Note: This is a reference cited in *AP 42, Compilation of Air Pollutant Emission Factors, Volume I Stationary Point and Area Sources.* AP42 is located on the EPA web site at www.epa.gov/ttn/chief/ap42/

The file name refers to the reference number, the AP42 chapter and section. The file name "ref02_c01s02.pdf" would mean the reference is from AP42 chapter 1 section 2. The reference maybe from a previous version of the section and no longer cited. The primary source should always be checked.

AP42 Section:	1.1
Reference:	34
Title:	Results of the March 28, 1990 Dioxin Emission Performance Test on Unit 3 at the NSP Sherco Plant in Becker, Minnesota. Interpoll Laboratories, Inc., Circle Pines, Minnesota. July 11, 1990.

(1)

Rep 1

RESULTS OF THE MARCH 28, 1990
DIOXIN EMISSION PERFORMANCE TEST
ON UNIT 3 AT THE NSP SHERCO
PLANT IN BECKER, MINNESOTA

interpoll

Interpoll Laboratories, Inc.
4500 Ball Road N.E.
Circle Pines, Minnesota 55014-1819

TEL: (612) 786-6020
FAX: (612) 786-7854

RESULTS OF THE MARCH 28, 1990 DIOXIN EMISSION PERFORMANCE TEST ON UNIT 3 AT THE NSP SHERCO PLANT IN BECKER, MINNESOTA

Submitted to:

NORTHERN STATES POWER COMPANY
West of Highway 10
Becker, Minnesota 55308

Attention:

Bob Catron

Approved by:

Perry Lonnes, Ph.D.
Director of Field Testing

Report Number 0-3009
July 11, 1990
PL/prp

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ABBREVIATIONS

ACFM	actual cubic feet per minute
cc (ml)	cubic centimeter (milliliter)
DSCFM	standard cubic foot of dry gas per minute
DSML	dry standard milliliter
DEG-F (^{0}F)	degrees Fahrenheit
DIA.	diameter
FP	finished product for plant
FT/SEC	feet per second
g	gram
GPM	gallons per minute
GR/ACF	grains per actual cubic foot
GR/DSCF	grains per dry standard cubic foot
g/dscm	grams per dry standard cubic meter
HP	horsepower
HRS	hours
IN.	inches
IN.HG.	inches of mercury
IN.WC.	inches of water
LB	pound
LB/DSCF	pounds per dry standard cubic foot
LB/HR	pounds per hour
LB/10^6BTU	pounds per million British Thermal Units heat input
LB/MMBTU	pounds per million British Thermal Units heat input
LTPD	long tons per day
MW	megawatt
mg/Nm^3	milligrams per dry standard cubic meter
ug/Nm^3	micrograms per dry standard cubic meter
microns (um)	micrometer
MIN.	minutes
ng	nanograms
ohm-cm	ohm-centimeter
PM	particulate matter
PPH	pounds per hour
PPM	parts per million
ppmC	parts per million carbon
ppm.d	parts per million, dry
ppm.w	parts per million, wet
ppt	parts per trillion
PSI	pounds per square inch
SQ.FT.	square feet
ug	micrograms
v/v	percent by volume
w/w	percent by weight
<	$\leq$ (when following a number)

Standard conditions are defined as 68 ^{0}F (20 ^{0}C) and 29.92 IN. of mercury pressure.

Jeff Cole, RTI, says this is a dry bottom boiler, according to the UDI/EEI database. Jeff has been working with the NSP data for five years and, therefore, I am taking his word on this.

Jj

1 INTRODUCTION

On March 28, 1990, Interpoll Laboratories personnel conducted a dioxin emission performance test on Unit 3 at the Northern States Power Company (NSP) Sherco Plant located in Becker, Minnesota. On-site testing was performed by D. Van Hoever, M. Kaehler and S. Bainville. Coordination between testing activities and plant operation was provided by Bob Catron of NSP.

The Unit 3 is a Babcock and Wilcox 860 megawatt boiler which came on line in 1987. The boiler is fired with pulverized subbituminous coal from Montana. Particulate and sulfur dioxide emissions are controlled by a dry scrubber consisting of a spray dryer absorber (SDA) and baghouse. Scrubbed flue gas is exhausted to the atmosphere by a 650-foot high radial brick-lined concrete stack.

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) sampling was conducted using a Modified Method 5 (MM5) sampling train with 32 g of purified XAD-2 resin in accordance with EPA Method 0010 (SW 846, 3rd Ed.) A spike (2-component mixture of isotopically-labeled dioxin and furan surrogates) was added to the top of the XAD-2 resin cartridge by Twin City Testing at the time the cartridges were packed. The pre-sample spikes provide an overall evaluation of the accuracy of sampling, recovery and analysis. A field-biased blank was collected by loading the entire sampling train, leak checking it and then recovering the sample in a manner identical to that used for the field samples.

The recovered PCDDs and PCDFs samples were stored over ice, returned to the laboratory, carefully packed and then shipped to Twin City Testing where they were analyzed for tetra- through octachlorodibenzodioxins and chlorodibenzofurans homolog groups as well as for all of the 2,3,7,8-chlorinated dibenzodioxin and dibenzofuran isomers as per EPA Method 8290

using HRGC/HRMS. It is important to note that quantitation has been performed by isotope dilution mass spectrometry by Twin City Testing and thus the reported dioxin and furan masses and concentrations have already been corrected for the recoveries of internal quantification standards.

Integrated flue gas samples were extracted simultaneously with each dioxin sample from the sampling train exhaust ports as part of the overall project quality assurance using a specially designed gas sampling system. Integrated flue gas samples were collected in 44-liter Tedlar bags. Prior to sampling, the Tedlar bags are leak checked at 15 IN.HG. vacuum with an in-line rotameter. Bags with any detectable inleakage are discarded. After sampling was complete, the bags were sealed and returned to the laboratory for Orsat analysis.

Testing on the boiler was conducted from two test ports oriented at 90° on the stack. These test ports are located 7 stack diameters downstream of the breeching inlet and 11 diameters upstream of the stack exit. Dioxin sampling was conducted using a 24-point traverse. Each traverse point was sampled ten minutes for a total sampling time of 240 minutes per run.

A summary and discussion of all of the important results of this engineering test is given in the following section. More detailed results of the samplings are presented in Section 3 together with pertinent sampling parameters. Supplemental information such as field data sheets, laboratory results, procedures and calculation equations are presented in the appendices.

2 SUMMARY AND DISCUSSION

The results of the dioxin and furan stack tests presented by homolog or chlorination level are given in Table 1. The results of the 2,3,7,8-isomer specific analyses are presented in Table 2.

The US EPA has published toxicity equivalence factors (TEFs) for the various non-2,3,7,8-polychlorinated isomers and 2,3,7,8-polychlorinated isomers. These factors may be used to convert the weight of these moieties to an equivalent weight of 2,3,7,8-tetrachlorodibenzo-p-dioxin. After the equivalent weights are calculated, they may be summed to give a single estimate of the toxic impact of the entire group of compounds based on the equivalence of each isomer group to 2,3,7,8-TCDD. This analysis has been performed in Table 3 using the latest US EPA TEFs.

Examination of these results show that the concentration of 2,3,7,8-TCDD equivalents from Unit 3 averaged 0.020 ng/Nm^3 for the three tests using state of the art sampling and analytical procedures. The corresponding 2,3,7,8-TCDD equivalent emission rate averaged $\leq 1.8\ 10^{-8}$ g/sec.

No difficulties were encountered in the field or in the laboratory evaluation of the samples. On the basis of this fact and a complete review of all of the field data, calculations and laboratory documentation, it is our opinion that the concentrations and emission rates reported herein are accurate and closely reflect the actual values which existed at the time the test was performed.

Table 1
Results of the March 28, 1990 Dioxin Homolog Analysis on Unit 3 at the NSP Sherco Plant.

Homolog	RUN 1	RUN 2	RUN 3
(Concentration ng/Nm³)			
TCDD	.043	.022	.016
PeCDD	.084	.041	.018
HxCDD	.35	.17	.093
HpCDD	1.3	.50	.26
OCDD	3.8	1.3	.79
TCDF	.35	.11	.052
PeCDF	.61	.24	.13
HxCDF	1.5	.71	.35
HpCDF	5.5	2.1	1.3
OCDF	18	5.6	4.4
(Emission Rate 10^{-8}g/sec)			
TCDD	4.0	2.0	1.4
PeCDD	7.8	3.8	1.7
HxCDD	32	16	8.6
HpCDD	119	46	24
OCDD	351	116	72
TCDF	32	10	4.8
PeCDF	57	22	12
HxCDF	143	65	32
HpCDF	512	197	118
OCDF	1670	512	402

Table 2
Results of the March 28, 1990 2,3,7,8-Isomer Analysis on Unit 3 at the NSP Sherco Plant (presented in 2,3,7,8-TCDD Equivalents).

Isomer	RUN 1	RUN 2	RUN 3
(Concentration *ng/Nm³*)			
2,3,7,8-TCDD	.001	.001	.0008
1,2,3,7,8-PeCDD	.007	.005	.002
1,2,3,4,7,8-HxCDD	.014	.009	.007
1,2,3,6,7,8-HxCDD	.038	.018	.006
1,2,3,7,8,9-HxCDD	.032	.028	.006
1,2,3,4,6,7,8-HpCDD	.7	.26	.15
2,3,7,8-TCDF	.061	.022	.010
1,2,3,7,8-PeCDF	.027	.011	.006
2,3,4,7,8-PeCDF	.058	.028	.014
1,2,3,4,7,8-HxCDF	.32	.13	.073
1,2,3,6,7,8-HxCDF	.10	.053	.024
1,2,3,7,8,9-HxCDF	.02	.009	.008
2,3,4,6,7,8-HxCDF	.28	.10	.006
1,2,3,4,6,7,8-HpCDF	1.5	.59	.35
1,2,3,4,7,8,9-HpCDF	.7	.28	.19
(Emission Rate 10^{-6}g/sec)			
2,3,7,8-TCDD	.12	.11	.078
1,2,3,7,8-PeCDD	.67	.48	.23
1,2,3,4,7,8-HxCDD	1.3	.86	.62
1,2,3,6,7,8-HxCDD	3.5	1.7	.56
1,2,3,7,8,9-HxCDD	3	2.5	.56
1,2,3,4,6,7,8-HpCDD	65	24	14
2,3,7,8-TCDF	5.7	2.0	.97
1,2,3,7,8-PeCDF	2.5	.97	.51
2,3,4,7,8-PeCDF	5.4	2.5	1.3
1,2,3,4,7,8-HxCDF	30	12	6.7
1,2,3,6,7,8-HxCDF	9.7	4.8	2.2
1,2,3,7,8,9-HxCDF	1.8	.83	.7
2,3,4,6,7,8-HxCDF	26	9.2	.56
1,2,3,4,6,7,8-HpCDF	138	54	32
1,2,3,4,7,8,9-HpCDF	65	26	17

Table 3
Results of the March 28, 1990 Dioxin Determination on Unit 3 at the NSP Sherco Plant (presented in 2,3,7,8-TCDD Equivalents).

	TEF	2,3,7,8-TCDD Equivalents RUN 1	RUN 2	RUN 3
(Concentration ng/Nm³)				
2,3,7,8-TCDD	1	.001	.001	.0008
other TCDDs	.01	.0004	.0002	.0001
2,3,7,8-PeCDD	.5	.004	.003	.001
other PeCDDs	.005	.0004	.0002	.00008
2,3,7,8-HxCDD	.04	.003	.002	.0008
other HxCDDs	.0004	.0001	.00005	.00003
2,3,7,8-HpCDD	.001	.0007	.0003	.0002
other HpCDDs	.00001	.000006	.000002	.000001
2,3,7,8-TCDF	.1	.006	.002	.001
other TCDFs	.001	.0003	.00009	.00004
2,3,7,8-PeCDF	.1	.008	.004	.002
other PeCDFs	.001	.0005	.0002	.0001
2,3,7,8-HxCDF	.01	.007	.003	.001
other HxCDFs	.0001	.00008	.00004	.00002
2,3,7,8-HpCDF	.001	.002	.0009	.0005
other HpCDFs	.00001	.00003	.00001	.000007
Totals		.035	.017	.008
(Emission rate 10^{-8}g/sec)				
2,3,7,8-TCDD	1	.12	.11	.078
other TCDDs	.01	.039	.019	.014
2,3,7,8-PeCDD	.5	.34	.24	.11
other PeCDDs	.005	.036	.016	.007
2,3,7,8-HxCDD	.04	.31	.20	.07
other HxCDDs	.0004	.01	.004	.003
2,3,7,8-HpCDD	.001	.065	.024	.014
other HpCDDs	.00001	.0005	.0002	.0001
2,3,7,8-TCDF	.1	.57	.20	.097
other TCDFs	.001	.027	.008	.004
2,3,7,8-PeCDF	.1	.79	.35	.18
other PeCDFs	.001	.049	.018	.011
2,3,7,8-HxCDF	.01	.67<	.27<	.10<
other HxCDFs	.0001	.008<	.004<	.002<
2,3,7,8-HpCDF	.001	.20	.080	.049
other HpCDFs	.00001	.004	.001	.0009
Totals		3.2<	1.6<	.74<

Toxicity Equivalence Factors (TEFs) used in the calculation of the above TTEs are those currently recommended by 'Iterim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-Dioxins and Dibenzofurans (CDDs and CDFs)' J.S. Bellin and D.G. Barnes, USEPA Risk Assesment Forum, October 1986.

3 RESULTS

The results of all field and laboratory evaluations are presented in this section. Gas composition (Orsat and moisture) are presented first followed by the dioxins and furan sampling data. Preliminary measurements including test port locations are given in the appendices.

The results have been calculated on an IBM PC Computer using programs written in Extended BASIC specifically for source testing calculations. EPA-published equations have been used as the basis of the calculation techniques in these programs.

Emission rates have been calculated using the product of the concentration times flow method.

Test No. 3
Unit 3 Stack

3.1 Results of Orsat & Moisture Analyses-----Methods 3 & 4(%v/v)

	Run 1	Run 2	Run 3
Date of run	03-28-90	03-29-90	09-29-90
Dry basis (orsat)			
carbon dioxide.....	13.40	14.10	14.10
oxygen.....	6.30	5.80	5.80
nitrogen.....	80.30	80.10	80.10
Wet basis (orsat)			
carbon dioxide.....	11.42	12.00	11.95
oxygen.....	5.37	4.94	4.91
nitrogen.....	68.42	68.17	67.87
water vapor.....	14.79	14.90	15.27
Dry molecular weight.....	30.40	30.49	30.49
Wet molecular weight.....	28.56	28.63	28.58
Specific gravity.....	0.987	0.989	0.987
Water mass flow.....(LB/HR)	0.00	0.00	0.00
FO	1.090	1.071	1.071

Test No. 3
Unit 3 Stack

3.2 Sampling Data for PCDD and PCDF Test-------SW846 Method 0010

	Run 1	Run 2	Run 3
Date of run	03-28-90	03-29-90	09-29-90
Time run start/end.....(HRS)	1225/1644	815/1233	1405/1818
Static pressure.....(IN.WC)	-0.80	-0.80	-0.80
Cross sectional area (SQ.FT)	683.49	683.49	683.49
Pitot tube coefficient.....	0.840	0.840	0.840
Water in sample gas			
condenser.....(ML)	414.0	416.0	430.0
impingers.....(GRAMS)	0.0	0.0	1.0
desiccant.....(GRAMS)	35.0	30.0	33.0
total.....(GRAMS)	449.0	446.0	464.0
Gas meter coefficient.....	0.9980	0.9980	0.9980
Barometric pressure..(IN.HG)	28.83	28.85	28.85
Avg. orif.pres.drop..(IN.WC)	0.91	0.88	0.89
Avg. gas meter temp..(DEF-F)	80.3	77.6	75.2
Volume through gas meter....			
at meter conditions...(CF)	129.34	126.68	127.44
standard conditions.(DSCF)	121.78	119.95	121.21
standard conditions..(NM^3)	3.448	3.397	3.432
Total sampling time....(MIN)	240.00	240.00	240.00
Nozzle diameter.....(IN)	.180	.180	.180
Avg.stack gas temp ..(DEG-F)	164	169	168
Volumetric flow rate.....			
actual.....(ACFM)	2845685	2819906	2846579
dry standard.....(DSCFM)	1971603	1939776	1952851
Isokinetic variation.....(%)	99.6	99.7	100.1

APPENDIX A

SAMPLING TRAIN CALIBRATION DATA

Interpoll Laboratories, Inc.
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: June 27, 1990

Meter Box No. : 8 (Rockwell Dry Test Meter Serial No. 964552)

Date of Last Calibration: March 7, 1990
Calibration Technician: D. Van Hoever
Wet Test Meter No.: American Meter AL-20

Date of Use	Report No.	Initial Meter Reading	Final Meter Reading	Volume/Job (cu. ft.)	Total Volume* (cu. ft.)
March 15, 1990	0-2995	175.17	292.18	117.01	117.01
March 22, 1990	0-3000	294.00	511.10	217.10	334.11
March 27, 1990	0-3005	511.57	719.85	208.28	542.39
March 28, 1990	0-3009	726.20	1121.34	395.14	937.53

* Total volume through meter since last calibration.

