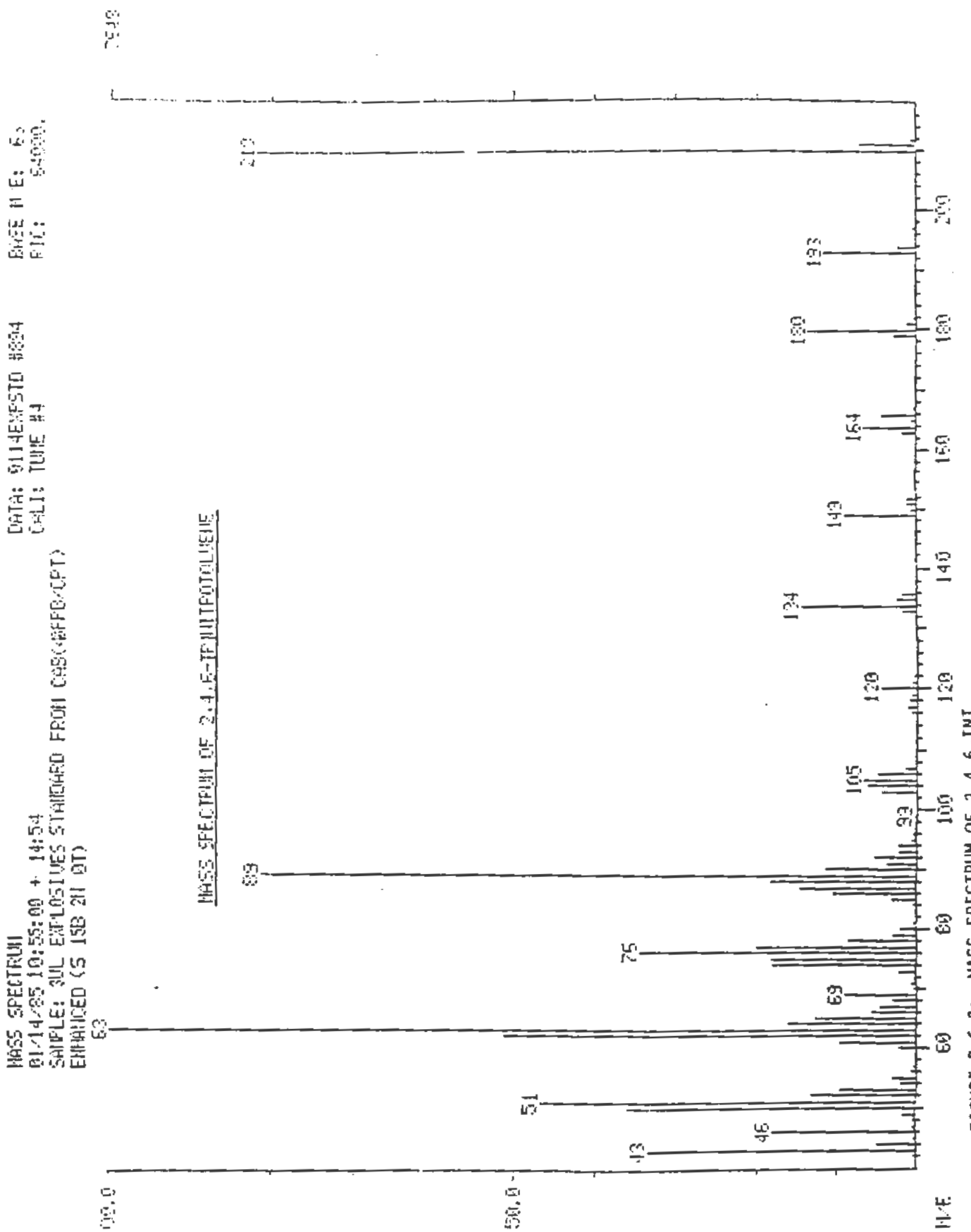


FIGURE B-6.2: MASS SPECTRUM OF 2,4,6-TNT

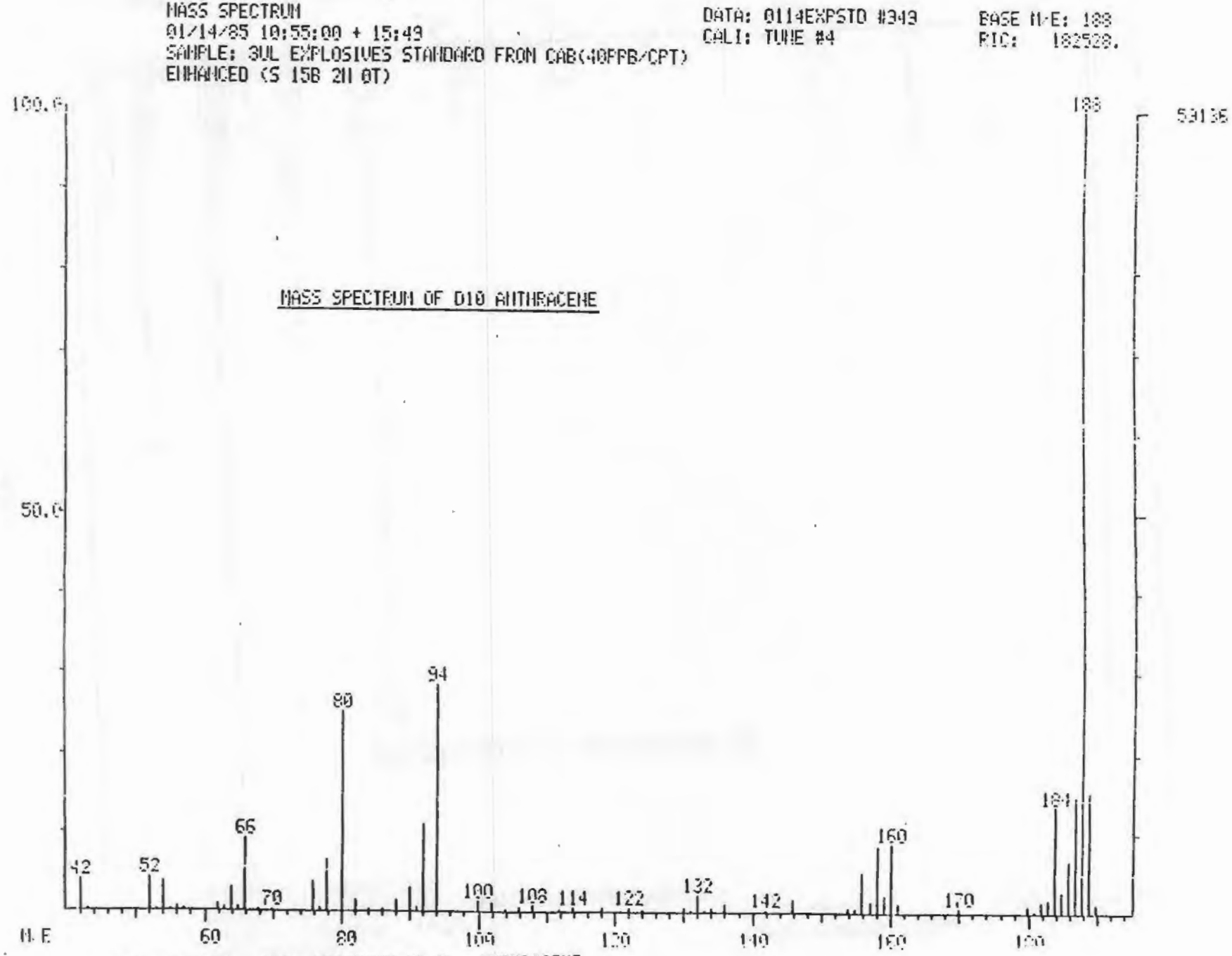


FIGURE B-6.3: MASS SPECTRUM OF D_{10}-ANTHRACENE

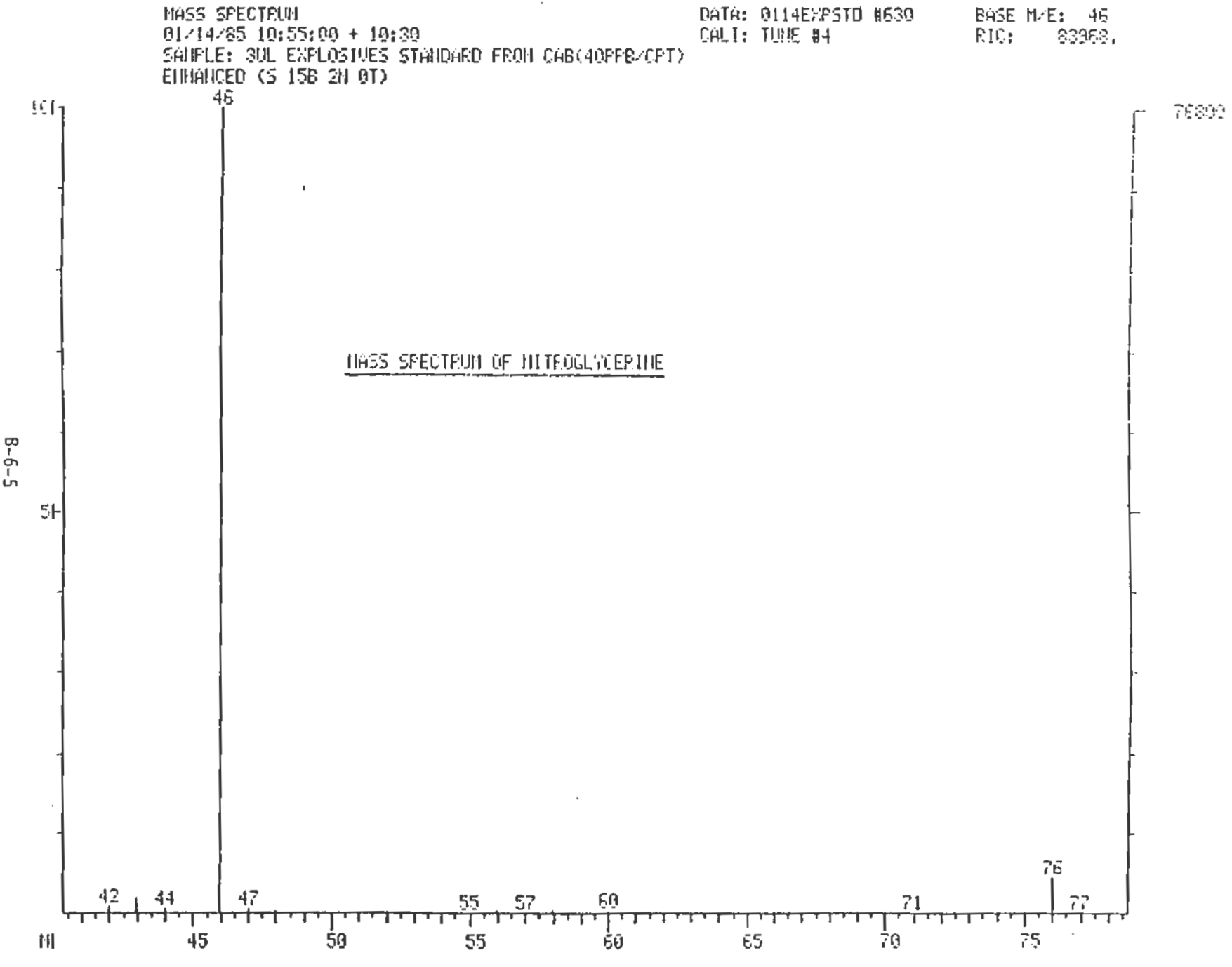


FIGURE B-6.4: MASS SPECTRUM OF NG

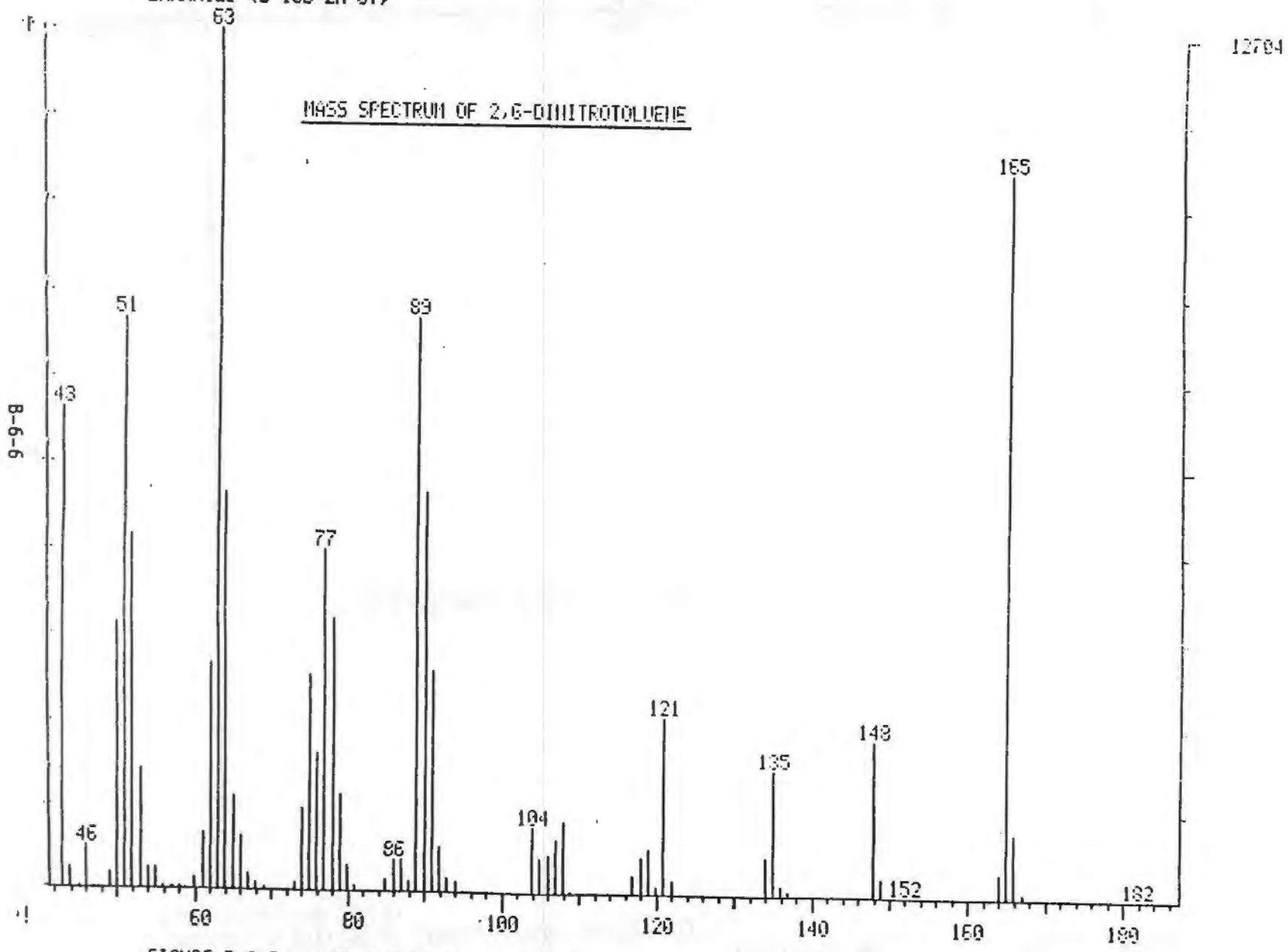


FIGURE B-6.5: MASS SPECTRUM OF 2,6-DNT

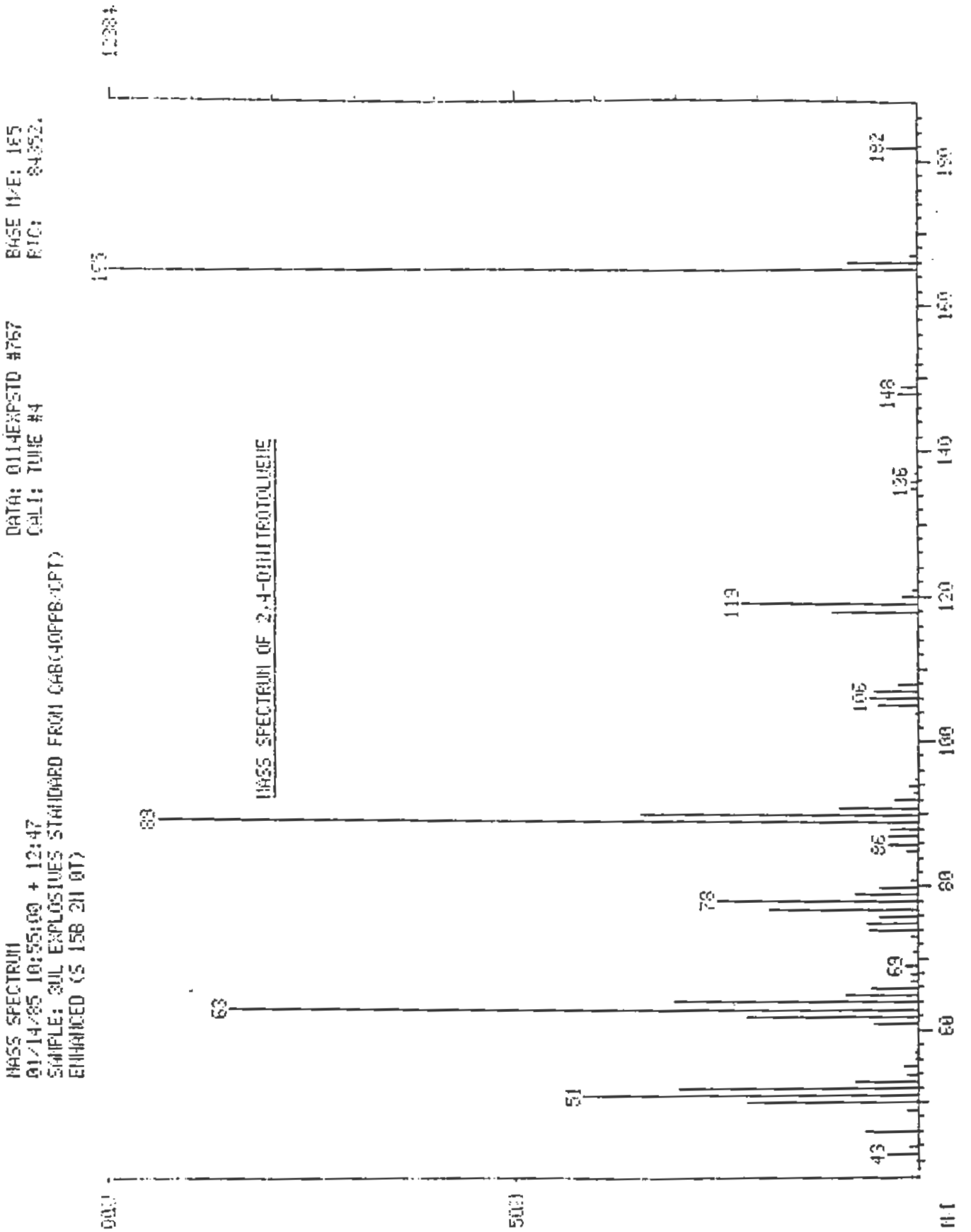


FIGURE B-6.6: MASS SPECTRUM OF 2,4-DNT

SEARCH AND QUANTITATION MASS CONDITIONS

Compound	Scan Range	Search Masses	Quantitation Mass
2,6-DNT	690-740	63;89;165	165
2,4-DNT	740-790	63;89;165	165
3,4-DNT	790-840	63;89;182	182
NG	600-650	46;76	46
RDX	980-1030	46;75;120	46
2,4,6-TNT	870-920	63;89;210	210
2,4,5-TNT	930-980	63;118;210	210
Anthracene-d10 (internal standard)	930-970	188	188

APPENDIX C

DOCUMENTATION REQUIREMENTS

1. Introduction. This appendix outlines the structure of the project file system and the type of information that will be contained in this system. For each energetic compound emission assessment, a project file will be constructed according to the guidance in this appendix and stored. The purpose of establishing these documentation requirements is to ensure that all data is accounted for once the project is completed and that the data/results may be re-analyzed in the future if the need should arise.

2. General. Before issuance of the final report on the assessment, the project engineer will assemble and cross-check all relevant data pertaining to the project to ensure the information is complete and that a hard copy is on file with the APED. For these projects, the APED will maintain the complete project file in hard copy form for a period of 5 years after the final report has been issued. If the sampling/analysis has been performed for a RCRA trial burn test, these hard copy files will be converted to microfiche by Central Files and the microfiche stored indefinitely.

3. Project File System Structure. The file system is organized into a master section containing project correspondence and four major data sections covering stack sampling data, process operational data, chain of custody documentation, and analytical data. Each file folder will contain the following information on the label: project number, installation, unit description (e.g., deactivation furnace), and file folder number. The file folder number will be used to provide a cross reference for the location of specific data within the project file. Each major section will not necessarily be restricted to a single folder. The number of folders will be dependent upon the quantity of data associated with the project.

3.1. Master Section. The master section will contain all general correspondence pertaining to the project along with a copy of the protocol/trial burn test plan and the final report. Any changes made to the protocol should be documented as a memorandum and contained in this section, along with the approval letter from the regulator for that change. The cross reference list for locating specific data within the file system should be located as the first item in the section.

3.2. Stack Sampling Data Section. This section will contain all equipment calibration data with a summary sheet containing the results from the pre- and post-test calibration on those items of equipment utilized. Additionally, all field data sheets for the meter box and sampling box operation will be contained in this section. The averages of this data which are used for the isokinetic calculations will also be summarized. The laboratory data sheets for the calibration of the Orsat analyzer, for the Orsat analysis of the stack gases, and the particulate determination will also be contained in this section. The final item in this section will be a complete set of calculations for all emission parameters (e.g., DRE, particulate).

3.3. Process Operational Data Section. As in the other sections of the project file system, the operational data in terms of process operation and continuous emission analyzer will be summarized at the beginning of the section. All raw operational data, to include tabulations of raw data, strip chart recordings or any other hard copy data, will be contained in this section. All information on settings for continuous emission analyzers should be summarized along with the daily pre- and post-test calibration of the analyzers. Raw data (strip charts) from these analyzers should also be contained in this section.

3.4. Chain of Custody Documentation Section. All forms and sheets which illustrate the movement of samples in the field, between the field and the laboratory, and within the laboratory should be maintained. The utilization of these forms is discussed in Section 7 of the QA/QC plan. Mail receipts, when appropriate, should also be kept.

3.5. Analytical Data Section. The analytical data section will be organized by the project chemist(s) in accordance with the outline in the following table. It is imperative that the analytical data package be of sufficient detail to allow technical review by a regulatory agency once the project has been completed. The listing below may not include all information areas that would be essential for assessment of the performance of the analytical method, and the analyst may enhance the data package as he deems necessary to provide all essential data/information for the assessment of the performance.

3.5.1. Case Narrative. This subsection should contain a description of the analytical method utilized, and those departures from the original protocol must be annotated in detail. If the protocol gave an option for a particular method, the options utilized should be discussed. This section should also contain a cross-referenced list of sample numbers as well as a chronological listing of sample analysis. Finally, the results of the analysis on the field samples should be tabularized. These results should be the final analytical results and should represent the quantitation of the target compounds in the samples (i.e., adjustments because of the serial dilution of the resin extracts should have been made).

3.5.2. Quality Control Summary. This subsection should give the results for the percent recovery of the surrogate compounds in all spiked samples, for the replicate analysis of samples, for the analysis of standards and for the analysis of method blanks. Additional data in this section should include summaries of calibration curve construction (concentration, chromatographic area/peak height, slope, intercept, and correlation coefficient) as well as summaries of any periodic checks of the calibration curve for each compound. Results from the tuning of the GC/MS instrument should appear in this section.

