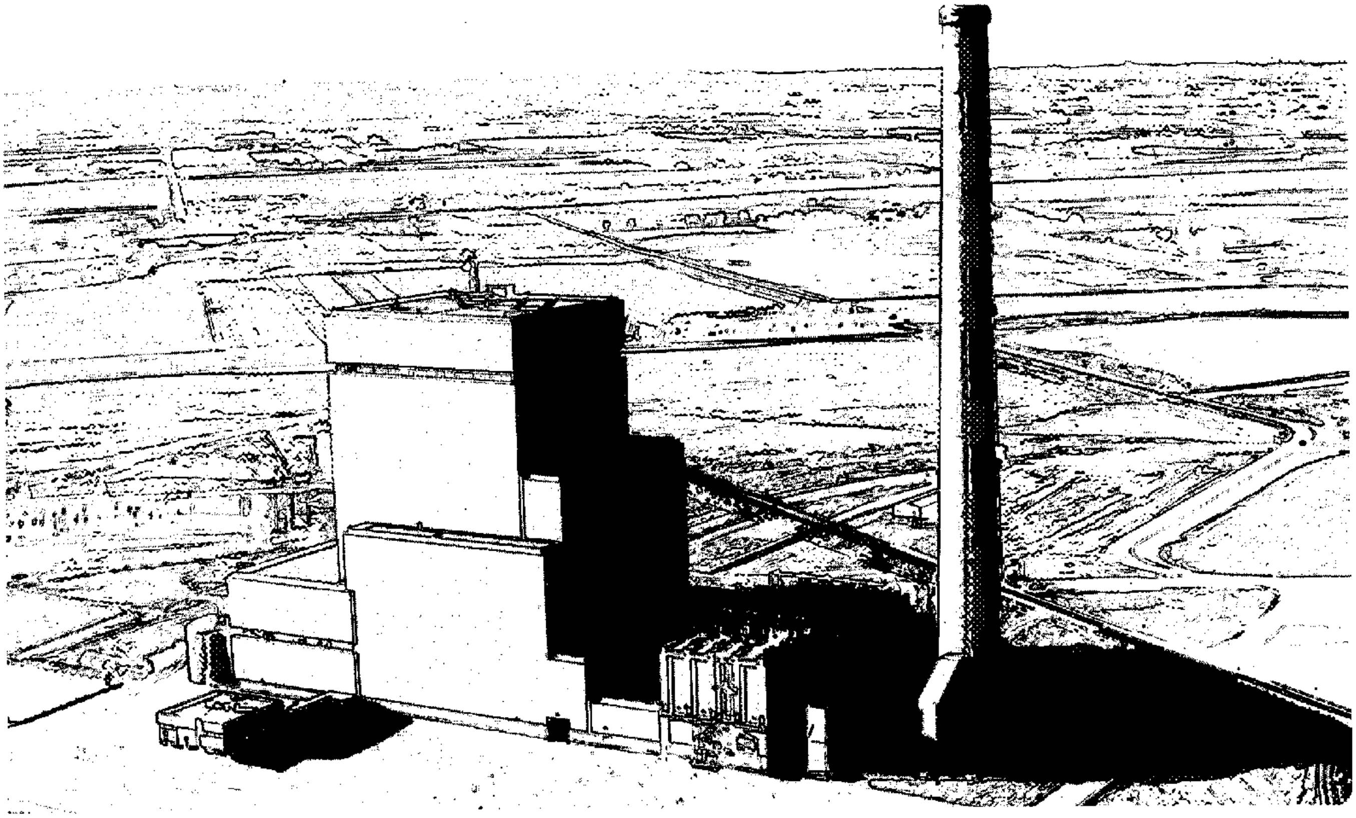
ANL/ESD-22
NREL/TP-430-6322c

Results of Combustion and Emissions Testing when Co-Firing Blends of Binder-Enhanced Densified Refuse-Derived Fuel (b-dRDF) Pellets and Coal in a 440 MW_e Cyclone Fired Combustor

Volume 3: Appendices



A Joint Effort

Energy Systems Division
Argonne National Laboratory

Operated by the University of Chicago
for the U.S. Department of Energy
under Contract W-31-109-Eng-38
Author: O. Ohlsson

National Renewable
Energy Laboratory

Managed by Midwest Research Institute
for the U.S. Department of Energy
under Contract DE-AC36-83CH10093
Technical Monitor: Philip B. Shepherd

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ANL/ESD-22
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O. Ohlsson
Energy Systems Division
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

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July, 1994

NOTICE

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Foreword

This report contains the data resulting from the co-firing of b-dRDF pellets and coal in a 440-MW_e cyclone-fired combustor. these tests were conducted under a Collaborative Research and Development Agreement (CRADA). The CRADA partners included the U.S. Department of Energy (DOE), National Renewable Energy Laboratory (NREL), Argonne National Laboratory (ANL), Otter Tail Power Company, Green Isle Environmental, Inc., XL Recycling Corporation, and Marblehead Lime Company. The report is made up of three volumes. Volume 1 contains a description of the test facility, the test program, test results, and study conclusions and recommendations; Volume 2 contains the field data and laboratory analysis of each individual run; Volume 3 contains other supporting information, along with quality assurance documentation and safety and test plans. With this multi-volume approach, readers can find information at the desired level of detail, depending on individual interest or need.

Table of Contents

Appendix A: Volumetric Flow Rate Determinations

Test No. 3
No. 1 Boiler Stack

Results of Volumetric Flow Rate Determination-------Method 2

Date of Determination............	10-26-92
Time of Determination.......(HRS)	800
Barometric pressure.......(IN.HG)	28.71
Pitot tube coefficient...........	.84
Number of sampling ports.........	4
Total number of points...........	12
Shape of duct....................	Round
Stack diameter...............(IN)	290
Duct area.................(SQ.FT)	458.69
Direction of flow................	UP
Static pressure...........(IN.WC)	-1.5
Avg. gas temp.............(DEG-F)	297
Moisture content..........(% V/V)	13.98
Avg. linear velocity.....(FT/SEC)	80.1
Gas density..............(LB/ACF)	.04953
Molecular weight......(LB/LBMOLE)	30.33
Mass flow of gas..........(LB/HR)	6554101
Volumetric flow rate.............	
actual....................(ACFM)	2205357
dry standard.............(DSCFM)	1264819

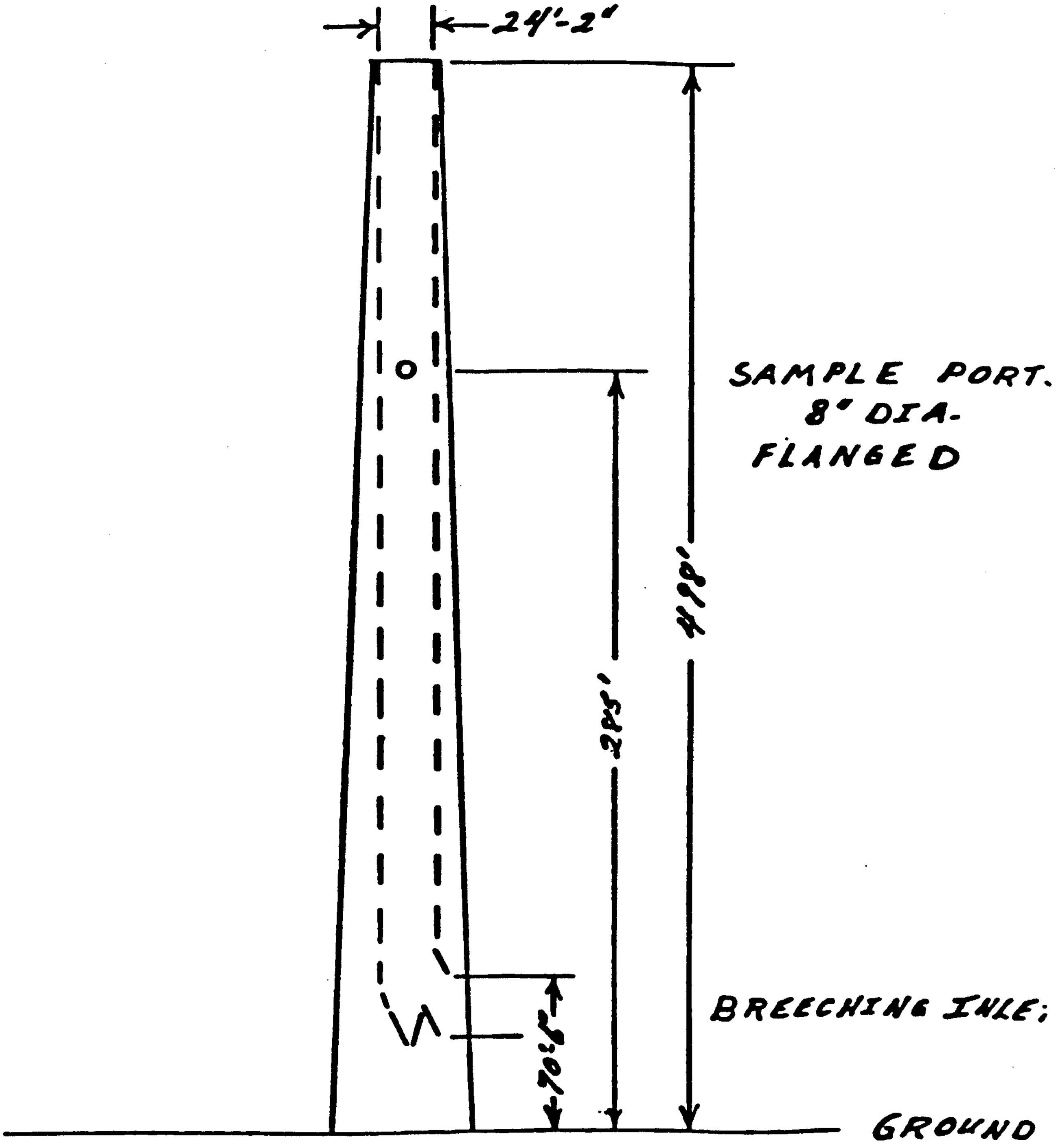
Test No. 8
No. 1 Boiler Stack

Results of Volumetric Flow Rate Determination-------Method 2

Date of Determination............	10-27-92
Time of Determination.......(HRS)	815
Barometric pressure.......(IN.HG)	28.46
Pitot tube coefficient...........	.84
Number of sampling ports.........	4
Total number of points...........	12
Shape of duct....................	Round
Stack diameter...............(IN)	290
Duct area.................(SQ.FT)	458.69
Direction of flow................	UP
Static pressure...........(IN.WC)	-1.5
Avg. gas temp.............(DEG-F)	294
Moisture content..........(% V/V)	15.25
Avg. linear velocity.....(FT/SEC)	78.2
Gas density..............(LB/ACF)	.04888
Molecular weight......(LB/LBMOLE)	30.24
Mass flow of gas..........(LB/HR)	6312633
Volumetric flow rate.............	
actual...................(ACFM)	2152316
dry standard............(DSCFM)	1210238

Appendix B: Stack Sampling Test Parts

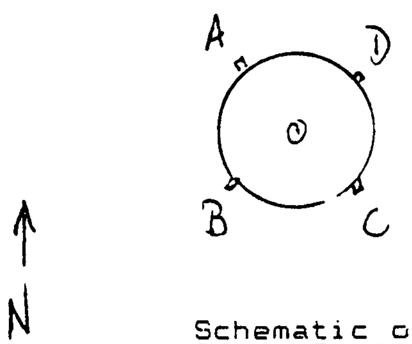
OTTER TAIL POWER COMPANY-BIG STONE PLANT
UNIT 1 STACK



Appendix C : Field Data Sheets

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job ANL/OTP BIG STONE
Source Boiler Stack
Test 1 Run 0 Date 10/26/92
Stack dimen. 290 IN.
Dry bulb ______ °F Wet bulb ______ °F
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.
Barometric pressure 28.71 in Hg
Static pressure -1.3 in WC
Operators J. Van Hoever & K. Rosenthal
Pitot No. 4M5-8 Cp .84



Schematic of Cross Section

Metals (Coal only)

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: 13 in.		Time start: hrs	
A 1	.044	12.75	25.75		
2	.146	42.34	55.34		
3	.296	85.84	98.84		
B 1					
2					
3					
C 1					
2					
3					
D 1					
2					
3					
Temp. meas. tool & S/N: PDT #18				Time end: hrs	

R or nothing= [✓ reg.] manometer; S= expanded; E = electronic S-392.1

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job ANL/OTP - Bigstone
Date 10/26/92 Test 1 Run 1
Source Boiler Stack
No. of traverse points 12
Method CC12 Filter holder: teflon
Filter type: Pallflex glass fiber

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: 0 cfm at 13 in. Hg. (vac) ☒

Particulate Catch Data:

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

☐ acetone
☒ other(s) .1N HNO$_3$ & MECL$_2$

No. of probe wash bottles: 2
Sample recovered by: Dail

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	719	400	319
Impinger No. 3			
Condenser			
Desiccant	1414	1383	27
Total	▨		346

Integrated Gas Sampling Data:

Bag Pump No. 29B Box No. 1 Bag No. 1
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 14 in. Hg.
Time start: 0935 (HRS) Time end: 1146 (HRS)
Sampling rate: 200 cc/min Operator:
S/N of O_2 Analyzer used to monitor train outlet: 4

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job ANL/COTF-BIG STONE
Source Boiler Stack
Date 10/26/92 Test 1 Run 1

Operators Dick Q
Meter Box No. 3 ^H@ 1.81 IN WC
Gasmeter coeff. .9984

Pitot No. 4MS-8 Cp .84
Bar. Press. 28.77 inHg H2O x
Nozzle No. 9L55 Nozzle Dia .232 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgm. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	Oxygen (xv/v)
	0930	350.65											
D 3	5	355.27	1.50	2.71	529	12	292	243	248	41	58	58	8.2
3	10	359.73	1.40	2.52	9.76	11	292				61	59	8.2
2	15	364.12	1.30	2.35	4.08	11	292				64	60	8.0
2	20	368.55	1.35	2.45	8.50	11	293	246	250	40	66	61	8.0
1	25	372.75	1.15	2.09	2.60	9	293				68	62	8.2
1	30	376.68	1.10	2.01	6.62	9	292				70	63	8.0
A 3	35	381.01	1.30	2.38	1.00	9	292	250	251	41	67	63	7.9
3	40	385.55	1.40	2.56	552	11	292				72	65	7.9
2	45	390.07	1.35	2.51	0.06	11	292				72	65	8.0
2	50	394.48	1.35	2.47	4.53	11	294	252	253	41	73	66	7.9
1	55	398.65	1.15	2.12	8.68	9	293				74	66	7.8
1	60	402.63	1.08	1.99	2.70	9	293				75	67	8.0
B 3	65	407.25	1.40	2.58	7.27	11	293	253	251	41	70	67	8.2
3	70	411.81	1.40	2.57	1.83	11	293				74	68	8.1
2	75	416.37	1.40	2.58	6.41	11	293				75	69	8.0
2	80	420.87	1.40	2.59	0.99	11	293	254	251	41	76	70	7.9
1	85	425.36	1.30	2.40	5.42	10	294				77	70	8.0
1	90	429.80	1.25	2.31	9.77	10	294				77	71	8.1
C 3	95	434.45	1.45	2.68	4.45	11	294	254	255	41	73	70	8.2
3	100	439.17	1.45	2.68	9.11	11	293				76	71	8.0
2	105	443.65	1.35	2.50	3.62	11	293				77	71	8.0
2	110	448.08	1.35	2.50	8.15	11	293	253	256	42	78	72	7.9
1	115	452.48	1.25	2.32	2.51		293				79	72	8.4
1	120	456.88	1.15	2.14	6.70		293				80	73	8.3
	(1146)												
	Θ = 120	Vm = 106.23		^H = 2.36							Avg. = 69.6		

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job ANL/OTP BIG STONE
Source Boiler Stack
Method 0012 Filter holder: teflon

Date 10/26/92 Test 1 Run 2
No. of traverse points 12
Filter type: pallflex glass fiber

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: 0 cfm at 13 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used:

Recovery solvent(s)

☐ acetone
☒ other(s) $MeCl_2$ " , $1N\ HNO_3$

No. of probe wash bottles: 2
Sample recovered by: DLH

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	719	400	319
Impinger No. 3			
Condenser			
Desiccant	1397	1366	31
Total	▨	▨	350

Integrated Gas Sampling Data:

Bag Pump No. 29 Box No. B Bag No. 2
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 14 in. Hg.
Time start: 1238 (HRS) Time end: 1445 (HRS)
Sampling rate: 300 cc/min Operator: DLH
S/N of O_2 Analyzer used to monitor train outlet: 4

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job ANL/OTF BIG STONE
Source Boiler Stack
Date 10/26/92 Test 1 Run 2

Operators Dick & KR
Meter Box No. 3 ^H@ 1.81 IN WC
Gasmeter coeff. .9984

Pitot No. 4MS-8 Cp .84
Bar. Press. 28.71 inHg H2O 14 %
Nozzle No. 9kss Nozzle Dia .239 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Des. Vol. (cf)	VAC. inHg	Temperatures (°F)						Oxygen
							Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	(%v/v)
	1234	457.24											
C 3	5	461.94	1.50	2.75	1.95	11	295	241	256	43	68	68	8.1
3	10	466.60	1.50	2.74	6.66	11	295				73	70	8.0
2	15	471.29	1.50	2.76	1.39	11	294				75	70	8.1
2	20	475.99	1.40	2.59	5.98	11	294	246	251	42	71	71	8.0
1	25	480.16	1.10	2.04	0.06	8	294				79	72	8.0
B 1	30	484.30	1.10	2.04	4.16	8	294				80	73	8.0
3	35	488.85	1.50	2.79	8.94	11	294				76	73	8.2
3	40	493.57	1.45	2.69	3.63	11	294				80	74	8.0
2	45	498.40	1.50	2.78	8.41	11	296				81	75	8.1
2	50	503.10	1.45	2.70	3.12	11	296				82	75	8.1
1	55	507.51	1.30	2.42	7.58	11	295				82	76	8.1
D 1	60	512.14	1.35	2.52	2.14	11	295	235	254	44	83	76	8.0
3	65	516.91	1.50	2.80	6.95	11	295				79	76	8.3
3	70	521.68	1.45	2.70	1.66	11	295				82	77	8.0
2	75	526.36	1.45	2.70	6.37	11	298	237	254	44	83	77	8.1
2	80	531.11	1.45	2.70	1.09	11	298				84	78	8.1
1	85	535.31	1.15	2.15	5.31	9	298				84	78	8.0
A 1	90	539.46	1.10	2.05	9.43	8	300	241	256	48	85	79	8.0
3	95	544.02	1.40	2.60	4.07	11	302				86	79	8.1
3	100	548.68	1.40	2.61	8.72	11	302				86	79	8.1
2	105	553.22	1.35	2.51	3.29	11	302	244	255	48	86	79	8.0
2	110	557.80	1.40	2.61	7.94	11	302				86	80	8.1
1	115	562.61	1.40	2.61	2.59	11	302				86	80	8.1
1	120	567.15	1.35	2.52	7.16		302	245	253	47	86	80	8.1
	1445												
	θ = 120	Vm = 109.91		^H = 2.56							Avg. = 78.4		

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job ANL/OTP BIG STONE
Source 1 Boiler Stack
Method 0012 Filter holder: teflon
Date 10/26/92 Test 1 Run 3
No. of traverse points 12
Filter type: pallflex glass fiber

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: 0 cfm at 12 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used:

Recovery solvent(s)
☐ acetone
☒ other(s) MECL2 & .1N HNO3

No. of probe wash bottles: 2
Sample recovered by: DvH

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	708	400	308
Impinger No. 3			
Condenser			
Desiccant	1401	1356	45
Total			353

Integrated Gas Sampling Data:

Bag Pump No. 29B Box No. 1 Bag No. 1
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 14 in. Hg.
Time start: 1535 (HRS) Time end: 1739 (HRS)
Sampling rate: 300 cc/min Operator: DvH
S/N of O_2 Analyzer used to monitor train outlet: 4

CF-023

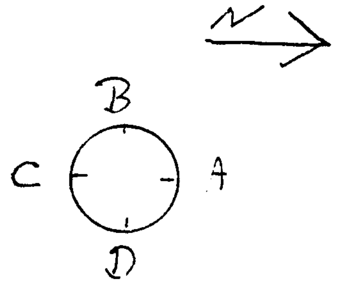
INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job ANL/OTP Big Stone
Source Boiler Stack
Date 10/26/92 Test 1 Run 3
Operators DWH & KE
Meter Box No. 3 ^H@ 1.81 IN WC
Gasmeter coeff. .9984
Pitot No. 4MS-8 Cp .84
Bar. Press. 28.71 inHg H2O 13.7 %
Nozzle No. Glass Nozzle Dia .237 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgs. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	Oxygen (%v/v)
	1530	561.40											
D 3	5	571.90	1.35	2.50	1.93	11	304	248	250	46	75	74	8.0
3	10	576.48	1.35	2.47	6.43	11	307				79	75	8.0
2	15	580.99	1.35	2.49	0.95	11	307				80	76	8.0
2	20	585.42	1.30	2.40	5.39	11	307	252	251	47	81	76	8.0
1	25	589.61	1.15	2.12	9.58	9	307				82	75	8.0
1	30	593.81	1.15	2.13	3.77	9	305				82	76	8.0
B 3	35	598.26	1.35	2.49	8.29	11	310	251	252	48	82	78	8.0
3	40	602.84	1.35	2.49	2.83	11	310				85	79	8.0
2	45	607.25	1.35	2.50	7.38	11	310				86	79	8.1
2	50	611.88	1.35	2.50	1.94	11	310	253	252	48	87	80	8.0
1	55	616.08	1.10	2.04	6.06	9	310				87	81	8.0
1	60	620.42	1.20	2.23	0.37	9	310				87	81	8.0
A 3	65	624.91	1.30	2.42	4.85	11	310	254	250	47	87	82	8.1
3	70	629.41	1.35	2.52	9.43	11	308				88	82	8.0
2	75	634.00	1.35	2.52	4.01	11	308				88	82	8.0
2	80	638.52	1.35	2.52	8.59	11	308				88	82	8.0
1	85	642.84	1.15	2.15	2.83	9	308	253	251	48	88	82	8.0
1	90	647.19	1.20	2.24	7.15	9	308				88	83	8.0
C 3	95	651.65	1.30	2.43	1.65	11	308				89	83	8.0
3	100	656.17	1.30	2.43	6.16	11	308	251	252	47	90	84	8.0
2	105	660.61	1.30	2.44	0.67	11	308				90	84	8.0
2	110	665.18	1.30	2.44	5.19	11	308				90	84	8.0
1	115	669.50	1.20	2.25	9.52		308	251	256	48	90	84	8.0
1	120	673.86	1.20	2.25	3.86		308				90	84	8.3
	1759												
	Θ = 120	Vm = 106.46		^H = 2.34							Avg. = 83		

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job HNL / OTP - Bigstone
Source Boiler Stack
Test 2 Run O-3 Date 10-26-92
Stack dimen. 290.0 IN.
Dry bulb _____ °F Wet bulb _____ °F
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.
Barometric pressure 28.71 in Hg
Static pressure -1.30 in WC
Operators R. Rosenthal, B. Aschenbach
Pitot No. MM5-8 Cp .840



Schematic of Cross Section

Dioxin (Coal only) (PAH/DCB Aliquot)

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: 13 in.			Time start: hrs	
	1	.044	12.75	25.75		
	2	.146	42.34	55.34		
	3	.296	85.84	98.84		
	1					
	2					
	3					
	1					
	2					
	3					
	1					
	2					
	3					
Temp. meas. tool & S/N: Digital T/C P07-20					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 ANL/OTP-BigSTONE
Source BOILER STACK
Cyclone: ☐ Yes ☒ No Filter holder: MM5
Analytes:
Field recovery spike added: ☒ Yes ☐ No
NOTE DIOXIN, PAH, PCB SAmple TRAIN

Date 10-26-92 Test 2 Run 1
No. of traverse points 12
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: .005 cfm at 11 in. Hg. (vac) ☒
Pitot @ IN W.C.

Semivolatile Catch Data:

No.s of filters used: 0251

Recovery solvent(s)
☒ MeCl2 * MeOH (50:50 v/v)
☐ other(s)

No. of bottles for condensate trap catch:
Samples recovered by:

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Condensate trap	735	449	286
Condensate trap			
Condensate trap			
Impingers			
Desiccant	1390	1348	42
Total	▨	▨	328

Integrated Gas Sampling Data:

Bag Pump No. 23A Box No. 22 Bag No. 1
Bag Material: 4-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 22 in. Hg.
Time start: 930 (HRS) Time end: 1145 (HRS)
Sampling rate: 200 cc/min O2 Analyzer S/N: 7

Rev.1 CF-026

Job ANC/OTP BIGSTONE
Source Boiler STACK
Date 10/26/92 Test 2 Run 1
Operators K.K & M.K.
Meter Box No. 9
Gasmeter coeff. 1.0001
Nozzle Dia. .234 in.
Bar. Pres. 28.71 in. Hg
Cp .840
H_2O 14 %

NOTE: DIOXIN PAH PCB SAMPLE TRAIN

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Del. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out) In	Meter (In/Out) Out	Oxygen (%v/v)
	930	778.23												
C-3	5	782.56	1.40	2.28	2.52	8	292	248	230	40	40	55	55	7.9
3	10	786.77	1.40	2.25	6.78	8.5	292					59	56	7.5
2	15	790.82	1.35	2.18	0.98	8.5	292	235	233	42	40	62	57	7.5
2	20	795.03	1.35	2.19	5.19	8.5	293					63	57	7.5
1	25	799.15	1.25	2.03	9.25	8.5	293	236	235	43	40	64	58	7.5
1	30	803.14	1.20	1.96	3.24	8	292					65	59	7.5
A-3	35	807.30	1.30	2.12	7.39	9	292	236	235	44	39	61	59	7.3
3	40	811.63	1.40	2.28	1.69	9	292					65	60	7.5
2	45	815.90	1.35	2.20	5.92	10	294	247	233	45	41	66	60	7.4
2	50	820.12	1.35	2.20	0.16	9.5	294					67	61	7.5
1	55	824.24	1.20	1.96	4.17	9	293	244	230	43	41	68	62	7.4
1	60	828.22	1.20	1.98	8.21	9	293					69	63	7.5
B 3	65	832.52	1.35	2.22	2.47	10	293	244	228	43	41	64	62	7.5
3	70	836.79	1.35	2.20	6.71	10	293					66	63	7.5
2	75	841.00	1.30	2.13	0.89	9.5	293	246	230	46	45	68	63	7.5
2	80	845.24	1.35	2.21	5.14	9.5	295					69	64	7.5
1	85	849.30	1.25	2.05	9.25	9	295	245	230	53	46	70	64	7.5
1	90	853.28	1.20	1.97	3.27	8.5	295					71	65	7.5
D-3	95	857.57	1.35	2.22	7.55	10	295	240	233	50	46	69	64	7.9
3	100	861.82	1.35	2.22	1.82	10	293				L	70	65	7.5
2	105	866.11	1.30	2.14	6.02	9	292	238	230	50	46	71	65	7.5
2	110	870.30	1.30	2.15	0.23	9	292					72	66	7.5
1	115	874.39	1.25	2.06	4.35	9	293	235	228	50	46	72	67	7.5
1	120	878.46	1.20	1.98	8.40	9	293					72	67	7.5
	1145													
	θ=120	Vm= 100.23		ΔH=2.13								Avg.= 64.2		

C-10

Rev. 1

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job ANL/OTP-BIGSTONE
Source BOILER STACK
Cyclone: ☐ Yes ☐ No Filter holder: MM5
Analytes:
Field recovery spike added: ☒ Yes ☐ No
NOTE DIOXIN PAH PCB sample TRAIN

Date 10-26-92 Test 2 Run 2
No. of traverse points 12
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: .00 cfm at 9 in. Hg. (vac) ☐
Pitot @

Semivolatile Catch Data:

No.s of filters used: 0252

Recovery solvent(s)
☒ MeCl2 * MeOH (50:50 v/v)
☐ other(s) ____

No. of bottles for condensate trap catch: ____
Samples recovered by: M.K.

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Condensate trap	565	261	304
Condensate trap			
Condensate trap			
Impingers			
Desiccant	1403	1374	29
Total	▨	▨	333

Integrated Gas Sampling Data:

Bag Pump No. 23A Box No. 22 Bag No. 2
Bag Material: 4-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 20 in. Hg.
Time start: 1225 (HRS) Time end: 1435 (HRS)
Sampling rate: 400 cc/min O2 Analyzer S/N: 7

Rev.1 CF-026

Job ANL/OTP-BIG STONE
Operators
Source BOILER STACK
Meter Box No. 9
Nozzle Dia. .234 in.
Cp .840
Date 10-26-92 Test 2 Run 2
Gasmeter coeff. 1.0001
Bar.Pres. 28.71 in.Hg
H_2O 14 %
NOTE DIOXIN PCB, PAH SAMPLE TRAIN

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)
	1225	878.95												
D- 3	5	883.35	1.45	2.37	3.35	6	293	235	228	42	40	67	64	7.7
3	10	887.71	1.45	2.38	7.76	6	293					69	65	7.9
2	15	891.95	1.35	2.22	2.04	6	293	232	234	45	41	71	66	7.9
2	20	896.22	1.35	2.22	6.31	6	296					73	67	7.9
1	25	900.26	1.20	1.98	0.36	5	296	232	234	45	42	74	68	7.9
1	30	904.49	1.25	2.07	4.50	6	295					75	68	7.9
A- 3	35	908.85	1.35	2.24	8.81	6	294	238	230	49	44	72	68	7.9
3	40	913.28	1.45	2.40	3.25	6	294					74	68	7.7
2	45	917.65	1.35	2.23	7.56	6	294	240	230	52	46	75	69	7.9
2	50	921.91	1.40	2.32	1.94	6	295					76	69	7.9
1	55	926.00	1.20	1.99	6.01	5	296	247	232	52	46	76	70	7.9
1	60	929.99	1.15	1.90	9.98	5	299					77	71	7.9
C- 3	65	934.41	1.40	2.32	4.38	6	296	248	235	54	47	73	70	7.7
3	70	938.80	1.40	2.31	8.76	6	296					75	70	7.9
2	75	943.16	1.40	2.32	3.15	6	295	248	235	54	49	77	71	7.7
2	80	947.55	1.40	2.33	7.56	6	295					77	72	7.7
1	85	951.79	1.25	2.08	1.72	6	295	240	237	53	49	78	72	7.7
1	90	955.93	1.30	2.15	5.96	6	300					78	72	7.7
B- 3	95	960.44	1.45	2.40	0.43	6	300	242	239	48	47	74	71	7.9
3	100	964.76	1.40	2.30	4.81	6	301					75	72	7.8
2	105	969.11	1.35	2.22	9.11	6	302	245	240	50	49	78	72	7.5
2	110	973.44	1.35	2.23	3.42	6	302					79	73	7.5
1	115	977.70	1.30	2.14	7.65	6	304	245	240	52	49	79	73	7.5
1	120	981.89	1.25	2.06	1.81	5.5	303					80	74	7.5
	(1435)													
	θ= 120	Vm= 102.94		ΔH= 2.22								Avg.= 72.4		

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job ANL/OTP-Bigstone
Source BOILER STACK
Cyclone: ☐ Yes ☒ No Filter holder: MM5
Analytes:
Field recovery spike added: ☒ Yes ☐ No
NOTE: DIOXIN, PAH, PCB Sample TRAIN

Date 10-26-92 Test 2 Run 3
No. of traverse points 12
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: ___ cfm at ___ in. Hg. (vac) ☐
Pitots @ ___ IN W.C.

Semivolatile Catch Data:

No.s of filters used: 0253

Recovery solvent(s)
☐ MeCl2 * MeOH (50:50 v/v)
☐ other(s) ___

No. of bottles for condensate trap catch: 1
Samples recovered by: mk

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Condensate trap	580	273	307
Condensate trap			
Condensate trap			
Impingers			
Desiccant	1414	1390	24
Total	▨	▨	331

Integrated Gas Sampling Data:

Bag Pump No. 23A Box No. 22 Bag No. 3
Bag Material: 4-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 20 in. Hg.
Time start: 1515 (HRS) Time end: 1730 (HRS)
Sampling rate: 200 cc/min O2 Analyzer S/N: 7

Rev.1 CF-026

Job ANL/OTP - BIGSTONE Operators K.K. & M.R.
Source BOILER STACK Meter Box No. 9 Nozzle Dia. .234 in. Cp .840
Date 10-26-92 Test 2 Run 3 Gasmeter coeff. 1.0001 Bar. Pres. 28.71 in. Hg H_2O 14 %
NOTE DIOXIN, PCB, PAH SAMPLE TRAIN

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) In	Meter (In/Out) Out	Oxygen (%v/v)
	1515	982.20												
B-3	5	986.65	1.45	2.39	6.65	6	300	238	229	49	49	69	69	7.5
3	10	990.98	1.40	2.28	0.99	7.5	302					72	70	7.5
2	15	995.42	1.45	2.37	5.42	8	302	242	235	50	49	74	70	7.5
2	20	999.78	1.45	2.37	9.86	8	304					76	71	7.7
1	25	1004.01	1.30	2.13	4.07	7	305	242	235	51	49	77	72	7.5
1	30	1008.23	1.25	2.05	8.21	7	305					78	72	7.5
A-3	35	1012.49	1.40	2.30	2.59	8	305	249	235	52	50	76	72	7.5
3	40	1016.90	1.40	2.30	6.97	8	305					78	72	7.5
2	45	1021.27	1.35	2.22	1.27	8	305	250	240	49	48	79	73	7.5
2	50	1025.66	1.40	2.29	5.65	8	310					80	74	7.5
1	55	1029.89	1.30	2.13	9.87	8	310	253	237	49	48	80	74	7.5
1	60	1034.02	1.25	2.05	4.02	8	310					81	75	7.5
D-3	65	1038.49	1.40	2.30	8.41	8.5	309	256	245	51	48	79	74	7.7
3	70	1042.90	1.45	2.38	2.87	9	309					80	75	7.5
2	75	1047.27	1.35	2.22	7.18	9	310	255	245	51	49	81	75	7.5
2	80	1051.53	1.35	2.22	1.50	8	310					81	75	7.7
1	85	1055.50	1.15	1.89	5.48	7.5	310	250	250	52	49	81	76	7.7
1	90	1059.58	1.20	1.97	9.56	7.5	310					81	76	7.7
C-3	95	1063.91	1.40	2.32	3.97	8.5	305	253	255	50	48	79	75	7.5
3	100	1068.15	1.35	2.11	8.06	8	304					81	76	7.5
2	105	1072.45	1.35	2.24	2.40	8	304	250	255	50	48	82	76	7.5
2	120	1076.82	1.40	2.32	6.82	8	303					82	76	7.5
1	115	1081.16	1.30	2.15	1.07	8	307	250	255	50	48	82	76	7.5
1	120	1085.24	1.25	2.07	5.24	8	307					82	76	
	1730													
	θ= 120	Vm=103.04		ΔH=2.21								Avg.=75.1		

Rev. 1

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job OTP BIG STONE

Source NO 1 BOILER STACK

Test 3 Run 0 Date 10-26-92

Stack dimen. 290 IN.

Dry bulb ______°F Wet bulb ______°F

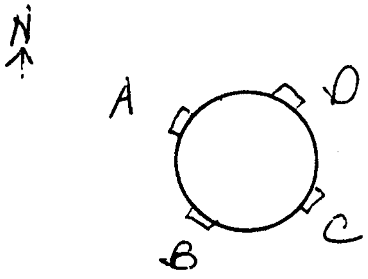
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.

Barometric pressure 2871 in Hg

Static pressure -1.5 in WC

Operators GH & JB

Pitot No. U23-8 Cp .84



Schematic of Cross Section

Particulate - Coal Only

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: 13 in.		Time start: 0800 hrs	
A 1	.044	12.75	25.75	1.22	298
2	.146	42.34	55.34	1.37	298
3	.296	85.84	98.84	1.40	296
B 1				1.24	296
2				1.40	299
3				1.50	299
C 1				1.26	300
2				1.39	298
3				1.48	298
D 1				1.20	298
2				1.31	297
3				140	297
Temp. meas. tool & S/N: NO 24				Time end: 0815 hrs	

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

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job OTP BIG STONE
Source NO 1 BOILER STACK
Method 5 Filter holder: GLASS

Date 10-26-92 Test 3 Run 1
No. of traverse points 12
Filter type: GLASS FIBER

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: .00 cfm at 10 in. Hg. (vac) ☐

Particulate Catch Data:

No.s of filters used: 4725

Recovery solvent(s)
☒ acetone
☐ other(s)

No. of probe wash bottles: 1
Sample recovered by: GH

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	656	493	163
Impinger No. 3			
Condenser			
Desiccant	1390	1373	17
Total			180

Integrated Gas Sampling Data:

Bag Pump No. 3 Box No. 2 Bag No. 1
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: .00 cc/min at 15 in. Hg.
Time start: 0930 (HRS) Time end: 1040 (HRS)
Sampling rate: 400 cc/min Operator: GH
S/N of O_2 Analyzer used to monitor train outlet: 3

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job OTP BIG STONE
Source NO 1 BOILER STACK
Date 10-26-92 Test 3 Run 1

Operators GH & TB
Meter Box No. 4 ^H@ 1.91 IN WC
Gasmeter coeff. 1.0000

Pitot No. 22-Y Cp .84
Bar. Press. 28.71 inHg H2O 15 x
Nozzle No. 23-4 Nozzle Dia .274 IN.

Traverse Point No.		Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgm. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	Oxygen (%v/v)
		(0930)	828.90											
B	3	5	833.49	1.35	2.82	349	6	292	255	243	39	60	60	8.2
	2	10	837.85	1.25	2.58	7.84	5	292	256	245	40	61	58	7.9
	1	15	842.22	1.25	2.57	222	5	293	257	246	42	65	60	7.9
A	3	20	846.84	1.40	2.90	6.86	8	293	257	248	45	69	63	8.0
	2	25	851.38	1.32	2.75	1.39	7	293	259	245	46	69	63	8.0
	1	30	855.80	1.25	2.61	5.81	7	292	260	247	45	71	65	8.1
D	3	35	860.47	1.38	289	0.47	7	292	265	250	46	74	65	7.9
	2	40	864.83	1.20	252	483	7	292	265	255	46	74	66	7.9
	1	45	869.36	1.29	2.71	9.35	8	292	260	252	44	76	68	7.9
C	3	50	874.10	1.43	2.99	410	8	293	259	256	40	79	70	7.9
	2	55	878.69	1.31	2.77	869	7	292	260	259	42	79	71	7.7
	1	60	883.00	1.15	2.44	3.00	7	293	261	258	42	80	72	7.9
		(1040)												
		Θ = 60	Vm = 54.10		^H = 2.71							Avg. = 68.2		

S-0037R

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job OTP BIG STONE
Source NO 1 BOILER STACK
Method 5 Filter holder: GLASS
Date 10-26-93 Test 3 Run 2
No. of traverse points 12
Filter type: GLASS FIBER

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: .00 cfm at 10 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used: 4726

Recovery solvent(s)
☒ acetone
☐ other(s)

No. of probe wash bottles: 1
Sample recovered by: GH

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	656	496	160
Impinger No. 3			
Condenser			
Desiccant	1349	1322	27
Total			187

Integrated Gas Sampling Data:

Bag Pump No. 23B Box No. 2 Bag No. 2
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: .00 cc/min at 15 in. Hg.
Time start: 1105 (HRS) Time end: 1220 (HRS)
Sampling rate: 400 cc/min Operator: GH
S/N of O_2 Analyzer used to monitor train outlet: 3

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job OTP BIG STONE
Source UNIT NO 1
Date 10-27-92 Test 3 Run 3

Operators GH & JB
Meter Box No. 4 ^H@ 1.91 IN WC
Gasmeter coeff. 1.000

Pitot No. 1-22-S Cp .84
Bar. Press. 28.91 inHg H2O 14 %
Nozzle No. 23-4 Nozzle Dia .249 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgs. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	Oxygen (%v/v)
	1105	883.30											
C 3	5	888.01	1.39	2.97	8.01	7	292	262	255	39	68	67	8.2
2	10	892.51	1.3	2.77	2.51	7	292	263	254	40	75	69	7.9
1	15	897.08	1.25	2.69	7.08	8	292	264	257	46	76	70	7.7
A 3	20	901.86	1.40	3.02	1.86	8	292	265	258	45	74	71	7.9
2	25	906.59	1.38	2.97	6.60	8	293	266	259	43	78	72	7.9
1	30	910.86	1.1	2.38	0.86	8	293	265	254	44	81	74	7.9
D 3	35	915.70	1.42	3.09	5.71	9	292	266	254	43	77	72	8.1
2	40	920.32	1.30	2.80	0.32	8	295	265	255	44	81	74	8.1
1	45	924.69	1.15	2.49	4.69	8	294	266	254	43	83	76	7.9
B - 3	50	929.38	1.32	2.87	9.38	7	294	265	255	44	82	75	8.0
2	55	933.95	1.25	2.72	3.94	7	294	266	254	43	84	76	7.7
1	60	938.43	1.21	2.63	8.44	7	295	262	255	44	85	78	7.8
	(1220)												
	θ = 60	Vm 55.13		^H 2.78							Avg. = 75.8		

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job OTP - BIG STONE
Source NO 1 BOILER
Method 5 Filter holder: GLASS

Date 10-26-92 Test 3 Run 3
No. of traverse points 12
Filter type: GLASS FIBER

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: .00 cfm at 10 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used: 4723

Recovery solvent(s)
☒ acetone
☐ other(s)

No. of probe wash bottles: 1
Sample recovered by: GH

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	658	492	160
Impinger No. 3			
Condenser			
Desiccant			
	1409	1390	19
Total	▨	▨	179

Integrated Gas Sampling Data:

Bag Pump No. 3 Box No. 2 Bag No. 3
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: .00 cc/min at 15 in. Hg.
Time start: 1255 (HRS) Time end: 1412 (HRS)
Sampling rate: 400 cc/min Operator: GH
S/N of O_2 Analyzer used to monitor train outlet: 3

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job OTP - BIG STONE	Operators GH & JB	Pitot No. V23-8	Cp .84
Source NO 1 BOILER STACK	Meter Box No. 4 ^H@ 1.91 IN WC	Bar. Press. 28.71 inHg	H2O 14 %
Date 10-27-93 Test 3 Run 3	Gasmeter coeff. 1.0000	Nozzle No. 23-4	Nozzle Dia .244 IN.

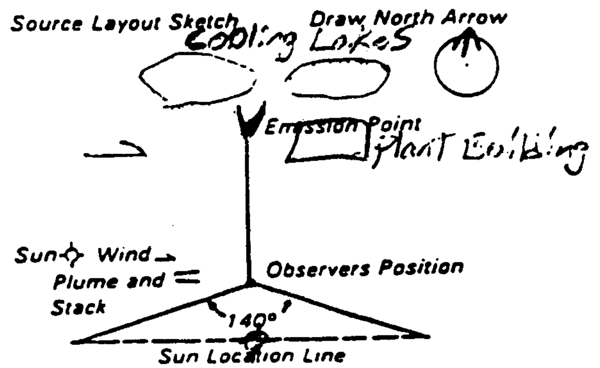
Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Des. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	Oxygen (%v/v)
	1255	938.64											
B - 3	5	943.57	1.50	3.22	3.58	9	298	260	255	39	74	73	7.9
2	10	948.32	1.39	2.98	8.33	8	297	262	256	40	79	75	7.9
1	15	952.60	1.1	2.38	2.60	7	295	261	257	40	81	76	7.8
D - 3	20	957.44	1.41	3.05	7.43	9	296	262	258	42	81	76	8.0
2	25	961.70	1.31	2.38	1.70	8	299	260	255	45	84	77	7.9
1	30	966.18	1.20	2.61	6.18	8	295	259	261	41	85	78	8.0
A 3	35	971.04	1.41	3.07	1.04	9	295	259	262	42	83	78	7.9
2	40	975.84	1.38	3.00	5.84	8	296	255	261	45	86	79	8.0
1	45	980.35	1.21	2.65	0.36	8	290	256	262	45	86	80	8.0
C 3	50	985.36	1.49	3.26	5.36	8	295	259	255	42	81	77	8.1
2	55	990.17	1.41	3.03	0.17	9	301	258	256	41	85	79	8.0
1	60	994.75	1.25	2.72	4.75	9	298	260	255	42	89	81	8.0
	(1412)												
	θ = 60	Vm = 56.11		^H 2.86							Avg. = 80.0		

Interpoll Laboratories
(612)786-6020

Visible Emissions Form

Test #1

SOURCE NAME Otter Tail Power
ADDRESS
CITY Big Stone
STATE S.D.
ZIP
PHONE
SOURCE ID NUMBER
PROCESS EQUIPMENT No.1 Boiler
OPERATING MODE 100%
CONTROL EQUIPMENT Precip.
OPERATING MODE 100%
DESCRIBE EMISSION POINT
START Large Round Stack STOP ✓
HEIGHT ABOVE GROUND LEVEL
START 600' STOP ✓
HEIGHT RELATIVE TO OBSERVER
START 600' STOP ✓
DISTANCE FROM OBSERVER
START 1200' STOP ✓
DIRECTION FROM OBSERVER
START N STOP ✓
DESCRIBE EMISSIONS
START Coning STOP ✓
EMISSION COLOR
START Light Brown STOP ✓
PLUME TYPE: CONTINUOUS ☒ FUGITIVE ☐ INTERMITTENT ☐
WATER DROPLETS PRESENT: NO ☒ YES ☐
IF WATER DROPLET PLUME: ATTACHED ☐ DETACHED ☐
POINT IN THE PLUME AT WHICH OPACITY WAS DETERMINED
START Exit Point STOP ✓
DESCRIBE BACKGROUND
START Blue Sky STOP ✓
BACKGROUND COLOR
START Blue STOP ✓
SKY CONDITIONS
START Clear STOP ✓
WIND SPEED
START 0-5 STOP ✓
WIND DIRECTION
START W STOP ✓
AMBIENT TEMP.
START 53° STOP ✓
WET BULB TEMP.
RH.percent 35%



COMMENTS Fuel — Lignite Coal

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TITLE
DATE

OBSERVATION DATE 10-26-92
START TIME 1300
STOP TIME 1306

SEC / MIN	0	15	30	45	SEC / MIN	0	15	30	45
1	10	10	10	10	31				
2	10	15	15	10	32				
3	10	10	10	10	33				
4	10	15	15	15	34				
5	10	10	10	10	35				
6	10	10	10	15	36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
19					49				
20					50				
21					51				
22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				
30					60				

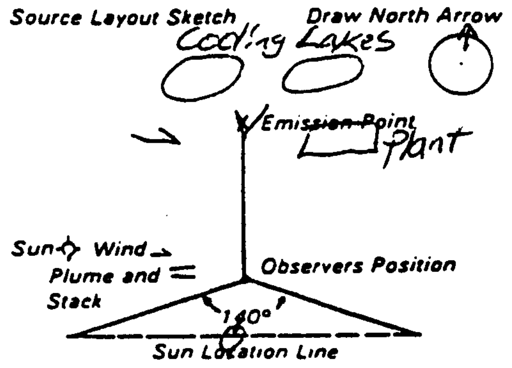
AVERAGE OPACITY FOR HIGHEST PERIOD 11.25
NUMBER OF READINGS ABOVE 11.25 % WERE 6
RANGE OF OPACITY READINGS MINIMUM 10 MAXIMUM 15
OBSERVER'S NAME (PRINT) JEFF BERGSTROM
OBSERVER'S SIGNATURE Jeff Bergstrom
DATE 10-26-92
ORGANIZATION Interpoll Labs
CERTIFIED BY E.T.A. Minneapolis MN.
DATE Sept. 1992
VERIFIED BY
DATE

Interpoll Laboratories
(612)786-6020

Visible Emissions Form

Test #2

SOURCE NAME Otter Tail Power
ADDRESS
CITY Big Stone
STATE S.D.
ZIP
PHONE
SOURCE ID NUMBER
PROCESS EQUIPMENT No. 1 Boiler
OPERATING MODE 100%
CONTROL EQUIPMENT Precip.
OPERATING MODE 100%
DESCRIBE EMISSION POINT
START Round Stack STOP ✓
HEIGHT ABOVE GROUND LEVEL
START 600' STOP ✓
HEIGHT RELATIVE TO OBSERVER
START 600' STOP ✓
DISTANCE FROM OBSERVER
START 1800' STOP ✓
DIRECTION FROM OBSERVER
START N STOP ✓
DESCRIBE EMISSIONS
START Light Brown/[illegible] STOP ✓
EMISSION COLOR
START Light Brown STOP ✓
PLUME TYPE: CONTINUOUS ☑ FUGITIVE ☐ INTERMITTENT ☐
WATER DROPLETS PRESENT: NO ☑ YES ☐
IF WATER DROPLET PLUME: ATTACHED ☐ DETACHED ☐
POINT IN THE PLUME AT WHICH OPACITY WAS DETERMINED
START Exit Point STOP ✓
DESCRIBE BACKGROUND
START Blue Sky STOP ✓
BACKGROUND COLOR
START Blue STOP ✓
SKY CONDITIONS
START Clear STOP ✓
WIND SPEED
START 0-5 STOP ✓
WIND DIRECTION
START W STOP ✓
AMBIENT TEMP.
START 53° STOP ✓
WET BULB TEMP.
RH.percent 35%



OBSERVATION DATE 10-26-92
START TIME 1320
STOP TIME 1326

MIN \ SEC	0	15	30	45	MIN \ SEC	0	15	30	45
1	10	10	10	10	31				
2	10	10	10	10	32				
3	10	10	10	10	33				
4	10	10	10	10	34				
5	10	10	10	10	35				
6	10	10	10	10	36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
19					49				
20					50				
21					51				
22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				
30					60				

AVERAGE OPACITY FOR HIGHEST PERIOD 10
NUMBER OF READINGS ABOVE 10 % WERE 0
RANGE OF OPACITY READINGS
MINIMUM 10 MAXIMUM 10
OBSERVER'S NAME (PRINT) JEFF BERGSTROM
OBSERVER'S SIGNATURE Jeff Bergstrom
DATE 10-26-92
ORGANIZATION Interpoll Labs
CERTIFIED BY ETA Minneapolis MN
DATE Sept. 1992
VERIFIED BY
DATE

COMMENTS Fuel - Lignite Coal

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TITLE
DATE

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Visible Emissions Form

Test #3

SOURCE NAME OtterTail Power

ADDRESS

CITY Big Stone
STATE S.D.
ZIP

PHONE
SOURCE ID NUMBER

PROCESS EQUIPMENT No. 1 Boiler
OPERATING MODE 100%

CONTROL EQUIPMENT Precip
OPERATING MODE 100%

DESCRIBE EMISSION POINT
START Round Stack STOP ✓

HEIGHT ABOVE GROUND LEVEL START 600' STOP ✓
HEIGHT RELATIVE TO OBSERVER START 600' STOP ✓

DISTANCE FROM OBSERVER START 1800' STOP ✓
DIRECTION FROM OBSERVER START ✓ STOP ✓

DESCRIBE EMISSIONS
START Coning STOP ✓

EMISSION COLOR START Light Brown STOP ✓
PLUME TYPE: CONTINUOUS ☑ FUGITIVE ☐ INTERMITTENT ☐

WATER DROPLETS PRESENT: NO ☑ YES ☐
IF WATER DROPLET PLUME: ATTACHED ☐ DETACHED ☐

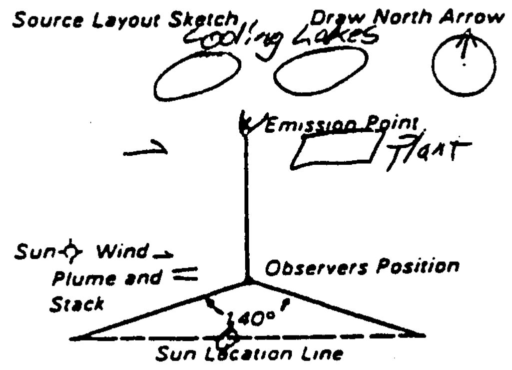
POINT IN THE PLUME AT WHICH OPACITY WAS DETERMINED
START Exit Point STOP ✓

DESCRIBE BACKGROUND
START Sky STOP

BACKGROUND COLOR START Blue STOP ✓
SKY CONDITIONS START Clear STOP ✓

WIND SPEED START 5-8 STOP ✓
WIND DIRECTION START W STOP ✓

AMBIENT TEMP. START 59° STOP ✓
WET BULB TEMP.
RH. percent 36%



OBSERVATION DATE 10-26-92
START TIME 1345
STOP TIME 1351

MIN \ SEC	0	15	30	45	MIN \ SEC	0	15	30	45
1	10	10	10	10	31				
2	10	10	10	10	32				
3	10	10	10	10	33				
4	10	10	10	10	34				
5	10	10	10	10	35				
6	10	10	10	10	36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
19					49				
20					50				
21					51				
22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				
30					60				

AVERAGE OPACITY FOR HIGHEST PERIOD 10
NUMBER OF READINGS ABOVE 10 % WERE 0

RANGE OF OPACITY READINGS
MINIMUM 10 MAXIMUM 10

OBSERVER'S NAME (PRINT) JEFF BERGSTROM

OBSERVER'S SIGNATURE Jeff Bergstrom
DATE 10-26-92

ORGANIZATION Interpoll Labs

CERTIFIED BY E.I.A. Minneapolis mn.
DATE Sept. 1992

VERIFIED BY
DATE

COMMENTS Fuel - Lignite Coal

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SIGNATURE

TITLE
DATE

Interpoll Laboratories
(612)786-6020

Field Data Sheet For HCL, HBR, HFCL

Job O.T.P. / Big Stone S.D
Test Location No. 1 Boiler Stack

Date 10-26-92
Operator(s) JEFF BERGSTROM
Test 4 Run 1
Console No. 25
Bar. Pressure 28.71 in. Hg.

γ .9939

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1530)	108.670	///////	///////	///////	///////	///////	///////
5	108.866	1000		2.5	294	77	
10	109.060	1000		2.5	293	78	
15	109.256	1000		2.5	304	80	
20	109.451	1000		2.5	305	82	
25	109.644	1000		2.5	305	83	
30	109.838	1000		2.5	304	85	
35	110.023	1000		2.5	305	87	
40	110.216	1000		2.5	309	88	
45	110.410	1000		2.5	309	90	
50	110.610	1000		2.5	308	92	
55	110.810	1000		2.5	308	94	
60	111.002	1000		2.5	308	95	

V_m: 2.332 CF

t_m: 85.916 °F

S-290BL-R

Interpoll Laboratories
(612)786-6020

Field Data Sheet For HCL, HBR, HF, CL

Job O.T.P. / Big Stone S.D

Test Location No. 1 Boiler Stack

Date 10-26-92

Operator(s) JEFF BERGSTROM

Test 4 **Run** 2

Console No. 25

Bar. Pressure 28.71 **in. Hg.**

γ .9939

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (^0F)	Meter Temp. (^0F)	
(1650)	111.050	///////	///////	///////	///////	///////	///////
5	111.250	1000		2.5	306	98	
10	111.450	1000		2,5	306	99	
15	111.647	1000		2.5	305	99	
20	111.845	1000		2.5	305	100	
25	112.039	1000		2,5	304	100	
30	112,235	1000		2.5	304	101	
35	112.435	1000		2.5	305	102	
40	112.636	1000		2.5	305	102	
45	112.833	1000		2,5	305	102	
50	113.030	1000		2,5	305	102	
55	113.227	1000		2.5	305	103	
60	113,422	1000		2.5	305	104	

(1750) V_m: 2.372 CF t_m: 101 °F

S-290BL-R

Interpoll Laboratories
(612)786-6020

Field Data Sheet For HCL, HBR, HF, CL

Job O.T.P./ Big Stone S.D
Test Location No.1 Boiler Stack

Date 10-26-92
Operator(s) JEFF BERGSTROM
Test 4 **Run** 3
Console No. 25
Bar. Pressure 28.71 **in. Hg.**

Y .9939

☑ **Pretest Leak Check**
(0 **cc/min at** 17 **IN.HG. VAC)**

☑ **Post Test Leak Check**
(0 **cc/min at** 17 **IN.HG. VAC)**

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1845)	113.491	///////	///////	///////	///////	///////	///////
5	113.683	1000		2.5		103	
10	113.845	1000		2.5		104	
15	114.041	1000		2.5		104	
20	114.288	1000		2.5		104	
25	114.487	1000		2.5		104	
30	114.686	1000		2.5		104	
35	114.884	1000		2.5		105	
40	115.080	1000		2.5		105	
45	115.277	1000		2.5		105	
50	115.475	1000		2.5		106	
55	115.673	1000		2.5		106	
60	115.873	1000		2.5		106	

V_m: 2.382 CF

t_m: 104.666 °F

S-290BL-R

Interpoll Laboratories
(612)786-6020

Field Data Sheet For BTX

Job O.T.P. / BigStone S.D.

Test Location No. 1 Boiler Stack

Date 10-26-92

Operator(s) JEFF BERGSTROM

Test 5 Run 1

Console No. 55

Bar. Pressure 28.71 in. Hg.

γ .9941

☑ Pretest Leak Check
(0 cc/min at 20 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1530)	196.255	///////	///////	///////	///////	///////	///////
5	196.441	1000		2.5	294	76	
10	196.630	1000		2.5	294	78	
15	196.816	1000		2.5	304	80	
20	197.005	1000		2.5	305	83	
25	197.195	1000		3.5	305	85	
30	197.387	1000		5	304	88	
35	197.579	1000		2.5	305	90	
40	197.776	1000		3	304	92	
45	197.971	1000		3.5	309	94	
50	198.163	1000		4.5	308	96	
55	198.355	1000		5	308	98	
60	198.547	1000		5	308	99	

(1630) V_m: 2.292 CF t_m: 88.25 °F

V_{STD} = 2.1057

S-290BL-R

Interpoll Laboratories
(612)786-6020

Field Data Sheet For B,T,X

Job O.T.P / Big Stone S.D
Test Location No. 1 Boiler Stack

Date 10-26-92
Operator(s) JEFF BERGSTROM
Test 5 Run 2
Console No. 55
Bar. Pressure 28.71 in. Hg.

γ .9941

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1650)	198.560	///////	///////	///////	///////	///////	///////
5	198.752	1000		2.5	306	98	
10	198.947	1000		2.5	306	99	
15	199.141	1000		3	305	100	
20	199.332	1000		3.5	305	101	
25	199.523	1000		4.5	304	102	
30	199.715	1000		5.5	304	103	
35	199.915	1000		2.5	305	103	
40	200.110	1000		2.5	305	103	
45	200.305	1000		2.5	305	103	
50	200.496	1000		3.5	305	105	
55	200.687	1000		4	305	106	
60	200.883	1000		4.5	304	107	

V_m: 2.323 CF

t_m: 102.5 °F

V_{STD} = 2.0801

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Field Data Sheet For BTX

Job O.T.P./Bigstone S.D.
Test Location No. 1 Boiler Stack

Date 10-26-92
Operator(s) JEFF BERGSTROM
Test 5 Run 3
Console No. 55
Bar. Pressure 28.71 in. Hg.

γ ,9941

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1815)	200.895	///////	///////	///////	///////	///////	///////
5	201.095	1000		2,5		103	
10	201.292	1000		2,5		104	
15	201.485	1000		2,5		104	
20	201.678	1000		2,5		105	
25	201.872	1000		2,5		105	
30	202.066	1000		2,5		106	
35	202,260	1000		2,5		106	
40	202.453	1000		2.5		107	
45	202.655	1000		2.5		107	
50	202.850	1000		2.5		107	
55	203.046	1000		2.5		108	
60	203.245	1000		2,5		108	

V_m: 2,35 CF

t_m: 105,833 °F

V_{STD} = 2.0919

S-290BL-R

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job ANL/OTF BIG STONE

Source Boiler Stack

Test 6 Run 0 Date 10/27/92

Stack dimen. 290 IN.

Dry bulb ____ °F Wet bulb ____ °F

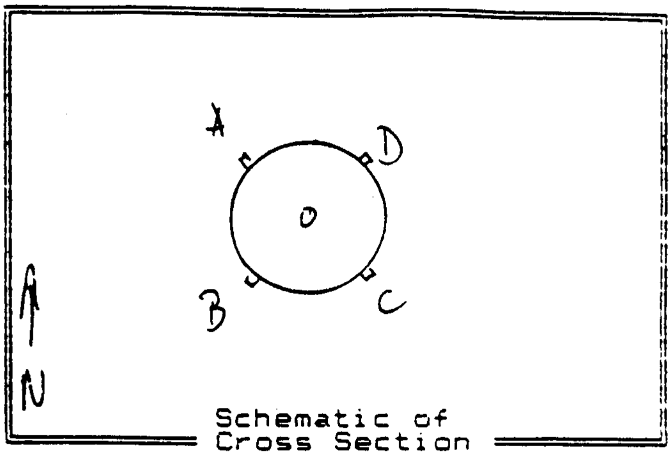
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.

Barometric pressure 28.46 in Hg

Static pressure -1.3 in WC

Operators D VanHoecke & K. Rosenthal

Pitot No. 4M5-8 Cp .84



Schematic of Cross Section

Metals (Coal + RDF)

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: in.		Time start: hrs	
A	1					
	2					
	3					
B	1					
	2					
	3					
C	1					
	2					
	3					
D	1					
	2					
	3					
Temp. meas. tool & S/N: PDT #18					Time end: hrs	

R or nothing=[✓ reg] manometer: S= expanded: E = electronic S-392.1

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job ANL/OTR? Big Stone
Source Boiler Stack
Method 0012 Filter holder: teflon

Date 10/27/92 Test 6 Run 1
No. of traverse points 12
Filter type: pallflex glass fiber

Sample Train Leak Check:

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

Particulate Catch Data:

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

☐ acetone
☒ other(s) .1NHNO$_3$ + MECL$_2$

No. of probe wash bottles:
Sample recovered by:

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	716	400	316
Impinger No. 3			
Condenser			
Desiccant	1439	1410	29
Total	▨	▨	345

Integrated Gas Sampling Data:

Bag Pump No. 24h Box No. 1 Bag No. 1
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 14 in. Hg.
Time start: 0934 (HRS) Time end: 1141 (HRS)
Sampling rate: 300 cc/min Operator: DLH
S/N of O$_2$ Analyzer used to monitor train outlet: 4

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job ANL/OTP BIG STONE
Source BOILER STACK
Date 10/27/87 Test 2 Run 1

Operators DUN + KR
Meter Box No. 3 ^H@ 1.51 IN WC
Gasmeter coeff. .9954

Pitot No. 4115-8 Cp .84
Bar. Press. 28.46 inHg H2O 13 %
Nozzle No. Glass Nozzle Dia .32 IN.

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	Impg.	Gas/In	Gas/Out	Oxygen (%v/v)
	0930	683.61											
B 3	5	688.07	1.40	2.56	8.12	11	290	253	251	42	54	54	8.0
3	10	692.72	1.45	2.65	2.70	12	290				56	55	8.0
2	15	697.16	1.40	2.56	7.20	12	292				58	55	8.0
2	20	701.55	1.35	2.47	1.63	12	292	255	256	43	58	55	8.0
1	25	705.82	1.25	2.29	5.90	12	292				59	57	8.0
1	30	710.16	1.25	2.29	0.19	11	292				61	58	8.0
C 3	35	714.58	1.35	2.48	4.65	12	292	257	258	43	62	58	8.0
3	40	719.17	1.40	2.58	9.20	12	291				62	59	7.7
2	45	723.58	1.35	2.48	3.66	12	295				63	60	8.0
2	50	728.07	1.35	2.48	8.13	12	295	256	257	43	64	60	7.9
1	55	732.32	1.25	2.31	2.45	11	293				65	60	7.6
1	60	736.71	1.25	2.31	6.77	11	293				66	61	7.8
D 3	65	741.23	1.35	2.50	1.26	12	293	253	256	44	67	62	7.8
3	70	745.71	1.35	2.51	5.76	12	293				68	63	7.8
2	75	750.16	1.35	2.51	0.27	12	293				69	64	7.7
2	80	754.80	1.35	2.51	4.79	12	293	254	256	44	69	65	7.6
1	85	759.16	1.25	2.33	9.15	11	293				70	66	7.5
1	90	763.48	1.25	2.34	3.51	11	293				71	67	7.6
A 3	95	768.06	1.35	2.53	8.05	12	293	256	257	43	72	68	7.6
3	100	772.54	1.35	2.53	2.60	13	293				72	68	7.7
2	105	777.14	1.35	2.53	7.15	13	293				73	69	7.6
2	110	781.66	1.35	2.54	1.71	13	293	257	258	43	73	70	7.6
1	115	786.02	1.20	2.26	6.01	13	293				73	70	7.5
1	120	790.30	1.25	2.34	0.39		298				75	71	7.6
	1141												
	θ = 120	Vm = 106.69		^H = 2.45							Avg. = 64		

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job ANL/OTP BIG STONE
Date 10/27/82 Test 6 Run 3
Source Boiler Stack
No. of traverse points 12
Method 0012 Filter holder: teflon
Filter type: Pallflex glass fiber

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Posttest: 0 cfm at 10 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used:
Recovery solvent(s)
☐ acetone
☒ other(s) .1N HNO3 + MeCl2

No. of probe wash bottles: 2
Sample recovered by: DrH

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Impinger No. 1	679	488	191
Impinger No. 2		400	
Impinger No. 3	499	497	2
Condenser			
Desiccant	1410	1392	18
		13	
Total	▨		211

Integrated Gas Sampling Data:

Bag Pump No. 29b Box No. 1 Bag No. 3
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 14 in. Hg.
Time start: 1508 (HRS) Time end: 1717 (HRS)
Sampling rate: 300 cc/min Operator: DrH
S/N of O_2 Analyzer used to monitor train outlet: 4

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job ANL/OTP BIG STONE
Source BOILER STACK
Date 10/27/92 Test 6 Run 2

Operators DAH & KR
Meter Box No. 3 *H@ 1.81 IN WC
Gasmeter coeff. .9984

Pitot No. 4175-8 Cp .84
Bar. Press. 28.47 inHg H2O 13.6 %
Nozzle No. Glass Nozzle Dia .186 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgs. Vol. (cf)	VAC. inHg	Temperatures (°F)						Oxygen
							Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	(%v/v)
	1220	790.86											
B 3	5	793.57	1.35	.86	3.50	7	304	257	260	43	71	70	7.3
3	10	796.31	1.40	.90	6.22	6	304				75	72	7.6
2	15	798.96	1.35	.87	8.91	6	304				78	73	7.6
2	20	801.61	1.35	.88	1.61	6	304	257	261	42	79	73	7.4
1	25	804.22	1.30	.84	4.27	6	304				81	74	7.4
1	30	806.87	1.30	.85	6.93	6	304				82	75	7.5
C 3	35	809.66	1.35	.88	9.64	6	304	261	260	42	82	76	7.5
3	40	812.31	1.35	.88	2.36	6	306				83	76	7.5
2	45	815.06	1.35	.88	5.08	6	303				83	77	7.6
2	50	817.84	1.40	.92	7.86	6	303	260	261	43	83	77	7.6
1	55	820.53	1.35	.88	0.58	6	304				83	77	7.7
1	60	823.29	1.30	.85	3.25	6	304				83	18	7.5
D 3	65	826.02	1.40	.92	6.03	6	304	260	263	43	83	78	7.7
3	70	828.84	1.40	.92	8.80	6	304				84	78	7.7
2	75	831.63	1.45	.95	1.63	6	306				84	78	7.6
2	80	834.46	1.40	.92	4.40	6	306	258	261	43	84	79	7.8
1	85	837.08	1.25	.82	7.03	6	306				84	79	7.8
1	90	839.69	1.25	.82	9.65	6	308				84	79	7.7
A 3	95	842.49	1.45	.95	2.47	6	308	257	260	43	84	79	7.8
3	100	845.36	1.50	.98	5.34	6	308				85	79	7.9
2	105	848.23	1.45	.95	8.17	6	308				85	80	7.7
2	110	851.06	1.45	.95	1.00	6	308	260	262	43	85	80	7.9
1	115	853.78	1.35	.88	3.73	6	308				86	80	7.9
1	120	856.45	1.35	.85	6.46		308				86	80	7.8
	(1432)												
	Θ = 130	Vm = 65.59		^H = .89							Avg. = 79.6		

INTERFOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job ANL/OTP Big Stone
Source Boiler Stack
Method 0012 Filter holder: teflon
Date 10/27/92 Test 6 Run 2
No. of traverse points 12
Filter type: pallflex glass fiber

Sample Train Leak Check:

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

Particulate Catch Data:

No.s of filters used:

Recovery solvent(s)
☐ acetone
☒ other(s) 1N HNO3 + MeCl2

No. of probe wash bottles: 2
Sample recovered by: DW

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	589	400	189
Impinger No. 3			
Condenser			
Desiccant	1345	1308	37
Total			226

Integrated Gas Sampling Data:

Bag Pump No. 29b Box No. 1 Bag No. 2
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 14 in. Hg.
Time start: 1225 (HRS) Time end: 1432 (HRS)
Sampling rate: 300 cc/min Operator: DWH
S/N of O_2 Analyzer used to monitor train outlet: 4

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job ANL/OTP BIG STONE
Source BOILER STACK
Date 10/27/92 Test B Run 3

Operators ~~BK~~ DLH + KF
Meter Box No. 3 ^H@ 1.81 IN WC
Gasmeter coeff. .9987

Pitot No. 4m5-8 Cp .84
Bar. Press. 28.92 inHg H2O ___ %
Nozzle No. ___ Nozzle Dia ___ IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dec. Vol. (cf)	VAC. inHg	Temperatures (°F)						Oxygen
							Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	(%v/v)
	1505	856.78											
A 3	5	859.60	1.5	.97	9.63	5	308	237	243	46	76	76	8.1
3	10	862.53	1.55	.99	2.50	5	308				79	77	8.1
2	15	865.87	1.30	0.87	5.88	5	308				81	77	9.1
2	20	868.74	1.50	.77	8.73	5	308	243	246	47	82	78	8.2
1	25	871.63	1.40	.90	1.49	5	308				84	79	7.9
1	30	874.13	1.35	.87	4.20	5	308				84	79	8.1
D 3	35	877.18	1.60	1.04	7.15	6	308	245	246	48	84	80	8.0
3	40	880.19	1.60	1.03	0.10	6	312				85	80	8.3
2	45	883.04	1.60	1.03	3.05	6	312				85	80	8.0
2	50	886.02	1.60	1.03	6.00	6	315	247	251	49	86	80	8.0
1	55	888.90	1.50	.97	8.85	6	315				86	80	8.4
1	60	891.81	1.50	.96	1.70	6	317				87	81	8.2
B 3	65	894.82	1.70	1.09	4.74	6	317	251	252	49	87	81	8.5
3	70	897.83	1.70	1.09	7.78	6	317				88	82	8.4
2	75	900.86	1.65	1.06	0.78	6	317				88	83	8.7
2	80	903.76	1.50	.97	3.65	6	317	256	254	49	89	83	8.3
1	85	906.55	1.50	.97	6.51	6	318				90	84	8.0
1	90	909.39	1.50	.97	9.38	6	318				90	84	8.2
C 3	95	912.36	1.60	1.04	2.35	6	316	257	256	48	90	84	8.2
3	100	915.31	1.60	1.04	5.32	6	316				90	84	8.3
2	105	918.26	1.60	1.04	8.29	6	316				90	85	8.0
2	110	921.27	1.60	1.04	1.26	6	316	258	257	47	90	85	8.0
1	115	924.26	1.50	.97	4.13		316				90	85	7.9
1	120	927.40	1.50	.97	7.01		316				90	85	7.9
	(1717)												
	θ = 120	Vm = 70.62		^H = 1.02							Avg. = 80.5		

S-0037R

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job ANL / OTP - BIGSTONE

Source BOILER STACK

Test 7 Run O-3 Date 10-27-92

Stack dimen. 290.0 IN.