Date 3/7/90

Bar. Press. 29.56 in. Hg

INTERPOLL LABORATORIES, INC.
METER CALIBRATION SHEET
EPA METHOD 5

Control Module No. 8
Serial No. DTM 964.552
Wet Test Meter No. AL-20
Technician [illegible]

ΔH (in. WC) nominal	ΔH (in. WC) actual	Gas Volume Wet Test Meter (ft^3)	* Cal. Index θ (%)	* Diff. Wet Test Meter ΔP_w (in.WC)	Gas Volume Dry test meter (ft^3) V_{di}	Gas Volume Dry test meter (ft^3) V_{df}	Gas temperatures Wet Test t_w (°F)	Gas temperatures Dry Test t_{di} (°F)	Gas temperatures Dry Test t_{do} (°F)	Time θ (min/sec)	Meter Coeff. Y	Orifice Const. ΔHθ	C_f
0.5	0.5	2	99.70	0.01	161.76	163.78	73.0	81	77	5/5.24	.9970	1.84	
1.2	1.2	~~3~~ 10.5	99.77	0.03	164.29	174.95	73.1	83	78	17/16.71	.9933	1.84	
2.0	2.0	3	99.87	0.05	141.20	144.21	73.6	79	75	3/54.40	.9966	1.87	
3.3	3.3	5	100.00	0.08	146.22	151.23	73.5	82	76	5/2.82	.9998	1.90	
4.7	4.7	5	100.20	0.11	152.22	157.22	73.3	84	77	4/17.27	1.0035	1.94	
											.9980	1.88	

Meter was in tolerance ☒
Meter was not in tolerance ☐; readjusted linkage
Meter was not in tolerance ☐; changed dry test meters

Approved by Perry Lonnes Date 3-15-90

* Based on AL-20 wet test meter calibration in August 1989 against Bell Prover (NBS Traceable) - Carl Poe Co.

Interpoll Laboratories, Inc.
(612) 786-6020

Nozzle Calibration
Data Sheet

Date of Calibration: March 28, 1990 Nozzle Number MM5-3
Technician: D. Van Hoever

Nozzle rotated by 60 degree increments and diameter measured to nearest 0.001 inch. Observed readings and average:

Position	Diameter (inches)
1	0.180
2	0.177
3	0.183
Average:	0.180

Interpoll Laboratories
(612)786-6020

S-Type Pitot Tube Inspection Sheet

Pitobe No. 8-14

Pitot tube dimensions:

1. External tubing diameter (D_t) .316 IN.
2. Base to Side A opening plane (P_A) .462 IN.
3. Base to Side B opening plane (P_B) .462 IN.

Alignment:

4. $\alpha_1 < 10^o$ 0
5. $\alpha_2 < 10^o$ 0

6. $B_1 < 5^o$ 1
7. $B_2 < 5^o$ 1

8. Z <.125" .03
9. W <.0625" .02

Distance from Pitot to Probe Components:

10. Pitot to 0.500 IN. nozzle .760 IN.
11. Pitot to probe sheath 3.00 IN.
12. Pitot to thermocouple (parallel to probe) 3.00 IN.
13. Pitot to thermocouple (perpendicular to probe) .762 IN.

Date of Inspection: 12-8-89

Inspected by:

S-348(1)

Interpoll Laboratories, Inc.

Temperature Measurement Device
Calibration Sheet

Unit under test:

Vendor GORDON

Model 5310K

Range -112°F to 1999°F °F

Date of Calibration 3-5-90

Serial Number PDT-10

Thermocouple Type K

Technician S. BAINVILLE

Method of Calibration:

☐ Comparison against ASTM mercury in glass thermometer using a thermostatted and insulated aluminum block designed to provide uniform temperature. The temperature is adjusted by adjusting the voltage on the block heater cartridge.

☒ Omega Model CL-300 Type K Thermocouple Simulator which provides 22 precise temperature equivalent millivolt signals. The CL-300 is cold junction compensated. Calibration accuracy is ± 0.1% of span (2100°F) ± 1 degree (for negative temperatures add ± 2 degrees. The CL-300 simulates exactly the millivoltage of a Type K thermocouple at the indicated temperature.

Desired Temp (°F) Nominal	Temperature of Standard or Simulated Temp (°F)	Response of Unit Under Test (°F)	Deviation	
			Δt (°F)	(%)
0	0	.8	.8	.17
100	100	97.7	2.3	.41
200	200	201	1	.15
300	300	298	2	.26
400	400	395	5	.58
500	500	494	6	.62
600	600	596	4	.38
700	700	696	4	.34
800	800	800	0	—
900	900	901	1	.07
1000	1000	1004	4	.27
1100	1100	1106	6	.38
1200	1200	1211	11	.66
1300	1300	1310	10	.57
1400	1400	1413	13	.70
1500	1500	1509	9	.46
1600	1600	1608	8	.39
1700	1700	1702	2	.09
1800	1800	1798	2	.09
1900	1900	1889	11	.47
2000	2000	1983	17	.69
2100	2100	OF	OF	OF
		Averages:	6.0	.39%

OF = off scale response by unit under test (°F)

% dev = 100 Δt / (460 + t)

☒ Unit in tolerance

☐ Unit was not in tolerance; recalibrated - See new calibration sheet.

S-433

Interpoll Laboratories, Inc.

Temperature Measurement Device
Calibration Sheet

Unit under test:
Vendor ATKINS
Model 39658
Range -112.0°F to +1990°F °F
Date of Calibration 2-5-90
Serial Number PDT-1
Thermocouple Type
Technician E. Trowbridge

Method of Calibration:

☐ Comparison against ASTM mercury in glass thermometer using a thermostatted and insulated aluminum block desi[illegible] to provide uniform temperature. The temperature is adjusted by adjusting the voltage on the block heat[illegible] cartridge.

☐ Omega Model CL-300 Type K Thermocouple Simulator which provides 22 precise temperature equivalent milliv[illegible] signals. The CL-300 is cold junction compensated. Calibration accuracy is ± 0.1% of span (2100 °F) ± 1 deg[illegible] (for negative temperatures add ± 2 degrees. The CL-300 simulates exactly the millivoltage of a Type K thermocouple at the indicated temperature.

Desired Temp (°F) Nominal	Temperature of Standard or Simulated Temp (°F)	Response of Unit Under Test (°F)	Deviation Δt (°F)	Deviation (%)
0	0	2.9	2.9	.63
100	100	100.0	—	—
200	200	203	3	.45
300	300	301	1	.13
400	400	398	2	.23
500	500	497	3	.31
600	600	599	1	.09
700	700	699	1	.08
800	800	803	3	.23
900	900	905	5	.36
1000	1000	1008	8	.54
1100	1100	1111	11	.70
1200	1200	1216	16	.96
1300	1300	1315	15	.85
1400	1400	1418	18	.96
1500	1500	1514	14	.71
1600	1600	1614	14	.67
1700	1700	1708	8	.37
1800	1800	1804	4	.17
1900	1900	1895	5	.27
2000	2000	1988	12	.48
2100				
		Averages:	6.6	.43

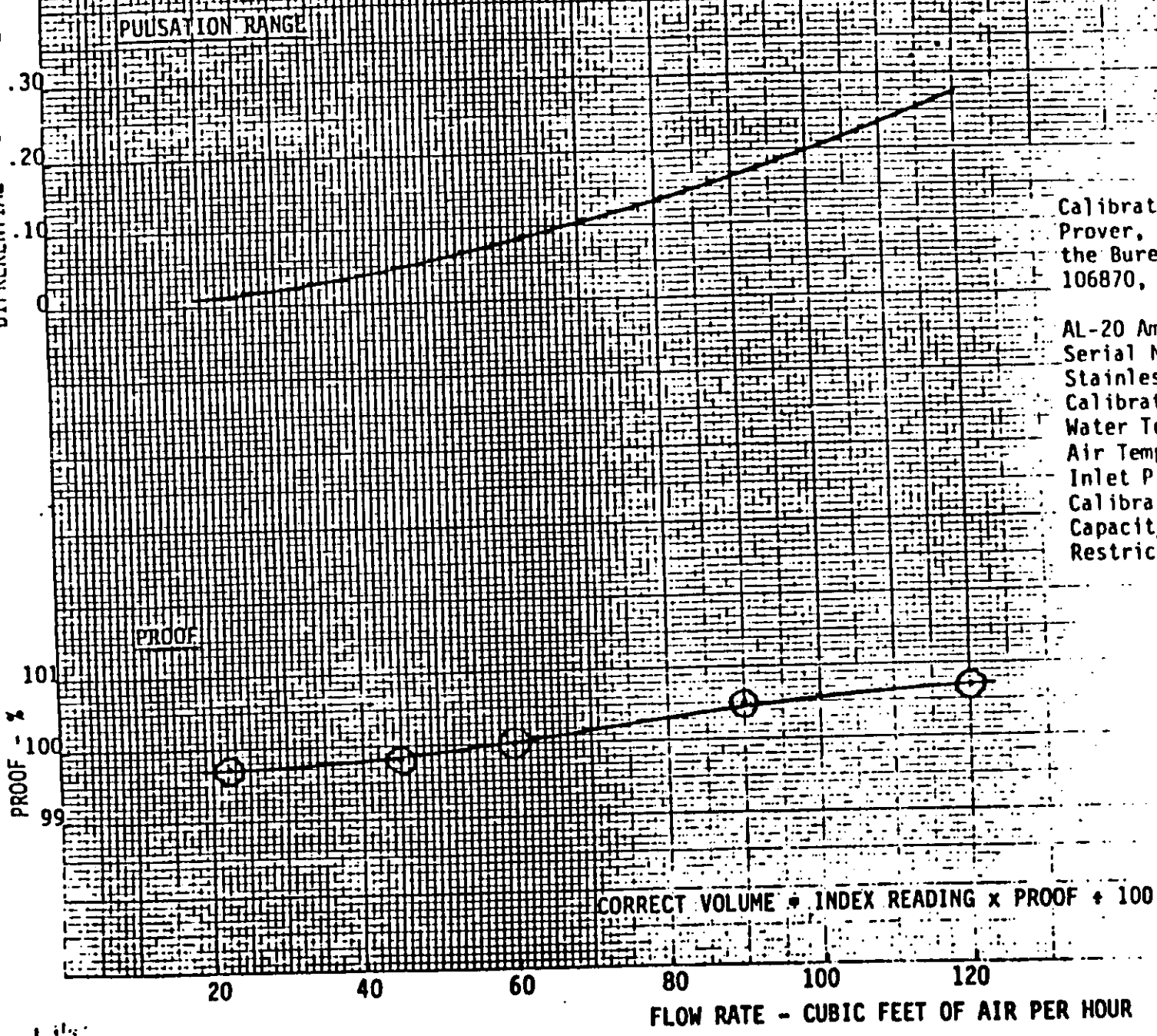
OF = off scale response by unit under test (°F)
% dev = 100 Δt / (460 + t)

☒ Unit in tolerance
☐ Unit was not in tolerance; recalibrated - See new calibration sheet.

S-433

DIFFERENTIAL PRESSURE AND PROOF CALIBRATION CURVES

WET TEST METER



Calibrated with a 10 Ft. American Bell Prover, Serial No. 3157. Traceable the Bureau of Standards. Reference No 106870, PI-TAPE.

AL-20 American Wet Test Meter
Serial No. P-717
Stainless Steel w/Removable Back
Calibrated w/Saturated Air
Water Temp. 74° F.
Air Temp. 74° F.
Inlet Pressure 2" H_2O Constant
Calibration Rate: 60 CFH Per/Hr.
Capacity Rate: 120 CFH Per/Hr.
Restricted Outlet for Rate Deviation

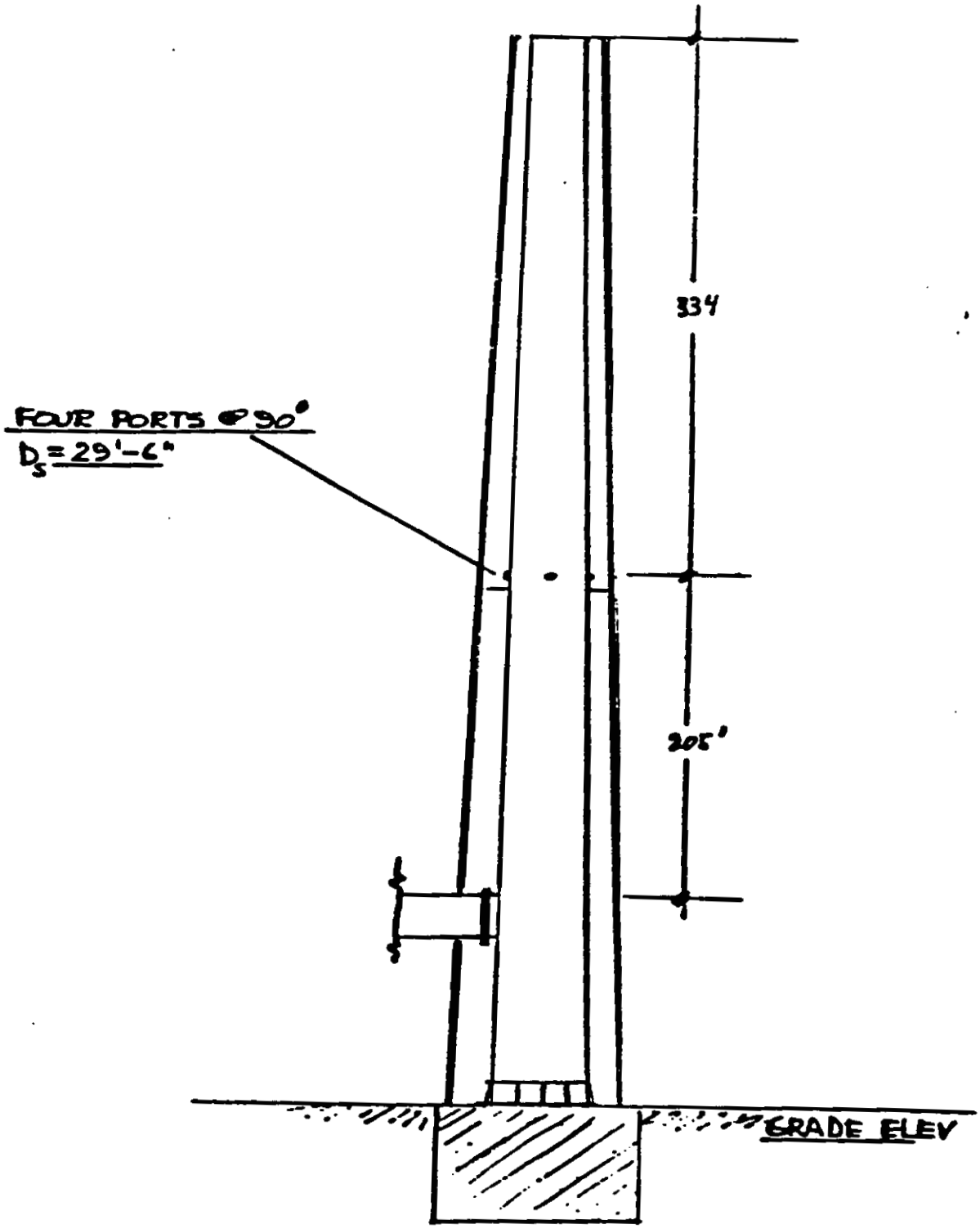
CORRECT VOLUME = INDEX READING x PROOF ÷ 100

DAVID BANKS
AUGUST, 1989

APPENDIX B

LOCATION OF TEST PORTS

NSP SHERCO UNIT 3



LP 11/87
REV 3/88

NOT TO SCALE

APPENDIX C

FIELD DATA SHEETS FOR DIOXIN SAMPLING

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job N.S.P. SHEROO

Source Unit #3 Stack

Test 3 Run 0 Date 3/28

Stack dimen. 354 IN.

Dry bulb ____ °F Wet bulb ____ °F

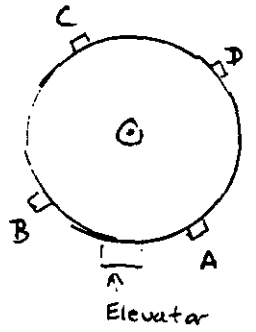
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.

Barometric pressure 28.83 in Hg

Static pressure -.80 in WC

Operators D. Van Hoever & S. Bainville & M. Kuhl

Pitot No. V14-8 Cp .84



Schematic of Cross Section

Traverse Point No.	Fraction of Diameter	Distance from Stack Wall (in)	Distance from End of Port (in)	Velocity Pressure (in WC)	Temperature of gas (°F)
		Port length: 40 in.		Time start: hrs	
A 4	.032	11.33	51.33		
2	.105	37.17	77.17		
3	.194	68.68	108.68		
4	.323	114.34	154.34		
B 1					
2					
3					
4					
B 1					
2					
3					
4					
C 1					
2					
3					
4					
Temp. meas. tool & S/N:				Time end: hrs	

R or nothing= reg. manometer: S= expanded: E = electronic S-392.1

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job N.S.P. SHERCO
Source Unit # 3 Stack
Cyclone: ☐ Yes ☒ No Filter holder: MM5
Analytes:
Field recovery spike added: ☐ Yes ☐ No

Date 3/28/90 Test 3 Run 1
No. of traverse points 48
Filter type: 4" Glass fiber
XAD-2 resin: ___ g Batch No. ___
Reference: EPA SW-846 Method 001

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: 0 cfm at 8 in. Hg. (vac) ☒

Semivolatile Catch Data:

No.s of filters used: Recovery solvent(s)

☒ MeCl2 * ~~MeOH (50:50~~ v/v)
☒ other(s) Acetone

No. of bottles for condensate trap catch: ___
Samples recovered by: DAN & SLB & M.K

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Condensate trap			
Condensate trap	414	0	414
Condensate trap			
Impingers	200	200	0
Desiccant	1415	1380	35
Total			449

Integrated Gas Sampling Data:

Bag Pump No. B3 Box No. 23 Bag No. 1

Bag Material: 4-layer Aluminized Tedlar Size: 44 L

Pretest leak check: 0 cc/min at 14 in. Hg.