3.5.3. Raw Sample Data. The chromatograms (photocopies) from the analysis of all field samples should be filed in hard copy form in this subsection as well as the quantitation reports (integrator print-outs) for those samples. Copies of the calculations performed if there was a detection of the target compounds should be included as well as copies of laboratory notebook pages pertaining to these samples. If the data contained in the laboratory pages is located elsewhere in the data file system (in hard copy), it is not necessary to file the copy of that notebook page.

3.5.4. Raw Standards Data. The preparation of all stock and working calibration standards as well as the preparation of all surrogate solutions should be recorded in the laboratory notebook. Copies of the pertinent laboratory pages should be included in the project file. As in the case of the field sample analysis, hard copies of the chromatograms used for the construction of the calibration curve for the target compounds and for the determination of retention time windows should also be filed in this section with their respective quantitation reports (integrator print-outs). Copies of the calculations performed should also be included. Chromatograms and quantitation reports for calibration curve check analysis will be placed in this subsection.

3.5.5. Raw Quality Control Data. As in the previous two subsections, all chromatograms and quantitation reports (integrator print-outs) associated with the analysis of quality control samples, method blanks, insert samples, and replicate samples will be filed. In addition, any applicable QC charts, generated during the analysis of the samples, for the analysts associated with the project will also be contained in this section. Finally, raw data from the tuning of the GC/MS instrument will be provided.

OUTLINE OF CONTENTS FOR ANALYTICAL DATA SECTION

1. Case Narrative

   a. Methodology Summary
   b. Cross-referenced List of Sample Numbers
   c. Results of Analysis

2. Quality Control Summary

   a. Summary of Surrogate Recovery
   b. Results from the Replicate Analysis of Samples
   c. Calibration Curve Summaries
   d. Calibration Curve Check Summaries
   e. Retention Time Window Summaries
   f. GC/MS System Tuning Reports

3. Raw Sample Data

   a. Chromatograms and Integrator Print-outs
   b. Calculation Sheets
   c. Laboratory Notebook Pages (Xerox copies)

4. Raw Standards Data

   a. Calibration Curve/Retention Time Window Raw Data
      (1) Chromatograms and Integrator Print-outs
      (2) Calibration Sheets
   b. Calibration Curve Check Raw Data - Chromatograms and Integrator Print-outs
   c. Laboratory Notebook Pages (Xerox copies)

5. Raw Quality Control Data

   a. Analysis of Quality Control Samples
   b. Method Blank Analysis
   c. Replicate Run Raw Data
   d. Control Charts on Analysts
   e. MS Tuning Data

APPENDIX D

CALCULATIONS

1. Analytical Results. Analytical results are obtained for each sample based on the applicable calibration plot in the GC instrument integrator. (Detector response in either area count or peak height is plotted against the concentration of the target analyte.) The volume of the sample will have been recorded by the sample coordinator and these values supplied to the project chemist along with the Request for Chemical Analysis. The manipulation of analytical data for each section of the sampling train is discussed below. The discussion assumes one POHC compound and one surrogate compound. If the sampling train is being utilized for multiple POHC's and surrogates, then this calculation should be performed for each target compound.

a. Filter/Probe Rinses. This section of the sampling train generates one organic sample for analysis and for determining the compound emission rate. Therefore, the multiplication of the compound concentration in the sample and the sample volume yields the total quantity of the target compound captured in this section of the sampling train. In the event that the target compound is not detected, then the detection limit concentration will be used in the calculation instead of the measured concentration. The acetone rinse of this section of the train is not figured into the target compound capture, but the analytical result will be used to assess the percentage of compound not removed by the extraction solvent rinse as a QA/QC check.

b. Impinger Water/Rinses. This section of the sampling train generates three samples in the extraction solvent for analysis and, therefore, increases the complexity of the calculation. As in the case of the front half sample analysis, the quantity of POHC and surrogate in any given sample from the sampling train is obtained by multiplying the sample volume and the concentration of the compound. In addition to determining the quantity/recovery of surrogate and the quantity of the target compound, the detection limit quantity, which is equal to the product of the sample detection limit and the sample volume, is determined. However, since the results for the capture of the POHC by this section of the sampling train is dependent upon the analytical data from subsequent extracts of the same media, the calculation methodology is therefore more complex. Figure D-1 illustrates the logic diagram for obtaining the total quantity of POHC captured in the impinger section of the train. The acetone rinse of this section of the sampling train is not figured into the calculation of the quantity captured but is used in assessing the effectiveness of the water rinses of this glassware.

c. Resin Section. The resin section also generates multiple samples (12 samples per resin package). Subsequent extractions of the same resin section represent a serial dilution of the first extract of that particular

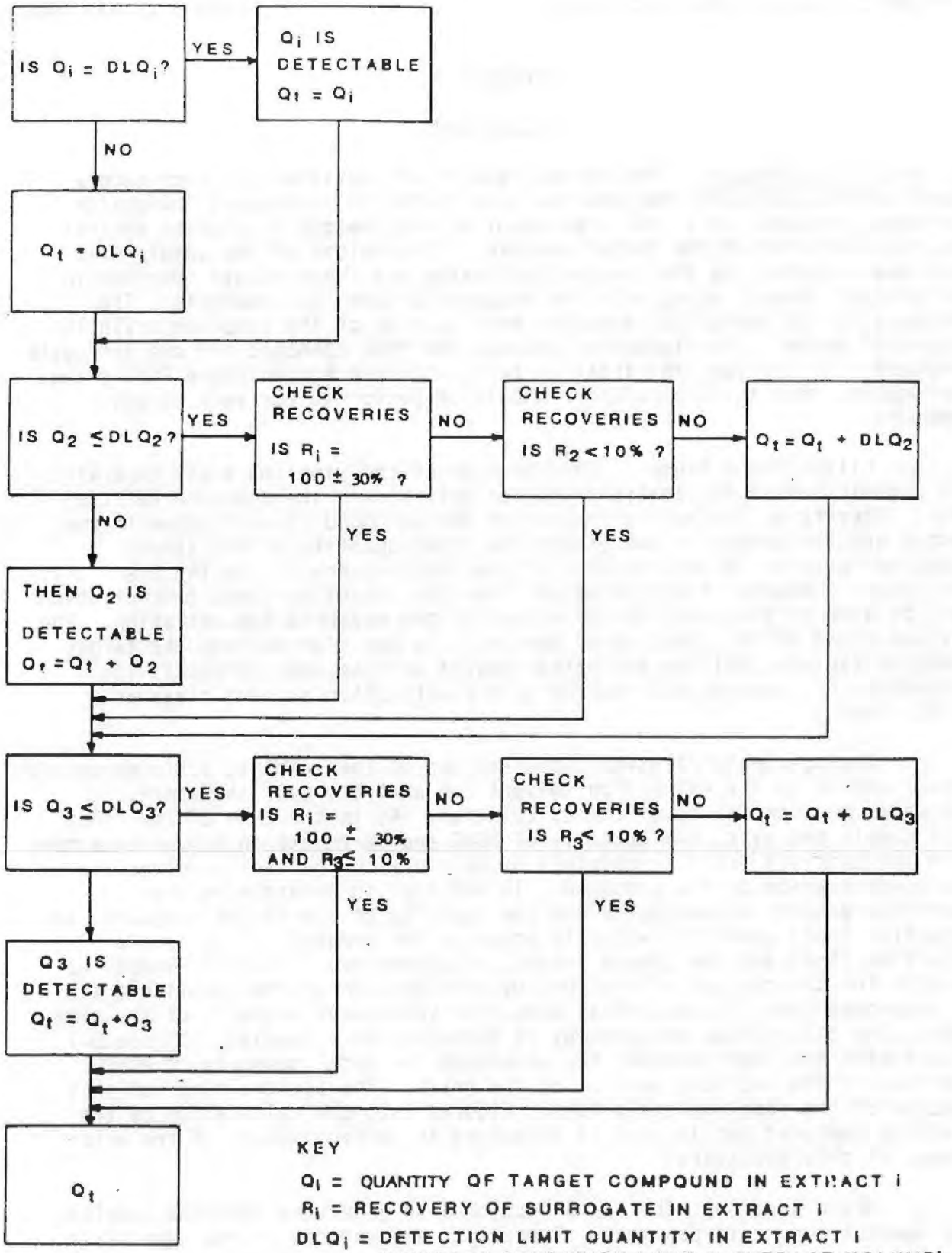


FIGURE D-1. CALCULATION LOGIC FLOWCHART

resin section. The quantity of the target compound in the samples generated from the extractions of a single resin section are calculated by the following equations:

Quantity (1st Extract) = Concentration x Sample Volume [30 mL]

Quantity (2nd Extract) = (Conc. x Sample Volume [30 mL]) - (66.67% of quantity in 1st Extract)

Quantity (3rd Extract) = (Conc. x Sample Volume [30 mL]) - (66.67% of (concentration x volume [30 mL] for 2d Extract))

The detection limit quantities of these extracts are calculated by multiplying the detection limit of the extract by the volume of the extract (30 mL), neglecting the dilution effects. Figure D-1 also represents the logic flow diagram for calculating the quantity of target compound captured by a resin section. This is performed for all four resin sections. The extraction solvent rinse of the resin tube is analyzed and the quantity of compound obtained is the product of the extract concentration and the concentration. The results from these calculations are then used to calculate the total quantity of the target compound captured by the resin section of the sampling train. The quantities from all four resin sections along with the quantity in the resin tube rinse are added together to yield the resin section total.

d. Total. The total quantity of an energetic compound captured on the sampling train is the sum of the quantities captured in the filter/probe wash section, the impinger section, and the resin section.

2. Sampling Results. The calculations for the sampling portion are the same as for Reference Method 5 sampling. The isokinetic factor must be calculated and compared with the Method 5 QA/QC sampling objective. The corrected volume sampled and the stack gas volumetric flow rate are required to calculate the stack gas concentration and the mass flow rate of the target compound. In the case of calculating the DRE for a target compound, the formula found in 40 CFR 264 Subpart O is utilized.

APPENDIX E

GLOSSARY OF TERMS

ACCURACY - The closeness of a measured or computed value to its true value.

ANALYSIS OF VARIANCE (ANOVA) - A technique of statistical analysis by which the components of variation for different elements of the data set are separated and estimated.

ANALYTE - Chemical component to be analyzed.

ARITHMETIC MEAN (X) - A measure of central tendency equal to the sum of the observations divided by the number of observations, usually called the mean or average.

CALIBRATION STANDARD - Solutions used in correlating instrument responses to analyte concentrations. See Section 8.3.

CENTRAL TENDENCY - The tendency of values in a set of data to group around a center value. The most common measures of central tendency are: the mean, the mode, and the median.

COEFFICIENT OF VARIATION - A measure of dispersion in which the standard deviation is expressed as a percentage of the arithmetic mean.

CONTROL CHART - A graphic record of values, usually averages or ranges, or both of sets of data, recorded successively with limits denoting out of control conditions. (Also called Shewhart Control Chart)

CONTROL LIMIT - Limits on a control chart establishing statistically, usually at ± 3 standard deviations from the mean. Plotted values falling outside the upper or lower control limits are said to be "out-of-control."

CONTROL SAMPLES - Samples that are introduced into a train of actual samples as a monitor on the performance of the analytical system.

DETECTION LIMIT - The lowest concentration that can be differentiated from zero, with a 90-percent confidence limit.

DUPLICATE SAMPLE - Identical samples that are carried through the entire analytical method.

F TEST - A statistical test based on the F sampling distribution. Used for testing for differences between variances.

FIELD BLANK - Samples prepared in standard matrices to which no analyte of interest has been added. Introduced into the sample train in the field. Used to detect contamination introduced in the field and laboratory.

MEASUREMENT STANDARDS - Standards traceable to primary standards maintained in the laboratory which are traceable to the National Bureau of Standards.

MEDIAN - A measure of central tendency. When the data are arranged in order of magnitude the median is the middle value, corresponding to the 50th percentile.

METHOD BLANK - Samples prepared in standard matrices to which no analyte of interest has been added. Used to detect contamination introduced in the laboratory.

NEGATIVE INTERFERENCES - A response, or lack thereof, indicating a less amount of analyte than actually present.

NORMAL CURVE - A continuous distribution, symmetrical and bell shaped, widely used in statistics. Also known as the Gaussian distribution.

PRECISION - The variability of repeated measurements taken under equivalent conditions. The relative standard deviation is the measure most often used to express precision.

POPULATION - The universe or collection from which a set of sample values is drawn.

POSITIVE INTERFERENCE - A response indicating the presence of an analyte in greater amounts than actually present.

RANGE - In a set of data, the difference between the largest and the smallest values.

REPLICATION - Independent testing or analysis of the same samples by same technicians, or by different technicians.

SAMPLE - An environmental specimen collected and submitted to the laboratory for analysis.

SAMPLE SIZE - 1. Number of units in a sample, often prescribed by a sampling plan.

2. The physical size or volume of a sample submitted, i.e., mL of water, or 3 ft of air sampled, or kg of soil, etc.

SPIKED SAMPLE - A sample into which a known quantity of analyte has been introduced.

STANDARD SAMPLE - Samples prepared in standard matrices as defined in Appendix B.

SURROGATE STANDARD - A compound or species used in lieu of actual analytes of interest to monitor recoveries from various matrices and to provide reference points for quantitation. Surrogate standards are spiked into original matrices.

t TEST - A test for the significance of differences in sets of data based on the "t" distribution; a means of estimating the characteristics of a population from a sample with assurance of accuracy at a chosen level.

VARIANCE - The square of the standard deviation.

APPENDIX F

LIST OF ACRONYMS

ACS	American Chemical Society
ANOVA	Analysis of Variance
APED	Air Pollution Engineering Division
ASTM	American Society for Testing and Materials
CFR	Code of Federal Regulations
CV	Coefficient of Variation
DNT	Dinitrotoluene
DRE	Destruction and Removal Efficiency
ECD	Electron Capture Detector
EGDN	Ethylene Glycol Dinitrate
EPA	Environmental Protection Agency
GC	Gas Chromatograph
GC/MS	Gas Chromatography/Mass Spectrometry
HMX	Cyclotetramethylene Tetranitramine
NBS	National Bureau of Standards
NG	Nitroglycerin
NPD	Nitrogen - Phosphorus Detector
POHC	Principal Organic Hazardous Constituent
QA	Quality Assurance
QC	Quality Control
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
RDX	Cyclotrimethylene Trinitramine
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SARM	Standard Analytical Reference Material
SOP	Standing Operating Procedure
SRM	Standard Reference Material
TNT	Trinitrotoluene
USAEHA	U.S. Army Environmental Hygiene Agency

APPENDIX G

REFERENCES

1. Title 40, Code of Federal Regulations (CFR), 1987 rev, Part 60, Standards of Performance for New Stationary Sources, Appendix A, Reference Methods.

2. Title 40, CFR, 1987 rev, Part 261, Identification and Listing of Hazardous Wastes.

3. Title 40, CFR, 1987 rev, Part 264, Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities.

4. EPA Publication Number 600/4-77-027b, Quality Assurance Handbook For Air Pollution Measurement Systems, Volume II, Stationary Source Specific Methods, as updated, 1987.

5. EPA Publication Number 600/8-85-003, February 1985, Modified Method 5 Train and Source Assessment Sampling System Operator's Manual.

6. EPA, Publication Number SW-846, July 1982, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

7. EPA Publication Number 600/4-82-057, July 1982, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewaters.

8. EPA Guidance Document Number QAMS-006/80, December 1980, Interim Guidelines and Specifications for Quality Assurance Project PLans.

9. EPA Report for EPA Contract Number 68-02-3111, February 1983, Sampling and Analysis Methods for Hazardous Waste Combustion, 1st edition, Arthur D. Little, Inc.

10. EPA Manual Number APTD-0576, March 1982, Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment.

11. EPA Manual Number APTD-0581, April 1971, Construction Details of Isokinetic Source Sampling Equipment.

12. TM 9-1300-214, 20 September 1984, Military Explosives.

13. Memorandum for Record, USAEHA, HSHB-ME-AS, 13 August 1986, subject: Meeting Regarding Sampling and Analysis Methods for RCRA Principal Hazardous Organic Constituents.

14. Memorandum for Record, USAEHA, HSHB-ME-AS, 15 June 1987, subject: Determination of Temperature Profile in USAEHA Stack Sampling Train for High Temperature Stacks.

ATTACHMENT C

Calibration Procedures

*TB 9-6685-319-35

SUPERSEDED COPY DATED 1 JULY 1973

DEPARTMENT OF THE ARMY TECHNICAL BULLETIN

CALIBRATION PROCEDURE FOR DIAL INDICATING PRESSURE GAGES (GENERAL)

Headquarters, Department of the Army, Washington, DC
27 June 1988

Approved for public release; distribution is unlimited.

REPORTING OF ERRORS

You can help improve this publication by calling attention to errors and by recommending improvements and stating your reasons for the recommendations. Your letter or DA Form 2028, Recommended Changes to Publications, should be mailed directly to Commander, U.S. Army TMDE Support Group, ATTN: AMXTM-LPP, Redstone Arsenal, AL 35898-5400. A reply will be furnished directly to you.

*This bulletin supersedes TB 9-6685-319-50, 1 July 1973, including all changes.

SECTION I

IDENTIFICATION AND DESCRIPTION

1. Test Instrument Identification. This bulletin provides instructions for the calibration of Dial Indicating Pressure Gages (General). Various manufacturers' manuals were used as the prime data sources in compiling these instructions. The equipment being calibrated will be referred to as the TI (test instrument) throughout this bulletin.

a. Model Variations. Variations among models are indicated in the text.

b. Time and Technique. The time required for this calibration is approximately 2 hours, using the physical technique.

2. Forms, Records, and Reports

a. Forms, records, and reports required for calibration personnel at all levels are prescribed by TB 750-25.

b. Adjustments to be reported are designated (R) at the end of the sentence in which they appear. When adjustments are in tables, the (R) follows the designated adjustment. Report only those adjustments made and designated with (R).

3. Calibration Description. TI parameters and performance specifications which pertain to this calibration are listed in table 1.

Table 1. Calibration Description

Test instrument parameters	Performance specifications
Range and accuracy[1]	Performance at cardinal points in accordance with specifications for gage being tested.

[1] In those cases where accuracy of TI cannot be determined, TI will be certified to one graduation of TI scale.

SECTION II

EQUIPMENT REQUIREMENTS

4. Equipment Required. Table 2 identifies the specific equipment to be used in this calibration procedure. This equipment is issued with Secondary Transfer Calibration Standards Set AN/GSM-286. Alternate items may be used by the calibrating activity when the equipment listed in table 2 is not available. The items selected must be verified to perform satisfactorily prior to use and must bear evidence of current calibration. The equipment must meet or exceed the minimum use specifications listed in table 2. The accuracies listed in table 2 provide a four-to-one ratio between the standard and TI. Where the four-to-one ratio cannot be met, the actual accuracy of the equipment selected is shown in parenthesis.

5. Accessories Required. The accessories listed in table 3 are issued as indicated in paragraph 4 above and are used in this calibration procedure. When necessary, these items may be substituted by equivalent items, unless specifically prohibited.

Table 2. Minimum Specifications of Equipment Required

Item	Common name	Minimum use specifications	Manufacturer and model (part number)
A1	PNEUMATIC PRESSURE STANDARD	Range: 0 to 200 psi Accuracy:[1] ±0.025% (±0.125% of FS)	Cybersystems, Inc., Model ZA00225A1 (MIS-30859)
A2	PRESSURE GAGE TESTER	Range: 0 to 10,000 psi Accuracy:[2] ±0.025% (±0.15% of reading)	Mansfield and Green, Model 10-10525 (10-10525)

[1]Equipment limitation: ±0.05% of FS.
[2]Equipment limitation: ±0.015% of reading.

Table 3. Accessories Required

Item	Common name	Description (part number)
B1	ADAPTER[1]	Male 1/4-in., SP to male 7/16-20 UNF, 37° flare (part of MIS-26326)
B2	ADAPTER	Male 3/8-18 NPT to female 1/4-18 NPT (part of 8598963)
B3	ADAPTER	Male 7/8-14 UNF w/"o" ring to female 3/8-18 NPT (part of 8598963)
B4	ADAPTER	Male 7/16-20 UNF, 37° flare to female 1/8-18 NPT (part of 7913310)
B5	ADAPTER	Male 1/2-20 UNF to female 7/8-14 UNF (part of 8598963)
B6	CONNECTOR	Stainless steel, female 1/4-18 NPT to male 7/16-20 UNF, 37° flare (part of 7913310)
B7	DISTILLED WATER	Additional equipment required
B8	FLUID SEPARATOR	MIS-26326
B9	HOSE	3-ft, 3000 psi operating pressure, female 7/16-20 UNF, 37° flare angle fittings (7913310)
B10	HOSE	5-ft, 5000 psi operating pressure (part of 7913310)
B11	HOSE	Male 1/4-18 NPT ends (part of 8598963)
B12	NITROGEN TANK	Water pumper nitrogen (7910373)

See footnote at end of table.

Table 3. Accessories Required - Continued

Item	Common name	Description (part number)
B13	PNEUMATIC PRESSURE CONTROLLER	MIS-10324
B14	REGULATOR	MIS-10325 Type II
B15	SQUEEZE BOTTLE	(Part of MIS-26326)
B16	TEE	Stainless steel, swivel nut (8491696) (part of 7913310)
B17	TUBE ASSEMBLY	1/4-in. AN back-to-back (female 7/16-20 UNF, 37° flare both ends (7913309) (part of 7913310)

[1]Two required.

SECTION III

CALIBRATION PROCESS FOR HYDRAULIC GAGES

6. Preliminary Instructions

a. The instructions outlined in paragraphs 6 and 7 are preparatory to the calibration process. Personnel should become familiar with the entire bulletin before beginning the calibration.

b. Items of equipment used in this procedure are referenced within the text by common name and item identification number as listed in tables 2 and 3. For the identification of equipment referenced by item numbers prefixed with A, see table 2, and for prefix B, see table 3.

c. Unless otherwise specified, verify the result of each test and, whenever the test requirement is not met, take corrective action before continuing with the calibration. Adjustments required to calibrate the TI are included in this procedure. Additional maintenance information is contained in the manufacturer's manual for this TI.

d. Unless otherwise specified all controls and controls settings refer to the TI.

7. Equipment Setup

a. Remove pressure gage tester (A2) from carrying case.

b. Remove filler plug and fill pressure gage tester with hydraulic oil (MIL-L-7870A).

c. Reinstall filler plug and secure pressure gage test to an adequate workbench. Level pressure gage tester.

d. Visually inspect TI for signs of damage or deterioration.

e. Throughly clean bourdon tube or diaphragm of TI. First, rotate TI and allow any liquid which may be in the bourdon tube or diaphragm area to flow out. Using an eye dropper or small glass tube, fill bourdon tube or diaphragm area with toluene, methyl alcohol, or freon TF solution. Allow solution to remain in the gage for 5 minutes. Drain gage and dry for at least 10 minutes.

f. If required, zero-adjust dial indicator of TI by removing retaining ring and coverglass and adjusting the calibration screw.

8. 0 to 10,000 Psi Hydraulic Gages (0.1 to 20 Percent Accuracy)

a. Performance Check

(1) Connect equipment as shown in figure 1.

(2) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(3) Place enough deadweights (supplied with pressure gage tester (A2)) on applicable low or high pressure piston to obtain a pressure equal to cardinal point nearest 10 percent of TI scale.

CAUTION

To avoid scoring the piston guide and damage to the deadweight cylinder, constantly rotate weights and piston when inserting or removing piston or when applying pressure.