Dry bulb ______ °F Wet bulb ______ °F

Manometer: ☒ Reg. ☐ Exp. ☐ Elec.

Barometric pressure 28.71 in Hg

Static pressure -1.3 in WC

Operators Ron Rosenthal, Mark Kaehler

Pitot No. MMS-8 Cp .840

Schematic of Cross Section

Dioxin (Coal + RDF) (PAH/PCB Aliquot)

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: 13 in.		Time start: hrs	
	1					
	2					
	3					
	1					
	2					
	3					
	1					
	2					
	3					
	1					
	2					
	3					
Temp. meas. tool & S/N:					Time end: hrs	

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

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job ANL/OTP-Bigstone
Date 10-27-92 Test 7 Run 1
Source BOILER STACK
No. of traverse points 12
Cyclone: ☐ Yes ☒ No Filter holder: MM5
Filter type: 4" Glass fiber
Analytes:
XAD-2 resin: ___ g Batch No. ___
Field recovery spike added: ☐ Yes ☐ No
Reference: EPA SW-846 Method 0010

NOTE: DIOXIN, PAH, PCB Sample TRAIN

Sample Train Leak Check:

Pretest: < 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: .00 cfm at 14 in. Hg. (vac) ☒
Pitot @ IN W.C.

Semivolatile Catch Data:

No.s of filters used: 0255

Recovery solvent(s)
☒ MeCl2 * MeOH (50:50 v/v)
☐ other(s)

No. of bottles for condensate trap catch: 1
Samples recovered by: M.K.

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Condensate trap	573	273	300
Condensate trap			
Condensate trap			
Impingers			
Desiccant	1420	1393	27
Total	▨	▨	327

Integrated Gas Sampling Data:

Bag Pump No. 23A Box No. 22 Bag No. 1
Bag Material: 4-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 20 in. Hg.
Time start: 930 (HRS) Time end: 1137 (HRS)
Sampling rate: 200 cc/min O2 Analyzer S/N: 7

Job ANL/OTP - Bigstone
Source BOILER STACK
Date 10-27-92 Test 17 Run 1
Operators R.R. & M.K.
Meter Box No. 9
Gasmeter coeff. 1.0001
Nozzle No. MM5-4
Nozzle Dia. .234 in.
Bar. Pres. 28.71 in. Hg
Pitot No. MM5-8
Cp .840
H₂O 14 %

NOTE DIOXIN PAH, PCB SAMPLE TRAIN

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgs. Vol. (cf)	VAC. inHg	Temperatures (°F)							Oxygen
							Stack	Probe	Oven	XAD2	Imp.	Meter(In/Out)		(%v/v)
	930	94.70												
D- 3	5	99.01	1.35	2.18	8.90	9	289	245	239	57	46	53	53	7.9
3	10	103.08	1.35	2.16	3.08	10	291					56	54	7.2
2	15	107.20	1.30	2.09	7.20	10	291	245	240	46	45	59	55	7.5
2	20	111.41	1.35	2.18	1.41	10	291					61	56	7.5
1	25	115.50	1.25	2.03	5.47	11	291	247	240	47	45	62	57	7.5
1	30	1199.39	1.15	1.86	9.38	10	293					64	58	7.5
A- 3	35	123.70	1.40	2.27	3.70	11	293	253	240	49	53	64	59	7.7
3	40	127.96	1.35	2.20	7.94	12	292					65	59	7.4
2	45	132.11	1.30	2.11	2.11	12	294	256	250	47	48	66	60	7.5
2	50	136.20	1.25	2.03	6.20	11	295					67	61	7.4
1	55	140.30	1.20	1.95	0.22	11	295	255	245	47	48	68	62	7.5
1	60	144.16	1.15	1.88	4.16	11	294					69	63	7.5
B- 3	65	148.39	1.30	2.12	8.35	12	297	250	255	48	52	68	63	7.5
3	70	152.66	1.35	2.20	2.61	12	297					70	64	7.6
2	75	156.92	1.35	2.21	6.89	12	297	250	270	49	47	70	65	7.7
2	80	161.20	1.35	2.21	1.17	12	297					71	65	7.5
1	85	165.15	1.15	1.87	5.12	11	301	249	265	50	50	71	66	7.7
1	90	169.04	1.15	1.87	9.07	11	301					72	66	7.7
C- 3	95	173.40	1.40	2.28	3.43	12	301	247	250	51	50	71	67	7.7
3	100	177.81	1.40	2.29	7.79	12	299					72	67	7.5
2	105	182.01	1.30	2.13	2.01	12	299	253	250	51	49	72	68	7.7
2	110	186.35	1.30	2.13	6.22	11	299					72	68	7.7
1	115	190.27	1.15	1.88	0.18	11	301	255	250	53	54	73	68	7.7
1	120	194.20	1.15	1.88	4.15	10	301					73	68	7.7
	1137													
	θ=120	Vm=99.50		ΔH=2.08								Avg.=64.6		

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job ANL/OTP- Bigstone. Date 10-27-92 Test 7 Run 2
Source BOILER STACK No. of traverse points 12
Cyclone: ☐ Yes ☐ No Filter holder: MM5 Filter type: 4" Glass fiber
Analytes: XAD-2 resin: ___ g Batch No. ___
Field recovery spike added: ☒ Yes ☐ No Reference: EPA SW-846 Method 0010
NOTE: DIOXIN PAH PCB SAmple TRAIN

Sample Train Leak Check:

Pretest: < 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: 100 cfm at 13 in. Hg. (vac) ☒

Semivolatile Catch Data:

No.s of filters used: 0256

Recovery solvent(s)
☐ MeCl2 * MeOH (50:50 v/v)
☐ other(s) ___

No. of bottles for condensate trap catch: 1
Samples recovered by: m.k.

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Condensate trap	575	273	302
Condensate trap			
Condensate trap			
Impingers			
Desiccant	1433	1410	23
Total	▨	▨	325

Integrated Gas Sampling Data:

Bag Pump No. 23A Box No. 22 Bag No. 2
Bag Material: 4-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 20 in. Hg.
Time start: 1245 (HRS) Time end: 1451 (HRS)
Sampling rate: 200 cc/min O2 Analyzer S/N: 7

Rev.1 CF-026

Job ANL/OTP - Bigstone
Source Boiler Stack
Date 10-27-92 Test D Run 2

Operators R.R. & M.K.
Meter Box No.
Gasmeter coeff. 1.0001

Nozzle No. mms-4
Nozzle Dia. .234 in.
Bar. Pres. 28.46 in. Hg

Pitot No. mms-8
Cp .840
H$_2$O 74 %

NOTE: DIOXIN; PCB; PAH; SAMPLE TRAIN

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Des. Vol. (cf)	VAC. inHg	Temperatures (°F)							Oxygen
							Stack	Probe	Oven	XAD2	Imp.	Meter(In/Out)		(%v/v)
	1245	194.75												
D- 3	5	199.12	1.40	2.26	9.06	11	303	256	245	48	54	67	66	7.4
3	10	203.41	1.40	2.27	3.40	10	303					69	67	7.5
2	15	207.65	1.35	2.18	7.66	10	306	260	255	50	55	73	68	7.5
2	20	211.94	1.35	2.19	1.94	10	306					74	69	7.5
1	25	216.04	1.25	2.03	6.06	9	308	260	255	53	57	75	69	7.5
1	30	220.07	1.20	1.95	0.10	9	308					76	70	7.5
A- 3	35	224.53	1.40	2.28	4.47	11	308	254	255	48	57	76	70	7.5
3	40	228.90	1.40	2.28	8.85	11	308					77	71	7.4
2	45	233.17	1.30	2.12	3.07	11	309	259	265	54	58	78	72	7.5
2	50	237.42	1.35	2.20	7.37	10	310					78	72	7.5
1	55	241.55	1.20	1.96	1.43	10	310	260	270	55	59	78	72	7.5
1	60	245.41	1.20	1.95	5.49	9	312					79	73	7.5
B- 3	65	249.74	1.35	2.20	9.80	11	311	251	255	50	52	78	73	7.7
3	70	254.14	1.40	2.28	4.18	11	311					80	73	7.9
2	75	258.60	1.45	2.37	8.65	10	311	247	230	47	54	80	74	8.3
2	80	263.10	1.40	2.29	3.05	11	311					80	74	7.9
1	85	267.19	1.30	2.12	7.28	11	312	240	228	58	54	81	74	7.9
1	90	271.54	1.35	2.20	1.60	10	314					81	74	7.9
C- 3	95	275.85	1.40	2.28	5.99	11	313	245	230	52	60	79	74	7.9
3	100	280.51	1.45	2.36	0.45	11	313					81	75	7.9
2	105	284.90	1.40	2.29	4.85	11	313	245	235	48	59	82	75	7.9
2	110	289.20	1.35	2.21	9.18	11	312					82	75	7.9
1	115	293.55	1.30	2.13	3.43	10	312	241	235	52	59	82	76	7.9
1	120	297.63	1.25	2.04	7.59	10	314					82	76	7.9
	1451													
	θ=120	Vm=102.88		ΔH=2.19								Avg. = 75		

INTERPOLL LABORATORIES EPA MODIFIED METHOD 5 SAMPLE LOG SHEET

Job ANL/OTP - BigStone
Date 10-27-92 Test 7 Run 3
Source BOILER STACK
No. of traverse points 12
Cyclone: ☐ Yes ☒ No Filter holder: MMS
Filter type: 4" Glass fiber
Analytes:
XAD-2 resin: ___ g Batch No. ___
Field recovery spike added: ☒ Yes ☐ No
Reference: EPA SW-846 Method 0010

NOTE DIOXIN, PAH, PCB, SAMPLE TRAIN

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Posttest: .00 cfm at 14 in. Hg. (vac) ☒

Semivolatile Catch Data:

No.s of filters used: 0257

Recovery solvent(s)
☐ MeCl2 * MeOH (50:50 v/v)
☐ other(s) ______

No. of bottles for condensate trap catch: 1
Samples recovered by: M.K.

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Condensate trap	563	262	301
Condensate trap			
Condensate trap			
Impingers			
Desiccant	1458	1433	25
Total	▨	▨	326

Integrated Gas Sampling Data:

Bag Pump No. 23A Box No. 22 Bag No. 3
Bag Material: 4-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 0 cc/min at 20 in. Hg.
Time start: 1530 (HRS) Time end: 1735 (HRS)
Sampling rate: 200 cc/min O2 Analyzer S/N: 7

Rev.1 CF-026

Job ANL/OTP- Bigstone
Source BOZIER STACK
Date 10-27-92 Test 7 Run 3

Operators R.R. & M.K.
Meter Box No. 7
Gasmeter coeff. 1.0001

Nozzle No. MMS-4
Nozzle Dia. .234 in.
Bar. Pres. 28.46 in. Hg

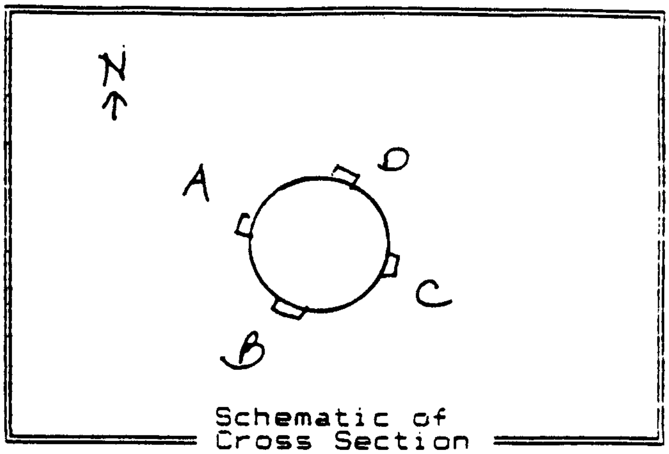
Pitot No. MMS-8
Cp .840
H_2O %

NOTE! DIOXIN, PAH, PCB SAMPLE TRAIN

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Del. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	XAD2	Imp.	Meter (In/Out)		Oxygen (%v/v)
	1530	297.80												
A - 3	5	302.38	1.45	2.37	2.26	5.5	314	250	240	46	55	70	70	7.9
3	10	306.89	1.50	2.43	6.77	6.0	314					74	71	8.0
2	15	311.16	1.40	2.28	1.13	5.5	315	258	246	46	53	76	72	8.1
2	20	315.50	1.40	2.28	5.52	5.5	315					78	73	8.1
1	25	319.79	1.35	2.21	9.83	5.75	315	260	250	44	54	79	73	7.9
1	30	324.03	1.30	2.12	4.06	5.75	317					80	74	8.3
B - 3	35	328.58	1.50	2.46	8.62	6.0	316	254	241	43	54	80	74	8.3
3	40	333.20	1.55	2.54	3.25	6.25	316					81	75	8.4
2	45	337.84	1.56	2.46	7.81	6.5	317	257	248	43	55	81	74	8.4
2	50	342.32	1.45	2.38	2.29	6.0	317					80	75	8.3
1	55	346.77	1.45	2.38	6.78	6.0	316	251	244	43	55	81	75	8.1
1	60	351.32	1.50	2.46	1.33	6.25	318					81	75	8.5
C - 3	65	355.79	1.45	2.37	5.82	6.25	318	259	251	42	54	81	75	8.4
3	70	360.25	1.40	2.30	0.23	6.0	316					81	75	8.3
2	75	364.58	1.35	2.22	4.56	6.0	315	260	245	42	55	81	76	8.2
2	80	368.84	1.30	2.14	8.82	6.0	314					81	76	8.3
1	85	373.21	1.40	2.29	3.23	6.0	319	253	247	43	56	81	76	8.0
1	90	377.61	1.35	2.21	7.56	6.0	318					81	76	7.9
D - 3	95	381.92	1.40	2.30	1.98	6.0	316	257	250	44	54	81	76	8.2
3	100	386.42	1.45	2.38	6.47	6.25	317					81	76	8.1
2	105	391.00	1.50	2.46	1.03	6.50	318	252	247	44	53	81	76	7.9
2	110	395.56	1.50	2.46	5.59	6.50	318					81	76	8.3
1	115	399.96	1.35	2.22	9.92	6.25	317	259	250	44	53	80	75	8.1
1	120	404.381	1.30	2.13	4.17	6.25	317					80	76	8.1
	1735													
	θ= 120	Vm= 106.58		ΔH= 2.33								Avg.= 77.1		

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job OTP BIG STONE
Source NO 1 BOILER STACK
Test 8 Run 0 Date 10-27-92
Stack dimen. 290 IN.
Dry bulb ____ °F Wet bulb ____ °F
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.
Barometric pressure 2846 in Hg
Static pressure -1.5 in WC
Operators GH & JB
Pitot No. U-23-8 Cp .84



Schematic of Cross Section

Particulate - Coal + RDF

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: 13 in.		Time start: 0815 hrs	
A	1	.094	12.75	25.75	1.15	294
	2	.146	4234	55.34	1.36	
	3	.296	85.84	98.84	1.41	
B	1				1.00	293
	2				1.31	
	3				1.30	
C	1				1.11	294
	2				1.32	
	3				1.40	
D	1				1.18	
	2				1.26	294
	3				1.42	
Temp. meas. tool & S/N:			24		Time end: 0830 hrs	

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

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job OTF BIG STONE
Source NC 1 BOILER STACK
Method 5 Filter holder: GLASS
Date 10-27-93 Test 8 Run 1
No. of traverse points
Filter type: GLASS FIBER

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: .00 cfm at 10 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used: 4732

Recovery solvent(s)
☒ acetone
☐ other(s)

No. of probe wash bottles: 1
Sample recovered by: GH

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	660	499	161
Impinger No. 3			
Condenser			
Desiccant	1307	1270	37
Total			198

Integrated Gas Sampling Data:

Bag Pump No. 3 Box No. 2 Bag No. 1
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: 00 cc/min at 15 in. Hg.
Time start: 0930 (HRS) Time end: 1037 (HRS)
Sampling rate: 400 cc/min Operator: GH
S/N of O_2 Analyzer used to monitor train outlet: 3

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job OTT BIG STONE
Source NO 1 BOILER STACK
Date 10-27-92 Test B Run 1

Operators GH & SB
Meter Box No. 4 ΔH@ 1.91 IN WC
Gasmeter coeff. 1.000

Pitot No. 132-8 Cp .84
Bar. Press. 28.46 inHg H2O 4 %
Nozzle No. 4 Nozzle Dia .38 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgs. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Imp.	Gas/In	Gas/Out	Oxygen (%v/v)
	0930	995.00											
C - 3	5	999.60	1.30	2.80	9.60	7	289	255	246	39	58	57	9.0
2	10	1004.01	1.25	2.62	4.01	7	291	255	247	40	61	59	8.2
1	15	1008.37	1.21	2.55	8.37	7	292	256	248	41	66	61	8.3
A - 3	20	1013.10	1.41	2.98	3.10	7	293	257	249	42	70	63	8.1
2	25	1017.60	1.26	2.68	7.60	8	292	258	248	44	71	64	8.1
1	30	1021.83	1.1	2.35	1.82	7	292	262	248	45	71	65	8.2
B - 3	35	1026.44	1.32	2.82	6.44	7	291	265	249	45	76	71	8.2
2	40	1031.02	1.28	2.34	1.03	7	292	266	249	45	76	67	8.0
1	45	1035.37	1.15	2.47	5.37	7	293	267	248	46	77	68	8.0
D - 3	50	1040.15	1.39	2.99	0.15	8	292	265	249	39	80	71	8.1
2	55	1044.75	1.28	2.77	4.76	9	292	266	250	39	80	72	8.0
1	60	1049.27	1.22	2.45	9.27	8	293	265	254	43	81	72	8.2
	(1037)												
	θ = 60	Vm = 54.27		ΔH 2.7							Avg. = 69.0		

S-0037R

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job OTP BIG STONE
Source NO. 1 BOILER STACK
Method 5 Filter holder: GLASS

Date 10-27-95 Test 8 Run 2
No. of traverse points 12
Filter type: GLASS FIBER

Sample Train Leak Check:

Pretest: < 0.02 cfm at 15 in. Hg. (vac) ☒
Posttest: .00 cfm at 10 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used:
4734

Recovery solvent(s)
☒ acetone
☐ other(s)

No. of probe wash bottles: 1
Sample recovered by: GH

Condensate Data:

Item	Weight (g)		
	Final	Tare	Difference
Impinger No. 1			
Impinger No. 2	665	499	166
Impinger No. 3			
Condenser			
Desiccant	1384	1369	15
Total			181

Integrated Gas Sampling Data:

Bag Pump No. 3 Box No. 2 Bag No. 2
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: .00 cc/min at 15 in. Hg.
Time start: 1100 (HRS) Time end: 1215 (HRS)
Sampling rate: 400 cc/min Operator: GH
S/N of O_2 Analyzer used to monitor train outlet: 3

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job OTP Big Stone
Source No 1 Boiler Stack
Date 10-27-92 Port B Run 3

Operators GH + JB
Meter Box No. 7 ^H@ 1.97 IN WC
Gasmeter coeff. 1.0000

Pitot No. 32-8 Cp .84
Bar. Press. 28.96 inHg H2O 14 %
Nozzle No. 23-7 Nozzle Dia .247 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dgs. Vol. (cf)	VAC.	Temperatures (°F)						Oxygen
						inHg	Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	(%v/v)
	1100	49.60											
D-3	5	54.25	1.36	2.85	4.25	7	292	258	265	39	69	69	8.0
2	10	58.96	1.40	2.92	8.92	7	297	260	261	40	74	71	8.0
1	15	63.57	1.30	2.73	35	7	297	262	262	42	78	72	8.0
C 3	20	68.23	1.37	2.89	822	7	297	263	262	44	79	73	8.0
2	25	72.84	1.31	2.79	284	7	297	265	266	47	81	74	8.1
1	30	77.36	1.25	2.65	736	7	298	267	269	46	82	75	8.0
A 3	35	82.13	1.35	2.95	2.13	7	298	262	258	39	82	78	8.0
2	40	86.57	1.20	2.54	654	7	299	260	255	40	81	77	8.0
1	45	90.61	1.0	2.12	061	7	299	256	257	42	85	79	8.2
B 3	50	95.59	1.5	3.19	559	8	299	257	258	44	84	77	8.1
2	55	99.85	1.1	2.34	985	7	300	256	257	44	87	80	8.1
1	60	103.96	1.0	2.14	392	6	300	257	559	46	87	80	8.1
	(1215)												
	θ = 60	Vm = 54.36		^H 2.67							Avg. = 78.1		

S-0037R

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job CTF BIG STONE
Source NCL BOILER STACK
Method 5 Filter holder: GLASS
Date 12 27 92 Test 8 Run 3
No. of traverse points 12
Filter type: GLASS FIBER

Sample Train Leak Check:

Pretest: ≤ 0.02 cfm at 15 in. Hg. (vac) ☒
Postest: .00 cfm at 10 in. Hg. (vac) ☒

Particulate Catch Data:

No.s of filters used: 4735

Recovery solvent(s)
☒ acetone
☐ other(s)

No. of probe wash bottles: 1
Sample recovered by: GH

Condensate Data:

Item	Weight(g)		
	Final	Tare	Difference
Impinger No. 1	652	494	158
Impinger No. 2			
Impinger No. 3			
Condenser			
Desiccant	1197	1167	30
Total	▨		188

Integrated Gas Sampling Data:

Bag Pump No. 3 Box No. 2 Bag No. 3
Bag Material: 5-layer Aluminized Tedlar Size: 44 L
Pretest leak check: .00 cc/min at 15 in. Hg.
Time start: 1240 (HRS) Time end: 1359 (HRS)
Sampling rate: 400 cc/min Operator: GH
S/N of O_2 Analyzer used to monitor train outlet: 3

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job OTF BIG STONE
Source NC ; BOILER STACK
Date 10-27-95 Test 8 Run 3
Operators GH & JB
Meter Box No. 4 ^H@ 1.71 IN WC
Calibrator coeff. 1.001
Pitot No. 22-5 Cp .84
Bar. Press. 28.96 inHg H2O 14 %
Nozzle No. 22-4 Nozzle Dia .247 IN.

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	Impg.	Gas/In	Gas/Out	Oxygen (%v/v)
	1240	104.37											
A - 3	5	109.10	1.40	3.01	9.10	9	302	260	251	39	77	77	8.1
2	10	113.94	1.35	2.90	3.94	8	302	262	254	40	79	77	8.0
1	15	118.17	1.1	2.56	8.17	8	303	260	255	41	84	78	7.5
B 3	20	122.79	1.28	2.76	2.79	8	302	258	252	44	84	79	7.9
2	25	127.46	1.30	2.80	7.46	8	304	259	259	43	86	79	7.7
1	30	131.57	1.0	2.17	1.57	8	302	262	255	42	87	79	8.1
C 3	35	136.43	1.4	3.03	6.43	8	302	260	258	41	86	81	8.0
2	40	141.12	1.31	2.83	1.12	8	305	259	259	40	86	82	7.9
1	45	145.18	1.1	2.98	5.18	8	306	258	258	39	88	82	8.0
D 3	50	150.34	1.43	3.09	0.35	10	307	260	255	39	89	82	8.0
2	55	154.95	1.25	2.70	4.95	9	307	261	256	39	89	82	8.0
1	60	159.45	1.2	2.57	9.45	8	309	258	257	40	89	82	8.0
	(1359)												
	θ = 60	Vm = 55.08		^H 2.72							Avg. = 82.7		

Interpoll Laboratories
(612)786-6020

Visible Emissions Form

Test #4

SOURCE NAME
Otter Tail Power

ADDRESS

CITY Big Stone
STATE S.D.
ZIP
PHONE
SOURCE ID NUMBER

PROCESS EQUIPMENT No. 1 Boiler
OPERATING MODE 100%

CONTROL EQUIPMENT Precip
OPERATING MODE 100%

DESCRIBE EMISSION POINT
START Round Stack STOP ✓

HEIGHT ABOVE GROUND LEVEL
START 600' STOP ✓

HEIGHT RELATIVE TO OBSERVER
START 600' STOP ✓

DISTANCE FROM OBSERVER
START 1800' STOP ✓

DIRECTION FROM OBSERVER
START N STOP ✓

DESCRIBE EMISSIONS
START Coning STOP ✓

EMISSION COLOR
START gray-white STOP ✓

PLUME TYPE: CONTINUOUS ☑ FUGITIVE ☐ INTERMITTENT ☐

WATER DROPLETS PRESENT: NO ☑ YES ☐

IF WATER DROPLET PLUME: ATTACHED ☐ DETACHED ☐

POINT IN THE PLUME AT WHICH OPACITY WAS DETERMINED
START Exit Point STOP ✓

DESCRIBE BACKGROUND
START Blue Sky STOP ✓

BACKGROUND COLOR
START Blue STOP ✓

SKY CONDITIONS
START Clear STOP ✓

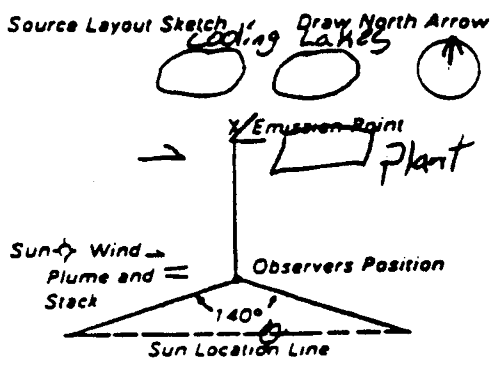
WIND SPEED
START 8-10 mph STOP ✓

WIND DIRECTION
START W STOP ✓

AMBIENT TEMP.
START 53 STOP ✓

WET BULB TEMP.

RH, percent 46%



OBSERVATION DATE 10-27-92
START TIME 1100
STOP TIME 1106

MIN \ SEC	0	15	30	45	MIN \ SEC	0	15	30	45
1	10	10	10	10	31				
2	10	10	10	10	32				
3	10	10	10	10	33				
4	10	10	10	10	34				
5	10	10	10	10	35				
6	10	10	10	10	36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
19					49				
20					50				
21					51				
22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				
30					60				

AVERAGE OPACITY FOR HIGHEST PERIOD 10

NUMBER OF READINGS ABOVE 10 % WERE 0

RANGE OF OPACITY READINGS
MINIMUM 10 MAXIMUM 10

OBSERVER'S NAME (PRINT) JEFF BERGSTROM

OBSERVER'S SIGNATURE Jeff Bergstrom
DATE 10-27-92

ORGANIZATION Interpoll Labs

CERTIFIED BY E.T.A. Minneapolis MN
DATE Sept. 1992

VERIFIED BY
DATE

COMMENTS Fuel = Lignite Coal
RdF pellets 15%

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SIGNATURE

TITLE
DATE

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Visible Emissions Form

Test #5

SOURCE NAME Otter Tail Power

ADDRESS

CITY Big Stone

STATE S.D

ZIP

PHONE

SOURCE ID NUMBER

PROCESS EQUIPMENT No. 1 Boiler

OPERATING MODE 100%

CONTROL EQUIPMENT Precip.

OPERATING MODE 100%

DESCRIBE EMISSION POINT
START Round Stack STOP ✓

HEIGHT ABOVE GROUND LEVEL
START 600' STOP ✓

HEIGHT RELATIVE TO OBSERVER
START 600' STOP ✓

DISTANCE FROM OBSERVER
START 1800' STOP ✓

DIRECTION FROM OBSERVER
START N STOP ✓

DESCRIBE EMISSIONS
START Coning STOP ✓

EMISSION COLOR
START gray-white STOP ✓

PLUME TYPE: CONTINUOUS ☑ FUGITIVE ☐ INTERMITTENT ☐

WATER DROPLETS PRESENT:
NO ☑ YES ☐

IF WATER DROPLET PLUME:
ATTACHED ☐ DETACHED ☐

POINT IN THE PLUME AT WHICH OPACITY WAS DETERMINED
START Exit Point STOP ✓

DESCRIBE BACKGROUND
START Sky STOP ✓

BACKGROUND COLOR
START Blue STOP ✓

SKY CONDITIONS
START Clear STOP ✓

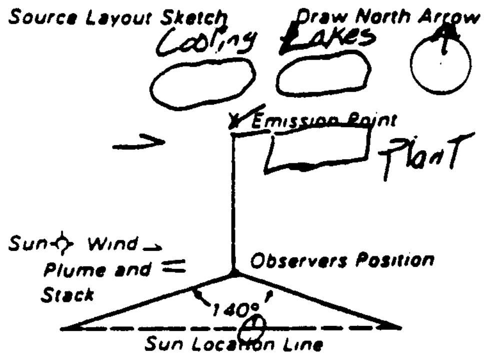
WIND SPEED
START 8-10 mph STOP ✓

WIND DIRECTION
START W STOP ✓

AMBIENT TEMP
START 53° STOP ✓

WET BULB TEMP

RH.percent 46%



COMMENTS Fuel = Lignite coal
and 15% pellets RDF

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SIGNATURE

TITLE

DATE

OBSERVATION DATE 10-27-92

START TIME 1130

STOP TIME 1136

MIN \ SEC	0	15	30	45	MIN \ SEC	0	15	30	45
1	10	10	10	10	31				
2	10	10	10	10	32				
3	10	10	10	10	33				
4	10	10	10	10	34				
5	10	10	10	10	35				
6	10	10	10	10	36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
19					49				
20					50				
21					51				
22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				
30					60				

AVERAGE OPACITY FOR HIGHEST PERIOD 10

NUMBER OF READINGS ABOVE 10 % WERE 0

RANGE OF OPACITY READINGS
MINIMUM 10 MAXIMUM 10

OBSERVER'S NAME (PRINT) JEFF BERGSTROM

OBSERVER'S SIGNATURE Jeff Bergstrom

DATE 10-27-92

ORGANIZATION Interpoll Lab

CERTIFIED BY E.T.A. Minneapolis Mn

DATE Sept. 1992

VERIFIED BY

DATE

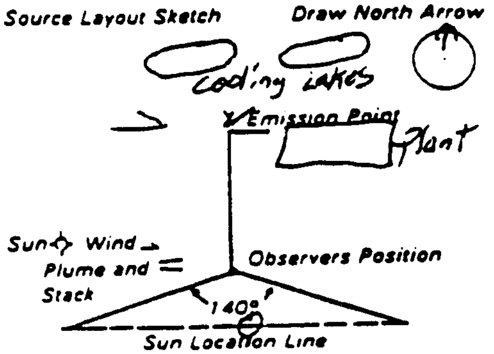
Interpoll Laboratories
(612)786-6020

Visible Emissions Form Test #6

SOURCE NAME: OTTerTail Power
ADDRESS:
CITY: Big Stone
STATE: S.D.
ZIP:
PHONE:
SOURCE ID NUMBER:
PROCESS EQUIPMENT: No. 1 Boiler
OPERATING MODE: 100%
CONTROL EQUIPMENT: Precip.
OPERATING MODE: 100%
DESCRIBE EMISSION POINT: START Round Stack STOP ✓
HEIGHT ABOVE GROUND LEVEL: START 600' STOP ✓
HEIGHT RELATIVE TO OBSERVER: START 600' STOP ✓
DISTANCE FROM OBSERVER: START 1800 STOP ✓
DIRECTION FROM OBSERVER: START N STOP ✓
DESCRIBE EMISSIONS: START Coning STOP ✓
EMISSION COLOR: START Gray-white STOP ✓
PLUME TYPE: CONTINUOUS ☑ FUGITIVE ☐ INTERMITTENT ☐
WATER DROPLETS PRESENT: NO ☑ YES ☐
IF WATER DROPLET PLUME: ATTACHED ☐ DETACHED ☐
POINT IN THE PLUME AT WHICH OPACITY WAS DETERMINED: START Exit Point STOP ✓
DESCRIBE BACKGROUND: START Sky STOP ✓
BACKGROUND COLOR: START Blue STOP ✓
SKY CONDITIONS: START Clear STOP ✓
WIND SPEED: START 8-10 mph STOP ✓
WIND DIRECTION: START W STOP ✓
AMBIENT TEMP: START 54° STOP ✓
WET BULB TEMP:
RH.percent: 42%

Source Layout Sketch

Draw North Arrow



OBSERVATION DATE: 10-27-92
START TIME: 1150
STOP TIME: 1156

MIN \ SEC	0	15	30	45	MIN \ SEC	0	15	30	45
1	10	10	10	10	31				
2	10	10	10	10	32				
3	10	10	10	10	33				
4	10	10	10	10	34				
5	10	10	10	10	35				
6	10	10	10	10	36				
7					37				
8					38				
9					39				
10					40				
11					41				
12					42				
13					43				
14					44				
15					45				
16					46				
17					47				
18					48				
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22					52				
23					53				
24					54				
25					55				
26					56				
27					57				
28					58				
29					59				
30					60				

AVERAGE OPACITY FOR HIGHEST PERIOD: 10
NUMBER OF READINGS ABOVE 10 % WERE 0
RANGE OF OPACITY READINGS: MINIMUM 10 MAXIMUM 10
OBSERVER'S NAME (PRINT): JEFF BERGSTROM
OBSERVER'S SIGNATURE: Jeff Bergstrom
DATE: 10-27-92
ORGANIZATION: Interpoll Labs
CERTIFIED BY: E.T.A. Minneapolis MN
DATE: Sept. 1992
VERIFIED BY:
DATE:

COMMENTS: Fuel = Lignite coal 85%
RDF pellets 15%

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SIGNATURE:
TITLE:
DATE:

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(612)786-6020

Field Data Sheet For HCL, HBR, HF, CL

Job O.T.P./Big Stone S.D.
Test Location No. 1 Boiler Stack

Date 10-27-92
Operator(s) JEFF BERGSTROM
Test 9 Run 1
Console No. 25
Bar. Pressure 28.46 in. Hg.

γ .9939

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1413)	115.916	///////	///////	///////	///////	///////	///////
5	116.119	1000		2.5	307	77	
10	116.316	1000		2.5	307	78	
15	116.516	1000		2.5	306	79	
20	116.712	1000		2.5	306	80	
25	116.909	1000		2.5	306	82	
30	117.103	1000		2.5	307	84	
35	117.300	1000		2.5	307	85	
40	117.498	1000		2.5	307	87	
45	117.696	1000		2.5	307	89	
50	117.895	1000		2.5	307	90	
55	118.092	1000		2.5	306	92	
60	118.289	1000		2.5	306	93	

(1513)

V_m: 2.373 CF

t_m: 84.666 °F

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Field Data Sheet For HCL, HBR, HF, CL

Job O.T.P./ Big STone S.D
Test Location No 1 Boiler STack

Date 10-27-92
Operator(s) JEFF BERGSTROM
Test 9 **Run** 2
Console No. 2 5
Bar. Pressure 28.46 **in. Hg.**

Y .9939

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1534)	118.341	///////	///////	///////	///////	///////	///////
5	118.543	1000		2.5	314	95	
10	118.743	1000		2,5	313	95	
15	118.942	1000		2,5	313	95	
20	114,141	1000		2,5	314	95	
25	119.343	1000		2,5	315	96	
30	119.539	1000		2.5	315	97	
35	119.735	1000		2.5	315	98	
40	119.941	1000		2,5	315	99	
45	120,143	1000		2.5	315	99	
50	120,342	1000		2,5	317	100	
55	120.541	1000		2.5	317	100	
60	120.742	1000		2,5	317	101	

(1634)

V_m: 2.401 CF

i_m: 97.5 °F

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Field Data Sheet For HCL, HBR, HF, CL

Job O.T.P./Big Stone S.D

Test Location No 1 Boiler Stack

Date 10-27-92

Operator(s) JEFF BERGSTROM

Test 9 Run 3

Console No. 25

Bar. Pressure 28.46 in. Hg.

γ .9939

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1657)	120.765	///////	///////	///////	///////	///////	///////
5	120.966	1000		2.5	317	99	
10	121.166	1000		2.5	317	99	
15	121.366	1000		2.5	315	99	
20	121.572	1000		2.5	315	100	
25	121.773	1000		2.5	315	100	
30	121.974	1000		2.5	315	100	
35	122.175	1000		2.5	316	100	
40	122.376	1000		2.5	316	101	
45	122.581	1000		2.5	315	101	
50	122.782	1000		2.5	315	101	
55	122.978	1000		2.5	315	102	
60	123.184	1000		2.5	315	102	

(1757) V_m: 2.419 CF t_m: 100.333 °F

C-57

Interpoll Laboratories
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Field Data Sheet For B.T.X.

Job O.T.P. / Big Stone S.D.
Test Location No 1 Boiler Stack

Date 10-27-92
Operator(s) JEFF BERGSTROM
Test 10 Run 1
Console No. 55
Bar. Pressure 28.46 in. Hg.

γ .9941

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1413)	203.255	///////	///////	///////	///////	///////	///////
5	203.436	1000		2.5	307	74	
10	203.623	1000		2.5	307	75	
15	203.809	1000		2.5	306	77	
20	204.000	1000		2.5	306	80	
25	204.185	1000		3.5	306	82	
30	204.374	1000		4.5	307	84	
35	204.563	1000		2.5	307	86	
40	204.755	1000		2.5	307	88	
45	204.944	1000		2.5	306	89	
50	205.138	1000		2.5	306	90	
55	205.321	1000		3.5	306	92	
60	205.520	1000		4	306	93	

(1513) V_m: 2.265 CF t_m: 84.166 °F

V_{STD} = 2.0783

S-290BL-R

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Field Data Sheet For B.T.X.

Job O.T.P. / Big Stone S.D.

Test Location No. 1 Boiler Stack

Date 10-27-92

Operator(s) JEFF BERGSTROM

Test 10 **Run** 2

Console No. 55

Bar. Pressure 28.46 **In. Hg.**

Y .9941

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1534)	205.537	///////	///////	///////	///////	///////	///////
5	205.726	1000		2.5	314	92	
10	205.920	1000		2.5	313	93	
15	206.114	1000		2.5	313	94	
20	206.306	1000		2.5	314	95	
25	206.498	1000		2.5	315	96	
30	206.683	1000		2.5	315	97	
35	206.875	1000		2.5	315	97	
40	207.063	1000		2.5	315	98	
45	207.263	1000		2.5	314	99	
50	207.452	1000		2.5	317	99	
55	207.644	1000		2.5	317	100	
60	207.840	1000		2.5	317	100	

(1634) V_m: 2.303 CF

t_m: 96.666 °F

VSTD = 2.0657

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Field Data Sheet For B.T.X.

Job O.T.P. / Big Stone S.D.
Test Location NO. 1 Boiler STACK

Date 10-27-92
Operator(s) JEFF BERGSTROM
Test 10 **Run** 3
Console No. 55
Bar. Pressure 28.46 **in. Hg.**

γ ,9941

☑ Pretest Leak Check
(0 cc/min at 17 IN.HG. VAC)

☑ Post Test Leak Check
(0 cc/min at 17 IN.HG. VAC)

Sampling Time (min.)	Sample Volume (CF)	Flow Rate (cc/min)		Vacuum (in. Hg.)	Stack Temp. (°F)	Meter Temp. (°F)	
(1657)	207.856	///////	///////	///////	///////	///////	///////
5	208.046	1000		2.5	317	96	
10	208.236	1000		2.5	317	96	
15	208.426	1000		2.5	315	97	
20	208.618	1000		2.5	315	98	
25	208.809	1000		2.5	315	98	
30	208.994	1000		2.5	315	98	
35	209.186	1000		2.5	316	99	
40	209.376	1000		2.5	316	99	
45	209.569	1000		2.5	315	100	
50	209.762	1000		2.5	315	101	
55	209.956	1000		2.5	315	101	
60	210.155	1000		2.5	315	102	

(1757) V_m: 2.299 CF

t_m: 98.75 °F

VSTD = 2.0544

Appendix D: Interpoll Laboratories Analytical Data

Interpoll Laboratories
(612) 786-6020

EPA Method 3 Data Reporting Sheet
Orsat Analysis

ob ANL/OTP Barstow
eam Leader DOH
ate Submitted 10-29-92
est No. 6
ate of Analysis 10-29-92

Source Boiler
Test Site Stack
Date of Test 10-27-92
No. of Runs Completed 3
Technician C. Helgeson

Test/ Run	Sample Log Number and Type	No. of An.	Buret Readings (ml)			Conc. CO₂ %v/v Dry	Conc. O₂ %v/v Dry	F₀
			Zero Pt.	After CO₂	After O₂			
6/1	7328-64	1	0.00	12.10	19.90	12.10	7.80	1.08
		2	0.00	12.10	19.90	12.10	7.80	1.08
	☑ B ☐ F	Avg				12.10	7.80	
6/2	-65	1	0.00	12.10	19.90	12.10	7.80	1.08
		2	0.00	12.10	19.90	12.10	7.80	1.08
	☑ B ☐ F	Avg				12.10	7.80	
6/3	-66	1	0.00	11.50	19.90	11.50	8.40	1.09
		2	0.00	11.50	19.90	11.50	8.40	1.09
	☑ B ☐ F	Avg				11.50	8.40	
		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
☑ F₀ Within EPA M-3 Guidelines for fuel type.

Where $F_0 = \frac{20.9 - O_2}{CO_2}$

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

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

Interpoll Laboratories
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EPA Method 3 Data Reporting Sheet
Orsat Analysis

Job ARGONNE NATIONAL / BIG STONE
Team Leader
Date Submitted 10-26-92
Test No. 1
Date of Analysis 10-26-92

Source NO 1 BOILER
Test Site STACK
Date of Test 10-26-92
No. of Runs Completed 3
Technician STROWBRIDGE

Test/ Run	Sample Log Number and Type	No. of An.	Buret Readings (ml)			Conc. CO_2 %v/v Dry	Conc. O_2 %v/v Dry	F_0
			Zero Pt.	After CO_2	After O_2			
1/1		1				12.5	7.9	1.04
		2						
	☐ B ☐ F	Avg						
1/2		1				12.6	7.8	1.04
		2						
	☐ B ☐ F	Avg						
1/3		1				12.6	7.8	1.04
		2						
	☐ B ☐ F	Avg						
2/1		1				12.6	7.8	1.04
		2						
	☐ B ☐ F	Avg						
2/2		1				12.6	7.9	
		2						
	☐ B ☐ F	Avg						
2/3		1				12.7	7.8	1.03
		2						
	☐ B ☐ F	Avg						
3/1		1				12.6	7.9	1.03
		2						
	☐ B ☐ F	Avg						
3/2		1				12.6	7.8	
		2						
	☐ B ☐ F	Avg						
3/3		1				10.7	9.4	
		2						
	☐ B ☐ F	Avg						

☐ Ambient Air QA Check
☐ Orsat Analyzer System Leak Check
☐ F_0 Within EPA M-3 Guidelines for fuel type.

Where $F_0 = \frac{20.9 - O_2}{CO_2}$

F=Flask (250 cc all glass)

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

Interpoll Laboratories
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EPA Method 3 Data Reporting Sheet
Orsat Analysis

ıb OTP Big Stone
ıam Leader GH
ıte Submitted 10-29-92
ıst No. 8
ıte of Analysis 10-29-92

Source #1 Boiler
Test Site Stack
Date of Test 10-27-92
No. of Runs Completed 3
Technician C. Helgeson

est/ 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
8/1		1	0.00	12.00	19.90	12.00	7.90	1.08
	7328-92	2	0.00	12.00	19.90	12.00	7.90	1.08
	☑ B ☐ F	Avg				12.00	7.90	
8/2		1	0.00	11.40	19.90	11.40	8.50	1.09
	-96	2	0.00	11.40	19.90	11.40	8.50	1.09
	☑ B ☐ F	Avg				11.40	8.50	
8/3		1	0.00	11.50	19.90	11.50	8.40	1.09
	-100	2	0.00	11.50	19.90	11.50	8.40	1.09
	☑ B ☐ F	Avg				11.50	8.40	
		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 $F_o = \frac{20.9 - O_2}{CO_2}$

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

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

Interpoll Laboratories
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EPA Method 3 Data Reporting Sheet
Orsat Analysis

Job ANL/OTP Big Stone
Team Leader KR
Date Submitted 10-29-92
Test No. 7
Date of Analysis 10-28-92

Source Boiler
Test Site Stack
Date of Test 10-27-92
No. of Runs Completed 3
Technician C. Helgeson

Test/ Run	Sample Log Number and Type	No. of An.	Buret Readings (ml)			Conc. CO_2 %v/v Dry	Conc. O_2 %v/v Dry	F_o
			Zero Pt.	After CO_2	After O_2			
7/1	7328-86	1	0.00	12.10	19.90	12.10	7.80	1.08
		2	0.00	12.10	19.90	12.10	7.80	1.08
	☑ B ☐ F	Avg				12.10	7.80	
7/2	-87	1	0.00	12.00	19.90	12.00	7.90	1.08
		2	0.00	12.00	19.90	12.00	7.90	1.08
	☑ B ☐ F	Avg				12.00	7.90	
7/3	-88	1	0.00	11.60	20.00	11.60	8.40	1.08
		2	0.00	11.60	20.00	11.60	8.40	1.08
	☑ B ☐ F	Avg				11.60	8.40	
		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)

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

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EPA Method 5 Data Reporting Sheet
Probe/Cyclone Wash

ob Big Stow OTP
eam Leader GH
Date Submitted 10-29-92
Test No. 3
Date of Analysis 11-11-92
Transport Leakage ☒ [✓ None] ☐ ______ ml

Source Unit #1
Test Site Stack
Date of Test 10-26-92
No. of Runs Completed 3
Technician C. Helgeson
Solvent Acetone

Test 3 Run 0 Field Blank Log Number 7323-32 Vol. of Solvent 130 ml *Solvent Residue 1.54 ug/ml	Dish No. 15 Dish Tare Wt. 49.2886 g Dish+Sample Wt. 49.2888 g Sample Wt. 0.0002 g
1 Test 3 Run 1 Vol. of Solvent 200 ml Log Number -35 Comments	Dish No. 22 Dish Tare Wt. 51.3615 g Dish+Sample Wt. 51.3682 g Sample Wt. 0.0067 g
2 Test 3 Run 2 Vol. of Solvent 180 ml Log Number -38 Comments	Dish No. 61A Dish Tare Wt. 51.4734 g Dish+Sample Wt. 51.4837 g Sample Wt. 0.0103 g
3 Test 3 Run 3 Vol. of Solvent 130 ml Log Number -41 Comments	Dish No. 111 Dish Tare Wt. 47.7798 g Dish+Sample Wt. 47.7966 g Sample Wt. 0.0168 g
4 Test ____ Run ____ Vol. of Solvent ____ ml Log Number Comments	Dish No. Dish Tare Wt. ____ g Dish+Sample Wt. ____ g Sample Wt. ____ g
5 Test ____ Run ____ Vol. of Solvent ____ ml Log Number Comments	Dish No. Dish Tare Wt. ____ g Dish+Sample Wt. ____ g Sample Wt. ____ g

*Solvent Residue ___ ug/ml = [(Sample Wt. 0.0002 g)(10^6)]/Vol. of Sol. 130 ml
EPA-M5 Acetone Residue Blank Spec. ≤ 7.8 ug/ml

Results:

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0064	0.0101 D-6	0.0166		

LSC-01Y

Interpoll Laboratories
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EPA Method 5 Data Reporting Sheet
Impinger Catch/Minnesota Protocol

Job Big Stone OTP
Team Leader GH
Date Submitted 10-29-92
Test No. 3
Date of Analysis 11-11-92

Source Unit #1
Test Site Stack
Date of Test 10-26-92
No. of Runs Completed 3
Technician C. Helgeson

0
Test 3 Run 0
Field Blank
Log Number 7328-34
Comments
Dish No. 3
Dish Tare Wt. 52.1196 g
Dish+Sample Wt. 52.1200 g
Sample Wt. 0.0004 g

1
Test 3 Run 1
Log Number -37
Comments
Dish No. 8
Dish Tare Wt. 48.6975 g
Dish+Sample Wt. 48.7031 g
Sample Wt. 0.0056 g

2
Test 3 Run 2
Log Number -40
Comments
Dish No. 11
Dish Tare Wt. 47.7579 g
Dish+Sample Wt. 47.7625 g
Sample Wt. 0.0046 g

3
Test 3 Run 3
Log Number -43
Comments
Dish No. 30
Dish Tare Wt. 47.2597 g
Dish+Sample Wt. 47.2659 g
Sample Wt. 0.0062 g

4
Test Run
Log Number
Comments
Dish No.
Dish Tare Wt. g
Dish+Sample Wt. g
Sample Wt. g

5
Test Run
Log Number
Comments
Dish No.
Dish Tare Wt. g
Dish+Sample Wt. g
Sample Wt. g

Blank Solvent Wt. 0.0004 g

Results:

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0052	0.0042 D-5	0.0058		

LSC-0

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EPA Method 5 Data Reporting Sheet
Impinger Catch/Minnesota Protocol

Job Big Stone CTP
Team Leader GH
Date Submitted 10-29-92
Test No. 8
Date of Analysis 11-11-92

Source #1 Boiler
Test Site Stack
Date of Test 10-27-92
No. of Runs Completed 3
Technician C. Helgeson

0 Test 8 Run 0
Field Blank
Log Number 7328-138
Comments
Dish No. 62
Dish Tare Wt. 50.8874 g
Dish+Sample Wt 50.8878 g
Sample Wt. 0.0004 g

1 Test 8 Run 1
Log Number -91
Comments
Dish No. 66
Dish Tare Wt. 48.3089 g
Dish+Sample Wt. 48.3125 g
Sample Wt. 0.0036 g

2 Test 8 Run 2
Log Number -95
Comments
Dish No. 69
Dish Tare Wt. 47.9885 g
Dish+Sample Wt. 47.9915 g
Sample Wt. 0.0030 g

3 Test 8 Run 3
Log Number -99
Comments
Dish No. 93
Dish Tare Wt. 53.9833 g
Dish+Sample Wt. 53.9852 g
Sample Wt. 0.0019 g

4 Test Run
Log Number
Comments
Dish No.
Dish Tare Wt. g
Dish+Sample Wt. g
Sample Wt. g

5 Test Run
Log Number
Comments
Dish No.
Dish Tare Wt. g
Dish+Sample Wt. g
Sample Wt. g

Blank Solvent Wt. 0.0004 g

Results:

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0032	0.0026 D-8	0.0015		

LSC-03

Interpoll Laboratories
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EPA Method 5 Data Reporting Sheet
Filter Gravimetrics

Job OTP Big Stone	Source #1 Boiler
Team Leader GH	Test Site Stack
Date Submitted 10-29-92	Date of Test 10-26-92
Test No. 3	No. of Runs Completed 3
Date of Analysis 10-30-92	Technician C. Helgeson

0	**Test** 3 **Run** 0 Field Blank Log Number 7328-33 Comments	Filter No. 4733 Filter Type 4" GF Filter Tare Wt. .9032 g Filter+Sample Wt. .9032 g **Sample Wt.** 0.0000 g
1	**Test** 3 **Run** 1 Log Number -36 Comments	Filter No. 4725 Filter Type 4" GF Filter Tare Wt. .8999 g Filter+Sample Wt. .9109 g **Sample Wt.** 0.0110 g
2	**Test** 3 **Run** 2 Log Number -39 Comments	Filter No. 4272 Filter Type 4" GF Filter Tare Wt. .9022 g Filter+Sample Wt. .9178 g **Sample Wt.** 0.0156 g
3	**Test** 3 **Run** 3 Log Number -42 Comments	Filter No. 4723 Filter Type 4" GF Filter Tare Wt. .8970 g Filter+Sample Wt. .9127 g **Sample Wt.** 0.0157 g
4	**Test** **Run** Log Number Comments	Filter No. Filter Type Filter Tare Wt. g Filter+Sample Wt. g **Sample Wt.** g
5	**Test** **Run** Log Number Comments	Filter No. Filter Type Filter Tare Wt. g Filter+Sample Wt. g **Sample Wt.** g

Results:

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0110	0.0156	0.0157		

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0226	0.0299	0.0381		

Interpoll Laboratories
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EPA Method 5 Data Reporting Sheet
Filter Gravimetrics

Job OTP Big Stone
Team Leader GH
Date Submitted 10-29-92
Test No. 8
Date of Analysis 10-30-92

Source #1 Boiler
Test Site Stack
Date of Test 10-27-92
No. of Runs Completed 3
Technician C. Helgeson

0
Test ____ Run 0
Field Blank
Log Number ____
Comments ____

Filter No. ____
Filter Type ____
Filter Tare Wt. ____ g
Filter+Sample Wt. ____ g
Sample Wt. ____ g

1
Test 8 Run 1
Log Number 7328-90
Comments ____

Filter No. 4732
Filter Type 4"GF
Filter Tare Wt. .8964 g
Filter+Sample Wt. .9126 g
Sample Wt. 0.0162 g

2
Test 8 Run 2
Log Number -94
Comments ____

Filter No. 4734
Filter Type 4"GF
Filter Tare Wt. .9080 g
Filter+Sample Wt. .9268 g
Sample Wt. 0.0188 g

3
Test 8 Run 3
Log Number -98
Comments ____

Filter No. 4735
Filter Type 4"GF
Filter Tare Wt. .9009 g
Filter+Sample Wt. .9195 g
Sample Wt. 0.0186 g

4
Test ____ Run ____
Log Number ____
Comments ____

Filter No. ____
Filter Type ____
Filter Tare Wt. ____ g
Filter+Sample Wt. ____ g
Sample Wt. ____ g

5
Test ____ Run ____
Log Number ____
Comments ____

Filter No. ____
Filter Type ____
Filter Tare Wt. ____ g
Filter+Sample Wt. ____ g
Sample Wt. ____ g

Results:

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0162	0.0188	0.0186		

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0338	0.0365	0.0459		

Interpoll Laboratories
(612) 786-6020

EPA Method 5 Data Reporting Sheet
Probe/Cyclone Wash

Job OTP Big Stone
Team Leader GH
Date Submitted 10-24-92
Test No. 8
Date of Analysis 11-11-92
Transport Leakage ☒ None ☐ ______ ml

Source #1 Boiler
Test Site Stack
Date of Test 10-27-92
No. of Runs Completed 3
Technician C. Helgeson
Solvent Acetone

0
Test ____ Run 0
Field Blank
Log Number ______
Vol. of Solvent ____ ml
*Solvent Residue 1.54 ug/ml
Dish No. ______
Dish Tare Wt. ______ g
Dish+Sample Wt. ______ g
Sample Wt. ______ g

1
Test 8 Run 1
Vol. of Solvent 190 ml
Log Number 7328-89
Comments ______
Dish No. 601
Dish Tare Wt. 47.6657 g
Dish+Sample Wt. 47.6804 g
Sample Wt. 0.0147 g

2
Test 8 Run 2
Vol. of Solvent 210 ml
Log Number -93
Comments ______
Dish No. 623
Dish Tare Wt. 77.7622 g
Dish+Sample Wt. 77.7776 g
Sample Wt. 0.0154 g

3
Test 8 Run 3
Vol. of Solvent 180 ml
Log Number -97
Comments ______
Dish No. 624
Dish Tare Wt. 48.5261 g
Dish+Sample Wt. 48.5522 g
Sample Wt. 0.0261 g

4
Test ____ Run ____
Vol. of Solvent ____ ml
Log Number ______
Comments ______
Dish No. ______
Dish Tare Wt. ______ g
Dish+Sample Wt. ______ g
Sample Wt. ______ g

5
Test ____ Run ____
Vol. of Solvent ____ ml
Log Number ______
Comments ______
Dish No. ______
Dish Tare Wt. ______ g
Dish+Sample Wt. ______ g
Sample Wt. ______ g

*Solvent Residue ___ ug/ml=[(Sample Wt. _____ g)(10^{6})]/Vol. of Sol. _____ ml
EPA-M5 Acetone Residue Blank Spec. ≤7.8 ug/ml

Results:

Field Blk.	Run 1	Run 2	Run 3	Run 4	Run 5
	0.0144	0.0151 D-9	0.0258		

LSC-0

Interpoll Laboratories
(612) 786-6020

EPA Method 18/NIOSH Sampling
Data Reporting Sheet

Job/Project Argonne National Laboratory
Source No. 1 Boiler
Test Site Stack
Date of Sampling 10-26-92
Analyst KDS
Date of Analysis 11/12/92
Method of Analysis NIOSH-1501

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size	Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
7328-54	Test 5 Run 2	☑ C\C Coal ☐ P\C Coal ☐ S. Gel ☐ XAD-2 ☐ ☐ ☐ ☐ ☐	☐ 100/50 ☐ 300/150 ☐ 400/200 ☑ 800/200 ☐ 1060/240 ☐ 1800/200 ☐ ☐ ☐	Benzene	<1.5	<.45
				Toluene	↓	↓
				Xylenes	↓	↓

Comments: Tube #1

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size	Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
7328-55	Test 5 Run 3	☑ C\C Coal ☐ P\C Coal ☐ S. Gel ☐ XAD-2 ☐ ☐ ☐ ☐ ☐	☐ 100/50 ☐ 300/150 ☐ 400/200 ☑ 800/200 ☐ 1060/240 ☐ 1800/200 ☐ ☐ ☐	Benzene	<1.5	<.45
				Toluene	↓	↓
				Xylenes	↓	↓

Comments: Tube #1

Detection Limits:

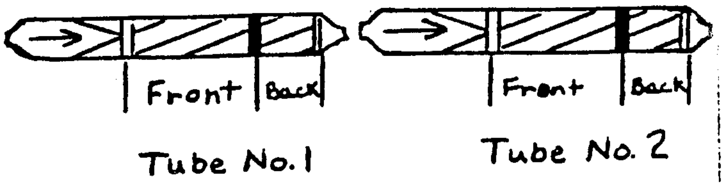
Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
Benzene	1.5	.45
Toluene	↓	↓
Xylene	↓	↓

C\C Coal = Coconut Charcoal
P\C Coal = Petroleum Charcoal
S. Gel = Silica Gel

Tube Schematics:

Single Tube ☐

Two Tubes "Piggy-back" ☐



LOA-13

Interpoll Laboratories
(612) 786-6020

EPA Method 18/NIOSH Sampling
Data Reporting Sheet

Job/Project Argonne National Laboratory
Source No. 1 Boiler
Test Site Stack
Date of Sampling 10-26-92
Analyst KDS
Date of Analysis 11/12/92
Method of Analysis NIOSH-1501

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size
7328-52	Test 5 Run 0	☑ C\C Coal ☐ P\C Coal ☐ S. Gel ☐ XAD-2 ☐ ☐ ☐ ☐	☐ 100/50 ☐ 300/150 ☐ 400/200 ☑ 800/200 ☐ 1060/240 ☐ 1800/200 ☐ ☐

Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug
Benzene	<1.5	<.45
Toluene	↓	↓
Xylenes		

Comments:

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size
7328-53	Test 5 Run 1	☑ C\C Coal ☐ P\C Coal ☐ S. Gel ☐ XAD-2 ☐ ☐ ☐ ☐	☐ 100/50 ☐ 300/150 ☐ 400/200 ☑ 800/200 ☐ 1060/240 ☐ 1800/200 ☐ ☐

Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug
Benzene	<1.5	<.45
Toluene	↓	↓
Xylenes		

Comments: Tube #1

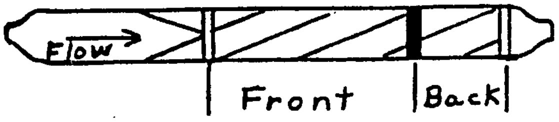
Detection Limits:

Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
Benzene	1.5	~~.30~~ .45
Toluene	↓	↓
Xylene		

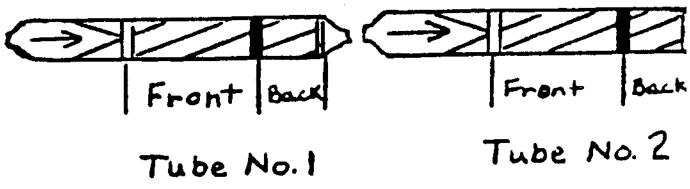
C\C Coal = Coconut Charcoal
P\C Coal = Petroleum Charcoal
S. Gel = Silica Gel

Tube Schematics:

Single Tube ☐



Two Tubes "Piggy-back" ☐



Interpoll Laboratories
(612) 786-6020

EPA Method 18/NIOSH Sampling Data Reporting Sheet

Job/Project Argonne National Laboratory
Source No. 1 Boiler
Test Site Stack
Date of Sampling 10-27-92
Analyst KDS
Date of Analysis 11/12/92
Method of Analysis NIOSH-1501

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size	Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
7328-109	Test 10	☑ C\C Coal	☐ 100/50	Benzene	< 1.5	<.45
	Run 3	☐ P\C Coal	☐ 300/150	Tolmene	↓	↓
		☐ S. Gel	☐ 400/200	Xylenes	↓	↓
		☐ XAD-2	☑ 800/200			
		☐	☐ 1060/240			
		☐	☐ 1800/200			
		☐	☐			
		☐	☐			
		☐	☐			

Comments: Tube #1

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size	Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
	Test	☐ C\C Coal	☐ 100/50			
	Run	☐ P\C Coal	☐ 300/150			
		☐ S. Gel	☐ 400/200			
		☐ XAD-2	☐ 800/200			
		☐	☐ 1060/240			
		☐	☐ 1800/200			
		☐	☐			
		☐	☐			
		☐	☐			

Comments:

Detection Limits:

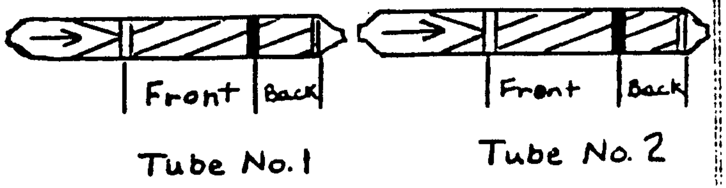
Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
Benzene	1.5	.45
Toluene	↓	↓
Xylene	↓	↓

C\C Coal = Coconut Charcoal
P\C Coal = Petroleum Charcoal
S. Gel = Silica Gel

Tube Schematics:

Single Tube ☐

Two Tubes "Piggy-back" ☐



LOA-13

Interpoll Laboratories
(612) 786-6020

EPA Method 18/NIOSH Sampling Data Reporting Sheet

Job/Project Argonne National Laboratory
Source No. 1 Boiler
Test Site Stack
Date of Sampling 10-27-92
Analyst KDS
Date of Analysis 11/12/92
Method of Analysis NIOSH-1501

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size	Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug
7328-107	Test 10 Run 1	☑ C\C Coal ☐ P\C Coal ☐ S. Gel ☐ XAD-2 ☐ ☐ ☐ ☐ ☐	☐ 100/50 ☐ 300/150 ☐ 400/200 ☑ 800/200 ☐ 1060/240 ☐ 1800/200 ☐ ☐ ☐	Benzene	< 1.5	<.45
				Toluene	↓	↓
				Xylenes	↓	↓

Comments: Tube #1

Sample Log Number	Test/Run	Sampling Media	Sample Tube Size	Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug
7328-108	Test 10 Run 2	☑ C\C Coal ☐ P\C Coal ☐ S. Gel ☐ XAD-2 ☐ ☐ ☐ ☐ ☐	☐ 100/50 ☐ 300/150 ☐ 400/200 ☑ 800/200 ☐ 1060/240 ☐ 1800/200 ☐ ☐ ☐	Benzene	<1.5	<.45
				Toluene	↓	↓
				Xylenes	↓	↓

Comments: Tube#1

Detection Limits:

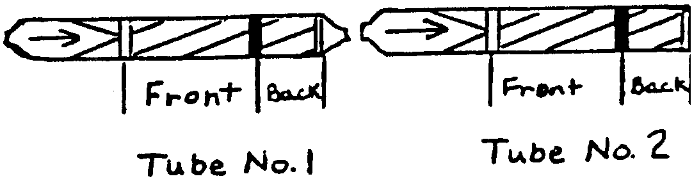
Analytes:	Mass Front Section (Total ug)	Mass Back Section (Total ug)
Benzene	1.5	.45
Toluene	↓	↓
Xyleme	↓	↓

C\C Coal = Coconut Charcoal
P\C Coal = Petroleum Charcoal
S. Gel = Silica Gel

Tube Schematics:

Single Tube ☐

Two Tubes "Piggy-back" ☐



Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 4000I WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: SMS

Date of Analysis: 11-11-92

Job: Argonne National Laboratory Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$
X	AS4A	2 ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$
	AS5	ml/min	100 mM NaOH
		ml/min	

Flow Rate	Suppressor Acid
10 ml/min	25 mM Sulfuric Acid
ml/min	

☒ Isocratic

☐ Gradient(List program below)

Gradient Program	Time (Min.)									
Eluent	0.0									
% A										
% B										

Impinger Catch

Results of Chloride Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T9R1	7328-101	100	50	0.619	3095
T9R2	-102	100	50	0.662	3310
T9R3	-103	100	50	0.630	3150

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-11R

Collected in 0.1N H_2SO_4

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 4000I WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: SMS

Date of Analysis: 11-23-92

Job: Argonne National Laboratory / Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

	Column	Flow Rate	Eluent	Flow Rate	Suppressor Acid
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$	10 ml/min	25 mM Sulfuric Acid
X	AS4A	2 ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$	ml/min	
	AS5	ml/min	100 mM NaOH	☐	Isocratic
X	AS4A	2 ml/min	5mM & 70mM Sodium Borate	☒	Gradient(List program below)

Gradient Program	Time (Min.)									
Eluent	0.0	5.0	5.1	7.0	15					
% A 5mM	100	100	0	0	100					
% B 70mM	0	0	100	100	0					

Impinger catch

Results of Chloride Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T4 R0	7328-44	100	1	<0.01	<1.00
T4 R1	-45	100	10	0.397	397
T4 R2	-46	100	10	0.353	353
T4 R3	-47	100	2	0.308	61.6

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-11R

collected in 0.1N H_2SO_4

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 40001 WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: BMS

Date of Analysis: 11-24-92

Job: Argonne National Laboratory
Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$
X	AS4A	2 ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$
	AS5	ml/min	100 mM NaOH
X	AS4A	2 ml/min	5&70mM Sodium Borate

Flow Rate	Suppressor Acid
10 ml/min	25 mM Sulfuric Acid
ml/min	
~~X~~	Isocratic
X	Gradient(List program below)

Gradient Program	Time (Min.)									
Eluent	0.0	5.0	5.1	7.0	7.1	15				
% A 5mM	100	100	0	0	100	100				
% B 70mM	0	0	100	100	0	0				

Impinger Catch

Results of Chloride Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T9R1	7328-104	100	1	0.109	10.9
T9R2	-105	100	1	0.178	17.8
T9R3	-106	100	1	0.068	6.80

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-11R

Collected in 0.1 N NaOH

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 40001 WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: SMS

Date of Analysis: 11-23-92

Job: Argonne National Laboratory / Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent	Flow Rate	Suppressor Acid
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$	10 ml/min	25 mM Sulfuric Acid
~~2~~	AS4A	~~2~~ ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$	ml/min	
	AS5	ml/min	100 mM NaOH	~~X~~	Isocratic
2	AS4A	2 ml/min	5 & 70mM Sodium Borate	X	Gradient(List program below)

Gradient Program	Time (Min.)									
Eluent	0.0	5.0	5.1	7.0	15					
% A 5mM	100	100	0	0	100					
% B 70mM	0	0	100	100	0					

Impinger Catch

Results of Chloride Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T4R0	7328-48	100	1	<0.010	<1.00
T4R1	-49	100	10	0.402	402
T4R2	-50	100	10	0.121	121
T4R3	-51	100	10	0.117	117

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-11R

Collected in 0.1N NaOH

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 40001 WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: SMS

Date of Analysis: 11-23-92

Job: Argonne National Laboratory
Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent	Flow Rate		Suppressor Acid
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$	10 ml/min		25 mM Sulfuric Acid
~~X~~	AS4A	~~3~~ ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$	ml/min		
	AS5	ml/min	100 mM NaOH	~~X~~	Isocratic	
X	AS4A	2 ml/min	5&70mM Sodium Borate	X	Gradient(List program below)	

Gradient Program	Time (Min.)									
Eluent	0.0	5.0	5.1	7.0	15					
% A 5mM	100	100	0	0	100					
% B 70mM	0	0	100	100	0					

Impinger Catch

Results of Fluoride Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T4R1	7328-101	100	2	0.392	78.4
T4R2	-102	100	2	0.552	110
T4R3	-103	100	2	0.604	121

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-12R

Collected in 0 1N H_2SO_4

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 40001 WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: BMS

Date of Analysis: 11-23-92

Job: Argonne National/Laboratory Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent	Flow Rate	Suppressor Acid
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$	10 ml/min	25 mM Sulfuric Acid
~~X~~	AS4A	~~2~~ ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$	ml/min	
	AS5	ml/min	100 mM NaOH	~~X~~	Isocratic
X	AS4A	2 ml/min	5:70 mM Sodium Borate	X	Gradient (List program below)

Gradient Program	Time (Min.)									
Eluent	0.0	5.0	5.1	7.0	15					
% A 5mM	100	100	0	0	100					
% B 70mM	0	0	100	100	0					

Impinger Catch

Results of Fluoride Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T4 R0	7328-44	100	1	0.054	5.4
T4 R1	-45	100	2	0.350	70
T4 R2	-46	100	5	0.240	120
T4 R3	-47	100	2	0.264	52.8

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-12R

collected in 0.1N H_2SO_4

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 40001 WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: sms
Date of Analysis: 11-11-92

Job: Argonne National/Laboratory
Big Stone
Source: Boiler
Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent	Flow Rate	Suppressor Acid
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$	10 ml/min	25 mM Sulfuric Acid
X	AS4A	2 ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$	ml/min	
	AS5	ml/min	100 mM NaOH	X	Isocratic
		ml/min			Gradient(List program below)

Gradient Program	Time (Min.)									
Eluent	0.0									
% A										
% B										

Impinger Catch

Results of Bromide Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
T4R0	7328-44	100	1	<0.025	<2.50
T4R1	-45	100	1	0.043	4.30
T4R2	-46	100	1	sms 11-11-92 ~~0.038~~ 0.042	4.20
T4R3	-47	100	1	0.041	4.10

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-13R

collected in 0.1N H_2SO_4

Interpoll Laboratories, Inc.
(612)786-6020

Ion Chromatography Laboratory

DIONEX MODEL 4000i WITH ANION MICRO MEMBRANE SUPRESSION

Analyst: SMS

Date of Analysis: 11-11-92

Job: Argonne National Laboratory Big Stone

Source: Boiler

Site: Stack

Chromatography Conditions

Column		Flow Rate	Eluent
	AS3	ml/min	2.4 mM Na_2CO_3 & 3.0 mM $NaHCO_3$
X	AS4A	2 ml/min	1.8 mM Na_2CO_3 & 1.7 mM $NaHCO_3$
	AS5	ml/min	100 mM NaOH
		ml/min	

Flow Rate	Suppressor Acid
10 ml/min	25 mM Sulfuric Acid
ml/min	
X	Isocratic
	Gradient(List program below)

Gradient Program	Time (Min.)									
Eluent	0.0									
% A										
% B										

Impinger catch

Results of Bromide Determination

Sample Name	Interpoll Log Number	Total Sample Vol. in mls	Dilution	Solution Conc. in ug/ml	Total ug
TgR1	7328-101	100	~~0~~ 1	0.217	21.7
TgR2	-102	100	SMS 11-11-92 1	0.194	19.4
TgR3	-103	100	1	0.200	20.0

Total ug = (Sample Vol.) x (Dilution) x (Solution Conc.)