Time start: 12 30 (HRS) Time end: 16 43 (HRS)

Sampling rate: 100 cc/min O2 Analyzer S/N: 8

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INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 FIELD DATA SHEET

Job N.S.P SHERLD
Source UNIT # 3 Stack
Date 3/28/90 Test 3 Run 1

Operators [illegible] & SLBX MK
Meter Box No. 8 → AH@ 1.88
Gasmeter coeff. .9980

Nozzle No. MM5-3
Nozzle Dia. .180 in.
Bar. Pres. 28.83 in. Hg

Pitot No. V14-8
Cp .89
H_2O 15 %

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dec. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
	(12.25)	(726.20)												
A 4	5	728.80	1.25	.90	8.84	4.5	163	254	259	47	42	77	77	6.0
4	10	731.57	1.30	.96	1.59	5	163					86	80	5.7
4	15	734.33	1.25	.93	4.33	5	163	257	260	44	41	88	80	5.8
3	20	737.12	1.30	.97	7.12	6	163					88	81	5.9
3	25	739.92	1.35	1.01	9.97	6	163	255	260	40	39	88	82	5.8
3	30	742.73	1.30	.97	2.76	6	165					89	82	5.9
2	35	745.56	1.30	.97	5.56	6	165	258	258	40	39	89	83	5.9
2	40	748.40	1.35	1.01	8.41	6	163					89	83	5.8
2	45	751.23	1.30	.97	1.22	5.8	163					88	81	5.9
1	50	753.64	.94	.70	3.59	4.7	163	256	257	42	40	88	82	5.9
1	55	756.16	1.10	.82	6.17	5.0	163					88	82	5.9
1	60	758.65	1.05	.78	8.68	5.0	163	257	259	43	40	88	82	5.9
B 4	65	761.30	1.20	.89	1.37	6.0	165					83	80	6.2
4	70	763.95	1.15	.85	3.98	6.0	163	259	258	41	41	84	80	6.2
4	75	766.53	1.10	.81	6.54	5.5	165					83	79	6.2
3	80	769.20	1.20	.89	9.20	6.0	165	259	260	40	41	82	78	6.2
3	85	771.77	1.10	.81	1.75	5.5	165					81	77	6.2
3	90	774.29	1.10	.81	4.29	5.2	165	258	259	42	43	78	75	6.2
2	95	776.83	1.10	.81	6.82	5.2	165					78	75	6.2
2	100	779.37	1.10	.81	9.35	5.2	165	258	259	43	42	78	75	6.2
2	105	781.93	1.15	.84	1.94	5.5	165					77	74	6.2
1	110	784.21	.86	.63	4.17	4.4	166	259	258	41	42	76	73	6.2
1	115	786.35	.82	.60	6.35	4.3	166					76	72	6.1
1	120	788.52	.80	.58	8.50	4.4	166	259	259	42	41	76	72	6.2
	θ=	Vm=		^H=								Avg.=		

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CF-023

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 FIELD DATA SHEET

Job NSP SPERCO
Source Unit No. 3 Stack
Date 3-28-90 Test 3 Run 1

Operators DW + SB + PK
Meter Box No. 8 → AHC 1.88
Geometer coeff. .9980

Nozzle No. MM5-3
Nozzle Dia. .180 in.
Bar. Pres. 28.83 in. Hg

Pitot No. V14A
Cp .84
H_2O 15 %

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Des. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
C 4	125	791.41	1.45	1.06	1.38	7.0	166	260	259	41	41	75	71	6.1
4	130	794.06	1.25	.91	4.06	6.0	166					76	72	6.1
4	135	796.74	1.25	.91	6.74	6.0	166	260	258	40	41	77	72	6.1
3	140	799.46	1.30	.95	9.48	6.0	166					78	72	6.0
3	145	802.13	1.20	.88	2.11	6.0	165	259	258	40	42	79	73	6.0
3	150	804.73	1.20	.88	4.75	5.9	165					79	73	6.0
2	155	807.41	1.30	.95	7.50	6.0	165	259	257	39	41	80	73	6.0
2	160	810.17	1.30	.95	0.25	6.0	165					80	74	6.0
2	165	812.93	1.25	.92	2.95	6.0	165	258	258	39	40	81	75	6.1
1	170	815.43	.99	.73	5.36	5.1	165					82	75	6.2
1	175	817.82	1.05	.77	7.84	5.1	165	259	258	40	41	82	75	6.0
1	180	820.33	1.05	.77	0.32	5.2	165					83	76	6.6
D 4	185	823.16	1.40	1.03	3.19	7.2	164	259	259	40	42	82	76	6.4
4	190	826.11	1.40	1.03	6.06	7.0	164					84	77	6.3
4	195	828.99	1.40	1.04	8.94	7.0	164	259	260	41	42	85	78	6.3
3	200	831.91	1.45	1.08	1.87	7.0	164					86	79	6.4
3	205	834.84	1.55	1.15	4.91	7.0	164	258	259	41	41	86	79	6.4
3	210	837.82	1.50	1.11	7.89	7.0	164					87	80	6.4
2	215	840.87	1.45	1.08	0.84	7.0	164	257	259	40	42	88	80	6.4
2	220	843.89	1.50	1.12	3.83	7.0	164					88	81	6.4
2	225	846.84	1.50	1.12	6.83	7.0	164	258	259	41	41	88	81	6.4
1	230	849.67	1.40	1.04	9.73	6.5	164					89	82	6.3
1	235	852.55	1.35	1.01	2.58	6.5	164	259	258	42	41	89	82	6.3
1	240	855.54	1.40	1.05	5.48	6.5	164					89	82	6.3
	(1644)													

[illegible] 24[illegible] Vm= [illegible].34 [illegible] H= [illegible] [illegible] g.= [illegible] % [illegible]

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job N.S.P. SHERCO
Source Unit #3 Stack
Cyclone: ☐ Yes ☒ No Filter holder: MM5
Analytes:
Field recovery spike added: ☒ Yes ☐ No

Date 3/29/90 Test 3 Run 2
No. of traverse points 48 (16)
Filter type: 4" Glass fiber
XAD-2 resin: g Batch No.
Reference: EPA SW-846 Method 0010

Sample Train Leak Check:

Pretest: < 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: 0.00 cfm at 10 in. Hg. (vac) ☒

Semivolatile Catch Data:

No.s of filters used:

Recovery solvent(s)

☒ MeCl2 ~~MeOH (50:50 v/v)~~
☒ other(s) toluene + ACETONE

No. of bottles for condensate trap catch: 1
Samples recovered by: [illegible] + S.L.B + M.K.

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Condensate trap			
Condensate trap	416	0	416
Condensate trap			
Impingers	200	200	0
Desiccant	1420	1390	30
Total			446

Integrated Gas Sampling Data:

Bag Pump No. 33 Box No. 23 Bag No. 2

Bag Material: 4-layer Aluminized Tedlar Size: 44 L

Pretest leak check: 0 cc/min at 15 in. Hg.

Time start: 0815 (HRS) Time end: 1233 (HRS)

Sampling rate: 100 cc/min O2 Analyzer ~~S/N:~~ [illegible]

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INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 FIELD DATA SHEET

Job N.S.P. SHERCO
Source UNIT #3 Stack
Date 3/29/90 Test 3 Run 2

Operators D.V.H + S.L.B + M.K
Meter Box No. 8 ΔH@ 1.88
Gasmeter coeff. .9980

Nozzle No. MMS-3
Nozzle Dia. .180 in.
Bar. Pres. 28.85 in. Hg

Pitot No. V14-8
Cp .84
H_2O 15 %

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Des. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
	08 15	865.90												
D 4	5	868.48	1.25	.90	8.55	5	170	251	247	43	44	69	69	5.7
4	10	871.17	1.25	.90	1.19	6	170					75	72	5.7
4	15	873.72	1.20	.87	3.80	6	170	252	248	40	37	77	72	5.7
3	20	876.51	1.30	.94	6.53	7	170					79	74	5.7
3	25	879.30	1.35	.98	9.31	8	173	252	250	39	37	80	74	5.7
3	30	882.07	1.35	.98	2.09	8	174					81	75	5.7
2	35	884.88	1.35	.98	4.88	8	174	253	252	39	36	82	76	5.6
2	40	887.74	1.35	.98	7.67	8	174					82	76	5.7
2	45	890.51	1.39	.98	0.46	8	174					83	77	5.7
1	50	893.24	1.30	.94	3.21	8	174	252	252	40	38	84	78	5.6
1	55	895.92	1.25	.91	5.90	8	174					84	78	5.7
1	60	898.63	1.25	.91	8.60	8	172					84	78	5.6
C 4	65	901.27	1.20	.88	1.25	8	172	254	251	40	37	80	76	5.8
4	70	903.90	1.20	.88	3.89	8	169					82	77	5.7
4	75	906.60	1.20	.88	6.54	8	168					82	77	5.7
3	80	909.23	1.20	.88	9.18	8	168	254	253	41	38	82	77	5.7
3	85	911.90	1.25	.92	1.88	8	168					82	77	5.7
3	90	914.57	1.25	.92	4.59	8	168					82	77	5.7
2	95	917.27	1.25	.92	7.29	8	168	256	254	40	36	81	77	5.8
2	100	919.96	1.20	.88	9.93	8	168					81	76	5.7
2	105	922.52	1.10	.80	2.46	7	168					81	76	5.8
1	110	924.95	1.05	.77	4.94	6.8	166	255	254	41	37	80	76	5.7
1	115	927.49	1.10	.81	7.47	7	166					80	76	5.7
1	120	929.91	1.05	.77	9.95	7	166					80	75	5.7

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 FIELD DATA SHEET

Job N.S.P. SHERCO
Source Unit #3 Stack
Date 3/29/90 Test 3 Run 2

Operators pg 2 of 2
Meter Box No.
Gasmeter coeff.

Nozzle No.
Nozzle Dia. in.
Bar. Pres. in. Hg

Pitot No.
Cp
H_2O %

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dec. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
C 4	125	932.38	1.05	.77	2.42	7	166	256	257	41	38	78	74	5.8
4	130	934.90	1.10	.80	4.94	7.2	166					79	75	5.7
4	135	937.40	1.05	.77	7.41	7.2	166					80	75	5.8
3	140	939.91	1.10	.81	9.94	7.2	166	255	258	42	39	80	75	5.8
3	145	942.45	1.10	.81	2.47	7.5	166					80	75	5.8
3	150	945.03	1.10	.81	5.00	7.5	166					80	75	5.7
2	155	947.65	1.20	.88	7.64	7.5	166	255	258	42	39	80	75	5.8
2	160	950.45	1.20	.88	0.29	7.5	166					80	75	5.8
2	165	952.91	1.20	.88	2.93	7.5	165	25				81	76	5.7
1	170	955.52	1.10	.81	5.47	8.0	165	255	258	42	38	81	76	5.7
1	175	957.99	1.05	.77	7.94	8.0	165					81	76	5.7
1	180	960.49	1.10	.81	0.48	8.0	165					81	76	5.7
A 4	185	963.16	1.25	.92	3.18	7.5	165	256	257	42	38	79	75	5.8
4	190	965.90	1.30	.95	5.93	7.5	165					79	75	5.7
4	195	968.62	1.25	.91	8.62	8.0	169					79	75	5.7
3	200	971.37	1.30	.95	1.35	8.0	169	257	258	40	38	79	75	5.7
3	205	974.10	1.30	.95	4.10	8.0	169					79	75	5.7
3	210	976.83	1.25	.91	6.79	8.0	169	257	260	40	38	79	75	5.7
2	215	979.56	1.30	.95	9.53	8.0	169					79	75	5.7
2	220	982.28	1.30	.95	2.27	8.0	169					79	75	5.7
2	225	985.50	1.30	.95	5.01	8.0	169	257	260	40	38	79	75	5.7
1	230	987.57	1.10	.80	7.53	8.0	169					79	75	5.8
1	235	990.10	1.10	.80	0.05	8.0	169	7				79	73	5.7
1	240	992.58	1.10	.80	2.57	8.0	169	257	260	40	39	78	72	5.7
	(1233)													
	θ= 240	Vm=126.68		^H=.88								Avg.=	77.6	

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CF-025

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job NSP / SHERCO
Source UNIT #3 STACK
Cyclone: ☐ Yes ☒ No Filter holder: MM5
Analytes:
Field recovery spike added: ☒ Yes ☐ No

Date 3-29-90 Test 3 Run 3
No. of traverse points 48
Filter type: 4" Glass fiber
XAD-2 resin: g Batch No.
Reference: EPA SW-846 Method 001

Sample Train Leak Check:

Pretest: < 0.02 cfm at 15 in. Hg. (vac)
Postest: 0 cfm at 9 in. Hg. (vac)

Semivolatile Catch Data:

No.s of filters used:

Recovery solvent(s)

☒ MeCl2 ~~+ MeOH (50:50 v/v)~~
☒ other(s) ACETONE

No. of bottles for condensate trap catch: 1
Samples recovered by: DUH + SLB + MK

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Condensate trap			
Condensate trap	430	0	430
Condensate trap			
Impingers	201	200	1
Desiccant	1493	1460	33
Total			464

Integrated Gas Sampling Data:

Bag Pump No. B3 Box No. 23 Bag No. 3

Bag Material: 4-layer Aluminized Tedlar Size: 44 L

Pretest leak check: 60 cc/min at 15 in. Hg.

Time start: 1410 (HRS) Time end: (HRS)

Sampling rate: 100 cc/min O2 Analyzer S/N: 8

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INTERPOLE LABORATORIES EPA QUALITY METHOD 5 FIELD DATA SHEET

Job NSP / SMERCO
Source UNIT #3 STACK
Date 3-29-90 Test 3 Run 3

Operators DUM + SLB
Meter Box No. 8
Gasmeter coeff. .9980

Nozzle No. 180 MM5-3 Pitot No. V14-8
Nozzle Dia. .180 in. Cp .840
Bar. Pres. 28.85 in. Hg H_2O 15 %

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dry Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
	(1405)	(993.90)												
A 4	5	996.57	1.25	.92	6.55	6	166	243	247	44	38	64	64	5.7
4	10	999.16	1.25	.89	9.22	6	166					68	65	5.7
4	15	1001.81	1.20	.86	1.81	6	166	245	248	42	38	68	65	5.7
3	20	1004.46	1.30	.93	4.50	6	166					69	65	5.7
3	25	1007.15	1.30	.93	7.20	6	166					69	65	5.6
3	30	1009.92	1.35	.97	9.94	7	167	247	251	40	37	70	65	5.7
2	35	1012.66	1.35	.97	2.69	7	167					70	65	5.7
2	40	1015.34	1.30	.93	5.39	7	167					70	66	5.7
2	45	1018.09	1.35	.97	8.14	7	167	247	251	40	37	71	66	5.7
1	50	1020.73	1.20	.86	0.73	7	167					73	67	5.7
1	55	1023.40	1.20	.87	3.34	7	166					72	67	5.6
1	60	1025.94	1.20	.87	5.94	7	166	247	253	41	39	72	66	5.7
B 4	65	1028.50	1.20	.84	8.54	7	166					69	66	5.8
4	70	1031.04	1.15	.83	1.03	7	166					68	72	5.6
4	75	1033.64	1.10	.79	3.57	7	167	251	251	40	38	75	69	5.6
3	80	1036.14	1.10	.80	6.07	7	167					76	69	5.7
3	85	1038.59	1.10	.80	8.58	7	166	250	254	39	37	77	70	5.7
3	90	1041.10	1.10	.80	1.09	7	166					78	71	5.6
2	95	1043.56	1.10	.80	3.60	7	166					79	72	5.6
2	100	1046.14	1.10	.80	6.12	7	166	251	257	38	37	72	80	5.6
2	105	1048.66	1.10	.80	8.65	7	166					80	73	5.7
1	110	1051.23	1.15	.84	1.23	7	167					80	74	5.7
1	115	1053.75	1.10	.81	3.76	7	165	254	253	39	37	80	74	5.7
1	120	1056.29	1.10	.81	6.29	7	166						74	5.7
	( )													
	θ=	Vm=		^H=								Avg.=		

C-9

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CF-025

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 FIELD DATA SHEET

Job NSP / SMERCO
Source UNIT #3 STACK
Date 3-29-90 Test 2 Run 2

Operators Pg 2 of 2
Meter Box No.
Gasmeter coeff.