(4) Using hand pump, apply pressure to deadweight cylinder until (low or high pressure) piston is approximately 9/16-inch above deadweight cylinder.

(5) Visually inspect equipment connection for leakage. If leakage appears, release pressure and tighten or seal connections as required.

CAUTION

Do not remove weights installed in (3) above from low or high pressure piston during remainder of this procedure.

(6) Place enough deadweights on applicable low or high pressure piston to obtain the 90 percent cardinal point of TI. Refer to table 4 for conversion table.

(7) Repeat (4) above.

(8) If TI does not indicate within limits calculated in (2) above perform b below.

(9) Repeat (2) and (6) through (8) above, using enough weights to obtain cardinal points nearest to 80, 60, 40, 20, and 10 percent of TI scale.

(10) Throughly clean TI in accordance with paragraph 7 above.

b. Adjustments (Typical, fig. 2).

NOTE

The following adjustment procedure applies specifically to solfront gages, but may be used as a guide for other gages. Manufacturers' instructions should be used if available.

(1) Deviations either plus or minus by a constant value are corrected by repositioning the pointer. Remove bezel ring, loosen locking screw (marked L) 1/4 turn, and adjust screw marked "A". Tighten locking screw and install bezel ring.

(2) Deviations varying linearly over range of TI are corrected by repositioning link screw. Remove protective back or case from mechanism, loosen link screw and move in slot of sector as required. Tighten link and install protective back of case.

(3) Deviations varying non-linearly over range of TI are corrected by repositioning arc-loc movement. Remove protective back or case, loosen the three locking screws, and rotate arc-loc movement as required. If indications are first increasingly plus and then decreasingly plus, rotate arc-loc movement in direction "A". If indictions are first increasingly minus and then decreasingly minus, rotate arc-loc movement in direction "B". Tighten locking screws.

(4) Repeat (1), (2) or (3) as necessary to obtain indications within tolerance.

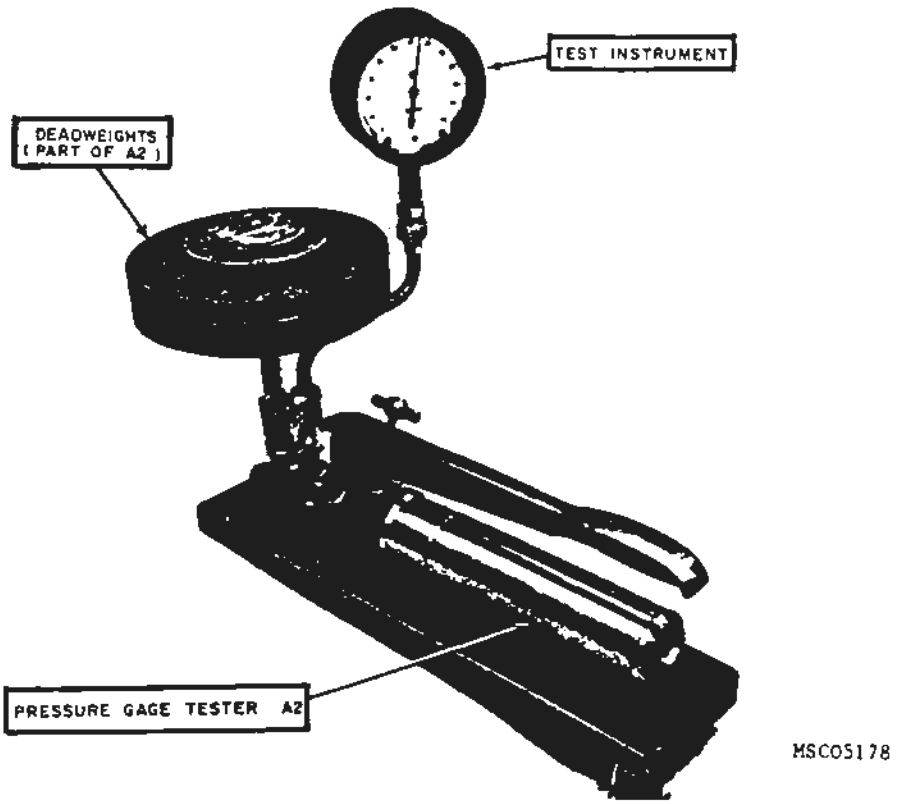


Figure 1. 1.0 to 10,0000 psi hydraulic gages (0.1 to 1.0 percent accuracy) - equipment setup.

Table 4. Conversion Table

Number of deadweights supplied with pressure gage tester (ea)	Value of each weight (lbs)	1 weight equal the equivalent pressure (psi)[1]	
		Low pressure piston	High pressure piston
18	10	100	500
1	9½	95	475
4	2	20	100
4	½	5	25

[1]Pressure generated by deadweight testers are affected by acceleration of gravity. Mansfield and Green, Model 10-1025 deadweight tester generates true pressures at locations where the acceleration of gravity is 980.217 cm/s^2. The acceleration of gravity must be considered when using the deadweight tester to calibrate TI's with an accuracy of ±0.35% of FS or better. To correct for the effect of gravity, multiply total pressure of weight combination by local gravity (units - cm/s^2) and divide by 980.217 cm/s^2.

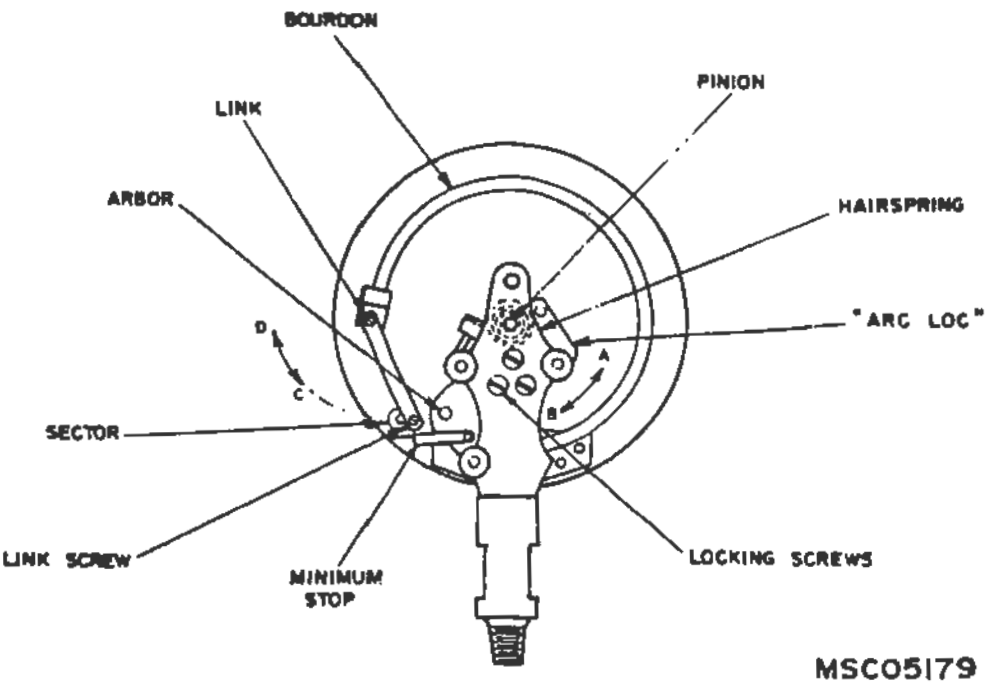


Figure 2. Typical pressure gage.

9. 0 to 5000 Psi Panel Mounted Hydraulic Gages (1.0 to 20 Percent Accuracy)

a. Performance Check

NOTE

Convert accuracies of TI and standard into pressure units (psi). If four-to-one accuracy ratio (in pressure units) cannot be attained, use procedure in paragraph 10 below.

(1) Select a standard pressure gage from pressure gage tester (A2) which will cover the same range as the TI.

(2) Connect appropriate standard pressure gage and TI in the system in a way that will insure that the same pressure will be applied to both gages.

NOTE

The standard pressure gage should be located so that reference plane of each instrument is the same height to eliminate an error caused by hydraulic head pressure.

(3) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(4) Using system pressure source, apply pressure to obtain an indication on standard pressure gage equal to 10 percent of TI scale.

NOTE

It is preferred that the system pressure source be utilized when calibrating panel mounted pressure gages, since TI and system pressure source are connected. If system pressure source is not available, utilize procedure in paragraph 8 above.

(5) If TI indication does not indicate within limits calculated in (3) above, perform b below.

(6) Repeat (1) through (5) above, using cardinal points nearest to 80, 60, 40 and 20 percent of TI scale.

b. Adjustments. Perform 8b above.

10. 0 to 10,000 Psi Panel Mounted Hydraulic Gages

a. Performance Check

WARNING

To prevent injury to personnel and/or damage to equipment, make certain that all components are within range of unit to be calibrated and all connections are securely sealed prior to applying pressure to TI.

(1) Connect equipment as listed in (a) through (d) below:

(a) Cap one vertical port on deadweight tester (part of A2).

(b) Connect applicable low or high pressure cylinder to other vertical port on deadweight tester and install appropriate piston.

(c) Connect horizontal outlet port of deadweight tester to input port of TI.

(d) Cap all other system outlet ports and close system shut-off valve.

NOTE

The deadweight tester should be located so that reference planes of TI and deadweight tester are same height to eliminate an error caused by hydraulic head pressure.

(2) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(3) Place enough deadweights (supplied with pressure gage tester) on the applicable low or high pressure piston to obtain an indication on standard pressure gage equal to 10 percent of TI scale.

(4) Using hand pump, apply pressure to system until (low or high pressure) piston is approximately 9/16-inch above deadweight cylinder.

(5) If TI indication does not indicate within limits calculated in (2) above, perform b below.

(6) Repeat (2) through (5) above, using cardinal points nearest to 80, 60, 40, and 20 percent of TI scale.

b. Adjustments. Perform 8b above.

11. Final Procedure

a. Deenergize and disconnect all equipment.

b. Annotate and affix DA Label/Form in accordance with TB 750-25.

SECTION IV

CALIBRATION PROCESS FOR

PNEUMATIC GAGES

12. Preliminary Instructions

a. The instructions outlined in paragraphs 12 and 13 are preparatory to the calibration process. Personnel should become familiar with the entire bulletin before beginning the calibration.

b. Items of equipment used in this procedure are referenced within the text by common name and item identification number as listed in tables 2 and 3. For the identification of equipment referenced by item numbers prefixed with A, see table 2, and for prefix B, see table 3.

c. Unless otherwise specified, verify the result of each test and, whenever the test requirement is not met, take corrective action before continuing with the calibration. Adjustments required to calibrate the TI are included in this procedure. Additional maintenance information is contained in the manufacturer's manual for this TI.

d. Unless otherwise specified, all controls and control settings refer to the TI.

13. Equipment Setup

a. Visually inspect TI for signs of damage or deterioration.

b. Throughly clean bourdon tube or diaphragm of TI. First, rotate TI and allow any liquid which may be in the bourdon tube or diaphragm area to flow out. Using an eye dropper or small glass tube, fill bourdon tube or diaphragm area with toluene, methyl alcohol, or freon TF solution. Allow solution to remain in the gage for 5 minutes. Drain gage and dry for at least 10 minutes.

c. If required, zero-adjust dial indicator of TI by removing retaining ring and coverglass and adjusting the calibration screw.

14. 0 to 235 Psi Pneumatic Gages (0.1 to 1.0 Percent Accuracy)

a. Performance Check

(1) Connect equipment as shown in figure 3, connection A.

NOTE
The maximum input to the 0 to 20 psia port should not exceed 6 psi. The maximum input to 0 to 250 psia port should not exceed 235 psi.

WARNING
To prevent injury to personnel and/or damage to equipment, make certain that all components are within range of unit to be calibrated and all connections are securely sealed prior to applying pressure to TI. Never attempt to tighten connection with pressure applied. Insure that TI is clean and free of oil or grease.

(2) Position controls on pneumatic pressure standard (A1) as listed in (a) through (d) below:

(a) **UNITS DISPLAYED** switch to **PSIA.**

(b) **RANGE** pushbutton to **0-250.**

(c) **SENSITIVITY** pushbutton to **HIGH.**

(d) **SOURCE** pushbutton to **INT.**

(3) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(4) Open exhaust, metering and shutoff valves on pneumatic pressure controller (B13).

(5) Press **RESET** and **ZERO** pushbutton on pneumatic pressure standard.

(6) Close exhaust, metering and shutoff valves on pneumatic pressure standard.

(7) Turn regulator control fully ccw.

(8) Open nitrogen tank (B12) valve and adjust regulator until outlet gage of regulator indicates maximum pressure of TI.

(9) Open inlet valve on pneumatic pressure controller.

(10) Slowly open shut off valve on pneumatic pressure controller.

(11) Increase pneumatic pressure, using metering valve on pneumatic pressure controller until TI reaches full scale.

(12) If TI indication does not indicate within limits calculated in (3) above, perform b below.

(13) Close inlet valve and slowly open exhaust valve.

(14) Release pressure by opening metering valve.

NOTE
Pressure changes can be made by proper use of inlet, exhaust, metering, and shut off valves.

(15) Repeat (2) and (6) through (14) above at cardinal points nearest to 80, 60, 40, and 20 percent of TI scale.

b. Adjustments. Perform 8b above.

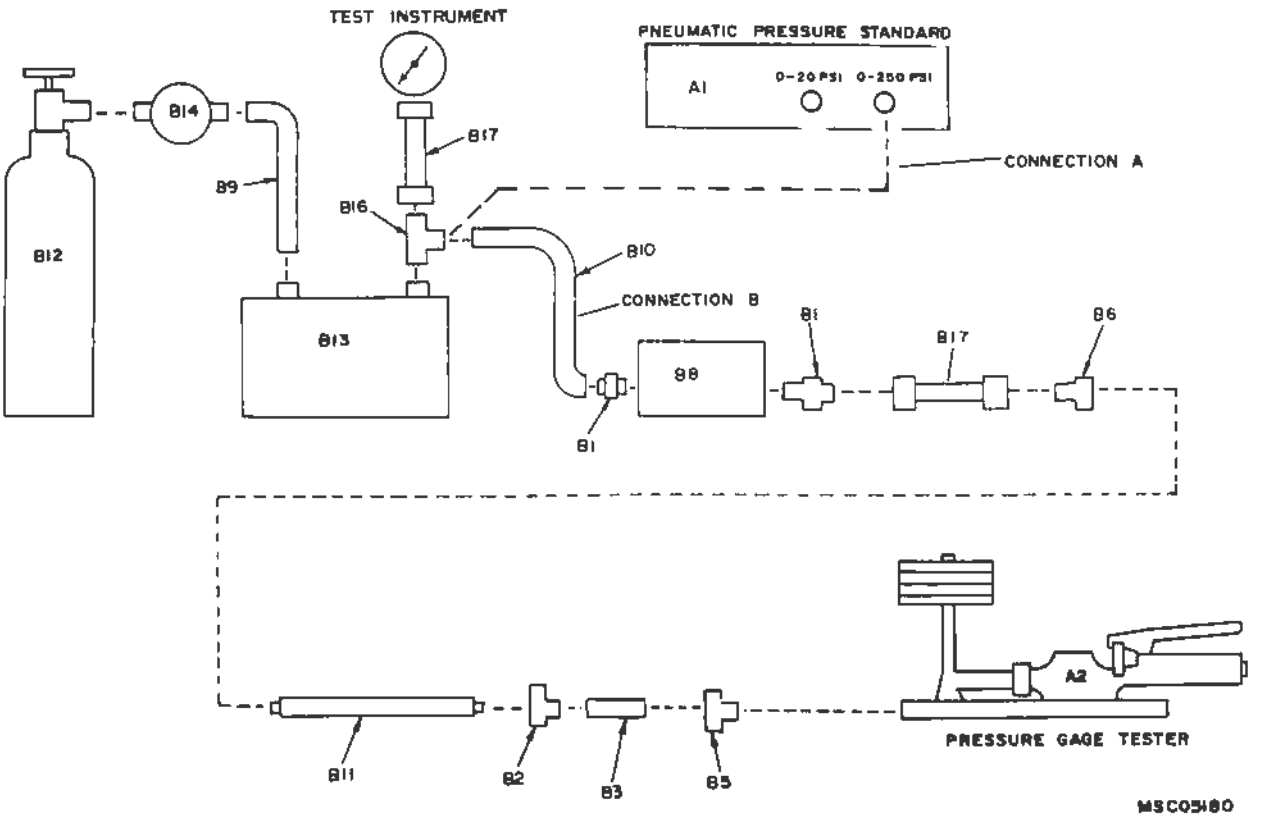


Figure 3. 0 to 1000 psi pneumatic gages (0.1 to 1.0 percent accuracy) - equipment setup.

15. 235 to 1000 Psi Pneumatic Gages (0.1 to 1.0 Percent Accuracy)

WARNING

To prevent injury to personnel or damage to equipment, make certain that all components used are clean and have not been contaminated with oil or grease.

a. Performance Check

(1) Connect equipment as shown in figure 3, connection B.

(2) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(3) Open exhaust, metering and shutoff valves on pneumatic pressure controller (B13).

(4) Close reservoir valve on deadweight tester, and inlet valves on pneumatic pressure controller.

(5) Loosen fill and vent plug on pressure gage tester.

(6) Operate hand pump until weight table rises (tare weight).

(7) Open reservoir valve, allowing weight table to fall to bottom stop; then wait about 10 seconds before closing reservoir valve.

(8) Close shutoff, metering, and exhaust valves on pneumatic pressure controller.

WARNING

To avoid scoring the piston guide and damage to the deadweight cylinder, constantly rotate weights and piston when inserting or removing piston or when applying pressure.

(9) Place enough deadweights (supplied with pressure gage tester (A2)) on applicable low or high pressure piston to obtain a pressure equal to cardinal point nearest to 10 percent of TI scale.

(10) Turn regulator (B14) fully ccw.

(11) Open nitrogen tank (B12) valve and adjust regulator until outlet gage of regulator indicates maximum pressure of TI.

(12) Insure that metering and exhaust valves on pneumatic pressure controller are closed.

(13) Open inlet valve on pneumatic pressure controller.

(14) Slowly open shutoff valve on pneumatic pressure controller.

(15) Increase pneumatic pressure, using metering valve on pneumatic pressure controller until weight rises approximately 9/16-inch above the deadweight cylinder.

NOTE

Making all pressure changes (however large or small) with pneumatic pressure controller. Vernier pressure changes can be made using screw-type pump on pneumatic pressure controller. The hydraulic hand pump should not be used after initial diaphragm positioning in (6) and (7) above.

(16) After reading and recording TI indication, a correction must be applied to each indication. The difference between reference plane of fluid separator (B8) and deadweight tester when both are resting on same table is 0.988-inch for high range position and 3.578-inches for low range position. The density of recommended oil (MIL-L-7870A) is 0.8653 gm/cc at 23 gm/cc at 23°C; therefore, 1-inch of this oil produces a pressure of 0.03125 psi. Thus, 0.031 psi (0.0312 psi x 0.98 inch) for high range position and 0.112 psi (0.3125 psi x 3.578 inch) for low range position must be subtracted from each TI indication.

(17) If TI indication after correction does not indicate within limits calculated in (2) above, perform b below.

(18) Close inlet valve and slowly open exhaust valve.

(19) Release pressure by opening metering valve.

(20) Repeat (2) and (12) through (19) above, using enough weights to obtain cardinal points nearest to 80, 60, 40, and 20 percent of TI scale. Pressure changes can be made by proper use of inlet, exhaust, metering, and shutoff valves.

b. Adjustments. Perform 8b above.

16. 1000 to 5000 Psi Pneumatic Gages (0.1 to 1 Percent Accuracy)

WARNING

To prevent injury to personnel or damage to equipment, make certain that all components used are clean and have not been contaminated with oil or grease.

a. Performance Check

(1) Connect equipment as shown in figure 4, except do not connect hose (B10) to fluid separator (B8).

(2) Open vent port on fluid separator.

(3) Operate hand pump until all the air is bled from hydraulic side of fluid separator.

(4) With fluid separator full of oil, install plug-in vent port.

(5) Connect appropriate standard gage to pressure gage tester (A2).

(6) Fill pneumatic side of fluid separator with distilled water (B7), using squeeze bottle (B15).

(7) Fill hose (B10) with distilled water and connect to fluid separator.

NOTE

In filling pneumatic side of fluid separator hose, connection, and TI; it is important that the components be filled completely and kept as full as possible during connection. Position diaphragm to pneumatic side and hydraulic side several times by manipulation of hand pump and reservoir valve on pressure gage tester.

(8) Position fluid separato. diaphragm to oil side by opening reservoir valve on pressure gage tester and holding hose (B10) above fluid separator, creating a head pressure. Refill hose as water falls.

(9) Fill TI with distilled water and connect to hose.

(10) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(11) Place enough deadweights (supplied with pressure gage tester) on applicable low or high pressure piston to obtain a pressure equal to cardinal point nearest to 10 percent of TI scale.

WARNING

To avoid scoring the piston guide and damage to the deadweight cylinder, constantly rotate weights and piston when inserting or removing piston or when applying pressure.

(12) Using hand pump, apply pressure to deadweight cylinder until (low or high pressure) piston is approximately 9/16-inch above deadweight cylinder.

(13) Visually inspect equipment connections for leakage. If leakage appears, release pressure and tighten or seal connections as required.

CAUTION

Do not remove weights installed in (11) above from low or high pressure pistons during remainder of this procedure.

(14) Place enough deadweights on applicable low or high pressure piston to obtain the 90 percent cardinal point of TI. Refer to table 4 for conversion table.

(15) Repeat (12) above.

(16) If TI indication does not indicate within limits calculated in (10 above, perform b below.

(17) Repeat (10) and (14) through (16) above, using enough weights to obtain cardinal points nearest to 80, 60, 40, 20, and 10 percent of TI scale.

b. **Adjustments.** Perform 8b above.

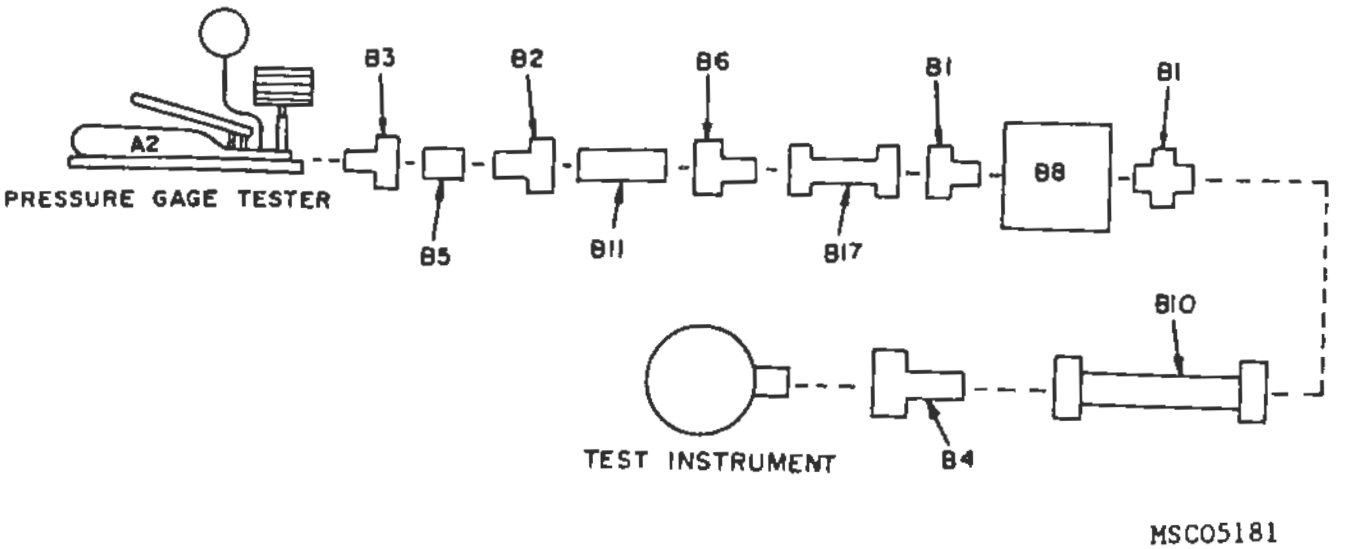


Figure 4. 1000 to 5000 psi pneumatic gages (0.1 to 1 percent accuracy) - equipment setup.