LSC-13R

Collected in 0.1N H_2SO_4

INTERPOLL LABORATORIES, INC.
(612)786-6020

Results of Trace Metals Analysis

Facility: Argonne National Laboratory/Big Stone
Test: 6
Source: Boiler Stack
Test Type: EPA Method 29

Element	Method	Total Mass of Elements in Sample (ug)				
		Field Blank 1	Field Blank 2	Run 1	Run 2	Run 3
(Log No.)		(7328-59)	(7328-60)	(7328-61)	(7328-62)	(7328-63)
(Wt. (g))	Method 5	NR	NR	NR	NR	NR
Ag	EPA 6010	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00
Al	EPA 6010	NR	NR	NR	NR	NR
As	EPA 7060	< 0.10	< 0.10	< 2.00	< 2.00	< 2.00
B	EPA 6010	NR	NR	NR	NR	NR
Ba	EPA 6010	3.80	3.10	16.80	41.00	39.50
Be	EPA 7091	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
C	ASTM D3178	NR	NR	NR	NR	NR
Ca	EPA 6010	NR	NR	NR	NR	NR
Cd	EPA 6010	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00
Co	EPA 6010	NR	NR	NR	NR	NR
Cr	EPA 6010	1.90	1.20	2.30	3.60	3.60
Cu	EPA 6010	2.40	2.40	9.60	10.60	16.80
Fe	EPA 6010	NR	NR	NR	NR	NR
Hg	Fluor.	0.12	0.06	24.40	34.80	42.30
K	EPA 6010	NR	NR	NR	NR	NR
Li	SM 317A	NR	NR	NR	NR	NR
Mg	EPA 6010	NR	NR	NR	NR	NR
Mn	EPA 6010	NR	NR	NR	NR	NR
Mo	EPA 6010	NR	NR	NR	NR	NR
Na	EPA 6010	NR	NR	NR	NR	NR
Ni	EPA 6010	2.00	2.00	1.30	3.90	3.50
P	EPA 6010	NR	NR	NR	NR	NR
Pb	EPA 7421	0.87	0.67	2.67	4.12	11.40
S	ASTM D1552	NR	NR	NR	NR	NR
Sb	EPA 7041	< 0.20	< 0.20	< 1.00	< 1.00	< 1.00
Se	EPA 7740	< 0.10	< 0.10	< 20.00	< 10.00	12.00
Si	EPA 6010	NR	NR	NR	NR	NR
Sn	EPA 6010	NR	NR	NR	NR	NR
Sr	SM 326A	NR	NR	NR	NR	NR
Ti	EPA 6010	NR	NR	NR	NR	NR
Tl	EPA 7841	< 60.00	< 60.00	< 60.00	< 60.00	< 60.00
V	EPA 6010	NR	NR	NR	NR	NR
Zn	EPA 6010	25.60	17.40	69.00	94.70	109.00

INTERPOLL LABORATORIES, INC.
(612)786-6020

Results of Trace Metals Analysis

Facility: Argonne National Laboratory/Big Stone
Test: 1
Source: Boiler Stack
Test Type: EPA Method 29

		Total Mass of Elements in Sample (ug)				
Element	Method	Field Blank 1	Field Blank 2	Run 1	Run 2	Run 3
(Log No.)		(7328-01)	(7328-02)	(7328-04)	(7328-05)	(7328-06)
(Wt. (g))	Method 5	NR	NR	NR	NR	NR
Ag	EPA 6010	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00
Al	EPA 6010	NR	NR	NR	NR	NR
As	EPA 7060	< 0.10	< 0.10	< 2.00	< 2.00	< 2.00
B	EPA 6010	NR	NR	NR	NR	NR
Ba	EPA 6010	2.80	2.70	17.40	18.70	17.40
Be	EPA 7091	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
C	ASTM D3178	NR	NR	NR	NR	NR
Ca	EPA 6010	NR	NR	NR	NR	NR
Cd	EPA 6010	< 1.00	< 1.00	1.10	< 1.00	< 1.00
Co	EPA 6010	NR	NR	NR	NR	NR
Cr	EPA 6010	< 1.00	< 1.00	3.40	2.00	2.00
Cu	EPA 6010	3.00	2.50	8.20	7.10	7.10
Fe	EPA 6010	NR	NR	NR	NR	NR
Hg	Fluor.	< 0.06	< 0.06	56.80	53.00	55.50
K	EPA 6010	NR	NR	NR	NR	NR
Li	SM 317A	NR	NR	NR	NR	NR
Mg	EPA 6010	NR	NR	NR	NR	NR
Mn	EPA 6010	NR	NR	NR	NR	NR
Mo	EPA 6010	NR	NR	NR	NR	NR
Na	EPA 6010	NR	NR	NR	NR	NR
Ni	EPA 6010	< 1.00	< 1.00	4.10	2.40	2.60
P	EPA 6010	NR	NR	NR	NR	NR
Pb	EPA 7421	0.62	0.59	2.51	2.52	2.30
S	ASTM D1552	NR	NR	NR	NR	NR
Sb	EPA 7041	< 0.20	< 0.20	< 1.00	< 1.00	< 1.00
Se	EPA 7740	< 0.10	< 0.10	25.20	< 20.00	< 10.00
Si	EPA 6010	NR	NR	NR	NR	NR
Sn	EPA 6010	NR	NR	NR	NR	NR
Sr	SM 326A	NR	NR	NR	NR	NR
Ti	EPA 6010	NR	NR	NR	NR	NR
Tl	EPA 7841	< 60.00	< 60.00	< 60.00	< 60.00	< 60.00
V	EPA 6010	NR	NR	NR	NR	NR
Zn	EPA 6010	12.60	6.95	105.00	81.80	62.10

Job ANL/Big Stone Source No. 1 Boiler Date of Test 10/27/92
(Test #6)

			MICROGRAMS											
Element	GF/AA	ICP	Field Blanks			Analytical Detection Limit †	Minimum Detectable Mass	Best Estimate of of Field Blank	Total Mass in Sample (µg) (Not corrected for blank)			Total Mass in Sample (µg) (Corrected for blank)		
			H*	1	2				Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Silver		X	<1.0	<1.0	<1.0			0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Arsenic	X		<0.05	<.10	<.10			0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Barium		X	2.3	3.8	3.1	(4.23)		2.3	16.8	41.0	39.5	14.5	38.7	37.2
Beryllium	X		<.05	<.03	<.03			0	<.03	<.03	<.03	<.03	<.03	<.03
Cadmium		X	<1.0	<1.0	<1.0			0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Chromium		X	<1.0	1.9	1.2			0	2.3	3.6	3.6	2.3	3.6	3.6
Copper		X	1.0	2.4	2.4	(4.36)		1.0	9.6	10.6	16.8	8.6	9.6	15.8
Nickel		X	<1.0	2.0	2.0			0	1.3	3.9	3.5	1.3	3.9	3.5
Lead	X		.26	.37	.67	(1.09)		.26	2.67	4.12	11.4	2.41	3.86	11.14
Antimony	X		0.1	<.20	<.20			0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Selenium	X		<.05	<.10	<.10			0	<20.0	<10.0	12.0	<20.0	<10.0	12.0
Thallium		X	<.05	<60	<60			0	<60	<60	<60	<60	<60	<60
Zinc		X	3.1	25.6	17.4	(14.01)		3.1	69.0	94.7	109.0	65.9	91.6	105.9
Mercury Front	CV/AA											▨	▨	▨
Mercury KMnO4												▨	▨	▨
Mercury Total				0.12	0.06			0	24.4	34.8	42.3	24.4	34.8	42.3

* - Filter Background

H = historical average mass of the trace element in a Pallaflex quartz filter

PL & DD
12/29/92

Job ANL/Big Stone Source No. 1 Boiler Date of Test 10/26/92 (Test #1)

			MICROGRAMS						Total Mass in Sample (µg) (Not corrected for blank)			Total Mass in Sample (µg) (Corrected for blank)		
Element	GF/AA	ICP	Field Blanks *H	1	2	Analytical Detection Limit †	Minimum Detectable Mass	Best Estimate of Field Blank	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Silver		X	<1.0	<1.0	<1.0			0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Arsenic	X		<.05	<0.1	<0.1			0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Barium		X	2.3	2.8	2.7	(4.23)		2.3	17.4	18.7	17.4	15.1	16.4	15.1
Beryllium	X		<.05	<0.05	<.05			0	<0.03	<0.03	<0.03	<.03	<.03	<.03
Cadmium		X	<1.0	<1.0	<1.0			0	1.10	<1.0	<1.0	1.10	<1.0	<1.0
Chromium		X	<1.0	<1.0	<1.0			0	3.40	2.00	2.00	3.4	2.0	2.0
Copper		X	1.0	3.0	2.5	(4.36)		1.0	8.2	7.1	7.1	7.2	6.1	6.1
Nickel		X	<1.0	<1.0	<1.0			0	4.1	2.4	2.6	4.1	2.4	2.6
Lead	X		0.26	0.62	0.59	(1.07)		0.26	2.51	2.52	2.30	2.25	2.26	2.04
Antimony	X		0.1	<.20	<.20			0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Sellenium	X		<.05	<.10	<.10			0	25.2	<20.0	<10.0	25.2	<20.0	<10.0
Thallium		X	<.05	<60	<60			0	<60	<60	<60	<60	<60	<60
Zinc		X	3.1	12.6	6.95	(14.01)		3.1	105	81.8	62.1	101.9	78.7	59.0
Mercury Front	CV/AA											▨	▨	▨
KMnO4												▨	▨	▨
Total	F/vor.			<.06	<.06			0	56.8	53.0	55.4	56.3	53.0	55.4

* - Filter background

H - historical average mass of the trace element in a Pallaflex quartz filter

AL & DD
12/29/92

Argonne National Laboratory,
Big Stone Plant
Laboratory Log No. 7328

Results of Polynuclear Aromatic Hydrocarbons Analysis[1]

Test:	7
Source:	Boiler Stack
Sample Type:	MM-5 Train Catch

	Total Mass of PAHs (ug)			
Analyte	Field Blank	Run 1	Run 2	Run 3
(Log No.)	(7328-70)	(7328-74)	(7328-79)	(7328-84)
Naphthalene	0.76	10.4	0.56	0.33
Acenaphthylene	< 0.088	< 0.088	< 0.088	< 0.088
Acenaphthene	< 0.10	< 0.10	< 0.10	< 0.10
Fluorene	< 0.10	< 0.10	< 0.10	< 0.10
Phenanthrene	0.20	0.31	0.19	0.22
Anthracene	< 0.080	< 0.080	< 0.080	< 0.080
Fluoranthene	< 0.12	< 0.12	< 0.12	< 0.12
Pyrene	< 0.15	< 0.15	< 0.15	< 0.15
Benzo(a)anthracene	< 0.18	< 0.18	< 0.18	< 0.18
Chyrsene	< 0.092	< 0.092	< 0.092	< 0.092
Benzo(b)fluoranthene	< 0.10	< 0.10	< 0.10	< 0.10
Benzo(k)fluoranthene	< 0.17	< 0.17	< 0.17	< 0.17
Benzo(a)pyrene	< 0.12	0.12	< 0.12	< 0.12
Dibenzo(a,h)anthracene	< 0.088	< 0.088	< 0.088	< 0.088
Benzo(g,h,i)perylene	< 0.12	< 0.12	< 0.12	< 0.12
Indeno(1,2,3-cd)pyrene	< 0.092	< 0.092	< 0.092	< 0.092
Fluoranthene-d10	18.8	18.0	22.4	20.0

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[1] EPA Method SW-846, 8270.

INTERPOLL LABORATORIES, INC.
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Argonne National Laboratory,
Big Stone Plant
Laboratory Log No. 7328

Results of Polynuclear Aromatic Hydrocarbons Analysis[1]

Test: 2
Source: Boiler Stack
Sample Type: MM-5 Train Catch

	Total Mass of PAHs (ug)			
Analyte	Field Blank	Run 1	Run 2	Run 3
(Log No.)	(7328-13)	(7328-17)	(7328-22)	(7328-27)
Naphthalene	6.4	5.2	1.3	5.2
Acenaphthylene	< 0.088	< 0.088	< 0.088	< 0.088
Acenaphthene	< 0.10	< 0.10	< 0.10	< 0.10
Fluorene	< 0.10	< 0.10	< 0.10	< 0.10
Phenanthrene	0.11	0.36	0.52	0.36
Anthracene	< 0.080	< 0.080	< 0.080	< 0.080
Fluoranthene	< 0.12	< 0.12	< 0.12	< 0.12
Pyrene	< 0.15	< 0.15	< 0.15	< 0.15
Benzo(a)anthracene	< 0.18	< 0.18	< 0.18	< 0.18
Chyrsene	< 0.092	< 0.092	< 0.092	< 0.092
Benzo(b)fluoranthene	< 0.10	< 0.10	< 0.10	< 0.10
Benzo(k)fluoranthene	< 0.17	< 0.17	< 0.17	< 0.17
Benzo(a)pyrene	< 0.12	< 0.12	< 0.12	< 0.12
Dibenzo(a,h)anthracene	< 0.088	< 0.088	< 0.088	< 0.088
Benzo(g,h,i)perylene	< 0.12	< 0.12	< 0.12	< 0.12
Indeno(1,2,3-cd)pyrene	< 0.092	< 0.092	< 0.092	< 0.092
Fluoranthene-d10	21.6	19.2	20.8	17.6

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[1] EPA Method SW-846, 8270.

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Argonne National Laboratory,
Big Stone Plant
Laboratory Log No. 7328

Results of PCB Analysis[1]

Test: 2

Source: Boiler Stack

Sample Type: MM-5 Train Catch

	Total Mass of PCBs[2] (ug)			
Analyte	Field Blank	Run 1	Run 2	Run 3
(Log No.)	(7328-13)	(7328-17)	(7328-22)	(7328-27)
Aroclor 1016	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1221	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1232	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1242	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1248	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1254	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1260	< 0.01	< 0.01	< 0.01	< 0.01
Total PCB	< 0.01	< 0.01	< 0.01	< 0.01

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[1] EPA Method SW-846, 8080.

[2] Sample values represent only one-fourth of total sample.

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Argonne National Laboratory,
Big Stone Plant
Laboratory Log No. 7328

Results of Polynuclear Aromatic Hydrocarbons Analysis[1]

Sample Identification: Triangle Labs Extraction Blank

Analyte	Total Mass of PAHs (ug)
(Log No.)	(7328-137)
Naphthalene	1.0
Acenaphthylene	< 0.088
Acenaphthene	< 0.10
Fluorene	< 0.10
Phenanthrene	< 0.092
Anthracene	< 0.080
Fluoranthene	< 0.12
Pyrene	< 0.15
Benzo(a)anthracene	< 0.18
Chyrsene	< 0.092
Benzo(b)fluoranthene	< 0.10
Benzo(k)fluoranthene	< 0.17
Benzo(a)pyrene	< 0.12
Dibenzo(a,h)anthracene	< 0.088
Benzo(g,h,i)perylene	< 0.12
Indeno(1,2,3-cd)pyrene	< 0.092
Fluoranthene-d10	0.72

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[1] EPA Method SW-846, 8270.

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Argonne National Laboratory,
Big Stone Plant
Laboratory Log No. 7328

Results of PCB Analysis[1]

Sample Identification: Triangle Labs Extraction Blank

Analyte	Total Mass of PCBs[2] (ug)
(Log No.)	(7328-137)
Aroclor 1016	< 0.01
Aroclor 1221	< 0.01
Aroclor 1232	< 0.01
Aroclor 1242	< 0.01
Aroclor 1248	< 0.01
Aroclor 1254	< 0.01
Aroclor 1260	< 0.01
Total PCB	< 0.01

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[1] EPA Method SW-846, 8080.

[2] Sample values represent only one-fourth of total sample.

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Argonne National Laboratory,
Big Stone Plant
Laboratory Log No. 7328

Results of PCB Analysis[1]

Test: 7

Source: Boiler Stack

Sample Type: MM-5 Train Catch

	Total Mass of PCBs[2] (ug)			
Analyte	Field Blank	Run 1	Run 2	Run 3
(Log No.)	(7328-70)	(7328-74)	(7328-79)	(7328-84)
Aroclor 1016	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1221	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1232	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1242	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1248	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1254	< 0.01	< 0.01	< 0.01	< 0.01
Aroclor 1260	< 0.01	< 0.01	< 0.01	< 0.01
Total PCB	< 0.01	< 0.01	< 0.01	< 0.01

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[1] EPA Method SW-846, 8080.

[2] Sample values represent only one-fourth of total sample.

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Chain of Custody
Sample Deposition Sheet

Job ANL/OTP - Big Stone
Team Leader R. Rosenthal
Date Submitted
Test No. 2

Source Boiler
Test Site Stack
Date of Test 10-26-92
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)
	☐ Fuel Sample ☐ Aggregate	☐Attached fuel Form #S-0163RRR	
—	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

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Chain of Custody
Sample Deposition Sheet

Job ANL/OTF Big Stone
Team Leader Dick
Date Submitted 10/28/92
Test No. 1

Source Boiler
Test Site Stack
Date of Test 10/26/92
No. of Runs Completed ______

No. of Samples	Type of Sample	Analysis Required	Comments
5 5	Probe Wash: ☒ ~~Acetone~~ $MeCl_2$ ☒ ~~D.I. Water~~ .1N HNO_3	☐ As per EPA M-5 ☒ Other METALS	
5	Filter: ☐ 4" G.F. ☐ S.S. Thimble ☐ 2.5" G.F. ☐ 47 mm G.F. ☒ 3.52" PALLFLEX	☐ As per EPA M-5 ☐ As per EPA M-17 ☒ Other METALS	
5 5	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 Hg	
	Integrated Gas sample	☐ As per EPA M-3 ☐ As per EPA M-10 ☐ Other ______	DONE IN FIELD
	Oxides of Nitrogen (NO_x)	☐ As per EPA M-7A ☐ Other ______	Date ______ Time (HRS) ______
	☐ Fuel Sample ☐ Aggregate	☐ Attached fuel Form #S-0163RRR	
	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

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Chain of Custody
Sample Deposition Sheet

Job OTTer Tail Power/Big Stone S.D
Team Leader D VanHoever
Date Submitted 10-28-92
Test No. 4
Source No. 1 Boiler
Test Site Stack
Date of Test 10-26-92
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	
3 pairs (sample) 2 Blanks	Impinger Catch: ☐D.I. Water ☐3% H_2O_2 ☐4M5 Hg Only ☐4M5 Metals ☑1.0 N NaOH ☑Other .1N H_2SO_4	☐MN Protocol ☐WI Protocol ☐EPA M-6 or 8 ☐Acid Gases ☐Formaldehyde ☐Metals ☐Other	HCL, HBR HF From 1N H_2SO_4 CL From NaOH
	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)
	☐ Fuel Sample ☐ Aggregate	☐Attached fuel Form #S-0163RRR	
	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 Lignite
3) Is sample combustible? ☑ No ☐ Yes
4) Does sample need special handling? ☑ No ☐ Yes If yes, explain

S-278RRRR

Interpoll Laboratories
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Chain of Custody
Sample Deposition Sheet

Job OTP BIG STONE

Team Leader GN

Date Submitted 10-2

Test No. 3

Source NO 1 BOILER STACK

Test Site STACK

Date of Test 10-26-92

No. of Runs Completed 3

No. of Samples	Type of Sample	Analysis Required	Comments
4	Probe Wash: ☒ Acetone ☐ D.I. Water	☒ As per EPA M-5 ☐ Other	
4	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	
4	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	
0	Integrated Gas sample	☐ As per EPA M-3 ☐ As per EPA M-10 ☐ Other	
0	Oxides of Nitrogen (NO_x)	☐ As per EPA M-7A ☐ Other	Date Time (HRS)
	☐ Fuel Sample ☐ Aggregate	☐ Attached fuel Form #S-0163RRR	
0	Particle Size	☐ X-Ray Sedigraph ☐ Bahco Method ☐ Other	
0	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 LIGNITE
3) Is sample combustible? ☒ No ☐ Yes
4) Does sample need special handling? ☒ No ☐ Yes If yes, explain

S-278RRRR

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Chain of Custody
Sample Deposition Sheet

Job ANL/OTP Big Stone
Team Leader Dan
Date Submitted 10/27/92
Test No. 6

Source Boiler
Test Site Stack
Date of Test 10/27/92
No. of Runs Completed 3

No. of Samples	Type of Sample	Analysis Required	Comments
5 5	Probe Wash: ☒ ~~Acetone~~ MeCl2 ☒ ~~D.I. Water~~, 1N HNO3	☐ As per EPA M-5 ☒ Other METALS	
5	Filter: ☐ 4" G.F. ☐ S.S. Thimble ☐ 2.5" G.F. ☐ 47 mm G.F. ☒ 3.52" Pallflex	☐ As per EPA M-5 ☐ As per EPA M-17 ☐ Other	
5 5	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 Hg	
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)
	☐ Fuel Sample ☐ Aggregate	☐ Attached fuel Form #S-0163RRR	
	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

Chain of Custody
Sample Deposition Sheet

Job Otter Tail Power/Big Stone S.D. Source No. 1 Boiler
Team Leader D. VanHoever Test Site Stack
Date Submitted 10-28-92 Date of Test 10-26-92
Test No. 5 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	
	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)
	☐ Fuel Sample ☐ Aggregate	☐Attached fuel Form #S-0163RRR	
	Particle Size	☐X-Ray Sedigraph ☐Bahco Method ☐Other	
3 Samples 1 Blank	~~Audit Samples~~ ☐Sulfur Dioxide ☐Oxides of Nit. ☑Other BTX	☐As per EPA M-6 ☐As per EPA M-7A ☑Other M-18	

Source Information

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

Interpoll Laboratories
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Chain of Custody
Sample Deposition Sheet

Job OTP BIG STONE
Team Leader G HELE
Date Submitted 11-
Test No. 8

Source NO 1 BOILER
Test Site STACK
Date of Test 10-27-92
No. of Runs Completed 3

No. of Samples	Type of Sample	Analysis Required	Comments
3	Probe Wash: ☒ Acetone ☐ D.I. Water	☒ As per EPA M-5 ☐ Other ______	
3	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 ______	
3	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 ______	
0	Oxides of Nitrogen (NO_x)	☐ As per EPA M-7A ☐ Other ______	Date ______ Time(HRS) ______
0	☐ Fuel Sample ☐ Aggregate	☐ Attached fuel Form #S-0163RRR	
0	Particle Size	☐ X-Ray Sedigraph ☐ Bahco Method ☐ Other ______	
0	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 RDF
3) Is sample combustible? ☒ No ☐ Yes
4) Does sample need special handling? ☒ No ☐ Yes If yes, explain ______

S-278RRRR

Interpoll Laboratories
(612) 786-6020

Chain of Custody
Sample Deposition Sheet

Job ANL/OTP-BIGSTONE
Team Leader R. ROSENTHAL
Date Submitted 10- -92
Test No. 7

Source BOILER
Test Site STACK
Date of Test 10-27-92
No. of Runs Completed

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)
—	☐ Fuel Sample ☐ Aggregate	☐Attached fuel Form #S-0163RRR	
—	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

Chain of Custody
Sample Deposition Sheet

Job O.T.P. / Big STONE S.D
Team Leader D. Van Hoever
Date Submitted 10-28-92
Test No. 10

Source NO. 1 Boiler
Test Site STACK
Date of Test 10-27-92
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 ____	
	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) ____
	☐ Fuel Sample ☐ Aggregate	☐ Attached fuel Form #S-0163RRR	
	Particle Size	☐ X-Ray Sedigraph ☐ Bahco Method ☐ Other ____	
3	[illegible] Samples ☐ Sulfur Dioxide ☐ Oxides of Nit. ☑ Other BTX	☐ As per EPA M-6 ☐ As per EPA M-7A ☑ Other M-18	

Source Information

1) Type of Source: ☑ Boiler ☐ Asphalt Plant ☐ Incinerator ☐ Dryer
☐ Other ____
2) Fuel: ☑ Coal ☐ Wood ☐ Gas ☐ Oil ☑ RDF ☐ Other pellets 15%
3) Is sample combustible? ☑ No ☐ Yes
4) Does sample need special handling? ☑ No ☐ Yes If yes, explain ____

S-278RRRR

Interpoll Laboratories
(612) 786-6020

Chain of Custody
Sample Deposition Sheet

Job O.T.P./Big Stone S.D.
Team Leader D. VanHoever
Date Submitted 10-28-92
Test No. 9

Source No. 1 Boiler
Test Site Stack
Date of Test 10-27-92
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 ____	
3 pairs	Impinger Catch: ☐ D.I. Water ☐ 3% H_2O_2 ☐ 4M5 Hg Only ☐ 4M5 Metals ☑ 1.0 N NaOH ☑ Other 1N H_2SO_4	☐ MN Protocol ☐ WI Protocol ☐ EPA M-6 or 8 ☑ Acid Gases ☐ Formaldehyde ☐ Metals ☐ Other ____	HCL, HBR HF, [illegible] From 1N H_2SO_4 CL From NaOH
	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) ____
	☐ Fuel Sample ☐ Aggregate	☐ Attached fuel Form #S-0163RRR	
	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 Pellets 15%
3) Is sample combustible? ☑ No ☐ Yes
4) Does sample need special handling? ☑ No ☐ Yes If yes, explain ____

Appendix E: Continuous Emission Monitoring Data

The CEM data is reported in this appendix. The data logger printed reports for the SO_2, NO_x, CO, CO_2, and O_2 are presented first followed by copies of the strip charts. The SO_2 readings for October 27 exhibit a discrepency between the data logger data and the strip charts. The data collected on the strip chart is considered valid and was the data reported. All other data logger parameters were cross-checked and found to be valid.

Following the SO_2, NO_x, CO, CO_2, and O_2 printouts and strip charts is the total hydrocarbon data. The charts were read and the results tabularized. copies of the strip charts follow these results.

Interpoll Laboratories, Inc.
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Printout of ESC Model 80 DAS
for CEM Trailer No. 1
- 1992 -

File Name: agnbs1
Job Number: 2-7328
Client: Argonne National Labs - Otter Tail Power - Big Stone
Location: Big Stone, South Dakota

No. 1 Boiler Stack -- Run 1

Julian Date	Time (Hrs)	Conc. (dry basis unless noted) SO2 (ppmv)	NOx (ppmv)	CO (ppmv)	O2 (%v/v)
300	09:45:00	869	508	6.2	8.16
300	10:00:00	951	442	6.2	7.92
300	10:15:00	971	520	5.2	8.11
300	10:30:00	967	519	4.5	8.15
300	10:45:00	968	518	4.7	8.18
300	11:00:00	968	516	3.7	8.19
300	11:15:00	971	518	4.0	8.17
300	11:30:00	960	513	3.5	8.30
300	11:45:00	957	519	3.6	8.37
300	12:00:00	974	520	2.8	8.17
300	12:15:00	974	521	2.8	8.17
300	12:30:00	965	523	4.3	8.28
300	12:45:00	963	524	4.3	8.30
300	13:00:00	965	526	3.2	8.26
300	13:15:00	966	526	4.0	8.26
300	13:30:00	968	528	4.5	8.24
300	13:45:00	969	530	3.7	8.23
300	14:00:00	971	530	4.2	8.22
300	14:15:00	971	530	4.0	8.20
300	14:30:00	973	532	4.5	8.19
300	14:45:00	971	531	4.0	8.19
300	15:00:00	975	528	4.1	8.17
300	15:15:00	979	528	3.8	8.10
300	15:30:00	977	525	3.1	8.11
300	15:45:00	980	527	3.4	8.07
300	16:00:00	976	530	2.8	8.09
300	16:15:00	975	528	2.6	8.11
300	16:30:00	970	524	3.8	8.16
300	16:45:00	975	523	2.5	8.08
300	17:00:00	975	525	3.5	8.05
300	17:15:00	975	522	2.8	8.04
300	17:30:00	974	523	2.5	8.04
300	17:45:00	973	521	2.0	8.05
300	18:00:00	974	522	1.4	8.05

Printout of ESC Model 80 DAS
for CEM Trailer No. 1
- 1992 -

File Name: agnbs1
Job Number: 2-7328
Client: Argonne National Labs - Otter Tail Power - Big Stone
Location: Big Stone, South Dakota

No. 1 Boiler Stack -- Run 1

Julian Date	Time (Hrs)	Conc. (dry basis unless noted) SO2 (ppmv)	NOx (ppmv)	CO (ppmv)	O2 (%v/v)
300	18:15:00	977	522	1.7	8.01
300	18:30:00	976	519	2.7	8.03
300	18:45:00	968	517	1.8	8.04
300	19:00:00	968	520	1.6	8.06
	Run Average	968	521	3.5	8.15

Printout of ESC Model 80 DAS
for CEM Trailer No. 1
- 1992 -

File Name: agnbs2
Job Number: 2-7328
Client: Argonne National Labs - Otter Tail Power - Big Stone
Location: Big Stone, South Dakota

No. 1 Boiler Stack -- Run 2

Julian Date	Time (Hrs)	Conc. (dry basis unless noted) SO2 (ppmv)	NOx (ppmv)	CO (ppmv)	O2 (%v/v)
301	15:05:00	981	534	2.2	7.63
301	15:10:00	925	497	2.5	8.19
301	15:15:00	966	489	1.0	7.68
301	15:20:00	1003	521	2.1	7.26
301	15:25:00	963	526	1.1	7.82
301	15:30:00	976	532	1.4	7.68
301	15:35:00	973	531	2.0	7.70
301	15:40:00	971	529	2.2	7.73
301	15:45:00	960	524	2.8	7.85
301	15:50:00	979	532	2.1	7.65
301	15:55:00	957	523	2.9	7.89
301	16:00:00	951	518	1.9	7.97
301	16:05:00	951	522	1.1	7.97
301	16:10:00	923	512	0.1	8.26
301	16:15:00	944	518	-0.4	8.04
301	16:20:00	965	528	1.0	7.81
301	16:25:00	936	518	0.0	8.13
301	16:30:00	950	516	-0.9	7.98
301	16:35:00	963	524	-0.3	7.83
301	16:40:00	965	529	-0.2	7.81
301	16:45:00	965	521	-0.4	7.84
301	16:50:00	990	527	-0.5	7.56
301	16:55:00	1000	541	-0.9	7.47
301	17:00:00	979	534	-0.4	7.70
301	17:05:00	983	529	-0.5	7.69
301	17:10:00	997	541	-1.0	7.51
301	17:15:00	958	521	-0.2	7.96
301	17:20:00	983	532	-0.9	7.67
301	17:25:00	976	532	-0.7	7.76
301	17:30:00	970	523	-0.9	7.82
301	17:35:00	984	530	-0.7	7.65
301	17:40:00	987	532	-0.7	7.65
301	17:45:00	983	528	-0.1	7.69
301	17:50:00	981	524	-0.9	7.72

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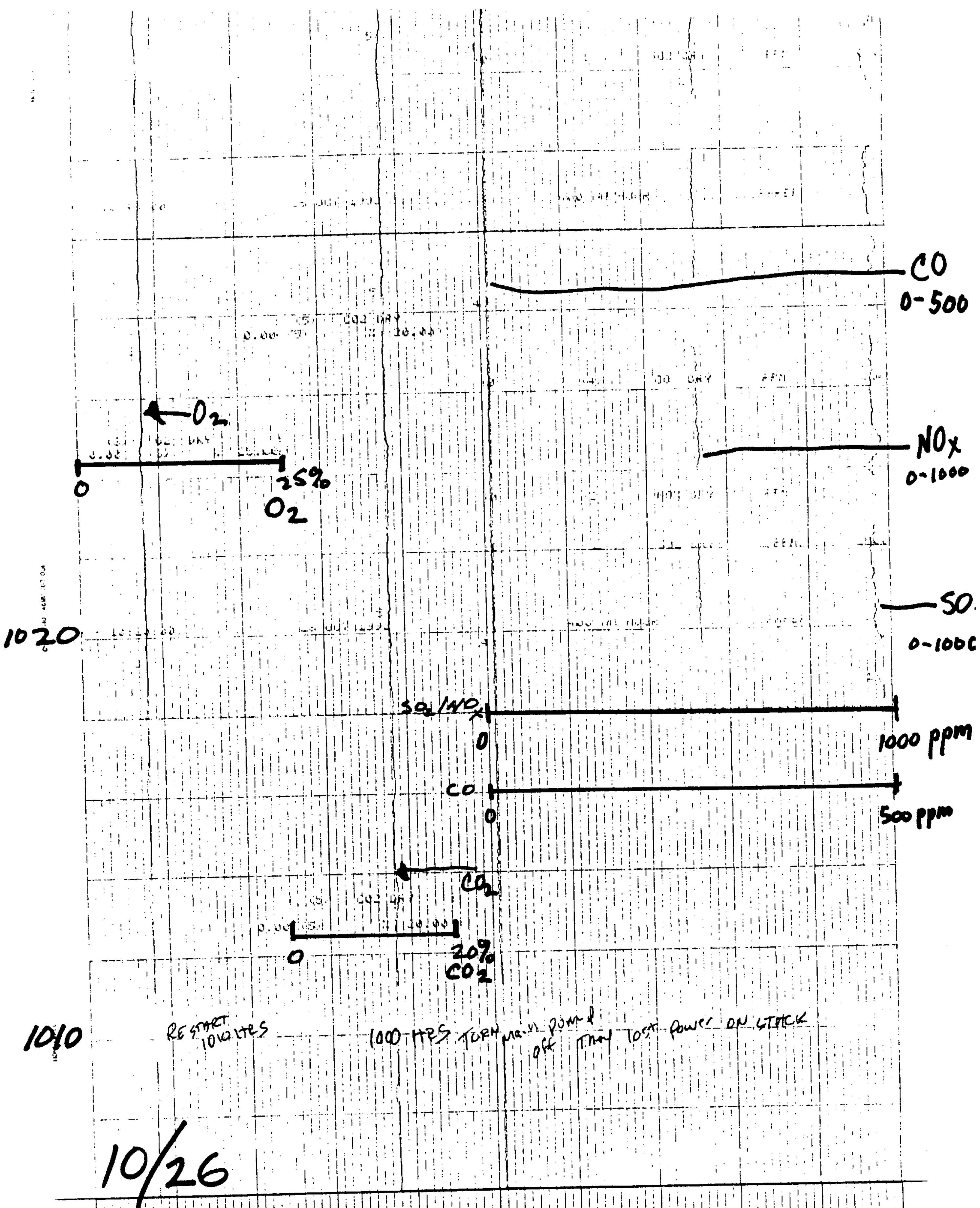
(612) 786-6020

Printout of ESC Model 80 DAS
for CEM Trailer No. 1
- 1992 -

File Name: agnbs2
Job Number: 2-7328
Client: Argonne National Labs - Otter Tail Power - Big Stone
Location: Big Stone, South Dakota

No. 1 Boiler Stack -- Run 2

Julian Date	Time (Hrs)	Conc. (dry basis unless noted) SO2 (ppmv)	NOx (ppmv)	CO (ppmv)	O2 (%v/v)
301	17:55:00	969	520	-1.2	7.82
	Run Average	969	524	0.4	7.78



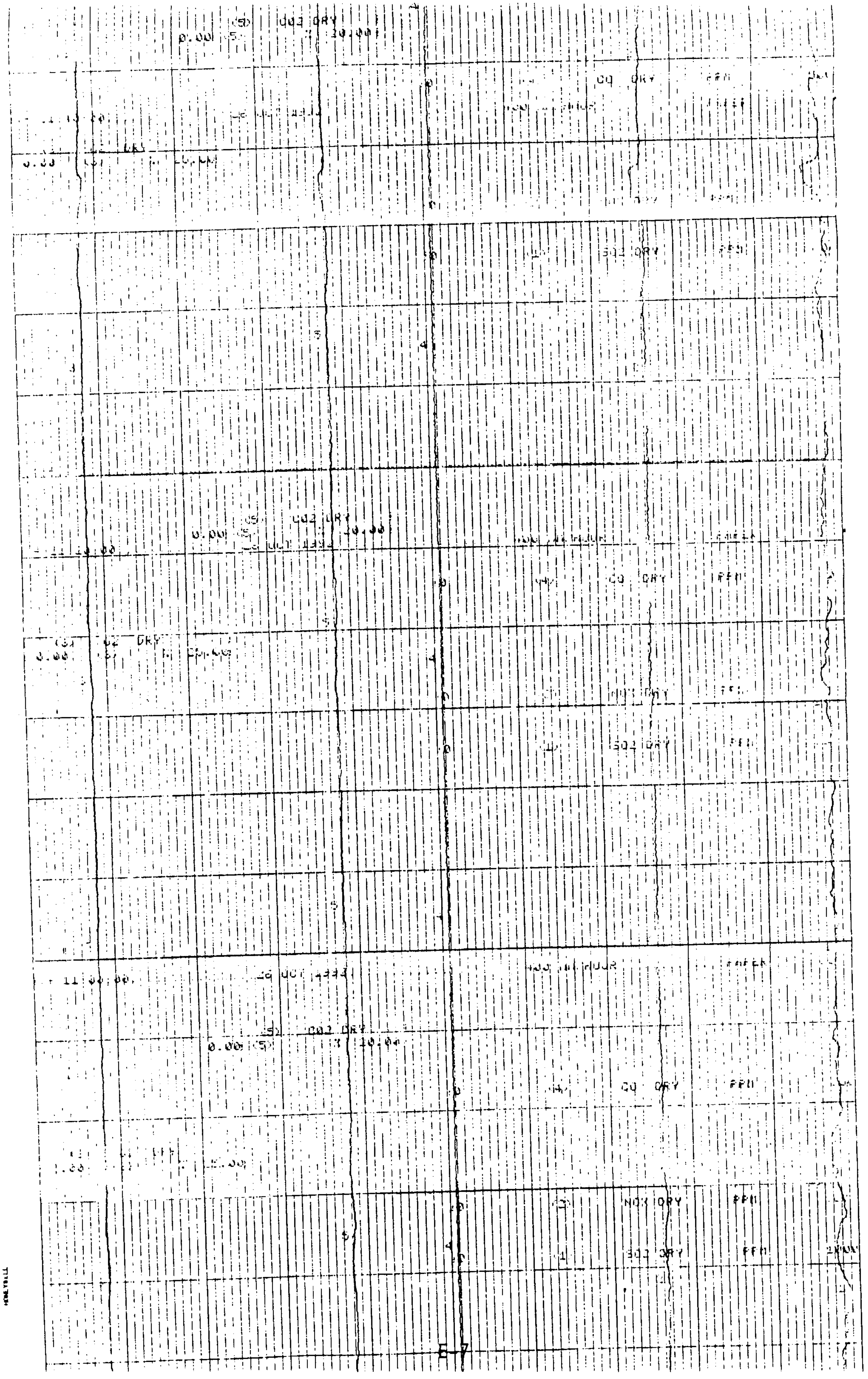
RESTART 1010 HRS

1000 HRS TURN main pump off They lost power on stack

ARGONNE NATIONAL LABORATORY

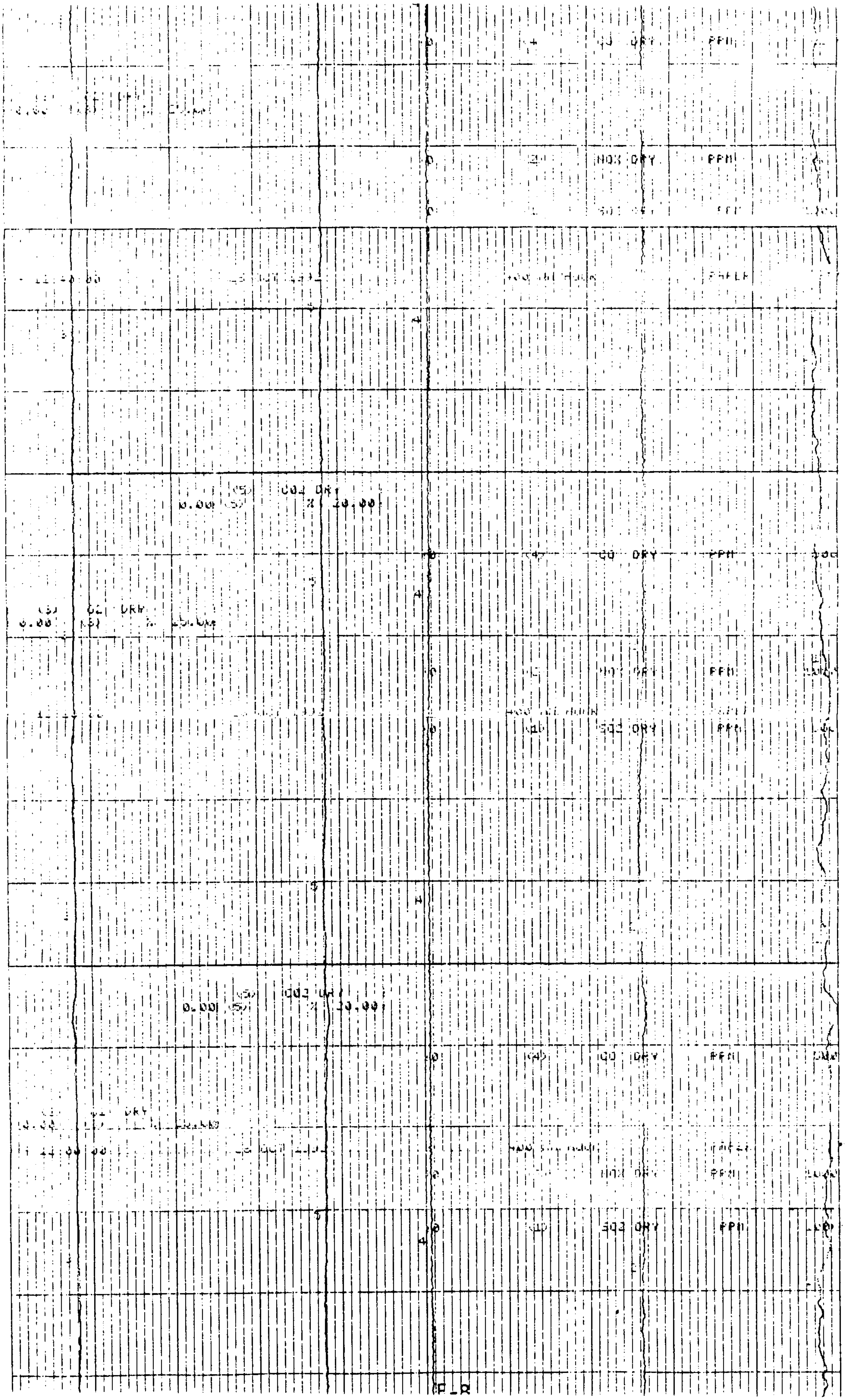
BIG STONE 10/25 CALIBRATION & SETUP

10/26 BASELINE STUDY - COAL ONLY


(5) CO2 DRY
0.00 (5) 3 20.00
CO DRY
PPM
11:00:00
29 OCT 1999
NOX DRY
SO2 DRY
HONEYWELL

10/26

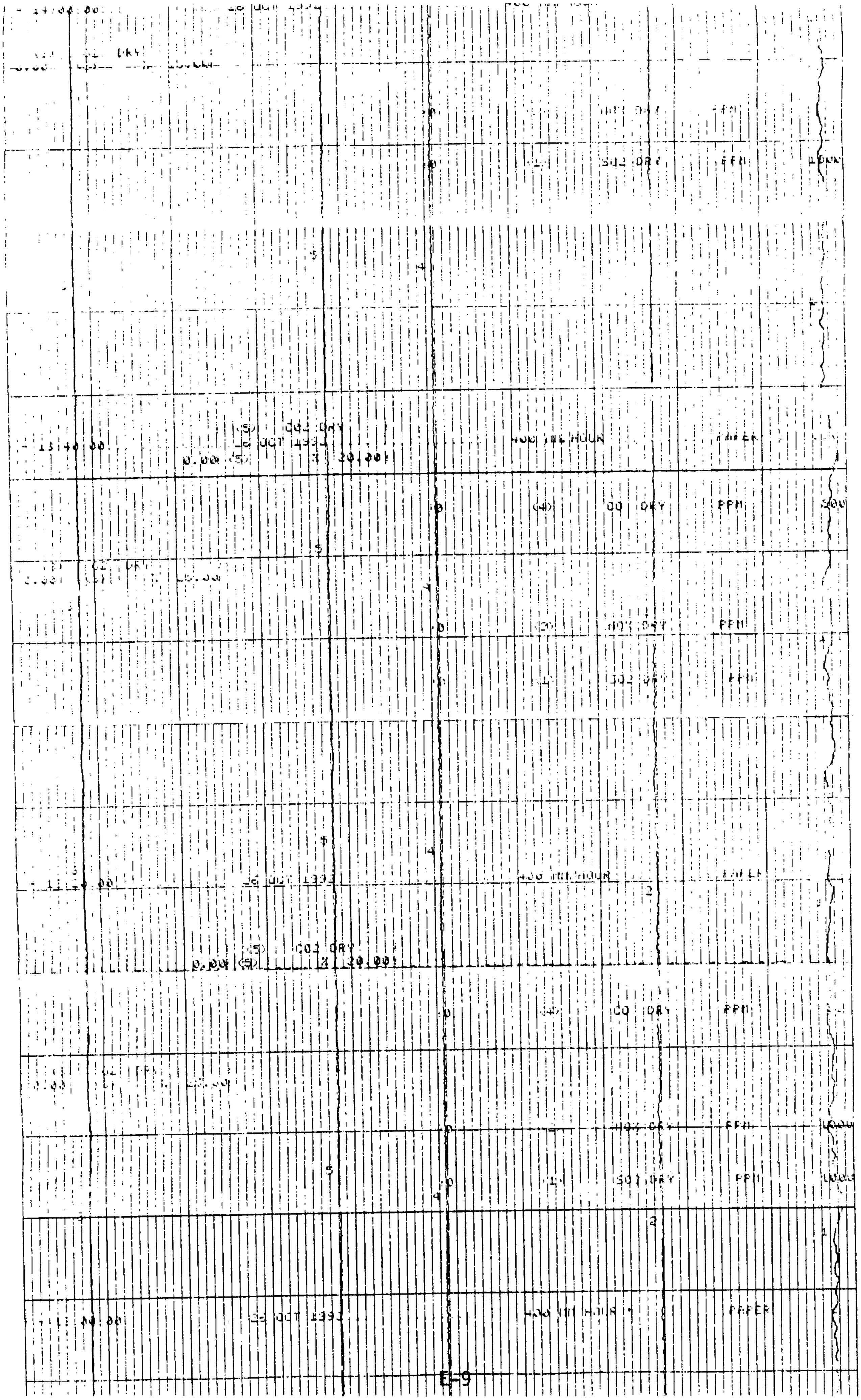
E-7



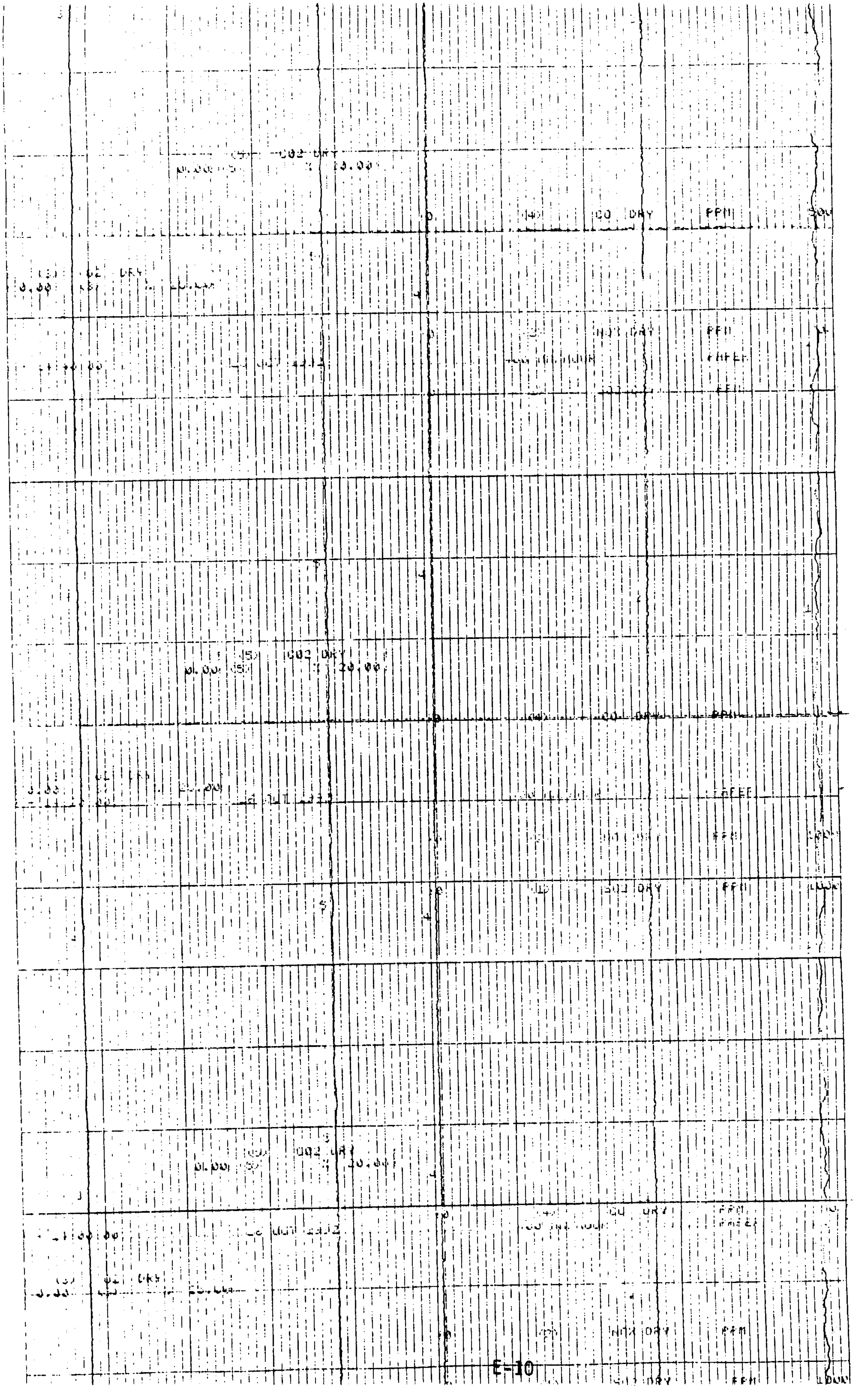
0 (4) CO DRY PPM
0 (2) NOX DRY PPM
0 (1) SO2 DRY PPM
11:40:00
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
0 NOX DRY PPM
11:42:00
0 (1) SO2 DRY PPM
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM
(3) O2 DRY
11:44:00
0 NOX DRY PPM
0 (1) SO2 DRY PPM

10/26

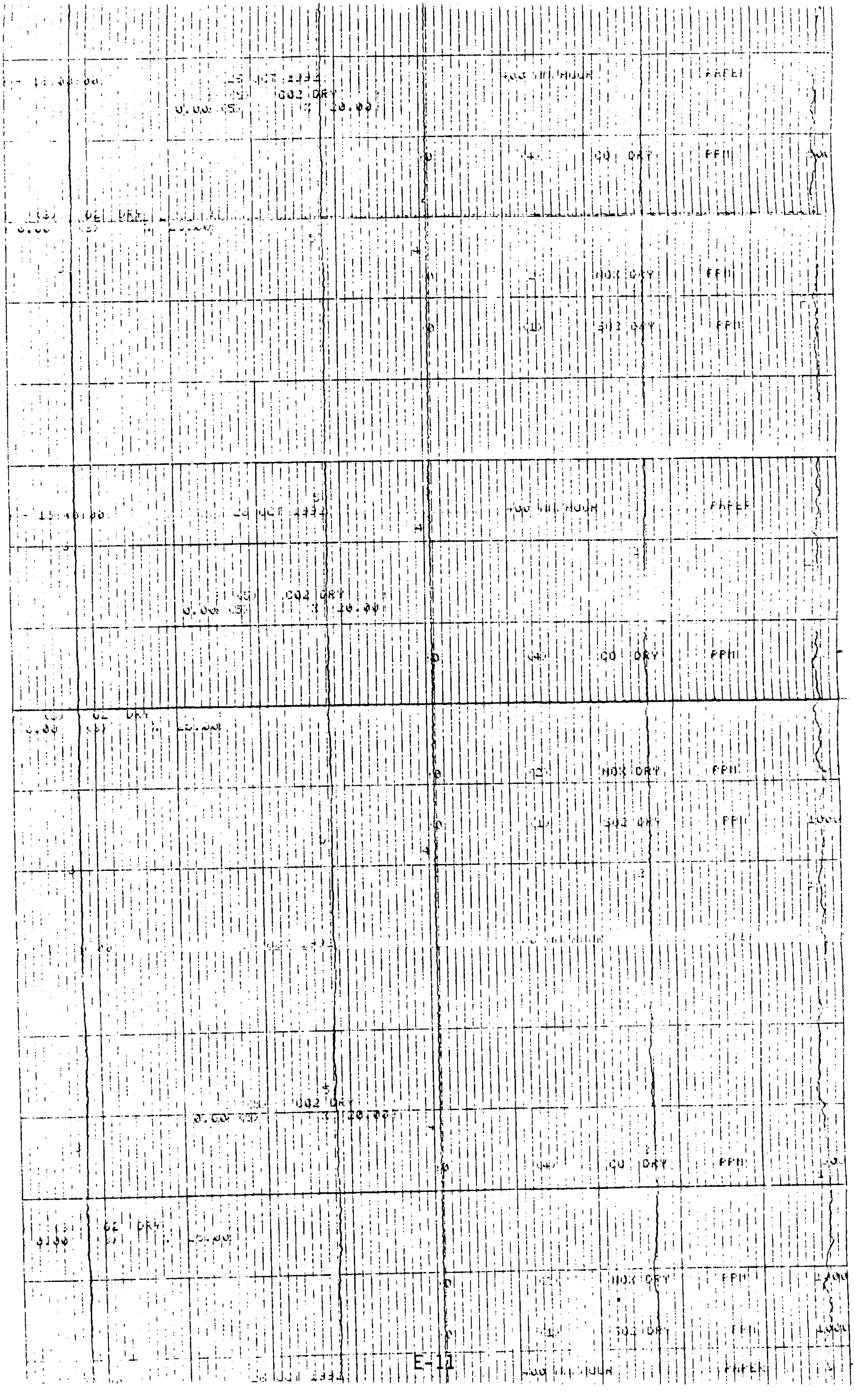
F-8


13:40:00
(5) CO2 DRY
26 OCT 1993
0.00 (5) 3 20.00
400 MM/HOUR
PAPER
0 (4) CO DRY PPM 500
(2) O2 DRY
0 (3) NOX DRY PPM
0 (1) SO2 DRY PPM
12:40:00
26 OCT 1993
400 MM/HOUR
PAPER
(5) CO2 DRY
0.00 (5) 3 20.00
0 (4) CO DRY PPM
(2) O2 DRY
0 (3) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
11:40:00
26 OCT 1993
400 MM/HOUR
PAPER

10/26



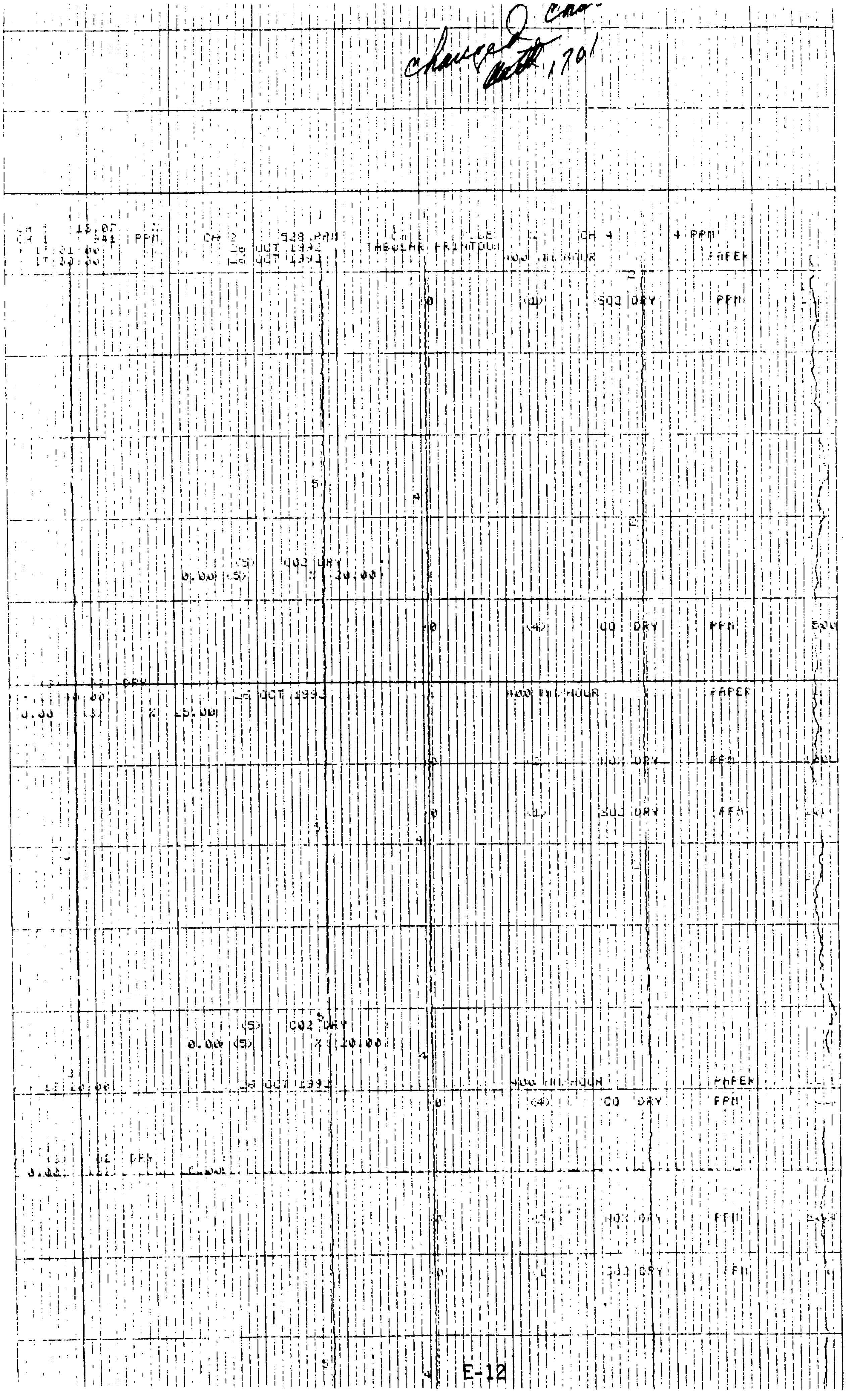
10/26



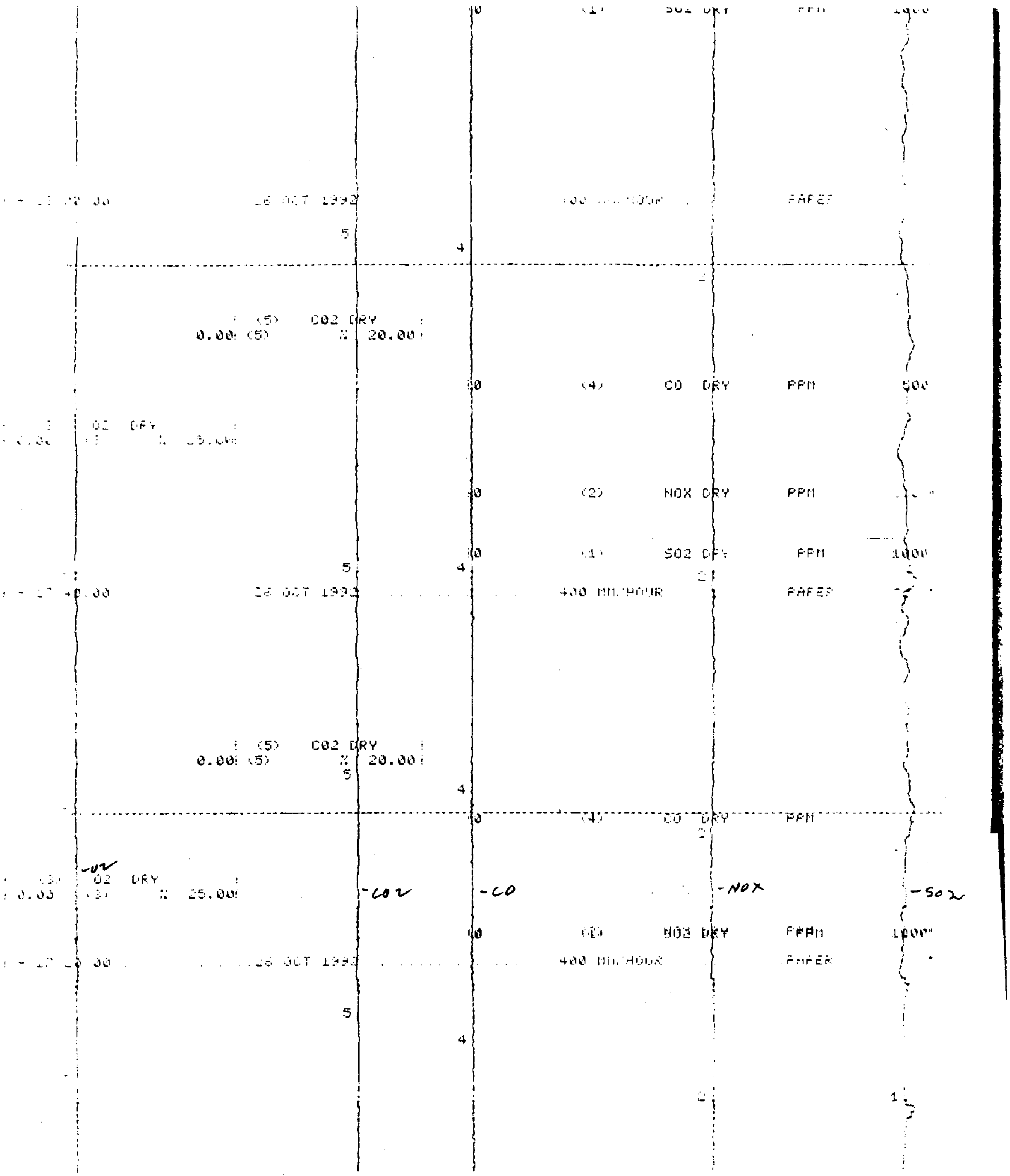
002 DRY
0.00 (S)
10.00
CO DRY
PPM
NOX DRY
SO2 DRY
O2 DRY
25.00
HOUR
PAPER
15:40:00
CO2 DRY