Nozzle No.
Nozzle Dia. in.
Bar. Pres. in. Hg

Pitot No.
Cp
H_2O %

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dec. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
[redacted]			[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
4	125	1056.85	1.20	.88	8.93	7	166	249	253	42	39	80	75	5.7
4	130	1061.55	1.20	.88	1.57	7	166					80	75	5.7
4	135	1069.27	1.20	.88	4.21	7	166					81	75	5.8
3	140	1066.91	1.30	.95	6.46	7	166	249	257	41	39	82	76	5.7
3	145	1069.57	1.20	.88	9.61	7	166					82	76	5.7
3	150	1072.29	1.30	.95	2.37	7	166					82	76	5.7
2	155	1075.05	1.30	.95	5.12	7.5	168	250	257	40	38	82	76	5.7
2	160	1077.70	1.20	.87	7.76	7.5	172					82	76	5.8
2	165	1080.31	1.20	.87	0.39	7.5	173					82	76	5.7
1	170	1082.89	1.10	.80	2.92	7	173	250	257	40	39	82	76	5.8
1	175	1085.44	1.10	.80	5.43	7	173					82	76	5.8
1	190	1087.96	1.10	.80	7.96	7	172					82	76	5.7
4	185	1090.54	1.20	.87	0.60	7	171	252	255	41	37	80	76	6.0
4	190	1093.24	1.25	.91	3.29	7	170					81	76	5.8
4	155	1095.94	1.25	.91	5.98	7	169					82	77	5.7
3	200	1098.69	1.30	.95	1098.74	7	169	254	253	40	37	83	77	5.7
3	205	1101.55	1.40	1.03	1.60	8	168					83	77	5.7
3	210	1104.42	1.40	1.03	4.46	8	168					84	78	5.7
2	215	1107.32	1.40	1.03	7.33	8	168	252	256	39	36	84	78	5.8
2	220	1110.09	1.35	.99	0.14	8	168					84	78	5.7
2	225	1112.97	1.40	1.03	3.01	8	168	252	255	38	36	84	78	5.7
1	230	1115.78	1.30	.96	5.80	8	168					85	78	5.7
1	235	1118.59	1.30	.96	8.56	9	168					84	79	5.7
1	240	1121.34	1.30	.96	1.33	9	168	253	256	39	37	84	78	5.7
	(1818)													

[redacted]

APPENDIX D

LABORATORY DATA SHEETS FOR METHOD 3

Interpoll Laboratories
(612) 786-6020

Chain of Custody
Sample Deposition Sheet

Job N.S.P. SHERCO
Team Leader [illegible]
Date Submitted 3/30/90
Test No. 3

Source Unit # 3
Test Site Stack
Date of Test 3/28 & 3/29/90
No. of Runs Completed 3

No. of Samples	Type of Sample	Analysis Required	Comments
	Probe Wash: ☐Acetone ☐D.I. Water	☐As per EPA M-5 ☐Other _____	
	Filter: ☐4" G.F. ☐S.S. Thimble ☐2.5" G.F. ☐47 mm G.F.	☐As per EPA M-5 ☐As per EPA M-17 ☐Other _____	
	Impinger Catch: ☐D.I. Water ☐3% H_2O_2 ☐4M5 Hg Only ☐4M5 Metals ☐1.0 N NaOH ☐Other _____	☐MN Protocol ☐WI Protocol ☐EPA M-6 or 8 ☐Acid Gases ☐Formaldehyde ☐Metals ☐Other _____	
3	Integrated Gas sample	☒As per EPA M-3 ☐As per EPA M-10 ☐Other _____	
	Oxides of Nitrogen (NO_x)	☐As per EPA M-7A ☐Other _____	Date _____ Time(HRS) _____
2	☒ Fuel Sample ☐ ~~Aggregate~~	☐~~Attached fuel form~~ ~~45-0183RRR~~	Lime samples see propa
	Particle Size	☐X-Ray Sedigraph ☐Bahco Method ☐Other _____	
	Audit Samples ☐Sulfur Dioxide ☐Oxides of Nit. ☐Other _____	☐As per EPA M-6 ☐As per EPA M-7A ☐Other _____	

Source Information

1) Type of Source: ☒ Boiler ☐ Asphalt Plant ☐ Incinerator ☐ Dryer ☐ Other _____
2) Fuel: ☒ Coal ☐ Wood ☐ Gas ☐ Oil ☐ RDF ☐ Other _____
3) Is sample combustible? ☒ No ☐ Yes
4) Does sample need special handling? ☒ No ☐ Yes If yes, explain _____

S-278RRRR

Interpoll Laboratories
(612) 786-6020

EPA Method 3 Data Reporting Sheet
Orsat Analysis

Job NSP / Sherco
Team Leader MVH
Date Submitted 3-29-90
Test No. 3
Date of Analysis 3-30-90

Source Unit #3
Test Site Stack
Date of Test 3-29-90
No. of Runs Completed 3
Technician RE

Test/ Run	Sample Log Number and Type	No. of An.	Buret Readings (ml) Zero Pt.	After CO_2	After O_2	Conc. CO_2 %v/v Dry	Conc. O_2 %v/v Dry	F_o
3/1		1	0.00	13.40	19.70	13.40	6.30	1.09
		2	0.00	13.40	19.70	13.40	6.30	1.09
	☑ B ☐ F	Avg				13.40	6.30	
3/2		1	0.00	14.10	19.90	14.10	5.80	1.07
		2	0.00	14.10	19.90	14.10	5.80	1.07
	☑ B ☐ F	Avg				14.10	5.80	
3/3		1	0.00	14.10	19.90	14.10	5.80	1.07
		2	0.00	14.10	19.90	14.10	5.80	1.07
	☑ B ☐ F	Avg				14.10	5.80	
		1						
		2						
	☐ B ☐ F	Avg						
		1						
		2						
	☐ B ☐ F	Avg						
		1						
		2						
	☐ B ☐ F	Avg						
		1						
		2						
	☐ B ☐ F	Avg						
		1						
		2						
	☐ B ☐ F	Avg						
		1						
		2						
	☐ B ☐ F	Avg						

☑ Ambient Air QA Check
☑ Orsat Analyzer System Leak Check
☑ F_o Within EPA M-3 Guidelines for fuel type.

Where $F_o = \frac{20.9 - O_2}{CO_2}$

F=Flask (250 cc all glass)
B=Tedlar Bag (5-layer)

EPA Method 3 Guidelines

Fuel Type	F0 Range
Coal:	
Anthracite/Lignite	1.016-1.130
Bituminous	1.083-1.230
Oil:	
Distillate	1.260-1.413
Residual	1.210-1.370
Gas:	
Natural	1.600-1.836
Propane	1.434-1.586
Butane	1.405-1.553
Wood/Wood Bark	1.000-1.130

LSC-04-BR

APPENDIX E

TWIN CITY TESTING DIOXIN AND FURAN ANALYSES

REPORT OF CHEMICAL ANALYSES

Reported to:

Interpoll Laboratories, Inc.
Attn: Ms. Kathleen Eickstadt
4500 Ball Road N.E.
Circle Pines, MN 55014-1819

By:

Twin City Testing Corporation
Organic Chemistry Department
662 Cromwell Avenue
St. Paul, MN 55114

RECEIVED
JUN 20 1990
INTERPOLL LABORATORIES

twin city testing
corporation
662 CROMWELL AVENUE
ST. PAUL, MN 55114
PHONE 612/645-3601

REPORT OF: CHEMICAL ANALYSES

PROJECT: NSP/SHERCO

DATE: June 15, 1990

ISSUED TO: Interpoll Laboratories, Inc.
Attn: Ms. Kathleen Eickstadt
4500 Ball Road N.E.
Circle Pines, MN 55014-1819

INVOICE NO: 4410 90-4052

INTRODUCTION

This report summarizes the results from the analyses performed on four multi-component stack emission samples which were submitted by a representative of Interpoll Laboratories, Inc. The samples were analyzed for the presence or absence of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) using a modified version of EPA Method 8290.

SAMPLE IDENTIFICATION

Client ID	TCT ID
T3R0 Field Blank 9242-(54-57)	187226
T3R1 9242-(58-61)	187228
T3R2 9242-(62-65)	187229
T3R3 9242-(66-69)	187234

METHODOLOGY

PCDD/PCDF Extraction

The XAD-2 resin portion of each MM-5 sample component set was spiked with $^{13}C_{12}$-labeled PCDD/PCDF internal standards (Table 1), combined with the filter and placed in a Soxhlet extractor. The probe rinse solvents, which did not contain particulate, were added to the Soxhlet without filters and continuously extracted with benzene for 16 hours. The condensate portion of each sample was extracted with methylene chloride in a separatory funnel and the extracts were combined with the XAD-2 extracts. The combined extracts were quantitatively transferred to Kuderna Danish concentrators, concentrated, and solvent exchanged to hexane. The hexane extracts were then spiked with 2,3,7,8-TCDD-$^{37}Cl_4$ enrichment efficiency standard (Table 1) and processed through the analyte enrichment procedures described below.

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REPORT OF: CHEMICAL ANALYSES

PROJECT: NSP/SHERCO

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Analyte Enrichment for PCDD/PCDF Analyses

The extraction procedure often removes a variety of compounds, in addition to the PCDDs and PCDFs, from the sample matrix. Some of these compounds can directly interfere with the analyses while others can overload the capillary column causing degradation in chromatographic resolution or sensitivity. The analyte enrichment steps described below were used to remove interferences from the extracts.

The extracts were diluted to 100 ml with hexane and washed with 1N sodium hydroxide, concentrated sulfuric acid, and distilled water. The hexane layers were concentrated to 1 ml and quantitatively transferred to liquid chromatography columns containing alternating layers of silica gel, 44% concentrated sulfuric acid on silica gel, and 33% 1 N sodium hydroxide on silica gel. The columns were eluted with 60 ml of hexane and each entire eluate was collected and concentrated, under a gentle stream of dry nitrogen, to a volume of 1 ml.

The extracts were then fractionated on liquid chromatography columns containing 4 g of activated alumina. The columns were eluted with 10 ml of hexane followed by 7 ml of 2.0% methylene chloride/hexane and 25 ml of 60% methylene chloride in hexane. The 60% methylene chloride/hexane fractions were concentrated to 1 ml under a stream of dry nitrogen and applied to the tops of chromatography columns containing 1 g of 5% AX-21 activated carbon on silica gel. Each column was eluted with cyclohexane/methylene chloride (50:50 V/V) and cyclohexane/methanol/benzene (75:20:5 V/V) in the forward direction, and then with benzene in the reverse direction. Each benzene fraction was collected, spiked with recovery standards (1,2,3,4-TCDD-$^{13}C_{12}$ and 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$) and concentrated to a final volume of 20 uL.

PCDD/PCDF Analyses

The extracts were analyzed for the presence of PCDDs and PCDFs using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The instrumentation consisted of a Hewlett Packard Model 5890 gas chromatograph and a VG Model 70SE high resolution mass spectrometer. The capillary column was interfaced directly into the ion source of the mass spectrometer, thus providing the highest possible sensitivity while minimizing degradation of the chromatographic resolution.

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REPORT OF: CHEMICAL ANALYSES

PROJECT: NSP/SHERCO

DATE: June 15, 1990

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PCDD/PCDF Analyses (Cont.)

The mass spectrometer was operated in the electron impact ionization mode at a mass resolution of 10,000-11,000 (M/ΔM, 10 percent valley definition). This resolution is sufficient to resolve most interferences, such as PCBs, thus providing the highest level of confidence that the detected levels of PCDD/PCDF are not false positives resulting from interferences. Typical operating parameters for the HRGC/HRMS analyses are summarized in Table 2.

The data were acquired by selected-ion-recording (SIR) monitoring the groups of ion masses described in EPA method 8290. The five groups corresponded to the tetrachlorinated through octachlorinated congener classes. Each group contained three ion masses for the PCDDs (with the exception of TCDD which contained two ion masses), two ion masses for the PCDFs, the corresponding ion masses from the two isotopically labeled internal standards, and the ion mass characteristic of the polychlorinated diphenylether (PCDPE) which, if present, could cause false responses in the dibenzofuran channels. The third PCDD ion mass monitored in the pentachloro through octachlorodibenzo-p-dioxin groups prevented the possibility of misinterpretation of a polychlorinated biphenylene isomer as a PCDD. The two ion masses monitored for TCDD also fulfilled this purpose.

Each group of ion masses also contained a lock mass which was monitored during the analyses to detect suppressive interferences. It is particularly important to detect this type of interference since it can cause the quantification of congener class levels to be artificially high if it occurs during the elution of an internal standard or low if it occurs during the elution of the native analytes.

The lock mass was also used by the data system to automatically correct the mass focus of the instrument. The data system determined the centroid of the lock mass during each data acquisition cycle and corrected the mass focus of the analyte and internal standard ion masses to assure that the centers of the mass peaks were being monitored.

The criteria used to judge positive responses for the PCDD/PCDF isomer included:

- Simultaneous response at both ion masses of the PCDD or PCDF
- Signal to noise ratio equal to or greater than 2.5:1.0 for both ion masses

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REPORT OF: CHEMICAL ANALYSES

PROJECT: NSP/SHERCO

DATE: June 15, 1990

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PCDD/PCDF Analyses (Cont.)

The criteria used to judge positive responses for the PCDD/PCDF isomer included:

- Chlorine isotope ratio within 15% of the theoretical value
- Chromatographic retention time within -1 to +3 seconds of the authentic standards (where applicable)
- Chromatographic retention times within elution windows determined from analyses of standard mixtures
- Absence of simultaneous response between the PCDF and diphenylether ion traces

A list of the exact ion masses monitored for the determination of PCDD/PCDF isomers and the PCDPE interferences is presented in Table 3. Also included are the theoretical chlorine isotope ratios for the ten congener classes.

Quantification and Calculations

The PCDD/PCDF isomers were quantified by comparison of their responses to the responses of the labeled internal standards as described in the draft version of EPA Method 8290. Relative response factors were calculated from analyses of standard mixtures containing representatives of each of the PCDD/PCDF congener classes at five concentration levels, and each of the internal standards at one concentration level, as shown in Table 4. The PCDD/PCDF response factors were calculated by comparing the sum of the responses from the two ion masses monitored for each chlorine congener class to the sum of the responses from the two ion masses of the corresponding isotopically labeled internal standard. Table 5 shows the response factor at each of the calibration levels as well as the average response factors and the relative percent deviation for each. The formula for the response factor calculation is:

$$Rf = \frac{An \times Qis}{Ais \times Qn}$$

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Quantification and Calculations (Cont.)

where:

Rf = Response factor
An = Sum of integrated areas for native isomer
Qis = Quantity of labeled internal standard
Ais = Sum of integrated areas for labeled internal standard
Qn = Quantity of native isomer

The levels of PCDD/PCDF in the samples were quantified using the following equation:

$$C = \frac{An \times Qis}{Ais \times W \times Rf}$$

where:

C = Concentration of target isomer or congener class
An = Sum of integrated area for the target isomer or congener class
Qis = Amount of labeled internal standard added to the sample
Ais = Sum of integrated areas for the labeled internal standard
W = Sample weight, volume or area
Rf = Response factor

Each pair of ion mass peaks in the selected-ion-current chromatograms was evaluated manually to determine if it met the criteria for a PCDD or PCDF isomer. Areas of all peaks exhibiting correct ion ratios and having retention times within the correct windows were then summed for calculations of total congener concentrations. A summary of the high resolution initial calibration chlorine isotope ratios is presented in Table 6.

A limit of detection (LOD) based on producing a signal that is 2.5 times the noise level, was calculated for each undetected 2,3,7,8-substituted isomer of any tetra through octa chlorinated congener class. The noise heights used to calculate the detection limits were measured at the retention time of the specific isomer. The formula used for calculating the LOD is:

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Quantification and Calculations (Cont.)

$$LOD = \frac{Hn \times Qis \times 2.5}{His \times W \times Rf}$$

where:

LOD = Single isomer limit of detection
Hn = Sum of noise heights at native isomer retention time
Qis = Quantity of labeled internal standard
His = Sum of peak heights for labeled internal standard
W = Sample weight, volume or surface area
Rf = Response factor

The recovery of the 2,3,7,8-TCDD-$^{37}Cl_4$ enrichment efficiency standard and each $^{13}C_{12}$-labeled internal standard, relative to either 1,2,3,4-TCDD-$^{13}C_{12}$ or 1,2,3,7,8,9-HxCDD-$^{13}C_{12}$, was calculated using the following equation:

$$\%R = \frac{Ais \times Wrs \times 100\%}{Rfr \times Ars \times Wis}$$

where:

%R = Percent recovery of labeled internal standard
AIS = Sum of integrated areas of internal standards
Wrs = ng of recovery standard
Ars = Sum of integrated areas of recovery standard
Rfr = Response factor of the specific labeled internal standard relative to the recovery standard
Wis = ng of the labeled internal standard congener added to the sample prior to extraction

RESULTS

Sample analysis results are included in the following:

Table 7 - Summary of Pre-Sampling Spike Recoveries
Appendix A - PCDD/PCDF Analysis Results
Appendix B - Chain of Custody Documentation

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DISCUSSION

The recoveries of the isotopically labeled PCDD/PCDF internal standards in the MM-5 samples were typically in the range of 80-120%, indicating a high level of efficiency through the extraction and enrichment steps. Recoveries of several isomers were affected by interferences in the extract; these are distinguished by apparent recoveries of 140-150 percent. Since the method is based on isotope dilution, the quantifications are automatically corrected for variations in recovery and accurate measurements are obtained for the 2,3,7,8-substituted native isomers in the samples.