17. Final Procedure

a. Deenergize and disconnect all equipment and reinstall protective cover on TI.

b. Annotate and affix DA Label/Form in accordance with TB 750-25.

By Order of the Secretary of the Army:

CARL E. VUONO
General, United States Army
Chief of Staff

Official:

R. L. DILWORTH
Brigadier General, United States Army
The Adjutant General

Distribution:

To be distributed in accordance with DA Form 12-34C, Block No. 319, requirements for calibration procedures publications.

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TO 33K5-4-267-1

TECHNICAL MANUAL

CALIBRATION PROCEDURE

FOR

THERMOCOUPLE INSTRUMENT CALIBRATOR

AN6520( )

(ANALOGIC)

This publication replaces TO 33K5-4-267-1 dated 13 November 1989.

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(ANALOGIC)

THERMOCOUPLE INSTRUMENT CALIBRATOR

1 CALIBRATION DESCRIPTION:

Table 1.

Test Instrument (TI) Characteristics	Performance Specifications	Test Method
Temperature Iron-Constantan (Type J)	Range: -346 to 2192°F	Compared with a known mV Input.
	Accuracy: ± 1.0°F	
	Range: -210 to 1200°C	
	Accuracy: ± 0.6°C	
Chromel-Alumel (Type K)	Range: -328 to 2501°F	
	Accuracy: ± 1.0°F	
	Range: -200 to 1372°C	
	Accuracy: ± 0.6°C	
Copper-Constantan (Type T)	Range: -337 to 752°F	
	Accuracy: ± 1.0°F	
	Range: -205 to 400°C	
	Accuracy: ± 0.6°C	
Chromel-Constantan (Type E)	Range: -389 to 1832°F	
	Accuracy: ± 1.0°F	
	Range: -422 to -389°F	
	Accuracy: ± 5.0°F	
	Range: -234 to 1000°C	
	Accuracy: ± 0.6°C	
	Range: -252 to -234°C	
	Accuracy: ± 2.8°C	

Table 1. (Cont)

Test Instrument (TI) Characteristics	Performance Specifications	Test Method
Platinum 13% Rhodium vs Platinum (Type R)	Range: -58 to 3214°F	
	Accuracy: ±2.0°F	
	Range: -50 to 1768°C	
	Accuracy: ±1.1°C	
Platinum 10% Rhodium vs Platinum (Type S)	Range: -58 to 3214°F	
	Accuracy: ±2.0°F	
	Range: -50 to 1768°C	
	Accuracy: ±1.1°C	
Tungsten 5% Rhenium vs Tungsten 26% Rhenium (Type C)	Range: 32 to 4200°F	
	Accuracy: ± 3.0°F	
	Range: 0 to 2315°C	
	Accuracy: ±1.7°C	
Millivolt	Range: -101.10 to +101.10 mV	
	Accuracy: ±0.03% of Rdg +5 digits	

2 EQUIPMENT REQUIREMENTS:

	Noun	Minimum Use Specifications	Calibration Equipment	Sub-Item
2.1	DIGITAL VOLTMETER	Range: 0 to 100 mVDC	Keithley 181	L&N 7553-K3
		Accuracy: ±0.0075%		
2.2	DC VOLTAGE STANDARD	Range: 0 to 100 mVDC	Fluke 332D	
		Accuracy: N/A		
2.3	ICE BATH	Range: 32°F	Local Manufacture	
		Accuracy: N/A		
2.4	THERMOCOUPLE LEAD WIRE	As required by TI	Local Purchase	

3 PRELIMINARY OPERATIONS:

3.1 Review and become familiar with entire procedure before beginning calibration process.

WARNING

Unless otherwise designated, and prior to beginning the Calibration Process, ensure all test equipment voltage and/or current outputs are set to zero (0) or turned off, where applicable. Ensure all equipment switches are set to the proper position before making connections or applying power.

3.2 Connect TI and test equipment to 115 V/60 Hz power source. Set POWER switch to ON or STANDBY and allow 20 minutes warm-up.

3.3 The voltage vs temperature values used in this procedure were taken from NBS Monograph 125 based on IPTS-68.

3.4 Using distilled water, prepare an Ice Bath, and maintain in a slushy condition throughout calibration process.

3.5 Where negative voltage is required, set DC Voltage Standard controls to ZERO and reverse output leads.

3.6 Use only that portion of the procedure applicable to TI type and range.

4 CALIBRATION PROCESS:

NOTE

Unless otherwise specified, verify the results of each test and take corrective action whenever the test requirement is not met, before proceeding.

4.1 TEMPERATURE CALIBRATION: (Type J)

4.1.1 Connect equipment as shown in Figure 1, using Type J thermocouple wire.

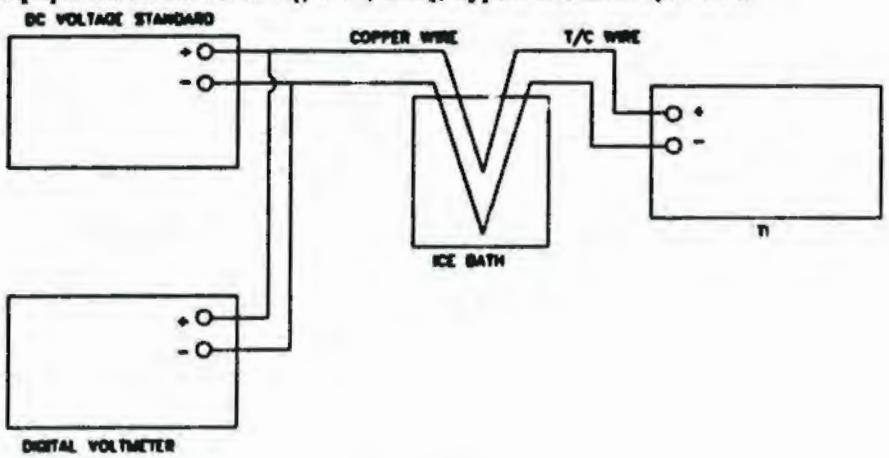

Figure 1

4.1.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.1.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.1.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 2.

4.1.5 The TI must indicate within the applicable limits listed in the Limits column of Table 2.

4.1.6 Repeat steps 4.1.4 and 4.1.5 for each of the remaining applied values listed in Table 2.

Table 2

Applied (mV)	Limits (°F)	(°C)
-7.519	-299 to -301	-183.9 to -185.1
1.942	99 to 101	37.1 to 38.3
14.108	499.0 to 501.0	259.6 to 260.8
29.515	999.0 to 1001.0	537.3 to 538.5
46.503	1499.0 to 1501.0	814.9 to 816.1
63.392	1999.0 to 2001.0	1092.7 to 1093.9

4.2 TEMPERATURE CALIBRATION: (Type K)

4.2.1 Connect equipment as shown in Figure 1, using Type K thermocouple wire.

4.2.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.2.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.2.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 3.

4.2.5 The TI must indicate within the applicable limits listed in the Limits column of Table 3.

4.2.6 Repeat steps 4.2.4 and 4.2.5 for each of the remaining applied values listed in Table 3.

Table 3

Applied (mV)	Limits (°F)	(°C)
-2.699	-99.0 to -101.0	-72.8 to -74.0
12.854	599.0 to 601.0	314.9 to 316.1
26.975	1199.0 to 1201.0	648.4 to 649.6
52.939	2399.0 to 2401.0	1315.0 to 1316.2

4.3 TEMPERATURE CALIBRATION: (Type T)

4.3.1 Connect equipment as shown in Figure 1, using Type T thermocouple wire.

4.3.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.3.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.3.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 4.

4.3.5 The TI must indicate within the applicable limits listed in the Limits column of Table 4.

4.3.6 Repeat steps 4.3.4 and 4.3.5 for each of the remaining applied values listed in Table 4.

Table 4

Applied (mV)	Limits (°F)	(°C)
-4.149	-199.0 to -201.0	-128.3 to -129.5
1.518	99.0 to 101.0	37.2 to 38.4
9.523	399.0 to 401.0	203.8 to 205.0
19.095	699.0 to 701.0	370.6 to 371.8

4.4 TEMPERATURE CALIBRATION: (Type E)

4.4.1 Connect equipment as shown in Figure 1, using Type E thermocouple wire.

4.4.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.4.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.4.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 5.

4.4.5 The TI must indicate within the applicable limits listed in the Limits column of Table 5.

4.4.6 Repeat steps 4.4.4 and 4.4.5 for each of the remaining applied values listed in Table 5.

Table 5.

Applied (mV)	Limits (°F)	(°C)
-9.604	-395.0 to -405.0	-236.9 to -242.5
13.748	399.0 to 401.0	203.9 to 205.1
49.020	1199.0 to 1201.0	648.3 to 649.5
75.024	1799.0 to 1801.0	981.6 to 982.8

4.5 TEMPERATURE CALIBRATION: (Type R)

4.5.1 Connect equipment as shown in Figure 1, using Type R thermocouple wire.

4.5.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.5.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.5.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 6.

4.5.5 The TI must indicate within the applicable limits listed in the Limits column of Table 6.

4.5.6 Repeat steps 4.5.4 and 4.5.5 for each of the remaining applied values listed in Table 6.

Table 6.

Applied (mV)	Limits (°F)	(°C)
0.598	198.0 to 202.0	92.5 to 94.7
6.143	1198.0 to 1202.0	647.6 to 649.8
13.286	2198.0 to 2202.0	1203.7 to 1205.9
20.275	3098.0 to 3102.0	1703.7 to 1705.9

4.6 TEMPERATURE CALIBRATION: (Type S)

4.6.1 Connect equipment as shown in Figure 1, using Type S thermocouple wire.

4.6.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.6.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.6.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 7.

4.6.5 The TI must indicate within the applicable limits listed in the Limits column of Table 7.

4.6.6 Repeat steps 4.6.4 and 4.6.5 for each of the remaining applied values listed in Table 7.

Table 7.

Applied (mV)	Limits (°F)	(°C)
0.597	198.0 to 202.0	92.7 to 94.9
5.740	1198.0 to 1202.0	647.8 to 650.0
12.001	2198.0 to 2202.0	1203.3 to 1205.5
17.993	3098.0 to 3102.0	1703.1 to 1705.3

4.7 TEMPERATURE CALIBRATION: (Type C)

4.7.1 Connect equipment as shown in Figure 1, using Type C thermocouple wire.

4.7.2 With no voltage applied, ensure that TI indicates ZERO or lowest applicable value.

4.7.3 Set Digital Voltmeter controls to measure DC millivolts, and verify DC Voltage Standard POWER switch is in the ON position.

4.7.4 Adjust the voltage controls of the DC Voltage Standard until the Digital Voltmeter indicates the first value listed in the Applied column of Table 8.

4.7.5 The TI must indicate within the applicable limits listed in the Limits column of Table 8.

4.7.6 Repeat steps 4.7.4 and 4.7.5 for each of the remaining applied values listed in Table 8.

NOTE

Voltage vs temperature values for Type C Thermocouple in Table 8, were taken from Table XVI of Omega Temperature Measurement Handbook.

Table 8

Applied (mV)	Limits (°F)	Limits (°C)
4.140	497.0 to 503.0	258.3 to 261.7
15.851	1597.0 to 1603.0	869.4 to 872.8
27.301	2797.0 to 2803.0	1536.1 to 1539.5
35.978	3997.0 to 4003.0	2202.7 to 2206.1

4.7.7 Disconnect equipment.

4.8 MILLIVOLT CALIBRATION:

4.8.1 Observing polarity and using copper wire leads, connect the output of the DC Voltage Standard to the input of the Digital Voltmeter and TI.

4.8.2 Set TI Function to switch MEASURE, and RANGE switch to mV.

4.8.3 Adjust DC Voltage Standard controls until the Digital Voltmeter indicates +15.00 mVDC.

4.8.4 The TI display must agree with the Digital Voltmeter, within the accuracy listed in Table 1.

4.8.5 Repeat steps 4.8.3 and 4.8.4 using applied values of +40.00, 70.00, and 90.00 mVDC.

4.8.6 Set DC Voltage Standard controls for ZERO output, reverse output leads, and repeat steps 4.8.3 through 4.8.5 for negative indications.

4.8.7 Set all switches to OFF. Disconnect and secure all equipment.

CALIBRATION PERFORMANCE TABLE

Not Required

*TB 9-6685-319-35

SUPERSEDED COPY DATED 1 JULY 1973

DEPARTMENT OF THE ARMY TECHNICAL BULLETIN

CALIBRATION PROCEDURE FOR DIAL INDICATING PRESSURE GAGES (GENERAL)

Headquarters, Department of the Army, Washington, DC
27 June 1988

Approved for public release; distribution is unlimited.

REPORTING OF ERRORS

You can help improve this publication by calling attention to errors and by recommending improvements and stating your reasons for the recommendations. Your letter or DA Form 2028, Recommended Changes to Publications, should be mailed directly to Commander, U.S. Army TMDE Support Group, ATTN: AMXTM-LPP, Redstone Arsenal, AL 35898-5400. A reply will be furnished directly to you.

*This bulletin supersedes TB 9 6685-319-50, 1 July 1973, including all changes.

SECTION I

IDENTIFICATION AND DESCRIPTION

1. Test Instrument Identification. This bulletin provides instructions for the calibration of Dial Indicating Pressure Gages (General). Various manufacturers' manuals were used as the prime data sources in compiling these instructions. The equipment being calibrated will be referred to as the TI (test instrument) throughout this bulletin.

a. Model Variations. Variations among models are indicated in the text.

b. Time and Technique. The time required for this calibration is approximately 2 hours, using the physical technique.

2. Forms, Records, and Reports

a. Forms, records, and reports required for calibration personnel at all levels are prescribed by TB 750-25.

b. Adjustments to be reported are designated (R) at the end of the sentence in which they appear. When adjustments are in tables, the (R) follows the designated adjustment. Report only those adjustments made and designated with (R).

3. Calibration Description. TI parameters and performance specifications which pertain to this calibration are listed in table 1.

Table 1. Calibration Description

Test instrument parameters	Performance specifications
Range and accuracy[1]	Performance at cardinal points in accordance with specifications for gage being tested.

[1]In those cases where accuracy of TI cannot be determined, TI will be certified to one graduation of TI scale.

SECTION II

EQUIPMENT REQUIREMENTS

4. Equipment Required. Table 2 identifies the specific equipment to be used in this calibration procedure. This equipment is issued with Secondary Transfer Calibration Standards Set AN/GSM-286. Alternate items may be used by the calibrating activity when the equipment listed in table 2 is not available. The items selected must be verified to perform satisfactorily prior to use and must bear evidence of current calibration. The equipment must meet or exceed the minimum use specifications listed in table 2. The accuracies listed in table 2 provide a four-to-one ratio between the standard and TI. Where the four-to-one ratio cannot be met, the actual accuracy of the equipment selected is shown in parenthesis.

5. Accessories Required. The accessories listed in table 3 are issued as indicated in paragraph 4 above and are used in this calibration procedure. When necessary, these items may be substituted by equivalent items, unless specifically prohibited.

Table 2. Minimum Specifications of Equipment Required

Item	Common name	Minimum use specifications	Manufacturer and model (part number)
A1	PNEUMATIC PRESSURE STANDARD	Range: 0 to 200 psi Accuracy:[1] ±0.025% (±0.125% of FS)	Cybersystems, Inc., Model ZA00225A1 (MIS-30859)
A2	PRESSURE GAGE TESTER	Range: 0 to 10,000 psi Accuracy:[2] ±0.025% (±0.15% of reading)	Mansfield and Green, Model 10-10525 (10-10525)

[1]Equipment limitation: ±0.05% of FS.
[2]Equipment limitation: ±0.015% of reading.

Table 3. Accessories Required

Item	Common name	Description (part number)
B1	ADAPTER[1]	Male 1/4-in., SP to male 7/16-20 UNF, 37° flare (part of MIS-26326)
B2	ADAPTER	Male 3/8-18 NPT to female 1/4-18 NPT (part of 8598963)
B3	ADAPTER	Male 7/8-14 UNF w/"o" ring to female 3/8-18 NPT (part of 8598963)
B4	ADAPTER	Male 7/16-20 UNF, 37° flare to female 1/8-18 NPT (part of 7913310)
B5	ADAPTER	Male 1/2-20 UNF to female 7/8-14 UNF (part of 8598963)
B6	CONNECTOR	Stainless steel, female 1/4-18 NPT to male 7/16-20 UNF, 37° flare (part of 7913310)
B7	DISTILLED WATER	Additional equipment required
B8	FLUID SEPARATOR	MIS-26326
B9	HOSE	3-ft, 3000 psi operating pressure, female 7/16-20 UNF, 37° flare angle fittings (7913310)
B10	HOSE	5-ft, 5000 psi operating pressure (part of 7913310)
B11	HOSE	Male 1/4-18 NPT ends (part of 8598963)
B12	NITROGEN TANK	Water pumper nitrogen (7910373)

See footnote at end of table.

Table 3. Accessories Required - Continued

Item	Common name	Description (part number)
B13	PNEUMATIC PRESSURE CONTROLLER	MIS-10324
B14	REGULATOR	MIS-10325 Type II
B15	SQUEEZE BOTTLE	(Part of MIS-26326)
B16	TEE	Stainless steel, swivel nut (8491696) (part of 7913310)
B17	TUBE ASSEMBLY	1/4-in. AN back-to-back (female 7/16-20 UNF, 37° flare both ends (7913309) (part of 7913310)

[1]Two required.

SECTION III

CALIBRATION PROCESS FOR HYDRAULIC GAGES

6. Preliminary Instructions

a. The instructions outlined in paragraphs 6 and 7 are preparatory to the calibration process. Personnel should become familiar with the entire bulletin before beginning the calibration.

b. Items of equipment used in this procedure are referenced within the text by common name and item identification number as listed in tables 2 and 3. For the identification of equipment referenced by item numbers prefixed with A, see table 2, and for prefix B, see table 3.

c. Unless otherwise specified, verify the result of each test and, whenever the test requirement is not met, take corrective action before continuing with the calibration. Adjustments required to calibrate the TI are included in this procedure. Additional maintenance information is contained in the manufacturer's manual for this TI.

d. Unless otherwise specified all controls and controls settings refer to the TI.

7. Equipment Setup

a. Remove pressure gage tester (A2) from carrying case.

b. Remove filler plug and fill pressure gage tester with hydraulic oil (MIL-L-7870A).

c. Reinstall filler plug and secure pressure gage test to an adequate workbench. Level pressure gage tester.

d. Visually inspect TI for signs of damage or deterioration.

e. Throughly clean bourdon tube or diaphragm of TI. First, rotate TI and allow any liquid which may be in the bourdon tube or diaphragm area to flow out. Using an eye dropper or small glass tube, fill bourdon tube or diaphragm area with toluene, methyl alcohol, or freon TF solution. Allow solution to remain in the gage for 5 minutes. Drain gage and dry for at least 10 minutes.

f. If required, zero-adjust dial indicator of TI by removing retaining ring and coverglass and adjusting the calibration screw.

8. 0 to 10,000 Psi Hydraulic Gages (0.1 to 20 Percent Accuracy)

a. Performance Check

(1) Connect equipment as shown in figure 1.

(2) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(3) Place enough deadweights (supplied with pressure gage tester (A2)) on applicable low or high pressure piston to obtain a pressure equal to cardinal point nearest 10 percent of TI scale.

CAUTION

To avoid scoring the piston guide and damage to the deadweight cylinder, constantly rotate weights and piston when inserting or removing piston or when applying pressure.

(4) Using hand pump, apply pressure to deadweight cylinder until (low or high pressure) piston is approximately 9/16-inch above deadweight cylinder.

(5) Visually inspect equipment connection for leakage. If leakage appears, release pressure and tighten or seal connections as required.

CAUTION

Do not remove weights installed in (3) above from low or high pressure piston during remainder of this procedure.

(6) Place enough deadweights on applicable low or high pressure piston to obtain the 90 percent cardinal point of TI. Refer to table 4 for conversion table.

(7) Repeat (4) above.

(8) If TI does not indicate within limits calculated in (2) above perform b below.

(9) Repeat (2) and (6) through (8) above, using enough weights to obtain cardinal points nearest to 80, 60, 40, 20, and 10 percent of TI scale.

(10) Throughly clean TI in accordance with paragraph 7 above.

b. Adjustments (Typical, fig. 2).

NOTE

The following adjustment procedure applies specifically to solfront gages, but may be used as a guide for other gages. Manufacturers' instructions should be used if available.

(1) Deviations either plus or minus by a constant value are corrected by repositioning the pointer. Remove bezel ring, loosen locking screw (marked L) 1/4 turn, and adjust screw marked "A". Tighten locking screw and install bezel ring.

(2) Deviations varying linearly over range of TI are corrected by repositioning link screw. Remove protective back or case from mechanism, loosen link screw and move in slot of sector as required. Tighten link and install protective back of case.

(3) Deviations varying non-linearly over range of TI are corrected by repositioning arc-loc movement. Remove protective back or case, loosen the three locking screws, and rotate arc-loc movement as required. If indications are first increasingly plus and then decreasingly plus, rotate arc-loc movement in direction "A". If indictions are first increasingly minus and then decreasingly minus, rotate arc-loc movement in direction "B". Tighten locking screws.

(4) Repeat (1), (2) or (3) as necessary to obtain indications within tolerance.

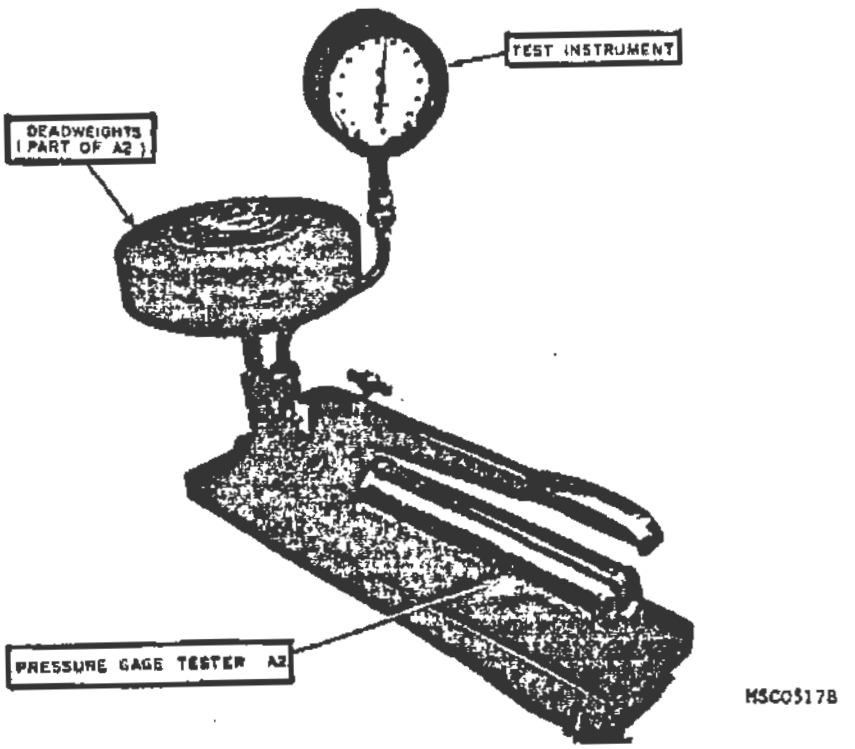

Figure 1. 1.0 to 10,0000 psi hydraulic gages (0.1 to 1.0 percent accuracy) - equipment setup.