10/26

changed cha- 1701



10/26



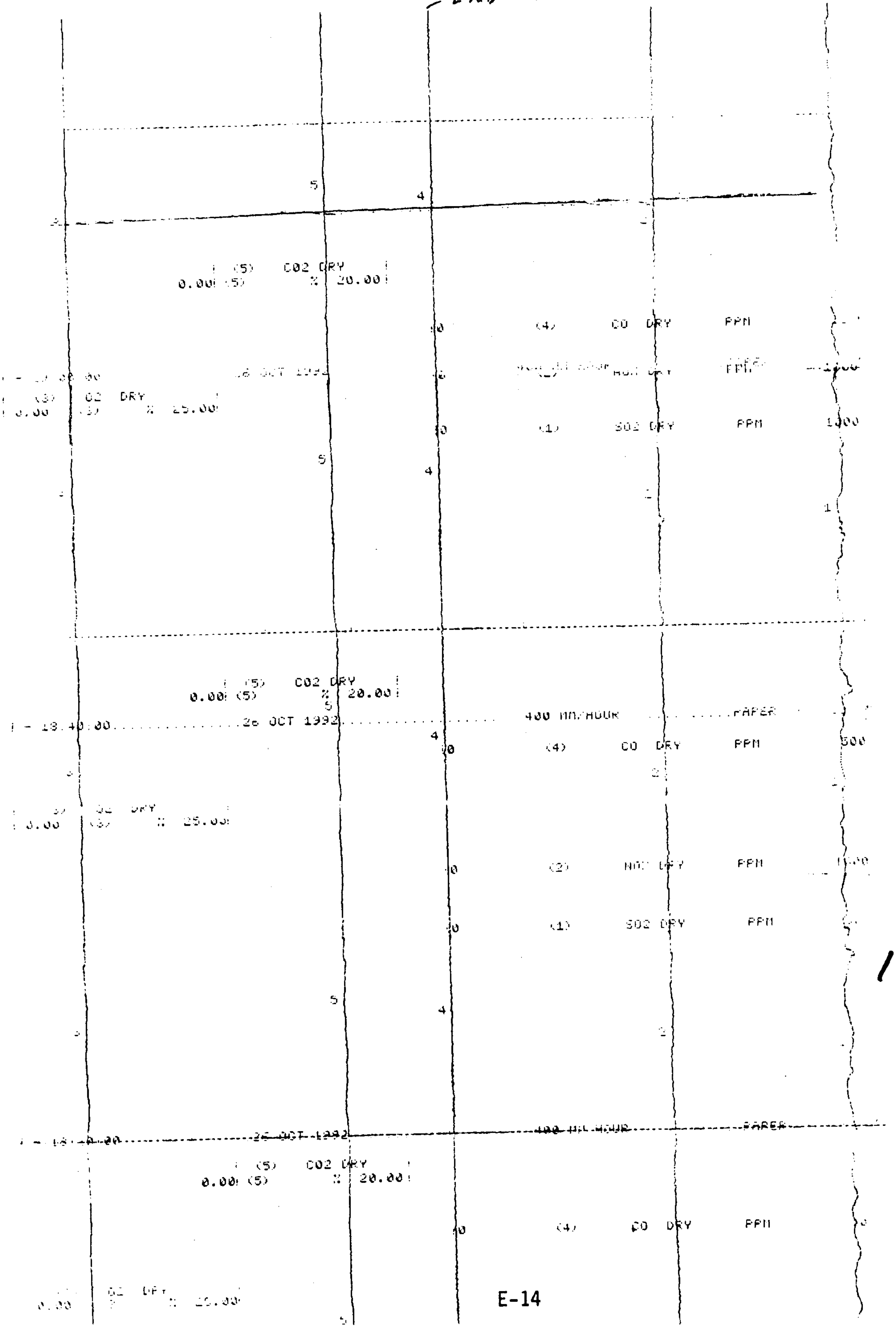
```
CH 5   13.06   %
CH 1     418  PPM     CH 2     528 PPM     CH 3    7.81   %     CH 4     5 PPM
   .11.00 ............26 OCT 1992.....TABULAR PRINTOUT
```

10/26

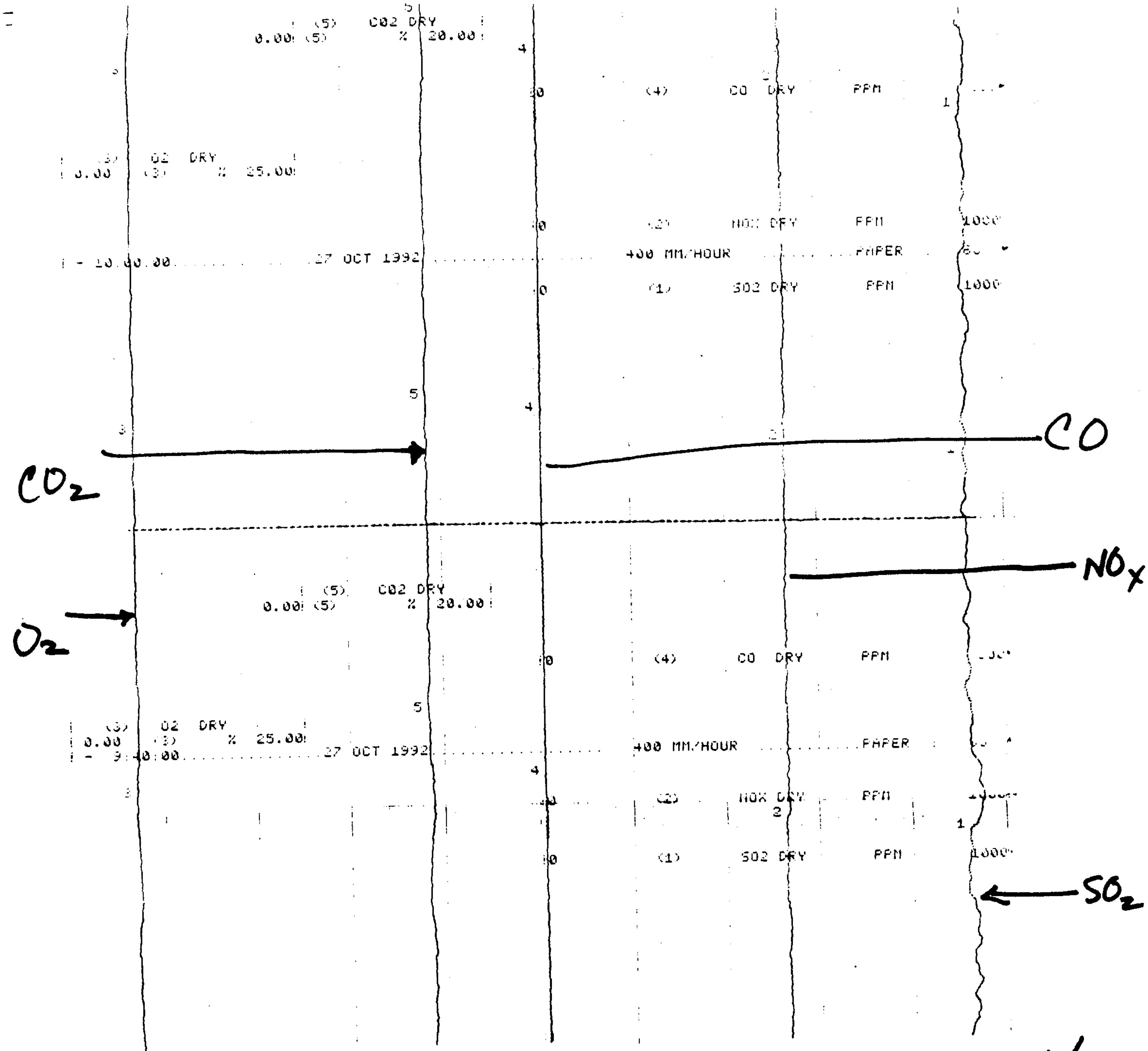
BASELINE STUDY - COAL ONLY E-13

System wir

— END TEST 1915



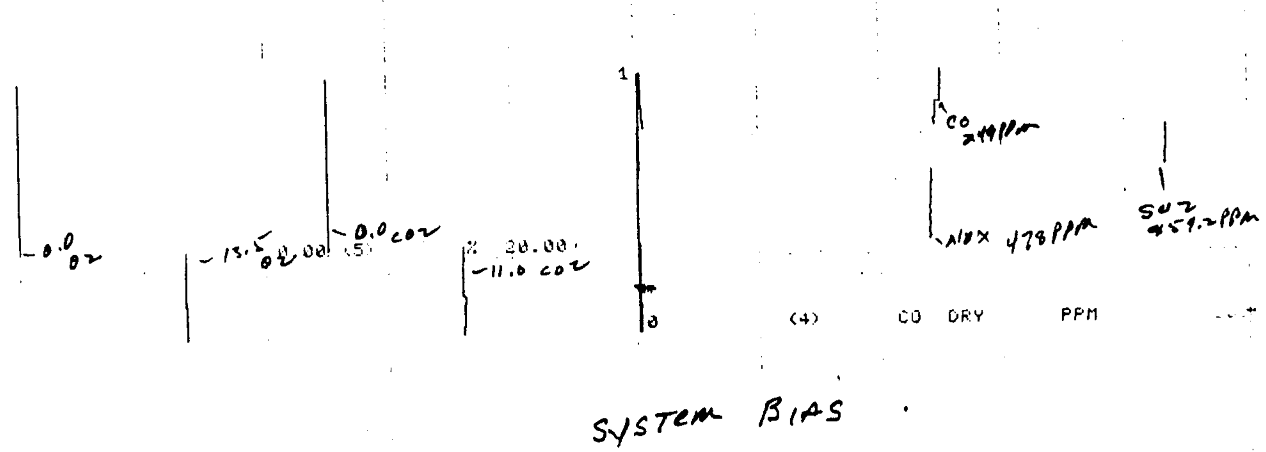
10/26



```
CH 5   13.32   %                        CH 3    7.18   %      CH 4       4 PPM
CH 1     910  PPM      CH 2     525 PPM
- 9:28:00              27 OCT 1992      TABULAR PRINTOUT
```

10/27

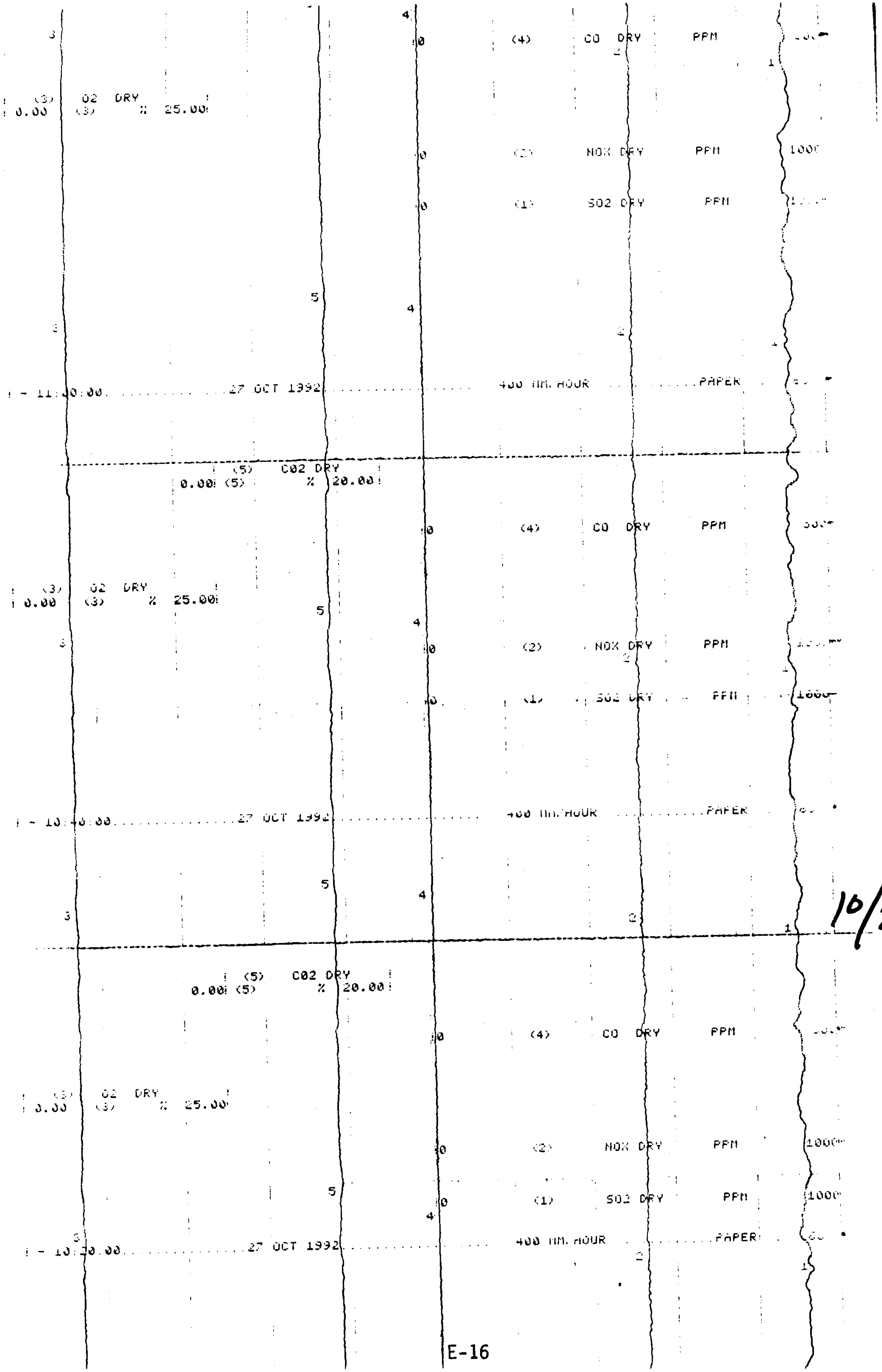
Run 1



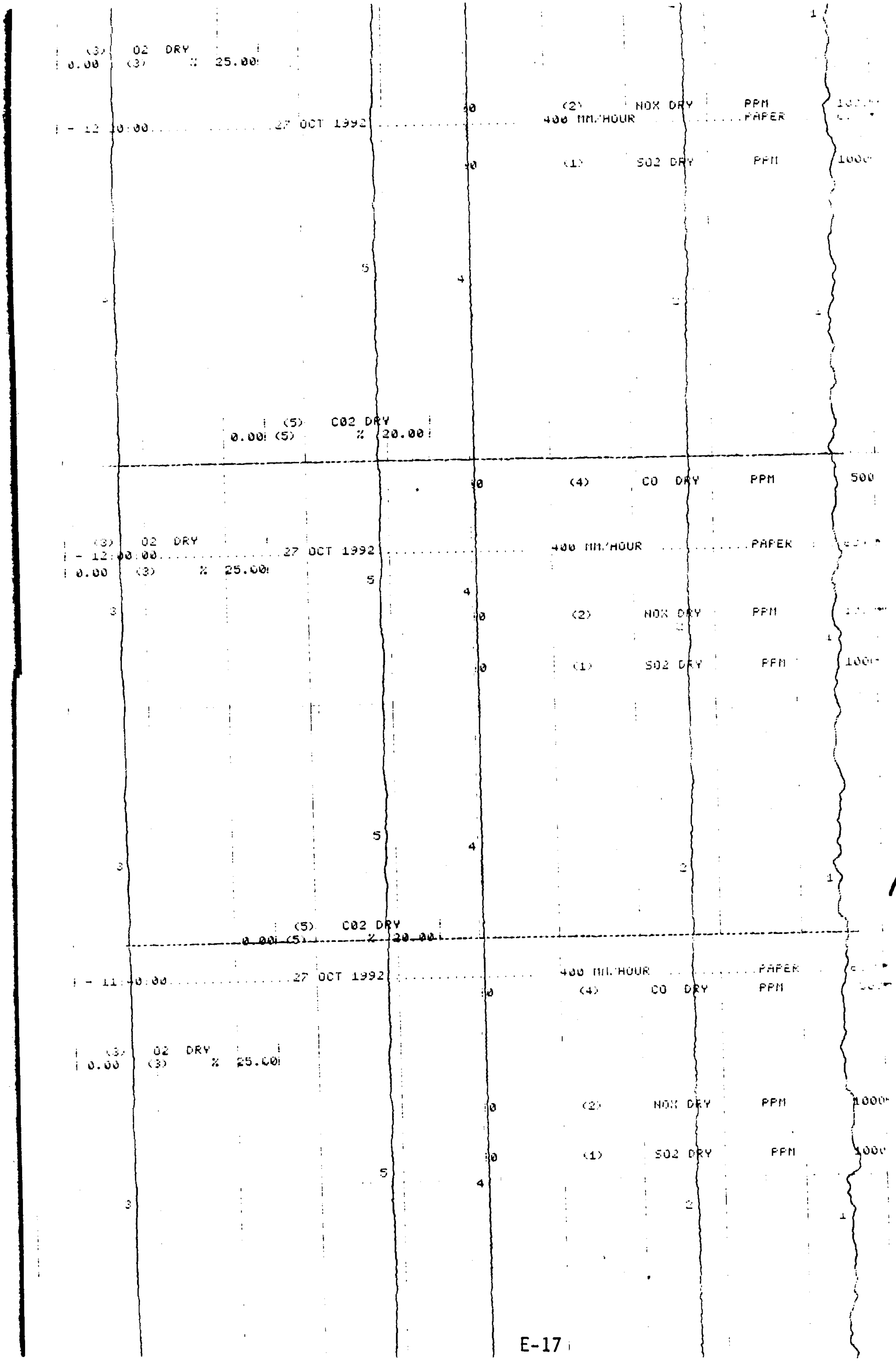
SYSTEM BIAS

```
CH 5   -0.17   %                        CH 3  -20.92   %      CH 4       1 PPM
CH 1      -6  PPM      CH 2      -4 PPM
+ 8:21:00              27 OCT 1992      TABULAR PRINTOUT
```

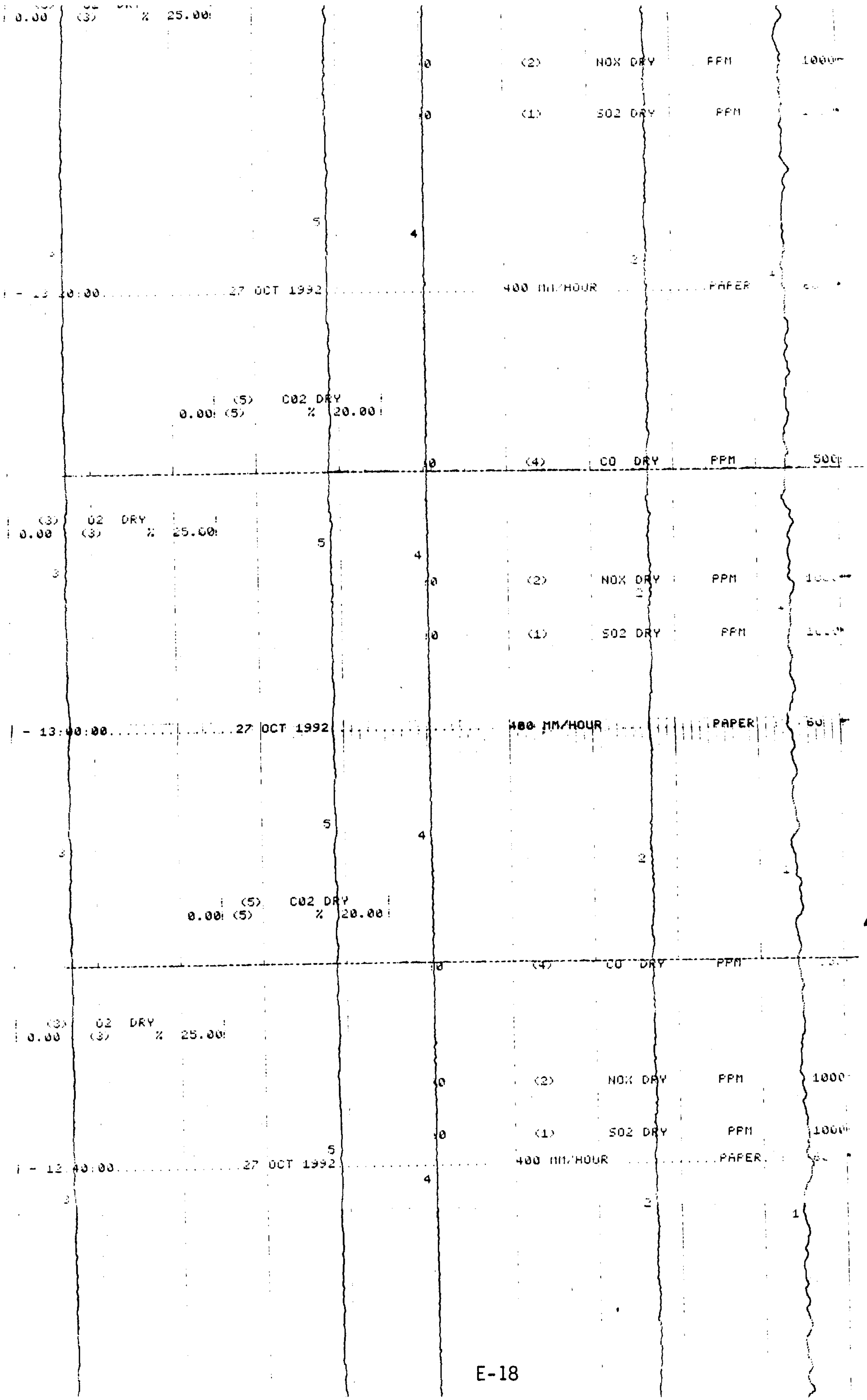
... Boiler stack


(4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM
- 11:40:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM
0 (1) SO2 DRY PPM 1000
- 10:40:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
- 10:20:00 27 OCT 1992 400 MM/HOUR PAPER
1
2
3
4
5

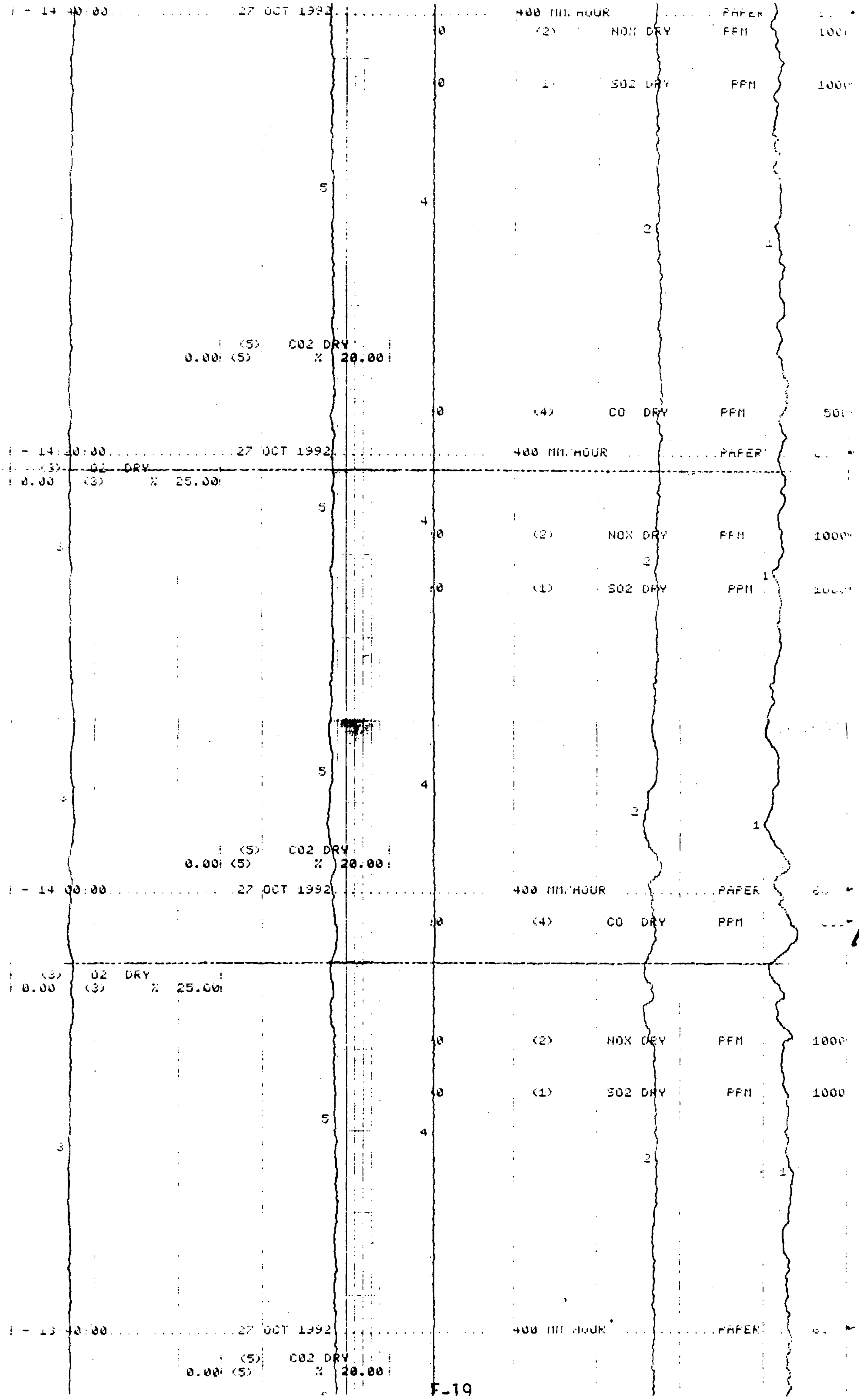
10/27


(3) O2 DRY
0.00 (3) % 25.00
(2) NOX DRY PPM
400 MM/HOUR PAPER
- 12:10:00 27 OCT 1992
(1) SO2 DRY PPM 1000
(5) CO2 DRY
0.00 (5) % 20.00
(4) CO DRY PPM 500
(3) O2 DRY
- 12:00:00 27 OCT 1992 400 MM/HOUR PAPER
0.00 (3) % 25.00
(2) NOX DRY PPM
(1) SO2 DRY PPM 1000
(5) CO2 DRY
0.00 (5) % 20.00
- 11:50:00 27 OCT 1992 400 MM/HOUR PAPER
(4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
(2) NOX DRY PPM 1000
(1) SO2 DRY PPM 1000
1
2
3
4
5

10/27

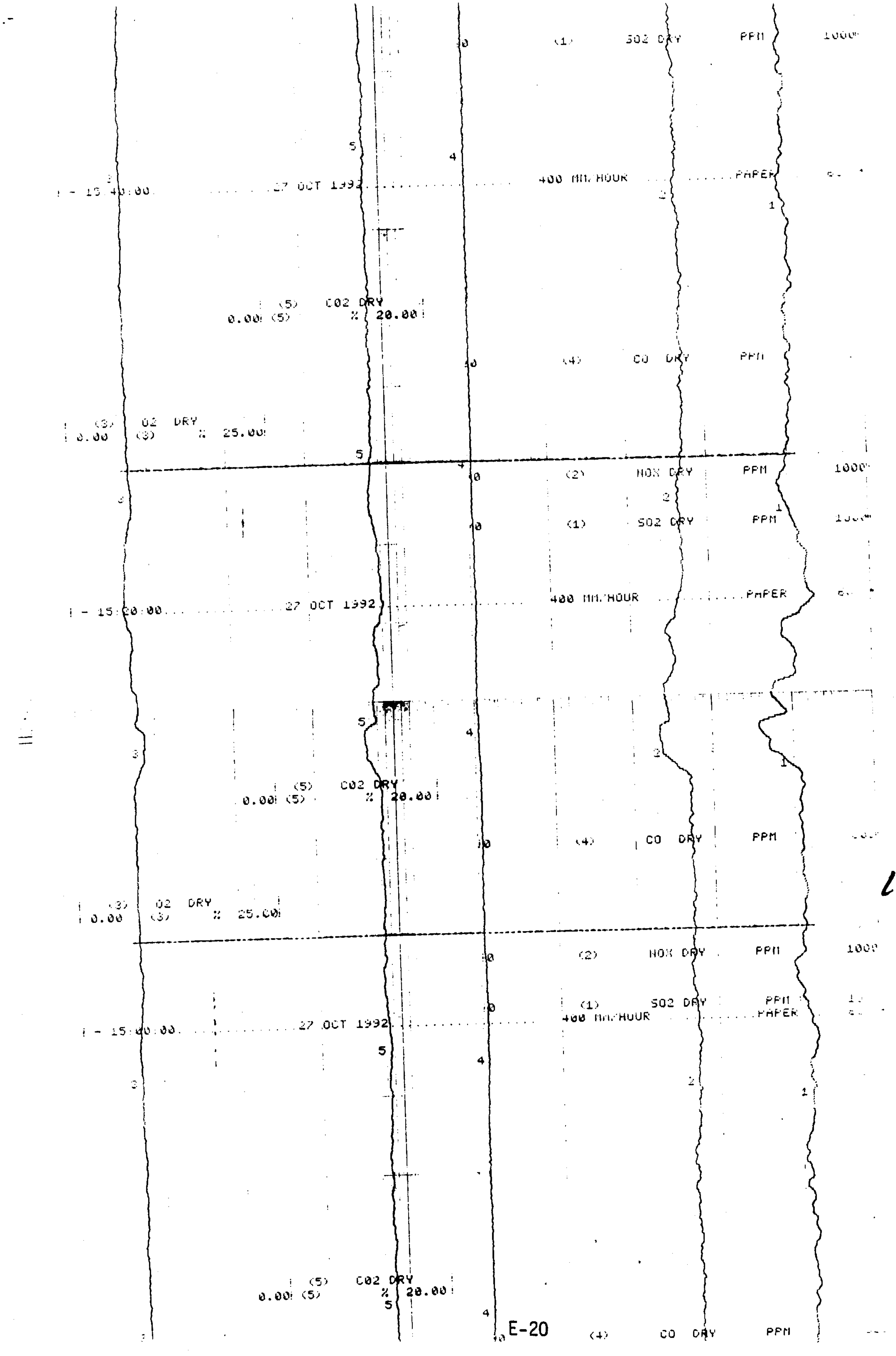

0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM
5
4
3
2
1
- 13:20:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM 500
(3) O2 DRY
0.00 (3) % 25.00
5
4
3
0 (2) NOX DRY PPM
2
0 (1) SO2 DRY PPM
- 13:00:00 27 OCT 1992 400 MM/HOUR PAPER 60
5
4
3
2
1
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
5
- 12:40:00 27 OCT 1992 400 MM/HOUR PAPER
4
3
2
1

10/27

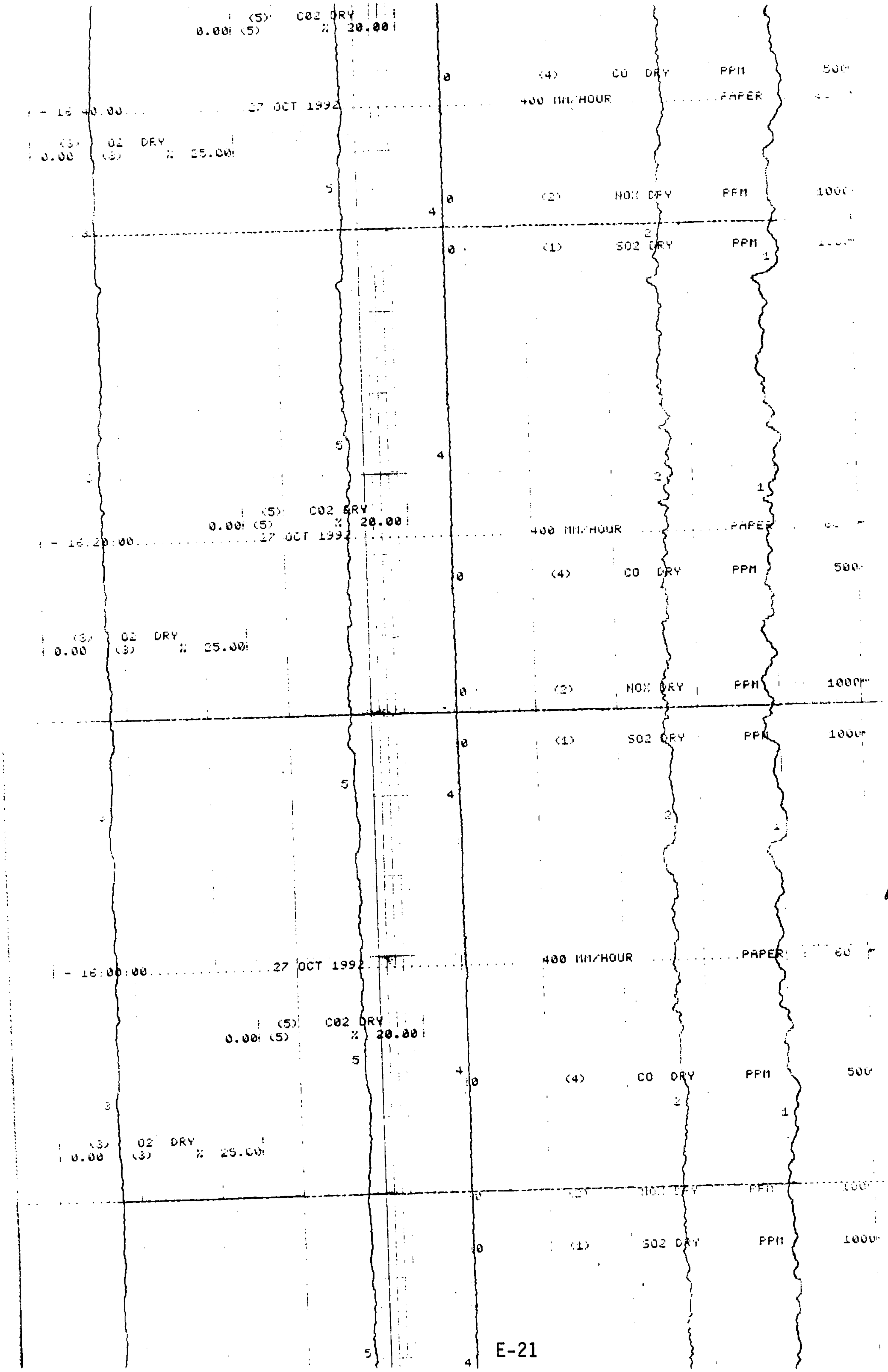


- 14 40:00 27 OCT 1992 400 MM/HOUR PAPER
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM 500
- 14 20:00 27 OCT 1992 400 MM/HOUR PAPER
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
(5) CO2 DRY
0.00 (5) % 20.00
- 14 00:00 27 OCT 1992 400 MM/HOUR PAPER
0 (4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
- 13 40:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
1
2
3
4
5

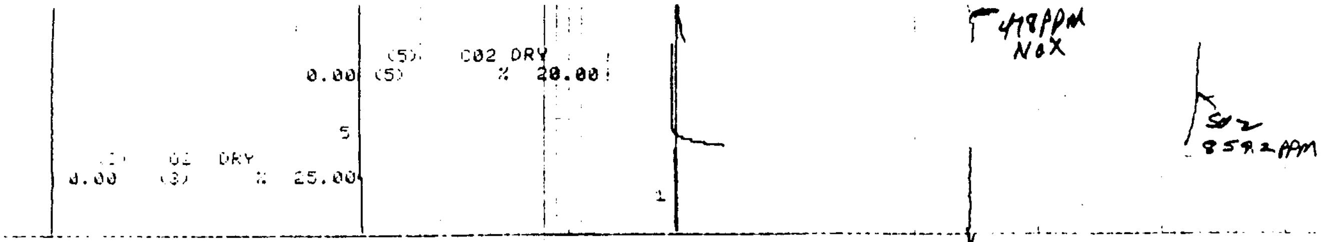
10/27


(1) SO2 DRY PPM 1000
I - 15:40:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
(4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
(2) NOX DRY PPM 1000
(1) SO2 DRY PPM 1000
I - 15:20:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
(4) CO DRY PPM
(3) O2 DRY
0.00 (3) % 25.00
(2) NOX DRY PPM 1000
(1) SO2 DRY PPM
I - 15:00:00 27 OCT 1992 400 MM/HOUR PAPER
(5) CO2 DRY
0.00 (5) % 20.00
(4) CO DRY PPM

10/2


(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM 500
- 16:40:00 27 OCT 1992 400 MM/HOUR PAPER
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
(5) CO2 DRY
0.00 (5) % 20.00
- 16:20:00 27 OCT 1992 400 MM/HOUR PAPER
0 (4) CO DRY PPM 500
(3) O2 DRY
0.00 (3) % 25.00
0 (2) NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000
- 16:00:00 27 OCT 1992 400 MM/HOUR PAPER 60
(5) CO2 DRY
0.00 (5) % 20.00
0 (4) CO DRY PPM 500
(3) O2 DRY
0.00 (3) % 25.00
NOX DRY PPM 1000
0 (1) SO2 DRY PPM 1000

10/27



478PPM NOx

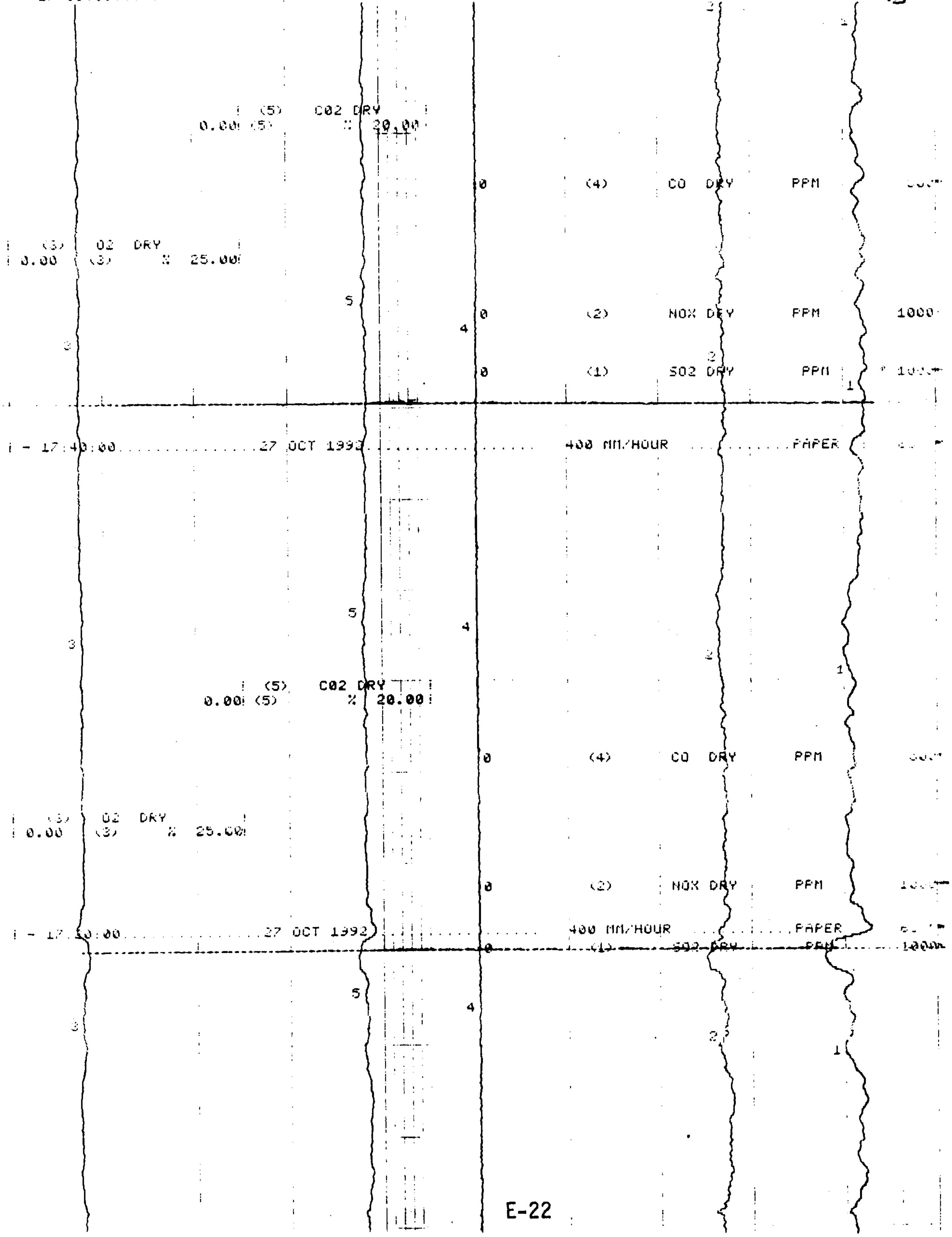
SO2 859.2 PPM

CO 749 PPM

SYSTEM BIAS

```
CH 5   11.70   %
CH 1     755  PPM      CH 2     486 PPM      CH 3   8.85    %      CH 4        1 PPM
* 17:59:00..................27 OCT 1992.....TABULAR PRINTOUT
```

END TEST 1800 HRS



10/2

ARGONNE NATIONAL LABS - OTP

Total Hydrocarbons (ppm as propane,wet)
DAY 1 (10-26-92)
TIME
(0940 - 1910)

1
1
1
1
1.5
1.5
1.5
1.5
1.5
1.5
1
0.5
1.5
1
1.5
1
1
1
1
1
1
1.5
1.5
1.5
2
2
2
1.5
2.5
2
2
2
2
2.5
3
3
3
3
3
2.5
2
2
2
2
2
0.5
2

2.5
3
3
3.5
4
4
4
4
4
4
4
3.5
3
3
1
3.5
3
3
3
3
3
3
3.5
3.5
3.5
2
0.5
0.5
0.5

Total 165
Avg 2.17
X 3 (To convert to ppmC,w)

6.51 ppmC,w

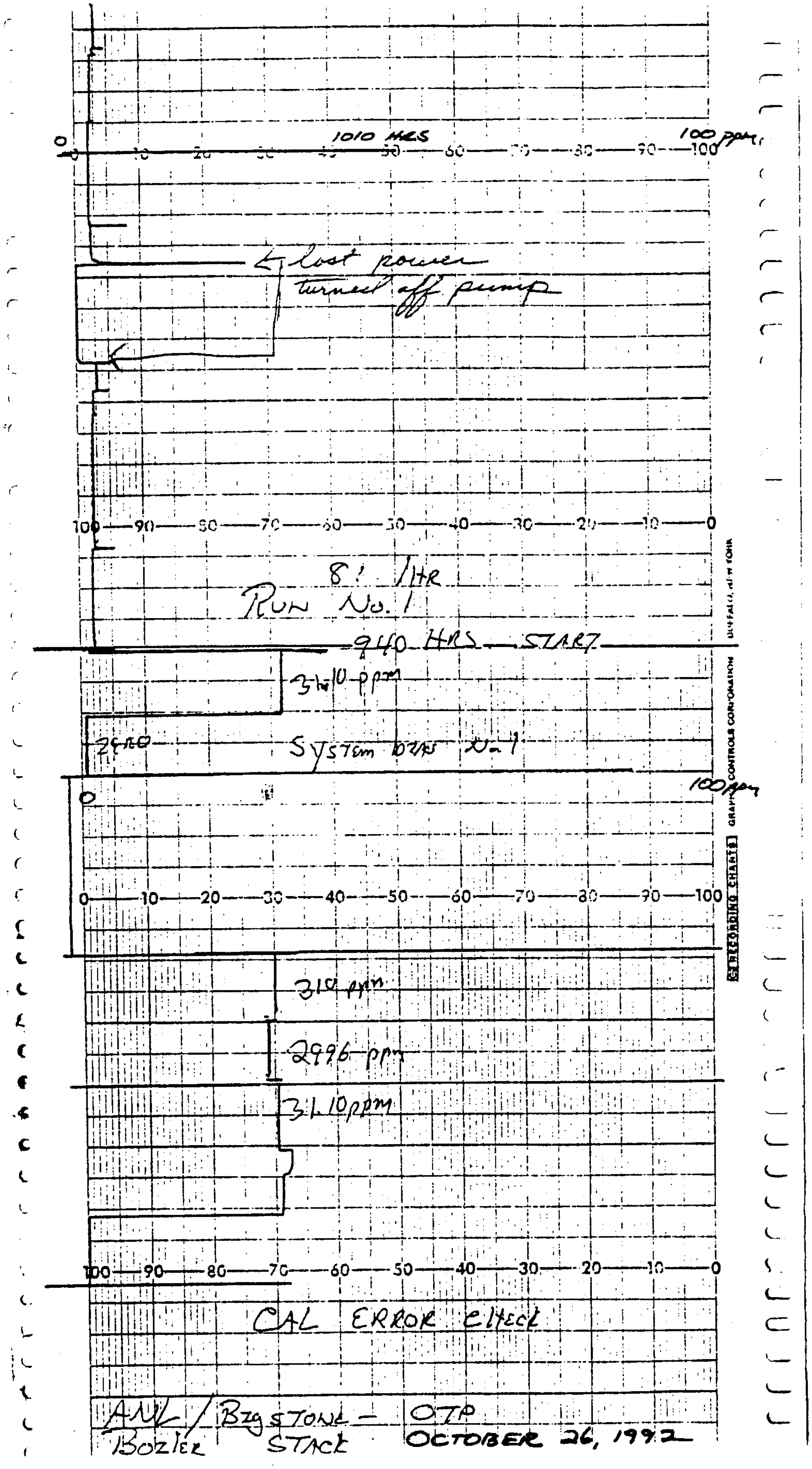
ARGONNE NATIONAL LABS - OTP

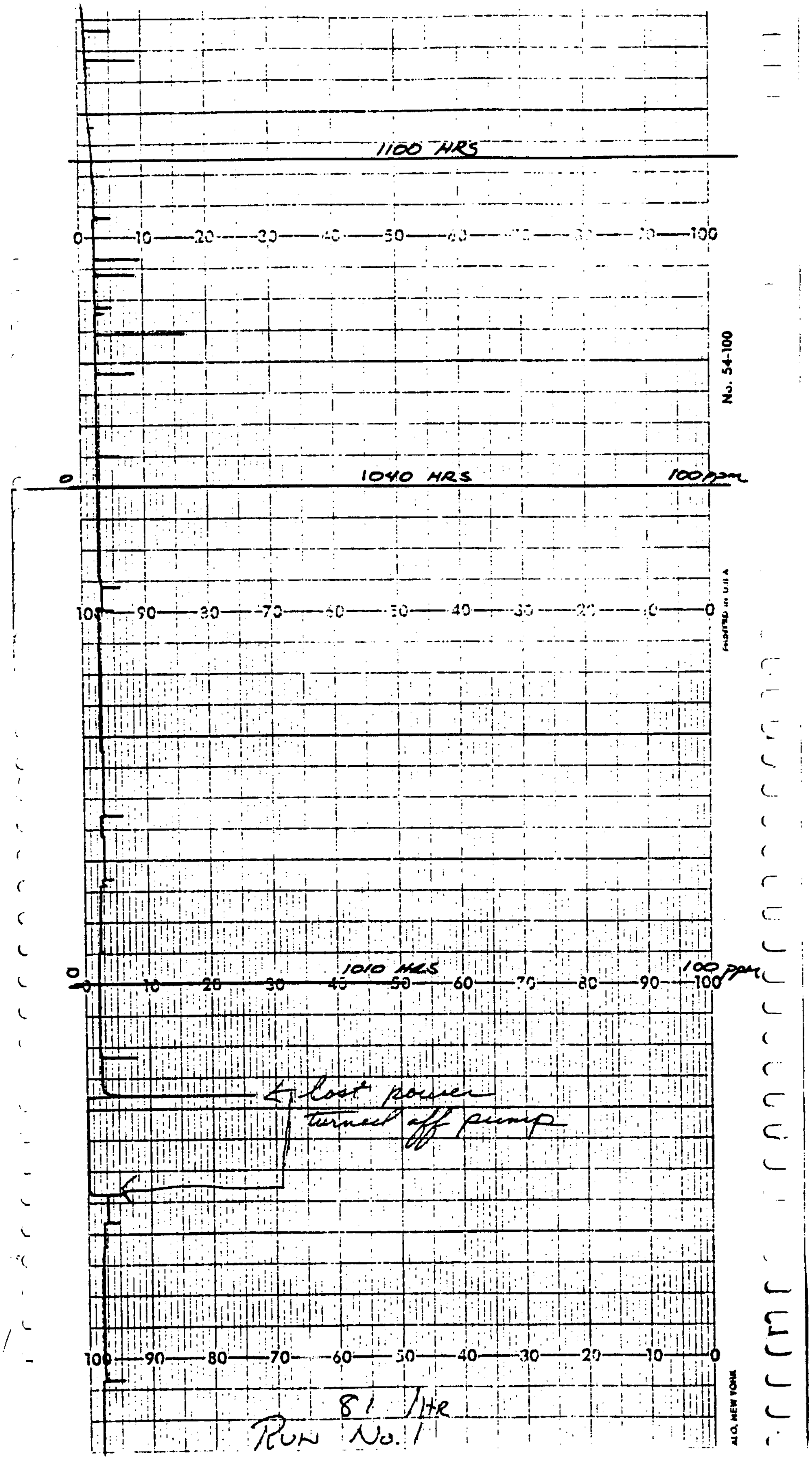
Total Hydrocarbons (ppm as propane,wet)
DAY 2 (10-27-92)
TIME
(1500-1800)

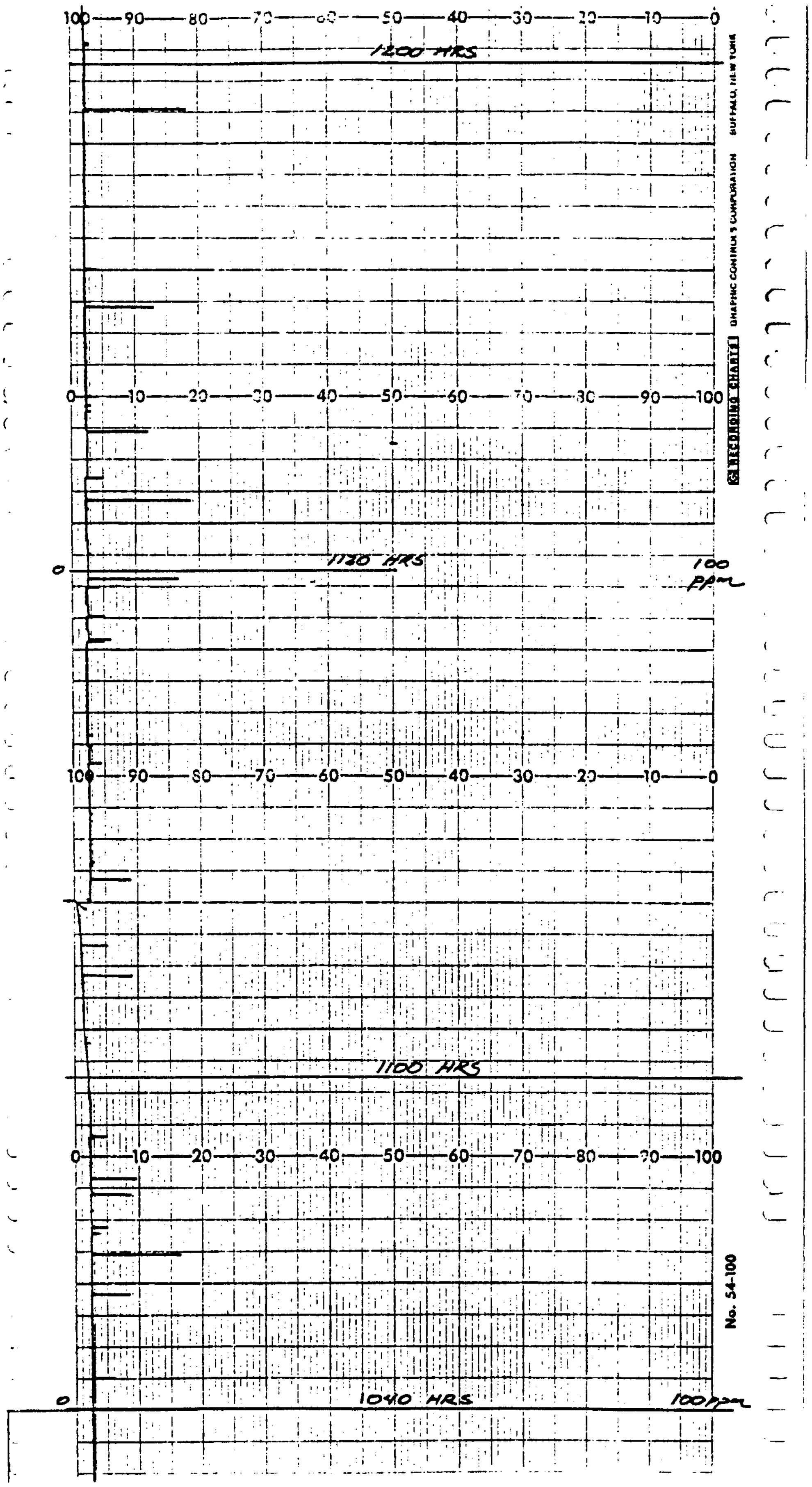
4
4.5
4.5
4.5
5
5
5.5
5.5
5.5
5.5
5.5
5.5
3.5
3.5
3.5
3.5
3.5
2.5
2.5
3
3
3
3
3

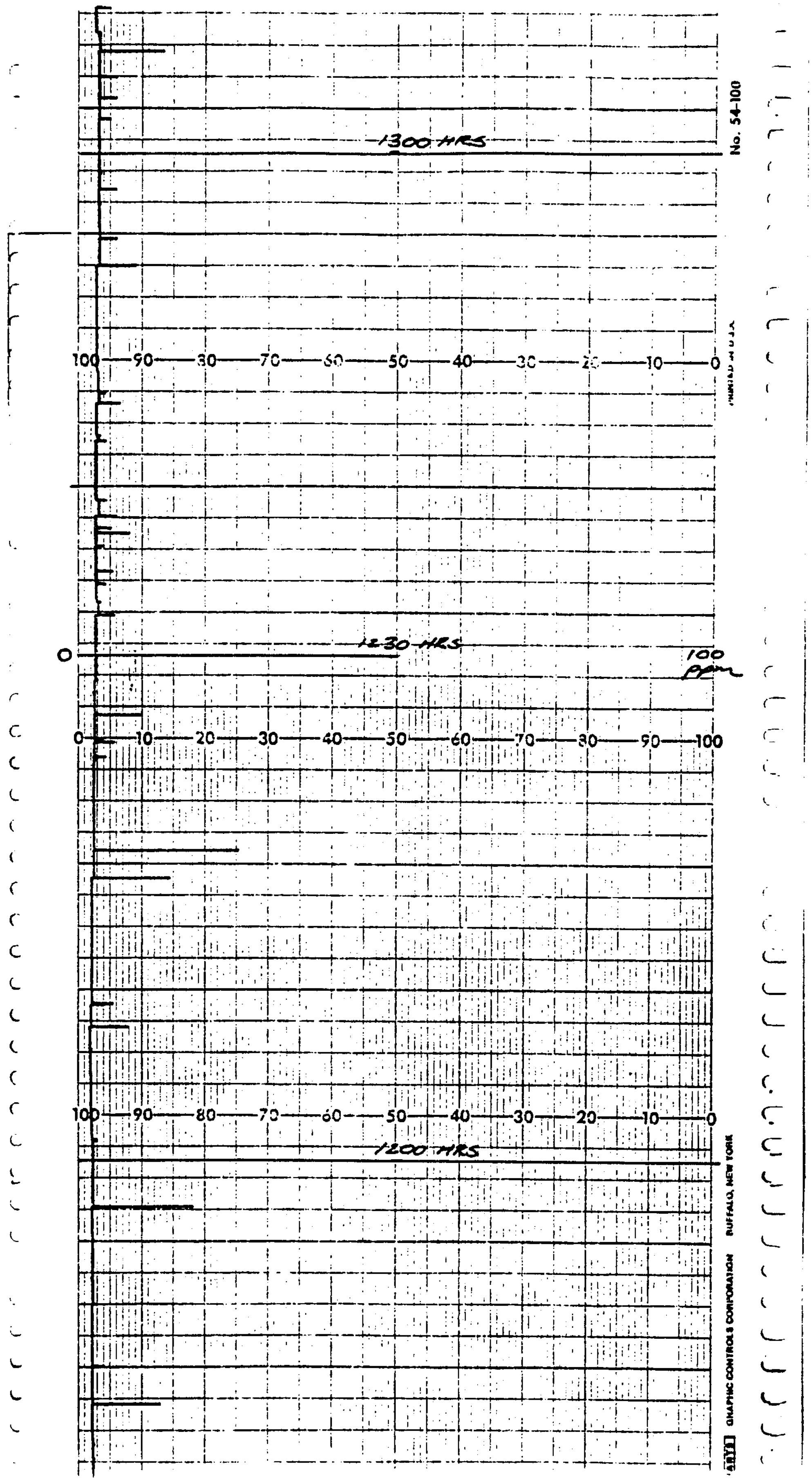
Total 98
Avg 4.08
X 3 (To convert to ppmC,w)

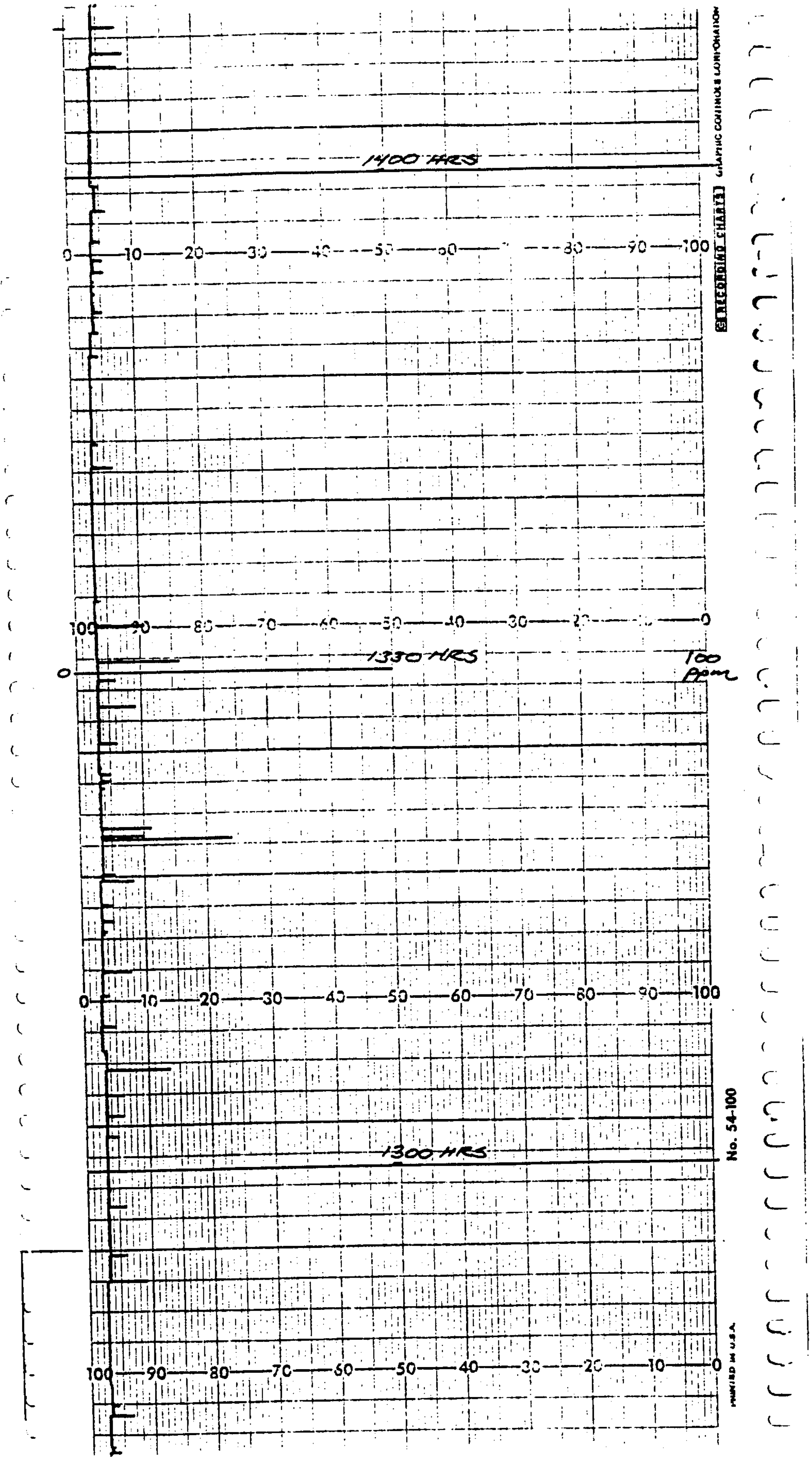
12.24 ppmC,w

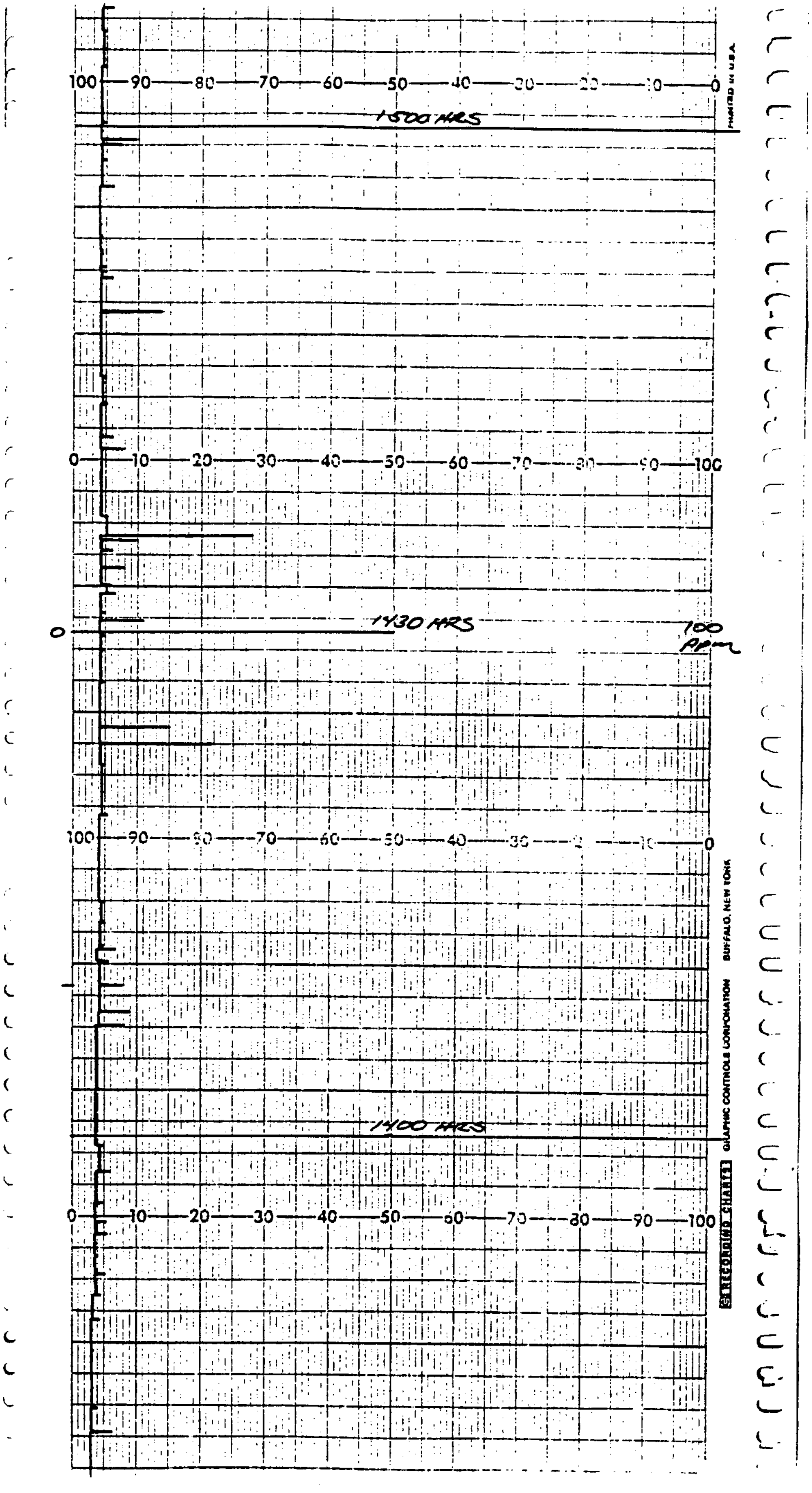

1010 HRS
100 ppm
0
0 10 20 30 40 50 60 70 80 90 100
lost power
turned off pump
100 90 80 70 60 50 40 30 20 10 0
8! 1HR
RUN No. 1
940 HRS START
3110 ppm
zero
SYSTEM BIAS No. 1
0
100ppm
0 10 20 30 40 50 60 70 80 90 100
310 ppm
2996 ppm
3110ppm
100 90 80 70 60 50 40 30 20 10 0
CAL ERROR CHECK
ANL / BIG STONE - OTP
BOILER STACK
OCTOBER 26, 1992
GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK
RECORDING CHARTS

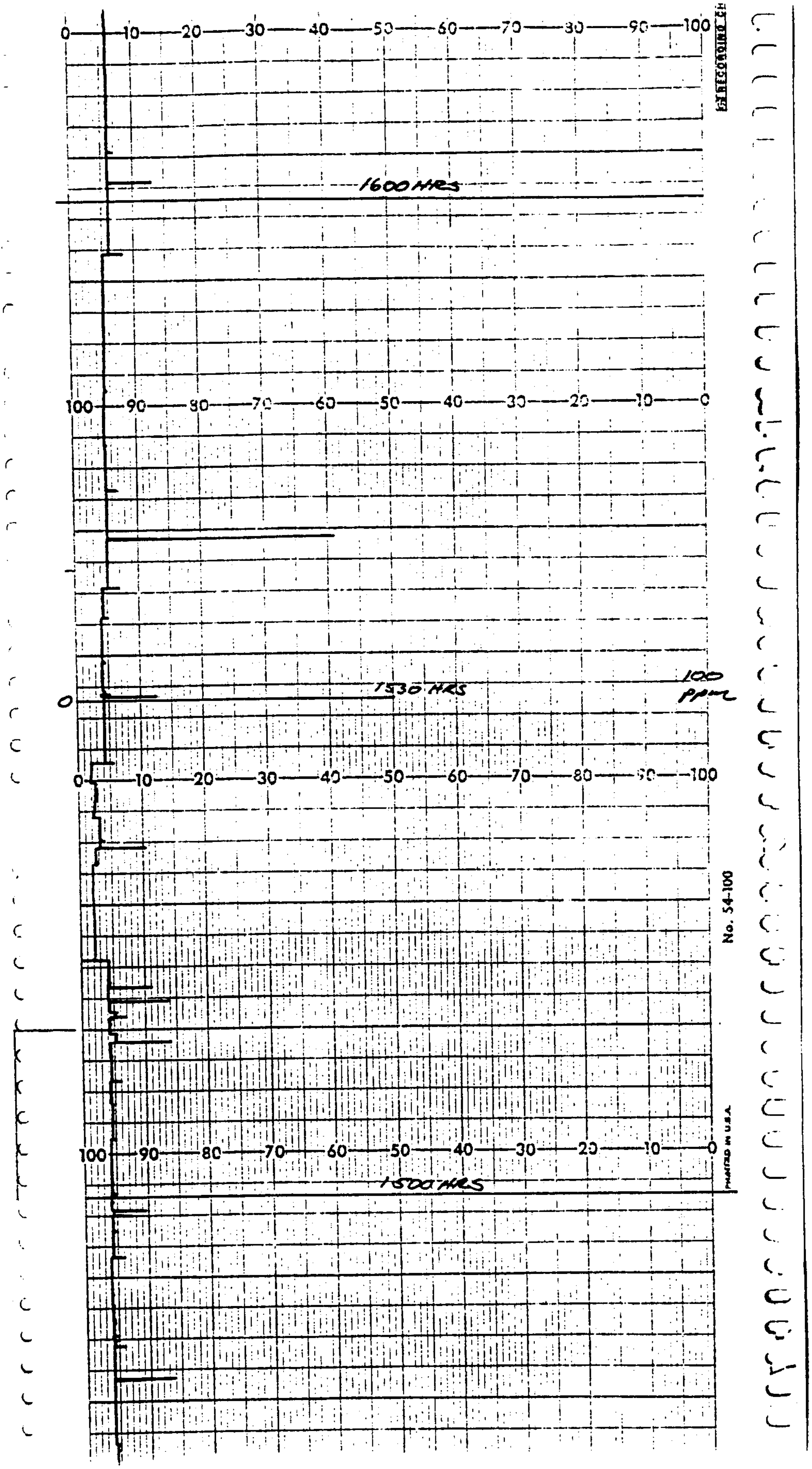

1100 HRS
0—10—20—30—40—50—60—70—80—90—100
No. 54-100
0
1040 HRS
100ppm
100—90—80—70—60—50—40—30—20—10—0
PRINTED IN U.S.A.
0
1010 HRS
100 ppm
0—10—20—30—40—50—60—70—80—90—100
← lost power
turned off pump
100—90—80—70—60—50—40—30—20—10—0
ALO, NEW YORK
81 l/Hr
Run No. 1