A laboratory method blank was prepared and analyzed with the sample extraction batch as part of our routine quality assurance/quality control procedures. The data sheet, included at the beginning of Appendix A, shows that it contained low background levels (typically less than 1 picograms/isomer) of selected PCDD/PCDF, with a somewhat higher level OCDD (130 picograms). These levels were typically one or more orders of magnitude below the levels measured for the same isomers in the actual samples. This indicates that the sample processing steps did not contribute significantly to the levels determined for the samples.

A laboratory matrix spike sample was also prepared and analyzed with the sample batch. The native analyte recoveries from this analysis, included at the end of Appendix A, typically ranged from 102-125%. This indicates a high degree of accuracy in the determination of the native isomers.

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REPORT OF: CHEMICAL ANALYSES

PROJECT: NSP/SHERCO

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REMARKS

The sample extracts will be retained for a period of 60 days from the date of this report and then discarded unless other arrangements are made. The raw mass spectral data will be archived on magnetic tape for a period of not less than one year.

TWIN CITY TESTING CORPORATION

Steven W. Hannan, Scientist
Mass Spectrometry Section

Charles V. Sueper
Dioxin Program Coordinator

Approved by:

Fred L. DeRoos, Ph.D.
Vice President
Chemistry

DJD/CVS/tls

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TABLE 1

Spike Levels of PCDD/PCDF Standards

Internal Standards:	Spike Level (ng)
2,3,7,8-TCDF-$^{13}C_{12}$	2.2*
2,3,7,8-TCDD-$^{13}C_{12}$	2.3*
1,2,3,7,8-PeCDF-$^{13}C_{12}$	2.4
2,3,4,7,8-PeCDF-$^{13}C_{12}$	2.2
1,2,3,4,7,8-PeCDD-$^{13}C_{12}$	1.7
1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	4.6
1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	5.1
1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	4.8
2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	5.0
1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	5.0
1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	5.2
1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	5.0
1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	5.0
1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	4.8
OCDD-$^{13}C_{12}$	14.3
Recovery Standards	
1,2,3,4-TCDD-$^{13}C_{12}$	2.0
1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	2.0
Enrichment Efficiency Standard	
2,3,7,8-TCDD-$^{37}Cl_4$	0.8

*These standards were spiked into the sampling media prior to sample collection.

TABLE 2

High Resolution PCDD/PCDF Analyses
HRGC/HRMS Operating Parameters

Mass Resolution	10,000-11,000 ($M/\Delta M$, 10% valley)
Electron Energy	32 electron volts
Accelerating Voltage	8,000 volts
Source Temperature	275°C
Preamplifier Gain	10^{-6} amp/volt
Multiplier Gain	~10^{5}
Chromatographic Column	60 M DB-5
Transfer Line Temperature	300°C
Injection Mode	Splitless
Carrier Gas	Helium
Carrier Flow Velocity	~30 cm/sec

TABLE 3

Exact Ion Masses Monitored for the Determination of PCDDs, PCDFs, and PCDPEs

Compound	Accurate Mass Mass 1	Mass 2	Mass 3	Theoretical Ratio Mass 2/Mass 3
Tetra-CDDs		319.8965	321.8936	0.77
Tetra-CDFs		303.9016	305.8987	0.77
Hexa-CDPEs	375.8364			
Penta-CDDs	353.8576	355.8546	357.8517	1.54
Penta-CDFs		339.8597	341.8567	1.54
Hepta-CDPEs	409.7974			
Hexa-CDDs	387.8187	389.8156	391.8127	1.23
Hexa-CDFs		373.8207	375.8178	1.23
Octa-CDPEs	445.7555			
Hepta-CDDs	421.7798	423.7766	425.7737	1.03
Hepta-CDFs		407.7817	409.7788	1.03
Nona-CDPEs	479.7165			
Octa-CDD	455.7409	457.7377	459.7347	0.88
Octa-CDF		441.7428	443.7398	0.88
Deca-CDPE	513.6775			

CDDs = Chlorinated Dibenzo-p-dioxins
CDFs = Chlorinated Dibenzofurans
CDPEs = Chlorinated Diphenylethers

TABLE 4

High Resolution Calibration Solutions

	Concentration (pg/uL)				
Native CDDs/CDFs	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
2,3,7,8-TCDD	0.5	2	10	40	200
2,3,7,8 TCDF	0.5	2	10	40	200
1,2,3,7,8-PeCDD	2.5	10	50	200	1000
1,2,3,7,8-PeCDF	2.5	10	50	200	1000
2,3,4,7,8-PeCDF	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
OCDD	5.0	20	100	400	2000
OCDF	5.0	20	100	400	2000
Internal Standards					
2,3,7,8-TCDD-$^{13}C_{12}$	100	100	100	100	100
2,3,7,8-TCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8-PeCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8-PeCDF-$^{13}C_{12}$	100	100	100	100	100
2,3,4,7,8-PeCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,7,8-HxCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,6,7,8-HxCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,6,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8,9-HxCDF-$^{13}C_{12}$	100	100	100	100	100
2,3,4,6,7,8-HxCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,6,7,8-HpCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,6,7,8-HpCDF-$^{13}C_{12}$	100	100	100	100	100
1,2,3,4,7,8,9-HpCDF-$^{13}C_{12}$	100	100	100	100	100
OCDD-$^{13}C_{12}$	200	200	200	200	200
Recovery Standards					
1,2,3,4-TCDD-$^{13}C_{12}$	100	100	100	100	100
1,2,3,7,8,9-HxCDD-$^{13}C_{12}$	100	100	100	100	100
Enrichment Efficiency Standard					
2,3,7,8-TCDD-$^{37}Cl_4$	0.5	2	10	40	200

TABLE #5
HRGC/HRMS Analyses VG70-VSE
HRGC/HRMS Initial Calibration (5-3-90) Summary of Response Factors

Isomer TCT Std #	LOW CS-1	CS-2	CS-3	CS-4	HIGH CS-5	Average RF	Percent RSD
2,3,7,8-TCDF	1.0054	0.9532	0.8615	0.8560	0.9161	0.9184	6.14
2,3,7,8-TCDD	0.8828	0.9231	0.9438	1.0287	1.0675	0.9692	7.07
1,2,3,7,8-PnCDF	1.0491	1.0913	1.1478	1.0457	1.1755	1.1019	4.73
2,3,4,7,8-PnCDF	0.8686	0.9565	0.9265	0.9175	0.9880	0.9314	4.29
1,2,3,7,8-PnCDD	0.9776	0.9754	0.9938	0.9424	1.0779	0.9934	4.57
1,2,3,4,7,8-HxCDF	1.1183	1.0610	1.0962	1.1378	1.0869	1.1000	2.40
1,2,3,6,7,8-HxCDF	1.0284	1.3237	1.1457	1.0879	1.3036	1.1779	9.94
1,2,3,7,8,9-HxCDF	1.2373	1.1448	0.9676	1.0951	1.2316	1.1353	8.77
2,3,4,6,7,8-HxCDF	1.2391	1.0650	1.0697	1.0977	1.1769	1.1297	6.01
1,2,3,4,7,8-HxCDD	0.9091	0.9446	0.8451	0.7785	0.8159	0.8586	7.06
1,2,3,6,7,8-HxCDD	0.7154	0.8030	1.0959	0.9727	1.0141	0.9202	15.22
1,2,3,7,8,9-HxCDD	0.6603	0.7438	0.8576	0.7933	1.0008	0.8112	14.13
1,2,3,4,6,7,8-HpCDF	1.5680	1.4974	1.5593	1.5355	1.5116	1.5344	1.76
1,2,3,4,7,7,8-HpCDF	1.1991	1.1825	1.2030	1.2035	1.3044	1.2185	3.58
1,2,3,4,6,7,8-HpCDD	0.9202	0.8727	0.9302	0.8771	0.9167	0.9034	2.62
OCDF	1.1504	1.2758	1.2374	1.2323	1.4273	1.2646	7.20
OCDD	1.1569	1.1227	1.1288	1.1342	1.1866	1.1459	2.05
TOTAL PeCDFs	0.9589	1.0239	1.0372	0.9816	1.0817	1.0167	4.24
TOTAL HxCDFs	1.1558	1.1486	1.0698	1.1046	1.1997	1.1357	3.93
TOTAL HxCDDs	0.7616	0.8305	0.9329	0.8482	0.9436	0.8633	7.85
TOTAL HpCDFs	1.3836	1.3400	1.3812	1.3695	1.4080	1.3764	1.61
2,3,7,8-TCDF*	1.4075	1.4323	1.3773	1.5208	1.4300	1.4336	3.34
2,3,7,8-TCDD*	0.8550	0.9081	0.8789	0.9232	1.0225	0.9175	6.27
2,3,7,8,-TCDD-C137	0.7311	0.6985	0.8267	0.9249	1.0575	0.8477	15.49
1,2,3,7,8-PnCDF*	1.2050	1.1707	0.7765	1.0126	0.9841	1.0298	14.86
2,3,4,7,8-PnCDF*	1.1567	1.1830	0.9732	1.1492	1.2310	1.1386	7.69
1,2,3,7,8-PnCDD*	0.6996	0.5533	0.5667	0.7354	0.6715	0.6453	11.26
1,2,3,4,7,8-HxCDF*	1.0120	1.1342	1.0667	1.0975	0.9947	1.0610	4.90
1,2,3,6,7,8-HxCDF*	1.3484	1.2448	1.2204	1.3120	1.0159	1.2283	9.41
1,2,3,7,8,9-HxCDF*	1.0905	1.1474	1.2890	1.1556	0.8813	1.1127	11.94
2,3,4,6,7,8-HxCDF*	0.7878	0.8228	0.8594	0.8150	0.8088	0.8188	2.86
1,2,3,4,7,8-HxCDD*	0.7387	0.7151	0.8569	0.8750	0.8440	0.8059	8.15
1,2,3,6,7,8-HxCDD*	1.0169	1.0802	0.9557	1.0085	0.7929	0.9708	10.03
1,2,3,4,6,7,8-HpCDF*	0.8090	0.8000	0.7908	0.7643	0.7500	0.7828	2.84
1,2,3,4,7,8,9-HpCDF*	0.6067	0.6418	0.6869	0.6530	0.6803	0.6538	4.41
1,2,3,4,6,7,8-HpCDD*	0.7500	0.7373	0.7285	0.7473	0.7683	0.7463	1.79
OCDD*	0.4730	0.4180	0.5019	0.4298	0.4816	0.4608	6.91

* = C13 Labeled Isomer

TABLE #6
HRGC/HRMS Analyses VG70-VSE
HRGC/HRMS Initial Calibration (5-3-90) Summary of Isotope Ratios

Isomer TCT Std #	LOW CS-1	CS-2	CS-3	CS-4	HIGH CS-5	Limits
2,3,7,8-TCDF	0.71	0.84	0.77	0.77	0.79	0.65-0.89
2,3,7,8-TCDD	0.78	0.71	0.75	0.76	0.75	0.65-0.89
1,2,3,7,8-PnCDF	1.55	1.79	1.67	1.54	1.73	1.32-1.78
2,3,4,7,8-PnCDF	1.63	1.76	1.62	1.56	1.67	1.32-1.78
1,2,3,7,8-PnCDD	1.59	1.78	1.64	1.49	1.69	1.32-1.78
1,2,3,4,7,8-HxCDF	1.07	1.28	1.42	1.24	1.27	1.05-1.43
1,2,3,6,7,8-HxCDF	1.42	1.32	1.08	1.26	1.28	1.05-1.43
1,2,3,7,8,9-HxCDF	1.41	1.19	1.22	1.23	1.24	1.05-1.43
2,3,4,6,7,8-HxCDF	1.34	1.27	1.27	1.28	1.25	1.05-1.43
1,2,3,4,7,8-HxCDD	1.35	1.15	1.28	1.18	1.31	1.05-1.43
1,2,3,6,7,8-HxCDD	1.09	1.16	1.31	1.20	1.37	1.05-1.43
1,2,3,7,8,9-HxCDD	1.25	1.15	1.30	1.22	1.31	1.05-1.43
1,2,3,4,6,7,8-HpCDF	1.10	1.02	1.00	1.01	1.01	0.88-1.20
1,2,3,4,7,7,8-HpCDF	0.89	1.06	0.98	1.02	1.01	0.88-1.20
1,2,3,4,6,7,8-HpCDD	0.95	1.07	1.00	1.02	1.04	0.88-1.20
OCDF	0.86	0.99	0.89	0.90	0.90	0.76-1.02
OCDD	0.89	0.92	0.86	0.86	0.89	0.76-1.02
1234-TCDD-REC	0.80	0.78	0.77	0.75	0.77	0.65-0.89
123789-HxCDD-REC	1.23	1.31	1.22	1.29	1.21	1.05-1.43
2,3,7,8-TCDF*	0.77	0.78	0.73	0.75	0.81	0.65-0.89
2,3,7,8-TCDD*	0.78	0.75	0.75	0.75	0.75	0.65-0.89
2,3,7,8,-TCDD-C137	****	****	****	****	****	*********
1,2,3,7,8-PnCDF*	1.71	1.68	1.65	1.50	1.53	1.32-1.78
2,3,4,7,8-PnCDF*	1.71	1.71	1.60	1.48	1.52	1.32-1.78
1,2,3,7,8-PnCDD*	1.56	1.47	1.58	1.57	1.49	1.32-1.78
1,2,3,4,7,8-HxCDF*	0.57	0.56	0.53	0.55	0.58	0.43-0.59
1,2,3,6,7,8-HxCDF*	0.59	0.55	0.53	0.57	0.58	0.43-0.59
1,2,3,7,8,9-HxCDF*	0.58	0.53	0.43	0.55	0.57	0.43-0.59
2,3,4,6,7,8-HxCDF*	0.58	0.54	0.51	0.55	0.59	0.43-0.59
1,2,3,4,7,8-HxCDD*	1.23	1.32	1.21	1.25	1.22	1.05-1.43
1,2,3,6,7,8-HxCDD*	1.27	1.38	1.25	1.32	1.22	1.05-1.43
1,2,3,4,6,7,8-HpCDF*	0.46	0.45	0.46	0.46	0.45	0.37-0.51
1,2,3,4,7,8,9-HpCDF*	0.45	0.45	0.44	0.46	0.46	0.37-0.51
1,2,3,4,6,7,8-HpCDD*	0.99	1.02	1.04	1.03	1.01	0.88-1.20
OCDD*	0.90	0.90	0.92	0.92	0.92	0.76-1.02

* = C13 Labeled Isomer

TABLE 7

INTERPOLL LABORATORIES, INC.
SUMMARY OF PRE-SAMPLING* SPIKE RECOVERIES

Client ID	2,3,7,8-TCDF-$^{13}C_{12}$	2,3,7,8-TCDD-$^{13}C_{12}$
T3R0 Field Blank	90%	91%
T3R1	92%	93%
T3R2	92%	94%
T3R3	92%	96%

All values are expressed as percent recovered and were calculated using the following equation:

$$\% \text{ Recovery} = \frac{\% \text{ Recovery of } ^{13}C_{12} \text{ Standard}}{\% \text{ Recovery of } ^{37}Cl_4 \text{ Standard}} \times 100\%$$

*2.2 ng of $^{13}C_{12}$-2,3,7,8-TCDF and 2.3 ng of $^{13}C_{12}$-2,3,7,8-TCDD were spiked into the sampling media prior to field sampling.

Laboratory No: 4410 90-4052

APPENDIX A

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*******************************
*TWIN CITY TESTING CORPORATION*
* PCDF/PCDD ANALYSIS RESULTS  *
*******************************
 Client....INTERPOLL
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Sample ID (TCT#).....METHOD BLANK 4-17-90
Analysis Date.....5-8-90
Filename.....V00508E
Analyst.....DJD
Sample Amount.....1.0000 sample
ICAL Date.....5-3-90
CCAL Filename.....V00508A

NATIVE ISOMERS	CONC. ng/sample	DL ng/sample
2378-TCDF	0.0120	-----
TOTAL TCDF	0.0290	-----
2378-TCDD	nd	0.0074
TOTAL TCDD	0.0430	-----
12378-PeCDF	0.0035	-----
23478-PeCDF	0.0046	-----
TOTAL PeCDF	0.0420	-----
12378-PeCDD	nd	0.0021
TOTAL PeCDD	0.0170	-----
123478-HxCDF	0.0130	-----
123678-HxCDF	0.0034	-----
123789-HxCDF	0.0073	-----
234678-HxCDF	nd	0.0015
TOTAL HxCDF	0.0450	-----
123478-HxCDD	nd	0.0012
123678-HxCDD	nd	0.0031
123789-HxCDD	nd	0.0024
TOTAL HxCDD	0.0370	-----
1234678-HpCDF	nd	0.0150
1234789-HpCDF	nd	0.0024
TOTAL HpCDF	0.0074	-----
1234678-HpCDD	0.0150	-----
TOTAL HpCDD	0.0280	-----
OCDF	0.0200	-----
OCDD	0.1300	-----

INTERNAL STANDARDS	ng's ADDED	PERCENT RECOVERY
2378-TCDF-C13....	2.00	46
2378-TCDD-C13....	2.00	40
12378-PeCDF-C13..	2.00	51
23478-PeCDF-C13..	2.00	69
12378-PeCDD-C13..	2.00	72
123478-HxCDF-C13.	2.00	63
123678-HxCDF-C13.	2.00	53
123789-HxCDF-C13.	2.00	65
234678-HxCDF-C13.	2.00	65
123478-HxCDD-C13.	2.00	59
123678-HxCDD-C13.	2.00	65
1234678-HpCDF-C13	2.00	59
1234789-HpCDF-C13	2.00	62
1234678-HpCDD-C13	2.00	55
OCDD-C13.....	4.00	38
1234-TCDD-C13....	2.00	na
123789-HxCDD-C13.	2.00	na
2378-TCDD-C137...	0.80	53

Total 2378-TCDD
Equivalence = 0.0029 ng/sample
(Using EPA 8290 Factors)

CONC= Concentrations, calculated as described in EPA method 8290.