Table 4. Conversion Table

Number of deadweights supplied with pressure gage tester (ea)	Value of each weight (lbs)	1 weight equal the equivalent pressure (psi)[1]	
		Low pressure piston	High pressure piston
18	10	100	500
1	9½	95	475
4	2	20	100
4	½	5	25

[1]Pressure generated by deadweight testers are affected by acceleration of gravity. Mansfield and Green, Model 10-1025 deadweight tester generates true pressures at locations where the acceleration of gravity is 980.217 cm/s². The acceleration of gravity must be considered when using the deadweight tester to calibrate TI's with an accuracy of ±0.35% of FS or better. To correct for the effect of gravity, multiply total pressure of weight combination by local gravity (units - cm/s²) and divide by 980.217 cm/s².

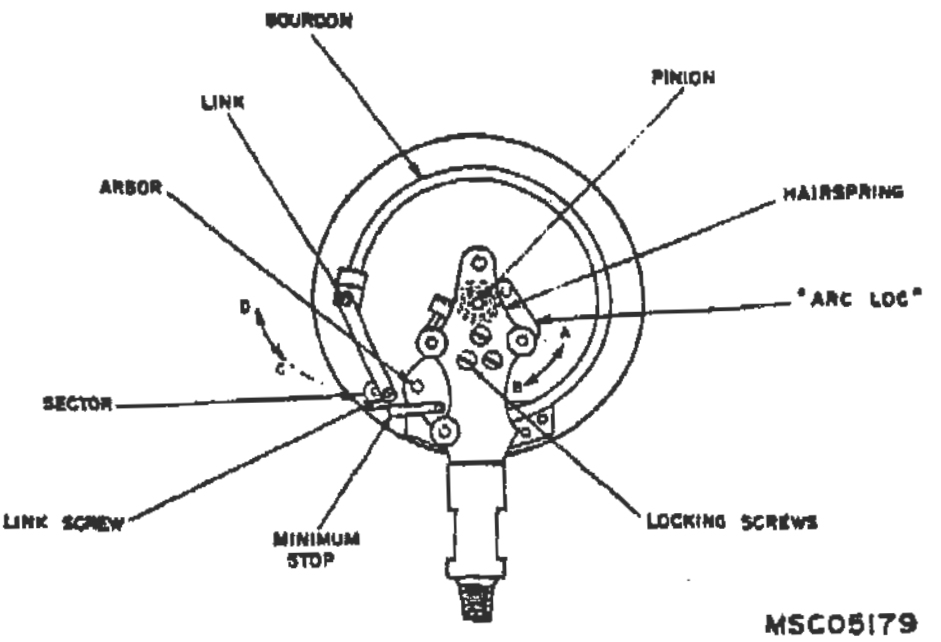

Figure 2. Typical pressure gage..

9. 0 to 5000 Psi Panel Mounted Hydraulic Gages (1.0 to 20 Percent Accuracy)

a. Performance Check

NOTE

Convert accuracies of TI and standard into pressure units (psi). If four-to-one accuracy ratio (in pressure units) cannot be attained, use procedure in paragraph 10 below.

(1) Select a standard pressure gage from pressure gage tester (A2) which will cover the same range as the TI.

(2) Connect appropriate standard pressure gage and TI in the system in a way that will insure that the same pressure will be applied to both gages.

NOTE

The standard pressure gage should be located so that reference plane of each instrument is the same height to eliminate an error caused by hydraulic head pressure.

(3) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(4) Using system pressure source, apply pressure to obtain an indication on standard pressure gage equal to 10 percent of TI scale.

NOTE

It is preferred that the system pressure source be utilized when calibrating panel mounted pressure gages, since TI and system pressure source are connected. If system pressure source is not available, utilize procedure in paragraph 8 above.

(5) If TI indication does not indicate within limits calculated in (3) above, perform b below.

(6) Repeat (1) through (5) above, using cardinal points nearest to 80, 60, 40 and 20 percent of TI scale.

b. Adjustments. Perform 8b above.

10. 0 to 10,000 Psi Panel Mounted Hydraulic Gages

a. Performance Check

WARNING

To prevent injury to personnel and/or damage to equipment, make certain that all components are within range of unit to be calibrated and all connections are securely sealed prior to applying pressure to TI.

(1) Connect equipment as listed in (a) through (d) below:

(a) Cap one vertical port on deadweight tester (part of A2).

(b) Connect applicable low or high pressure cylinder to other vertical port on deadweight tester and install appropriate piston.

(c) Connect horizontal outlet port of deadweight tester to input port of TI.

(d) Cap all other system outlet ports and close system shut-off valve.

NOTE

The deadweight tester should be located so that reference planes of TI and deadweight tester are same height to eliminate an error caused by hydraulic head pressure.

(2) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(3) Place enough deadweights (supplied with pressure gage tester) on the applicable low or high pressure piston to obtain an indication on standard pressure gage equal to 10 percent of TI scale.

(4) Using hand pump, apply pressure to system until (low or high pressure) piston is approximately 9/16-inch above deadweight cylinder.

(5) If TI indication does not indicate within limits calculated in (2) above, perform b below.

(6) Repeat (2) through (5) above, using cardinal points nearest to 80, 60, 40, and 20 percent of TI scale.

b. Adjustments. Perform 8b above.

11. Final Procedure

a. Deenergize and disconnect all equipment.

b. Annotate and affix DA Label/Form in accordance with TB 750-25.

SECTION IV

CALIBRATION PROCESS FOR

PNEUMATIC GAGES

12. Preliminary Instructions

a. The instructions outlined in paragraphs 12 and 13 are preparatory to the calibration process. Personnel should become familiar with the entire bulletin before beginning the calibration.

b. Items of equipment used in this procedure are referenced within the text by common name and item identification number as listed in tables 2 and 3. For the identification of equipment referenced by item numbers prefixed with A, see table 2, and for prefix B, see table 3.

c. Unless otherwise specified, verify the result of each test and, whenever the test requirement is not met, take corrective action before continuing with the calibration. Adjustments required to calibrate the TI are included in this procedure. Additional maintenance information is contained in the manufacturer's manual for this TI.

d. Unless otherwise specified, all controls and control settings refer to the TI.

13. Equipment Setup

a. Visually inspect TI for signs of damage or deterioration.

b. Throughly clean bourdon tube or diaphragm of TI. First, rotate TI and allow any liquid which may be in the bourdon tube or diaphragm area to flow out. Using an eye dropper or small glass tube, fill bourdon tube or diaphragm area with toluene, mathyl alcohol, or freon TF solution. Allow solution to remain in the gage for 5 minutes. Drain gage and dry for at least 10 minutes.

c. If required, zero-adjust dial indicator of TI by removing retaining ring and coverglass and adjusting the calibration screw.

14. 0 to 235 Psi Pneumatic Gages (0.1 to 1.0 Percent Accuracy)

a. Performance Check

(1) Connect equipment as shown in figure 3, connection A.

NOTE
The maximum input to the 0 to 20 psia port should not exceed 6 psi. The maximum input to 0 to 250 psia port should not exceed 235 psi.

WARNING
To prevent injury to personnel and/or damage to equipment, make certain that all components are within range of unit to be calibrated and all connections are securely sealed prior to applying pressure to TI. Never attempt to tighten connection with pressure applied. Insure that TI is clean and free of oil or grease.

(2) Position controls on pneumatic pressure standard (A1) as listed in (a) through (d) below:

(a) UNITS DISPLAYED switch to PSIA.

(b) RANGE pushbutton to 0-250.

(c) SENSITIVITY pushbutton to HIGH.

(d) SOURCE pushbutton to INT.

(3) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(4) Open exhaust, metering and shutoff valves on pneumatic pressure controller (B13).

(5) Press **RESET** and **ZERO** pushbutton on pneumatic pressure standard.

(6) Close exhaust, metering and shutoff valves on pneumatic pressure standard.

(7) Turn regulator control fully ccw.

(8) Open nitrogen tank (B12) valve and adjust regulator until outlet gage of regulator indicates maximum pressure of TI.

(9) Open inlet valve on pneumatic pressure controller.

(10) Slowly open shut off valve on pneumatic pressure controller.

(11) Increase pneumatic pressure, using metering valve on pneumatic pressure controller until TI reaches full scale.

(12) If TI indication does not indicate within limits calculated in (3) above, perform b below.

(13) Close inlet valve and slowly open exhaust valve.

(14) Release pressure by opening metering valve.

NOTE
Pressure changes can be made by proper use of inlet, exhaust, metering, and shut off valves.

(15) Repeat (2) and (6) through (14) above at cardinal points nearest to 80, 60, 40, and 20 percent of TI scale.

b. Adjustments. Perform 8b above.

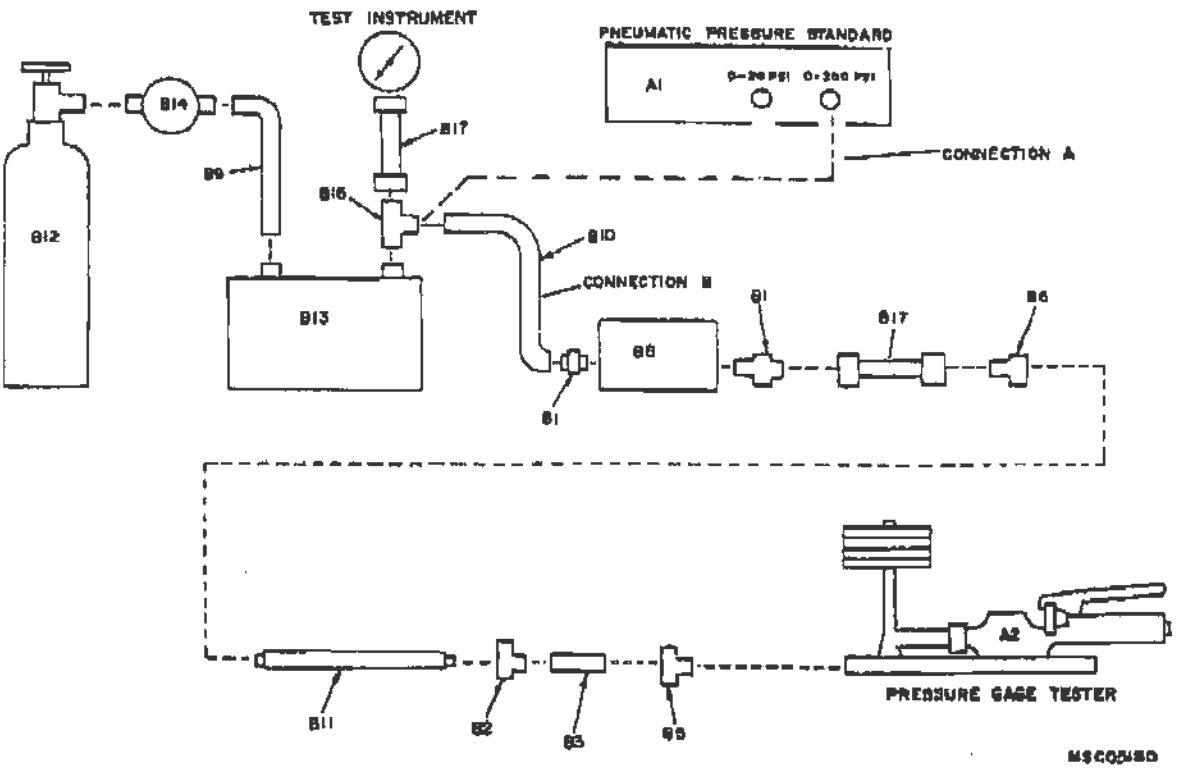

Figure 3. 0 to 1000 psi pneumatic gages (0.1 to 1.0 percent accuracy) - equipment setup.

15. 235 to 1000 Psi Pneumatic Gages (0.1 to 1.0 Percent Accuracy)

WARNING

To prevent injury to personnel or damage to equipment, make certain that all components used are clean and have not been contaminated with oil or grease.

a. Performance Check

(1) Connect equipment as shown in figure 3, connection B.

(2) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(3) Open exhaust, metering and shutoff valves on pneumatic pressure controller (B13).

(4) Close reservoir valve on deadweight tester, and inlet valves on pneumatic pressure controller.

(5) Loosen fill and vent plug on pressure gage tester.

(6) Operate hand pump until weight table rises (tare weight).

(7) Open reservoir valve, allowing weight table to fall to bottom stop; then wait about 10 seconds before closing reservoir valve.

(8) Close shutoff, metering, and exhaust valves on pneumatic pressure controller.

WARNING

To avoid scoring the piston guide and damage to the deadweight cylinder, constantly rotate weights and piston when inserting or removing piston or when applying pressure.

(9) Place enough deadweights (supplied with pressure gage tester (A2)) on applicable low or high pressure piston to obtain a pressure equal to cardinal point nearest to 10 percent of TI scale.

(10) Turn regulator (B14) fully ccw.

(11) Open nitrogen tank (B12) valve and adjust regulator until outlet gage of regulator indicates maximum pressure of TI.

(12) Insure that metering and exhaust valves on pneumatic pressure controller are closed.

(13) Open inlet valve on pneumatic pressure controller.

(14) Slowly open shutoff valve on pneumatic pressure controller.

(15) Increase pneumatic pressure, using metering valve on pneumatic pressure controller until weight rises approximately 9/16-inch above the deadweight cylinder.

NOTE

Making all pressure changes (however large or small) with pneumatic pressure controller. Vernier pressure changes can be made using screw-type pump on pneumatic pressure controller. The hydraulic hand pump should not be used after initial diaphragm positioning in (6) and (7) above.

(16) After reading and recording TI indication, a correction must be applied to each indication. The difference between reference plane of fluid separator (B8) and deadweight tester when both are resting on same table is 0.988-inch for high range position and 3.578-inches for low range position. The density of recommended oil (MIL-L-7870A) is 0.8653 gm/cc at 23 gm/cc at 23°C; therefore, 1-inch of this oil produces a pressure of 0.03125 psi. Thus, 0.031 psi (0.0312 psi x 0.98 inch) for high range position and 0.112 psi (0.3125 psi x 3.578 inch) for low range position must be subtracted from each TI indication.

(17) If TI indication after correction does not indicate within limits calculated in (2) above, perform b below.

(18) Close inlet valve and slowly open exhaust valve.

(19) Release pressure by opening metering valve.

(20) Repeat (2) and (12) through (19) above, using enough weights to obtain cardinal points nearest to 80, 60, 40, and 20 percent of TI scale. Pressure changes can be made by proper use of inlet, exhaust, metering, and shutoff valves.

b. Adjustments. Perform 8b above.

16. 1000 to 5000 Psi Pneumatic Gages (0.1 to 1 Percent Accuracy)

WARNING

To prevent injury to personnel or damage to equipment, make certain that all components used are clean and have not been contaminated with oil or grease.

a. Performance Check

(1) Connect equipment as shown in figure 4, except do not connect hose (B10) to fluid separator (B8).

(2) Open vent port on fluid separator.

(3) Operate hand pump until all the air is bled from hydraulic side of fluid separator.

(4) With fluid separator full of oil, install plug-in vent port.

(5) Connect appropriate standard gage to pressure gage tester (A2).

(6) Fill pneumatic side of fluid separator with distilled water (B7), using squeeze bottle (B15).

(7) Fill hose (B10) with distilled water and connect to fluid separator.

NOTE

In filling pneumatic side of fluid separator hose, connection, and TI; it is important that the components be filled completely and kept as full as possible during connection. Position diaphragm to pneumatic side and hydraulic side several times by manipulation of hand pump and reservoir valve on pressure gage tester.

(8) Position fluid separator diaphragm to oil side by opening reservoir valve on pressure gage tester and holding hose (B10) above fluid separator, creating a head pressure. Refill hose as water falls.

(9) Fill TI with distilled water and connect to hose.

(10) Calculate tolerance limits for TI, using accuracy specified for applicable gage.

(11) Place enough deadweights (supplied with pressure gage tester) on applicable low or high pressure piston to obtain a pressure equal to cardinal point nearest to 10 percent of TI scale.

WARNING

To avoid scoring the piston guide and damage to the deadweight cylinder, constantly rotate weights and piston when inserting or removing piston or when applying pressure.

(12) Using hand pump, apply pressure to deadweight cylinder until (low or high pressure) piston is approximately 9/16-inch above deadweight cylinder.

(13) Visually inspect equipment connections for leakage. If leakage appears, release pressure and tighten or seal connections as required.

CAUTION

Do not remove weights installed in (11) above from low or high pressure pistons during remainder of this procedure.

(14) Place enough deadweights on applicable low or high pressure piston to obtain the 90 percent cardinal point of TI. Refer to table 4 for conversion table.

(15) Repeat (12) above.

(16) If TI indication does not indicate within limits calculated in (10 above, perform b below.

(17) Repeat (10) and (14) through (16) above, using enough weights to obtain cardinal points nearest to 80, 60, 40, 20, and 10 percent of TI scale.

b. Adjustments. Perform 8b above.

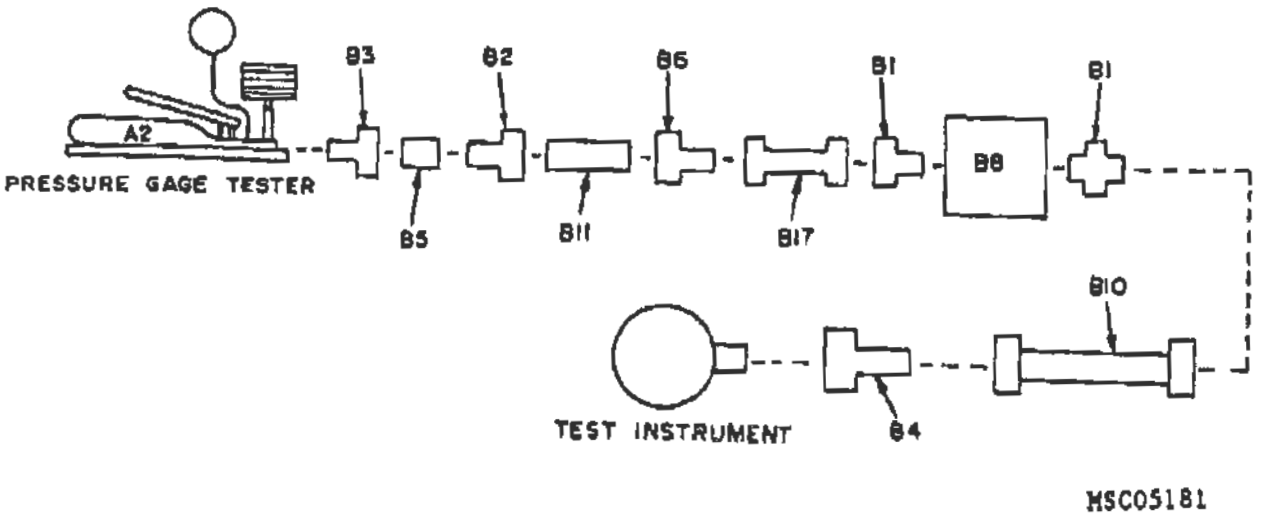

Figure 4. 1000 to 5000 psi pneumatic gages (0.1 to 1 percent accuracy) - equipment setup.

17. Final Procedure

a. Deenergize and disconnect all equipment and reinstall protective cover on TI.

b. Annotate and affix DA Label/Form in accordance with TB 750-25.

By Order of the Secretary of the Army:

CARL E. VUONO
General, United States Army
Chief of Staff

Official:

R. L. DILWORTH
Brigadier General, United States Army
The Adjutant General

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APPENDIX H

AMMUNITION TERMINOLOGY

APPENDIX H

AMMUNITION TERMINOLOGY

1. API	Designation of an armor-piercing incendiary munition which is designed to penetrate armor and destroy the target by fire.
2. AP-T	Designation for armor-piercing munition designed to penetrate armor which has a tracer added to aid the gunner in following the projectile path to the target.
3. Booster	A component of an explosive train which, by exploding, amplifies the action of the detonator providing the initiating force necessary for the explosion of the burster or main charge.
4. Cartridge (CTG)	Complete small arms munition containing a propellant, primer, and projectile, and in some cases, an incendiary or tracer component.
5. Detonator	A component of an explosive train which amplifies the action of the primer and provides the initiating force necessary to explode the booster.
6. Explosive Train	A sequence of components in a munition which are designed to generate an explosion and/or flight of a projectile. For example, in the cartridges listed previously, the primer is activated by percussion and/or an electrical charge. The combustion of primer ignites the propellant which, by its action, propels the projectile toward the target. The projectile may also contain incendiary and tracer components. In larger munitions, the explosive train is started by the activation of a fuze containing a primer. The primer combustion causes the detonator to explode. The explosion of the detonator amplifies the force of the primer combustion and causes the booster charge to explode. The booster charge explosion activates the burster and/or the main charge.

7. Fuze	Component of ammunition which, when activated, initiates the explosive train reaction. TYPES PD Fuze: Point-detonating fuze, located on front of the projectile, and is activated by impact and/or time. BD Fuze: Base-detonating fuze, located at the rear of the projectile and is activated after impact with the target. MTSQ Fuze: Mechanical time-super quick fuze, fuze has a mechanical linkage and various time setting for detonation
8. HEI	Designation for a high explosive incendiary munition designed to destroy a target by fire and explosion.
9. HEI-T	Designation for a high explosive incendiary munition with a tracer added to aid the gunner in following the projectile path to the target.
10. HV-TP-T	Designation for a high velocity, target practice munition with a tracer added to aid the gunner in following the projectile path to the target.
11. Igniter	Portion of the rocket motor which causes the ignition of the propellant.
12. INC	Designation for an incendiary munition designed to destroy a target by fire.
13. Incendiary	Mixture of chemical compounds contained in a projectile designed to burn and destroy a target by fire.
14. Primer	Component of a munition containing a small amount of sensitive high explosive which starts the explosive chain reaction by rapid combustion.
15. Propellant	A chemical mixture designed for rapid combustion with a large evolution of gas which used to propel a projectile out of the weapon.
16. Rocket Motor	The propellant portion of a rocket munition.

17. TP-T	Designation for a target practice munition with a tracer added to aid the gunner in following the projectile path to the target.
18. Tracer	Mixture of chemical compounds contained in a projectile designed to burn and produce color and light.

APPENDIX I

Performance Specification Test

APPENDIX I

SENECA ARMY DEPOT
PERFORMANCE SPECIFICATION TEST

I-1 INTRODUCTION

The Seneca Army Depot maintains and operates a Continuous Emissions Monitor (CEM) system which measures the gas concentrations of oxygen (O2) and carbon monoxide (CO) from the APE 1236 deactivation furnace (DF). During the trial burn, these instruments will be the monitors of record for O_2 and CO. Before the data from the CEM system can be used however, the system must be certified to meet the specifications outlined in Methods Manual for Compliance with the BIF Regulations. Specifically, a Performance Specification Test (PST) must be performed to evaluate the acceptability of Seneca Army Depot's CEM system and the data that it generates.

This document provides a description of the CEM system installed on the Deactivation Furnace and the procedures that will be used to evaluate its acceptability. The procedures described in this document are based on those outlined in "Performance Specifications for Continuous Emissions Monitoring Systems" in the BIF Manual.