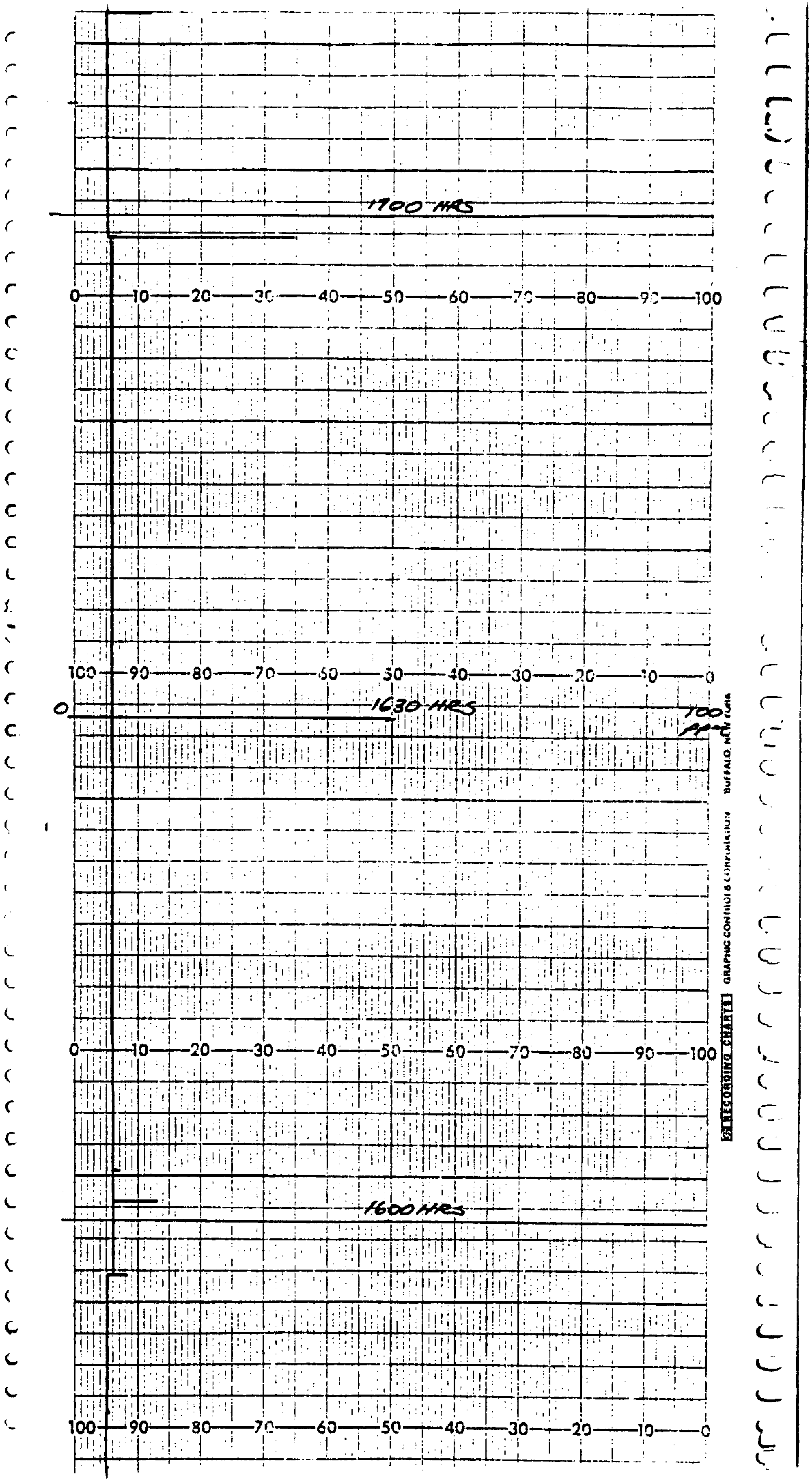

100 90 80 70 60 50 40 30 20 10 0
1200 HRS
0 10 20 30 40 50 60 70 80 90 100
1130 HRS
0
100 ppm
100 90 80 70 60 50 40 30 20 10 0
1100 HRS
0 10 20 30 40 50 60 70 80 90 100
1040 HRS
0
100 ppm
RECORDING CHARTS
GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK
No. 54-100

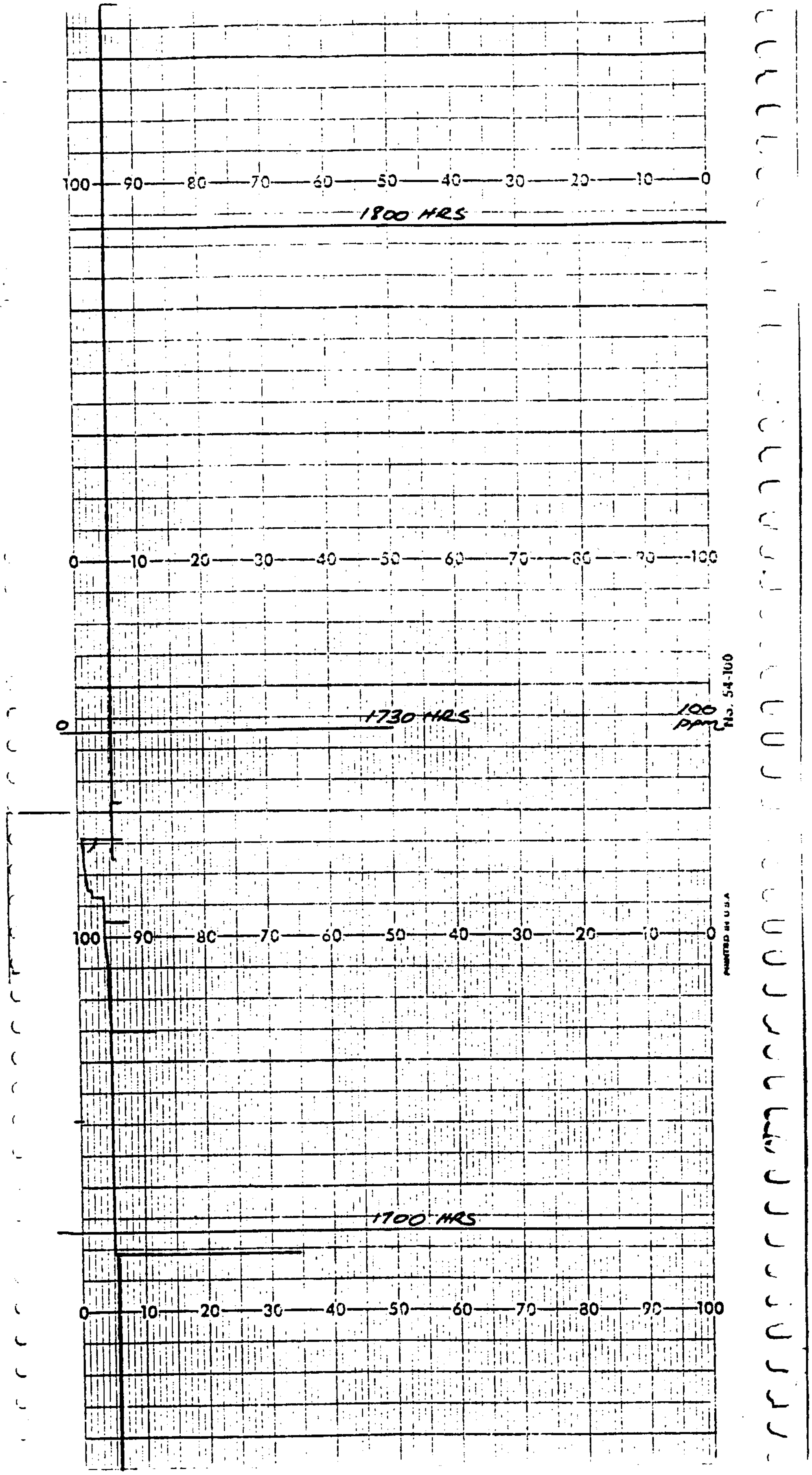

1300 HRS
100 90 80 70 60 50 40 30 20 10 0
1230 HRS
0
100 ppm
0 10 20 30 40 50 60 70 80 90 100
100 90 80 70 60 50 40 30 20 10 0
1200 HRS
No. 54-100
PRINTED IN U.S.A.
GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK

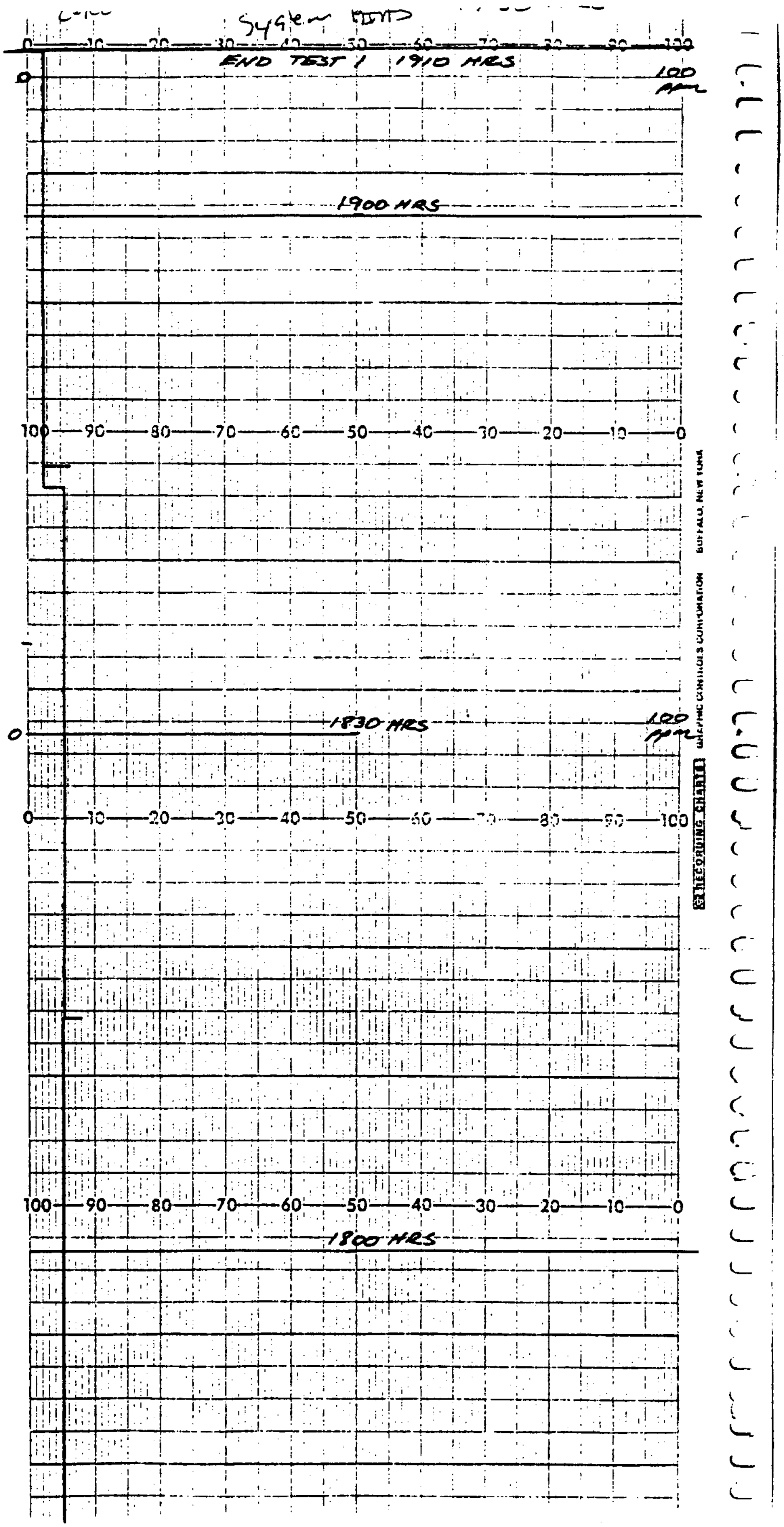

1400 HRS
GRAPHIC CONTROLS CORPORATION
RECORDING CHARTS
0 10 20 30 40 50 60 80 90 100
100 90 80 70 60 50 40 30 20 0
1330 HRS
0
100 ppm
0 10 20 30 40 50 60 70 80 90 100
No. 54-100
1300 HRS
100 90 80 70 60 50 40 30 20 10 0
PRINTED IN U.S.A.

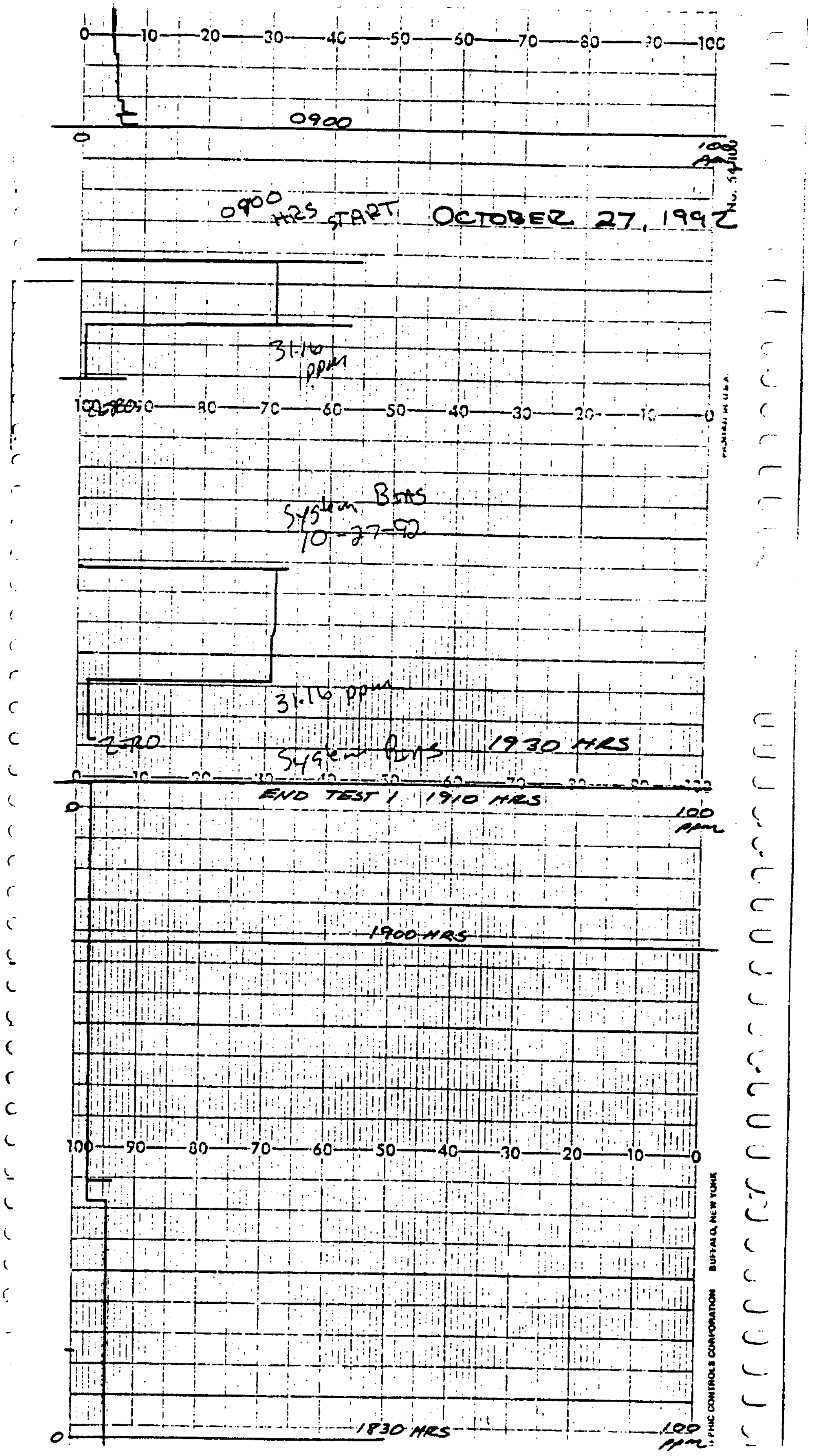

100 90 80 70 60 50 40 30 20 10 0
PRINTED IN U.S.A.
1500 HRS
0 10 20 30 40 50 60 70 80 90 100
1430 HRS
0
100 PPM
100 90 80 70 60 50 40 30 20 10 0
GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK
1400 HRS
RECORDING CHARTS
0 10 20 30 40 50 60 70 80 90 100


0—10—20—30—40—50—60—70—80—90—100
1600 HRS
100—90—80—70—60—50—40—30—20—10—0
1530 HRS
100 ppm
0
0—10—20—30—40—50—60—70—80—90—100
No. 54-100
100—90—80—70—60—50—40—30—20—10—0
1500 HRS
PRINTED IN U.S.A.


1700 HRS
0 10 20 30 40 50 60 70 80 90 100
100 90 80 70 60 50 40 30 20 10 0
0
1630 HRS
700 ppm
GC RECORDING CHARTS GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK
0 10 20 30 40 50 60 70 80 90 100
1600 HRS
100 90 80 70 60 50 40 30 20 10 0


100—90—80—70—60—50—40—30—20—10—0
1800 HRS
0—10—20—30—40—50—60—70—80—90—100
No. 54-100
0
1730 HRS
100 ppm
PRINTED IN U.S.A.
100—90—80—70—60—50—40—30—20—10—0
1700 HRS
0—10—20—30—40—50—60—70—80—90—100


System
0 10 20 30 40 50 60 70 80 90 100
END TEST 1 1910 HRS
0
100 ppm
1900 HRS
100 90 80 70 60 50 40 30 20 10 0
0
1830 HRS
100 ppm
0 10 20 30 40 50 60 70 80 90 100
100 90 80 70 60 50 40 30 20 10 0
1800 HRS
RECORDING CHARTS GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK


0900

0900 HRS START OCTOBER 27, 1992

31.16 ppm

System Bias 10-27-92

31.16 ppm

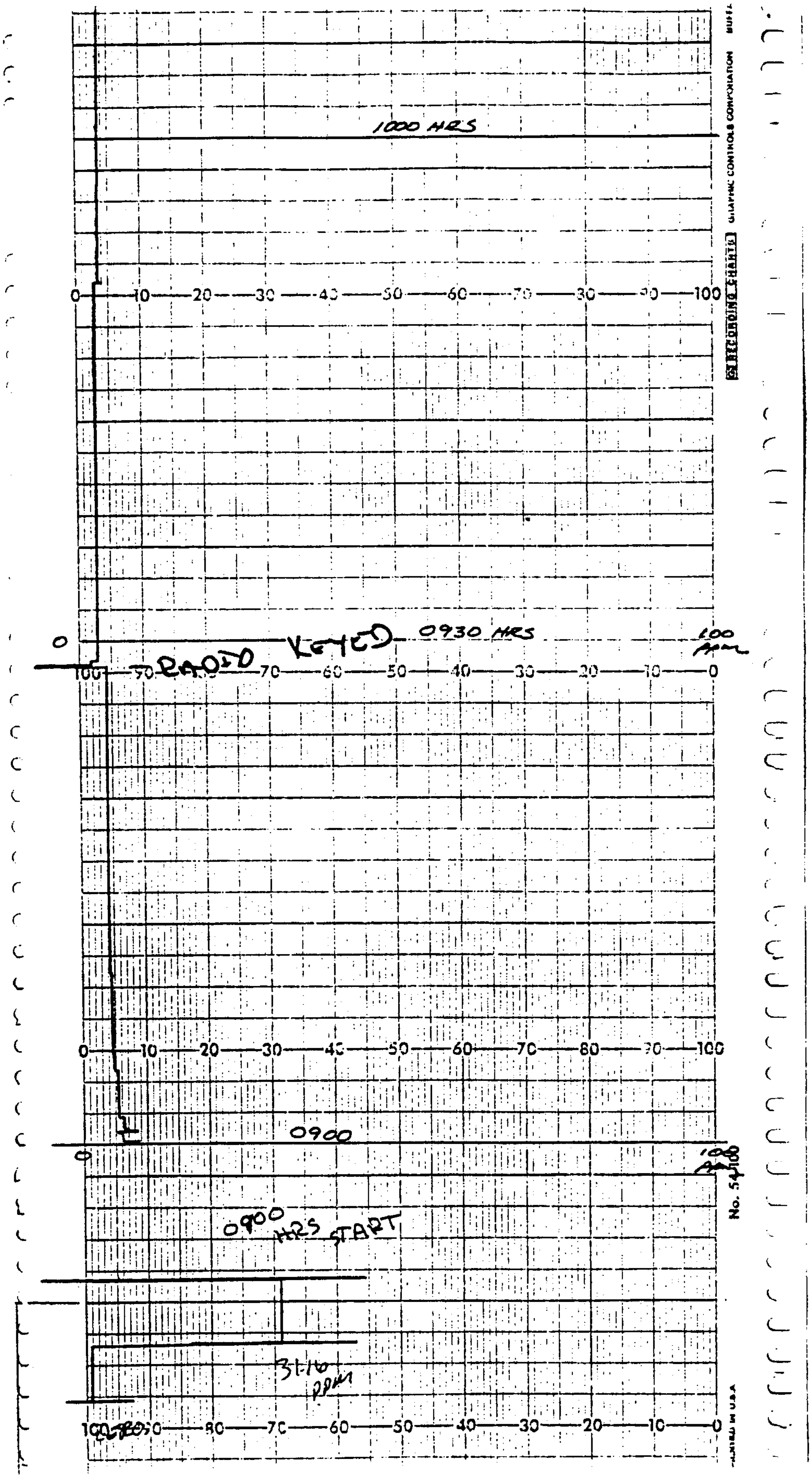
Zero

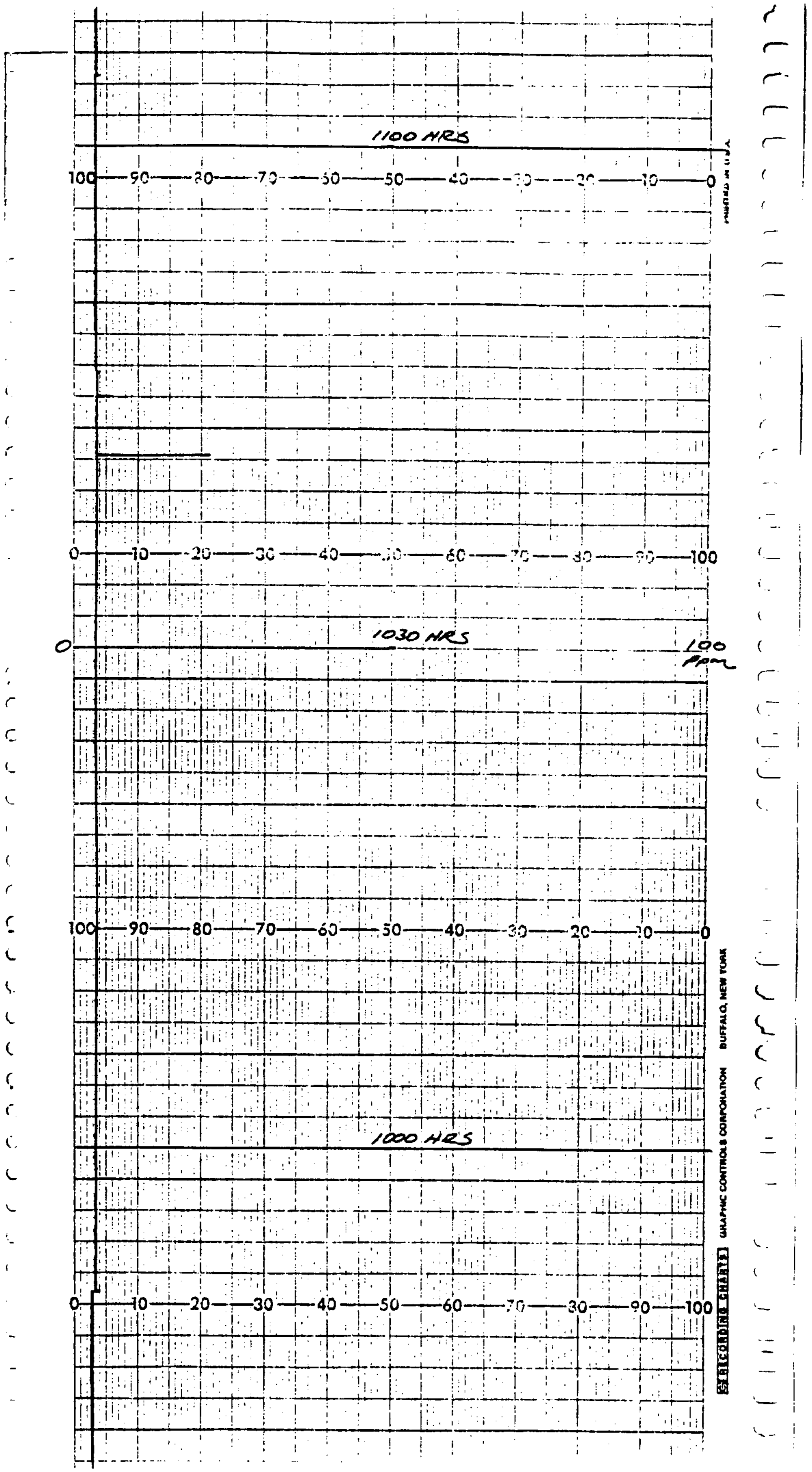
System Bias 1930 HRS

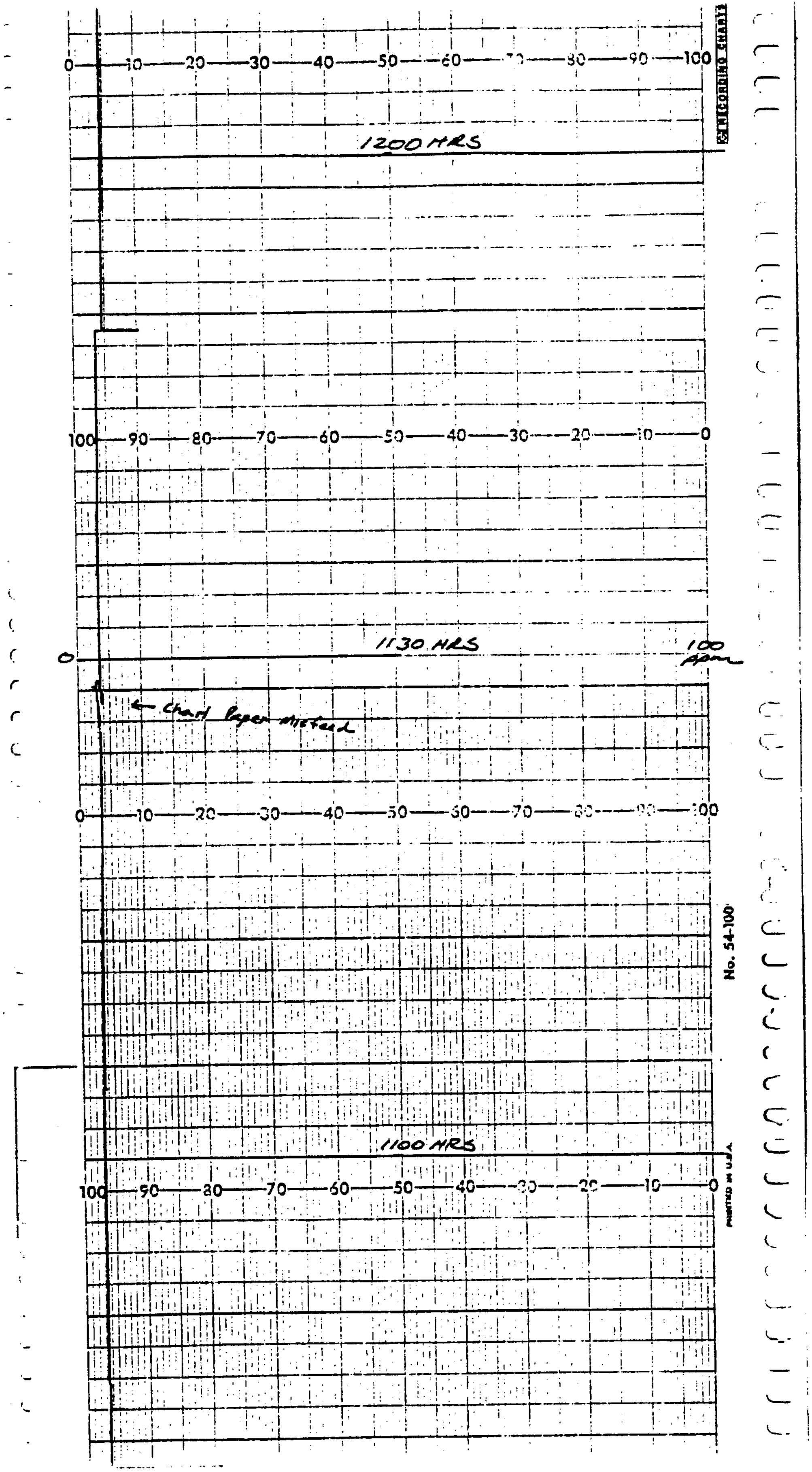
END TEST 1 1910 HRS

1900 HRS

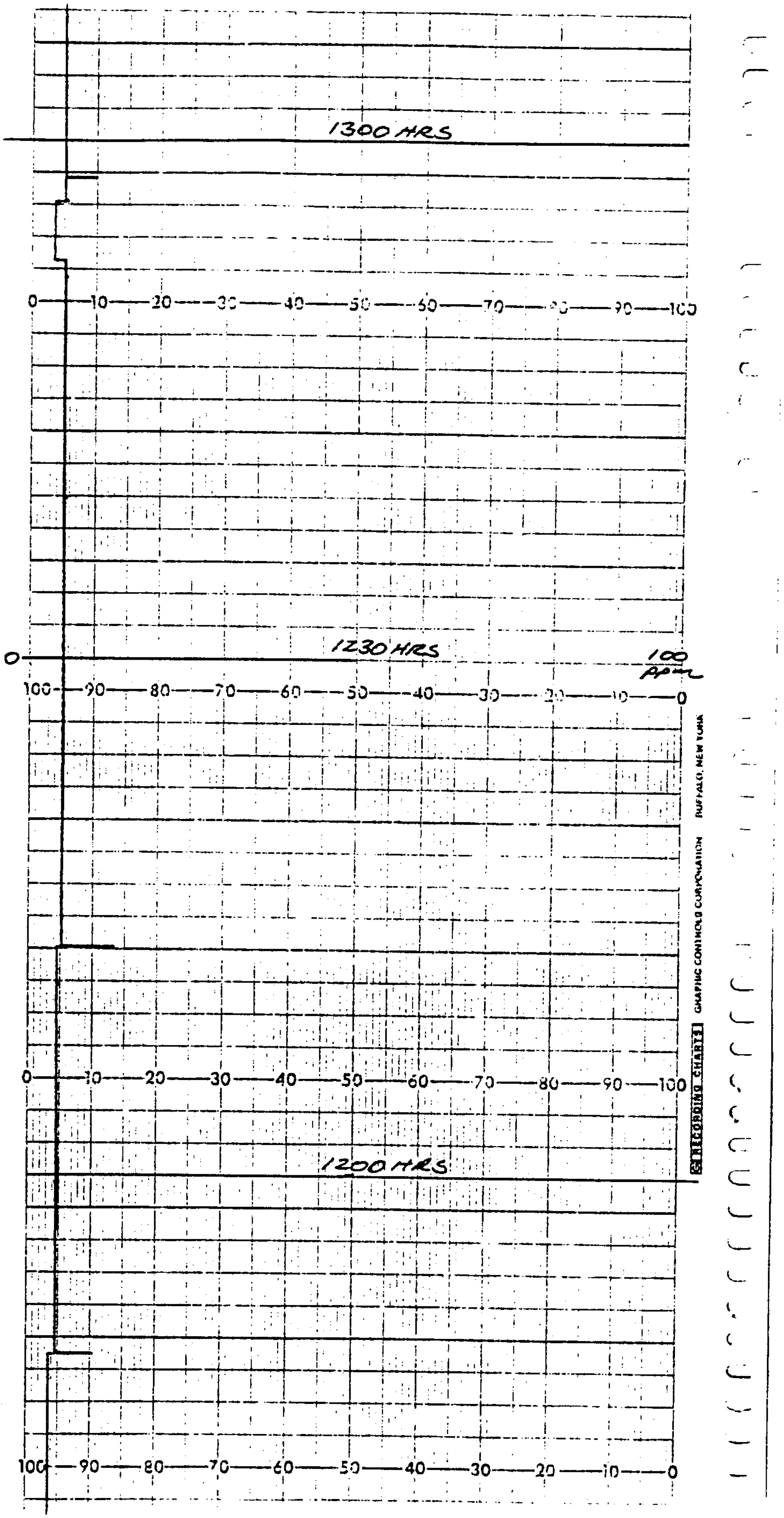
1830 HRS

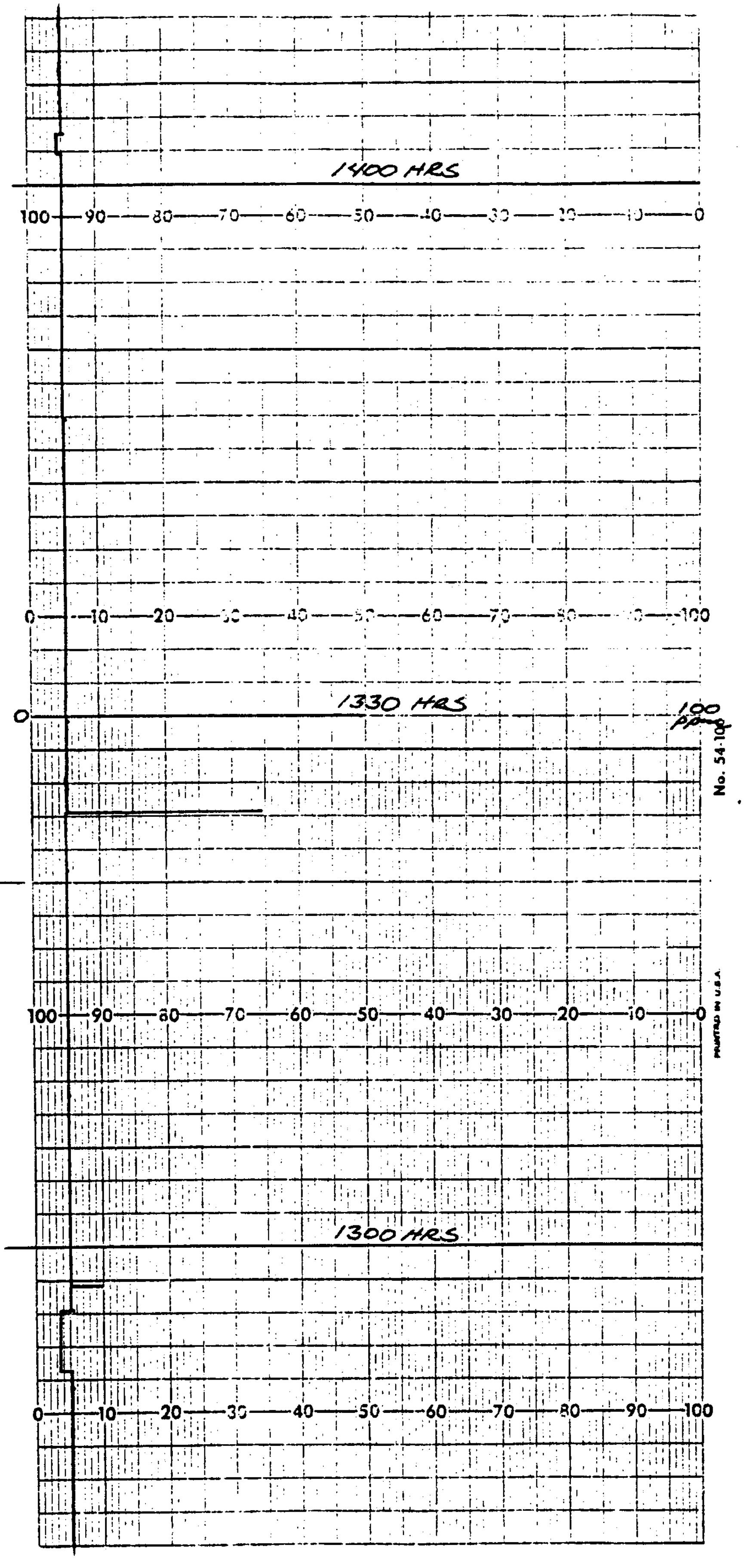

GRAPHIC CONTROLS CORPORATION
RECORDING CHARTS
1000 HRS
0—10—20—30—40—50—60—70—80—90—100
0
RADIO KEYED
0930 HRS
100 PPM
100—90—80—70—60—50—40—30—20—10—0
0—10—20—30—40—50—60—70—80—90—100
0900
0
100 PPM
No. 54100
0900 HRS START
3116 ppm
100—90—80—70—60—50—40—30—20—10—0
PRINTED IN U.S.A.

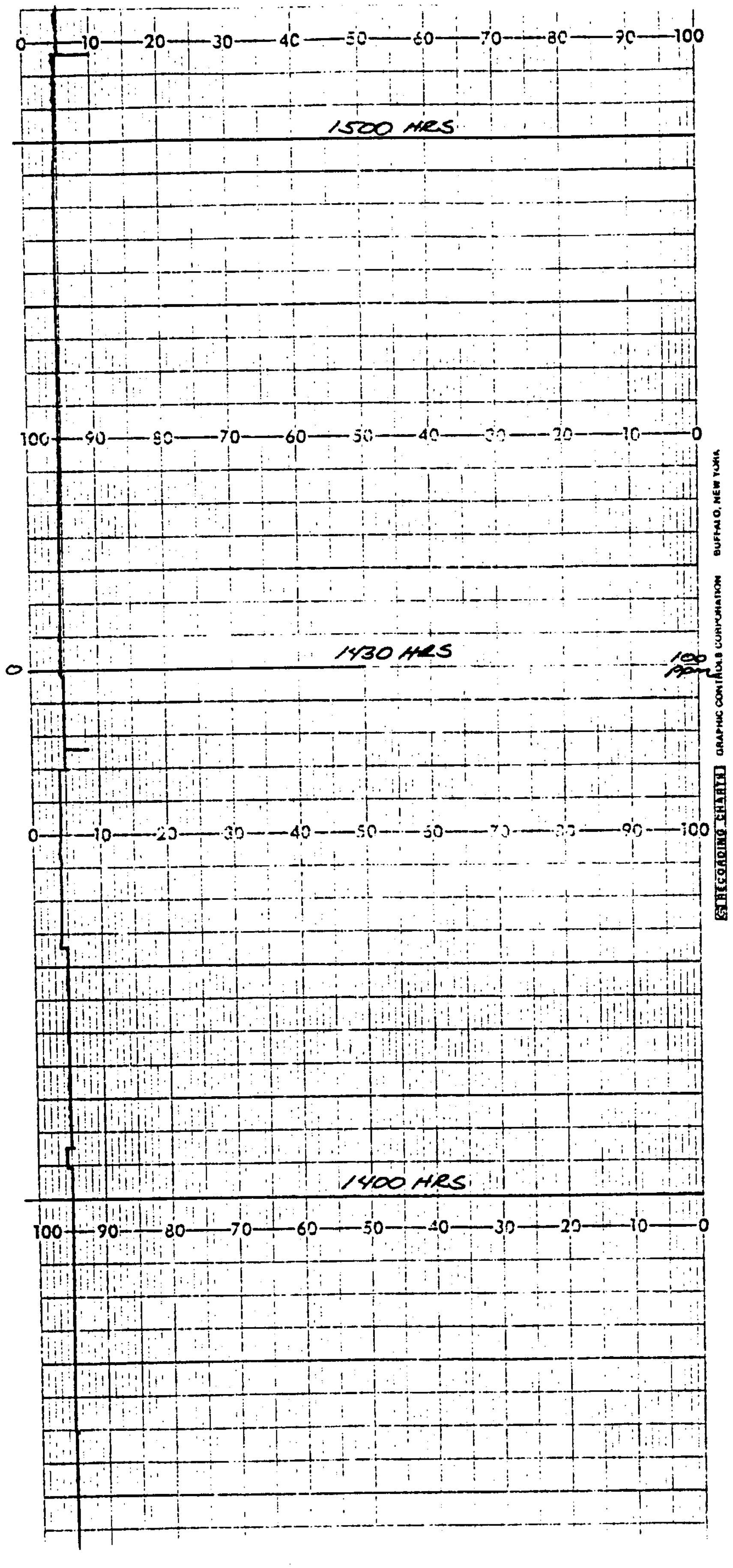

1100 HRS
100 90 80 70 60 50 40 30 20 10 0
PRINTED IN U.S.A.
0 10 20 30 40 50 60 70 80 90 100
1030 HRS
0
100 ppm
100 90 80 70 60 50 40 30 20 10 0
BUFFALO, NEW YORK
1000 HRS
GRAPHIC CONTROLS CORPORATION
GC RECORDING CHARTS
0 10 20 30 40 50 60 70 80 90 100



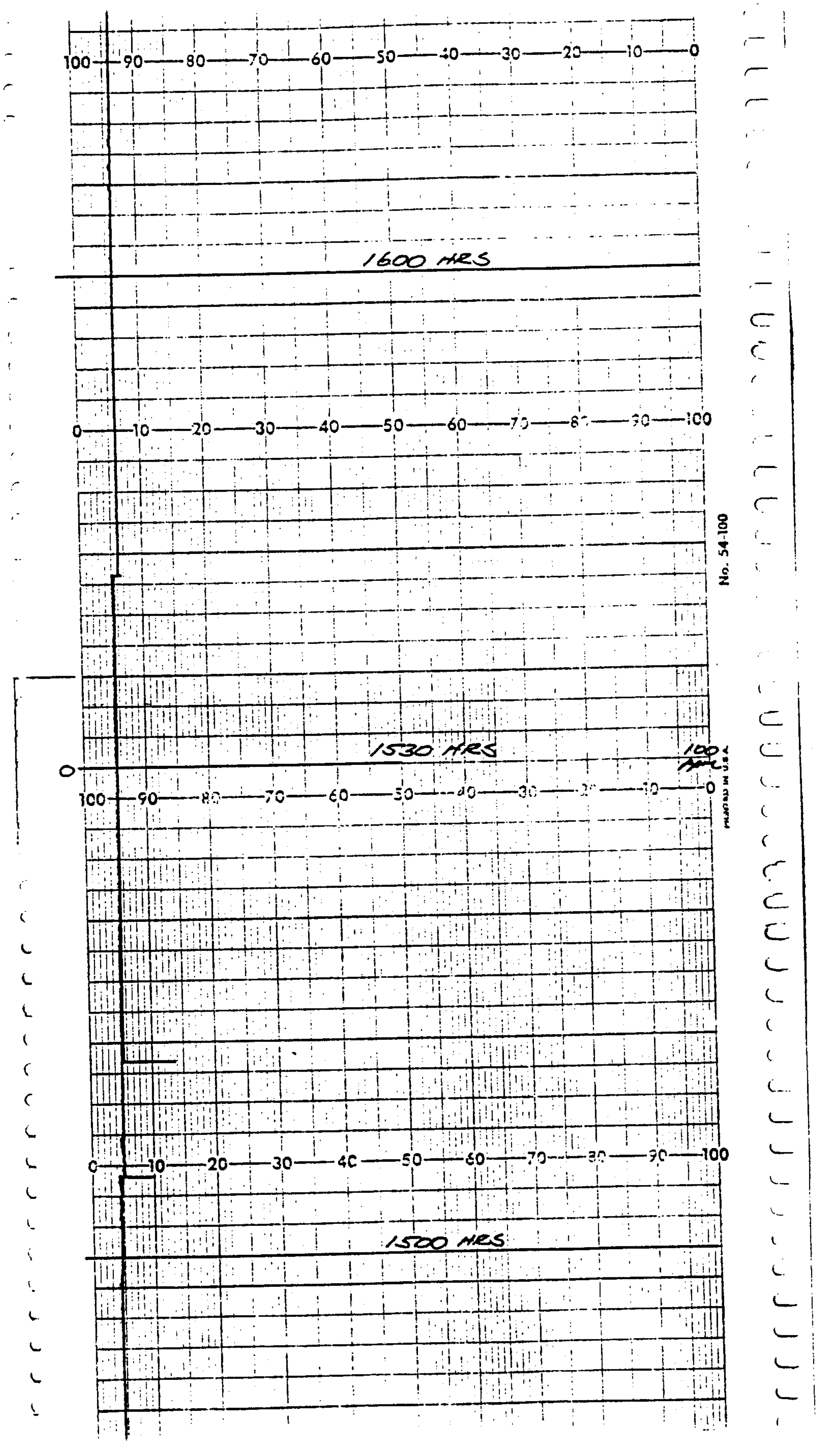
0—10—20—30—40—50—60—70—80—90—100
RECORDING CHARTS
1200 HRS
100—90—80—70—60—50—40—30—20—10—0
0
1130 HRS
100 gpm
← Chart Paper misfeed
0—10—20—30—40—50—60—70—80—90—100
No. 54-100
1100 HRS
100—90—80—70—60—50—40—30—20—10—0
PRINTED IN U.S.A.

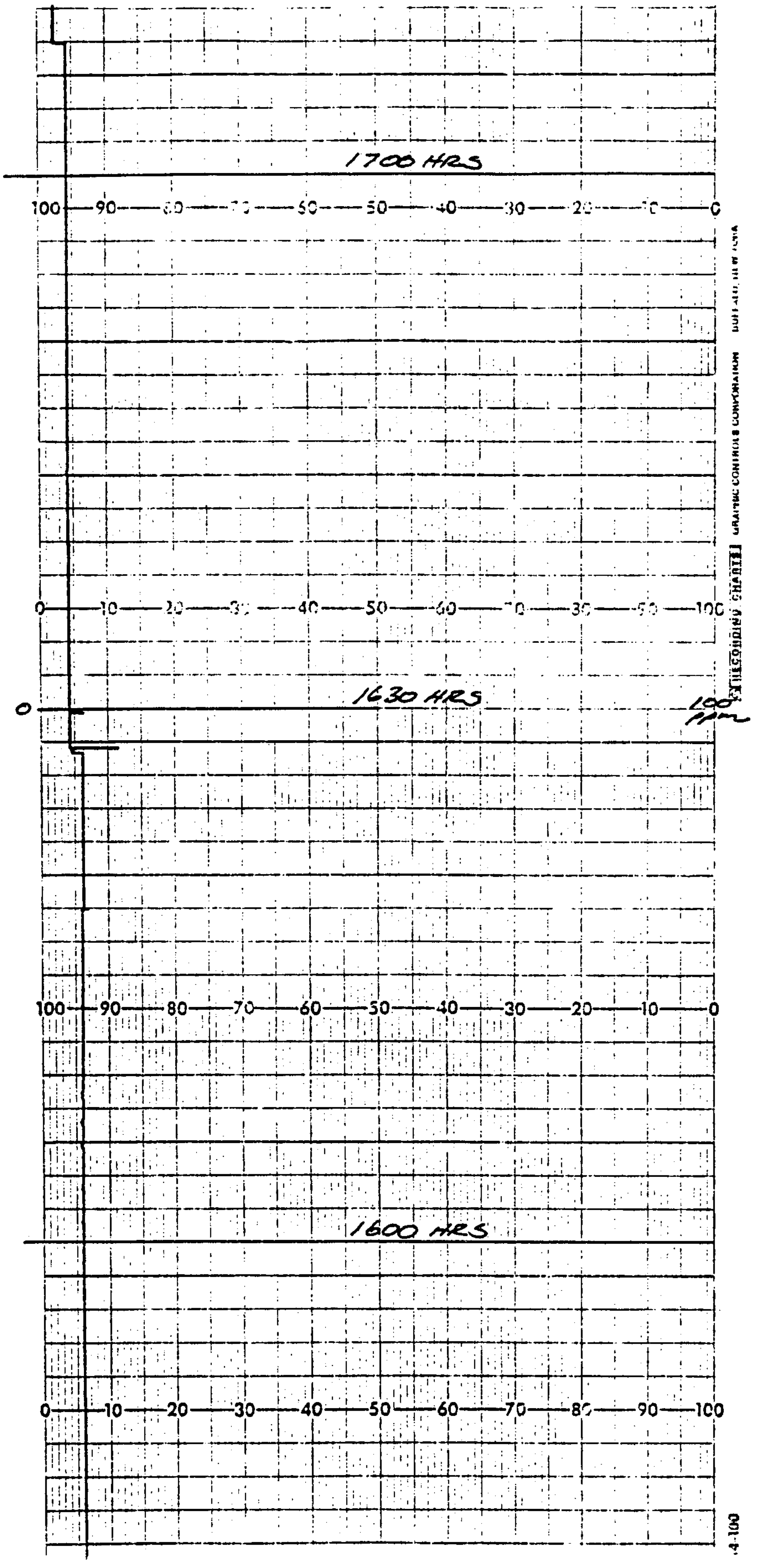

1300 HRS
0—10—20—30—40—50—60—70—80—90—100
1230 HRS
0
100 ppm
100—90—80—70—60—50—40—30—20—10—0
GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK
RECORDING CHARTS
0—10—20—30—40—50—60—70—80—90—100
1200 HRS
100—90—80—70—60—50—40—30—20—10—0

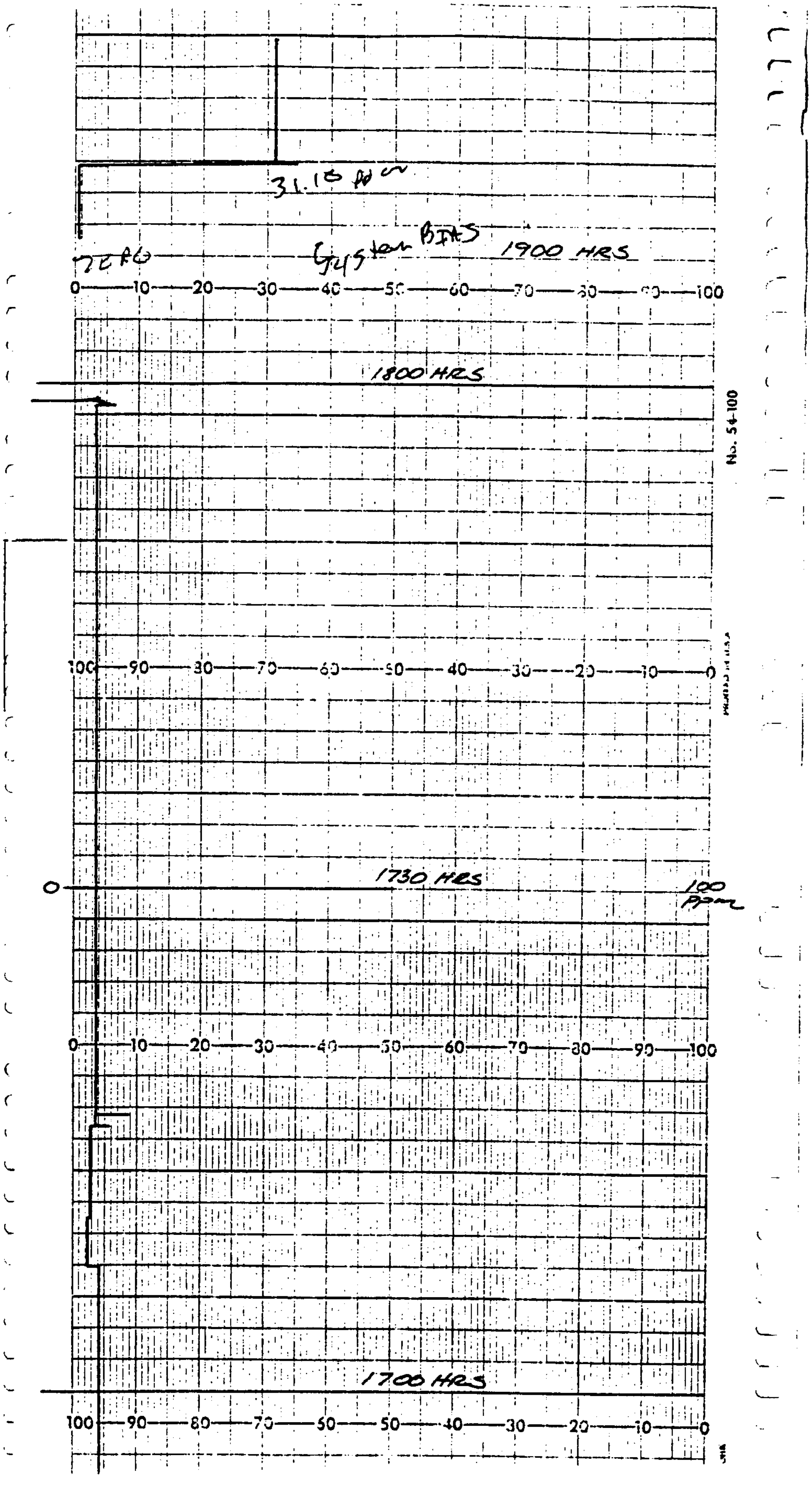

1400 HRS
100 90 80 70 60 50 40 30 20 10 0
0 10 20 30 40 50 60 70 80 90 100
1330 HRS
0
100 ppm
No. 54-100
100 90 80 70 60 50 40 30 20 10 0
PRINTED IN U.S.A.
1300 HRS
0 10 20 30 40 50 60 70 80 90 100



0 10 20 30 40 50 60 70 80 90 100
1500 HRS
100 90 80 70 60 50 40 30 20 10 0
1430 HRS
0
100 ppm
0 10 20 30 40 50 60 70 80 90 100
1400 HRS
100 90 80 70 60 50 40 30 20 10 0
RECORDING CHARTS GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK


100 90 80 70 60 50 40 30 20 10 0
1600 HRS
0 10 20 30 40 50 60 70 80 90 100
No. 54-100
1530 HRS
0
100
100 90 80 70 60 50 40 30 20 10 0
PRINTED IN U.S.A.
0 10 20 30 40 50 60 70 80 90 100
1500 HRS


1700 HRS
100 90 80 70 60 50 40 30 20 10 0
0 10 20 30 40 50 60 70 80 90 100
1630 HRS
0
100 ppm
100 90 80 70 60 50 40 30 20 10 0
1600 HRS
0 10 20 30 40 50 60 70 80 90 100
RECORDING CHARTS GRAPHIC CONTROLS CORPORATION BUFFALO, NEW YORK
4-100


31.10 ppm
ZERO
System BIAS
1900 HRS
0—10—20—30—40—50—60—70—80—90—100
1800 HRS
No. 54-100
100—90—80—70—60—50—40—30—20—10—0
0
1730 HRS
100 ppm
0—10—20—30—40—50—60—70—80—90—100
1700 HRS
100—90—80—70—60—50—40—30—20—10—0

Appendix F: Triangle Laboratories Analytical Data

TRIANGLE LABORATORIES OF RTP, INC.
801 CAPITOLA DRIVE
DURHAM, NC 27713

Phone: (919) 544-5729
Fax: (919) 544-5491

DATE : * NOVEMBER 30, 1992

CLIENT ID : * INTERPOLL LABS

P.O. NUMBER : * 16934KE

TLI PROJECT No. : * 22314

CASE NARRATIVE

MODEL 8290X

ANALYSIS OF SAMPLES FOR THE PRESENCE OF

POLYCHLORINATED DIBENZO-p-DIOXINS

AND

DIBENZOFURANS

BY

HIGH-RESOLUTION GAS CHROMATOGRAPHY / HIGH-RESOLUTION MASS SPECTROMETRY

Eight MM5 samples were received from INTERPOLL LABS in good condition November 4, 1992 and stored in a refrigerator at 4°C. The samples were extracted and analyzed according to procedures described in the Triangle Labs User Manual provided with this data package. Any particular difficulties encountered during the sample handling by Triangle Labs will be discussed in the QC remark section below.

Quality Control Samples

A laboratory method blank -- identified as the TLI MM5 Blank -- is prepared along with the samples.

QC Remarks

The release of this particular set of INTERPOLL LABS analytical data by Triangle Labs was authorized by the Quality Control Chemist who has reviewed each sample data package individually following a series of inspections/reviews. When applicable, general deviations from acceptable QC requirements are identified below. Comments on the effect of these deviations upon the validity and reliability of the results can be obtained from the User Manual (Data Quality Objectives; Section 5). Specific QC Problems Associated with this Particular Project are:

Sample Preparation Laboratory: After extraction in MeCl2 (see flowchart), 50% of the extract was sent back to Interpoll for PAH and PCB analysis.

Mass Spectrometry: None

Data Review: Concentrations reported are based on 100% of the extract (on a per train basis).

Any analytes found in the TLI Blank are detected at a level at or below the Target Detection Limit. This level of contamination is acceptable as per TLI Guidelines. OCDD is not subject to blank contamination criteria as per TLI Guidelines.

Addendum to Section 2.3 of the Data User's Manual

All sample and calibration analyses are conducted using a 2 uL injection volume, unless otherwise specified.

Addendum to Section 4.6 of the Data User's Manual

Effective December 15, 1989, Triangle Laboratories has adopted a new procedure for calculating the analyte specific detection limits. Under this new policy two representative noise height determinations are summed and multiplied by an empirically determined factor before the detection limit equation given in Section 4.6 of the Data User's Manual is applied. The effect of this procedure is to increase DB-225 analyses detection limits by a factor of 3.5, and DB-5 analyses detection limits by a factor of 5. This procedure will result in a more accurate estimate of detection limits.

The revised formula for DB-225 analyses is:

$$DL_{\delta i}^{DB\text{-}225} = \frac{2 * 2.5 * (3.5 * H_{\delta i}) * Q_{\beta j}}{A_{\beta j} * \overline{RRF} * W}$$

$$3.5 * H_{\delta i} = A_{noise}$$

The revised formula for DB-5 analyses is:

$$DL_{\delta i}^{DB\text{-}5} = \frac{2 * 2.5 * (5 * H_{\delta i}) * Q_{\beta j}}{A_{\beta j} * \overline{RRF} * W}$$

$$5 * H_{\delta i} = A_{noise}$$

The symbols are defined in Section 4.6 of the Data User's Manual.

As an example, consider a DB-225 confirmation analysis of a 10 g sample. The internal standard spike is 2000 pg, the 13C<u>12-</u> 2,3,7,8-TCDF internal standard area is 12000 area units, the RRF is 1.215 and the noise height is 11 units. The 2,3,7,8-TCDF detection limit is:

$$DL_{TCDF}^{DB\text{-}225} = \frac{2 * 2.5 * (3.5 * 11) * 2000}{12000 * 1.215 * 10.0} = 2.6 \text{ ppt}$$

Addendum to Section 3.3.1 in the Data User's Manual

A "B" flag is used to indicate that an analyte has been detected in the laboratory method blank as well as in an associated field sample. This flag denotes possible contribution of background laboratory contamination to the concentration or amount of that analyte detected in the field sample. Under Triangle Laboratories of RTP guidelines, a laboratory blank is acceptable if one of the following conditions is satisfied: 1) the tetra through hepta CDD/CDF levels are all below the target detection limits (TDLs), 2) the analyte levels found are all below 1/3 the theoretical method detection limit (TMQL), or 3) the contamination levels are less than 5% of the levels detected in the associated field samples. If these conditions are satisfied or if the blank is unable to be reextracted, the interpretation of the contamination levels relative to the samples should be as follows: 1) analyte quantitations should be considered valid if the level of blank contamination is less than five percent of the level detected in the field sample, 2) analyte quantitations should be considered estimated if the analyte level in the sample is five to twenty times the level of the analyte in the blank, or 3) analytes whose level in a sample is the same as or less than five times the level detected in the associated blank should be considered present likely due to laboratory contamination and not native to the sample.

A "Q" flag is used to indicate the presence of QC ion instabilities caused by quantitative interferences. Affected analytes may be overestimated or underestimated as a result of this interference. A peak is flagged "Q" only if it is affected by a QC ion deviation greater than 20% full scale as determined relative to the labeled standard against which it is quantitated. Total PCDD/PCDF quantitations will be flagged "Q" if the interferences affect ten percent or more of the total PCDD/PCDF peak areas.

A "U" flag is used to indicate that a specific isomer cannot be resolved from a large, coeluting interferent GC peak. The specific isomer is reported as not detected as a valid concentration/amount cannot be determined. The calculated detection limit, therefore, should be considered an underestimated value.

A "V" flag is used to indicate that, although the percent recovery of a labeled standard may be below a specific QC limit, the signal-to-noise ratio of the peak is greater than 10:1. The standard is considered reliably quantifiable. All quantitations derived from the standard are considered valid as well.

A "PR" flag is used to indicate that a GC peak is poorly resolved. This resolution problem may be seen as two closely eluting peaks without a reasonable valley between the peak tops, overly broad peaks, or peaks whose shapes vary greatly from a normal distribution. The concentrations or amounts reported for such peaks are most likely overestimated.

An "I" flag is used to indicate labeled standards have been interfered with on the GC column by coeluting, interferent peaks. The interference may have caused the standard's area to be overestimated. All quantitations relative to this standard, therefore, may be underestimated.

An "E" flag is used to indicate that a PCDF peak has eluted at the same time as the associated diphenyl ether (DPE) and that the DPE peak intensity is ten percent or more of the PCDF peak intensity. Total PCDF values are flagged "E" if the total DPE contribution to the total PCDF value is greater than ten percent. All PCDF peaks that are significantly influenced by the presence of DPE peaks are quantitated with EMPC values, regardless of the isotopic abundance ratio. These EMPC values are most likely overestimated due to the DPE contribution to the peak area.

An "RO" flag is used to indicate that a labeled standard has an ion-abundance ratio that is outside of the acceptable QC limits, most likely due to a coeluting interference. This may have caused the percent recovery of the standard to be overestimated. All quantitations versus this standard, therefore, may be underestimated.

Should INTERPOLL LABS have any questions or comments regarding this data package please feel free to contact us.

For Triangle Laboratories of RTP, Inc.,

Report Preparation

Deborah E Hage

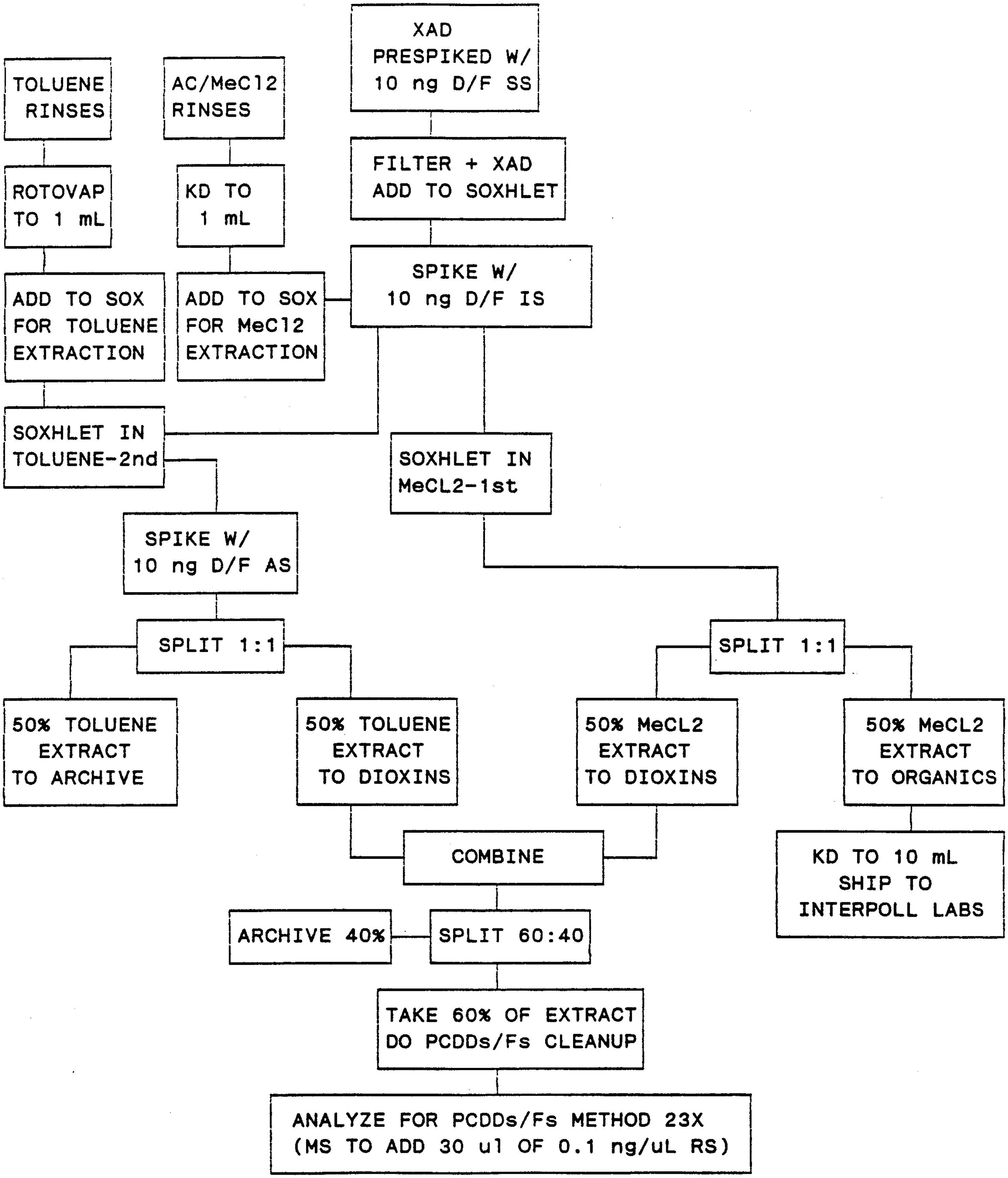
Deborah E. Hage
Report Preparation Chemist

Quality Control

Sheila C. Lee

Report Preparation Chemist

CHART #:M23-INT
CLIENT : INTERPOLL
TLI #'s: 22314
NO IMPINGER; TOLUENE RINSES COMBINED
FLOW CHART FOR :PCDDs/PCDFs BY TLI, AND PAHs/PCBs BY INTERPOLL LABS



TLI rev-hk1-NOV 92

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

Page 1 of 2
11/30/92

LE NAME.: S925324
ROJECT...: 22314
IENT PROJECT: n/a
I ID....: TLI BLANK
LLECTED.: n/a
CEIVED..: n/a
TRIX....: MM5UNITS
T. SIZE.: 1.000
J. SIZE.: 1.000
T. DATE.: 11/12/92
T. VOL..: 30.00 ul
IKE FILE: SPX23710
JECT VOL: 2.0 ul

SAMPLE ID.: TLI MM5 BLANK
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 11/24/92
ANALYSIS TIME.: 04;05
ANALYST.......: SA
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2493345
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925323
CONCAL DATE...: 11/24/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925324
% LIPID......: n/a
% SOLIDS.....: n/a
% MOISTURE...: n/a
ORIGIN.......: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.008				
378-PeCDD	ND		0.01				
3478-HxCDD	ND		0.01				
3678-HxCDD	ND		0.008				
3789-HxCDD	ND		0.01				
34678-HpCDD	0.05				0.98	36:17	
D	0.51				0.77	39:50	
78-TCDF	EMPC			0.009			
378-PeCDF	ND		0.008				
478-PeCDF	ND		0.005				
3478-HxCDF	ND		0.008				
3678-HxCDF	ND		0.005				
4678-HxCDF	EMPC			0.02			
3789-HxCDF	ND		0.01				
34678-HpCDF	ND		0.008				
34789-HpCDF	ND		0.01				
F	ND		0.02				
TAL TCDD	ND		0.008				
TAL PeCDD	ND		0.01				
TAL HxCDD	ND		0.01				
TAL HpCDD	0.07	2			1.01		
TAL TCDF	EMPC			0.009			
TAL PeCDF	ND		0.008				
TAL HxCDF	EMPC			0.02			Q
TAL HpCDF	ND		0.01				

viewed By: | DH 11/30/92|

X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

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11/30/92

FILE NAME.:	S925324	SAMPLE ID.:	TLI MM5 BLANK		
PROJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
CLIENT PROJECT:	n/a				
TLI ID....:	TLI BLANK	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
COLLECTED.:	n/a	ANALYSIS TIME.:	04;05	DILUTION.....:	n/a
RECEIVED..:	n/a	ANALYST.......:	SA	BLANK FILE...:	S925324
MATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
ADJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
EXT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	n/a
EXT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

URROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	9.3	93.3		25:37	___
13C12-PeCDF 234	10.5	105	1.49	29:33	___
13C12-HxCDF 478	10.0	100	0.50	32:19	___
13C12-HxCDD 478	10.1	101	1.18	33:00	___
13C12-HpCDF 789	11.0	110	0.43	36:46	___

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	10.5	105	0.49	33:38	___
13C12-HxCDF 234	9.5	95.2	0.50	32:54	Q

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	9.3	92.9	0.76	24:52	___
13C12-2378-TCDD	8.6	85.7	0.78	25:36	___
13C12-PeCDF 123	9.8	98.4	1.48	28:52	___
13C12-PeCDD 123	9.0	89.6	1.50	29:55	Q
13C12-HxCDF 678	9.8	98.4	0.50	32:25	___
13C12-HxCDD 678	11.0	110	1.20	33:05	___
13C12-HpCDF 678	8.2	81.6	0.45	35:16	___
13C12-HpCDD 678	10.0	99.6	0.99	36:17	___
13C12-OCDD	18.0	89.8	0.86	39:50	___

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.78	25:25	___
13C12-HxCDD 789	1.20	33:24	___

Reviewed By: DH 11/30/92

X237_RPT 4.01, LARS 5.