DL= Detection limits, calculated as described in EPA method 8290.

na= not applicable

nd= not detected

TCT Invoice Number....4410 90-4052

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*********************************
*TWIN CITY TESTING CORPORATION*
* PCDF/PCDD ANALYSIS RESULTS   *
*********************************
 Client....INTERPOLL
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Sample ID (Client's#)....T3R0 FIELD BLANK 9242-(54-57)
Sample ID (TCT#).....187226
Analysis Date.....5-8-90
Filename.....V00508F
Analyst.....BB
Sample Amount.....1.0000 sample
ICAL Date.....5-3-90
CCAL Filename.....V00508A

NATIVE ISOMERS	CONC. ng/sample	DL ng/sample
2378-TCDF	0.0270	-----
TOTAL TCDF	0.1300	-----
2378-TCDD	nd	0.0036
TOTAL TCDD	0.0450	-----
12378-PeCDF	0.0095	-----
23478-PeCDF	0.0280	-----
TOTAL PeCDF	0.2200	-----
12378-PeCDD	0.0070	-----
TOTAL PeCDD	0.0580	-----
123478-HxCDF	0.0740	-----
123678-HxCDF	0.0330	-----
123789-HxCDF	0.0510	-----
234678-HxCDF	0.0055	-----
TOTAL HxCDF	0.2700	-----
123478-HxCDD	0.0120	-----
123678-HxCDD	0.0210	-----
123789-HxCDD	0.0280	-----
TOTAL HxCDD	0.1900	-----
1234678-HpCDF	0.1600	-----
1234789-HpCDF	0.0480	-----
TOTAL HpCDF	0.3300	-----
1234678-HpCDD	0.2000	-----
TOTAL HpCDD	0.3400	-----
OCDF	0.6000	-----
OCDD	0.5200	-----

INTERNAL STANDARDS	ng's ADDED	PERCENT RECOVERY
2378-TCDF-C13....	4.20	88
2378-TCDD-C13....	4.30	89
12378-PeCDF-C13..	2.00	77
23478-PeCDF-C13..	2.00	100
12378-PeCDD-C13..	2.00	94
123478-HxCDF-C13.	2.00	108
123678-HxCDF-C13.	2.00	90
123789-HxCDF-C13.	2.00	106
234678-HxCDF-C13.	2.00	122
123478-HxCDD-C13.	2.00	111
123678-HxCDD-C13.	2.00	104
1234678-HpCDF-C13	2.00	96
1234789-HpCDF-C13	2.00	114
1234678-HpCDD-C13	2.00	83
OCDD-C13.....	4.00	90
1234-TCDD-C13....	2.00	na
123789-HxCDD-C13.	2.00	na
2378-TCDD-Cl37...	0.80	98

Total 2378-TCDD
Equivalence = 0.0155 ng/sample
(Using EPA 8290 Factors)

CONC= Concentrations, calculated as described in EPA method 8290.

DL= Detection limits, calculated as described in EPA method 8290.

na= not applicable

nd= not detected

TCT Invoice Number....4410 90-4052

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*******************************
*TWIN CITY TESTING CORPORATION*
* PCDF/PCDD ANALYSIS RESULTS  *
*******************************
 Client....INTERPOLL
```

Sample ID (Client's#)....T3R1 9242-(58-61)
Sample ID (TCT#).....187228
Analysis Date.....5-8-90
Filename.....V00508G
Analyst.....BB
Sample Amount.....1.0000 sample
ICAL Date.....5-3-90
CCAL Filename.....V00508A

NATIVE ISOMERS	CONC. ng/sample	DL ng/sample
2378-TCDF	0.2100	-----
TOTAL TCDF	1.2000	-----
2378-TCDD	0.0043	-----
TOTAL TCDD	0.1500	-----
12378-PeCDF	0.0920	-----
23478-PeCDF	0.2000	-----
TOTAL PeCDF	2.1000	-----
12378-PeCDD	0.0250	-----
TOTAL PeCDD	0.2900	-----
123478-HxCDF	1.1000	-----
123678-HxCDF	0.3600	-----
123789-HxCDF	0.9700	-----
234678-HxCDF	0.0680	-----
TOTAL HxCDF	5.3000	-----
123478-HxCDD	0.0470	-----
123678-HxCDD	0.1300	-----
123789-HxCDD	0.1100	-----
TOTAL HxCDD	1.2000	-----
1234678-HpCDF	5.1000	-----
1234789-HpCDF	2.4000	-----
TOTAL HpCDF	19.0000	-----
1234678-HpCDD	2.4000	-----
TOTAL HpCDD	4.4000	-----
OCDF	62.0000	-----
OCDD	13.0000	-----

INTERNAL STANDARDS	ng's ADDED	PERCENT RECOVERY
2378-TCDF-C13....	4.20	101
2378-TCDD-C13....	4.30	102
12378-PeCDF-C13..	2.00	57
23478-PeCDF-C13..	2.00	87
12378-PeCDD-C13..	2.00	80
123478-HxCDF-C13.	2.00	117
123678-HxCDF-C13.	2.00	109
123789-HxCDF-C13.	2.00	116
234678-HxCDF-C13.	2.00	141
123478-HxCDD-C13.	2.00	151
123678-HxCDD-C13.	2.00	115
1234678-HpCDF-C13	2.00	108
1234789-HpCDF-C13	2.00	137
1234678-HpCDD-C13	2.00	104
OCDD-C13.....	4.00	127
1234-TCDD-C13....	2.00	na
123789-HxCDD-C13.	2.00	na
2378-TCDD-C137...	0.80	110

Total 2378-TCDD
Equivalence = 0.1200 ng/sample
(Using EPA 8290 Factors)

CONC= Concentrations, calculated as described in EPA method 8290.

DL= Detection limits, calculated as described in EPA method 8290.

na= not applicable

nd= not detected

TCT Invoice Number....4410 90-4052

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*******************************
*TWIN CITY TESTING CORPORATION*
* PCDF/PCDD ANALYSIS RESULTS  *
*******************************
 Client....INTERPOLL
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Sample ID (Client's#)....T3R2 9242-(62-65)
Sample ID (TCT#).....187229
Analysis Date.....5-8-90
Filename.....V00508H
Analyst.....BB
Sample Amount.....1.0000 sample
ICAL Date.....5-3-90
CCAL Filename.....V00508A

NATIVE ISOMERS	CONC. ng/sample	DL ng/sample
2378-TCDF	0.0760	-----
TOTAL TCDF	0.3800	-----
2378-TCDD	0.0042	-----
TOTAL TCDD	0.0760	-----
12378-PeCDF	0.0360	-----
23478-PeCDF	0.0940	-----
TOTAL PeCDF	0.8100	-----
12378-PeCDD	0.0180	-----
TOTAL PeCDD	0.1400	-----
123478-HxCDF	0.4400	-----
123678-HxCDF	0.1800	-----
123789-HxCDF	0.3400	-----
234678-HxCDF	0.0310	-----
TOTAL HxCDF	2.4000	-----
123478-HxCDD	0.0320	-----
123678-HxCDD	0.0620	-----
123789-HxCDD	0.0940	-----
TOTAL HxCDD	0.5900	-----
1234678-HpCDF	2.0000	-----
1234789-HpCDF	0.9600	-----
TOTAL HpCDF	7.3000	-----
1234678-HpCDD	0.8800	-----
TOTAL HpCDD	1.7000	-----
OCDF	19.0000	-----
OCDD	4.3000	-----

INTERNAL STANDARDS	ng's ADDED	PERCENT RECOVERY
2378-TCDF-C13....	4.20	89
2378-TCDD-C13....	4.30	91
12378-PeCDF-C13..	2.00	61
23478-PeCDF-C13..	2.00	74
12378-PeCDD-C13..	2.00	68
123478-HxCDF-C13.	2.00	84
123678-HxCDF-C13.	2.00	27
123789-HxCDF-C13.	2.00	75
234678-HxCDF-C13.	2.00	102
123478-HxCDD-C13.	2.00	83
123678-HxCDD-C13.	2.00	61
1234678-HpCDF-C13	2.00	71
1234789-HpCDF-C13	2.00	85
1234678-HpCDD-C13	2.00	61
OCDD-C13.....	4.00	89
1234-TCDD-C13....	2.00	na
123789-HxCDD-C13.	2.00	na
2378-TCDD-C137...	0.80	97

Total 2378-TCDD
Equivalence = 0.0579 ng/sample
(Using EPA 8290 Factors)

CONC= Concentrations, calculated as described in EPA method 8290.

DL= Detection limits, calculated as described in EPA method 8290.

na= not applicable

nd= not detected

TCT Invoice Number....4410 90-4052

```
*********************************
*TWIN CITY TESTING CORPORATION*
* PCDF/PCDD ANALYSIS RESULTS   *
*********************************
 Client....INTERPOLL
```

Sample ID (Client's#)....T3R3 9242-(66-99)
Sample ID (TCT#).....187234
Analysis Date.....5-8-90
Filename.....V00508I
Analyst.....BB
Sample Amount.....1.0000 sample
ICAL Date.....5-3-90
CCAL Filename.....V00508A

NATIVE ISOMERS	CONC. ng/sample	DL ng/sample
2378-TCDF	0.0360	-----
TOTAL TCDF	0.1800	-----
2378-TCDD	0.0029	-----
TOTAL TCDD	0.0540	-----
12378-PeCDF	0.0190	-----
23478-PeCDF	0.0480	-----
TOTAL PeCDF	0.4600	-----
12378-PeCDD	0.0084	-----
TOTAL PeCDD	0.0630	-----
123478-HxCDF	0.2500	-----
123678-HxCDF	0.0820	-----
123789-HxCDF	0.2000	-----
234678-HxCDF	0.0260	-----
TOTAL HxCDF	1.2000	-----
123478-HxCDD	0.0230	-----
123678-HxCDD	0.0210	-----
123789-HxCDD	0.0210	-----
TOTAL HxCDD	0.3200	-----
1234678-HpCDF	1.2000	-----
1234789-HpCDF	0.6400	-----
TOTAL HpCDF	4.4000	-----
1234678-HpCDD	0.5300	-----
TOTAL HpCDD	0.8900	-----
OCDF	15.0000	-----
OCDD	2.7000	-----

INTERNAL STANDARDS	ng's ADDED	PERCENT RECOVERY
2378-TCDF-C13....	4.20	78
2378-TCDD-C13....	4.30	82
12378-PeCDF-C13..	2.00	43
23478-PeCDF-C13..	2.00	57
12378-PeCDD-C13..	2.00	52
123478-HxCDF-C13.	2.00	135
123678-HxCDF-C13.	2.00	113
123789-HxCDF-C13.	2.00	121
234678-HxCDF-C13.	2.00	146
123478-HxCDD-C13.	2.00	97
123678-HxCDD-C13.	2.00	100
1234678-HpCDF-C13	2.00	96
1234789-HpCDF-C13	2.00	127
1234678-HpCDD-C13	2.00	104
OCDD-C13.....	4.00	127
1234-TCDD-C13....	2.00	na
123789-HxCDD-C13.	2.00	na
2378-TCDD-Cl37...	0.80	85

Total 2378-TCDD
Equivalence = 0.0295 ng/sample
(Using EPA 8290 Factors)

CONC= Concentrations, calculated as described in EPA method 8290.

DL= Detection limits, calculated as described in EPA method 8290.

na= not applicable

nd= not detected

TCT Invoice Number....4410 90-4052