I-2 APE 1236 DEACTIVATIONFURNACE CEM DESCRIPTION

I-2.1 CO Monitoring System

When the APE 1236 deactivation furnace is operating, stack gas is drawn continuously from a port in the stack at approximately 20 feet above grade and delivered to the analyzer in the control room. The gas is pulled through a heated line (constructed of material that is non-reactive to the gas) at a rate specific to the analyzer. The sample is conditioned prior to analysis since the NDIR analyzer requires a cool and dry sample. Sample gas conditioning involves removal of particles and moisture from the sample prior to introduction to the analyzer. The sample conditioning system consists of a dual channel refrigerated condenser and a multiple-stage filtration unit.

The CO analyzer is a Rosemount/Beckman 880 NDIR; a dual-range model that has a 0-200 ppm range and a 0-3000 ppm range. When CO concentrations exceed the lower range, the

instrument automatically switches to the higher range. After the sampling system is ready for use, calibration gases are directly introduced into the instrument for an analyzer calibration error check. When this has been completed, calibration gas is introduced into the sampling system at the probe to provide a sampling system bias check. A calibration error check and a sampling system bias check is performed prior to every run. At the completion of each run, a calibration drift check and a sampling system bias is performed. These procedures are used to validate the analyzers responses during the sampling period. Further details regarding calibration gases, operating procedures, QA/QC procedures and corrective actions are provided in **Appendix G**. Manufacturer's information for the CO analyzer is included in **Appendix B**.

I-2.2 O_2 Monitoring System

When the APE 1236 deactivation furnace is operating, stack gas is drawn continuously from a port in the stack at approximately 20 feet above grade and delivered to the analyzer in the control room. The gas is pulled through a heated line (constructed of material that is non-reactive to the gas) at a rate specific to the analyzer. The sample is analyzed by a paramagnetic Oxygen analyzer, which requires a cool and dry sample. Sample gas conditioning involves removal of particles and moisture from the sample prior to introduction to the analyzer. The sample conditioning system consists of a dual channel refrigerated condenser and a multiple-stage filtration unit.

The analyzer operating range is chosen such that the expected sample gas concentration is on-scale. The O_2 analyzer is a Rosemount Analytical 755R Paramagnetic Oxygen Analyzer. The standard full-scale operating ranges for this instrument are 0-5%, 0-10%, 0-25%, 0-50%, and 0-100%. During normal operation the system will be operating in the 0-25% range. After the sampling system is ready for use, calibration gases are directly introduced into the instrument for an analyzer calibration error check. When this has been completed, calibration gas is introduced into the sampling system at the probe to provide a sampling system bias check. A calibration error check and a sampling system bias check is performed prior to every run. At the completion of each run, a calibration drift check and a sampling system bias is performed. These procedures are used to validate the analyzer response during the sampling period. Further details regarding calibration gases, operating procedures, QA/QC procedures and corrective actions are provided in **Appendix G**. Manufacturer's information for the Oxygen analyzer is included in **Appendix B**.

I-3 PERFORMANCE SPECIFICATION TEST PROCEDURES

I-3.1 Overview

This section describes specific testing procedures that will be used to evaluate the CEM system. There are four discrete tests conducted on the CEM system itself and one test on the effluent gas at the CEM sampling location. Each analyzer will be subjected to a 7-day calibration drift test, a response time test, and a calibration error test. In addition, the analyzers and the recording system will be subjected to a relative accuracy (RA) test by sampling the effluent gas simultaneously alongside another CEM system and comparing the results for O_2 and CO. The effluent gas at the CEM sampling location will be tested to determine if significant stratification exists at the sampling location.

I-3.2 7-day Calibration Drift Test Procedure

The 7-day Calibration Drift (CD) test is conducted to determine the magnitude of the system drift over a period of seven operating days. At 24-hour intervals, the CEM system is challenged with zero gas and upscale gas standards for both O_2 and CO. The observed system drift per 24-hour period must not exceed 0.5 percent O_2 or three percent of the CO span value.

Engineering-Science will procure zero and upscale gas standards for the 7-day calibration drift test. Ultra-high purity (UHP) nitrogen will be used as the zero gas for both the O_2 and CO analyzers. This zero standard is certified by the manufacturer to contain less than 0.5 ppm O_2 and 1 ppm CO. Upscale standards will consist of approximately 18.75 percent O_2 in nitrogen, 150 ppm CO in nitrogen, and 2250 ppm CO in nitrogen. Each of the up-scale gas standards will be prepared and analyzed according to EPA Traceability Protocol-1.

At the start of the 7-day calibration drift test, each of the four calibration gas standards will be introduced into the CEM system, one at a time, at a location near the probe. The calibration gases will be allowed to flow through the same flow path as a stack sample normally would. Each of the gases will be allowed to flow for approximately five minutes, in order to ensure a stable system response. The CEM system will be returned to normal operation after the last gas has been introduced. This procedure will be repeated six times at 24-hour intervals, and six values of calibration drift will be calculated according to the following equation:

$$\% \text{ Drift} = \{(R_{(n-1)} - R_{(n)}) / \text{Span value}\} \times 100$$

where:

$R_{(n)}$ = analyzer response to cylinder gas
$R_{(n-1)}$ = previous analyzer response to cylinder gas
Span value = analyzer full-scale span (e.g. 25% for O_2)

Analyzer responses will be recorded and the results calculated on a data sheet similar to the one shown in **Figure I-1**.

I-3.3 Response Time Test Procedure

The system response time will be measured to ensure that the emission rate calculated by the CEM system is a reasonable estimate of the true stack conditions at the time of the calculation. System response time is the maximum value observed between the average upscale response time and the average downscale response time.

The system response time test will be conducted one time prior to the Relative Accuracy test. A zero gas standard (UHP N_2) will be introduced into the system at a point as close as possible to the CEM sampling location. The zero gas standard will flow until the CEM system response for O_2 and CO stabilizes, which is defined as a change of no more than one percent of the span range in any thirty-second interval. After the system has stabilized, stack gas will be allowed to enter the system, and the CEM system allowed to reach a stable value. The upscale system response time will then be recorded, which is defined as the time for the system to reach 95 percent of the stable stack gas value. A high level span gas, corresponding to approximately 75 percent of the span range, will then be introduced into the system and the system will be allowed to stabilize. Stack gas will then be allowed to enter the system, and the time to reach 95 percent of a stable stack gas value will be recorded. This procedure will be repeated three times for the zero gas and for each of the high level calibration gases for the O_2 and CO analyzers. The four values of the upscale and downscale responses will be averaged for each analyzer, and the maximum response time will be recorded as the system response time. An example system response time data sheet is presented in **Figure I-2**.

FIGURE I-1

CALIBRATION DRIFT DETERMINATION

Source:	Date:
Monitor:	Location:
Serial Number:	Span:

Low Range	
High Range	

Day	Date	Time	Calibration Value	Monitor Response	Difference	Percent of Span*
Zero/Low Level						
1						
2						
3						
4						
5						
6						
7						
High Level						
1						
2						
3						
4						
5						
6						

I.3.4 Calibration Error Test Procedure

The purpose of the calibration error test is to determine the analyzer accuracy over the measurement span. To determine the calibration error, each analyzer will be challenged with a zero and two upscale gas standards for each measurement range. Gas standards will be certified by the manufacturer as being prepared according to US EPA Traceability Protocol-1. Gas standards will be introduced into the CEM system at a point as close as possible to the CEM probe (one at a time for three non-consecutive times) and the CEM response is recorded. The mean difference between the calibration gas and the CEM response will be calculated for each gas injected, and the calibration error calculated in terms of the percent of the span range. The calibration error must be no more than 5 percent of the span range for any gas standard in order for the system to meet the calibration error specification. Calibration error will be calculated using the following equation:

$$\% \text{ CE} = \{(\text{R} - \text{CV}) / \text{Span Value}\} \times 100$$

where:

R = analyzer response to cylinder gas
CV = certified cylinder gas value
Span value = analyzer full-scale span (e.g. 25% for O_2)

An example data sheet for the O_2 and CO calibration error tests is presented in **Figure I-3.**

I-3.5 Relative Accuracy Test Procedure

The purpose of the relative accuracy test is to determine the accuracy of the installed CEMs with respect to US EPA Reference Methods. ES will use a mobile CEM system to sample the stack gas effluent independent of, and simultaneously with the installed CEM system. The CO concentration will be calculated and corrected to seven percent O_2. These results will then be compared with the results of the Seneca CEM system to determine the percent relative accuracy.

I-3.6 Relative Accuracy Test Strategy

Engineering-Science, Inc. will conduct twelve EPA Reference Method tests to determine the oxygen and carbon monoxide concentrations in the stack gas. Oxygen will be determined

FIGURE I-2
RESPONSE TIME DETERMINATION

Parameter _____

Person Conducting Test _____

Affiliation _____

Date _____

Analyzer Manufacturer _____

Model Serial No. _____

Location _____

Analyzer Span Setting _____

Upscale Span Gas Concentration (_____)

Upscale Gas Response 1_____seconds

2_____seconds

3_____seconds

4_____seconds

Average Upscale Gas Response _____seconds

Zero Gas Response 1_____seconds

2_____seconds

3_____seconds

4_____seconds

Average Zero Gas Response _____seconds

Average System Response Time _____seconds

Maximum System Response Time _____seconds

FIGURE I-3

CALIBRATION ERROR DETERMINATION

Source:	Date:
Monitor:	Location:
Serial Number:	Span:

Low Range	
High Range	

Run Number	Calibration Value	Monitor Response	Difference Zero/Low	Mid	High
1-Zero					
2-Mid					
3-High					
4-Mid					
5-Zero					
6-High					
7-Zero					
8-Mid					
9-High					
		Mean Difference=			
		Calibration Error =	%	%	%

using Reference Method 3A and carbon monoxide will be determined using Reference Method 10. Each of the sample runs will last for approximately thirty minutes. Simultaneously with the Reference Method sampling, the DF CEM system will record oxygen and carbon monoxide concentrations in the stack gas. To determine the relative accuracy of the installed CEM system, a minimum of nine sets of data will be used.

I-3.7 Description of Engineering-Science CEM System

The mobile stack gas monitoring system employed by Engineering-Science consists of the following components:

1. Heated stainless steel probe fitted with a 100-micron in-stack stainless steel sintered filter with particle shield.

2. Heated CEM stack gas interface box. Capable of maintaining a temperature of 250 °F by a temperature controller located in the CEM van. The stack gas sample is split after the probe and directed to two three-way valves, which allow the introduction of calibration gas or stack gas into the system.

3. Multi-tube heat-traced sample line for transport of stack gas to the moisture condenser. Line consists of one heated sample line and one unheated calibration gas line. The sample line is a self-limiting heated line which is designed to limit the sample gas temperature to ambient plus 250 °F.

4. Sample gas moisture condenser, designed to cool the sample gas in order to condense and collect entrained moisture. The condenser is constructed of a PVC plastic condensate reservoir and a 10-foot coil of Teflon tubing. The sample gas is cooled using an ice bath.

5. Sample gas manifold, for distribution of sample gas to continuous analyzers. The inlet of the sample gas manifold is fitted with a 10-micron Balston filter. Each outlet leg of the manifold is fitted with an in-line filter.

6. Continuous analyzers, for the measurement of target diluent and pollutant gases.

7. Data Acquisition System (DAS), to measure and record analyzer responses. The DAS consists of an analog-to-digital signal converter and a personal computer. Analyzer responses are recorded on the computer's hard disk drive as well as on a paper printout.

Prior to each day of sampling, each analyzer is calibrated using Protocol-1 gas standards. After calibration the stack interface box and the heated sample line are checked to determine the system bias by introducing a zero and upscale gas standard for each analyzer. The sample system bias is acceptable if the analyzer response to the bias gas differs from the calibration response by no more than five percent of the operating range. At the conclusion of a test period, the system zero and upscale bias are again determined to calculate the zero and upscale system drift during the course of the sampling period.

I-3.7.1 ES CO Monitoring System

The CO analyzer used by Engineering-Science is the Teco Model 48 Carbon Monoxide Analyzer, which has full-scale ranges 0-1, 2, 5, 10, 20, 50, 100, 200, 500 and 1000 ppm CO. It is typically operated in the 0-1000 ppm range with standard gases that are 300 ppm, 600 ppm and 900 ppm CO in N_2.

The Teco Model 48 CO Monoxide analyzer operates on the principles of gas filter correlation spectroscopy. The technique of gas filter correlation (GFC) offers improved specificity and sensitivity over conventional nondispersive infrared (NDIR) techniques. GFC spectroscopy is based upon comparison of the detailed structure of the infrared absorption spectrum of the measured gas to that of other gases also present in the sample being analyzed. The technique is implemented by using a high concentration sample of the measured gas, i.e., CO, as a filter for the infrared radiation transmitted through the analyzer, hence the term GFC.

Radiation from an IR source is chopped and then passed through a gas filter alternating between CO and N_2 due to rotation of the filter wheel. The radiation then passes through a narrow bandpass interference filter and enters a multiple optical pass cell where absorption by the sample gas occurs. The IR radiation then exits the sample cell and falls on an IR detector.

The CO gas filter acts to produce a reference beam which cannot be further attenuated by CO in the sample cell. The N_2 side of the filter wheel is transparent to the IR radiation and therefore produces a measure beam which can be absorbed by CO in the cell. The chopped detector signal is modulated by the alternation between the two gas filters with an amplitude related to the concentration of CO in the sample cell. Other gases do not cause modulation of the detector signal since they absorb the reference and measure beams equally. Thus the GFC system responds specifically to CO.

With the improved rejection of interferences afforded by the GFC technique, it is possible to increase the sensitivity of the analyzer. This is achieved by the multiple pass optics (White Cell) used in the sample cell which leads to a large path length, and thus an improved sensitivity, in a small physical space. This allows full scale sensitivity down to 1 PPM with a lower detectable limit (LDL) of 0.1 PPM.

The analyzer used for this analysis meets the following specifications:

- Analyzer calibration error - Less than $\pm$ 2 percent of the span for zero, mid-range, and high-range calibration gases.
- Sampling system bias - Less than $\pm$ 5 percent of the span for the zero, and mid- or high-range calibration gases.
- Zero drift - Less than $\pm$ 3 percent of span over the period of each run.
- Calibration drift - Less than $\pm$ 3 percent of span over the period of each run.

I-3.7.2 Engineering-Science O_2 Monitoring System

The O_2 analyzer to be used is a Horiba Model PMA-200 O_2 analyzer that measures O_2 concentration using the paramagnetic dumbbell technique. The PMA-200 has full-scale range 0 to 25 percent O_2.

O_2 concentration will be determined using procedures outlined in EPA Method 3A. O_2 is detected by using Faraday's principle that comparatively measures the magnetic susceptibility of a gas volume by the force acting upon a nonmagnetic test body suspended in a disproportionate magnetic field. A glass dumbbell is horizontally suspended by a platinum ribbon in a strong, relatively disproportionate magnetic field. Because oxygen has a higher magnetic field susceptibility in the magnetic field compared to the other gases, the magnetic

force acts to reject the dumbbell from the magnetic field. As oxygen is introduced into the analyzer, a force manifests itself upon the platinum ribbon in the form of torque as the magnetic susceptibility of the gas volume changes. A magnetic feedback system is used to measure the magnitude of the torque applied and the torque is proportional to the amount of O_2 present in the sample.

The analyzer and sampling system used for this analysis meets the following specifications.

- Analyzer calibration error - Less than $\pm$ 2 percent of the span for zero and calibration gases.
- Sampling system bias - Less than $\pm$ 5 percent of the span for the zero and calibration gases.
- Zero drift - Less than $\pm$ 3 percent of span over the period of each run.
- Calibration drift - Less than $\pm$ 3 percent of span over the period of each run.

I-3.8 Summary of ES CEM Operating Procedures

Engineering-Science performs O2 and CO analyses according to the methods and procedures outlined EPA Reference Methods 3A and 10. Engineering-Science operating and calibration procedures are briefly summarized in this section. EPA Reference Methods 3A and 10 are provided in **Appendix E**. After the ES CEM measurement system is prepared for use, calibration gases are introduced directly into each instrument. The analyzers are calibrated to $\pm$ 2 percent of the instrument span for each standard introduced. A gas standard is then introduced into the sample system at the probe to conduct a system bias check. The system bias must not be greater than five percent of the instrument range.

After the sampling run, a zero and calibration drift check is performed by introducing a zero and a mid-range calibration gas into each analyzer. If either the zero or upscale value has drifted by more than 3 percent of the span, the run is considered invalid. In the event that an analyzer fails any of the above tests, the system is diagnosed to determine the source of the problem, corrective maintenance is performed, and the procedure is repeated. All measurements are corrected for bias and drift using appropriate equations as described in the reference methods.

After each analyzer is calibrated and system bias and leak checks are performed, sample gas is introduced into the instrument. Sampling runs can be of different lengths depending on the testing requirements. Analyzer responses to stack gas concentrations are processed with a digital data acquisition system which uses an analog to digital signal converter, a personal computer and a hard copy printer. Spread sheet files of all measurements and calibration results are also saved onto a hard disk.

I-4 REPORTING

Results of the PST will be reported in a document that summarizes the test results in tables. Summarized data will be presented for the calibration drift, calibration error, response time, and relative accuracy tests. Narrative will be provided to discuss the results and to present conclusions as to the performance of the CEM system being tested. Also included in the final report will be the field data sheets, calculations, CEMS data records, and cylinder gas certifications.

REFERENCES

1. Methods Manual for Compliance with the BIF Regulations, USEPA, EPA/530-SW-91-010, December 1990.

2. EPA Reference Methods 3A and 10, 40CFR Part 60 Appendix A.

APPENDIX J

Emissions Modeling

APPENDIX J

Emissions Modeling

J.1.0 INTRODUCTION

This appendix summarizes the results of screening modeling analysis, conducted for the deactivation furnace at Seneca Army Depot. Using the model USEPA SCREEN, an impact for a generic pollutant emitted to the atmosphere at the rate of 1 lb/hr was calculated. The calculated impact will be the basis for all allowable pollutant emission rates and munition feed rates. These analyses were conducted in accordance with the recommendations and guidance received from NYSDEC staff in telephone conversations. Analysis of land type and the identification of critical features was done after referring to Figure B-2, Area Map, from SEAD's RCRA Part B Permit Application. Figure B-2 which is located in map pocket one of that document locates the deactivation furnace and depicts area topography.

J.2.0 MODEL

SCREEN is a computer model which encompasses current techniques recommended by USEPA for estimating the air quality impacts of stationary sources. SCREEN is designed to provide a conservative estimate of the maximum impact on air quality due to an individual stationary source. More realistic (i.e., lower) estimates can be obtained by using more sophisticated air quality models with actual meteorological data. SCREEN is a Gaussian plume model which can be used to estimate pollutant impacts from continuous sources. It is most applicable to chemically stable gaseous or fine particulate pollutants. No pollutant transformation or removal mechanisms are considered.

SCREEN examines a wide range of meteorological conditions, as represented by various combinations of wind speed and stability class, to determine the conditions which yield the maximum predicted concentrations at specified receptors. SCREEN uses standard methods to calculate plume rise from point sources. Algorithms for stack tip downwash, buoyancy induced dispersion, and building wake and cavity effects are also included. SCREEN contains algorithms to estimate pollutant impacts in both simple terrain (below stack top) and in complex terrain (above stack top). SCREEN yields estimates of maximum 1-hour impacts in simple terrain and maximum 24-hour impacts in complex terrain.

The SCREEN model is described in the draft document "Screening Procedures for Estimating The Air Quality Impact of Stationary Sources" (EPA-450/4-88-010).

J.3.0 SOURCE DATA

J.3.1 STACK PARAMETERS

Most parameters necessary for analysis of the deactivation furnace stack were readily available. The actual flow rate and exit velocity were calculated from this information. Stack parameters used are summarized below:

Stack height	31 ft (9.45 m)
Stack diameter	20 in (0.508 m)
Stack Temperature	200 degrees F (366.5 K or 660 R)
Flow rate	3000 dscfm
Moisture	6% (by volume)
Stack base elevation	725 ft MSL

The actual flow rate was calculated by correcting the standard dry flow rate to actual temperature and moisture conditions:

$$\text{Actual flow} = 3000 \text{ scfm} \times (660/520) \times [1/(1-0.06)] = 4050.7 \text{ acfm}$$

The stack gas exit velocity was then calculated based on the actual flow rate. The resulting exit velocity of 30.95 ft/s (9.43 m/s) was used in the screening modeling analyses.

J.3.2 BUILDING DIMENSIONS

The deactivation furnace stack is below the Good Engineering Practice (GEP) formula height. Plumes emitted from stacks below GEP height are presumed to be subject to the effects of the added turbulence associated with building wakes and eddies generated by the interaction of wind flow with structures. Accordingly, the screening modeling analysis accounted for the potential for these building downwash effects on emissions.

An examination of available plot plans and photographs for the Seneca Army Depot indicates that the deactivation furnace is isolated from other activities at the base. Additionally, there is a small control building adjacent to a wall which houses system controls and partially encloses the deactivation furnace. The furnace is housed along with some ancillary equipment (e.g., furnace shroud, vent system, and afterburner) within four vertical walls but is not enclosed on top. The stack and some associated control equipment is adjacent to the area in which the furnace resides.

Although the furnace is not within an enclosed building, the surrounding walls and the fairly massive structures within the walls could be considered to represent enough of an obstruction to wind flow that aerodynamic wake effects could result. The structure represented by the deactivation furnace and other ancillary equipment within the surrounding walls is larger than the adjacent control building and would yield the greater GEP formula height. Accordingly, this structure was considered explicitly in the screening modeling analyses. Approximate dimensions, as determined from available plot plans, drawings, and photographs were:

Height	17 ft (5.18 m)
Width	26 ft (7.92 m)
Length:	50 ft (15.24 m)

J.3.3 OPERATING AND CONTROL ASSUMPTIONS

The deactivation furnace will operate no more than 2080 hours per year. This is consistent with operation for 8 hours each day for five days each week. The screening modeling analyses assumed that the furnace would be limited by its permit to operating no more than 2080 hours per year or 520 hours per calendar quarter. The analyses did not assume that there would be any limit on the number of hours of operation per day.

J.4.0 RECEPTOR DATA

Topographic maps were examined to select receptors at which impacts from the furnace would be predicted. Six receptors were selected to encompass receptor elevations both below and above stack top (756 ft MSL). One receptor was selected at the nearest property line, approximately 2700 ft from the furnace stack. This receptor had an elevation of 745 ft MSL. Next, the closest occurrences of terrain elevations of 750 ft MSL, 756 ft MSL, 760 ft MSL, 780 ft MSL, and 800 ft MSL were determined and used as receptors. The downwind distances of these additional receptors were 3100 ft, 3450 ft, 3600 ft, 16000 ft, and 16750 ft, respectively.

J.5.0 MODELING ANALYSES

J.5.1 MODELING APPROACH

The SCREEN model was used with sources and receptor data described in previous sections. A unit emission rate of 1.0 lb/hr (0.126 g/s) was used to predict a maximum 1-hour impact for a generic pollutant.

J.5.2 MODELING RESULTS FOR UNIT EMISSION RATE

The SCREEN model was run as described above. The maximum predicted impact assuming an emission rate of 1 lb/hr (0.126 g/s), of the source was 18.55 ug/m^3. This was predicted at a receptor located approximately 3450 ft (1052 m) downwind of the source with an elevation equal to stack top elevation of 756 ft MSL. This is 9.5 meters above the stack base elevation.

New York DEC modeling staff recommend the use of multiplicative factors of 0.4, 0.2, and 0.1 to obtain conservative estimates of maximum 24-hour, quarterly, and annual impacts from maximum predicted 1-hour impacts. These factors all assume continuous operation of the source. The DEC allows limitations on hours of operation to be factored into the estimate of maximum long-term impacts from short-term screening modeling results.