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

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11/30/92

LE NAME.:	S925325	SAMPLE ID.:	TEST 2/0-F.B.-7328		
OJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
IENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
I ID....:	61-11-1A-C	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
LLECTED.:	n/a	ANALYSIS TIME.:	04:52	DILUTION.....:	n/a
CEIVED..:	11/04/92	ANALYST.......:	ML	BLANK FILE...:	S925324
TRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
T. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
J. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
T. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
T. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
IKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
JECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

E	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
3-TCDD	ND		0.04				
78-PeCDD	ND		0.05				
478-HxCDD	ND		0.08				
578-HxCDD	ND		0.05				
789-HxCDD	ND		0.07				
4678-HpCDD	ND		0.1				
D	ND		0.3				
3-TCDF	ND		0.03				
78-PeCDF	ND		0.04				
78-PeCDF	ND		0.03				
478-HxCDF	ND		0.05				
578-HxCDF	ND		0.03				
578-HxCDF	ND		0.04				
789-HxCDF	ND		0.06				
4678-HpCDF	ND		0.05				
4789-HpCDF	ND		0.08				
F	EMPC			0.97			
AL TCDD	ND		0.04				
AL PeCDD	ND		0.05				
AL HxCDD	ND		0.06				
AL HpCDD	ND		0.1				
AL TCDF	ND		0.03				
AL PeCDF	ND		0.04				
AL HxCDF	ND		0.04				
AL HpCDF	ND		0.06				

iewed By: DH 11/30/92

X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

FILE NAME.: S925325 SAMPLE ID.: TEST 2/0-F.B.-7328
PROJECT...: 22314 CLIENT NAME...: INTERPOLL LABS
CLIENT PROJECT: ANL-OTP/BIG STONE, S.D.

TLI ID....:	61-11-1A-C	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
COLLECTED.:	n/a	ANALYSIS TIME.:	04:52	DILUTION.....:	n/a
RECEIVED..:	11/04/92	ANALYST.......:	ML	BLANK FILE...:	S925324
MATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
ADJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
EXT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
EXT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	9.1	90.7		25:38	
13C12-PeCDF 234	9.3	93.0	1.41	29:33	
13C12-HxCDF 478	9.5	95.3	0.49	32:19	
13C12-HxCDD 478	9.7	96.9	1.17	33:00	
13C12-HpCDF 789	7.6	75.7	0.43	36:46	

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	6.6	66.1	0.56	33:39	
13C12-HxCDF 234	8.5	85.3	0.50	32:54	

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	4.7	46.8	0.75	24:54	
13C12-2378-TCDD	4.3	42.7	0.76	25:37	
13C12-PeCDF 123	4.5	44.6	1.37	28:52	
13C12-PeCDD 123	5.1	50.7	1.49	29:53	
13C12-HxCDF 678	11.1	111	0.51	32:25	
13C12-HxCDD 678	11.4	114	1.15	33:05	
13C12-HpCDF 678	8.4	83.6	0.45	35:16	
13C12-HpCDD 678	6.5	64.6	1.02	36:17	
13C12-OCDD	7.0	35.0	0.76	39:50	

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.80	25:25	
13C12-HxCDD 789	1.14	33:24	

Reviewed By: DH 11/30/92 X237_RPT 4.01, LARS 5.0

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

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11/30/92

ILE NAME.:	S925326	SAMPLE ID.:	TEST 2/1-STACK-7328		
ROJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
LIENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
LI ID....:	61-11-2A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
OLLECTED.:	n/a	ANALYSIS TIME.:	05:40	DILUTION.....:	n/a
ECEIVED..:	11/04/92	ANALYST.......:	SA	BLANK FILE...:	S925324
ATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
XT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
DJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
XT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
XT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
PIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
NJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.03				
378-PeCDD	ND		0.03				
3478-HxCDD	ND		0.04				
3678-HxCDD	ND		0.03				
3789-HxCDD	ND		0.03				
34678-HpCDD	0.07				0.92	36:16	B
DD	EMPC			0.52			B
78-TCDF	ND		0.02				
378-PeCDF	ND		0.02				
478-PeCDF	ND		0.02				
3478-HxCDF	ND		0.02				
3678-HxCDF	ND		0.02				
4678-HxCDF	ND		0.02				
3789-HxCDF	ND		0.03				
34678-HpCDF	ND		0.02				
34789-HpCDF	ND		0.04				
DF	ND		0.05				
TAL TCDD	ND		0.03				
TAL PeCDD	ND		0.03				
TAL HxCDD	ND		0.03				
TAL HpCDD	0.07	1			0.92		
TAL TCDF	ND		0.02				
TAL PeCDF	ND		0.02				
TAL HxCDF	ND		0.02				
TAL HpCDF	ND		0.03				

eviewed By: | 11/30/92

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TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

FILE NAME.:	S925326	SAMPLE ID.:	TEST 2/1-STACK-7328		
PROJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
CLIENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
TLI ID....:	61-11-2A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
COLLECTED.:	n/a	ANALYSIS TIME.:	05:40	DILUTION.....:	n/a
RECEIVED..:	11/04/92	ANALYST.......:	SA	BLANK FILE...:	S925324
MATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
ADJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
EXT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
EXT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	9.0	89.7		25:37	___
13C12-PeCDF 234	11.1	111	1.54	29:33	___
13C12-HxCDF 478	10.2	102	0.50	32:18	___
13C12-HxCDD 478	10.5	105	1.16	33:00	___
13C12-HpCDF 789	11.3	113	0.41	36:45	___

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	9.6	95.8	0.49	33:38	___
13C12-HxCDF 234	9.1	91.0	0.50	32:53	___

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	7.5	75.2	0.81	24:52	___
13C12-2378-TCDD	7.2	72.1	0.77	25:36	___
13C12-PeCDF 123	7.5	74.9	1.54	28:51	___
13C12-PeCDD 123	10.2	102	1.51	29:53	___
13C12-HxCDF 678	8.6	85.8	0.50	32:25	___
13C12-HxCDD 678	9.5	95.1	1.17	33:04	___
13C12-HpCDF 678	7.8	77.7	0.44	35:15	___
13C12-HpCDD 678	9.0	89.5	1.01	36:16	___
13C12-OCDD	14.4	71.8	0.84	39:49	___

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.78	25:24	___
13C12-HxCDD 789	1.16	33:23	___

Reviewed By: | DH 11/30/92

X237_RPT 4.01, LARS 5.

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

ILE NAME.: S925327 SAMPLE ID.: TEST 2/2-STACK-7328
ROJECT...: 22314 CLIENT NAME...: INTERPOLL LABS
LIENT PROJECT: ANL-OTP/BIG STONE, S.D.

LI ID....:	61-11-3A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
OLLECTED.:	n/a	ANALYSIS TIME.:	06:33	DILUTION.....:	n/a
ECEIVED..:	11/04/92	ANALYST.......:	SA	BLANK FILE...:	S925324
ATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
XT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
DJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
XT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
XT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
PIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
NJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.008				
378-PeCDD	ND		0.008				
3478-HxCDD	ND		0.01				
3678-HxCDD	ND		0.005				
3789-HxCDD	ND		0.008				
34678-HpCDD	EMPC			0.04			B
DD	0.41				0.84	39:50	B
78-TCDF	0.02				0.79	24:54	B
378-PeCDF	ND		0.005				
478-PeCDF	ND		0.005				
3478-HxCDF	ND		0.005				
3678-HxCDF	ND		0.005				
4678-HxCDF	0.02				1.15	32:54	B
3789-HxCDF	ND		0.008				
34678-HpCDF	EMPC			0.007			
34789-HpCDF	ND		0.008				
DF	0.02				0.93	40:02	
TAL TCDD	ND		0.008				
TAL PeCDD	ND		0.008				
TAL HxCDD	ND		0.008				
TAL HpCDD	0.03	1		0.06	1.02		
TAL TCDF	0.02	1		0.03	0.79		
TAL PeCDF	0.01	1			1.70		
TAL HxCDF	0.02	1			1.15		
TAL HpCDF	EMPC			0.009			

eviewed By: | 11/30/92

X237_RPT 4.01, LARS 5.00

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                    TRIANGLE LABORATORIES OF RTP, INC.              Page 2 of 2
                PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY            11/30/92


 FILE NAME.: S925327      SAMPLE ID.: TEST 2/2-STACK-7328
 PROJECT...: 22314        CLIENT NAME...: INTERPOLL LABS
 CLIENT PROJECT: ANL-OTP/BIG STONE, S.D.
 TLI ID....: 61-11-3A-D   ANALYSIS DATE.: 11/24/92  CLIENT CODE..: IPL01
 COLLECTED.: n/a          ANALYSIS TIME.: 06:33     DILUTION.....: n/a
 RECEIVED..: 11/04/92     ANALYST.......: SA        BLANK FILE...: S925324
 MATRIX....: MM5UNITS     INSTRUMENT....: S         % LIPID......: n/a
 EXT. SIZE.: 1.000        GC COLUMN.....: DB-5      % SOLIDS.....: n/a
 ADJ. SIZE.: 1.000        GC COLUMN ID..: 2493345   % MOISTURE...: n/a
 EXT. DATE.: 11/12/92     ICAL NAME.....: SC50072   ORIGIN.......: SEE COC
 EXT. VOL..: 30.00 ul     ICAL DATE.....: 10/07/92  CONTRACT.....: n/a
 SPIKE FILE: SPX23710     CONCAL NAME...: S925323   SAS NUMBER...: n/a
 INJECT VOL: 2.0 ul       CONCAL DATE...: 11/24/92  EPISODE......: n/a
================================================================================

SURROGATE RECOVERY SUMMARY (TYPE AA)
==============================================================================
NAME                 AMT (ng )          % REC.     RATIO          RT       FLAGS
------------------------------------------------------------------------------
37Cl-TCDD               8.9              89.3                    25:36      ___
13C12-PeCDF 234        11.2             112          1.51        29:33      ___
13C12-HxCDF 478        10.1             101          0.50        32:19      ___
13C12-HxCDD 478        11.1             111          1.18        33:00      ___
13C12-HpCDF 789        12.1             121          0.42        36:45      ___

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)
==============================================================================
NAME                 AMT (ng )          % REC.     RATIO          RT       FLAGS
------------------------------------------------------------------------------
13C12-HxCDF 789         8.9              88.5        0.51        33:38      ___
13C12-HxCDF 234         8.6              85.6        0.50        32:54      ___

INTERNAL STANDARDS RECOVERY SUMMARY
==============================================================================
NAME                 AMT (ng )          % REC.     RATIO          RT       FLAGS
------------------------------------------------------------------------------
13C12-2378-TCDF         7.3              72.6        0.80        24:51      ___
13C12-2378-TCDD         7.0              70.1        0.79        25:36      ___
13C12-PeCDF 123         7.7              77.0        1.46        28:51      ___
13C12-PeCDD 123        11.1             111          1.53        29:53      ___
13C12-HxCDF 678         7.8              77.8        0.50        32:25      ___
13C12-HxCDD 678         8.7              87.4        1.17        33:05      ___
13C12-HpCDF 678         7.6              75.6        0.44        35:15      ___
13C12-HpCDD 678         9.3              92.5        0.98        36:16      ___
13C12-OCDD             16.8              83.9        0.84        39:49      ___

RECOVERY STANDARDS RECOVERY SUMMARY
==============================================================================
NAME                                               RATIO          RT       FLAGS
------------------------------------------------------------------------------
13C12-1234-TCDD                                      0.78        25:24      ___
13C12-HxCDD 789                                      1.19        33:23      ___
--------------------------------------------------------------------------------
Reviewed By: |_____DH_____   11/30/92                 X237_RPT 4.01, LARS 5.0
```

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

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ILE NAME.:	S925328	SAMPLE ID.:	TEST 2/3-STACK-7328		
ROJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
LIENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
LI ID....:	61-11-4A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
OLLECTED.:	n/a	ANALYSIS TIME.:	07:24	DILUTION.....:	n/a
ECEIVED..:	11/04/92	ANALYST........:	SA	BLANK FILE...:	S925324
ATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
XT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
DJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
XT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
XT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
PIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
NJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.03				
378-PeCDD	ND		0.03				
3478-HxCDD	ND		0.04				
3678-HxCDD	ND		0.03				
3789-HxCDD	ND		0.03				
34678-HpCDD	EMPC			0.06			B
DD	0.66				0.77	39:51	B
78-TCDF	ND		0.02				
378-PeCDF	ND		0.02				
478-PeCDF	ND		0.02				
3478-HxCDF	ND		0.03				
3678-HxCDF	ND		0.02				
4678-HxCDF	EMPC			0.03			B
3789-HxCDF	ND		0.03				
34678-HpCDF	ND		0.02				
34789-HpCDF	ND		0.04				
DF	ND		0.05				
TAL TCDD	ND		0.03				
TAL PeCDD	ND		0.03				
TAL HxCDD	ND		0.03				
TAL HpCDD	EMPC			0.11			
TAL TCDF	ND		0.02				
TAL PeCDF	ND		0.02				
TAL HxCDF	EMPC			0.03			
TAL HpCDF	ND		0.03				

eviewed By: DH 11/30/92

X237_RPT 4.01, LARS 5.00

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TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

FILE NAME.:	S925328	SAMPLE ID.:	TEST 2/3-STACK-7328		
PROJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
CLIENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
TLI ID....:	61-11-4A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
COLLECTED.:	n/a	ANALYSIS TIME.:	07:24	DILUTION.....:	n/a
RECEIVED..:	11/04/92	ANALYST.......:	SA	BLANK FILE...:	S925324
MATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
ADJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
EXT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
EXT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	8.9	88.9		25:37	
13C12-PeCDF 234	10.9	109	1.46	29:33	
13C12-HxCDF 478	10.7	107	0.51	32:19	
13C12-HxCDD 478	10.1	101	1.19	33:00	
13C12-HpCDF 789	11.1	111	0.45	36:46	

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	9.2	92.2	0.50	33:38	
13C12-HxCDF 234	9.2	92.1	0.50	32:54	

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	6.4	63.8	0.75	24:52	
13C12-2378-TCDD	6.3	63.1	0.81	25:36	
13C12-PeCDF 123	6.5	65.1	1.46	28:52	
13C12-PeCDD 123	8.5	84.6	1.48	29:53	
13C12-HxCDF 678	6.9	69.3	0.52	32:25	
13C12-HxCDD 678	8.4	83.6	1.16	33:05	
13C12-HpCDF 678	7.1	70.7	0.43	35:16	
13C12-HpCDD 678	7.7	77.4	1.01	36:16	
13C12-OCDD	12.8	64.2	0.83	39:50	

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.81	25:25	
13C12-HxCDD 789	1.18	33:23	

Reviewed By: | 11/30/92

X237_RPT 4.01, LARS 5.

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

```
ILE NAME.: S925329       SAMPLE ID.: TEST 7/0-STACK-7328
ROJECT...: 22314         CLIENT NAME...: INTERPOLL LABS
LIENT PROJECT: ANL-OTP/BIG STONE, S.D.
LI ID....: 61-11-5A-C    ANALYSIS DATE.: 11/24/92   CLIENT CODE..: IPL01
OLLECTED.: n/a           ANALYSIS TIME.: 08:08      DILUTION.....: n/a
ECEIVED..: 11/04/92      ANALYST.......: SA         BLANK FILE...: S925324
ATRIX....: MM5UNITS      INSTRUMENT....: S          % LIPID......: n/a
XT. SIZE.: 1.000         GC COLUMN.....: DB-5       % SOLIDS.....: n/a
DJ. SIZE.: 1.000         GC COLUMN ID..: 2493345    % MOISTURE...: n/a
XT. DATE.: 11/12/92      ICAL NAME.....: SC50072    ORIGIN.......: SEE COC
XT. VOL..: 30.00 ul      ICAL DATE.....: 10/07/92   CONTRACT.....: n/a
PIKE FILE: SPX23710      CONCAL NAME...: S925323    SAS NUMBER...: n/a
NJECT VOL: 2.0 ul        CONCAL DATE...: 11/24/92   EPISODE......: n/a
```

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.04				—
378-PeCDD	ND		0.04				—
3478-HxCDD	ND		0.05				—
3678-HxCDD	ND		0.03				—
3789-HxCDD	ND		0.04				—
34678-HpCDD	ND		0.05				—
DD	EMPC			0.61			B
78-TCDF	ND		0.02				—
378-PeCDF	ND		0.03				—
478-PeCDF	ND		0.03				—
3478-HxCDF	ND		0.03				—
3678-HxCDF	ND		0.03				—
4678-HxCDF	ND		0.03				—
3789-HxCDF	ND		0.04				—
34678-HpCDF	ND		0.03				—
34789-HpCDF	ND		0.05				—
DF	ND		0.07				—
TAL TCDD	ND		0.04				—
TAL PeCDD	ND		0.04				—
TAL HxCDD	ND		0.04				—
TAL HpCDD	ND		0.05				—
TAL TCDF	ND		0.02				—
TAL PeCDF	ND		0.03				—
TAL HxCDF	ND		0.03				—
TAL HpCDF	ND		0.04				—

eviewed By: | DH 11/30/92 X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

FILE NAME.: S925329
PROJECT...: 22314
CLIENT PROJECT: ANL-OTP/BIG STONE, S.D.
TLI ID....: 61-11-5A-C
COLLECTED.: n/a
RECEIVED..: 11/04/92
MATRIX....: MM5UNITS
EXT. SIZE.: 1.000
ADJ. SIZE.: 1.000
EXT. DATE.: 11/12/92
EXT. VOL..: 30.00 ul
SPIKE FILE: SPX23710
INJECT VOL: 2.0 ul

SAMPLE ID.: TEST 7/0-STACK-7328
CLIENT NAME...: INTERPOLL LABS
ANALYSIS DATE.: 11/24/92
ANALYSIS TIME.: 08:08
ANALYST.......: SA
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2493345
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925323
CONCAL DATE...: 11/24/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925324
% LIPID......: n/a
% SOLIDS.....: n/a
% MOISTURE...: n/a
ORIGIN.......: SEE COC
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	8.7	86.7		25:36	___
13C12-PeCDF 234	10.1	101	1.50	29:33	___
13C12-HxCDF 478	10.1	101	0.50	32:18	___
13C12-HxCDD 478	10.1	101	1.19	33:00	___
13C12-HpCDF 789	11.0	110	0.44	36:45	___

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	8.0	80.5	0.49	33:38	___
13C12-HxCDF 234	7.8	78.3	0.49	32:53	___

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	4.7	47.3	0.76	24:51	___
13C12-2378-TCDD	4.6	46.4	0.77	25:35	___
13C12-PeCDF 123	5.3	52.6	1.48	28:51	___
13C12-PeCDD 123	6.4	63.6	1.50	29:52	___
13C12-HxCDF 678	5.9	59.3	0.50	32:24	___
13C12-HxCDD 678	7.0	69.8	1.19	33:04	___
13C12-HpCDF 678	5.7	57.3	0.42	35:15	___
13C12-HpCDD 678	6.4	64.3	0.98	36:15	___
13C12-OCDD	10.3	51.4	0.84	39:48	___

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.81	25:24	___
13C12-HxCDD 789	1.16	33:23	___

Reviewed By: | DH 11/30/92 |

X237_RPT 4.01, LARS 5.0

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

LE NAME.:	S925330	SAMPLE ID.:	TEST 7/1-STACK-7328		
OJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
IENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
I ID....:	61-11-6A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
LLECTED.:	n/a	ANALYSIS TIME.:	08:59	DILUTION.....:	n/a
CEIVED..:	11/04/92	ANALYST.......:	SA	BLANK FILE...:	S925324
TRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
T. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
J. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
T. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
T. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
IKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
JECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

E	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.01				
78-PeCDD	ND		0.01				
478-HxCDD	ND		0.02				
678-HxCDD	ND		0.01				
789-HxCDD	ND		0.02				
4678-HpCDD	0.05				1.04	36:16	B
D	0.58				0.81	39:49	B
78-TCDF	EMPC			0.01			B
378-PeCDF	ND		0.01				
78-PeCDF	ND		0.01				
3478-HxCDF	0.02				1.05	32:18	
3678-HxCDF	ND		0.01				
678-HxCDF	EMPC			0.02			B
3789-HxCDF	ND		0.02				
34678-HpCDF	0.03				0.99	35:15	
34789-HpCDF	ND		0.02				
F	0.07				0.86	40:02	
TAL TCDD	ND		0.01				
TAL PeCDD	ND		0.01				
TAL HxCDD	EMPC			0.02			
TAL HpCDD	0.05	1		0.10	1.04		
TAL TCDF	EMPC			0.01			
TAL PeCDF	ND		0.01				
TAL HxCDF	0.02	1		0.05	1.05		
TAL HpCDF	0.04	1		0.05	0.99		

viewed By: | Dr 11/30/92|

X237_RPT 4.01, LARS 5.00

FILE NAME.:	S925330	SAMPLE ID.:	TEST 7/1-STACK-7328		
PROJECT...:	22314	CLIENT NAME...:	INTERPOLL LABS		
CLIENT PROJECT:	ANL-OTP/BIG STONE, S.D.				
TLI ID....:	61-11-6A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
COLLECTED.:	n/a	ANALYSIS TIME.:	08:59	DILUTION.....:	n/a
RECEIVED..:	11/04/92	ANALYST.......:	SA	BLANK FILE...:	S925324
MATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
ADJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
EXT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
EXT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	9.1	90.7		25:37	
13C12-PeCDF 234	10.5	105	1.48	29:33	
13C12-HxCDF 478	10.5	105	0.50	32:18	
13C12-HxCDD 478	10.2	102	1.18	33:00	
13C12-HpCDF 789	10.2	102	0.42	36:45	

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	11.0	110	0.50	33:38	
13C12-HxCDF 234	11.3	113	0.51	32:54	

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	9.1	90.8	0.76	24:52	
13C12-2378-TCDD	8.3	83.1	0.79	25:36	
13C12-PeCDF 123	8.1	81.1	1.50	28:51	
13C12-PeCDD 123	11.3	113	1.50	29:53	
13C12-HxCDF 678	9.9	98.6	0.50	32:25	
13C12-HxCDD 678	11.0	110	1.20	33:04	
13C12-HpCDF 678	8.6	85.8	0.43	35:15	
13C12-HpCDD 678	9.2	91.7	1.01	36:16	
13C12-OCDD	15.2	76.0	0.86	39:49	

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.78	25:24	
13C12-HxCDD 789	1.18	33:23	

Reviewed By: | TDH 11/30/92 X237_RPT 4.01, LARS 5.0

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TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

LE NAME.: S925331 SAMPLE ID.: TEST 7/2-STACK-7328
OJECT...: 22314 CLIENT NAME...: INTERPOLL LABS
IENT PROJECT: ANL-OTP/BIG STONE, S.D.

I ID....:	61-11-7A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
LLECTED.:	n/a	ANALYSIS TIME.:	09:49	DILUTION.....:	n/a
CEIVED..:	11/04/92	ANALYST.......:	NB	BLANK FILE...:	S925324
TRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
T. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
J. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
T. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
T. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
IKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
JECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.02				—
378-PeCDD	ND		0.02				—
3478-HxCDD	ND		0.02				—
3678-HxCDD	ND		0.02				—
3789-HxCDD	ND		0.02				—
34678-HpCDD	0.04				1.02	36:10	B
DD	0.49				0.81	39:43	B
78-TCDF	0.03				0.66	24:48	B
378-PeCDF	ND		0.02				—
478-PeCDF	ND		0.01				—
3478-HxCDF	ND		0.02				—
3678-HxCDF	ND		0.01				—
4678-HxCDF	EMPC			0.02			B
3789-HxCDF	ND		0.02				—
34678-HpCDF	ND		0.01				—
34789-HpCDF	ND		0.02				—
DF	0.03				0.77	39:54	—
TAL TCDD	ND		0.02				—
TAL PeCDD	ND		0.02				—
TAL HxCDD	ND		0.02				—
TAL HpCDD	0.06	2			1.09		—
TAL TCDF	0.04	2		0.06	0.74		—
TAL PeCDF	0.02	1			1.65		—
TAL HxCDF	EMPC			0.02			—
TAL HpCDF	ND		0.02				—

viewed By: | DH 11/30/92 X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

FILE NAME.: S925331 SAMPLE ID.: TEST 7/2-STACK-7328
PROJECT...: 22314 CLIENT NAME...: INTERPOLL LABS
CLIENT PROJECT: ANL-OTP/BIG STONE, S.D.
TLI ID....: 61-11-7A-D ANALYSIS DATE.: 11/24/92 CLIENT CODE..: IPL01
COLLECTED.: n/a ANALYSIS TIME.: 09:49 DILUTION.....: n/a
RECEIVED..: 11/04/92 ANALYST.......: NB BLANK FILE...: S925324
MATRIX....: MM5UNITS INSTRUMENT....: S % LIPID......: n/a
EXT. SIZE.: 1.000 GC COLUMN.....: DB-5 % SOLIDS.....: n/a
ADJ. SIZE.: 1.000 GC COLUMN ID..: 2493345 % MOISTURE...: n/a
EXT. DATE.: 11/12/92 ICAL NAME.....: SC50072 ORIGIN.......: SEE COC
EXT. VOL..: 30.00 ul ICAL DATE.....: 10/07/92 CONTRACT.....: n/a
SPIKE FILE: SPX23710 CONCAL NAME...: S925323 SAS NUMBER...: n/a
INJECT VOL: 2.0 ul CONCAL DATE...: 11/24/92 EPISODE......: n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	2.0	20.1		25:30	√
13C12-PeCDF 234	2.7	26.8	1.62	29:26	√
13C12-HxCDF 478	2.8	27.5	0.50	32:12	√
13C12-HxCDD 478	2.6	25.8	1.16	32:53	√
13C12-HpCDF 789	3.1	30.7	0.41	36:38	

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	7.8	78.4	0.50	33:31	
13C12-HxCDF 234	7.5	74.9	0.50	32:46	

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	5.8	57.7	0.74	24:45	
13C12-2378-TCDD	5.7	56.6	0.77	25:29	
13C12-PeCDF 123	5.8	57.9	1.62	28:43	
13C12-PeCDD 123	7.7	76.7	1.55	29:46	
13C12-HxCDF 678	5.7	56.7	0.51	32:17	
13C12-HxCDD 678	6.8	68.4	1.18	32:58	
13C12-HpCDF 678	5.8	58.3	0.42	35:08	
13C12-HpCDD 678	7.2	71.6	1.00	36:09	
13C12-OCDD	13.1	65.3	0.86	39:42	

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.79	25:17	
13C12-HxCDD 789	1.20	33:16	

Reviewed By: | DH 11/30/92

X237_RPT 4.01, LARS 5.0

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa)

```
ILE NAME.: S925332        SAMPLE ID.: TEST 7/3-STACK-7328
ROJECT...: 22314          CLIENT NAME...: INTERPOLL LABS
LIENT PROJECT: ANL-OTP/BIG STONE, S.D.
LI ID....: 61-11-8A-D     ANALYSIS DATE.: 11/24/92    CLIENT CODE..: IPL01
OLLECTED.: n/a            ANALYSIS TIME.: 10:43       DILUTION.....: n/a
ECEIVED..: 11/04/92       ANALYST.......: NB          BLANK FILE...: S925324
ATRIX....: MM5UNITS       INSTRUMENT....: S           % LIPID......: n/a
KT. SIZE.: 1.000          GC COLUMN.....: DB-5        % SOLIDS.....: n/a
OJ. SIZE.: 1.000          GC COLUMN ID..: 2493345     % MOISTURE...: n/a
KT. DATE.: 11/12/92       ICAL NAME.....: SC50072     ORIGIN.......: SEE COC
KT. VOL..: 30.00 ul       ICAL DATE.....: 10/07/92    CONTRACT.....: n/a
PIKE FILE: SPX23710       CONCAL NAME...: S925323     SAS NUMBER...: n/a
NJECT VOL: 2.0 ul         CONCAL DATE...: 11/24/92    EPISODE......: n/a
```

ME	AMT(ng)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
78-TCDD	ND		0.03				
378-PeCDD	ND		0.04				
3478-HxCDD	ND		0.06				
3678-HxCDD	ND		0.04				
3789-HxCDD	ND		0.05				
34678-HpCDD	ND		0.08				
DD	0.57				1.00	39:43	B
78-TCDF	0.06				0.77	24:49	B
378-PeCDF	ND		0.03				
478-PeCDF	ND		0.03				
3478-HxCDF	ND		0.04				
3678-HxCDF	ND		0.03				
4678-HxCDF	ND		0.04				
3789-HxCDF	ND		0.05				
34678-HpCDF	ND		0.04				
34789-HpCDF	ND		0.07				
DF	ND		0.1				
TAL TCDD	ND		0.03				
TAL PeCDD	ND		0.04				
TAL HxCDD	ND		0.05				
TAL HpCDD	ND		0.08				
TAL TCDF	0.13	2		0.17	0.77		
TAL PeCDF	ND		0.03				
TAL HxCDF	ND		0.04				
TAL HpCDF	ND		0.05				

viewed By: DH 11/30/92

X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (aa) QA/QC SUMMARY

FILE NAME.: S925332
SAMPLE ID.: TEST 7/3-STACK-7328
PROJECT...: 22314
CLIENT NAME...: INTERPOLL LABS
CLIENT PROJECT: ANL-OTP/BIG STONE, S.D.

TLI ID....:	61-11-8A-D	ANALYSIS DATE.:	11/24/92	CLIENT CODE..:	IPL01
COLLECTED.:	n/a	ANALYSIS TIME.:	10:43	DILUTION.....:	n/a
RECEIVED..:	11/04/92	ANALYST.......:	NB	BLANK FILE...:	S925324
MATRIX....:	MM5UNITS	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	1.000	GC COLUMN.....:	DB-5	% SOLIDS.....:	n/a
ADJ. SIZE.:	1.000	GC COLUMN ID..:	2493345	% MOISTURE...:	n/a
EXT. DATE.:	11/12/92	ICAL NAME.....:	SC50072	ORIGIN.......:	SEE COC
EXT. VOL..:	30.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX23710	CONCAL NAME...:	S925323	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	11/24/92	EPISODE......:	n/a

SURROGATE RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
37Cl-TCDD	7.9	79.5		25:31	
13C12-PeCDF 234	10.2	102	1.45	29:27	
13C12-HxCDF 478	10.1	101	0.49	32:12	
13C12-HxCDD 478	10.2	102	1.19	32:54	
13C12-HpCDF 789	9.8	97.6	0.44	36:39	

ALTERNATE STANDARDS RECOVERY SUMMARY (TYPE AA)

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-HxCDF 789	7.2	71.7	0.49	33:32	
13C12-HxCDF 234	8.3	83.3	0.50	32:48	

INTERNAL STANDARDS RECOVERY SUMMARY

NAME	AMT (ng)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	5.7	57.0	0.71	24:46	
13C12-2378-TCDD	5.5	54.9	0.79	25:30	
13C12-PeCDF 123	5.0	49.9	1.51	28:45	
13C12-PeCDD 123	6.9	69.0	1.53	29:47	
13C12-HxCDF 678	7.6	75.8	0.49	32:18	
13C12-HxCDD 678	8.5	85.2	1.22	32:58	
13C12-HpCDF 678	6.0	59.8	0.45	35:09	
13C12-HpCDD 678	6.2	62.2	1.00	36:10	
13C12-OCDD	8.1	40.7	0.87	39:43	

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.79	25:19	
13C12-HxCDD 789	1.20	33:17	

Reviewed By: | DH 11/30/92 |

X237_RPT 4.01, LARS 5.0

SLP

Job Argonne Labs-BigStone **Test No.** 2
Report Number 2-7328 **Reviewer**

Analyst: ☐ Twin City Testing
☒ Triangle Labs

OK
12-14-92

Note: All masses are in nanograms

Native Isomers	Raw Data (By Run Numbers)								To be Entered in Computer			
	0 Mass	1 Mass	2 Mass	3 Mass	0 DL	1 DL	2 DL	3 DL	0 Mass	1 Mass	2 Mass	3 Mass
2378-TCDF	ND	ND	0.02	ND	0.03	0.02		0.02	<0.03	<0.02	0.02	<0.02
TOTAL TCDF	ND	ND	0.02	ND	0.03	0.02		0.02	<0.03	<0.02	0.02	<0.02
2378-TCDD	ND	ND	ND	ND	0.04	0.03	0.008	0.03	<0.04	<0.03	<0.008	<0.03
TOTAL TCDD	ND	ND	ND	ND	0.04	0.03	0.008	0.03	<0.04	<0.03	<0.008	<0.03
12378-PeCDF	ND	ND	ND	ND	0.04	0.02	0.005	0.02	<0.04	<0.02	<0.005	<0.02
23478-PeCDF	ND	ND	ND	ND	0.03	0.02	0.005	0.02	<0.03	<0.02	<0.005	<0.02
TOTAL PECDF	ND	ND	0.01	ND	0.04	0.02		0.02	<0.04	<0.02	0.01	<0.02
12378-PeCDD	ND	ND	ND	ND	0.05	0.03	0.008	0.03	<0.05	<0.03	<0.008	<0.03
TOTAL PeCDD	ND	ND	ND	ND	0.05	0.03	0.008	0.03	<0.05	<0.03	<0.008	<0.03
123478-HxCDF	ND	ND	ND	ND	0.05	0.02	0.005	0.03	<0.05	<0.02	<0.005	<0.03
123678-HxCDF	ND	ND	ND	ND	0.03	0.02	0.005	0.02	<0.03	<0.02	<0.005	<0.02
123789-HxCDF	ND	ND	ND	ND	0.06	0.03	0.008	0.03	<0.06	<0.03	<0.008	<0.03
234678-HxCDF	ND	ND	0.02	EMPC	0.04	0.02		0.03	<0.04	<0.02	0.02	<0.03
TOTAL HxCDF	ND	ND	0.02	EMPC	0.04	0.02		0.03	<0.04	<0.02	0.02	<0.03
123478-HxCDD	ND	ND	ND	ND	0.08	0.04	0.01	0.04	<0.08	<0.04	<0.01	<0.04
123678-HxCDD	ND	ND	ND	ND	0.05	0.03	0.005	0.03	<0.05	<0.03	<0.005	<0.03
123789-HxCDD	ND	ND	ND	ND	0.07	0.03	0.008	0.03	<0.07	<0.03	<0.008	<0.03
TOTAL HxCDD	ND	ND	ND	ND	0.06	0.03	0.008	0.03	<0.06	<0.03	<0.008	<0.03
1234678-HpCDF	ND	ND	EMPC	ND	0.05	0.02	0.007	0.02	<0.05	<0.02	<0.007	<0.02
1234789-HpCDF	ND	ND	ND	ND	0.08	0.04	0.008	0.04	<0.08	<0.04	<0.008	<0.04
TOTAL HpCDF	ND	ND	EMPC	ND	0.06	0.03	0.009	0.03	<0.06	<0.03	<0.009	<0.03
1234678-HpCDD	ND	0.07	EMPC	EMPC	0.1		0.04	0.06	<0.1	0.07	<0.04	<0.06
TOTAL HpCDD	ND	0.07	0.03	EMPC	0.1			0.11	<0.1	0.07	0.03	<0.11
OCDF	EMPC	ND	0.02	ND	0.97	.05		0.05	<0.97	<0.05	0.02	<0.05
OCDD	ND	EMPC	0.41	0.66	0.3	0.52			<0.3	<0.52	0.41	0.66

PCDD/PCDF Data Reduction

SP

Job Argonne Labs Big Stone Test No. 7
Report Number 2-2328 Reviewer ____

Analyst: ___ Twin City Testing
X Triangle Labs

12-14-92
OK PL

Note: All masses are in nanograms

Native Isomers	Raw Data (By Run Numbers)								To be Entered in Computer			
	0 Mass	1 Mass	2 Mass	3 Mass	0 DL	1 DL	2 DL	3 DL	0 Mass	1 Mass	2 Mass	3 Mass
2378-TCDF	ND	EMPC	0.03	0.06	0.02	0.01			< 0.02	< .01	0.03	0.06
TOTAL TCDF	ND	EMPC	0.04	0.13	0.02	0.01			< 0.02	< .01	0.04	0.13
2378-TCDD	ND	ND	ND	ND	0.04	0.01	0.02	0.03	< .04	< .01	< .02	< .03
TOTAL TCDD	ND	ND	ND	ND	0.04	0.01	0.02	0.03	< .04	< .01	< .02	< .03
12378-PeCDF	ND	ND	ND	ND	0.03	0.01	0.02	0.03	< .03	< .01	< .02	< .03
23478-PeCDF	ND	ND	ND	ND	0.03	0.01	0.01	0.03	< .03	< .01	< .01	< .03
TOTAL PECDF	ND	ND	0.02	ND	0.03	0.01		0.03	< .03	< .01	.02	< .03
12378-PeCDD	ND	ND	ND	ND	0.04	0.01	0.02	0.04	< .04	< .01	< .02	< .04
TOTAL PeCDD	ND	ND	ND	ND	0.04	0.01	0.02	0.04	< .04	< .01	< .02	< .04
123478-HxCDF	ND	0.02	ND	ND	0.03		0.02	0.04	< .03	0.02	< .02	< .04
123678-HxCDF	ND	ND	ND	ND	0.03	0.01	0.01	0.03	< .03	< .01	< .01	< .03
123789-HxCDF	ND	ND	ND	ND	0.04	0.02	0.02	0.05	< .04	< .02	< .02	< .05
234678-HxCDF	ND	EMPC	EMPC	ND	0.03	0.02	0.02	0.04	< .03	< .02	< .02	< .04
TOTAL HxCDF	ND	0.02	EMPC	ND	0.03		0.02	0.04	< .03	0.02	< .02	< .04
123478-HxCDD	ND	ND	ND	ND	0.05	0.02	0.02	0.06	< .05	< .02	< .02	< .06
123678-HxCDD	ND	ND	ND	ND	0.03	0.01	0.02	0.04	< .03	< .01	< .02	< .04
123789-HxCDD	ND	ND	ND	ND	0.04	0.02	0.02	0.05	< .04	< .02	< .02	< .05
TOTAL HxCDD	ND	EMPC	ND	ND	0.04	0.02	0.02	0.05	< .04	< .02	< .02	< .05
1234678-HpCDF	ND	0.03	ND	ND	0.03		0.01	0.04	< .03	0.03	< .01	< .04
1234789-HpCDF	ND	ND	ND	ND	0.05	0.02	0.02	0.07	< .05	< 0.02	< .02	< .07
TOTAL HpCDF	ND	0.04	ND	ND	0.04		0.02	0.05	< .04	0.04	< .02	< .05
1234678-HpCDD	ND	0.05	0.04	ND	0.05			0.08	< .05	0.05	0.04	< .08
TOTAL HpCDD	ND	0.05	0.06	ND	0.05			0.08	< .05	0.05	0.06	< .08
OCDF	ND	0.07	0.03	ND	0.07			0.1	< 0.07	0.07	0.03	< 0.1
OCDD	EMPC	0.58	0.49	0.57	0.61				< 0.61	0.58	0.49	0.57

DS-007

TRIANGLE LABORATORIES OF RTP, INC.
801 CAPITOLA DRIVE
DURHAM, NC 27713

Phone: (919) 544-5729
Fax: (919) 544-5491

DATE : * DECEMBER 19, 1992

CLIENT ID : * INTERPOLL LABS

P.O. NUMBER : * 16934KE

TLI PROJECT No. : * 22499

RECEIVED
DEC 22 1992
INTERPOLL LABORATORIES

CASE NARRATIVE
MODEL 8290X

ANALYSIS OF SAMPLES FOR THE PRESENCE OF

POLYCHLORINATED DIBENZO-p-DIOXINS

AND

DIBENZOFURANS

BY

HIGH-RESOLUTION GAS CHROMATOGRAPHY / HIGH-RESOLUTION MASS SPECTROMETRY

Four ash samples were received from INTERPOLL LABS in good condition November 30, 1992 and stored in a refrigerator at 4°C. The samples were extracted and analyzed according to procedures described in the Triangle Labs User Manual provided with this data package. Any particular difficulties encountered during the sample handling by Triangle Labs will be discussed in the QC remark section below.

Quality Control Samples

A laboratory method blank -- identified as the TLI Ash Blank -- is prepared along with the samples.

QC Remarks

The release of this particular set of INTERPOLL LABS analytical data by Triangle Labs was authorized by the Quality Control Chemist who has reviewed each sample data package individually following a series of inspections/reviews. When applicable, general deviations from acceptable QC requirements are identified below. Comments on the effect of these deviations upon the validity and reliability of the results can be obtained from the User Manual (Data Quality Objectives; Section 5). Specific QC Problems Associated with this Particular Project are:

Sample Preparation Laboratory: None

Mass Spectrometry: None

Data Review: None

Any analytes found in the TLI Blank are detected at a level at or below the Target Detection Limit. This level of contamination is acceptable as per TLI Guidelines. OCDD is not subject to blank contamination criteria as per TLI Guidelines.

Addendum to Section 2.3 of the Data User's Manual

All sample and calibration analyses are conducted using a 2 uL injection volume, unless otherwise specified.

Addendum to Section 4.6 of the Data User's Manual

Effective December 15, 1989, Triangle Laboratories has adopted a new procedure for calculating the analyte specific detection limits. Under this new policy two representative noise height determinations are summed and multiplied by an empirically determined factor before the detection limit equation given in Section 4.6 of the Data User's Manual is applied. The effect of this procedure is to increase DB-225 analyses detection limits by a factor of 3.5, and DB-5 analyses detection limits by a factor of 5. This procedure will result in a more accurate estimate of detection limits.

The revised formula for DB-225 analyses is:

$$DL_{\delta i}^{DB-225} = \frac{2 * 2.5 * (3.5 * H_{\delta i}) * Q_{\beta j}}{A_{\beta j} * \overline{RRF} * W}$$

$$3.5 * H_{\delta i} = A_{noise}$$

The revised formula for DB-5 analyses is:

$$DL_{\delta i}^{DB-5} = \frac{2 * 2.5 * (5 * H_{\delta i}) * Q_{\beta j}}{A_{\beta j} * \overline{RRF} * W}$$

$$5 * H_{\delta i} = A_{noise}$$

The symbols are defined in Section 4.6 of the Data User's Manual.

As an example, consider a DB-225 confirmation analysis of a 10 g sample. The internal standard spike is 2000 pg, the 13C12-2,3,7,8-TCDF internal standard area is 12000 area units, the RRF is 1.215 and the noise height is 11 units. The 2,3,7,8-TCDF detection limit is:

$$DL_{TCDF}^{DB-225} = \frac{2 * 2.5 * (3.5 * 11) * 2000}{12000 * 1.215 * 10.0} = 2.6 \text{ ppt}$$

Addendum to Section 3.3.1 in the Data User's Manual

A "B" flag is used to indicate that an analyte has been detected in the laboratory method blank as well as in an associated field sample. This flag denotes possible contribution of background laboratory contamination to the concentration or amount of that analyte detected in the field sample. Under Triangle Laboratories of RTP guidelines, a laboratory blank is acceptable if one of the following conditions is satisfied: 1) the tetra through hepta CDD/CDF levels are all below the target detection limits (TDLs), 2) the analyte levels found are all below 1/3 the theoretical method detection limit (TMQL), or 3) the contamination levels are less than 5% of the levels detected in the associated field samples. If these conditions are satisfied or if the blank is unable to be reextracted, the interpretation of the contamination levels relative to the samples should be as follows: 1) analyte quantitations should be considered valid if the level of blank contamination is less than five percent of the level detected in the field sample, 2) analyte quantitations should be considered estimated if the analyte level in the sample is five to twenty times the level of the analyte in the blank, or 3) analytes whose level in a sample is the same as or less than five times the level detected in the associated blank should be considered present likely due to laboratory contamination and not native to the sample.

A "Q" flag is used to indicate the presence of QC ion instabilities caused by quantitative interferences. Affected analytes may be overestimated or underestimated as a result of this interference. A peak is flagged "Q" only if it is affected by a QC ion deviation greater than 20% full scale as determined relative to the labeled standard against which it is quantitated. Total PCDD/PCDF quantitations will be flagged "Q" if the interferences affect ten percent or more of the total PCDD/PCDF peak areas.

A "U" flag is used to indicate that a specific isomer cannot be resolved from a large, coeluting interferent GC peak. The specific isomer is reported as not detected as a valid concentration/amount cannot be determined. The calculated detection limit, therefore, should be considered an underestimated value.

A "V" flag is used to indicate that, although the percent recovery of a labeled standard may be below a specific QC limit, the signal-to-noise ratio of the peak is greater than 10:1. The standard is considered reliably quantifiable. All quantitations derived from the standard are considered valid as well.

A "PR" flag is used to indicate that a GC peak is poorly resolved. This resolution problem may be seen as two closely eluting peaks without a reasonable valley between the peak tops, overly broad peaks, or peaks whose shapes vary greatly from a normal distribution. The concentrations or amounts reported for such peaks are most likely overestimated.

An "I" flag is used to indicate labeled standards have been interfered with on the GC column by coeluting, interferent peaks. The interference may have caused the standard's area to be overestimated. All quantitations relative to this standard, therefore, may be underestimated.

An "E" flag is used to indicate that a PCDF peak has eluted at the same time as the associated diphenyl ether (DPE) and that the DPE peak intensity is ten percent or more of the PCDF peak intensity. Total PCDF values are flagged "E" if the total DPE contribution to the total PCDF value is greater than ten percent. All PCDF peaks that are significantly influenced by the presence of DPE peaks are quantitated with EMPC values, regardless of the isotopic abundance ratio. These EMPC values are most likely overestimated due to the DPE contribution to the peak area.

An "RO" flag is used to indicate that a labeled standard has an ion-abundance ratio that is outside of the acceptable QC limits, most likely due to a coeluting interference. This may have caused the percent recovery of the standard to be overestimated. All quantitations versus this standard, therefore, may be underestimated.

Should INTERPOLL LABS have any questions or comments regarding this data package please feel free to contact us.

For Triangle Laboratories of RTP, Inc.,

Report Preparation	Quality Control
Amy J. Boehm	
Report Preparation Chemist	Report Preparation Chemist

```
                 TRIANGLE LABORATORIES OF RTP, INC.              Page 1 of
                   PCDD/PCDF 2378X ANALYSIS (b)                    12/19/9

FILE NAME.: S925716       SAMPLE ID.: TLI ASH BLANK
PROJECT...: 22499         CLIENT NAME...: INTERPOLL LABS
CLIENT PROJECT: n/a
TLI ID....: n/a           ANALYSIS DATE.: 12/15/92  CLIENT CODE..: IPL01
COLLECTED.: n/a           ANALYSIS TIME.: 07:18     DILUTION.....: n/a
RECEIVED..: n/a           ANALYST.......: DM        BLANK FILE...: S925716
MATRIX....: ASH           INSTRUMENT....: S         % LIPID......: n/a
EXT. SIZE.: 14.869 g      GC COLUMN.....: DB-5      % SOLIDS.....: n/a
ADJ. SIZE.: 14.869 g      GC COLUMN ID..: 2568817   % MOISTURE...: n/a
EXT. DATE.: 12/04/92      ICAL NAME.....: SC50072   ORIGIN.......: n/a
EXT. VOL..: 20.00 ul      ICAL DATE.....: 10/07/92  CONTRACT.....: n/a
SPIKE FILE: SPX2372S      CONCAL NAME...: S925711   SAS NUMBER...: n/a
INJECT VOL: 2.0 ul        CONCAL DATE...: 12/15/92  EPISODE......: n/a
==========================================================================
NAME               CONC(ppt) NUMBER     DL     EMPC     RATIO     RT    FLAGS
--------------------------------------------------------------------------
2378-TCDD             ND               0.3                              ___
12378-PeCDD           ND               0.4                              ___
123478-HxCDD          ND               0.6                              ___
123678-HxCDD          ND               0.4                              ___
123789-HxCDD          ND               0.5                              ___
1234678-HpCDD          1.6                               1.01    37:20  ___
OCDD                  22.4                               1.02    41:05  ___

2378-TCDF             ND               0.2                              ___
12378-PeCDF           ND               0.3                              ___
23478-PeCDF           ND               0.3                              ___
123478-HxCDF          ND               0.4                              ___
123678-HxCDF          ND               0.3                              ___
234678-HxCDF          ND               0.4                              ___
123789-HxCDF          ND               0.5                              ___
1234678-HpCDF         ND               0.4                              ___
1234789-HpCDF         ND               0.7                              ___
OCDF                  ND               1.2                              ___

TOTAL TCDD            ND               0.3                              ___
TOTAL PeCDD           ND               0.4                              ___
TOTAL HxCDD           ND               0.5                              ___
TOTAL HpCDD            1.6      1               2.6      1.01           ___

TOTAL TCDF            ND               0.2                              ___
TOTAL PeCDF           ND               0.3                              ___
TOTAL HxCDF           ND               0.4                              ___
TOTAL HpCDF           ND               0.5                              ___
--------------------------------------------------------------------------
Reviewed By: |DB____________    12/19/92|         X237_RPT 4.01, LARS 5.0
```

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b) QA/QC SUMMARY

ILE NAME.: S925716
ROJECT ..: 22499
IENT PROJECT: n/a
I ID....: n/a
OLLECTED.: n/a
ECEIVED..: n/a
ATRIX....: ASH
XT. SIZE.: 14.869 g
DJ. SIZE.: 14.869 g
XT. DATE.: 12/04/92
XT. VOL..: 20.00 ul
PIKE FILE: SPX2372S
NJECT VOL: 2.0 ul

SAMPLE ID.: TLI ASH BLANK
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 07:18
ANALYST.......: DM
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2568817
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925716
% LIPID......: n/a
% SOLIDS.....: n/a
% MOISTURE...: n/a
ORIGIN.......: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

RROGATE RECOVERY SUMMARY (TYPE B)

ME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
Cl-TCDD	10.5	78.3		26:42	___
C12-PeCDF 234	92.2	68.5	1.38	30:29	___
C12-HxCDF 478	125	92.9	0.51	33:12	___
C12-HxCDD 478	135	101	1.22	33:55	___
C12-HpCDF 789	97.7	72.6	0.43	37:51	___

TERNATE STANDARDS RECOVERY SUMMARY (TYPE B)

ME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
C12-HxCDF 789	131	97.7	0.50	34:35	___
C12-HxCDF 234	125	93.3	0.47	33:48	___

TERNAL STANDARDS RECOVERY SUMMARY

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-2378-TCDF	93.2	69.3	0.73	26:00	___
3C12-2378-TCDD	102	76.2	0.81	26:41	___
3C12-PeCDF 123	86.6	64.4	1.34	29:49	___
3C12-PeCDD 123	108	80.1	1.45	30:50	___
3C12-HxCDF 678	116	86.2	0.49	33:18	___
3C12-HxCDD 678	127	94.1	1.17	33:59	___
3C12-HpCDF 678	92.6	68.8	0.44	36:16	___
3C12-HpCDD 678	98.7	73.4	0.98	37:19	___
3C12-OCDD	130	48.3	0.89	41:04	___

ECOVERY STANDARDS RECOVERY SUMMARY

AME	RATIO	RT	FLAGS
3C12-1234-TCDD	0.82	26:31	___
3C12-HxCDD 789	1.17	34:19	___

eviewed By: | ______________ 12/19/92 |

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b)

FILE NAME.: S925717
PROJECT...: 22499
CLIENT PROJECT: n/a
TLI ID....: 61-155-1
COLLECTED.: 10/26/92
RECEIVED..: 11/30/92
MATRIX....: ASH
EXT. SIZE.: 12.881 g
ADJ. SIZE.: 12.572 g
EXT. DATE.: 12/04/92
EXT. VOL..: 20.00 ul
SPIKE FILE: SPX2372S
INJECT VOL: 2.0 ul

SAMPLE ID.: 7328-123
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 08:07
ANALYST.......: DM
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2568817
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925716
% LIPID......: n/a
% SOLIDS.....: 97.6
% MOISTURE...: 2.4
ORIGIN.......: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

NAME	CONC(ppt)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
2378-TCDD	ND		0.5				
12378-PeCDD	ND		0.8				
123478-HxCDD	ND		1.2				
123678-HxCDD	ND		0.8				
123789-HxCDD	ND		1.0				
1234678-HpCDD	EMPC			1.9			B
OCDD	24.3				0.90	41:07	B
2378-TCDF	ND		0.4				
12378-PeCDF	ND		0.6				
23478-PeCDF	ND		0.5				
123478-HxCDF	ND		0.7				
123678-HxCDF	ND		0.5				
234678-HxCDF	ND		0.7				
123789-HxCDF	ND		0.9				
1234678-HpCDF	ND		0.8				
1234789-HpCDF	ND		1.4				
OCDF	ND		2.4				
TOTAL TCDD	ND		0.5				
TOTAL PeCDD	ND		0.8				
TOTAL HxCDD	ND		1.0				
TOTAL HpCDD	EMPC			1.9			
TOTAL TCDF	ND		0.4				
TOTAL PeCDF	ND		0.5				
TOTAL HxCDF	ND		0.7				
TOTAL HpCDF	ND		1.0				

Reviewed By: | 12/19/92 |

X237_RPT 4.01, LARS 5.

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b) QA/QC SUMMARY

ILE NAME.: S925717
ROJECT...: 22499
LIENT PROJECT: n/a
LI ID....: 61-155-1
OLLECTED.: 10/26/92
ECEIVED..: 11/30/92
ATRIX....: ASH
XT. SIZE.: 12.881 g
DJ. SIZE.: 12.572 g
XT. DATE.: 12/04/92
XT. VOL..: 20.00 ul
PIKE FILE: SPX2372S
NJECT VOL: 2.0 ul

SAMPLE ID.: 7328-123
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 08:07
ANALYST........: DM
INSTRUMENT....: S
GC COLUMN......: DB-5
GC COLUMN ID..: 2568817
ICAL NAME......: SC50072
ICAL DATE......: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION......: n/a
BLANK FILE...: S925716
% LIPID.......: n/a
% SOLIDS......: 97.6
% MOISTURE...: 2.4
ORIGIN........: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

RROGATE RECOVERY SUMMARY (TYPE B)

ME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
Cl-TCDD	13.5	85.0		26:44	
C12-PeCDF 234	110	69.3	1.41	30:32	
C12-HxCDF 478	143	89.7	0.50	33:14	
C12-HxCDD 478	150	94.2	1.23	33:56	
C12-HpCDF 789	110	69.0	0.43	37:52	

TERNATE STANDARDS RECOVERY SUMMARY (TYPE B)

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-HxCDF 789	148	92.8	0.51	34:37	
3C12-HxCDF 234	145	90.9	0.50	33:49	

NTERNAL STANDARDS RECOVERY SUMMARY

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-2378-TCDF	115	72.4	0.74	26:01	
3C12-2378-TCDD	126	79.5	0.78	26:43	
3C12-PeCDF 123	108	68.0	1.42	29:51	
3C12-PeCDD 123	129	81.1	1.44	30:51	
3C12-HxCDF 678	142	89.0	0.50	33:19	
3C12-HxCDD 678	147	92.6	1.15	34:01	
3C12-HpCDF 678	105	66.2	0.42	36:17	
3C12-HpCDD 678	111	69.8	1.02	37:21	
3C12-OCDD	152	47.7	0.90	41:06	

ECOVERY STANDARDS RECOVERY SUMMARY

AME	RATIO	RT	FLAGS
3C12-1234-TCDD	0.81	26:32	
3C12-HxCDD 789	1.22	34:21	

eviewed By: | ______________ 12/15/92 |

X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b)

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FILE NAME.: S925719
PROJECT...: 22499
CLIENT PROJECT: n/a
TLI ID....: 61-155-3
COLLECTED.: 10/26/92
RECEIVED..: 11/30/92
MATRIX....: ASH
EXT. SIZE.: 11.546 g
ADJ. SIZE.: 11.523 g
EXT. DATE.: 12/04/92
EXT. VOL..: 20.00 ul
SPIKE FILE: SPX2372S
INJECT VOL: 2.0 ul

SAMPLE ID.: 7328-126
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 09:48
ANALYST.......: NB
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2568817
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925716
% LIPID......: n/a
% SOLIDS.....: 99.8
% MOISTURE...: 0.2
ORIGIN.......: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

NAME	CONC(ppt)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
2378-TCDD	ND		0.5				
12378-PeCDD	ND		0.7				
123478-HxCDD	ND		1.0				
123678-HxCDD	ND		0.7				
123789-HxCDD	ND		0.9				
1234678-HpCDD	1.7				1.04	37:21	B
OCDD	25.4				0.88	41:07	B
2378-TCDF	ND		0.4				
12378-PeCDF	ND		0.5				
23478-PeCDF	ND		0.5				
123478-HxCDF	ND		0.6				
123678-HxCDF	ND		0.5				
234678-HxCDF	ND		0.6				
123789-HxCDF	ND		0.8				
1234678-HpCDF	ND		0.7				
1234789-HpCDF	ND		1.2				
OCDF	ND		2.1				
TOTAL TCDD	ND		0.5				
TOTAL PeCDD	ND		0.7				
TOTAL HxCDD	ND		0.8				
TOTAL HpCDD	1.7	1		2.8	1.04		
TOTAL TCDF	ND		0.4				
TOTAL PeCDF	ND		0.5				
TOTAL HxCDF	ND		0.6				
TOTAL HpCDF	ND		0.8				

Reviewed By: | 12/19/92 |

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TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b) QA/QC SUMMARY

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'ILE NAME.: S925719
'ROJECT...: 22499
:LIENT PROJECT: n/a
'LI ID....: 61-155-3
:OLLECTED.: 10/26/92
:ECEIVED..: 11/30/92
IATRIX....: ASH
:XT. SIZE.: 11.546 g
ADJ. SIZE.: 11.523 g
:XT. DATE.: 12/04/92
:XT. VOL..: 20.00 ul
;PIKE FILE: SPX2372S
:NJECT VOL: 2.0 ul

SAMPLE ID.: 7328-126
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 09:48
ANALYST........: NB
INSTRUMENT....: S
GC COLUMN......: DB-5
GC COLUMN ID..: 2568817
ICAL NAME......: SC50072
ICAL DATE......: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION......: n/a
BLANK FILE...: S925716
% LIPID.......: n/a
% SOLIDS......: 99.8
% MOISTURE...: 0.2
ORIGIN........: n/a
CONTRACT......: n/a
SAS NUMBER...: n/a
EPISODE.......: n/a

JRROGATE RECOVERY SUMMARY (TYPE B)

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
7Cl-TCDD	13.4	77.0		26:43	
3C12-PeCDF 234	129	74.0	1.37	30:30	
3C12-HxCDF 478	174	100	0.51	33:13	
3C12-HxCDD 478	191	110	1.18	33:56	
3C12-HpCDF 789	136	78.1	0.44	37:52	

LTERNATE STANDARDS RECOVERY SUMMARY (TYPE B)

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-HxCDF 789	186	107	0.50	34:36	
3C12-HxCDF 234	180	104	0.50	33:49	

NTERNAL STANDARDS RECOVERY SUMMARY

'AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-2378-TCDF	119	68.4	0.73	26:01	
3C12-2378-TCDD	127	72.9	0.82	26:42	
.3C12-PeCDF 123	118	67.8	1.40	29:51	
.3C12-PeCDD 123	141	81.4	1.45	30:51	
.3C12-HxCDF 678	160	92.0	0.51	33:19	
.3C12-HxCDD 678	170	98.1	1.22	34:01	
.3C12-HpCDF 678	120	68.9	0.43	36:17	
13C12-HpCDD 678	126	72.7	1.07	37:21	
13C12-OCDD	172	49.5	0.87	41:06	

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.81	26:32	
13C12-HxCDD 789	1.17	34:20	

Reviewed By: | 12/15/92 |

X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b)

FILE NAME.: S925718
PROJECT...: 22499
CLIENT PROJECT: n/a
TLI ID....: 61-155-2
COLLECTED.: 10/27/92
RECEIVED..: 11/30/92
MATRIX....: ASH
EXT. SIZE.: 12.864 g
ADJ. SIZE.: 12.594 g
EXT. DATE.: 12/04/92
EXT. VOL..: 20.00 ul
SPIKE FILE: SPX2372S
INJECT VOL: 2.0 ul

SAMPLE ID.: 7328-129
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 08:56
ANALYST.......: DM
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2568817
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925716
% LIPID......: n/a
% SOLIDS.....: 97.9
% MOISTURE...: 2.1
ORIGIN.......: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

NAME	CONC(ppt)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
2378-TCDD	ND		0.4				
12378-PeCDD	ND		0.7				
123478-HxCDD	ND		1.0				
123678-HxCDD	ND		0.7				
123789-HxCDD	ND		0.8				
1234678-HpCDD	1.3				1.05	37:22	B
OCDD	23.1				0.82	41:06	B
2378-TCDF	ND		0.3				
12378-PeCDF	ND		0.5				
23478-PeCDF	ND		0.5				
123478-HxCDF	ND		0.6				
123678-HxCDF	ND		0.5				
234678-HxCDF	ND		0.6				
123789-HxCDF	ND		0.7				
1234678-HpCDF	ND		0.6				
1234789-HpCDF	ND		1.1				
OCDF	ND		1.9				
TOTAL TCDD	ND		0.4				
TOTAL PeCDD	ND		0.7				
TOTAL HxCDD	ND		0.8				
TOTAL HpCDD	1.3	1			1.05		
TOTAL TCDF	ND		0.3				
TOTAL PeCDF	ND		0.5				
TOTAL HxCDF	ND		0.6				
TOTAL HpCDF	ND		0.8				

Reviewed By: | 12/15/92 |

X237_RPT 4.01, LARS 5.0

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b) QA/QC SUMMARY

ILE NAME.:	S925718	SAMPLE ID.:	7328-129		
ROJECT...:	22499	CLIENT NAME...:	INTERPOLL LABS		
LIENT PROJECT:	n/a				
LI ID....:	61-155-2	ANALYSIS DATE.:	12/15/92	CLIENT CODE..:	IPL01
OLLECTED.:	10/27/92	ANALYSIS TIME.:	08:56	DILUTION.....:	n/a
ECEIVED..:	11/30/92	ANALYST.......:	DM	BLANK FILE...:	S925716
ATRIX....:	ASH	INSTRUMENT....:	S	% LIPID......:	n/a
XT. SIZE.:	12.864 g	GC COLUMN.....:	DB-5	% SOLIDS.....:	97.9
DJ. SIZE.:	12.594 g	GC COLUMN ID..:	2568817	% MOISTURE...:	2.1
XT. DATE.:	12/04/92	ICAL NAME.....:	SC50072	ORIGIN.......:	n/a
XT. VOL..:	20.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
PIKE FILE:	SPX2372S	CONCAL NAME...:	S925711	SAS NUMBER...:	n/a
NJECT VOL:	2.0 ul	CONCAL DATE...:	12/15/92	EPISODE......:	n/a

RROGATE RECOVERY SUMMARY (TYPE B)

ME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
Cl-TCDD	12.6	79.6		26:43	
C12-PeCDF 234	97.9	61.7	1.40	30:30	
C12-HxCDF 478	173	109	0.50	33:13	
C12-HxCDD 478	183	115	1.21	33:55	
C12-HpCDF 789	126	79.6	0.42	37:52	

TERNATE STANDARDS RECOVERY SUMMARY (TYPE B)

ME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-HxCDF 789	172	108	0.51	34:36	
3C12-HxCDF 234	164	103	0.48	33:49	

TERNAL STANDARDS RECOVERY SUMMARY

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-2378-TCDF	112	70.8	0.77	26:01	
3C12-2378-TCDD	124	77.8	0.80	26:42	
3C12-PeCDF 123	81.4	51.3	1.35	29:51	
3C12-PeCDD 123	109	68.4	1.56	30:51	
3C12-HxCDF 678	154	96.9	0.50	33:19	
3C12-HxCDD 678	166	105	1.20	34:01	
3C12-HpCDF 678	117	73.4	0.44	36:17	
3C12-HpCDD 678	132	83.4	1.05	37:20	
3C12-OCDD	182	57.2	0.86	41:06	

ECOVERY STANDARDS RECOVERY SUMMARY

AME	RATIO	RT	FLAGS
3C12-1234-TCDD	0.82	26:32	
3C12-HxCDD 789	1.18	34:20	

eviewed By: | 12/16/92 |

X237_RPT 4.01, LARS 5.00

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b)

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FILE NAME.:	S925720	SAMPLE ID.:	7328-134		
PROJECT...:	22499	CLIENT NAME...:	INTERPOLL LABS		
CLIENT PROJECT:	n/a				
TLI ID....:	61-155-4	ANALYSIS DATE.:	12/15/92	CLIENT CODE..:	IPL01
COLLECTED.:	10/27/92	ANALYSIS TIME.:	10:41	DILUTION.....:	n/a
RECEIVED..:	11/30/92	ANALYST.......:	NB	BLANK FILE...:	S925716
MATRIX....:	ASH	INSTRUMENT....:	S	% LIPID......:	n/a
EXT. SIZE.:	10.757 g	GC COLUMN.....:	DB-5	% SOLIDS.....:	99.9
ADJ. SIZE.:	10.746 g	GC COLUMN ID..:	2568817	% MOISTURE...:	0.1
EXT. DATE.:	12/04/92	ICAL NAME.....:	SC50072	ORIGIN.......:	n/a
EXT. VOL..:	20.00 ul	ICAL DATE.....:	10/07/92	CONTRACT.....:	n/a
SPIKE FILE:	SPX2372S	CONCAL NAME...:	S925711	SAS NUMBER...:	n/a
INJECT VOL:	2.0 ul	CONCAL DATE...:	12/15/92	EPISODE......:	n/a

NAME	CONC(ppt)	NUMBER	DL	EMPC	RATIO	RT	FLAGS
2378-TCDD	ND		0.5				
12378-PeCDD	ND		0.6				
123478-HxCDD	ND		1.0				
123678-HxCDD	ND		0.7				
123789-HxCDD	ND		0.9				
1234678-HpCDD	EMPC			1.8			B
OCDD	EMPC			29.7			B
2378-TCDF	ND		0.3				
12378-PeCDF	ND		0.4				
23478-PeCDF	ND		0.4				
123478-HxCDF	ND		0.6				
123678-HxCDF	ND		0.4				
234678-HxCDF	ND		0.6				
123789-HxCDF	ND		0.7				
1234678-HpCDF	ND		0.6				
1234789-HpCDF	ND		1.0				
OCDF	ND		1.8				
TOTAL TCDD	1.2	1		2.0	0.73		
TOTAL PeCDD	ND		0.6				
TOTAL HxCDD	EMPC			0.61			
TOTAL HpCDD	EMPC			3.2			
TOTAL TCDF	ND		0.3				
TOTAL PeCDF	ND		0.4				
TOTAL HxCDF	ND		0.6				
TOTAL HpCDF	ND		0.8				

Reviewed By: | 12/19/92 |

X237_RPT 4.01, LARS 5.C

TRIANGLE LABORATORIES OF RTP, INC.
PCDD/PCDF 2378X ANALYSIS (b) QA/QC SUMMARY

FILE NAME.: S925720
PROJECT...: 22499
CLIENT PROJECT: n/a
TLI ID....: 61-155-4
COLLECTED.: 10/27/92
RECEIVED..: 11/30/92
MATRIX....: ASH
EXT. SIZE.: 10.757 g
ADJ. SIZE.: 10.746 g
EXT. DATE.: 12/04/92
EXT. VOL..: 20.00 ul
SPIKE FILE: SPX2372S
INJECT VOL: 2.0 ul

SAMPLE ID.: 7328-134
CLIENT NAME...: INTERPOLL LABS

ANALYSIS DATE.: 12/15/92
ANALYSIS TIME.: 10:41
ANALYST.......: NB
INSTRUMENT....: S
GC COLUMN.....: DB-5
GC COLUMN ID..: 2568817
ICAL NAME.....: SC50072
ICAL DATE.....: 10/07/92
CONCAL NAME...: S925711
CONCAL DATE...: 12/15/92

CLIENT CODE..: IPL01
DILUTION.....: n/a
BLANK FILE...: S925716
% LIPID......: n/a
% SOLIDS.....: 99.9
% MOISTURE...: 0.1
ORIGIN.......: n/a
CONTRACT.....: n/a
SAS NUMBER...: n/a
EPISODE......: n/a

URROGATE RECOVERY SUMMARY (TYPE B)

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
7Cl-TCDD	18.2	97.6		26:43	___
3C12-PeCDF 234	161	86.7	1.41	30:30	___
3C12-HxCDF 478	221	119	0.50	33:13	___
3C12-HxCDD 478	237	127	1.17	33:55	___
3C12-HpCDF 789	175	93.8	0.43	37:52	___

LTERNATE STANDARDS RECOVERY SUMMARY (TYPE B)

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
3C12-HxCDF 789	231	124	0.49	34:36	___
3C12-HxCDF 234	219	118	0.50	33:48	___

NTERNAL STANDARDS RECOVERY SUMMARY

AME	CONC (ppt)	% REC.	RATIO	RT	FLAGS
13C12-2378-TCDF	181	97.1	0.76	26:01	___
13C12-2378-TCDD	179	96.0	0.78	26:42	___
13C12-PeCDF 123	161	86.3	1.45	29:51	___
13C12-PeCDD 123	191	103	1.42	30:50	___
13C12-HxCDF 678	215	115	0.49	33:19	___
13C12-HxCDD 678	217	117	1.21	34:00	___
13C12-HpCDF 678	165	88.7	0.44	36:16	___
13C12-HpCDD 678	172	92.5	1.04	37:20	___
13C12-OCDD	246	66.0	0.89	41:05	___

RECOVERY STANDARDS RECOVERY SUMMARY

NAME	RATIO	RT	FLAGS
13C12-1234-TCDD	0.80	26:31	___
13C12-HxCDD 789	1.19	34:19	___

Reviewed By: | ____________ 12/19/92|

X237_RPT 4.01, LARS 5.00

Appendix G: Chain of Custody Forms

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-OTP / Big Stone, S.D.

Date of Sampling 10-26-92 Site Boiler / Stack

Test 2 Run 0 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Kaehler Title Field Engineer

Location at which recovery was done Resource Room

Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-10
2.	4" G.F.F. No. 0250	1	-11
3.	XAD-2 Resin	1	-12
4.	Condensate Trap	1	-13
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 10-29-92 Time of receipt 0802 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.	7328-13			
2.		1/8/93	EPA 8080	WAO
3.		12/28/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WAO	
RWW	

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-OTP / Big Stone, S.D.

Date of Sampling 10-26-92 Site Boiler / Stack

Test 2 Run 1 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koehler Title Field Engineer

Location at which recovery was done Resource Room

Special transport/storage requirements? Teflon & Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-14
2.	4" G.F.F. No. 0251	1	-15
3.	XAD-2 Resin	1	-16
4.	Condensate Trap	1	-17
5.	Toluene Rinse	1	-18
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 10-29-92 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.	7328-17			
2.		1/8/93	EPA 8080	WWO
3.		12/28/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WWO	
RWG	Robert W. [illegible]

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-OTP / Big Stone, S.D.

Date of Sampling 10-26-92 Site Boiler / Stack

Test 2 Run 2 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koehler Title Field Engineer

Location at which recovery was done Resource Room

Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-19
2.	4" G.F.F. No. 0252	1	-20
3.	XAD-2 Resin	1	-21
4.	Condensate Trap	1	-22
5.	Toluene Rinse	1	-23
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 10-29-92 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.	7328-22			
2.		1/8/93	EPA 8080	WO
3.		12/28/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WO	Wayne [illegible]
RWW	Robert W. [illegible]

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-OTP / Big Stone, S.D.
Date of Sampling 10-26-92 Site Boiler / Stack
Test 2 Run 3 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:
Signature Mark Kaahler Title Field Engineer
Location at which recovery was done Resource Room
Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-24
2.	4" G.F.F. No. 0253	1	-25
3.	XAD-2 Resin	1	-26
4.	Condensate Trap	1	-27
5.	Toluene Rinse	1	-28
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 10-29-92 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.	7328-27			
2.		1/8/93	EPA 8080	WNO
3.		12/28/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WNO	Wayne
RWW	

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL - OTP / Big Stone, S.D.

Date of Sampling 10-26-92 Site Boiler / Stack

Test 7 Run 0 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Kaehler Title Field Engineer

Location at which recovery was done Resource Room

Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-67
2.	4" G.F.F. No. 0254	1	-68
3.	XAD-2 Resin	1	-69
4.	Condensate Trap	1	-70
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 10-29-92 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.	7328-70			
2.		1/8/93	EPA 8080	
3.		12/29/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
RWW	Robert W. Wynne

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-DTP / Big Stone, S.D.

Date of Sampling 10-27-92 Site Boiler / Stack

Test 7 Run 1 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:

Signature Mark Koehler Title Field Engineer

Location at which recovery was done Resource Room

Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-71
2.	4" G.F.F. No. 0255	1	-72
3.	XAD-2 Resin	1	-73
4.	Condensate Trap	1	-74
5.	Toluene Rinse	1	-75
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 10-29-92 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.	7328-74			
2.		1/8/93	EPA 8080	WDO
3.		12/29/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WDO	
RWW	Robert W. Wayne

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-OTP / Big Stone, S.D.
Date of Sampling 10-27-92 Site Boiler / Stack
Test 7 Run 2 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:
Signature Mark Koehler Title Field Engineer
Location at which recovery was done Recovery Room
Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-76
2.	4" G.F.F. No. 0256	1	-77
3.	XAD-2 Resin	1	-78
4.	Condensate Trap	1	-79
5.	Toluene Rinse	1	-80
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 Duke Title Lab Analyst

Date of receipt 10-24-92 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.	7328-79			
2.		1/8/93	EPA 8080	WAD
3.		12/29/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WAD	Wayne [illegible]
RWW	Robert W. Wynne

S-0157RR-2

Interpoll Laboratories
(612)786-6020

Field Sample Chain Of Custody Sheet

Job ANL-OTP / Big Stone, S.D.
Date of Sampling 10-27-92 Site Boiler / Stack
Test 7 Run 3 Team Leader Ron Rosenthal

Type of Sample Train: Modified Method 5

Sample Collection/Recovery

Person performing sample recovery:
Signature Mark Kaehler Title Field Engineer
Location at which recovery was done Resource Room
Special transport/storage requirements? Teflon + Custody Seals

	Sample Description	No. of Items	Assigned Log Number
1.	Solvent Rinse	1	7328-81
2.	4" G.F.F. No. 0257	1	-82
3.	XAD-2 Resin	1	-83
4.	Condensate Trap	1	-84
5.	Toluene Rinse	1	-85
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 10-24-92 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.	7328-84			
2.		1/8/93	~~EPA~~ 8080	WAO
3.		12/29/92	EPA 8270	RWW
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				

Signature of each analyst:

Initials	Signature
WAO	Wayne [illegible]
RWW	Robert W. Wayne

adprep rev. 3/23/92 [illegible]

TLI # 22133

TRIANGLE LABORATORIES OF RTP, INC.

AIR QUALITY PREPARATION WORK ORDER FORM

PLEASE RETURN THIS FORM WITH THE SAMPLES AFTER THE SAMPLING SESSION

TLI INFORMATION SECTION

Reference No:
P.O. Number: 16934 KE
Customer ID: Interpoll
Order Date: 10 / 14 / 92
Due Date: 10 / 21 / 92
Product Manager: Hani K.

Type & No. of Traps: 8 XAD ____ PUF ____ QA/QC ____ FILTER

SAMPLE HISTORY

XAD Resin Batch No.: Lot # T661-T640-T630 from Supelco
PUF > Sandwich with XAD resin: yes / no
> Screen Required: # T655 yes / no
Preparer G1
# of Blanks 1
Fortification: XAD: [✓ yes] / no → USC-C + USC-S
PUF: yes / no

Spiker/Observer: (D) BRGL exp. 4/16/93 (O) ____
Spiking Date: (D) 10/19/92 (O) ____
Spiking Sln ID: (D) $21-80-3C 100 μL (O) ____ ____ μL
Nominal con.: (D) 0.1 ng/μl (O) ____ ng/ml ____ μg/ml

Nature & Amount of Surrogates:

37C14-2,3,7,8 - TCDD	10	ng
13C12-2,3,4,7,8 - PeCDF	10	ng
13C12-1,2,3,4,7,8 - HxCDF	10	ng
13C12-1,2,3,4,7,8 - HxCDD	10	ng
13C12-1,2,3,4,7,8,9 - HpCDF	10	ng
Other:		ng
		ng

Terphenyl d^{14} ____ μg
____ ng
Other: ____

JOINT PROJECT
[✓ YES] / NO

Special Instructions:
no prespike for organics; Ck with PM when samples come back to the lab for analysis

SHIPPING INFORMATION

Ship to the attention of: Kathy Eickstadt by 10 / 20 / 92 (shipping date)

Mailing Address:
(street address)
Interpoll Labs
4500 Ball Rd N.E.
Circle Pines, MN 55014 - 1819

Air Bill No.:
Phone No: 612 - 786 - 6020

CLIENT INFORMATION SECTION
circle requested analysis

Selections circled by: ____
Date: ____ / ____ / ____

MM5 Front & Back Halves (pooled): yes / no
M23 Toluene Analysis (separate): yes / no
VOST Tandem: yes / no
VOST Separate: yes / no
PUF & Filter Pooled: yes / no

PCDD / PCDF	
Mono through Octachlorinated:	yes / no
Tri through Octachlorinated:	yes / no
Tetra through Octachlorinated:	yes / no
Confirmation of 2,3,7,8-TCDF:	yes / no
Toxicity Equivalency Factor:	yes / no

ORGANICS	
HSL:	yes / no
PAH:	yes / no
PAHH:	yes / no
PCB:	yes / no
PCBH:	yes / no

ILI Project# 7328

Interpoll Laboratories
(612) 786-6020

XAD-2 & VOST Trap Preparation Work Order Form

Project Name: Argone, B. Stone
Order Date: 10/20/92
Date Required: 10/25/92
Delivery Date: 10/23/92

Number of XAD-2 Traps Required: 8

Number of VOST Tubes Required: ________ Tenax
________ Tenax/Charcoal

XAD-2 TRAP HISTORY

Traps Prepared by Tri. Labs

XAD-2 Lot Number: ↓
XAD-2 Lot Date:
XAD-2 Lot Technician:
Date of Spiking: 10/25/92
Technician: TTM

Nature of Spiking Material	Spike Concentration	Spike Volume	Total Mass of Spike
D_{10}-Fluorathene	4.0 ug/ul	5 ul	20.0 ug

VOST TUBE HISTORY

Date of Thermal Disorption: ________ Technician: ________

Total Hydrocarbon Analysis: ____ ppm

SPECIAL REQUIREMENTS

Note - Traps post spiked after prep ! Spiking by Tri Labs

**Please return this form with the samples.

LOA-10RR

RIANGLE LABORATORIES OF RTP, INC.
01 CAPITOLA DRIVE
URHAM, NC 27713

PHONE: (919) 544-5729
FAX: (919) 544-5491

THIS REPORT IS PROVIDED TO ACKNOWLEDGE RECEIPT OF YOUR SAMPLE(S), AND TO ADVISE YOU OF THE STATUS OF YOUR ORDER.

ATHY EICKSTADT
NTERPOLL LABS
500 BALL ROAD, N. E.
IRCLE PINES, MN 55014-1819

TYPE AND NUMBER OF SAMPLES

ASH_4

ROJECT NAME:
URCHASE ORDER NO.: 16934KE
EFERENCE NO.: 0100000625

TRIANGLE LABORATORIES INFORMATION

ATE SAMPLES RECEIVED: 11/30/92

PON RECEIPT, A TLI PROJECT WAS INITIATED TO PROCESS YOUR AMPLES. PLEASE REFER TO THE TLI PROJECT NUMBER GIVEN BELOW WHEN NQUIRING ABOUT THE STATUS OF YOUR SAMPLES:

```
+------------------------------+
|  TLI PROJECT NUMBER: 22499   |
+------------------------------+
```

NTICIPATED DATA PACKAGE SHIP DATE: 12/30/92

ARAMETERS TO BE ANALYZED:

DIOXIN DEPT		ORGANICS DEPT		INORGANICS DEPT		OTHER
D/DF		VOLATILES		M-M TRAIN		
378 TCDF ONLY		SEMIVOLATILES		TCLP		
378 TCDD ONLY		PESTICIDES		METALS IN WATER		
ETRA-OCTA	✓	PCB'S		NON-ROUTINE:		
ONO-OCTA		CBCP'S				
ONFIRMATION	✓	PAH'S				
CDD/DF		AOX/TOX				
THERS:		OTHERS:				

ONTACT(S):
ALES: Vicki Wray
ATA INTERPRETATION: Hani Karam
ROJECT STATUS: Robert Smith

Signature: Mary C. Cole Date: 11/30/92

PAGE 1 OF 1

Custody Seal : Present/Intact
Chain of Custody : Absent
Sample Tags : Present
Sample Tag Numbers : Listed
SMO Forms : Absent

TLI Project Number	22499		Book
Client: IPL01	INTERPOLL LABS		61
Date Received	11/30/92	By	Page
Carrier and Number	UPS#2136-0333-167		155

Ice Chest/Box Ice Temp 40.0 F

Sample ID TLI	Sample ID Client	Matrix	Storage Location	To Lab Date/Initial		To Storage Date/Initial		To Lab Date/Initial	
61-155-1	7328-123	BOTTOM ASH	COOLER#1						
61-155-2	7328-129	BOTTOM ASH	COOLER#1						
61-155-3	7328-126	ESP FLYASH	COOLER#1						
61-155-4	7328-134	ESP FLYASH	COOLER#1						

Receiving Remarks:	Archive Remarks:

TRIANGLE LABORATORIES OF RTP, INC.--CHAIN OF CUSTODY---REVISED 07/23/9

Interpoll Laboratories
(612)786-6020

Sample Custody Transfer Form

Job ANL-OTP / Big Stone, S.D. Date 11-2-92

Interlaboratory transfer:
Person in custody of samples ____ Init. ____
Person to receive custody ____ Init. ____

Out of laboratory transfer:
Date shipped 11-2-92 Date received ____
Method of shipment Air Carrier Federal Express
Person releasing samples Bill Drake Init. B.D.
Person receiving custody ____ Init. ____

		Sample Log Number	Initials of Person in Custody of Samples	Initials of Person Receiving Samples
T_7R_0	1.	7328-67	B.D.	BH
	2.	-68	B.D.	
	3.	-69	B.D.	
T_7R_1	4.	7328-71	B.D.	
	5.	-72	B.D.	
	6.	-73	B.D.	
	7.	-75	B.D.	
T_7R_2	8.	7328-76	B.D.	
	9.	-77	B.D.	
	10.	-78	B.D.	
	11.	-80	B.D.	
T_7R_3	12.	7328-81	B.D.	
	13.	-82	B.D.	
	14.	-83	B.D.	
	15.	-85	B.D.	
	16.			
	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.

TRIANGLE LABORATORIES OF RTP, INC.
801 CAPITOLA DRIVE
DURHAM, NC 27713

PHONE: (919) 544-5729
FAX: (919) 544-5491

THIS REPORT IS PROVIDED TO ACKNOWLEDGE RECEIPT OF YOUR SAMPLE(S), AND TO ADVISE YOU OF THE STATUS OF YOUR ORDER.

KATHY EICKSTADT
INTERPOLL LABS
4500 BALL ROAD, N. E.
CIRCLE PINES, MN 55014-1819

TYPE AND NUMBER OF SAMPLES

M23UNITS_8

PROJECT NAME: ANL-OTP/BIG STONE, S.D.
PURCHASE ORDER NO.: 16934KE
REFERENCE NO.: 0100000625

TRIANGLE LABORATORIES INFORMATION

DATE SAMPLES RECEIVED: 11/04/92

UPON RECEIPT, A TLI PROJECT WAS INITIATED TO PROCESS YOUR SAMPLES. PLEASE REFER TO THE TLI PROJECT NUMBER GIVEN BELOW WHEN INQUIRING ABOUT THE STATUS OF YOUR SAMPLES:

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+------------------------------+
| TLI PROJECT NUMBER: 22314    |
+------------------------------+
```

ANTICIPATED DATA PACKAGE SHIP DATE: 12/04/92

PARAMETERS TO BE ANALYZED:

DIOXIN DEPT
DD/DF ____
2378 TCDF ONLY ____
2378 TCDD ONLY ____
TETRA-OCTA ✓
MONO-OCTA ____
CONFIRMATION ✓
TCDD/DF ____
OTHERS: ____

ORGANICS DEPT
VOLATILES ____
SEMIVOLATILES ____
PESTICIDES ____
PCB'S ____
CBCP'S ____
PAH'S ____
AOX/TOX ____
OTHERS: ____

INORGANICS DEPT
M-M TRAIN ____
TCLP ____
METALS IN WATER ____
NON-ROUTINE: ____

OTHER

CONTACT(S):
SALES: Vicki Wray
DATA INTERPRETATION: Hani Karam
PROJECT STATUS: Robert Smith

Signature: Mary C. Collins Date: 11/6/92

Custody Seal : Present/Intact	TLI Project Number	22314	Book
Chain of Custody : Present	Client: IPL01	INTERPOLL LABS	51
Sample Tags : Present	Date Received	11/04/92 By B Hurst	Page
Sample Tag Numbers : Listed			
SMO Forms : Absent			
Ice Chest Ice Temp 40.0 F	Carrier and Number	FEDX.4645022756	11

Sample ID TLI	Client	Matrix	Storage Location	To Lab Date/Initial	To Storage Date/Initial	To Lab Date/Initial
61-11-1A	TEST 2/0-F.B.-7328-10	SOLVENT RINSE	COOLER#1			
61-11-1B	TEST 2/0-F.B.-7328-11	FILTER	COOLER#1			
61-11-1C	TEST 2/0-F.B.-7328-12	XAD RESIN	COOLER#1			
61-11-2A	TEST 2/1-STACK-7328-14	SOLVENT RINSE	COOLER#1			
61-11-2B	TEST 2/1-STACK-7328-15	FILTER	COOLER#1			
61-11-2C	TEST 2/1-STACK-7328-16	XAD RESIN	COOLER#1			
61-11-2D	TEST 2/1-STACK-7328-18	TOLUENE RINSE	COOLER#1			
61-11-3A	TEST 2/2-STACK-7328-19	SOLVENT RINSE	COOLER#1			
61-11-3B	TEST 2/2-STACK-7328-20	FILTER	COOLER#1			
61-11-3C	TEST 2/2-STACK-7328-21	XAD RESIN	COOLER#1			
61-11-3D	TEST 2/2-STACK-7328-23	TOLUENE RINSE	COOLER#1			
61-11-4A	TEST 2/3-STACK-7328-24	SOLVENT RINSE	COOLER#1			
61-11-4B	TEST 2/3-STACK-7328-25	FILTER	COOLER#1			
61-11-4C	TEST 2/3-STACK-7328-26	XAD RESIN	COOLER#1			
61-11-4D	TEST 2/3-STACK-7328-28	TOLUENE RINSE	COOLER#1			
61-11-5A	TEST 7/0-F.B.-7328-67	SOLVENT RINSE	COOLER#1			
61-11-5B	TEST 7/0-F.B.-7328-68	FILTER	COOLER#1			

Receiving Remarks:	Archive Remarks:

TRIANGLE LABORATORIES OF RTP, INC.--CHAIN OF CUSTODY---REVISED 07/23/92

Custody Seal	: Present/Intact	TLI Project Number	22314	Bo
Chain of Custody	: Present	Client: IPL01	INTERPOLL LABS	6.
Sample Tags	: Present	Date Received	11/04/92 ¦ By	Pa
Sample Tag Numbers	: Listed			
SMO Forms	: Absent			
Ice Chest	Ice Temp 40.0 F	Carrier and Number	FEDX.4645022756	1

Sample ID			Storage	To Lab		To Storage		To Lab	
TLI	Client	Matrix	Location	Date/Initial		Date/Initial		Date/Initial	
61-11-5C	TEST 7/0-F.B.-7328-69	XAD RESIN	COOLER#1						
61-11-6A	TEST 7/1-STACK-7328-71	SOLVENT RINSE	COOLER#1						
61-11-6B	TEST 7/1-STACK-7328-72	FILTER	COOLER#1						
61-11-6C	TEST 7/1-STACK-7328-73	XAD RESIN	COOLER#1						
61-11-6D	TEST 7/1-STACK-7328-75	TOLUENE RINSE	COOLER#1						
61-11-7A	TEST 7/2-STACK-7328-76	SOLVENT RINSE	COOLER#1						
61-11-7B	TEST 7/2-STACK-7328-77	FILTER	COOLER#1						
61-11-7C	TEST 7/2-STACK-7328-78	XAD RESIN	COOLER#1						
61-11-7D	TEST 7/2-STACK-7328-80	TOLUENE RINSE	COOLER#1						
61-11-8A	TEST 7/3-STACK-7328-81	SOLVENT RINSE	COOLER#1						
61-11-8B	TEST 7/3-STACK-7328-82	FILTER	COOLER#1						
61-11-8C	TEST 7/3-STACK-7328-83	XAD RESIN	COOLER#1						
61-11-8D	TEST 7/3-STACK-7328-85	TOLUENE RINSE	COOLER#1						

Receiving Remarks:	Archive Remarks:

CHAIN OF CUSTODY

SAMPLER'S SIGNATURE: ____________

CO. NAME: Interpoll Labs Inc

ADDRESS: 4500 Ball Road N.E.
Circle Pines,
Minnesota 55014-1819

PHONE #: 612, 786, 6020

CONTACT NAME: Daniel Despen

PROJECT NAME/#: 22314

PO#: 16934KE

STA NO.	DATE	TIME	COMP	~~GRAB~~ Extraction Date	SAMPLE I.D.#	NO. OF CONTAINERS	PAH	PCB					REMARKS
0	11/10/92	9:52	[illegible]	11/10/92	TLI BLANK	1	X	X					10 ml (1/2 EXTRACT)
1	↓	↓	↓	↓	TEST 2/0 - FB - 7328	1	↓	↓					↓ ↓
2					TEST 2/1 - STACK - 7328	1							
3					TEST 2/2 - STACK - 7328	1							
4					TEST - 2/3 - STACK - 7328	1							
5					TEST - 7/0 - FB - 7328	1							
6					TEST 7/1 - STACK - 7328	1							
7					TEST 7/2 - STACK - 7328	1							
8					TEST 7/3 - STACK - 7328	1							

RELINQUISHED BY: SIGN.	DATE / TIME 11/10/92 10:31	RECEIVED BY: SIGN.	RELINQUISHED BY: SIGN.	DATE / TIME	RECEIVED BY: SIGN.
RELINQUISHED BY: SIGN.	DATE / TIME	RECEIVE BY: SIGN.	RELINQUISHED BY: SIGN.	DATE / TIME	RECEIVED BY: SIGN.

RECEIVED FOR LABORATORY BY: SIGNATURE	DATE/TIME 11/13/92 0800	SEND SAMPLES TO: TRIANGLE LABORATORIES OF RTP, INC. 801 CAPITOLA DRIVE DURHAM, NORTH CAROLINA 27713

FT2/COC

5/21/92

Appendix H: CEM Calibration Specifications

INTERPOLL LABORATORIES

Calibration Error Check

Job ARGONNE/BIG STONE

Test 1 Run ____ Date 10-26-92

Operator ____

SO$_2$ Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0	0	0	500	0
Mid level	229	229	0	500	
High level	495	495	0	500	0

NO$_x$ Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0	0	0	500	0
Mid level	251	251		500	
High level	478	476	2	500	

O$_2$ Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0	0	0	25	0
Mid level	13.5	13.5		25	
High level	20.9	20.8	.1	25	

CO$_2$ Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0	0	0	20	
Mid level	11.0	10.9	.1	20	
High level	17.0	17.0		20	

Must be within 2% of the span for each calibration gas S-420-10

INTERPOLL LABORATORIES

Calibration Error Check

Job ARGONNE/BIG STONE

Test 1 Run ____ Date 10-26-92

Operator ____

SO_2 Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0				
Mid level					
High level					

NO_x Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0				
Mid level					
High level					

O_2 Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0				
Mid level					
High level					

CO Calibration: Time(HRS) ____

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0	0	0	500	
Mid level	249	250	1	500	
High level	142	142		500	

Must be within 2% of the span for each calibration gas

S-420-10

INTERPOLL LABORATORIES
(612) 786-6020

SO_2 System Bias Check

Job ARGONNE/BIG STONE Source Boiler No1
Test 1 Run 1 Date 10-26-92 Site Stack
Operator

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0830	Zero gas	0		0	0	500	0
		Upscale	229		229	0	500	0
2	1930	Zero gas	0				1000	
		Upscale	859.2		849	10	1000	1.0
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

NOx System Bias Check

Job ARGONNE/BIG STONE Source NO1 BOILER
Test 1 Run 1 Date 10-26-92 Site STACK
Operator

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0920	Zero gas	0		0	0	500	0
		Upscale	251		253	2	500	.4
2	1930	Zero gas	0		2	2	500	.2
		Upscale	478		477	1	500	.2
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

CO System Bias Check

Job ARGONNE/BIG STONE Source NO 1 BOILER
Test 1 Run 1 Date 10-26-92 Site STACK
Operator

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm) Cal Err	Analyzer Resp (ppm) Sys Bias	Diff. CE-SB (ppm)	Span Val (PPM)	% of span
1	1920	Zero gas	0		0	0	500	0
		Upscale	249		247	2	500	
2	1930	Zero gas	0		1	1	500	
		Upscale	249		247	7	500	
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

CO_2 System Bias Check

Job ARGONNE
Test 1 Run 1 Date 10-26-92
Operator

Source No 1 Boiler
Site Stack

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0830	Zero gas	0		0	0	20	0
		Upscale	11.0		11	0	20	0
2	1930	Zero gas	0		0	0	20	0
		Upscale	11.0		11.0	0	20	0
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

O2 System Bias Check

Job ARGONNE/BIG STONE Source No1 BOILER
Test 1 Run 1 Date 10-26-92 Site STACK
Operator

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm) Cal Err	Analyzer Resp (ppm) Sys Bias	Diff. CE-SB (ppm)	Span Val (PPM)	% of span
1	0920	Zero gas	0		0	0	25	0
		Upscale	13.5		13.6	.1	25	.4
2	1930	Zero gas	0		0	0	25	0
		Upscale	13.5		13.5	0	25	0
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

SO_2 System Bias Check

Job ARGONNE/BIG STONE Source NO 1 BOILER
Test 2 Run 1 Date 10-27-92 Site STACK
Operator E TROWBRIDGE

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0835	Zero gas	0		0	0	1000	0
		Upscale	859.2		859	.2	1000	.02
2	1800	Zero gas	0		4	4	1000	.4
		Upscale	859.2		853	6.2	1000	.62
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

NOx System Bias Check

Job ARGONNE / BIG STONE
Source No 1 BOILER
Test 2 Run 1 Date 10-27-92
Site STACK
Operator STROWBRIDGE

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0835	Zero gas	0		1	1	1000	.1
		Upscale	478		477	1	1000	.1
2	1800	Zero gas	0			1	1000	.1
		Upscale	478		479	1	1000	.1
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

CO System Bias Check

Job ARGONNE/BIG STONE Source NO1 BOILER
Test 2 Run 1 Date 10-27-92 Site STACK
Operator

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0835	Zero gas	0		0	0	500	0
		Upscale	249		247	2	500	.4
2	1800	Zero gas	0		2	2	500	.4
		Upscale	249		242	7	500	1.4
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

CO2 System Bias Check

Job ARGONNE / BIG STONE Source No 1 Boiler
Test 2 Run 1 Date 10-27-92 Site STACK
Operator

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0835	Zero gas	0		0	0	20	0
		Upscale	11.0		11.0	0	20	0
2	1000	Zero gas	0		0.1	0.1	20	.8
		Upscale	11.0		10.9	.1	20	.8
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
(612) 786-6020

O_2 System Bias Check

Job ARGONNE/BIG STONE
Test 2 Run 1 Date 10-29-92
Operator

Source NO1 BOILER
Site STACK

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0835	Zero gas	0		0	0	25	0
		Upscale	13.5		13.5	0	25	0
2	1800	Zero gas	0		0	0	25	0
		Upscale	13.5		13.4	.1	25	.4
3		Zero gas	0					
		Upscale						
4		Zero gas	0					
		Upscale						
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

INTERPOLL LABORATORIES
EPA Method 25A
Calibration Error Check & Drift Determination

Job ADL / OTP BIG STONE

Test 0+1 Run 1+2 Date 10-26 to 10-27
Operator BOB

THC Calibration (Low Range): Time(HRS) 0700

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0	1	1	100	1.0
Low level	31.10	31.0	.10	100	.10
Mid level	310	300	10	1000	1.0
High level	2996	2990	6	10000	.06

THC Calibration (High Range): Time(HRS) ______

***	Cylinder Value (ppm)	Analyzer Response (ppm)	Difference (ppm)	Span Value (ppm)	Percent of Span
Zero gas	0				
Span					

O_2 Calibration: Time(HRS) ______

***	Cylinder Value (%)	Analyzer Response (%)	Difference (%)	Span Value (%)	Percent of Span
Zero gas	0				
Mid level					
High level					

CO_2 Calibration: Time(HRS) ______

***	Cylinder Value (%)	Analyzer Response (%)	Difference (%)	Span Value (%)	Percent of Span
Zero gas	0				
Mid level					
High level					

Must be within 2% of the span for each calibration gas

9-420-14

INTERPOLL LABORATORIES
(612) 786-6020

THC System Bias Check

Job ANL 10TP Big Stone Source Boiler
Test 6+11 Run 1+2 Date 10-26 to 10-27 Site Stack
Operator Bob

Run	Time (HRS)	***	Cylinder Value (ppm)	Analyzer Resp (ppm)		Diff. CE-SB (ppm)	Span Val (PPM)	% of span
				Cal Err	Sys Bias			
1	0930	Zero gas	0	1	2	1	100	1.0
		Upscale	31.10	31.0	31.50	.50	100	.50
2	1930	Zero gas	0	1	3	2	100	2.0
		Upscale	31.10	31.0	31.0	0	100	0
3	0845	Zero gas	0	1	2	1	100	1.0
		Upscale	31.10	31.0	31.0	0	100	0
4	1900	Zero gas	0	1	1	1	100	0
		Upscale	31.10	31.0	31.0	0	100	0
5		Zero gas	0					
		Upscale						
6		Zero gas	0					
		Upscale						
7		Zero gas	0					
		Upscale						
8		Zero gas	0					
		Upscale						
9		Zero gas	0					
		Upscale						
10		Zero gas	0					
		Upscale						
11		Zero gas	0					
		Upscale						
12		Zero gas	0					
		Upscale						

Must be within 5% of the span for the zero or upscale cal. gas.

S420-11R

SPECIFICATIONS FOR SO_2 ANALYZER
WESTERN RESEARCH MODEL 721ATZ

Measuring principle	NDUV double beam method which uses 285 nm UV light for SO_2 measurement and 585 nm visible light to compensate for contamination of all windows, detector drift or changes in the intensity of the radiation source
Range	SO_2: 0 - 500 ppm and 0 - 1000 ppm; but low range may be reduced to 0 - 100 ppm with full scale analog output; total dynamic range of 0 - 5000 ppm with 1 ppm readability
Accuracy	±2% f.s., worst case. Typically better than ±1% f.s.
Temperature drift	≤0.5% f.s./°C
Noise	0.5% of full scale, worst case
Ambient temperature	0 to 40 °C
Ambient humidity	Less than 100% RH
Response time (90% of final reading)	<5 seconds
Optical cell length	35 cm
Output signal	Panel display is digital - direct reading in ppm,w; output signal: 7 field-selectable potentiometric outputs of 1V, 2V, 5V, 10V DC and 100, 200, 500 mV DC. Two outputs per range are provided at the rear of the

	instrument, standard. Unit equipped also with 4 - 20 mA
Interferences	No known interferences from O_2, CO_2, CO or hydrocarbons; internally compensated for NO interference
Linearity	±1.5% of full scale
Power supply	AC 115 V ± 10%, 60 Hz
Power consumption	Less than 575 watts
Electronic span value	Nominal 766 @ 77 °F and 29.92 in Hg
Sample gas flow	1.0 - 5.0 LPM to give desired response time
Sample gas temperature	0 to 40 °C
Warmup time	Approximately 30 minutes
External dimensions	7 x 19 x 22 (H x W x D) inch
Weight	40 LB

Thermo
Electron
CORPORATION

MODEL 10A
RACK-MOUNTED
CHEMILUMINESCENT
NO-NO_x GAS ANALYZER
SPECIFICATIONS

Sensitivity	Each instrument is equipped with the folling ranges: 0 - 2.5 ppm 0 - 10 ppm 0 - 25 ppm 0 - 100 ppm 0 - 250 ppm 0 - 1000 ppm 0 - 2500 ppm 0 - 10000 ppm
Accuracy	Derived from the NO or NO_2 calibration gas, ±1% of fullscale.
Response time (0-90%) Typical	1.5 seconds - NO Mode 1.7 seconds - NO_x Mode
Output	0 - 10mV and 0 - 10V standard. Other outputs available upon request.
Zero Drift	Negligible after 1/2-hour warm-up.
Linearity	±1% of full scale.
Input Power Requirements	Standard: 115v/50Hz; 115v/60Hz Optional: 220v/50Hz; 220v/60Hz

Thermo Electron Corporation
Instruments Division
8 West Forge Parkway
Franklin, Massachusetts 02038
(617) 520-0430

THERMO ENVIRONMENTAL INSTRUMENTS INC.
Model 10A
Chemiluminescent Gas Analyzer

INTERFERENCE RESPONSE TEST

DATE OF TEST JANUARY 18, 1980

ANALYZER TYPE 10A/R Range 0 - 2.5 PPM **SERIAL NO.** 10 A/R - 014R-80

TEST GAS TYPE	CONCENTRATION PPM	ANALYZER OUTPUT RESPONSE	% OF SPAN
CO	500	< .1 PPM	< .1 %
SO_2	201	< .1 PPM	< .1 %
CO_2	10%	< .1 PPM	< .1 %
O_2	20.9 %	< .1 PPM	< .1 %

SPECIFICATIONS FOR ACS MODEL 3300 CO NDIR

Measuring principle	NDIR single beam method
Operating ranges	0 - 500 ppm 0 - 1000 ppm
Reproducibility	±0.5% of full scale
Stability	Zero drift; ±% of full scale/24H Span drift; ±% of full scale/24H
Noise	0.5% of full scale
Ambient temperature	-5 to 45°C
Ambient humidity	Less than 90% RH
Response time (90% of final reading)	Electrical system; 2 sec, 3 sec, 5 sec (selectable with connector) Response of actual gas; Within 15 sec (depending on cell length)
Indicator	100 linear division
Output signal	OUTPUT 1; DC 0 - 1 V OUTPUT 2; DC 0 - 10 mV or DC 0 - 100 mV or DC 0 - 1 V or DC 4 - 20 mA (Allowable load resistance 500Ω max.)
Linearity	Better than ±2% of full scale (when linearizer is used)
Power supply	AC 115 V ± 10%, 60 Hz

Power consumption	Approx. 30 VA
Materials of gas-contacting parts	Measuring cell; SUS304 Window; CaF2 Piping; Polyethylene
Sample gas flow rate	1ℓ/min ± 0.5ℓ/min
Sample gas temperature	0 to 55°C
Purging gas flow rate	1ℓ/min (to be flowed as occasion demands)
Warmup time	Approx. 2 hours
External dimensions	200 x 250 x 541 (H x W x D) mm
Weight	Approx. 11 kg
Finish Color	MUNSELL N1.5

Remarks: For combinations of measuring ranges for the dualcomponent analyzer, inquiry should be made to the manufacturer.

Ratfisch
INSTRUMENTE

TOTAL HYDROCARBON ANALYZER (FLAME IONIZATIO
Model RS 55

TECHNICAL DATES

MAINS: 115V/60H
RECORDER OUTPUT: 0 - 5 V / 4-20mA

MODEL: X Manual switching
___ Solenoid valves
HOUSING: ___ Case, ___ 19"-Rack
MEASURING RANGES: 1 = 0 - 10 C_1-
2 = 0 - 100 C_1
3 = 0 - 1,000 C_1
4 = 0 - 10,000 C_1

SPECIAL OPTIONS:

Flame out alarm ....
1 Alarm ....
Sample line ....
....

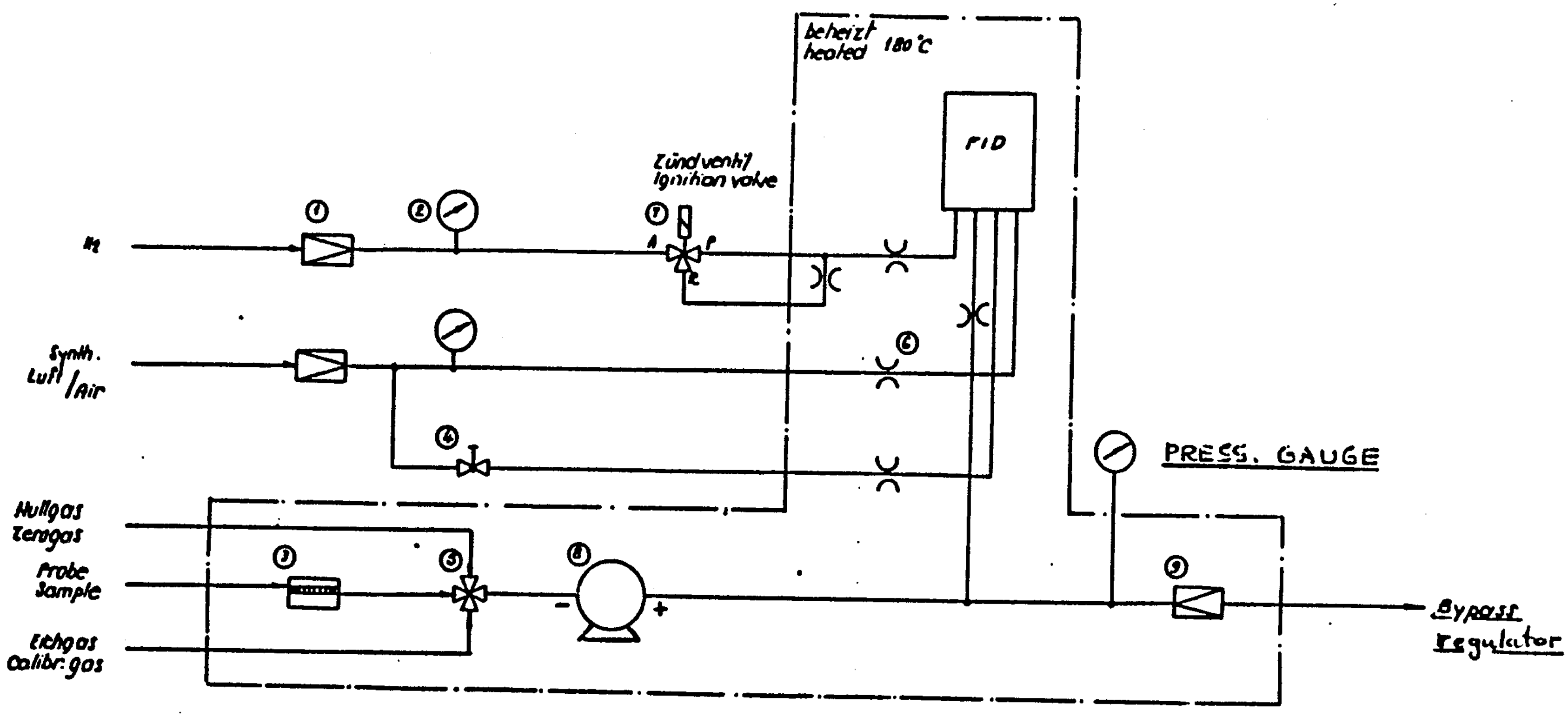
ANALYZER CONDITIONS:

Temperature: ..160.°C

Zero Point: 3,90...
Gain: ....7,70...

Pressure Setting: Sample/Spangas/Zerogas: 200 mbar
Fuel: Hydrogen: ....0,35 bar
Combustion Air: ....0,80 bar

Span Gases: ..300 ppm C_1
24000 ppm C_1



1 Druckregler
Pressure regulator
2 Manometer
Gauge
3 Filter
4 Nadelventil
Needle valve
5 3 Wege ventil
3 way valve
6 Kapillare
capillary
7 Magnet ventil
solenoid valve
8 Pumpe
pump
9 Rückdruckregler
Back pressure regulator

Zündventil
Ignition valve

P-A angezogen
energised
R-A stromlos
at rest

Handumschaltung
Manuel switching

Ratfisch
INSTRUMENTE

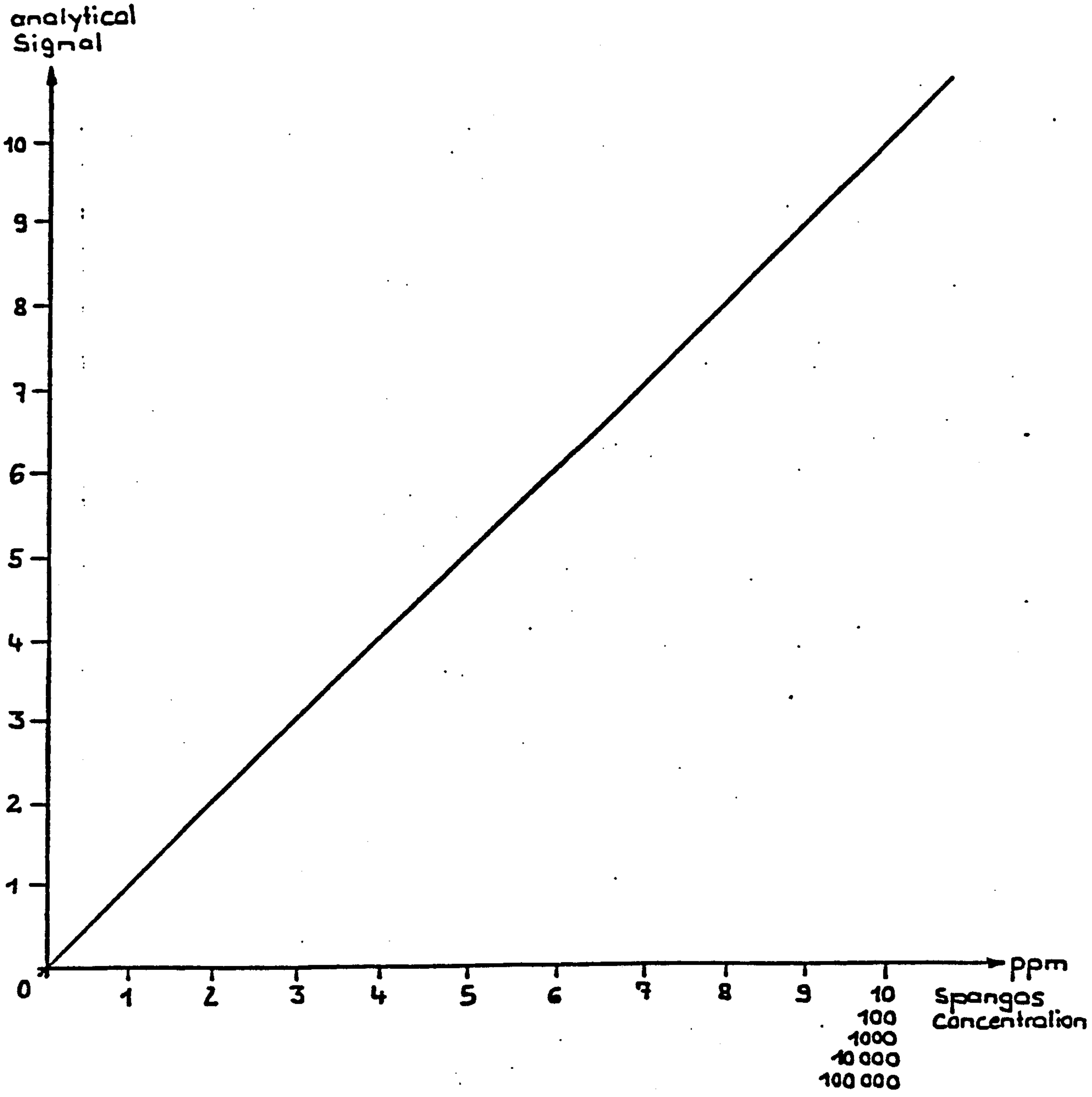
FLAMMEN IONISATIONS DETEKTOR
Flame Ionisation Detector

Fließplan
Flowdiagram

RS 55

13.04.88

CALIBRATION DIAGRAMM



SPECIFICATIONS FOR ACS MODEL 3300 CO_2 NDIR

Measuring principle	NDIR single beam method
Measurable gas components and measuring range	0 - 20%
Reproducibility	±0.5% of full scale
Stability	Zero drift; ±% of full scale/24H Span drift; ±% of full scale/24H
Noise	0.5% of full scale
Ambient temperature	-5 to 45°C
Ambient humidity	Less than 90% RH
Response time (90% of final reading)	Electrical system; 2 sec, 3 sec, 5 sec (selectable with connector) Response of actual gas; Within 15 sec (depending on cell length)
Indicator	100 linear division
Output signal	OUTPUT 1; DC 0 - 1 V OUTPUT 2; DC 0 - 10 mV or DC 0 - 100 mV or DC 0 - 1 V or DC 4 - 20 mA (Allowable load resistance 500Ω max.)
Linearity	Better than ±2% of full scale (when linearizer is used)
Power supply	AC 115 V ± 10%, 60 Hz

Power consumption	Approx. 30 VA
Materials of gas-contacting parts	Measuring cell; SUS304 Window; CaF2 Piping; Polyethylene
Sample gas flow rate	1ℓ/min ± 0.5ℓ/min
Sample gas temperature	0 to 55°C
Purging gas flow rate	1ℓ/min (to be flowed as occasion demands)
Warmup time	Approx. 2 hours
External dimensions	200 x 250 x 541 (H x W x D) mm
Weight	Approx. 11 kg
Finish Color	MUNSELL N1.5

Remarks: For combinations of measuring ranges for the dualcomponent analyzer, inquiry should be made to the manufacturer.

INTERPOLL LABORATORIES
4500 BALL ROAD N.E.
CIRCLE PINES, MN 55014-1819
(612) 786-6020

Servomex

1420
Oxygen Analyser
Instruction Manual

Ref : 01420/001A/0

Order as part No. 01420001A

was (7982-2842)

INTERPOLL LABORATORIES
4500 BALL ROAD N.E.
CIRCLE PINES, MN 55014-1819
(612) 786-6020

1.3 Sampling System

The sampling system of the analyser includes a combination filter/automatic flow control device, designed to keep a constant flow of sample gas through the measuring cell for varying input pressures and to prevent the entrance of particulate matter into the measuring cell. Excess flow is vented to the by-pass.

1.4 Specification

Performance Specification (typical)

Repeatability:	Better than +/-0.2% O2 under constant conditions.
Drift:	Less than 0.2% O2 per week under constant conditions. (Excluding variation due to barometric pressure changes; reading is proportional to barometric pressure.)

Outputs

Display:	3 1/2 digit LCD reading 0.0 to 100.0% oxygen with overrange capability.
Output:	0 to 1V (non-isolated) for 0 to 100% oxygen available on 'D' type connector located on the back panel of the instrument. Output impedance is less than 10 ohms.
Option:	4 - 20mA isolated, Max impedance 500 ohms.
Flow alarm output:	Change over relay contact rated at 3A/115V ac, 1A/240V ac or 1A/28V dc. 4 sets of single pole changeover contacts. Alarm becomes active when sample gas flow through the analyser fails.

Sample requirements

Condition:	Clean, dry gas with dew point 5 deg C below ambient temperature.
Inlet pressure:	0.5 to 3psig (3.5 to 21kPa). Inlet pressure changes within this range will change the reading by less than 0.1% O2. May be operated up to 10psig (70kPa) with degraded stability.
Flowrate:	1.5 to 6 litres/minute approximately depending on sample pressure.
Filtering:	0.6 micron replaceable filter integral to the automatic flow control device.

Response time:	Less than 15 secs. to 90% at an inlet pressure of 3psig (21kPa).
Inlet/vent connections:	1/4 inch OD tube (stainless steel) suitable for 6mm ID flexible tubing or 1/4 inch OD compression fittings.
Materials exposed to the sample:	Stainless steel, Pyrex glass, brass, platinum, epoxy resin, Viton, polypropylene and glass fibre filter.

Physical Characteristics

Case:	Steel and aluminium finished in epoxy powder paint.
Case classification:	IP 20 (IEC 529) when fitted into the Servomex 1400 series 19 inch case.
Dimensions:	See Figure 2.1.
Weight:	10Kg (22lb) approximately.

Electrical

AC Supply:	110 to 120V AC or 220 to 240V AC, +/-10%, 48 to 62Hz. Voltage selected by a voltage selector integral to the IEC supply plug.
Power required:	15VA maximum.

Environmental Limits

Operating ambient temperature:	0 to +40 deg C (32 to 104 deg F)
Storage temp. range:	-20 to +70 deg C (-4 to 158 deg F)
Relative humidity:	0-85%, non-condensing.

LINDE

Union Carbide Industrial Gases, Inc.
Linde Division
4550 Kennedy Avenue
East Chicago, IN 46312

DATE : NOVEMBER 9, 1990

: OXYGEN SERVICE COMPANY
1111 PIERCE BUTLER ROUTE
ST PAUL, MN 55104

LINDE ORDER NUMBER : 310.031.02
CUSTOMER PO NUMBER : 3023SA
CUSTOMER REL NUMBER :

AR SIR/MADAM:

IS IS YOUR CERTIFICATE OF ANALYSIS FOR:

LINDER MBER	MIXTURE COMPONENTS	REQUESTED COMPOSITION	CERTIFIED COMPOSITION	CERTIFICATION ACCURACY
6433	CARBON DIOXIDE	17%	17.00%	± 0.02% ABS
	OXYGEN	21%	21.00%	± 0.02% ABS
	NITROGEN	BALANCE	BALANCE	

RECEIVED
NOV 15 1990
INTERPOLL LABORATORIES

APPROVED BY

OA-6MP

GENEX

GenEx
4018 Duncan Avenue
St. Louis, Missouri 63110
314 371 1016
314 534 6113 fax

July 30,1991
GenEx Order No.:559
Customer P.O. No.:4972SA

Genex Ltd.
2455 Cleveland Ave. N.
Roseville Mn. 55113

Dear Sir:

This is your Certificate of Analysis for:

Cylinder Number	Mixture Components	Requested Composition	Certified Composition
K 461889	Carbon Dioxide	11.0%	11.0%
K 307403	Oxygen	13.5%	13.5%
	Nitrogen	Balance	Balance

Very truly yours,

Randy L. Renner
Lab Technician

Key to Analytical Techniques
A. Gas Chromatograph with Thermal Conductivity Detector
B. Gas Chromatograph with Flame Ionization Detector
C. Gas Chromatograph with Ultrasonic Detector
D. Specific Oxygen Analyzer
E. Specific Water Analyzer
F. Total Hydrocarbon Analyzer
G. Infrared
H. Proprietary
I. Gas Chromatograph with Helium Ionization Detector
J. Flame Ionization with Methanizer
K. Gas Chromatograph — Photo Ionization
L. Gas Chromatograph — Flame Photometric
M. Mass Spectrometry
N. Wet Chemical

Scott Specialty Gases, Inc.

1290 COMBERMERE STREET, TROY, MI 48083-0000
PHONE: 313-589-2950 FAX:313-589-2134

8/02/91

GENEX
4018 DUNCAN AVENUE

ST LOUIS MO 63110-0000

PROJECT #: 05-26600
PO #: 4970SA

CYLINDER #: AAL19103

ANALYTICAL ACCURACY: +-2%

COMPONENT	REQUESTED CONCENTRATION		ANALYSIS 1 (MOLES)	U/M
CARBON MONOXIDE	250.0	PPM	249.	PPM
NITROGEN		BALANCE		N/A

NOTES: CERTIFIED MASTER GAS

ANALYTICAL METHOD: CMG

DATE OF ANALYSIS: 7/29/91

ANALYST: ANALYST

APPROVED BY: J. Shapiro SUPERVISOR

Scott Specialty Gases, Inc.

1290 COMBERMERE STREET, TROY, MI 48083-0000
PHONE: 313-589-2950 FAX:313-589-2134

8/02/91

GENEX
4018 DUNCAN AVENUE

ST LOUIS MO 63110-0000

PROJECT #: 05-29600
PO #: 4970SA

CYLINDER #: AAL20926

ANALYTICAL ACCURACY: +-2%

COMPONENT	REQUESTED CONCENTRATION		ANALYSIS 1 (MOLES)	U/M
SULFUR DIOXIDE	500.0	PPM	495.	PPM
NITROGEN		BALANCE		N/A

NOTES: CERTIFIED MASTER GAS

ANALYTICAL METHOD: CMG

DATE OF ANALYSIS: 7/29/91

ANALYST: ____________
ANALYST

APPROVED BY: J Shapiro
SUPERVISOR

Scott Specialty Gases, Inc.

1290 COMBERMERE STREET, TROY, MI 48083-0000 7/01/91
PHONE: 313-589-2950 FAX:313-589-2134

GENEX
2455 CLEVELAND AVENUE N.
ROSEVILLE MN 55113-0000

PROJECT #: 05-24722
PO #: 4922SA #108

CYLINDER #: AAL20839 ANALYTICAL ACCURACY: +-2%

COMPONENT	REQUESTED CONCENTRATION		ANALYSIS 1 (MOLES)	U/M
NITRIC OXIDE	475.0	PPM	476.	PPM
NITROGEN - OXYGEN FREE		BALANCE		N/A

NOTES: NOX 478 PPM NITROGEN DIOXIDE 2 PPM

CERTIF

ANALYTICAL METHOD: CMG

DATE OF ANALYSIS: 6/05/91

ANALYST: ______________ APPROVED BY: Bob ______________
ANALYST SUPERVISOR

RECEIVED
JUL 08 1991
GEN EX, LTD.
ROSEVILLE MN

Scott Specialty Gases, Inc.

1290 COMBERMERE STREET, TROY, MI 48083-0000 7/01/91
PHONE: 313-589-2950 FAX:313-589-2134

GENEX
2455 CLEVELAND AVENUE N.

ROSEVILLE MN 55113-0000

PROJECT #: 05-24939
PO #: 4930SA # 116

CYLINDER #: ALM013044

ANALYTICAL ACCURACY: +-2%

COMPONENT	REQUESTED CONCENTRATION		ANALYSIS 1 (MOLES)	U/M
NITRIC OXIDE	250.0	PPM	250.	PPM
NITROGEN - OXYGEN FREE		BALANCE		N/A

NOTES: CERTIFIED MASTER GAS
NOX 251 PPM NITROGEN DIOXIDE 1 PPM

ANALYTICAL METHOD: CMG

DATE OF ANALYSIS: 6/14/91

ANALYST: ____________ APPROVED BY: ____________
ANALYST SUPERVISOR

RECEIVE
JUL 08 1991
GEN EX, LTD.
ROSEVILLE MN

Scott Specialty Gases

TROY, MI 48083 PHONE: 313-589-2950 FAX NO.: 313-589-2134

RECEIVED
FEB 0[illegible] 1990
INTERPOOL LABORATORIES

1/20/90

SCOTT SPECIALTY GASES, INC.

CERTIFICATE OF ANALYSIS

CERTIFIED

PROJECT #: 05-[illegible]7722
O #: [illegible]7-SA

OXYGEN SERVICE
INTERPOOL
4500 BALL R2 N.E.
CIRCLE PINES MN 55014-0000

CYLINDER #: AAL10507

ANALYTICAL ACCURACY: +-1%

COMPONENT	REQUESTED CONCENTRATION		ANALYSIS 1 (MOLES)	U/M
ULFUR DIOXIDE	850.0	PPM	859.2	PPM
ITROGEN	BALANCE GAS			N/A

NOTES: ACUBLEND MASTER GAS

ANALYTICAL METHOD: ACMG

DATE OF ANALYSIS: 12/26/89

ANALYST: Rohit G. Patel
ANALYST

APPROVED BY: J. Shapiro
SUPERVISOR

THE ONLY LIABILITY OF THIS COMPANY FOR GAS WHICH FAILS TO COMPLY WITH THIS ANALYSIS SHALL BE THE REPLACEMENT THEREOF BY THE COMPANY WITHOUT EXTRA COST

CERTIFIED REFERENCE MATERIALS EPA PROTOCOL GASES
ACUBLEND® CALIBRATION & SPECIALTY GAS MIXTURES PURE GAS
ACCESSORY PRODUCTS CUSTOM ANALYTICAL SERVICES

PLUMSTEADVILLE, PENNSYLVANIA / TROY, MICHIGAN / HOUSTON, TEXAS / WHEELING, ILLINOIS
SOUTH PLAINFIELD, NEW JERSEY / FREMONT, CALIFORNIA / WAKEFIELD, MASACHUSETTS / LONGMONT, COLORADO

Interpoll Laboratories, Inc.
(612) 786-6020

CERTIFICATE OF ANALYSIS FOR STANDARD GAS FOR METHOD 6C

Vendor: Scott Specialty Gases
Cylinder No: AAL 10507
Date of Preparation: 12-26-89
Label: 859.2 PPM SO_2/N_2
Blend Specification: ± 1% Accublend Master Gas

Results Of Analyses Of Standard Gas

Date of Analysis	Run	SO_2 (ppm)
3-30-92	1	843
3-30-92	2	840
3-30-92	3	849
	4	
	5	
	6	
	Avg	844

Analyst: Rick Eidem

☒ Results are within 5% of the vendor tag value; use tag value.
☐ Results are not within 5% of the vendor tag value; conduct another set of triplicate analyses.
☐ All results within ± 5% of the average; relabel as above.
☐ All results not within ± 5% of the average; perform another set of triplicate analyses.

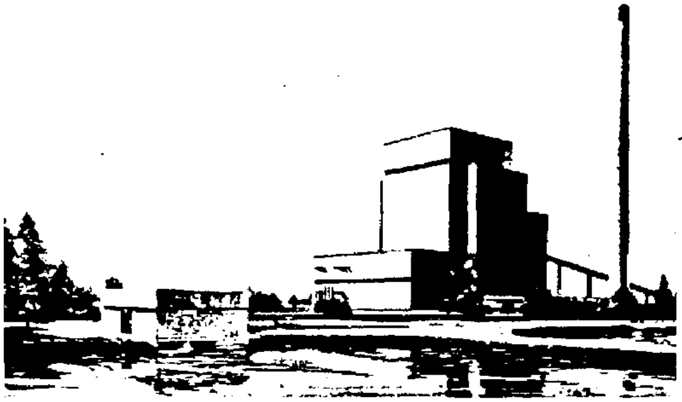
Approved by,

4-1-92
Date

Perry Lonnes
Dr. Perry Lonnes

S-443

Appendix I: Boiler Operational Data



BIG STONE PLANT

P. O. BOX 218
BIG STONE CITY, SOUTH DAKOTA 57216
(605) 862-8125

MONTANA-DAKOTA UTILITIES CO.
400 North Fourth Street
Bismarck, North Dakota 58501

OTTER TAIL POWER COMPANY
215 South Cascade Street
Fergus Falls, Minnesota 56537

NORTHWESTERN PUBLIC SERVICE COMPANY
3rd Street & Dakota Avenue South
Huron, South Dakota 57350

January 11, 1993

RECEIVED
JAN 14 1993
INTERPOLL LABORATORIES

Mr. Daniel Despen
Manager
Field Testing Support Group
Interpoll Laboratories, Inc.
4500 Ball Road, N.E.
Circle Pines, MN 55014-1819

Dear Mr. Despen:

This letter is in regards to the RDF cofire test performed at the Big Stone Power Plant. The heat input for the test on October 26, strictly coal, is 4562.8 MBTU/HR with a boiler efficiency of 80.03%. The heat input for the test on October 27, cofire test, is 4785.4 MBTU/HR with a boiler efficiency of 78.19%.

The test on the 26th started at 10:00 and continued until 19:00. Because the boiler and fuel were at a steady state from 10:00 to 18:00 we used this time frame to calculate the heat input. The attached graph, 100% lignite, shows the steady state of the boiler.

The test on the 27th ran from 09:30 to 18:00. However, the test did not run as smoothly as on the 27th. From 09:30 to 12:30 the feed of RDF into the boiler was a relatively small percent of the fuel. Our Unit Operations encountered numerous boiler upsets due to material handling problems with the RDF. The RDF blend leveled out at approximately 15:00 and stayed constant for the remainder of the test 18:00. The attached graph, RDF/lignite blend, shows the steady state of the boiler. I estimate a 12% RDF / 88% lignite ratio, by weight, from 15:00 to 18:00 hours. I therefore recommend that you use stack emission data corresponding to this time for the October 27 test.

Address Reply To: Otter Tail Power Company

BIG STONE PLANT

P. O. BOX 218
BIG STONE CITY, S.D. 57216

If you have any questions, please give Stu Schreurs or myself a call.

Sincerely,

Dean Pawlowski

Dean Pawlowski
Results Engineer

DP:ck

cc: Stu Schreurs

Attachments

Appendix J: Sampling and Analysis Methods

Particulate Loadings and Emission Rates

The particulate emission rates were determined per EPA Methods 1-5, CFR title 40, Part 60, Appendix A (revised July 1, 1987). In this procedure, a preliminary velocity profile of the gases in the flue is obtained by means of a temperature and velocity traverse. On the basis of these values, sampling nozzles of appropriate diameter are selected to allow isokinetic sampling, a necessary prerequisite for obtaining a representative sample.

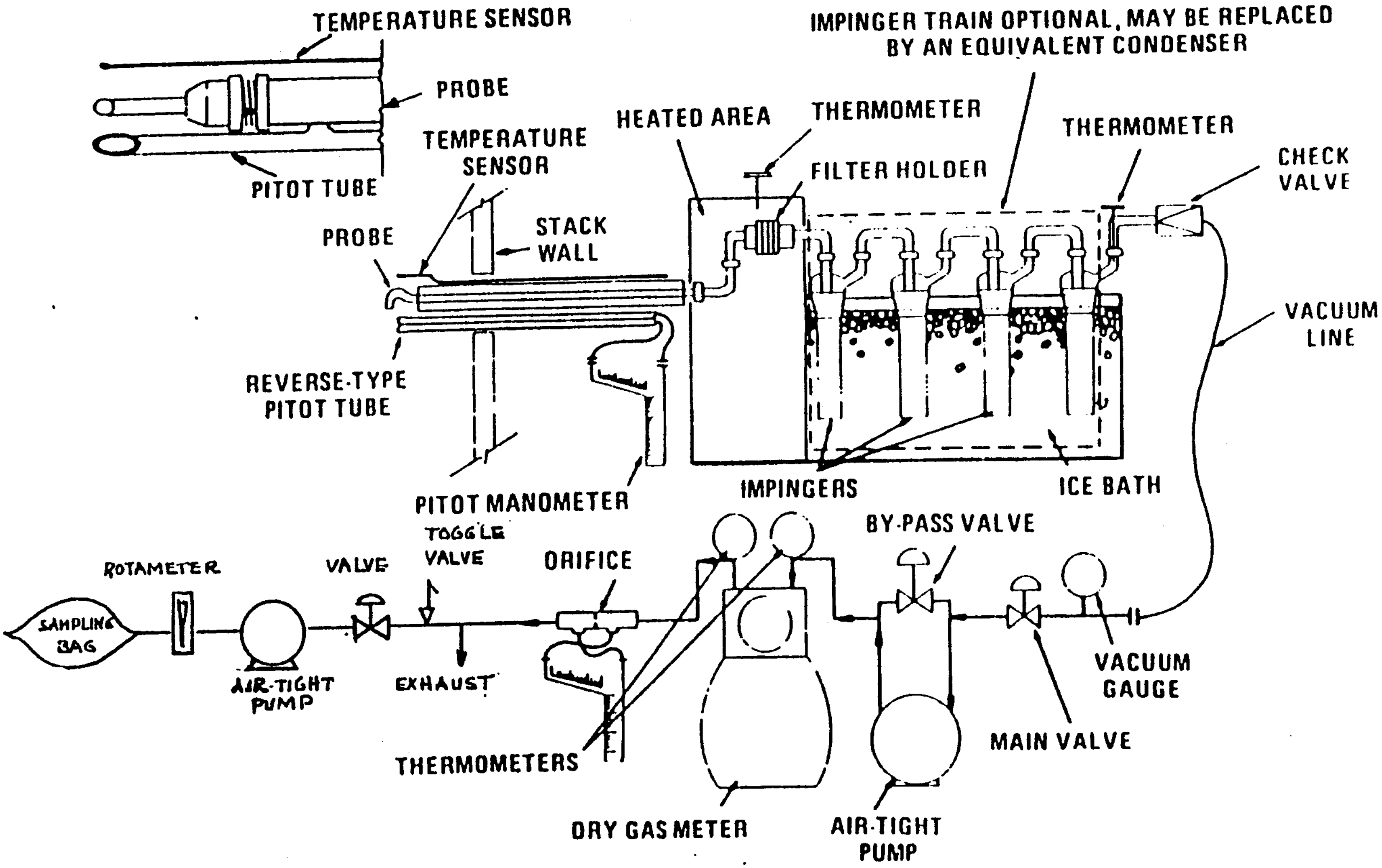
The sampling train consists of a heated stainless steel-lined sampling probe equipped with a Type S pitot and a thermocouple. The probe is attached to a sampling module which houses the all-glass in line filter holder in a temperature controlled oven. In addition, the sampling module also houses the impinger case and a Drierite drying column. The sampling module is connected by means of an umbilical cord to the control module which houses the dry test gasmeter, the calibrated orifice, a leakless pump, two inclined manometers, and all controls required for operating the sampling train.

Particulate samples were collected as follows: The sample gas was drawn in through the sampling probe isokinetically and passed through a 4-inch diameter Gelman Type A/E glass fiber filter. The particulates were removed at this point and collected on the filter. The gases then passed through an ice-cooled impinger train and a desiccant-packed drying column which quantitatively absorb all moisture from the sample gas stream after which the sample gas passes through the pump and the dry test gasmeter which integrates the sample gas flow throughout the course of the test. A calibrated orifice attached to the outlet of the gasmeter provides instantaneous flow rate data.

A representative particulate sample was acquired by sampling for equal periods of time at the centroid of a number of equal area regions in the duct. The sampling rate is adjusted at each site such that an isokinetic sampling condition prevails. Nomographs are used to aid in the rapid determination of the sampling rate.

After sampling is complete, the filter is removed and placed in a clean container. The nozzle and inlet side of the filter holder are quantitatively washed with acetone and the washings are stored in a second container. A brush is often used in the cleaning step to help dislodge deposits. The samples are returned to the laboratory where they are logged in and analyzed. The volume of the acetone rinse ("probe wash") is noted and then the rinse is quantitatively transferred to a tared 120 cc porcelain evaporating dish and the acetone evaporated off at 97-105 °F. This temperature is used to prevent condensation of atmospheric moisture due to the cooling effect induced by the evaporation of acetone. The acetone-free sample is then transferred to an oven and dried at 105 °C for 30 minutes, cooled in a desiccator over Drierite, and then weighed to the nearest .01 mg. The filter sample is quantitatively transferred to a 6-inch watch glass and dried in an oven at 105 °C for two hours. The filter and watch glass are then cooled in a desiccator and the filter weighed to the nearest .01 mg. All weighings are performed in a balance room where the relative humidity is hydrostatted to less than 50% relative humidity. Microscopic examination of the samples is performed if any unusual characteristics are observed. The weight of the acetone rinse is corrected for the acetone blank. The Drierite column is weighed on-site and the water collected by Drierite is added to the condensate so that the total amount of absorbed water may be ascertained.

Integrated flue gas samples for Orsat analysis were collected simultaneously from the stack and from the breeching at the inlet to the wet scrubber. The samples were collected in 15-liter gas sampling bags at a constant flow rate throughout each particulate run. The bags were then returned to the laboratory and analyzed by Orsat analysis. Standard commercially prepared solutions were used in the Orsat analyzer (sat. KOH for carbon dioxide and reduced methylene blue for oxygen).



Particulate sampling train.

Method 3A—Determination of Oxygen and Carbon Dioxide Concentrations in Emissions From Stationary Sources (Instrumental Analyzer Procedure)

1. Applicability and Principle

1.1 Applicability. This method is applicable to the determination of oxygen (O_2) and carbon dioxide (CO_2) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A sample is continuously extracted from the effluent stream: a portion of the sample stream is conveyed to an instrumental analyzer(s) for determination of O_2 and CO_2 concentration(s). Performance specifications and test procedures are provided to ensure reliable data.

2. Range and Sensitivity

Same as Method 6C, Sections 2.1 and 2.2, except that the span of the monitoring system shall be selected such that the average O_2 or CO_2 concentration is not less than 20 percent of the span.

3. Definitions

3.1 Measurement System. The total equipment required for the determination of the O_2 or CO_2 concentration. The measurement system consists of the same major subsystems as defined in Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, Response Time, and Calibration Curve. Same as Method 6C, Sections 3.2 through 3.8, and 3.10.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

4. Measurement System Performance Specifications

Same as Method 6C, Sections 4.1 through 4.4.

5. Apparatus and Reagents

5.1 Measurement System. Any measurement system for O_2 or CO_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe. A leak-free probe, of sufficient length to traverse the sample points.

5.1.2 Sample Line. Tubing, to transport the sample gas from the probe to the moisture removal system. A heated sample line is not required for systems that measure the O_2 or CO_2 concentration on a dry basis, or transport dry gases.

5.1.3 Sample Transport Line, Calibration Value Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.3 through 5.1.9, and 5.1.11, except that the requirements to use stainless steel, Teflon, and nonreactive glass filters do not apply.

5.1.4 Gas Analyzer. An analyzer to determine continuously the O2 or CO2 concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer. The requirements for measuring and controlling the analyzer flow rate are not applicable if data are presented that demonstrate the analyzer is insensitive to flow variations over the range encountered during the test.