```
*******************************
*TWIN CITY TESTING CORPORATION*
* PCDF/PCDD ANALYSIS RESULTS  *
*******************************
     Client....INTERPOLL
```

Sample ID (TCT#).....SPIKE
Analysis Date.....5-8-90
Filename.....V00508P
Analyst.....BB
Sample Amount.....1.0000 sample
ICAL Date.....5-3-90
CCAL Filename.....V00508A

NATIVE ISOMERS	Qs (NG)	Qm (NG)	% REC.
2378-TCDF	0.80	1.00	125
TOTAL TCDF	0.80	1.00	125
2378-TCDD	0.80	1.00	125
TOTAL TCDD	0.80	1.00	125
12378-PeCDF	4.00	4.80	120
23478-PeCDF	4.00	4.80	120
TOTAL PeCDF	8.00	9.60	120
12378-PeCDD	4.00	4.60	115
TOTAL PeCDD	4.00	4.60	115
12347t-HxCDF	4.00	4.40	110
123678-HxCDF	4.00	4.80	120
123789-HxCDF	4.00	4.70	118
234678-HxCDF	4.00	4.70	118
TOTAL HxCDF	16.00	19.00	119
123478-HxCDD	4.00	4.30	108
123678-HxCDD	4.00	4.90	123
123789-HxCDD	4.00	4.10	103
TOTAL HxCDD	12.00	13.00	108
1234678-HpCDF	4.00	4.20	105
1234789-HpCDF	4.00	4.50	113
TOTAL HpCDF	8.00	8.70	109
1234678-HpCDD	4.00	4.90	123
TOTAL HpCDD	4.00	4.90	123
OCDF	8.00	11.00	138
OCDD	8.00	9.50	119

INTERNAL STANDARD	ng's ADDED	PERCENT RECOVERY
2378-TCDF-C13....	2.00	48
2378-TCDD-C13....	2.00	48
12378-PeCDF-C13..	2.00	60
23478-PeCDF-C13..	2.00	73
12378-PeCDD-C13..	2.00	74
123478-HxCDF-C13.	2.00	68
123678-HxCDF-C13.	2.00	50
123789-HxCDF-C13.	2.00	62
234678-HxCDF-C13.	2.00	69
123478-HxCDD-C13.	2.00	80
123678-HxCDD-C13.	2.00	58
1234678-HpCDF-C13	2.00	71
1234789-HpCDF-C13	2.00	82
1234678-HpCDD-C13	2.00	60
OCDD-C13.....	4.00	61
1234-TCDD-C13....	2.00	XX
123789-HxCDD-C13.	2.00	XX
2378-TCDD-Cl37...	0.80	63

Qs= Quantity spiked.
Qm= Quantity measured.
%REC.= Percent recovered.

TCT Invoice Number....4410 90-4052

APPENDIX B

Interpoll Laboratories
(612)786-6020

Sample Custody Transfer Form

Job NSP / Sherco Date 3-30-90

Interlaboratory transfer:
Person in custody of samples _____ Init. _____
Person to receive custody [illegible] Init. LD.

Out of laboratory transfer:
Date shipped 3-30-90 Date received _____
Method of shipment Car Carrier Bill Drake
Person releasing samples Bill Drake Init. B.D.
Person receiving custody [illegible] Init. LD.

	Sample Log Number	Initials of Person in Custody of Samples	Initials of Person Receiving Samples
T_3R_0 Field Blank	1. 9242-54	BD	LD.
	2. -55	BD	LD.
	3. -56	BD	LD.
	4. -57	BD	LD
T_3R_1	5. 9242-58	BD	LD.
	6. -59	BD	LD.
	7. -60	BD	LD.
	8. -61	BD	LD.
T_3R_2	9. 9242-62	BD	LD.
	10. -63	BD	LD.
	11. -64	BD	LD
	12. -65	BD	LD.
T_3R_3	13. 9242-66	BD	LD.
	14. -67	BD	LD.
	15. -68	BD	LD
	16. -69	BD	LD.
	17.		
	18.		

Storage Instructions: _____

Instructions for Receiving Laboratory:

1. Fill out form and return to Interpoll Laboratories.
2. Initiate your laboratory chain-of-custody. When analysis is complete, mail copy of your chain-of-custody documents with analytical results.
3. Store samples for four (4) months.

S-365

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP / Sherco

Date of Sampling 3-28-90 Site Unit No. 3 Stack

Test 3 Run 0 . Team Leader Duane VanHoeier

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koehler Title Field Tech Eng

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon & Chain of Custody Sheet

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No. 0300	1 ✓	9242-54
2.	Solvent Rinse	1 ✓	-55
3.	Water Rinse	1 ✓	-56
4.	Condensate Trap	1 ✓	-57
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature [illegible] Title SAMPLE RECEIVING

Date of receipt 4/2/90 — 3/30/90 Time of receipt _____ HRS

S-0157RR-1

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP / Sherco

Date of Sampling 3-28-90 Site Unit No. 3 Stack

Test 3 Run 1 Team Leader Duane Venthoever

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Kaehler Title Field Tech Eng.

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon & Chain of Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No. 0301	1 ✓	9242-58
2.	Solvent Rinse	1 ✓	-59
3.	XAD-2 Resin	1 ✓	-60
4.	Condensate Trap	1 ✓	-61
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature [illegible] Title SAMPLE RECEIVING

Date of receipt 3/30/90 Time of receipt _____ HRS

S-0157RR-1

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP / Sherco

Date of Sampling 2-25-90 Site Unit No. 3 Stack

Test 3 Run 2 Team Leader Dennis Unstheuer

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koehler Title Field Tech Eng.

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon + Chain of Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No 0302	1 ✓	9242-62
2.	Solvent Rinse	1 ✓	-63
3.	XAD-2 Resin	1 ✓	-64
4.	Condensate Trap	1 ✓	-65
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature [illegible] Title SAMPLE RECEIVING

Date of receipt 3/30/90 Time of receipt _____ HRS

S-0157RR-1

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP/Sherco

Date of Sampling 3-29-90 Site Unit No 3 Stack

Test 3 Run 3 Team Leader Duane VanNoever

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Goebler Title Field Tech Eng

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon + Chain of Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No 0503	1 ✓	9242-66
2.	Solvent Rinse	1 ✓	-67
3.	XAD-2 Resin	1 ✓	-68
4.	Condensate Trap	1 ✓	-69
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

187234

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature [illegible] Title SAMPLE RECEIVING

Date of receipt 3/30/90 Time of receipt _____ HRS

S-0157RR-1

APPENDIX F

CHAIN OF CUSTODY SHEETS

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP / Sherco

Date of Sampling 3-28-90 Site Unit No. 3 Stack

Test 3 Run 0 Team Leader Duane Vanhoeier

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koebler Title Field Tech Eng

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon & Chain of Custody Sheets

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No. 0300	1	9242-54
2.	Solvent Rinse	1	-55
3.	Water Rinse	1	-56
4.	Condensate Trap	1	-57
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature _____ Title _____

Date of receipt _____ Time of receipt _____ HRS

S-0157RR-1

Sample Check-In at Interpoll Labs

Laboratory person receiving samples:

Signature Bill Dub Title Lab analyst

Date of receipt 3-30-90 Time of receipt 0800 HRS

Sample storage location secured walk-in cooler

Special storage conditions? _____

Analysis

	Sample Log No.	Date of Analysis	Method of Analysis	Initials of Analyst
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP / Sherco

Date of Sampling 3-28-90 Site Unit No. 3 Stack

Test 3 Run 1 Team Leader Duane Vanhoover

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Kaehler Title Field Tech Eng.

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon + Chain of Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No. 0301	1	9242-58
2.	Solvent Rinse	1	-59
3.	XAD-2 Resin	1	-60
4.	Condensate Trap	1	-61
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature _____ Title _____

Date of receipt _____ Time of receipt _____ HRS

S-0157RR-1

Sample Check-In at Interpoll Labs

Laboratory person receiving samples:

Signature Bill Drake Title Lab analyst

Date of receipt 3-30-90 Time of receipt 0800 HRS

Sample storage location secured walk-in cooler

Special storage conditions? _____

Analysis

	Sample Log No.	Date of Analysis	Method of Analysis	Initials of Analyst
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP / Sherco

Date of Sampling 3-29-90 Site Unit No 3 Stack

Test 3 Run 2 Team Leader Duane Vanhoever