1 09-04-92
14:40:56

*** SCREEN-1.1 MODEL RUN ***
*** VERSION DATED 88300 ***

SENECA ARMY DEPOT -- DEACTIVATION FURNACE -- SCREENING MODELING

COMPLEX TERRAIN INPUTS:

SOURCE TYPE	= POINT
EMISSION RATE (G/S)	= .1260
STACK HT (M)	= 9.45
STACK DIAMETER (M)	= .51
STACK VELOCITY (M/S)	= 9.43
STACK GAS TEMP (K)	= 366.50
AMBIENT AIR TEMP (K)	= 293.00
RECEPTOR HEIGHT (M)	= .00
IOPT (1=URB,2=RUR)	= 2

BUOY. FLUX = 1.20 M**4/S**3; MOM. FLUX = 4.59 M**4/S**2.

FINAL STABLE PLUME HEIGHT (M) = 28.7
DISTANCE TO FINAL RISE (M) = 151.3

			VALLEY 24-HR CALCS		**SIMPLE TERRAIN 24-HR CALCS**				
TERR HT (M)	DIST (M)	MAX 24-HR CONC (UG/M**3)	CONC (UG/M**3)	PLUME HT ABOVE STK BASE (M)	CONC (UG/M**3)	PLUME HT ABOVE STK HGT (M)	SC	U10M (M/S)	USTK
10.	1052.	7.420	.7247	28.7	7.420	26.2	6	1.0	1.0
11.	1097.	7.389	.7678	28.7	7.389	26.2	6	1.0	1.0
17.	4877.	2.440	.1441	28.7	2.440	26.2	6	1.0	1.0
23.	5105.	2.322	.1376	28.7	2.322	26.2	6	1.0	1.0

1 09-04-92
14:40:56

*** SCREEN-1.1 MODEL RUN ***
*** VERSION DATED 88300 ***

SENECA ARMY DEPOT -- DEACTIVATION FURNACE -- SCREENING MODELING

SIMPLE TERRAIN INPUTS:

SOURCE TYPE	= POINT
EMISSION RATE (G/S)	= .1260
STACK HEIGHT (M)	= 9.45
STK INSIDE DIAM (M)	= .51
STK EXIT VELOCITY (M/S)	= 9.43
STK GAS EXIT TEMP (K)	= 366.50
AMBIENT AIR TEMP (K)	= 293.00
RECEPTOR HEIGHT (M)	= .00
IOPT (1=URB,2=RUR)	= 2
BUILDING HEIGHT (M)	= 5.18
MIN HORIZ BLDG DIM (M)	= 7.92
MAX HORIZ BLDG DIM (M)	= 15.24

BUOY. FLUX = 1.20 M**4/S**3; MOM. FLUX = 4.59 M**4/S**2.

*** FULL METEOROLOGY ***

****
*** SCREEN DISCRETE DISTANCES ***
****

*** TERRAIN HEIGHT OF 6. M ABOVE STACK BASE USED FOR FOLLOWING DISTANCES ***

DIST (M)	CONC (UG/M**3)	STAB	U10M (M/S)	USTK (M/S)	MIX HT (M)	PLUME HT (M)	SIGMA Y (M)	SIGMA Z (M)	DWASH
823.	15.19	4	1.0	1.0	320.0	27.9	57.5	28.3	NO

****
*** SCREEN DISCRETE DISTANCES ***
****

*** TERRAIN HEIGHT OF 8. M ABOVE STACK BASE USED FOR FOLLOWING DISTANCES ***

DIST (M)	CONC (UG/M**3)	STAB	U10M (M/S)	USTK (M/S)	MIX HT (M)	PLUME HT (M)	SIGMA Y (M)	SIGMA Z (M)	DWASH
945.	15.00	6	1.0	1.0	5000.0	28.0	33.0	15.4	NO

****
*** SCREEN DISCRETE DISTANCES ***
****

*** TERRAIN HEIGHT OF 9. M ABOVE STACK BASE USED FOR FOLLOWING DISTANCES ***

DIST (M)	CONC (UG/M**3)	STAB	U10M (M/S)	USTK (M/S)	MIX HT (M)	PLUME HT (M)	SIGMA Y (M)	SIGMA Z (M)	DWASH
1052.	18.55	6	1.0	1.0	5000.0	26.2	36.3	16.2	NO

****
*** SCREEN DISCRETE DISTANCES ***
****

*** TERRAIN HEIGHT OF 9. M ABOVE STACK BASE USED FOR FOLLOWING DISTANCES ***

DIST (M)	CONC (UG/M**3)	STAB	U10M (M/S)	USTK (M/S)	MIX HT (M)	PLUME HT (M)	SIGMA Y (M)	SIGMA Z (M)	DWASH
1097.	18.47	6	1.0	1.0	5000.0	26.2	37.6	16.6	NO

****
* SCREEN DISCRETE DISTANCES ***
****

*** TERRAIN HEIGHT OF 9. M ABOVE STACK BASE USED FOR FOLLOWING DISTANCES ***

DIST (M)	CONC (UG/M**3)	STAB	U10M (M/S)	USTK (M/S)	MIX HT (M)	PLUME HT (M)	SIGMA Y (M)	SIGMA Z (M)	DWASH
4877.	6.101	6	1.0	1.0	5000.0	26.2	142.6	34.6	NO

****
*** SCREEN DISCRETE DISTANCES ***
****

*** TERRAIN HEIGHT OF 9. M ABOVE STACK BASE USED FOR FOLLOWING DISTANCES ***

DIST (M)	CONC (UG/M**3)	STAB	U10M (M/S)	USTK (M/S)	MIX HT (M)	PLUME HT (M)	SIGMA Y (M)	SIGMA Z (M)	DWASH
5105.	5.804	6	1.0	1.0	5000.0	26.2	148.6	35.3	NO

DWASH= MEANS NO CALC MADE (CONC = 0.0)
DWASH=NO MEANS NO BUILDING DOWNWASH USED
DWASH=HS MEANS HUBER-SNYDER DOWNWASH USED
DWASH=SS MEANS SCHULMAN-SCIRE DOWNWASH USED
DWASH=NA MEANS DOWNWASH NOT APPLICABLE, X<3*LB

****
SUMMARY OF TERRAIN HEIGHTS ENTERED FOR *
SIMPLE ELEVATED TERRAIN PROCEDURE *
****

TERRAIN HT (M)	DISTANCE RANGE (M) MINIMUM	MAXIMUM
6.	823.	--
8.	945.	--
9.	1052.	--
9.	1097.	--
9.	4877.	--
9.	5105.	--

*** CAVITY CALCULATION - 1 ***

CONC (UG/M**3)	=	.0000
CRIT WS @10M (M/S)	=	99.99
CRIT WS @ HS (M/S)	=	99.99
DILUTION WS (M/S)	=	99.99
CAVITY HT (M)	=	6.32
CAVITY LENGTH (M)	=	13.81
ALONGWIND DIM (M)	=	7.92

*** CAVITY CALCULATION - 2 ***

CONC (UG/M**3)	=	.0000
CRIT WS @10M (M/S)	=	99.99
CRIT WS @ HS (M/S)	=	99.99
DILUTION WS (M/S)	=	99.99
CAVITY HT (M)	=	5.36
CAVITY LENGTH (M)	=	10.03
ALONGWIND DIM (M)	=	15.24

CAVITY CONC NOT CALCULATED FOR CRIT WS > 20.0 M/S. CONC SET = 0.0

****
*** SUMMARY OF SCREEN MODEL RESULTS ***
****

CALCULATION PROCEDURE	MAX CONC (UG/M**3)	DIST TO MAX (M)	TERRAIN HT (M)

SIMPLE TERRAIN	18.55	1052.	9.
COMPLEX TERRAIN	7.420	1052.	10. (24-HR CONC)

****
** REMEMBER TO INCLUDE BACKGROUND CONCENTRATIONS **
****

APPENDIX K

TOXICOLOGICAL REVIEW OF STRONTIUM COMPOUNDS

APPENDIX K

Toxicological Review of Strontium Compounds

K.1.0 INTRODUCTION

The following toxicological assessment supports the classification of strontium compounds as low toxicity contaminants, allowing the use of a de-minimis screening value of 1.0 ug/m^3. Further, this assessment indicates that ambient air levels of strontium compounds greater than 1.0 ug/m^3 are protective of human health and the environment. This assessment was prepared in consultation with Thomas Gentile, Acting Section Chief of Toxics Assessment, Bureau of Air Toxics, New York State Department of Environmental Protection.

New York State Air Guide 1 [REF 1] (AG-1) provides guidance which uses current toxicological information to set ambient Annual Guideline Concentrations (AGCs) and Short-term Guideline Concentrations (SGCs) for compounds emitted to the air. AG-1 also provides methods for setting interim AGCs and SGCs for compounds not yet evaluated by the State. These interim values can be calculated from recognized occupational exposure limits published by the National Institute for Occupational Safety and Health (NIOSH) [REF 2] or the American Conference of Governmental Industrial Hygienists (ACGIH) [REF 3] Of the compounds considered in this Trial Burn Plan, only the strontium compounds have no federal, state or occupational exposure guidelines from which SGCs and AGCs could be established.

For compounds which do not have established exposure guidelines AG-1 provides for the use of "screening de-minimis values". If a compound is known not to fit the definition of a high toxicity contamninant, a conservative annual ambient impact of 0.1 micrograms/cubic meter (ug/m^3) is used as the de-minimis AGC for the compound. If a compound is known to be classifiable as a low toxicity contaminant, a conservative annual ambient impact of 1.0 ug/m^3 is used as the de-minimis AGC for the compound.

Section K.2 briefly describes the thermal treatment process, the forms of strontium that will be treated, and the anticipated forms of strontium in the thermal treatment effluent. **Section K.3** of this document

describes the data collection process conducted for this review. **Section K.4** is a review of the chemical and physical properties of strontium and compounds. **Section K.5** is a review of the toxicological data obtained. **Section K.6** is a review of environmental exposure levels that have been developed in previous work and **Section K.7** follows these procedures to develop an AGC for Strontium.

K.2.0 DEACTIVATION FURNACE FEED MATERIALS

The munitions containing strontium that will be treated in the deactivation furnace are primarily flares and tracer bullets. Three compounds of strontium are contained in these munitions: strontium nitrate ($Sr(NO_3)_2$), strontium peroxide (SrO_2), and strontium oxalate (SrC_2O_4).

It is anticipated that the predominant form of strontium formed within the deactivation furnace will be strontium oxide (SrO). Strontium nitrates may be emitted unchanged or may be reformed within the furnace. The presence of chlorine compounds in the feed materials may result in the formation of strontium chloride ($SrCl_2$).

K.3.0 DATA COLLECTION

The primary sources of toxiciological information for this review are six online databases included in the USEPA Toxnet System [REF4]:

1. Registry of Toxic Effects of Chemical Substances(RTECS);
2. Hazardous Substance Data Base (HSDB);
3. Integrated Risk Information Service (IRIS);
4. Chemical Carcinogenesis Research Information Service (CCRIS);
5. Developmental and Reproductive Toxicology bibliographic database (DART);
6. Gene-Tox, a database of mutagenicity and other genetic toxicological studies.

Each of these databases was searched for compounds of strontium. In general, organic compounds of strontium have not been considered in this review, as it is anticipated that organic compounds of strontium will not survive thermal treatment.

K.4.0 PHYSICAL AND CHEMICAL CHARACTERISTICS

Strontium is a silvery-white alkaline earth metal of Periodic Group II-A and its chemistry is very similar to that of calcium and barium [REF 5,6,7]. Strontium is a reactive, electropositive metal and is easily oxidized to the stable Sr^{+2} in most of its compounds. Water soluble compounds of strontium are SrX_2, where X = Cl, Br, I, NO_2, NO_3, ClO_3, ClO_4, BrO_3, CN. Water insoluble salts are SrF_2, $SrSO_3$, $SrSO_4$, $SrCO_3$, $SrMoO_4$, $SrHPO_4$, $SrSeO_4$[7]. The carbonate, bromide and fluoride are the most stable compounds of strontium.

In nature, strontium is found primarily as strontium carbonate ($SrCO_3$) and strontium sulfate ($SrSO_4$). Estimates of the natural abundance of strontium in the earth's crust range from 300 to 500 ppm. Strontium is the fifteenth most abundant element in nature and is the most abundant trace element in sea water (7 to 8 milligrams per litre (mg/l)) [REF 7,8,9]. Fresh water concentrations of strontium range from 0.007 to 15 mg/l, averaging about 0.5 to 0.7 mg/l [REF 7,8]. Strontium concentrations in finished drinking water in the United States range from 2.2 to 1200 mg/l with an average of about 10 mg/l [REF 8].

Strontium is ubiquitous in the biosphere, being found in all plant and animal tissues. The body burden of strontium in an adult male is estimated at 140 to 320 mg, varying with geographic location [REF 7,8,9]. The skeleton contains 99% of this body burden, the remainder distributed in soft tissues, principally the aorta, larynx, trachea, and lower gastrointestinal tract [REF 8].

Strontium has several naturally occuring isotopes, the principal ones being 88 (82.56%), 86 (9.86%), 87 (7.02%), 84 (0.56%) [REF 5]. Strontium 90 is created in nuclear explosions and is not naturally occuring. The naturally occurring isotopes do not constitute a radiation hazard [REF 8].

K.5.0 TOXICOLOGICAL REVIEW

Strontium absorption from the digestive tract in test species is generally 5 to 25% of orally administered dose depending upon compound, species and other dietary constituents, principally calcium [REF 8,11]. In humans, about 36% of the orally administered dose is absorbed [REF 9]. Absorption and retention of strontium is dependent upon concurrent calcium intake. Calcium is preferentially absorbed and retained in the body. Strontium body burdens tend to match strontium intake, suggesting an homeostatic regulatory mechanism [REF 9].

Strontium salts are generally considered to be of low toxicity, with the magnitude of the LD50 in test species dependent on the cation species [REF 7,8]. In Clinical Toxicology of Commercial Products [REF 9] inorganic salts of strontium are described as "remarkably benign." The LD50 values for various test species, exposure routes and strontium compounds are shown on **Table K-1**. LD50 values for inorganic salts of strontium range from about 1800 mg/Kg to greater than 10,000 mg/Kg for the oral route across species and compounds. LD50's by intraperitoneal and intravenous injection are generally lower, reflecting the poor gastrointestinal absorption of strontium.

Chronic feeding and inhalation studies using strontium nitrate have demonstrated effects related to the strontium toxicity and mineral metabolism; increases in blood and urine calcium, and an increase in serum alkaline phosphatase. Other effects; histological changes in kidney, liver, lungs, and heart, and renal function changes, are likely related to nitrate/nitrite toxicity. One inhalation exposure study determined the threshold of strontium nitrate toxicity at 3.2 mg/m^3 [REF 12] Another inhaltion study was conducted at about 44.6 +/- 1.4 mg/m^3 [REF 13]. In neither study was any lethality produced.

Chronic feeding studies of young test species with strontium nitrate produced severe skeletal deformities, though these effects are produced at very high levels: 0.2% strontium in the diet [REF 8], or at high strontium:calcium ratios (3:1) [REF 11]. In 90 day feeding studies at doses of 75, 300, 1200, 4800 mg/Kg of diet, minor changes in hematology and blood chemistry were noted at the highest dose. Liver glycogen was decreased in female rats at the highest dose and thyroid weights were increased in male rats at the two high doses [REF 8]. An 8 week feeding study of adult and weanling male rats found no signs of toxicity at levels in the diet up to 1000 mg/Kg [REF 13].

Irritation of the skin and conjunctival membrane is reported in an irritation study of topically applied strontium nitrate [REF 10]. However, no irritation was reported at the levels used in the inhalation or chronic feeding studies discussed above.

Teratological studies have been conducted on non-radioactive strontium, though most studies cited in the DART database employed strontium-90 in absorption and distribution studies or studies of devlopmental effects of the radioactive isotope [REF 11]. Strontium passes freely across the placenta and is found in embryos and newborns of test species [REF 9]. A study of non-radioactive strontium conducted at several maternal doses of strontium nitrate up to 200 mg/Kg/d showed no differences in size or body weight of progeny and no difference in litter sizes or number of resorption sites [REF 12].

TABLE K-1
ACUTE TOXICITY VALUES FOR COMPOUNDS OF STRONTIUM (MG/KG)

TEST ORGANISM	ROUTE OF ADMIN.	TOXICITY END POINT	STRONTIUM NITRATE	STRONTIUM CHLORIDE	STRONTIUM CHLORIDE HEAXAHYDRATE	STRONTIUM BROMIDE	STRONTIUM FLUORIDE	STRONTIUM IODIDE	STRONTIUM IODIDE HEXA-HYDRATE	STRONTIUM HYDROXIDE	STRONTIUM PEROXIDE	STRONTIUM OXIDE
Rat	Oral	LD50	2750	2250			10600					
Rat	IP	LD50	540	405		1000		800				
Rat	IV	LDlo		222		500	625		625			
Mouse	Oral IP IV Oral	LD50 LD50 DL50 LDlo	1826	1874 1643 148	1253 405		4400					
Rabbit	Oral IV	LD50 LDlo	3865	2735 1067								
Guinea Pig	Oral Oral SC	LD50 LDlo LDlo	7432	5143			>5000 >5000					
Mammal	Oral	LD50								>1500		

The Genetox database references one study which found no cell transformation in Syrian Hamster embryo cells exposed to strontium chloride[13]. The RTECs database cites a study using Saccharomyces cerevisiae which found no genetic effects from exposure to strontium chloride [REF 14]. The CCRIS database lists one study of the carcinogenicity of strontium chromate [REF 15]. The study results were positive, but the likely etiological agent in cancer formation is the chromate ion, not the strontium.

Human exposure

A described above, strontium is ubiquitous in the environment. Average daily human intake is estimated at 1.8 to 2.0 mg/day. Intake in humans is in balance with the amount secreted. Approximately 60% to 90% of the exposure is from food, with the remaining 10% to 40% from water. A negligible quantity is supplied by air [REF 8].

The National Occupational Exposure Survey [REF 16] indicates that upwards of 37,000 employees are exposed to strontium compounds in the work place. Interestingly, for strontium nitrate and strontium peroxide, a large majority of the reported exposed employees are female (1895 of 2667 and 31012 of 37145, respectively). No cases of occupational strontium intoxication have been reported [REF 17].

Strontium compounds have been used medicinally in the past and is still employed in food preparation [REF 5,11]. Strontium bromide ($SrBr_2$) was used as an anticonvulsant until 1960 and strontium fluoride was used in toothpastes. Strontium carbonate and strontium hydroxide are used in refining of beet sugar [REF 5].

Environmental Exposure levels

There are a number of points of departure for determining an acceptable level of environmental exposure for strontium. The National Research Council developed 24-hour and 7-day Suggested No Adverse Response Levels (SNARLs) based primarily on a No Observed Effect Level(NOEL) determined in a 90 rat feeding study [REF 8]. A 1974 study by Dawson [REF 18] suggested an acceptable strontium level in drinking water of 10 mg/l based on LD50 data, but evaluated this estimate as having the "lowest level of reliability." An acceptable level of exposure could also be developed as a percentage of the daily strontium intake of strontium.

Environmental exposure levels for air derived from these sources are shown on **Table K.2**. All calculations are based on a 70 Kilogram adult drinking 2 liters of water and breathing 20 cubic meters of air per day.

The calculated air concentrations based on the 7 day SNARL and on the suggested drinking water level of 10 mg/l result in similar values: 0.85 mg/m^3 and 1.0 mg/m^3.

The air concentration based on the average daily intake results in a much lower level of 0.1 mg/m^3. However, the basis of this estimate, 2 mg/d average intake, does not correlate with the reported average finished drinking water level of 10 mg/l. If the average adult drinks 2 litres of water per day, and the average strontium concentration in finished drinking water is 10 mg/l, the average intake of strontium should be at least 20 mg/d, in addition to the amounts of strontium contributed by food. If the reported 2 mg/d intake is intended to be the daily absorbed dose, indicating a daily intake from three to ten times higher, these calculated values are more in line. However, taken at face value, the average daily intake indicates that 0.1 mg/m^3 is an acceptable air concentration for strontium.

Each of the above derived values need to be adjusted for uncertainty. The SNARL is based on a subchronic study and extrapolation to a longer-term exposure introduces a degree of uncertainty. The Dawson drinking water estimate is stated to be very uncertain by the author. The daily strontium intake value, while it already appears conservative, represents contributions almost exclusively from water and food and should be adjusted to provide for a lesser incremental exposure above the normal intake.

Applying a 10 fold safety factor to each of these calculated environmental levels provides for a conservative value that will be protective of human health and the enviornment. The lowest of the calculated environmental values, 0.01 mg/m^3, represents a 10% increase in the average strontium exposure, if exposure is constant. However, the deactivation furnace will be permitted to operate 2080 hours per year, less than 25% of the time. Actual maximum impact would thus be less than a 2.5% incremental exposure over the average.

A 2.5% incremental increase in strontium exposure is not likely to produce adverse effetcs in the exposed populations. The preceding review has shown that the range of concentrations that have been recorded in surface waters and drinking waters span a much greater range than 2.5% above average; the range of human body burdens, which are under homeostatic control, also span a much greater range than this, all without apparent adverse effect on human populations. Further, this exposure level is approximately 10

TABLE K-2
ESTIMATION OF ALLOWABLE
STRONTIUM AMBIENT AIR CONCENTRATIONS

SOURCE OF STANDARD OR BASIS	STANDARD OR BASIS (MG/L)	ESTIMATED RfD (MG/KG/DAY)	ESTIMATED NO ADVERSE AFFECT AMBIENT CONCENTRATION 24-HR-AVERAGE (ug/m^3)	ESTIMATED AGC UG/M^3 ANNUAL AVERAGE
Concentration cited in drinking water by the National Research Council[8]	10	0.29	1000	250
Suggested No Adverse Response Level (SNARL) (7-day)	8.40	0.84	840	210
Daily human intake[8] through drinking water	2.0 (mg/day)	0.03	100	25
Estimated No Observed Effect Level (NOEL) for Humans	0.24 (mg/kg)	0.24	840	21

Note: Calculations were based on a 70 KG adult consuming:
(1) 2 liters of drinking water per day, and
(2) 20 $meter^3$ of air per day

times less than the other environmental levels, derived by standard extrapolation methods, based on animal experimental results.

K.6.0 CONCLUSION

Strontium and its inorganic compounds meet the requirements for a low toxicity air contaminant and the deminimis screening value of 1 ug/m^3 can be used in determining potential impacts. Oral LD50 values in all species studied are greater than 500 mg/Kg; no evidence of mutagenic or carcinogenic activity has been found; and irritation is not likely to occur at anticipated exposure levels.

Further, the deminmis screening value is conservative for strontium. The derived environmental exposure levels indicate that ambient air concentrations 10 to 100 times the deminimis value are still protective of the environment and human health.