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Kaehler Title Field Tech Eng.

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon + Chain of Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No 0302	1	9242-62
2.	Solvent Rinse	1	-63
3.	XAD-2 Resin	1	-64
4.	Condensate Trap	1	-65
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature _____ Title _____

Date of receipt _____ Time of receipt _____ HRS

S-0157RR-1

Sample Check-In at Interpoll Labs

Laboratory person receiving samples:

Signature Bill Drake Title Lab analyst

Date of receipt 3-30-90 Time of receipt 0800 HRS

Sample storage location secured walk-in cooler

Special storage conditions? _____

Analysis

	Sample Log No.	Date of Analysis	Method of Analysis	Initials of Analyst
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job NSP/Sherco

Date of Sampling 3-29-90 Site Unit No 3 Stack

Test 3 Run 3 Team Leader Duane VanHoeven

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koehler Title Field Tech Eng

Location at which recovery was done Sherco Meeting Room

Special transport/storage requirements? Teflon + Chain of Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Filter No 0303	1	9242-66
2.	Solvent Rinse	1	-67
3.	XAD-2 Resin	1	-68
4.	Condensate Trap	1	-69
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			

Sample Transport

Method of transport Company Van

Recipient of samples upon recovery (if not recovery person):

Signature _____ Title _____

Date of receipt _____ Time of receipt _____ HRS

S-0157RR-1

Sample Check-In at Interpoll Labs

Laboratory person receiving samples:

Signature Bill Dale Title Lab. Analyst

Date of receipt 3-30-90 Time of receipt 0500 HRS

Sample storage location secured walk-in cooler

Special storage conditions? _____

Analysis

	Sample Log No.	Date of Analysis	Method of Analysis	Initials of Analyst
1.	0			
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature

S-0157RR-2

APPENDIX G

PROCEDURES

PAH and Dioxin Procedures

Interpoll Laboratories
4500 Ball Road N.E.
Circle Pines, Minnesota 55014

Telephone (612)786-6020
Fascimile (612)786-7854

COLLECTION OF SEMIVOLATILE COMPOUNDS

EPA METHOD 0010

(Interpoll Method II-8788 - Version 1.0)

EPA Method SW846 USEPA Office of Solid Waste and Emergency Response 3rd Ed., November 1986: Test Methods for Evaluating Solid Waste Volume 2: Field Manual Physical/Chemical Methods Method 0010.

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

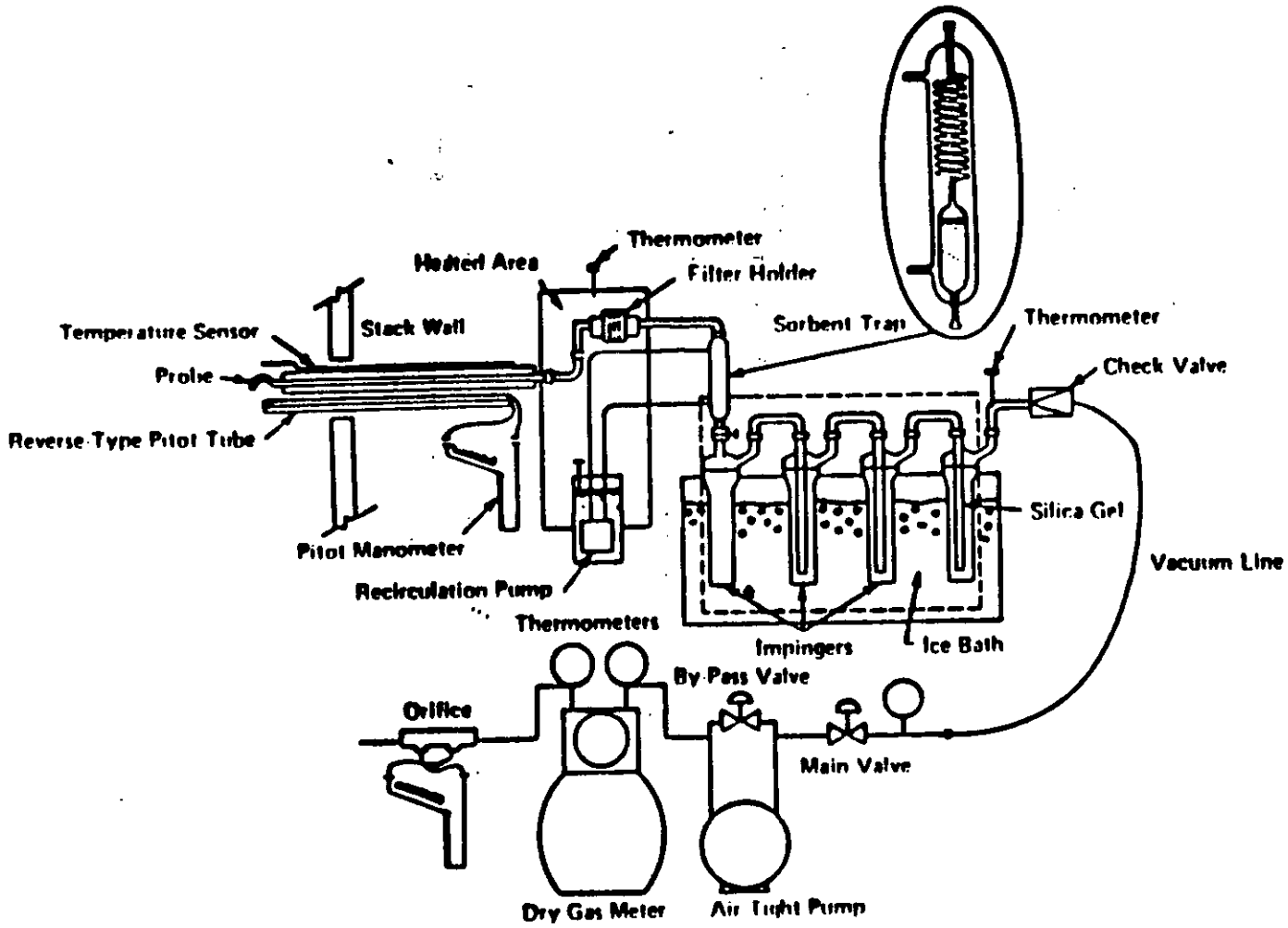

Figure 1. Modified Method 5 Sampling Train.

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 ± 14°C (248 ± 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 ± 3°C (62.5 ± 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.

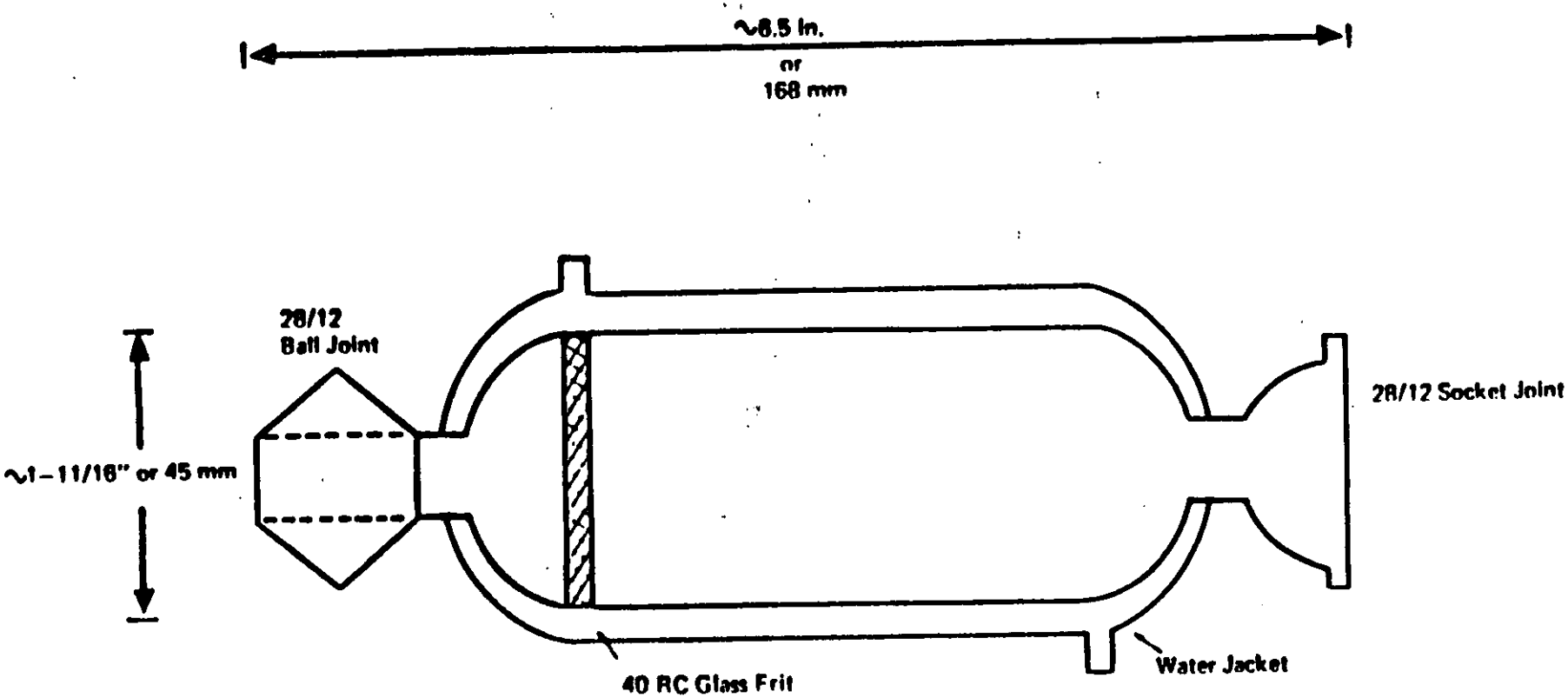

Figure 2. Adsorbent Sampling System.

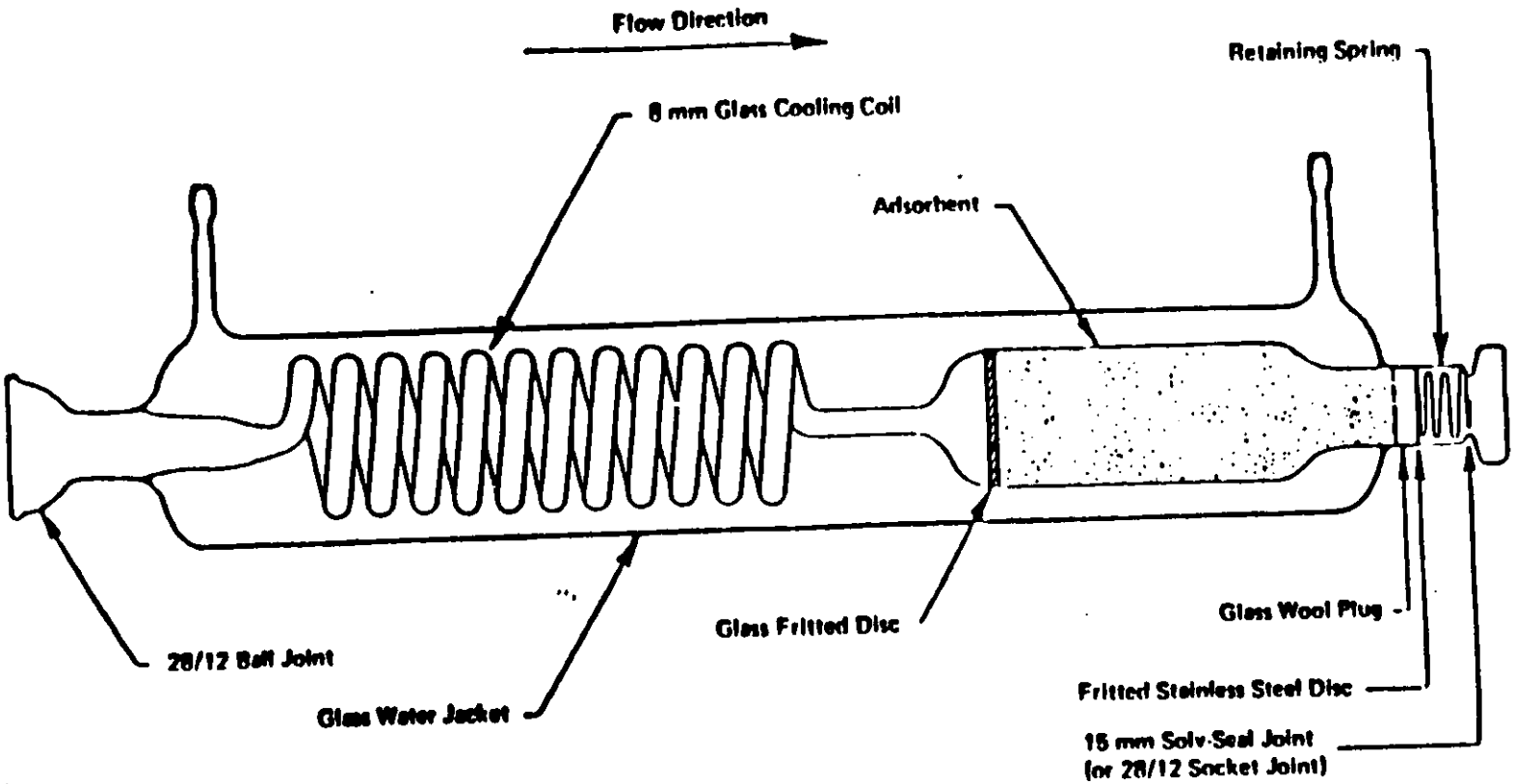

Figure 3. Adsorbent Sampling System.

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 ± 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 <u>not</u> 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

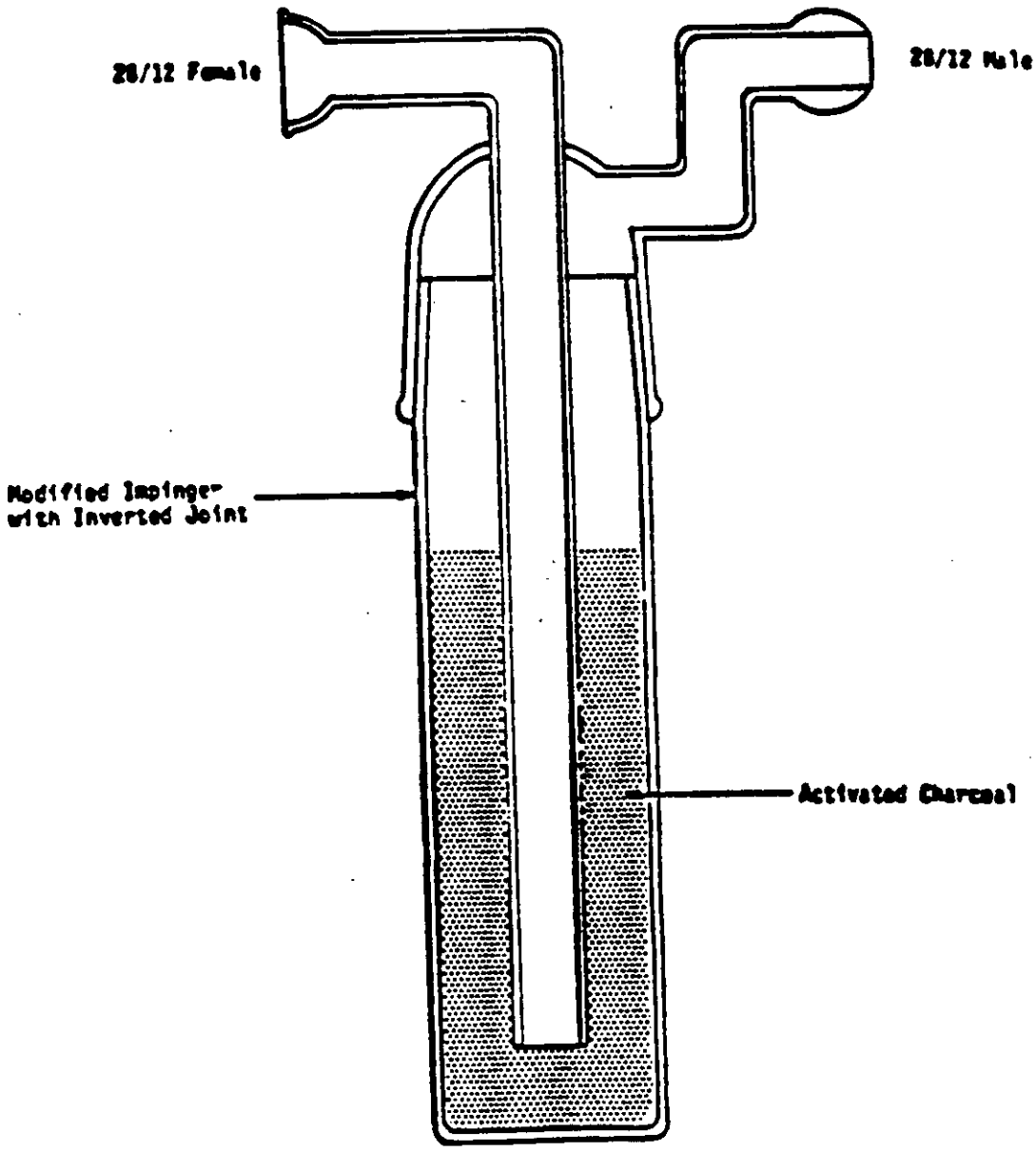

Figure 4. Leak-check impinger.

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 __________		Ambient Temperature __________
Location __________		Barometric Pressure __________
Operator __________		Assumed Moisture % __________
Date __________		Probe Length, m (ft) __________
Run No. __________		Nozzle Identification No. __________
Sample Box No. __________		Average Calibrated Nozzle Diameter, cm (in) __________
Meter Box No. __________		Probe Heater Setting __________
Meter ΔH@ __________		Leak Rate, m^3/min, (cfm) __________
C Factor __________	Schematic of Stack Cross Section	Probe Liner Material __________
Pitot Tube Coefficient C_p __________		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 (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 Sorbent 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 with 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 applicable). The particulate filter (and cyclone catch, if applicable) is transferred 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.

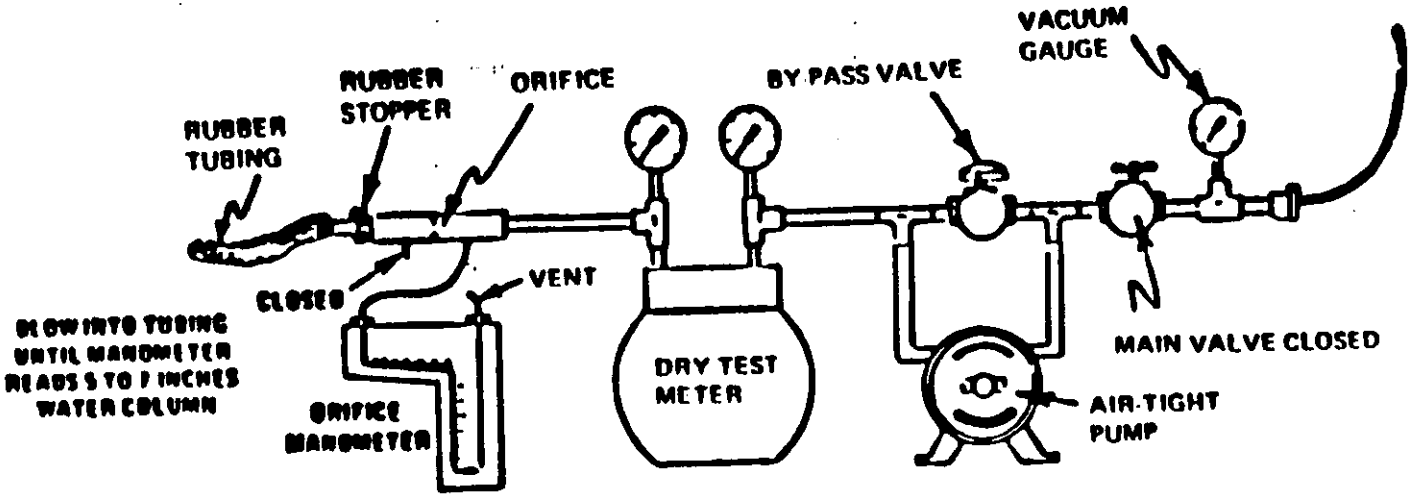

Figure 8. Leak check of meter box.

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).

9.7 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:

$$V_{m(std)} = V_m Y \frac{T_{std}}{T_m} \frac{P_{bar} + \Delta H/13.6}{P_{std}} = K_1 V_m Y \frac{P_{bar} + \Delta H/13.6}{T_m} \qquad (1)$$

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:

$$V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p$$

and substitute only for those leakage rates (L_i 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:

$$I = \frac{100\ T_s[K_3F_{1c} + (V_m/T_m)\ (P_{bar} + \Delta H/13.6)]}{60\theta V_s P_s A_n} \qquad (4)$$

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:

$$\frac{(WF)\ (POHC_1\ conc)}{100}\ \frac{(100-\%DRE)}{100} = \text{Max } POHC_1 \text{ Mass} \qquad (6)$$

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:

$$\frac{\text{Max POHC}_i \text{ Mass}}{DV_{eff(std)}} = \text{Max POHC}_i \text{ conc} \qquad (7)$$

where:

$DV_{eff(std)}$ = volumetric flow rate of exhaust gas, dscm (dscf).

$POHC_i$ 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:

$$\frac{LDL_{POHC} \times 10}{POHC_i \text{ conc}} = V_{TBC} \qquad (8)$$

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

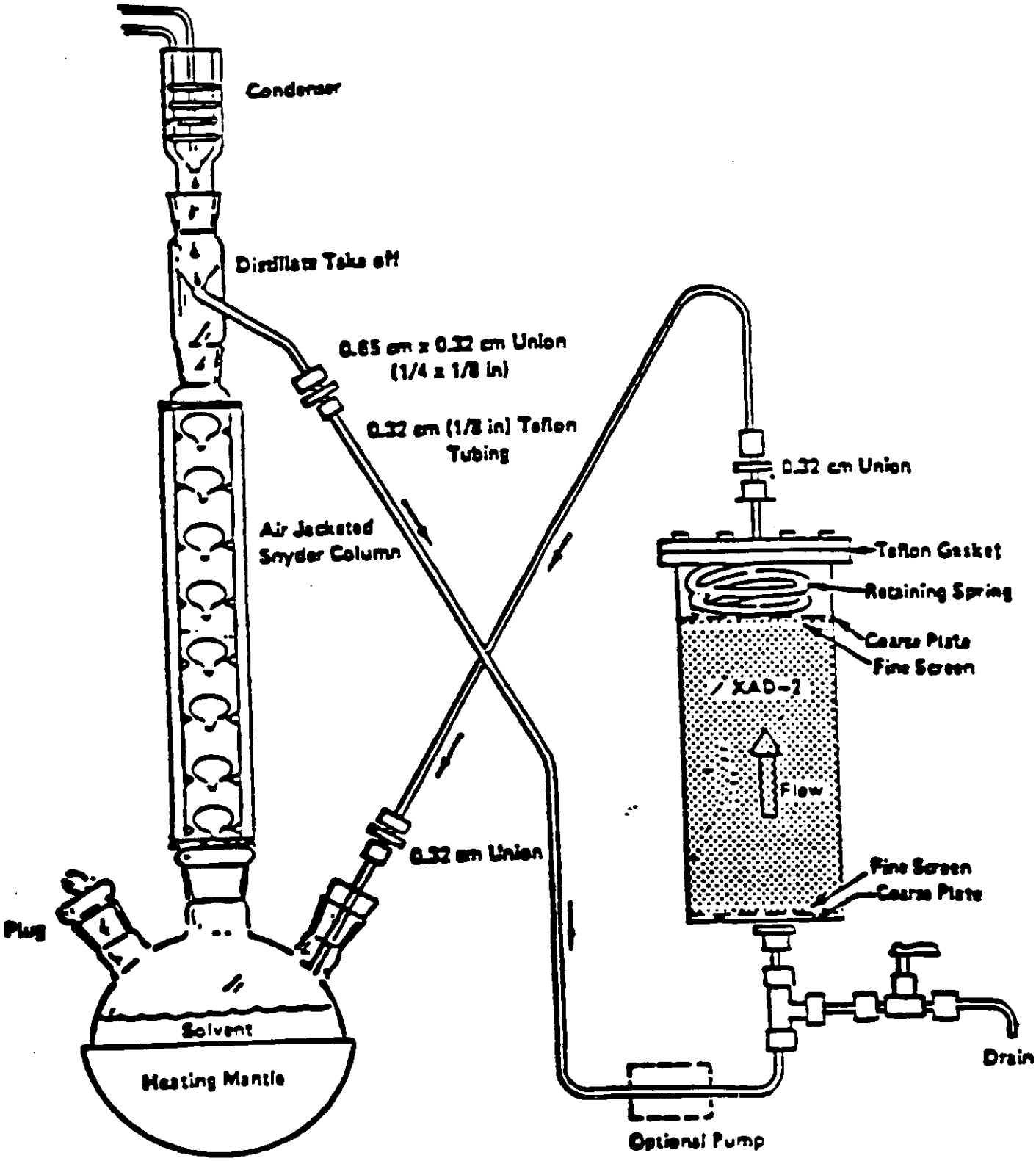

Figure A-1. XAD-2 cleanup extraction apparatus.

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 All materials of construction are glass, Teflon, or stainless steel. Pumps, if used, should not contain extractable materials. Pumps are not used with methanol and methylene 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 nitrogen cylinder has routinely proved to be a reliable source of large volumes of 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 heat source is a water bath heated from a steam line. The final nitrogen temperature 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

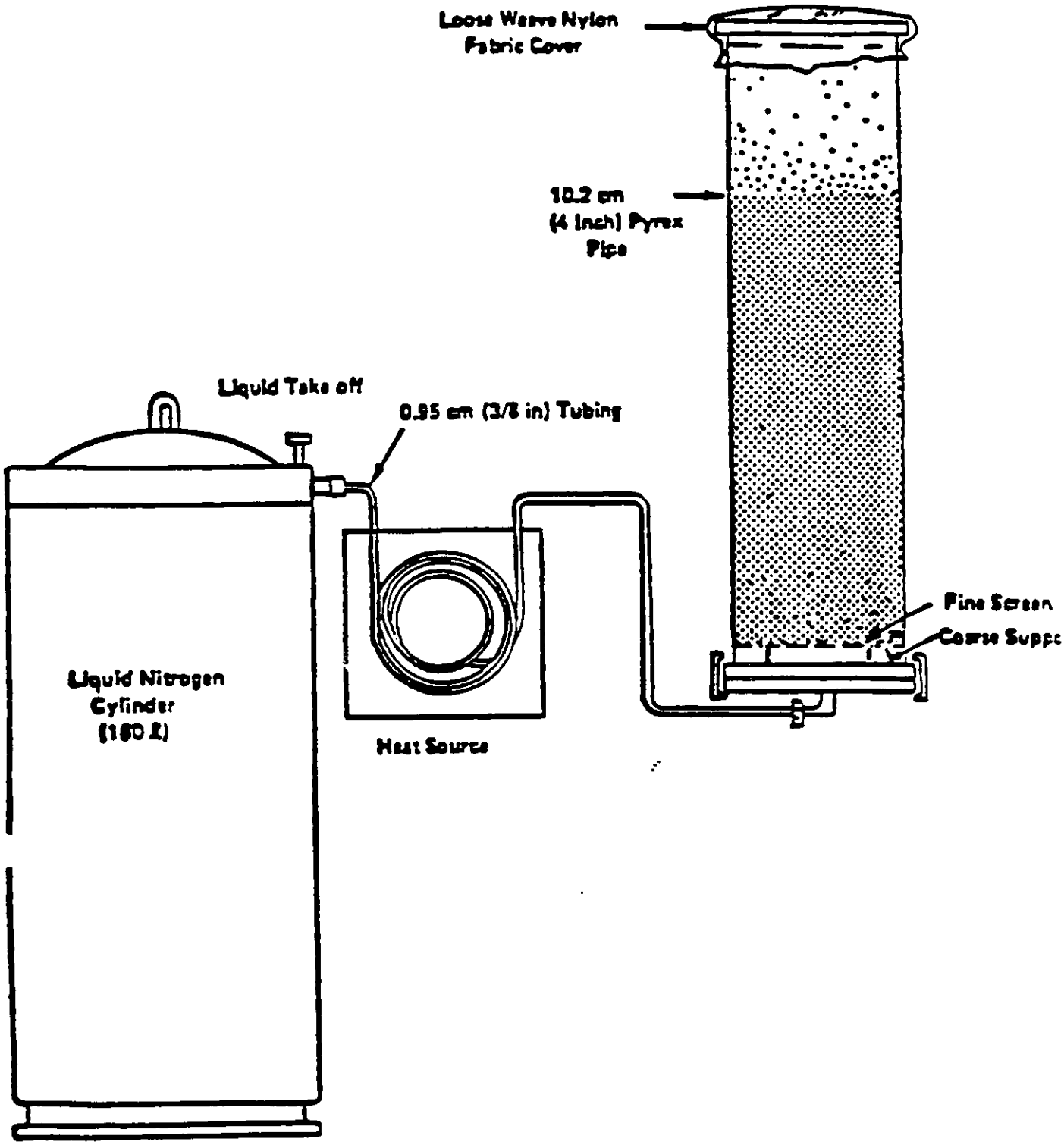

Figure A-2. XAD-2 fluidized-bed drying apparatus.

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.

1.2 Application: This procedure applies solely to the Level I 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.

APPENDIX H

CALCULATION EQUATIONS

Calculation of Dioxin Emission Rates in g/sec and 10^{-8} g/sec

$$\dot{m}\ (g/sec) = 4.716 \times 10^{-13}\ C_s(ng/m^3)\ Q_{s,e}(DSCFM)$$

$$\dot{m}\ (10^{-8}g/sec) = 4.716 \times 10^{-5}\ C_s(ng/m^3)\ Q_{s,e}(DSCFM)$$

where:

C_s = ng/dsm^3

$Q_{s,e}$ = DSCF/MIN

RESOLUTION OF CONFLICTS BETWEEN ISOMER AND HOMOLOG RESULTS

<u>Problem</u>: $\sum_{2,3,7,8}$ Isomers concentration > homolog concentration

This is caused by detection limits for isomers being too high (interference)
But clearly this is impossible so,

<u>Solution</u>: assign the homolog concentration value to the isomer sum.
i.e.

$$\sum_{2,3,7,8} \text{isomers} = \text{homolog}$$

This results in a worst case analysis of the higher toxicity 2,3,7,8 isomers.

<u>Example</u>:

	Homolog	1,2,3,4,6,7,8
HpCDD	2.37	< 3.5

Assign 2,3,7,8 HpCDD = 2.37
Other HpCDDs = 0

Net effect is to reduce the concentration estimate of the non-2,3,7,8-isomers and increase the concentration estimate of the 2,3,7,8-isomers using the homolog analysis as the best estimate of the sum of the 2,3,7,8- and non 2,3,7,8-isomers.