K.7.0 REFERENCES

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APPENDIX L

Kiln Operational Data

TABLE 3-4. Operating Parameters with Normal Input Conveyor

ITEM	TEMPERATURE RECORDER SET POINT	RETORT SPEED (RPM)	ITEMS/ CONVEYOR SECTION	ITEMS/ HOUR
Cartridge, Impulse				
MK12 Mod 1	250°F	2.5	17	22,500
MK12 Mod 2 (NM)	250°F	2.5	17	22,500
MK12 Mod 1 (NM)	250°F	2.5	17	22,500
For Cutter MK15 Mod 0, MK14 Mod 0	250°F	2.5	34	45,000
Cartridge, Tracer				
.30 cal	350°F	1.5	17	22,500
7.62mm, M62	400°F	1.5	17	22,500
.50 Cal	450°F	1.8	5	6,600
Cartridge, 20mm				
HEI	400°F	1.8	2 every 3rd section	900
HEI-T	400°F	1.8	2 every 3rd section	900
HE-T	400°F	1.8	2 every 3rd section	900
INC	400°F	1.8	2 every 3rd section	900
API	400°F	1.8	1 per section, skip 10th section	1,200
TP	400°F	1.8	1 per section, skip 10th section	1,200
Fuzes				
BD, M66A1	300°F	1.2	1 every 2nd section	600
BD, M66A2	400°F	1.2	1 every 2nd section	660
MTSQ, M501, M501A1	400°F	1.7	1 every 2nd section	660

TABLE 3-4. Operating Parameters with Normal Input Conveyor (Cont'd)

ITEM	TEMPERATURE RECORDER SET POINT	RETORT SPEED (RPM)	ITEMS/ CONVEYOR SECTION	ITEMS/ HOUR
MTSQ, M502 w/M21A4 Booster	~~300~~°F 450	~~1.7~~ 1.0	1 every 3rd section	440
PD, M557 w/M125A1 Booster	400°F	1.7	1 every 3rd section	440
PD, MK27, Mod 0	400°F	1.5	1 every 2nd section	660
Booster				
M21A4 - Assembled	400°F	1.5	1 every 3rd section.	440
M21A4 - Disassembled	300°F	2.1	2	2,640
Cartridge, AP				
7.62mm	400°F	1.5	17	22,500
.30 cal	350°F	1.5	17	22,500
.50 cal	450°F	1.8	7	8,000
Cartridge, Ball				
.22 cal	400°F	1.5	17	22,500
5.56mm	400°F	1.5	17	22,500
.38 cal	400°F	1.5	17	22,500
.45 cal	400°F	1.5	17	22,500
.30 cal	350°F	1.5	17	22,500
7.62mm	400°F	1.5	17	22,500
.60 cal	450°F	1.8	7	8,000
Cartridge, Blank				
5.56mm	400°F	1.5	17	22,500
7.62mm	400°F	1.5	17	22,500

TABLE 3-4. Operating Parameters with Normal Input Conveyor (Cont'd)

ITEM	TEMPERATURE RECORDER SET POINT	RETORT SPEED (RPM)	ITEMS/ CONVEYOR SECTION	ITEMS/ HOUR
.30 cal	400°F	1.5	17	22,500
.38 cal	400°F	1.5	17	22,500
.45 cal	400°F	1.5	17	22,500
.50 cal	450°F	1.8	6	8,000
Cartridge, Grenade				
5.56mm	400°F	1.5	17	22,500
.30 cal	400°F	1.5	17	22,500
7.62mm	400°F	1.5	17	22,500
Cartridge, Incendiary				
.30 cal	400°F	1.5	17	22,500
Cartridge, Ingition M66 for 60 & 81mm	400°F	1.5	9	11,880
Cartridge, Bomb-ejection CCU-1/B	400°F	2.5	5	6,600
Primers				
M28B2	350°F	1.4	2	2,640
M34	400°F	1.5	17	22,500
M40A2	350°F	1.5	2 every 2nd section	1,320
M57	350°F	1.5	1 every 2nd section	660
M71, M71A1E1	400°F	1.8	17	22,500
M82	400°F	1.5	17	22,500
Projectiles				
76mm HVTP-T,M315A1	400°F	1	1 every 2nd section	660
76mm AP-T, M339	400°F	1	1 every 4th section	330

TABLE 3-4. Operating Parameters with Input conveyor (Cont'd)

ITEM	TEMPERATURE RECORDER SET POINT	RETORT SPEED (RPM)	ITEMS/ CONVEYOR SECTION	ITEMS/ HOUR
40MM empty or AP-T	400°F	1.5	2	2,640
Miscellaneous				
Motor, Rocket, 3.5" Rocket (w/ Fuze removed and fed simultaneously w/motor).	400°F	1	1 every 6th section	240
Cutter, Cartridge Actuated, M2 Series	350°F	3.0	1	1,320
Cutter, M22	350°F	1.5	2	2,640
Detent, for CBU 9A/A 1325-00-209-6251	400°F	1.5	17	22,500
40MM Grenade, M385 Pract	400°F	1.5	1 every 2nd section	660
Fin Assy, w/Primer 81mm Mortar	300°F	2.0	2 (end to end)	2,640
Metal, Scrap (requiring flashing)	400°F	1	N.A.	2000 lb
Igniters, rocket Motor				
MK117, MK118	350°F	1.2	2 per section, skip 10th section (38 per min)	2,280
MK125-5	300°F	2.7	5	6,600
1340-00-862-3228	300°F	2.0	1 every 2nd section	660
Cartridge, 40MM				
M407	400°F	1.5	1 every 2nd section	660
Element, Ignition, Electric 1377-00-007-4880	300°F	2.0	1 every 2nd section	660

Appendix M

Tier I/Tier II Analysis

The following is the Tier I/Tier II Analysis of the Deactivation Furnace located at the Seneca Army Depot (SEAD). The analysis follows the steps described in Volume IV of the Hazardous Waste Incineration Guidance Series, "Guidance on Metals and Hydrogen Chloride Controls for Hazardous Waste Incinerators" published by EPA. Attached to this Appendix is worksheet 1 from Volume IV. Analysis of land type (complex, non-complex) and usage (rural, urban) in the area surrounding the deactivation furnace was done after referring to Figure B-2, Area Map, from SEAD's RCRA Part B Permit Application and the 7.5 minute USGS topo maps which depict the area around SEAD. Figure B-2 which is located in map pocket three of Appendix O, shows the location of the deactivation furnace and depicts area topography. USGS maps of the area are included in map pocket 4 of Appendix P.

Tab - A. Determine land use characteristics within 3Km of the stack using the AUER method provided in Appendix I of Volume IV.

Step 1. Fill out worksheet 1 in Appendix I of Volume IV (see attached) for stack, terrain, building parameters and feedrates.

Step 2. The area within the 3Km radius was inspected via topo map. Since less than 30% of the total area was urban the area is designated as rural.

Step. 3. Determine suitability of Tier I/Tier II screening tables

Is the facility located in valley < 1Km wide?	no
Does the facility have a stack > 20m and does terrain rise to the stack height within 1Km?	no
Does the facility have a stack > 20m and is it located within 5Km of shoreline or a large lake?	no
Is the physical stack height < 2.5 x building height and is distance from the stack to the closest boundary < 5 building heights?	no

Since the answer to all of the above questions is no, the Tier I/Tier II tables can be used.

Tab - B. Determine feedrates or emission limits (Tier I and Tier II)

Step 1. Determine Worst-Case Stack for Multiple Stack Sites:

Incinerator has only one stack go to Step 2.

Step 2. Define Terrain:

Does terrain rise greater than physical stack height within 5Km?

Yes - terrain rises 13.75M within 5Km of stack while physical stack height is 9.5M. Therefore by definition the terrain is complex for this analysis. (See attached worksheet 1).

Step 3. Determine Terrain - Adjusted Effective Stack Height

GEP Stack Height = H + 1.5L
H protective walls = 17
L = 17

Step 3A. GEP = 17 + 1.5 (17) = 42.5 feet

Since the stack is 31.0 feet tall and is less than GEP, the method recommends that a 4M stack height be used as the terrain adjusted stack height, and this is defined as the worst case stack for subsequent analysis.

Go to step 4A.

Step 4A. Determine compliance with Tier I Feed Rate Limits

A. For non-carcinogens read from:

Table B-3, For metals in complex terrain
Table B-10 for HCl

B. Compare proposed total pollutant feedrates with Tier I Limits:

For A 4M adjusted stack height and
For complex terrain

This information is presented in Table M-1.

Table M-1

Pollutant	Tier I Feed Limits lb/hr	Proposed Feed lb/hr	Tier II Emission Limits g/sec	Proposed Emission g/sec
Antimony	0.031	13.6	3.9×10^{-3}	1.72
Barium	5.2	45.0	0.66	5.68
Lead	0.0094	20.4	1.2×10^{-3}	2.57
Chlorine	0.26	3.0	0.033	0.38

Since the limits for each pollutant were exceeded: Go to Tab B - Step 4B.

Step 4B. Determine Compliance with Tier II Emission Limits

A. Using the following tables read Tier II Emission Limits from:

Table B-7 for metals in complex terrain
Table B-11 for HCl

B. Proposed emissions were calculated assuming no credit for partitoning to ash bottoms or APC removal. This information is presented in Table M-1.

Since all emission limits were exceeded, go to Tab C.

Tab - C. Site Specific Modeling and Risk Analysis (Tier III)

Tab C presents methods to determine, under Tier III, if the aggregate cancer risk to the Most Exposed Individual (MEI) resulting from the metals emissions is less than or equal to 10^{-5}, and if the ambient concentrations of non-carcinogenic metals and HCl are below the reference air concentrations (RACs). For some facilities, emission limits under Tier III can be a factor of 10 or more higher than those under Tier II Screening Limits. Within Tier III, the permit writer has the option of (a) performing an in-house dispersion analysis or (b) requiring the applicant to perform detailed site-specific modeling. (The preceding was abstracted almost verbatim from Volume IV's Tab C).

At this point in the analysis SEAD opted to perform site specific analysis and to take into account metals partitioning to ash bottoms and metals removal in the deactivation furnace's air pollution control equipment. The goal of this modeling was to demonstrate that impacts to the ambient air would be less than allowed by Federal and State standards and guidance for both carcinogen and non-carcinogenic metals and HCl. The site specific analysis performed is described in detail in Section 4 of this report.

Appendix M

WORKSHEET 1
Date Requirements
(if facility has more than five units, attach additional sheets)

I. REFERENCE INFORMATION

A. Facility Name SENECA ARMY DEPOT
B. Address ROMULUS, New York 14541-5001
C. Phone Number 607·869·1450
D. Date of Submission 12·1·92

II. SITE INFORMATION

Incinerator Stacks

A. Stack Parameters	1	2	3	4	5
1. Stack Height (meters)	11.0				
2. Exhaust Temperature (Kelvin)	366				
3. Inner Stack Diameter (meters)	0.51				
4. Exit Velocity (m/second)	9.42				
5. Flow Rate (cubic m/sec)	1.91				
6. Latitude	76°50'59"				
Longitude	42°43'43"				
UTM (easting)(km)	348.9				
UTM (northing) (km)	4733.3				
B. Terrain Parameters					
1. Maximum Terrain Rise(meters)					
0 – 0.5 km radius	13.75				
0 – 2.5 km radius	13.75				
0 – 5.0 km radius	13.75				
2. Shortest distance to fenceline	825 M				
*C. Dimensions of Tallest Buildings**					
1. Distance from Stack (meters)	0				
2. Distance from nearest fenceline (meters)	825				
3. Building height (meters)	5.2				
4. Building length (meters)	15.2				
5. Building width (meters)	9.9				

*Consider buildings within 5 building heights or 5 maximum projected widths of the stack.

III. REQUESTED MAXIMUM

Appendix M

WORKSHEET 1
Data Requirements
(If facility has more than five units, attach additional sheets)

FEED RATES
If waste blending is performed, the data provided in this section should be after blending. These feed rates will be written into the permit provided they pass the risk screening

The applicant must also submit copies of any supporting documentation of the waste feed rate calculations for each one of the feed (burner) systems

A. Maximum Feed Rates by Feed System

	Feed System 1	Feed System 2	Feed System 3	Feed System 4	Total for all Feed Systems
Antimony	13.6 lb/hr				13.6 lb/hr
Arsenic	NA				NA
Barium	45.0				46.0
Beryllium	NA				NA
Cadmium	NA				NA
Chromium	0.09				0.09
Lead	20.4				20.4
Mercury	NA				NA
Silver	NA				NA
Thallium	NA				NA
Chlorine	3.0				3.0

Appendix IV-5

APPENDIX N

Particulate Fugitive Emissions Monitoring Plan

Appendix N

Particulate Fugitive Emission Monitoring Plan

N.1.0 INTRODUCTION

During the trial burn ambient particulate concentrations will be measured. The goal of this work will be to demonstrate that no uncontrolled particulate (fugitive) emissions are generated. Sampling stations will be set up at the point where uncontrolled particulate emissions are most likely to occur, and at an upwind location representative of background. The resulting data will be included in the Trial Burn Report.

N.2.0 SAMPLING LOCATIONS

An ambient particulate monitoring station will be established at the point at which uncontrolled particulate emissions are most likely to occur. SEAD believes that this point is located where the discharge conveyor exits the kiln shroud. The approximate location can be seen in photographs located in Volume 1, Appendix 1, Map Pocket 7, of SEAD's 6NYCRR Part 373 Permit Application. SEAD will establish the monitoring station within 20 feet of this point. The exact location will be determined in the field and will be chosen so as to insure the integrity of the instrument, to insure reliable readings, to allow necessary access for reading and maintenance, and to take into account the prevailing wind direction. A second ambient particulate monitoring station will be established at an upwind location representative of background. The background monitoring station will be located outside the kiln wall and will be placed upwind of the furnace discharge. It will be located 20 feet from the kiln wall or the control building.

N.3.0 INSTRUMENT DESCRIPTION

The instrument which will be used to measure ambient particulate matter concentration will be a MINIRAM (Minature Real-time Aerosol Monitor) personal monitor model PDM-3 or equivalent. The MINIRAM is manufactured by Monitoring Instruments for the Environment, Inc. It is a compact particulate monitor whose operating principle is based on the detection of scattered electromagnetic radiation in the near infrared region of the spectrum. The MINIRAM detects both aerosols and particulate matter.

The MINIRAM preferentially detects particles in the 0.1 to 10 micron range (respirable or inhalable size). Air surrounding the instrument passes freely through the sensing chanber, requiring no pump

for operation. Every 10 seconds it records an average which is taken over the 10 second period. The information can be monitored continuously by a personal computer or can be recovered after the monitoring run. The instrument can also calculate a time weighted average for the run on a continuous basis. Measurements are in mg/m3. Concentrations are normally recorded in the 0-9.99 mg/m^3 range. When the value exceeds 9.99 mg/m3 the instrument automatically switches to the 0-99.9 range.

Before the instruments are run in the field they will be calibrated as described in MEI's operations manual and presented below. To insure accurate measurement in the field the instrument will be zeroed prior to each monitoring run. This will be accomplished by using the procedures described in MIE's operations manual and presented below.

N.4.0 AMBIENT PARTICULATE MONITORING PROTOCOL

As described previously two ambient particulate monitoring stations will be established in the vicinity of the kiln. The location of the stations will be determined with measuring tape (i.e. the distance from two benchmarks to each station will be recorded). The monitors will be set 4 to 6 feet above the ground.

The instruments will have been calibrated and zeroed as described in Sections N.5 and N.6 prior to their placement in the field. In the field the instruments will be zeroed daily. The instruments will be turned on at least 10 minutes before waste is fed to the deactivation furnace. Monitoring will continue until burning has ceased for the day. The instruments will be run off AC power, unless the location of the monitors prohibits the introduction of electrical chords. If this is the case the monitors will run off batteries. Extra batteries, a recharger and an extra monitor will be available during the trial burn.

Each monitor will be either hooked to a personal computer which will display and record each 10 second measurement or the data will be dumped from the instrument to the computer at the end of each day of monitoring. The time weighted average of ambient particulate matter concentration will be determined for each day. The daily time weighted average obtained from the instrument located near the junction of the discharge conveyor and kiln shroud will be compared to that obtained at the background monitoring station. If a significant difference between the time weighted averages between the two stations is not seen then it can be concluded that fugitive particulate emissions asociated with the deactivation furnace are negligible.

N.5.0 CALIBRATION OF THE INSTRUMENT

Although every MINIRAM has been factory-calibrated using a representative dust, the user may wish to change the calibration constant of the instrument for a specific type of aerosol. Such a calibration should be performed by obtaining a concurrent filter collection (e.g., by means of personal filter sampler), sampling from the same environment within which the MINIRAM is placed. The average concentration obtained by the MINIRAM (i.e. TWA reading) at the end of the test should be compared with the filter-gravimetric-determined concentration. The ratio of the two concentration values can then be used to correct the MINIRAM calibration. The comparison run should be replicated several times (to minimize errors) to obtain an average ratio.

To change the MINIRAM calibration proceed as follows:

5.1 Place MINIRAM in a Clean Environment (e.g. air conditioned office)

5.2 Remove batter pack

5.3 Disconnect battery connector (remember that all stored data will thus be lost/erased from MINIRAM memory).

5.4 While leaving battery pack lying next to MINIRAM, re-connect the two units (i.e. plug in connector).

5.5 Immediately observe MINIRAM display. It will be performing a slow segment-by-segment display checkout. As soon as it displays ".00", pres OFF, thus interrupting the initial automatic zero check. Wait until the display indicates "OFF" and then press MEAS and wait approximately 36 seconds.

5.6 Observe 10-second readings (typically in the range of 1 to 3 mg/m^3) and record manually a few consecutive readings. Calculate the average of these values.

5.7 Identify small potentiometer screw (visible through an opening in the foil shield of the open MINIRAM) opposite the digital output jack. Adjust this potentiometer, using a fine screw driver, until the average MINIRAM reading is increased or decreased by the desired ratio (e.g. as determined by previous gravimetric comparison runs).

5.8 Shut off MINIRAM, reposition and secure battery pack, and re-zero instrument as usual. All subsequent concentration readings are now corrected by the desired ratio.

If an optional Reference Scatterer (MIE model PDM-RS) is available, insert it in the MINIRAM instead of the normal sensing chamber and follow the same procedure (i.e., follow steps 5.1 through 5.8).

N.6.0 ZEROING OF THE INSTRUMENT

The interior walls of the MINIRAM sampling chamber reflect a small amount of the light from the infrared source into the detector. This background level is referred to as the "zero value," and it automatically subtracted from all aerosol concentration readings during the measurement mode. The result is that the displayed readings depend only on the actual dust concentration present within the sensing chamber.

The zero value varies from instrument to instrument as well as with different sensing chambers. It will increase somewhat as the chamber inner walls and windows become contaminated with dust. A zero update should be performed after cleaning the sensing chamber.

Pressing ZERO during a measurement period provides momentary display of the stored zero concentration value used by the MINIRAM to correct all digital concentration readings (the analog output signal is not zero-corrected). To update the ZERO value the MINIRAM must be in its off condition (press OFF in case of doubt). Then, press ZERO and wait until the display again indicates "OFF."

The average of 4 consecutive 10-second zero level measurements will then be stored by the MINIRAM as the new ZERO reference value. When operating the MINIRAM in high particle concentration environments (>5 mg/m^3) the zero value update should be performed approximately every 8 hours. At aerosol concentrations below approximately 1 mg/m^3 this update may only be required once a week, or even less frequently. The zero update should be performed either within a clean-air environment (ideally, a clean room or cleanbench) or using one of the accessories provided by MIE for that purpose: either the Z-Bag (standard accessory), or the Zero Check Module PDM-1FZ (optional accessory). The latter should be used for zeroing only when subsequent measurements are at concentrations greater than approximately 0.5 mg/m^3, or if the PDM-1FZ is left on the MINIRAM for active air sampling. Air conditioned offices (without smokers) usually have concentrations below approximately 0.05 mg/m^3 and can thus be used for zeroing purposes, if a Z-Bag is not available. When measurements are performed under essentially clean air conditions, e.g., in the same environment where the zero check was performed, the MINIRAM readings will indicate 0.00 mg/m^3 with small random fluctuations around

that value. Positive values (e.g., 0.02) will thus be indicated on the LCD display. Negative values (e.g.,-0.02) are suppressed and are also indicated as 0.00. The digital output, however, does include such negative values and these will be printed out by a digital printer.

APPENDIX O

POHC Spiking Procedure

In order to insure that the deactivation furnace has been tested under "worst-case" conditions, POHCs which are at least as difficult as the waste to be fed will be used in the trial burn. According to the Dayton ranking system DPA is the most difficult component of munition PEP to destroy. Under normal circumstances that would make DPA an ideal POHC for the trial burn. However, there are problems associated with the capture of DPA from stack gas in the presence of NO_x. This would make it difficult to determine whether a DRE of 99.99% had been achieved during the trial burn. Because there are no other components present in the waste feed which are contained in the same incinerability class (i.e., are as hard or harder to destroy), a constituent with another substance, must be fed (spiked) into the furnace which is at least as difficult to destroy based on the Dayton Ranking System as DPA.

DPA is listed as a Class II substance by the Dayton Ranking System. To demonstrate the DRE for DPA a substitute POHC would be required to be either a Class I or II compound. SEAD has selected both a Class I compound (Hexachlorobenzene (HCB)) and a Class II compound (Trichloroethylene (TCE)).

1. Prior to the trial burn POHCs chosen for spiking will be analyzed. The purity of the material will be determined and the major contaminants will be identified. If the supplier has analysis, this may be acceptable.

2. The required amount of materials are as follows:

Compound	No. of Tests	Duration of Test (hr)	lb/hr	Total lb
HCB	5	1	4.11	21
TCE	5	.33	4.11	7

Note feed rate calculations are presented in **Appendix D-3**.

3A. For HCB, which is a solid, portions will be weighed on a balance to an accuracy $\pm$ 0.1 g on weighing paper. Each portion will be numbered and its weight recorded. Once weighing of a portion is complete, the HCB will be wrapped or rolled up in its weighing paper. The packages will be rolled to approximately the same size as propellant "pencils" which are found in 3.5" rocket motors. This package will be secured with tape. Each weight will be recorded and each package numbered (see **Table O-1**). The HCB packages will then be stored in a cool dry place until munitions experts are ready to place (spike) them into rocket motors for the trial burn.

For TCE, which is a liquid, measured volumes with an accuracy ± 0.1 ml will be transferred to Polyethylene "pencils". These pencils will be approximately the same size as the propellant pencils which are found in 3.5" rocket motors. (Note that after the trial burn is complete rocket motors will be examined to ensure that the pencils where completely incinerated during the test). The pencils will be weighed on a balance to an accuracy of ± 0.1g prior to and after the TCE has transferred. The weight of TCE added will then be back calculated. Each pencil will be numbered and the weight of TCE recorded (see **Table O-2**). The TCE pencils will then be stored in a cool dry place until munitions experts are ready to place (spike) them into rocket motors for the trial burn.

4. Munitions experts will partially disassemble rocket motors, by removing individual "pencils" of propellant and will replace the "pencil" with a single POHC package. The resulting POHC spiked rocket motors will be fed to the incinerators at a rate of 4.11 lb/hr for HCB and 4.11 lb/hr for TCE.

5. Spiked rocket motors will be handled, disassembled, and reassembled by munitions experts. They will be stored according to Army requirements until they are needed for the trial burn.

6. Spiked rocket motors will be fed through the automatic waste feed measuring system. The kiln operating parameters will all be set for the destruction of rocket motors.

   Kiln Temperature: 400°F
   Retort Speed: 1 RPM

The after burner temperature will be maintained at a setting of 1600°F.

TABLE O-1
HCB SPIKING PROCEDURE

PENCIL NUMBER	WEIGHT OF PAPER	WEIGHT OF PAPER AND HCB	CALCULATED WEIGHT OF HCB

TABLE O-2
TCE SPIKING PROCEDURE

PENCIL NUMBER	WEIGHT OF PENCIL	WEIGHT OF PENCIL AND TCE	CALCULATED WEIGHT OF TCE

APPENDIX P

MAP POCKETS

CONTENTS OF APPENDIX P

MAP POCKET	FIGURE/DRAWINGNO.	DESCRIPTION
1		Cyclone
2	N741382	Baghouse
3		Area Map
4		7.5 minute Quadrange Maps for: 1. Geneva South, NY 2. Romulus, NY 3. Dresden, NY 4. Ovid, NY
5		Thermocouple and Fly Ash Sampling Locations