5.2 Calibration Gases. The calibration gases for CO_2 analyzers shall be CO_2 in N_2 or CO_2 in air. Alternatively, CO_2/SO_2, O_2/SO_2, or $O_2/CO_2/SO_2$ gas mixtures in N_2 may be used. Three calibration gases, as specified Section 5.3.1 through 5.3.3 of Method 6C, shall be used. For O_2 monitors that cannot analyze zero gas, a calibration gas concentration equivalent to less than 10 percent of the span may be used in place of zero gas.

6. Measurement System Performance Test Procedures

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Concentration Verification. Follow Section 6.1 of Method 6C, except if calibration gas analysis is required, use Method 3 and change the acceptance criteria for agreement among Method 3 results to 5 percent (or 0.2 percent by volume, whichever is greater).

6.2 Interference Response. Conduct an interference response test of the analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could alter the interference response (e.g., changes in the type of gas detector). Conduct the interference response in accordance with Section 5.4 of Method 20.

6.3 Measurement System Preparation, Analyzer Calibration Error, and Sampling System Bias Check. Follow Sections 6.2 through 6.4 of Method 6C.

7. Emission Test Procedure

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 3.

7.2 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the sampling system bias check. Maintain constant rate sampling (i.e., ± 10 percent) during the entire run. The sampling time per run shall be the same as for tests conducted using Method 3 plus twice the system response time. For each run, use only those measurements obtained after twice the response time of the measurement system has elapsed to determine the average effluent concentration.

7.3 Zero and Calibration Drift Test. Follow Section 7.4 of Method 6C.

8. Quality Control Procedures

The following quality control procedures are recommended when the results of this method are used for an emission rate correction factor, or excess air determination. The tester should select one of the following options for validating measurement results:

8.1 If both O_2 and CO_2 are measured using Method 3A, the procedures described in Section 4.4 of Method 3 should be followed to validate the O_2 and CO_2 measurement results.

8.2 If only O_2 is measured using Method 3A, measurements of the sample stream CO_2 concentration should be obtained at the sample by-pass vent discharge using an Orsat or Fyrite analyzer, or equivalent. Duplicate samples should be obtained concurrent with at least one run. Average the duplicate Orsat or Fyrite analysis results for each run. Use the average CO_2 values for comparison with the O_2 measurements in accordance with the procedures described in Section 4.4 of Method 3.

8.3 If only CO_2 is measured using Method 3A, concurrent measurements of the sample stream CO_2 concentration should be obtained using an Orsat or Fyrite analyzer as described in Section 8.2. For each run, differences greater than 0.5 percent between the Method 3A results and the average of the duplicate Fyrite analysis should be investigated.

9. Emission Calculation

For all CO_2 analyzers, and for O_2 analyzers that can be calibrated with zero gas, follow Section 8 of Method 6C, except express all concentrations as percent, rather than ppm.

For O_2 analyzers that use a low-level calibration gas in place of a zero gas, calculate the effluent gas concentration using Equation 3A-1.

$$C_{gas} = \frac{C_{ma} - C_{oa}}{C_{m} - C_{o}} (\bar{C} - C_{m}) + C_{ma} \qquad \text{Eq. 3A-1}$$

Where:

C_{gas} = Effluent gas concentration, dry basis, percent.
C_{ma} = Actual concentration of the upscale calibration gas, percent.
C_{oa} = Actual concentration of the low-level calibration gas, percent.
C_{m} = Average of initial and final system calibration bias check responses for the upscale calibration gas, percent.
C_{o} = Average of initial and final system calibration bias check responses for the low-level gas, percent.
$\bar{C}$ = Average gas concentration indicated by the gas analyzer, dry basis, percent.

10. Bibliography

Same as bibliography of Method 6C.

METHOD 6C—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

1. *Applicability and Principle*

1.1 *Applicability.* This method is applicable to the determination of sulfur dioxide (SO_2) concentrations in controlled and uncontrolled emissions from stationary sources only when specified within the regulations.

1.2 Principle. A gas sample is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental analyzer for determination of SO_2 gas concentration using an ultraviolet (UV), nondispersive infrared (NDIR), or fluorescence analyzer. Performance specifications and test procedures are provided to ensure reliable data.

2. *Range and Sensitivity*

2.1 Analytical Range. The analytical range is determined by the instrumental design. For this method, a portion of the analytical range is selected by choosing the span of the monitoring system. The span of the monitoring system shall be selected such that the pollutant gas concentration equivalent to the emission standard is not less than 30 percent of the span. If at any time during a run the measured gas concentration exceeds the span, the run shall be considered invalid.

2.2 Sensitivity. The minimum detectable limit depends on the analytical range, span, and signal-to-noise ratio of the measurement system. For a well designed system, the minimum detectable limit should be less than 2 percent of the span.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of gas concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface. That portion of a system used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the analyzers from the effects of the stack effluent.

3.1.2 Gas Analyzer. That portion of the system that senses the gas to be measured and generates an output proportional to its concentration.

3.1.3 Data Recorder. A strip chart recorder, analog computer, or digital recorder for recording measurement data from the analyzer output.

3.2 Span. The upper limit of the gas concentration measurement range displayed on the data recorder.

3.3 Calibration Gas. A known concentration of a gas in an appropriate diluent gas.

3.4 Analyzer Calibration Error. The difference between the gas concentration exhibited by the gas analyzer and the known concentration of the calibration gas when the calibration gas is introduced directly to the analyzer.

3.5 Sampling System Bias. The difference between the gas concentrations exhibited by the measurement system when a known concentration gas is introduced at the outlet of the sampling probe and when the same gas is introduced directly to the analyzer.

3.6 Zero Drift. The difference in the measurement system output reading from the initial calibration response at the zero concentration level after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

3.7 Calibration Drift. The difference in the measurement system output reading from the initial calibration response at a mid-range calibration value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

3.8 Response Time. The amount of time required for the measurement system to display 95 percent of a step change in gas concentration on the data recorder.

3.9 Interference Check. A method for detecting analytical interferences and excessive biases through direct comparison of gas concentrations provided by the measurement system and by a modified Method 6 procedure. For this check, the modified Method 6 samples are acquired at the sample by-pass discharge vent.

3.10 Calibration Curve. A graph or other systematic method of establishing the relationship between the analyzer response and the actual gas concentration introduced to the analyzer.

4. *Measurement System Performance Specifications*

4.1 Analyzer Calibration Error. Less than ±2 percent of the span for the zero, mid-range, and high-range calibration gases.

4.2 Sampling System Bias. Less than ±5 percent of the span for the zero, and mid- or high-range calibration gases.

4.3 Zero Drift. Less than ±3 percent of the span over the period of each run.

4.4 Calibration Drift. Less than ±3 percent of the span over the period of each run.

4.5 Interference Check. Less than ±7 percent of the modified Method 6 result for each run.

5. *Apparatus and Reagents*

5.1 Measurement System. Any measurement system for SO_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1. The essential components of the measurement system are described below:

5.1.1 Sample Probe. Glass, stainless steel, or equivalent, of sufficient length to traverse the sample points. The sampling probe shall be heated to prevent condensation.

5.1.2 Sample Line. Heated (sufficient to prevent condensation) stainless steel or

Teflon tubing, to transport the sample gas to the moisture removal system.

5.1.3 Sample Transport Lines. Stainless steel or Teflon tubing, to transport the sample from the moisture removal system to the sample pump, sample flow rate control, and sample gas manifold.

5.1.4 Calibration Valve Assembly. A three-way valve assembly, or equivalent, for blocking the sample gas flow and introducing calibration gases to the measurement system at the outlet of the sampling probe when in the calibration mode.

5.1.5 Moisture Removal System. A refrigerator-type condenser or similar device (e.g., permeation dryer), to remove condensate continuously from the sample gas while maintaining minimal contact between the condensate and the sample gas. The moisture removal system is not necessary for analyzers that can measure gas concentrations on a wet basis; for these analyzers, (1) heat the sample line and all interface components up to the inlet of the analyzer sufficiently to prevent condensation, and (2) determine the moisture content and correct the measured gas concentrations to a dry basis using appropriate methods, subject to the approval of the Administrator. The determination of sample moisture content is not necessary for pollutant analyzers that measure concentrations on a wet basis when (1) a wet basis CO_2 analyzer operated according to Method 3A is used to obtain simultaneous measurements, and (2) the pollutant/CO_2 measurements are used to determine emissions in units of the standard.

5.1.6 Particulate Filter. An in-stack or heated (sufficient to prevent water condensation) out-of-stack filter. The filter shall be borosilicate or quartz glass wool, or glass fiber mat. Additional filters at the inlet or outlet of the moisture removal system and inlet of the analyzer may be used to prevent accumulation of particulate material in the measurement system and extend the useful life of the components. All filters shall be fabricated of materials that are nonreactive to the gas being sampled.

5.1.7 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The pump may be constructed of any material that is nonreactive to the gas being sampled.

5.1.8 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent.

(NOTE: The tester may elect to install a back-pressure regulator to maintain the sample gas manifold at a constant pressure in order to protect the analyzer(s) from overpressurization, and to minimize the need for flow rate adjustments.)

5.1.9 Sample Gas Manifold. A sample gas manifold, to divert a portion of the sample gas stream to the analyzer, and the remainder to the by-pass discharge vent. The sample gas manifold should also include provisions for introducing calibration gases directly to the analyzer. The manifold may be constructed of any material that is nonreactive to the gas being sampled.

5.1.10 Gas Analyzer. A UV or NDIR absorption or fluorescence analyzer, to determine continuously the SO_2 concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer.

(NOTE: Housing the analyzer(s) in a clean, thermally-stable, vibration-free environment will minimize drift in the analyzer calibration.)

5.1.11 Data Recorder. A strip chart recorder, analog computer, or digital recorder, for recording measurement data. The data recorder resolution (i.e., readability) shall be 0.5 percent of span. Alternatively, a digital or analog meter having a resolution of 0.5 percent of span may be used to obtain the analyzer responses and the readings may be recorded manually. If this alternative is used, the readings shall be obtained at equally spaced intervals over the duration of the sampling run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive, shall be obtained. For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 96 measurements, whichever is less restrictive, shall be obtained.

5.2 Method 6 Apparatus and Reagents. The apparatus and reagents described in Method 6, and shown by the schematic of the sampling train in Figure 6C-2, to conduct the interference check.

5.3 SO_2 Calibration Gases. The calibration gases for the gas analyzer shall be SO_2 in N_2 or SO_2 in air. Alternatively, SO_2/CO_2, SO_2/O_2, or $SO_2/CO_2/O_2$ gas mixtures in N_2 may be used. For fluorescence-based analyzers, the O_2 and CO_2 concentrations of the calibration gases as introduced to the analyzer shall be within 1 percent (absolute) O_2 and 1 percent (absolute) CO_2 of the O_2 and CO_2 concentrations of the effluent samples as introduced to the analyzer. Alternatively, for fluorescence-based analyzers, use calibration blends of SO_2 in air and the nomographs provided by the vendor to determine the quenching correction factor (the effluent O_2 and CO_2 concentrations must be known). Use three calibration gases as specified below:

5.3.1 High-Range Gas. Concentration equivalent to 80 to 90 percent of the span.

5.3.2 Mid-Range Gas. Concentration equivalent to 50 to 60 percent of the span.

5.3.3 Zero Gas. Concentration of less than 0.25 percent of the span. Purified ambient air may be used for the zero gas by passing air through a charcoal filter, or through one or more impingers containing a solution of 3 percent H_2O_2.

6. *Measurement System Performance Test Procedures*

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. There are two alternatives for establishing the concentrations of calibration gases. Alternative Number 1 is preferred.

6.1.1 Alternative Number 1—Use of calibration gases that are analyzed following the Environmental Protection Agency Traceability Protocol Number 1 (see Citation 1 in the Bibliography). Obtain a certification from the gas manufacturer that Protocol Number 1 was followed.

6.1.2 Alternative Number 2—Use of calibration gases not prepared according to Protocol Number 1. If this alternative is chosen, obtain gas mixtures with a manufacturer's tolerance not to exceed ±2 percent of the tag value. Within 6 months before the emission test, analyze each of the calibration gases in triplicate using Method 6. Citation 2 in the Bibliography describes procedures and techniques that may be used for this analysis. Record the results on a data sheet (example is shown in Figure 6C-3). Each of the individual SO_2 analytical results for each calibration gas shall be within 5 percent (or 5 ppm, whichever is greater) of the triplicate set average; otherwise, discard the entire set, and repeat the triplicate analyses. If the average of the triplicate analyses is within 5 percent of the calibration gas manufacturer's cylinder tag value, use the tag value; otherwise, conduct at least three additional analyses until the results of six consecutive runs agree with 5 percent (or 5 ppm, whichever is greater) of their average. Then use this average for the cylinder value.

6.2 Measurement System Preparation. Assemble the measurement system by following the manufacturer's written instructions for preparing and preconditioning the gas analyzer and, as applicable, the other system components. Introduce the calibration gases in any sequence, and make all necessary adjustments to calibrate the analyzer and the data recorder. Adjust system components to achieve correct sampling rates.

6.3 Analyzer Calibration Error. Conduct the analyzer calibration error check by introducing calibration gases to the measurement system at any point upstream of the gas analyzer as follows:

6.3.1 After the measurement system has been prepared for use, introduce the zero, mid-range, and high-range gases to the analyzer. During this check, make no adjustments to the system except those necessary to achieve the correct calibration gas flow rate at the analyzer. Record the analyzer responses to each calibration gas on a form similar to Figure 6C-4.

NOTE: A calibration curve established prior to the analyzer calibration error check may be used to convert the analyzer response to the equivalent gas concentration introduced to the analyzer. However, the same correction procedure shall be used for all effluent and calibration measurements obtained during the test.

6.3.2 The analyzer calibration error check shall be considered invalid if the gas concentration displayed by the analyzer exceeds ±2 percent of the span for any of the calibration gases. If an invalid calibration is exhibited, take corrective action, and repeat the analyzer calibration error check until acceptable performance is achieved.

6.4 Sampling System Bias Check. Perform the sampling system bias check by introducing calibration gases at the calibration valve installed at the outlet of the sampling probe. A zero gas and either the midrange or high-range gas, whichever most closely approximates the effluent concentrations, shall be used for this check as follows:

6.4.1 Introduce the upscale calibration gas, and record the gas concentration displayed by the analyzer on a form similar to Figure 6C-6. Then introduce zero gas, and record the gas concentration displayed by the analyzer. During the sampling system bias check, operate the system at the normal sampling rate, and make no adjustments to the measurement system other than those necessary to achieve proper calibration gas flow rates at the analyzer. Alternately introduce the zero and upscale gases until a stable response is achieved. The tester shall determine the measurement system response time by observing the times required to achieve a stable response for both the zero and upscale gases. Note the longer of the two times as the response time.

6.4.2 The sampling system bias check shall be considered invalid if the difference between the gas concentrations displayed by the measurement system for the analyzer calibration error check and for the sampling system bias check exceeds ±5 percent of the span for either the zero or upscale calibration gas. If an invalid calibration is exhibited, take corrective action, and repeat the sampling system bias check until acceptable performance is achieved. If adjustment to the analyzer is required, first repeat the analyzer calibration error check, then repeat the sampling system bias check.

7. *Emission Test Procedure*

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to Method 6.

7.2 Interference Check Preparation. For each individual analyzer, conduct an interference check for at least three runs during the initial field test on a particular source category. Retain the results, and report them with each test performed on that source category.

If an interference check is being performed, assemble the modified Method 6 train (flow control valve, two midget impingers containing 3 percent H_2O_2, and dry gas meter) as shown in Figure 6C-2. Install the sampling train to obtain a sample at the measurement system sample by-pass discharge vent. Record the initial dry gas meter reading.

7.3 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the sampling system bias check. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as for Method 6 plus twice the system response time. For each run, use only those measurements obtained after twice response time of the measurement system has elapsed, to determine the average effluent concentration. If an interference check is being performed, open the flow control valve on the modified Method 6 train concurrent with the initiation of the sampling period, and adjust the flow to 1 liter per minute (±10 percent).

(NOTE: If a pump is not used in the modified Method 6 train, caution should be exercised in adjusting the flow rate since overpressurization of the impingers may cause leakage in the impinger train, resulting in positively biased results).

7.4 Zero and Calibration Drift Tests. Immediately preceding and following each run, or if adjustments are necessary for the measurement system during the run, repeat the sampling system bias check procedure described in Section 6.4 (Make no adjustments to the measurement system until after the drift checks are completed.) Record and analyzer's responses on a form similar to Figure 6C-5.

7.4.1 If either the zero or upscale calibration value exceeds the sampling system bias specification, then the run is considered invalid. Repeat both the analyzer calibration error check procedure (Section 6.3) and the sampling system bias check procedure (Section 6.4) before repeating the run.

7.4.2 If both the zero and upscale calibration values are within the sampling system bias specification, then use the average of the initial and final bias check values to calculate the gas concentration for the run. If the zero or upscale calibration drift value exceeds the drift limits, based on the difference between the sampling system bias check responses immediately before and after the run, repeat both the analyzer calibration error check procedure (Section 6.3) and the sampling system bias check procedure (Section 6.4) before conducting additional runs.

7.5 Interference Check (if performed). After completing the run, record the final dry gas meter reading, meter temperature, and barometric pressure. Recover and analyze the contents of the midget impingers, and determine the SO_2 gas concentration using the procedures of Method 6. (It is not necessary to analyze EPA performance audit samples for Method 6.) Determine the average gas concentration exhibited by the analyzer for the run. If the gas concentrations provided by the analyzer and the modified Method 6 differ by more than 7 percent of the modified Method 6 result, the run is invalidated.

8. *Emission Calculation*

The average gas effluent concentration is determined from the average gas concentration displayed by the gas analyzer, and is adjusted for the zero and upscale sampling system bias checks, as determined in accordance with Section 7.4. The average gas concentration displayed by the analyzer may be determined by integration of the area under the curve for chart recorders, or by averaging all of the effluent measurements. Alternatively, the average may be calculated from measurements recorded at equally spaced intervals over the entire duration of the run. For sampling run durations of less than 1 hour, measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive, shall be used. For sampling run durations greater than 1 hour, measurements at 2-minute intervals or a minimum of 96 measurements, whichever is less restrictive, shall be used. Calculate the effluent gas concentration using Equation 6C-1.

$$C_{gas} = (\bar{C} - C_o) \frac{C_{ma}}{C_m - C_o} \qquad \text{Eq. 6C-1}$$

Where:

C_{gas} = Effluent gas concentration, dry basis, ppm.

C = Average gas concentration indicated by gas analyzer, dry basis, ppm.
C_o = Average of initial and final system calibration bias check responses for the zero gas, ppm.
C_m = Average of initial and final system calibration bias check responses for the upscale calibration gas, ppm.
C_{ma} = Actual concentration of the upscale calibration gas, ppm.

9. *Bibliography*

1. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibrations and Audits of Continuous Source Emission Monitors: Protocol Number 1. U.S. Environmental Protection Agency, Quality Assurance Division. Research Triangle Park, NC. June 1978.

2. Westlin, Peter R. and J. W. Brown. Methods for Collecting and Analyzing Gas Cylinder Samples. Source Evaluation Society Newsletter. *3*(3):5-15. September 1978.

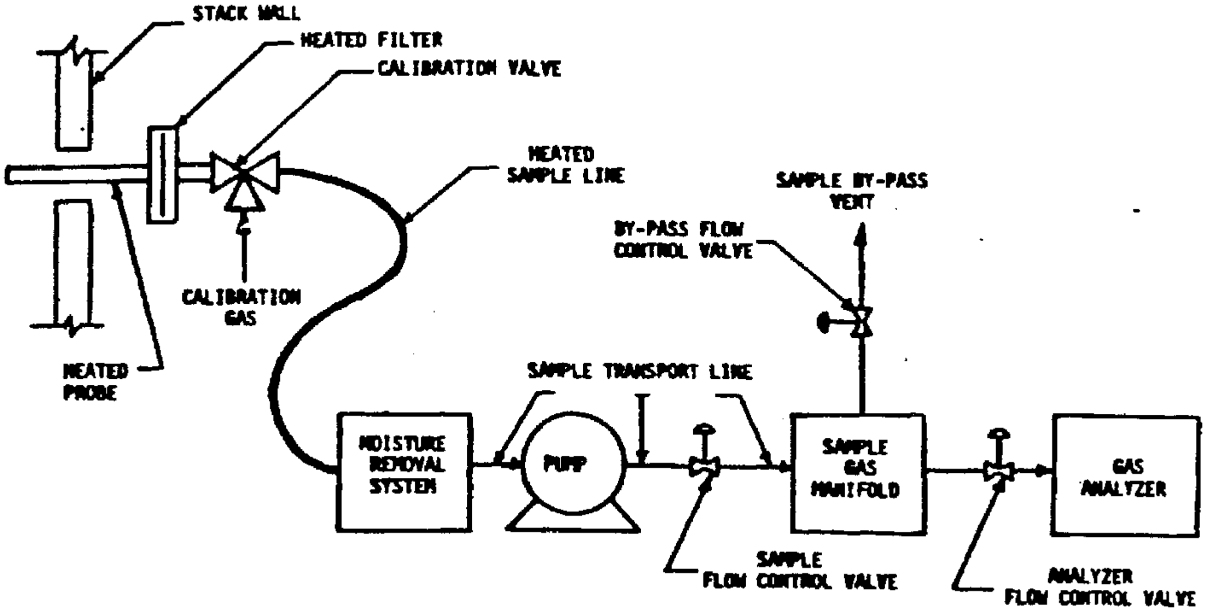


Figure 6C-1. Measurement system schematic.

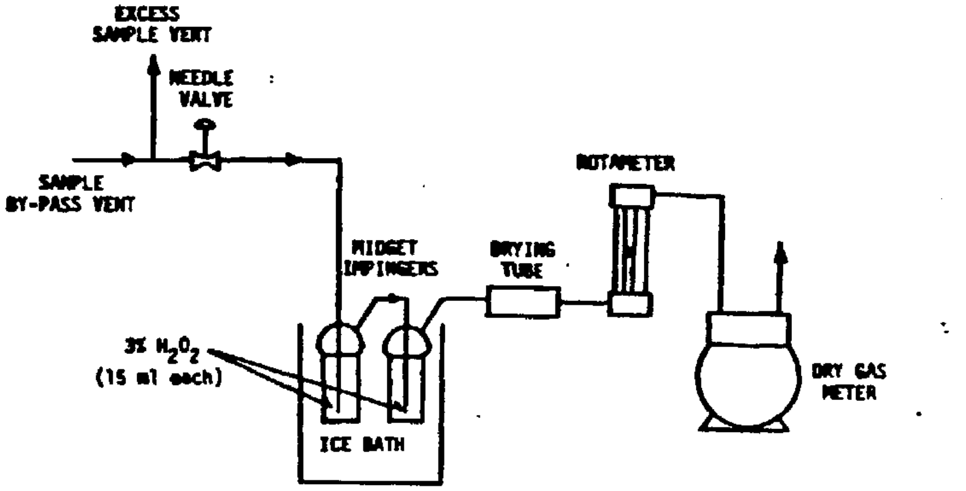


Figure 6C-2. Interference check sampling train.

FIGURE 6C-3—ANALYSIS OF CALIBRATION GASES

Date ____________________

Analytic method used ____________________

METHOD 7E—DETERMINATION OF NITROGEN OXIDES EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

1. *Applicability and Principle*

1.1 Applicability. This method is applicable to the determination of nitrogen oxides (NO_x) concentrations in emissions from stationary sources only when specified within the regulations.

1.2 Principle. A gas simple is continuously extracted from a stack, and a portion of the sample is conveyed to an instrumental chemiluminescent analyzer for determination of NO_x concentration. Performance specifications and test procedures are provided to ensure reliable data.

2. *Range and Sensitivity*

Same as Method 6C, Sections 2.1 and 2.2.

3. *Definitions*

3.1 Measurement System. The total equipment required for the determination of NO_x concentration. The measurement system consists of the following major subsystems:

3.1.1 Sample Interface, Gas Analyzer, and Data Recorder. Same as Method 6C, Sections 3.1.1, 3.1.2, and 3.1.3.

3.1.2 NO_2 to NO Converter. A device that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO).

3.2 Span, Calibration Gas, Analyzer Calibration Error, Sampling System Bias, Zero Drift, Calibration Drift, and Response Time. Same as Method 6C, Sections 3.2 through 3.8.

3.3 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

4. *Measurement System Performance Specifications*

Same as Method 6C, Sections 4.1 through 4.4.

5. *Apparatus and Reagents*

5.1 Measurement System. Any measurement system for NO_x that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 6C-1 of Method 6C. The essential components of the measurement system are described below:

5.1.1 Sample Probe, Sample Line, Calibration Valve Assembly, Moisture Removal System, Particulate Filter, Sample Pump, Sample Flow Rate Control, Sample Gas Manifold, and Data Recorder. Same as Method 6C, Sections 5.1.1 through 5.1.9, and 5.1.11.

5.1.2 NO_2 to NO Converter. That portion of the system that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO). An NO_2 to NO converter is not necessary if data are presented to demonstrate that the NO_2 portion of the exhaust gas is less than 5 percent of the total NO_x concentration.

5.1.3 NO_x Analyzer. An analyzer based on the principles of chemiluminescence, to determine continuously the NO_x concentration in the sample gas stream. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer.

5.2 NO_x Calibration Gases. The calibration gases for the NO_x analyzer shall be NO in N_2. Three calibration gases, as specified in Sections 5.3.1 through 5.3.3. of Method 6C, shall be used. Ambient air may be used for the zero gas.

6. *Measurement System Performance Test Procedures*

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. Follow Section 6.1 of Method 6C, except if calibration gas analysis is required, use Method 7, and change all 5 percent performance values to 10 percent (or 10 ppm, whichever is greater).

6.2 Interference Response. Conduct an interference response test of the analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could alter the interference response (e.g., changes in the gas detector). Conduct the interference response in accordance with Section 5.4 of Method 20.

6.3 Measurement System Preparation, Analyzer Calibration Error, and Sample System Bias Check. Follow Sections 6.2 through 6.4 of Method 6C.

6.4 NO_2 to NO Conversion Efficiency. Unless data are presented to demonstrate that the NO_2 concentration within the sample stream is not greater than 5 percent of the NO_x concentration, conduct an NO_2 to NO conversion efficiency test in accordance with Section 5.6 of Method 20.

7. *Emission Test Procedure*

7.1 Selection of Sampling Site and Sampling Points. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 7.

7.2 Sample Collection. Position the sampling probe at the first measurement point, and begin sampling at the same rate as used during the system calibration drift test. Maintain constant rate sampling (i.e., ±10 percent) during the entire run. The sampling time per run shall be the same as the total time required to perform a run using Method 7, plus twice the system response time. For each run, use only those measurements obtained after twice the response time of the measurement system has elapsed, to determine the average effluent concentration.

7.3 Zero and Calibration Drift Test. Follow Section 7.4 of Method 6C.

8. *Emission Calculation*

Follow Section 8 of Method 6C.

9. *Bibliography*

Same as bibliography of Method 6C.

METHOD 25A—DETERMINATION OF TOTAL GASEOUS ORGANIC CONCENTRATION USING A FLAME IONIZATION ANALYZER

1. *Applicability and Principle*

1.1 Applicability. This method applies to the measurement of total gaseous organic concentration of vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). The concentration is expressed in terms of propane (or other appropriate organic calibration gas) or in terms of carbon.

1.2 Principle. A gas sample is extracted from the source through a heated sample line, if necessary, and glass fiber filter to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalents of the calibration gas or as carbon equivalents.

2. *Definitions*

2.1 Measurement System. The total equipment required for the determination of the gas concentration. The system consists of the following major subsystems:

2.1.1 Sample Interface. That portion of the system that is used for one or more of the following: sample acquisition, sample transportation, sample conditioning, or protection of the analyzer from the effects of the stack effluent.

2.1.2 Organic Analyzer. That portion of the system that senses organic concentration and generates an output proportional to the gas concentration.

2.2 Span Value. The upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable part of the regulations. The span value is established in the applicable regulation and is usually 1.5 to 2.5 times the applicable emission limit. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration. For convenience, the span value should correspond to 100 percent of the recorder scale.

2.3 Calibration Gas. A known concentration of a gas in an appropriate diluent gas.

2.4 Zero Drift. The difference in the measurement system response to a zero level calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place.

2.5 Calibration Drift. The difference in the measurement system response to a mid-level calibration gas before and after a stated period of operation during which no unscheduled maintenance, repair or adjustment took place.

2.6 Response Time. The time interval from a step change in pollutant concentration at the inlet to the emission measurement system to the time at which 95 percent of the corresponding final value is reached as displayed on the recorder.

2.7 Calibration Error. The difference between the gas concentration indicated by the measurement system and the known concentration of the calibration gas.

3. *Apparatus*

A schematic of an acceptable measurement system is shown in Figure 25A-1. The essential components of the measurement system are described below:

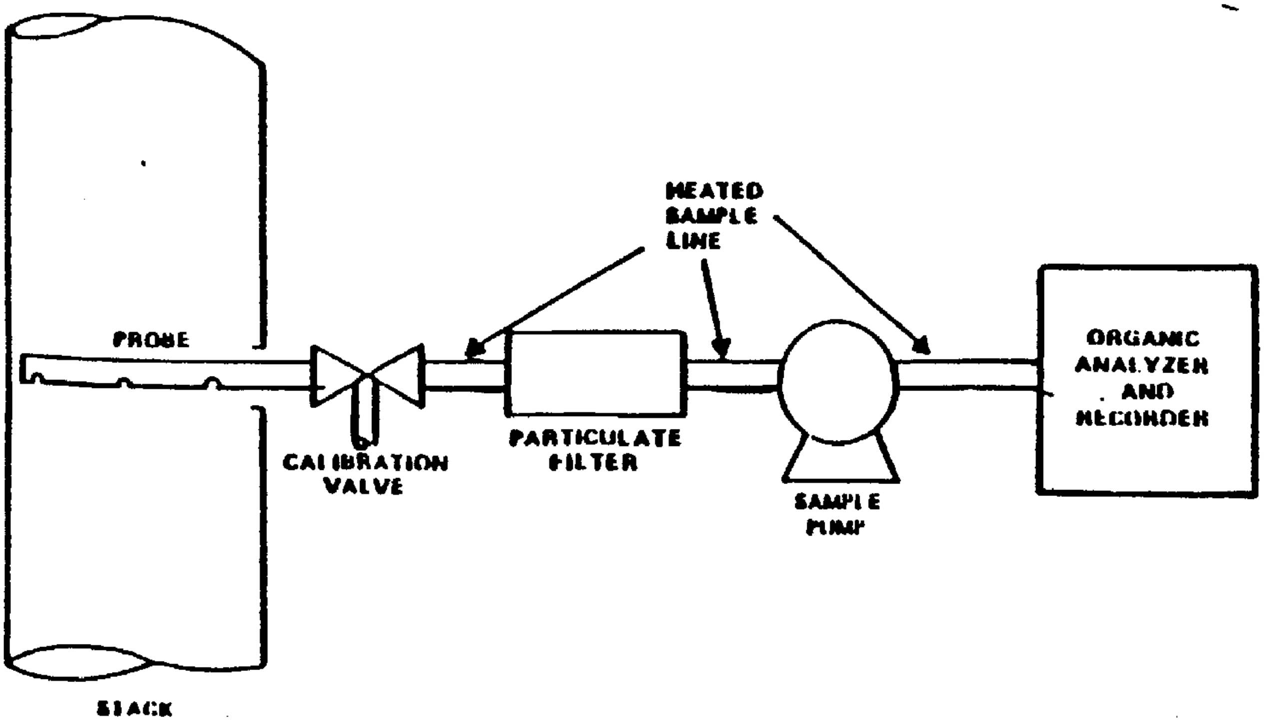


Figure 25A-1. Organic Concentration Measurement System.

3.1 Organic Concentration Analyzer. A flame ionization analyzer (FIA) capable of meeting or exceeding the specifications in this method.

3.2 Sample Probe. Stainless steel, or equivalent, three-hole rake type. Sample holes shall be 4 mm in diameter or smaller and located at 16.7, 50, and 83.3 percent of the equivalent stack diameter. Alternatively, a single opening probe may be used so that a gas sample is collected from the centrally located 10 percent area of the stack cross-section.

3.3 Sample Line. Stainless steel or Teflon* tubing to transport the sample gas to the analyzer. The sample line should be heated, if necessary, to prevent condensation in the line.

3.4 Calibration Valve Assembly. A three-way valve assembly to direct the zero and calibration gases to the analyzers is recommended. Other methods, such as quick-connect lines, to route calibration gas to the analyzers are applicable.

3.5 Particulate Filter. An in-stack or an out-of-stack glass fiber filter is recommended if exhaust gas particulate loading is significant. An out-of-stack filter should be heated to prevent any condensation.

3.6 Recorder. A strip-chart recorder, analog computer, or digital recorder for recording measurement data. The minimum data recording requirement is one measurement value per minute. Note: This method is often applied in highly explosive areas. Caution and care should be exercised in choice of equipment and installation.

4. *Calibration and Other Gases*

Gases used for calibrations, fuel, and combustion air (if required) are contained in compressed gas cylinders. Preparation of calibration gases shall be done according to the procedure in Protocol No. 1, listed in Reference 9.2. Additionally, the manufacturer of the cylinder should provide a recommended shelf life for each calibration gas cylinder over which the concentration does not change more than ±2 percent from the certified value. For calibration gas values not generally available (i.e., organics between 1 and 10 percent by volume), alternative methods for preparing calibration gas mixtures, such as dilution systems, may be used with prior approval of the Administrator.

Calibration gases usually consist of propane in air or nitrogen and are determined in terms of the span value. Organic compounds other than propane can be used following the above guidelines and making the appropriate corrections for response factor.

4.1 Fuel. A 40 percent H_2/60 percent He or 40 percent H_2/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

4.2 Zero Gas. High purity air with less than 0.1 parts per million by volume (ppmv) of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

4.3 Low-level Calibration Gas. An organic calibration gas with a concentration equivalent to 25 to 35 percent of the applicable span value.

4.4 Mid-level Calibration Gas. An organic calibration gas with a concentration equivalent to 45 to 55 percent of the applicable span value.

4.5 High-level Calibration Gas. An organic calibration gas with a concentration equivalent to 80 to 90 percent of the applicable span value.

5. *Measurement System Performance Specifications*

5.1 Zero Drift. Less than ±3 percent of the span value.

5.2 Calibration Drift. Less than ±3 percent of span value.

5.3 Calibration Error. Less than ±5 percent of the calibration gas value.

6. *Pretest Preparations*

6.1 Selection of Sampling Site. The location of the sampling site is generally specified by the applicable regulation or purpose of the test; i.e., exhaust stack, inlet line, etc. The sample port shall be located at least 1.5 meters or 2 equivalent diameters upstream of the gas discharge to the atmosphere.

6.2 Location of Sample Probe. Install the sample probe so that the probe is centrally located in the stack, pipe, or duct and is sealed tightly at the stack port connection.

6.3 Measurement System Preparation. Prior to the emission test, assemble the measurement system following the manufacturer's written instructions in preparing the sample interface and the organic analyzer. Make the system operable.

FIA equipment can be calibrated for almost any range of total organics concentrations. For high concentrations of organics (>1.0 percent by volume as propane) modifications to most commonly available analyzers are necessary. One accepted method of equipment modification is to decrease the size of the sample to the analyzer through the use of a smaller diameter sample capillary. Direct and continuous measurement of organic concentration is a necessary consideration when determining any modification design.

6.4 Calibration Error Test. Immediately prior to the test series, (within 2 hours of the start of the test) introduce zero gas and

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* Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

high-level calibration gas at the calibration valve assembly. Adjust the analyzer output to the appropriate levels, if necessary. Calculate the predicted response for the low-level and mid-level gases based on a linear response line between the zero and high-level responses. Then introduce low-level and mid-level calibration gases successively to the measurement system. Record the analyzer responses for low-level and mid-level calibration gases and determine the differences between the measurement system responses and the predicted responses. These differences must be less than 5 percent of the respective calibration gas value. If not, the measurement system is not acceptable and must be replaced or repaired prior to testing. No adjustments to the measurement system shall be conducted after the calibration and before the drift check (Section 7.3). If adjustments are necessary before the completion of the test series, perform the drift checks prior to the required adjustments and repeat the calibration following the adjustments. If multiple electronic ranges are to be used, each additional range must be checked with a mid-level calibration gas to verify the multiplication factor.

6.5 Response Time Test. Introduce zero gas into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas. Record the time from the concentration change to the measurement system response equivalent to 95 percent of the step change. Repeat the test three times and average the results.

7. *Emission Measurement Test Procedure*

7.1 Organic Measurement. Begin sampling at the start of the test period, recording time and any required process information as appropriate. In particular, note on the recording chart periods of process interruption or cyclic operation.

7.2 Drift Determination. Immediately following the completion of the test period and hourly during the test period, reintroduce the zero and mid-level calibration gases, one at a time, to the measurement system at the calibration valve assembly. (Make no adjustments to the measurement system until after both the zero and calibration drift checks are made.) Record the analyzer response. If the drift values exceed the specified limits, invalidate the test results preceding the check and repeat the test following corrections to the measurement system. Alternatively, recalibrate the test measurement system as in Section 6.4 and report the results using both sets of calibration data (i.e., data determined prior to the test period and data determined following the test period).

8. *Organic Concentration Calculations*

Determine the average organic concentration in terms of ppmv as propane or other calibration gas. The average shall be determined by the integration of the output recording over the period specified in the applicable regulation.

If results are required in terms of ppmv as carbon, adjust measured concentrations using Equation 25A-1.

$$C_c = K\,C_{meas} \qquad \text{Eq. 25A-1}$$

Where:

C_c=Organic concentration as carbon, ppmv.

C_{meas}=Organic concentration as measured, ppmv.

K=Carbon equivalent correction factor,

K=2 for ethane.

K=3 for propane.

K=4 for butane.

K=Appropriate response factor for other organic calibration gases.

9. *Bibliography*

9.1 Measurement of Volatile Organic Compounds—Guideline Series. U.S. Environmental Protection Agency. Research Triangle Park, NC. Publication No. EPA-450/2-78-041. June 1978. p. 46-54.

9.2 Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Continuous Source Emission Monitors (Protocol No. 1). U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory. Research Triangle Park, NC. June 1978.

9.3 Gasoline Vapor Emission Laboratory Evaluation—Part 2. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. EMB Report No. 75-GAS-6. August 1975.

delayed, timely notice will be published in the Federal Register.

ADDRESSES: Copies of the state submittal for this action are available for public inspection during normal business hours at: the Environmental Protection Agency, Region VII, Air Branch, 726 Minnesota Avenue, Kansas City, Kansas 66101; Public Information Reference Unit, Environmental Protection Agency, 401 M Street SW., Washington, DC 20460; Environmental Protection Division, Iowa Department of Natural Resources, Wallace State Office Building, 900 East Grand, Des Moines, Iowa 50319.

FOR FURTHER INFORMATION CONTACT: Wayne Kaiser at (913) 551-7603 (FTS 276-7603).

SUPPLEMENTARY INFORMATION: On October 17, 1988, EPA revised the prevention of significant deterioration (PSD) regulations at 40 CFR 52.21 (see 53 FR 40656) for nitrogen oxides. These regulations establish the maximum increase in ambient nitrogen dioxide concentrations allowed in an area above the baseline concentration; these maximum allowable increases are called increments. The intended effect of these regulations is to require all applicants for major new stationary sources and major modifications emitting nitrogen oxides to account for and, if necessary, restrict emissions so as not to cause or contribute to exceedances of the increment.

On November 20, 1990, the IDNR submitted an amendment to chapter 22.4(455B), "Special Requirements for Major Stationary Sources Located in Areas Designated Attainment or Unclassified (PSD)," which incorporates by reference the revisions to 40 CFR part 52.21, effective October 17, 1988. The state rule was effective November 21, 1990. The state also provided a demonstration that it meets the conditions for approval of adoption of the NO_x increment program as detailed in the EPA guidance memorandum on the subject dated August 17, 1990.

The above memorandum described specific conditions for EPA approval of a state's adoption of the NO_x increment rule. Those conditions pertained to regulatory language, increment consumption analysis, increment consumption for the transition period, and legal authority. EPA has evaluated the state's submittal in accordance with the August 17, 1990, guidance and finds that the state submittal is acceptable. EPA is publishing this action without prior proposal because the Agency views this as a noncontroversial amendment and anticipates no adverse comments. This action will be effective April 15, 1991, unless, within 30 days of its publication, notice is received that adverse or critical comments will be submitted.

If such notice is received, this action will be withdrawn before the effective date by publishing two subsequent notices. One notice will withdraw the final action and another will begin a new rulemaking by announcing a proposal of the action and establishing a comment period. If no such comments are received, the public is advised that this action will be effective April 15, 1991.

EPA Action: EPA is taking final action to approve a revision to Iowa rule 567-22.4(455B) which adopts by reference the PSD NO_x requirements of 40 CFR 52.21 at 53 FR 40656 (October 17, 1988). Nothing in this action should be construed as permitting or allowing or establishing a precedent for any future request for revision to any SIP. Each request for revision to the SIP shall be considered separately in light of specific technical, economic, and environmental factors and in relation to relevant statutory and regulatory requirements. Under 5 U.S.C. 605(b), I certify that this SIP revision will not have a significant economic impact on a substantial number of small entities (See 46 FR 8709).

This action has been classified as a Table 3 action by the Regional Administrator under the procedures published in the Federal Register on January 19, 1989 (54 FR 2214-2225). On January 6, 1989, the Office of Management and Budget waived Tables 2 and 3 SIP revisions (54 FR 2222) from the requirements of Section 3 of Executive Order 12291 until April 1991.

Under section 307(b)(1) of the Act, petitions for judicial review of this action must be filed in the U.S. Court of Appeals for the appropriate circuit by April 15, 1991. This action may not be challenged later in proceedings to enforce its requirements. (See section 307(b)(2).)

The Agency has reviewed this request for revision of the federally approved SIP for conformance with the provisions of the 1990 Amendments enacted on November 15, 1990. The Agency has determined that this action conforms with those requirements irrespective of the fact that the adoption of the revision by the state preceded the date of enactment.

List of Subjects in 40 CFR Part 52

Air pollution control, Incorporation by reference, Intergovernmental relations, Nitrogen dioxide, Particulate matter, Sulfur oxides.

Dated: January 28, 1991.

Morris Kay,

Regional Administrator.

40 CFR part 52, subpart Q, is amended as follows:

PART 52—[AMENDED]

1. The authority citation for part 52 continues to read as follows:

Authority: 42 U.S.C. 7401-7642.

2. Section 52.820 is amended by adding paragraph (c)(53) to read as follows:

§ 52.820 Identification of plan.

* * * * *

(c) * * *

(53) Revised chapter 22, rule 22.4(455B), submitted on November 8, 1990, incorporates by reference revised EPA PSD rules pertaining to NO_x increments.

(i) Incorporation by reference

(A) Amendment to chapter 22, "Controlling Pollution," Iowa Administrative Code, subrule 22.4, adopted by the Environmental Protection Commission on October 17, 1990, effective November 21, 1990.

(ii) Additional material

(A) Letter from the state dated November 8, 1990, pertaining to NO_x rules and analysis which certifies the material was adopted by the state on October 17, 1990.

[FR Doc. 91-3451 Filed 2-12-91; 8:45 am]

BILLING CODE 6560-50-M

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40 CFR Part 60

[AD-FRL-3867-1]

Standards of Performance for New Stationary Sources; Addition of Methods for Measurement of Polychlorinated Dibenzo-p-Dioxins, Polychlorinated Dibenzofurans, and Hydrogen Chloride Emissions From Stationary Sources

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: The purpose of this action is to add Method 23, "Determination to Polychlorinated Dibenzo-p-Dioxins (PCDD's) and Polychlorinated Dibenzofurans (PCDF's) from Stationary Sources," and Method 26, "Determination of Hydrogen Chloride Emissions from Stationary Sources" to appendix A of 40 CFR part 60. These methods are being promulgated to determine compliance with subparts Ca and Ea of part 60.

DATES: *Effective Date:* February 13, 1991.

Judicial Review: Under section 307(b)(1) of the Clean Air Act, judicial review of the actions taken by this notice is available only by the filing of a petition for review in the U.S. Court of Appeals for the District of Columbia Circuit within 60 days of today's publication of this rule. Under section 307(b)(2) of the Clean Air Act, the requirements that are the subject of today's notice may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

ADDRESSES: *Background Information Document.* The Background Information Document for the promulgated test methods may be obtained from Gary McAlister or Roger Shigehara, MD-19, U.S. EPA, Research Triangle Park, North Carolina 27711, telephone number (919) 541-1062. Please refer to "Summary of Comments and Responses for Methods 23 and 26."

Docket. Docket Number A-89-11, containing material relevant to this rulemaking, is available for public inspection and copying between 8:30 a.m. and 3:30 p.m. Monday through Friday, at EPA's Air Docket Section, room M-1500, 1st Floor, Waterside Mall, 401 M Street SW., Washington, DC 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: Gary McAlister or Roger Shigehara, Emission Measurement Branch (MD-19), Technical Support Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone number (919) 541-1062.

SUPPLEMENTARY INFORMATION:

I. The Rulemaking

Under Subparts Ca and Ea, the EPA is regulating emissions from municipal waste combustors (MWC's) including setting emission limits for polychlorinated dibenzo-p-dioxins (PCDD's), polychlorinated dibenzofurans (PCDF's), and hydrochloric acid (HCl). There are presently no methods published in 40 CFR part 60, appendix A, to measure any of these pollutants. This action would promulgate one method to measure the PCDD's and PCDF's and another method to measure the HCl.

Summary of Reference Methods

Method 23 is used to measure the emission of PCDD's and PCDF's from MWC's. A sample is withdrawn isokinetically from the stack through a probe, a filter, and a trap packed with a solid adsorbent. The PCDD's and PCDF's are collected in the probe, on the filter, and on the solid adsorbent. The PCDD's and PCDF's are extracted from the particulate matter and the adsorbent with a hot organic solvent. The extracted PCDD's and PCDF's are separated by capillary gas chromatography, and then, each isomer is identified and measured with mass spectrometry (GC/MS). The total PCDD's and PCDF's are the sum of the individual isomers. Toxicity factors are not used in the calculation.

Method 26 is used to measure the emission of HCl from MWC's. A sample is withdrawn at a constant rate from the stack through a probe and impingers filled with a dilute acid. The HCl is collected in the implinger solution. The chloride ion is separated by ion chromatography and measured by a conductivity detector.

Background

In 1983, the American Society of Mechanical Engineers (ASME) recognized that the testing for PCDD's and PCDF's needed to be standardized. In February of 1984, the ASME convened a committee of government representatives, testing consultants, equipment manufacturers, and incinerator operators to write a standard test procedure for PCDD's and PCDF's. This was eventually distributed as a draft ASME protocol in December of 1984. The procedure that we are promulgating was derived from this draft ASME protocol. There are some changes in the quality assurance (QA) requirements and the solvents used to recover the sample in the promulgated method. Because more labeled compounds are available, the method will require additional labeled internal standards and surrogate compounds which will provide better representation of the entire range of PCDD's and PCDF's. The filter and solid adsorbent are extracted in the laboratory with toluene to assure a high PCDD and PCDF recovery efficiency. Additionally the proposed sample recovery solvents used for rinsing the sample train glassware in the field would be acetone followed by methylene chloride with a final quality assurance rinse using toluene. However, the results from the toluene rinse are not used in calculating the total PCDD and PCDF emissions. EPA will continue to review the toluene field rinse quality assurance results and continue to evaluate the desirability of replacing methylene chloride with toluene for field rinsing sample glassware.

II. Public Participation

The opportunity to hold a public hearing at 10 a.m. on February 7, 1990 was presented, but no one requested a hearing. The public comment period was from December 20, 1989 to March 5, 1990.

III. Significant Comments and Changes to the Proposed Rulemaking

Thirteen comment letters were received on the proposed test methods. These comments have been carefully considered and, where deemed appropriate by the Administrator, changes have been made in the proposed test methods. A detailed discussion of these comments is contained in the background document entitled, "Summary of Comments and Responses for Methods 23 and 26" which is referred to in the **ADDRESSES** section of this preamble.

Several commenters thought that there were not enough stack sampling organizations that were experienced with Method 23 to avoid major delays in securing sampling and analysis contractors. Many of these commenters also thought that there would not be an adequate supply of calibration standards and audit samples. We believe that the number of tests required immediately after promulgation of the regulation will not exceed the capabilities of the available sampling and analytical laboratories and that there will be an adequate supply of labeled standards and audit samples. The full effect of the testing requirements for the new and existing sources will not be felt for about five years. We believe that this is adequate time to allow for the necessary expansion of testing capabilities.

Some commenters requested alternative procedures or methods to Method 23. While testers always have the option of requesting alternative methods, requests should be submitted after the method becomes final. Any request should be in writing and should be accompanied by any supporting data.

Many commenters thought that the gas chromatography columns specified in Method 23 were not the most appropriate choice. The column requirements in the method have been revised to allow the tester to use any column system provided that the tester can demonstrate through calibration and performance checks that the columns provide the necessary isomer separation.

One commenter thought that Method 26 should be modified to allow isokinetic sampling so that it could be lied to sources where hydrochloric . aerosols are present. We agree that inetic sampling may be important at ces other than MWC's. We have pared Method 26 and an isokinetic pling train and found that they erally give similar results at stack centrations above 20 PPM. At lower centrations the isokinetic sampling n seems to have a negative bias. We continuing to investigate this blem, and may be able to approve an rnative method using isokinetic pling for future use.

nother commenter wanted EPA to elop a QA audit sample for Method An audit sample is being developed will be available for validating the ilts of compliance tests.

Administrative

he docket is an organized and plete file of all the information mitted to or otherwise considered by in the development of this making. The principal purposes of docket are: (1) To allow interested ties to identify and locate documents hat they can effectively participate he rulemaking process and (2) to ve as the record in case of judicial iew (except for interagency review terials) (section 307(d)(7)(A)).

Inder Executive Order 12291, EPA st judge whether a regulation is ajor" and, therefore, subject to the uirement of a regulatory impact lysis. The Agency has determined t this rulemaking would not result in any of the adverse economic effects set forth in Section 1 of the Order as grounds for finding a "major rule." The Agency has, therefore, concluded that this regulation is not a "major rule" under Executive Order 12291.

The Regulatory Flexibility Act (RFA) of 1980 requires the identification of potentially adverse impacts of Federal regulations upon small business entities. The Act specifically requires the completion of an RFA analysis in those instances where small business impacts are possible. Because this rulemaking imposes no adverse economic impacts, an analysis has not been conducted.

Pursuant to the provisions of 5 U.S.C. 605(b), I hereby certify that this promulgated rule will not have an economic impact on small entities because no additional costs will be incurred from this action.

This rule does not contain any information collection requirements currently approved by OMB review under the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.*

List of Subjects in 40 CFR Part 60

Air pollution control, Municipal waste combustors, Polychlorinated dibenzo-p-dioxins, Polychlorinated dibenzofurans, Hydrogen chloride.

Dated: January 11, 1991.

F. Henry Habicht,

Acting Administrator.

Title 40, part 60 of the Code of Federal Regulations is amended as follows:

PART 60—[AMENDED]

1. The authority citation for part 60 continues to read as follows:

Authority: 42 U.S.C. 7401, 7411, 7414, 7416, and 7601.

2. By adding in numerical order Methods 23 and 26 to appendix A as follows:

Appendix A—Reference Methods

* * * * *

Method 23—Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans From Stationary Sources

1. Applicability and Principle

1.1 Applicability. This method is applicable to the determination of polychlorinated dibenzo-p-dioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's) from stationary sources.

1.2 Principle. A sample is withdrawn from the gas stream isokinetically and collected in the sample probe, on a glass fiber filter, and on a packed column of adsorbent material. The sample cannot be separated into a particle vapor fraction. The PCDD's and PCDF's are extracted from the sample, separated by high resolution gas chromatography, and measured by high resolution mass spectrometry.

2. Apparatus

2.1 Sampling. A schematic of the sampling train used in this method is shown in Figure 23-1. Sealing greases may not be used in assembling the train. The train is identical to that described in section 2.1 of Method 5 of this appendix with the following additions:

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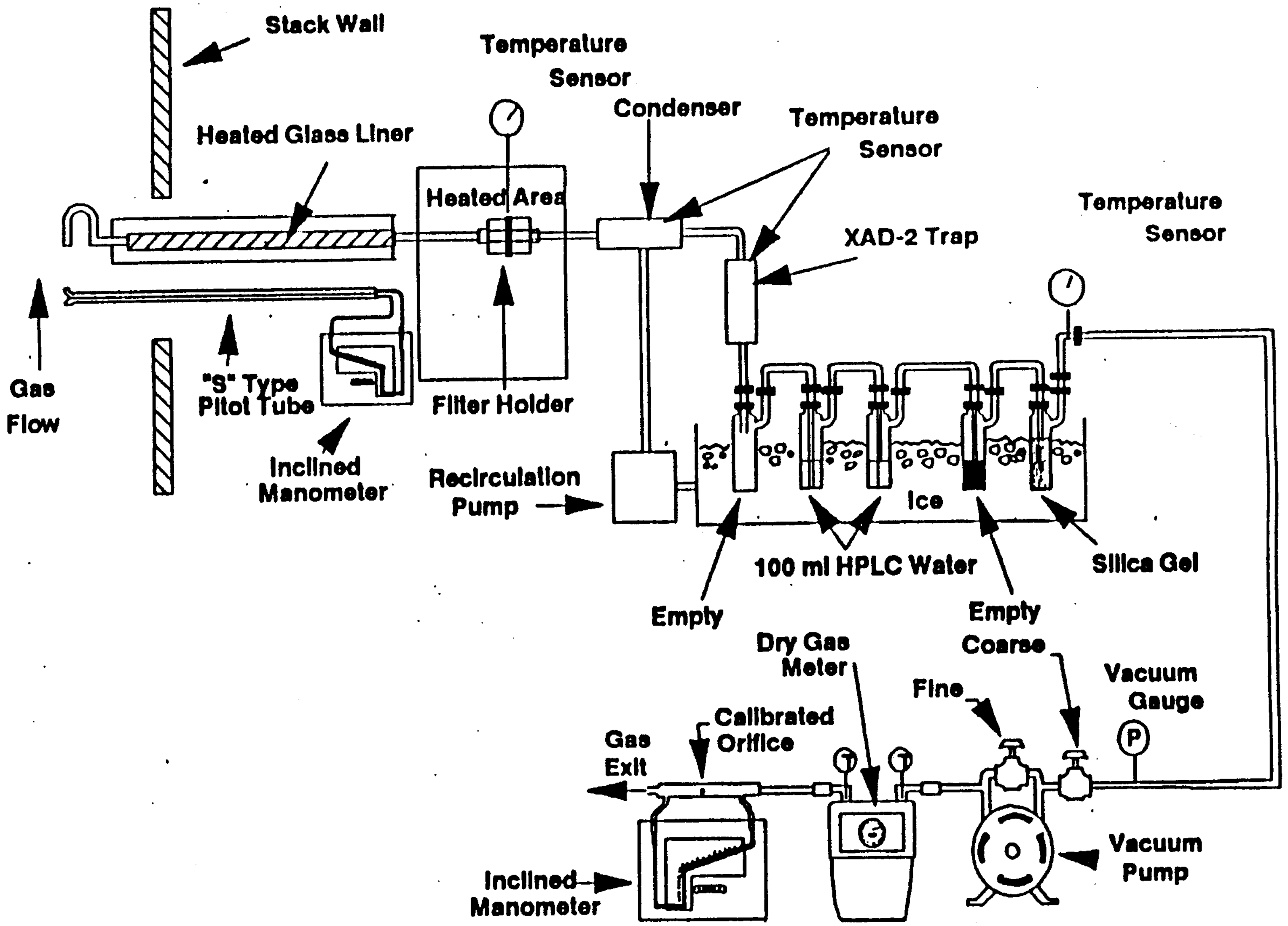

Figure 23.1 Sampling train

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2.1.1 Nozzle. The nozzle shall be made of ckel, nickel-plated stainless steel, quartz, or rosilicate glass.

2.1.2 Sample Transfer Lines. The sample nsfer lines, if needed, shall be heat traced. avy walled TFE (½ in. OD with ⅛ in. wall) th connecting fittings that are capable of rming leak-free, vacuum-tight connections thout using sealing greases. The line shall as short as possible and must be aintained at 120 °C.

2.1.1. Filter Support. Teflon or Teflon-ated wire.

2.1.2 Condenser. Glass, coil type with compatible fittings. A schematic diagram is shown in Figure 23–2.

2.1.3 Water Bath. Thermostatically controlled to maintain the gas temperature exiting the condenser at <20 °C (68 °F).

2.1.4 Adsorbent Module. Glass container to hold the solid adsorbent. A ahematic diagram is shown in Figure 23–2. Other physical configurations of the resin trap/condenser assembly are acceptable. The connecting fittings shall form leak-free, vacuum tight seals. No sealant greases shall be used in the sampling train. A coarse glass frit is included to retain the adsorbent.

2.2 Sample Recovery.

2.2.1 Fitting Caps. Ground glass, Teflon tape, or aluminum foil (Section 2.2.6) to cap off the sample exposed sections of the train.

2.2.2 Wash Bottles. Teflon, 500-ml.

2.2.3 Probe-Liner Probe-Nozzle, and Filter-Holder Brushes. Inert bristle brushes with precleaned stainless steel or Teflon handles. The probe brush shall have extensions of stainless steel or Teflon, at least as long as the probe. The brushes shall be properly sized and shaped to brush out the nozzle, probe liner, and transfer line, if used.

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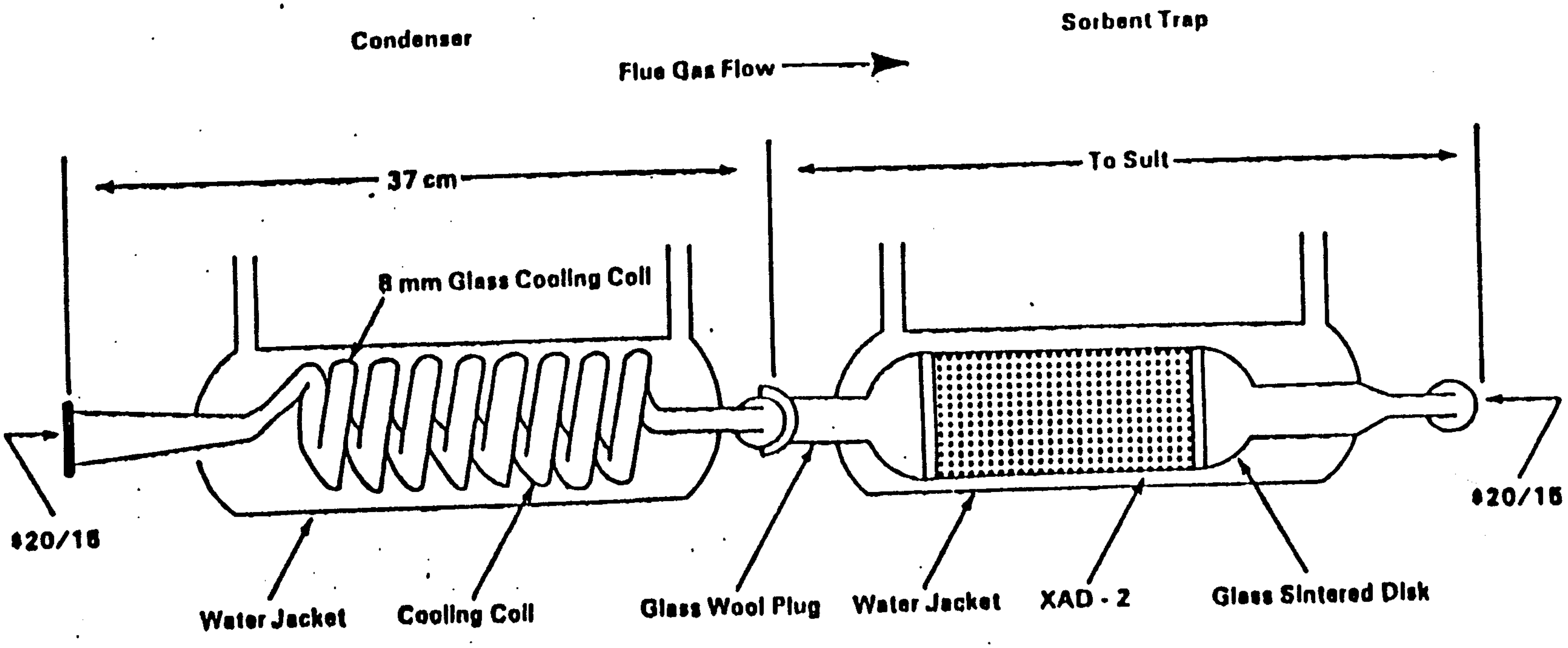

Figure 23.2. Condenser and adsorbent trap

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2.2.4 Filter Storage Container. Sealed ilter holder, wide-mouth amber glass jar with eflon-lined cap, or glass petri dish.

2.2.5 Balance. Triple beam.

2.2.6 Aluminum Foil. Heavy duty, hexane-insed.

2.2.7 Metal Storage Container. Air tight ontainer to store silica gel.

2.2.8 Graduated Cylinder. Glass, 250-ml vith 2-ml graduation.

2.2.9 Glass Sample Storage Container. mber glass bottle for sample glassware vashes, 500- or 1000-ml, with leak free eflon-lined caps.

2.3 Analysis.

2.3.1 Sample Container. 125- and 250-ml lint glass bottles with Teflon-lined caps.

2.3.2 Test Tube. Glass.

2.3.3 Soxhlet Extraction Apparatus. apable of holding 43 x 123 mm extraction himbles.

2.3.4 Extraction Thimble. Glass, recleaned cellulosic, or glass fiber.

2.3.5 Pasteur Pipettes. For preparing liquid hromatographic columns.

2.3.6 Reacti-vials. Amber glass, 2-ml, ilanized prior to use.

2.3.7 Rotary Evaporator. Buchi/Brinkman F–121 or equivalent.

2.3.8 Nitrogen Evaporative Concentrator. -Evap Analytical Evaporator Model III or quivalent.

2.3.9 Separatory Funnels. Glass, 2-liter.

2.3.10 Gas Chromatograph. Consisting of he following components:

2.3.10.1 Oven. Capable of maintaining the eparation column at the proper operating emperature ± °C and performing rogrammed increases in temperature at ates of at least 40 °C/min.

2.3.10.2 Temperature Gauge. To monitor olumn oven, detector, and exhaust emperatures ± 1 °C.

2.3.10.3 Flow System. Gas metering ystem to measure sample, fuel, combustion as, and carrier gas flows.

2.3.10.4 Capillary Columns. A fused silica column, 60 × 0.25 mm inside diameter (ID), coated with DB–5 and a fused silica column, 30 m × 0.25 mm ID coated with DB–225. Other column systems may be used provided that the user is able to demonstrate using calibration and performance checks that the column system is able to meet the specifications of section 6.1.2.2.

2.3.11. Mass Spectrometer. Capable of routine operation at a resolution of 1:10000 with a stability of ± 5 ppm.

2.3.12 Data System. Compatible with the mass spectrometer and capable of monitoring at least five groups of 25 ions.

2.3.13 Analytical Balance. To measure within 0.1 mg.

3. Reagents

3.1 Sampling.

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D 2986–71 (Reapproved 1978) (incorporated by reference—see § 60.17).

3.1.1.1 Precleaning. All filters shall be cleaned before their initial use. Place a glass extraction thimble and 1 g of silica gel and a plug of glass wool into a Soxhlet apparatus, charge the apparatus with toluene, and reflux for a minimum of 3 hours. Remove the toluene and discard it, but retain the silica gel. Place no more than 50 filters in the thimble onto the silica gel bed and top with the cleaned glass wool. Charge the Soxhlet with toluene and reflux for 16 hours. After extraction, allow the Soxhlet to cool, remove the filters, and dry them under a clean N_2 stream. Store the filters in a glass petri dish sealed with Teflon tape.

3.1.2 Adsorbent Resin. Amberlite XAD–2 resin. Thoroughly cleaned before initial use.

3.1.2.1 Cleaning Procedure. This procedure may be carried out in a giant Soxhlet extractor. An all-glass filter thimble containing an extra-course frit is used for extraction of XAD–2. The frit is recessed 10–15 mm above a crenelated 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 a stainless steel ring 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 water, and discard. Fill with water a second time, let stand overnight, and discard.
Water	Extract with water for 8 hours.
Methanol	Extract for 22 hours.
Methylene Chloride	Extract for 22 hours.
Toluene	Extract for 22 hours.

3.1.2.2 Drying.

3.1.2.2.1 Drying Column. Pyrex pipe, 10.2 cm ID by 0.6 m long, with suitable retainers.

3.1.2.2.2 Procedure. The adsorbent must be dried with clean inert gas. Liquid nitrogen from a standard commercial liquid nitrogen cylinder has proven to be a reliable source of large volumes of gas free from organic contaminants. Connect the liquid nitrogen cylinder to the column by a length of cleaned copper tubing, 0.95 cm ID, coiled to pass through a heat source. 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. Continue flowing nitrogen through the adsorbent until all the residual solvent is removed. The flow rate should be sufficient to gently agitate the particles but not so excessive as the cause the particles to fracture.

3.1.2.3 Quality Control Check. The adsorbent must be checked for residual toluene.

3.1.2.3.1 Extraction. Weigh 1.0 g sample of dried resin into a small vial, add 3 ml of toluene, cap the vial, and shake it well.

3.1.2.3.2 Analysis. Inject a 2 µl sample of the extract into a gas chromatograph operated under the following conditions:

Column: 6 ft × ⅛ in stainless steel containing 10 percent OV–101 on 100/120 Supelcoport.
Carrier Gas: Helium at a rate of 30 ml/min.
Detector: Flame ionization detector operated at a sensitivity of 4×10^{-11} A/mV.
Injection Port Temperature: 250 °C.
Detector Temperature: 305 °C.
Oven Temperature: 30 °C for 4 min; programmed to rise at 40 °C/min until it reaches 250 °C; return to 30 °C after 17 minutes.

Compare the results of the analysis to the results from the reference solution. Prepare the reference solution by injection 2.5 µl of methylene chloride into 100 ml of toluene. This corresponds to 100 µg of methylene chloride per g of adsorbent. The maximum acceptable concentration is 1000 µg/g of adsorbent. If the adsorbent exceeds this level, drying must be continued until the excess methylene chloride is removed.

3.1.2.4 Storage. The adsorbent must be used within 4 weeks of cleaning. After cleaning, it may be stored in a wide mouth amber glass container with a Teflon-lined cap or placed in one of the glass adsorbent modules tightly sealed with glass stoppers. If precleaned adsorbent is purchased in sealed containers, it must be used within 4 weeks after the seal is broken.

3.1.3 Glass Wool. Cleaned by sequential immersion in three aliquots of methylene chloride, dried in a 110 °C oven, and stored in a methylene chloride-washed glass jar with a Teflon-lined screw cap.

3.1.4 Water. Deionized distilled and stored in a methylene chloride-rinsed glass container with a Teflon-lined screw cap.

3.1.5 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 °C (350 °F) for two hours. New silica gel may be used as received. Alternately other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.6 Chromic Acid Cleaning Solution. Dissolve 20 g of sodium dichromate in 15 ml of water, and then carefully add 400 ml of concentrated sulfuric acid.

3.2 Sample Recovery.

3.2.2 Acetone. Pesticide quality.

3.2.2 Methylene Chloride. Pesticide qualtity.

3.2.3 Toluene. Pesticide quality.

3.3 Analysis.

3.3.1 Potassium Hydroxide. ACS grade, 2-percent (weight/volume) in water.

3.3.2 Sodium Sulfate. Granulated, reagent grade. Purify prior to use by rinsing with methylene chloride and oven drying. Store the cleaned material in a glass container with a Teflon-lined screw cap.

3.3.3 Sulfuric Acid. Reagent grade.

3.3.4 Sodium Hydroxide. 1.0 N. Weigh 40 g of sodium hydroxide into a 1-liter volumetric flask. Dilute to 1 liter with water.

3.3.5 Hexane. Pesticide grade.

3.3.6 Methylene Chloride. Pesticide grade.

3.3.7 Benzene. Pesticide Grade.

3.3.8 Ethyl Acetate.

3.3.9 Methanol. Pesticide Grade.

3.3.10 Toluene. Pesticide Grade.

3.3.11 Nonane. Pesticide Grade.

3.3.12 Cyclohexane. Pesticide Grade.

3.3.13 Basic Alumina. Activity grade 1, 100–200 mesh. Prior to use, activate the alumina by heating for 16 hours at 130 °C before use. Store in a desiccator. Pre-activated alumina may be purchased from a supplier and may be used as received.

3.3.14 Silica Gel. Bio-Sil A, 100–200 mesh. Prior to use, activate the silica gel by heating for at least 30 minutes at 180 °C. After cooling, rinse the silica gel sequentially with methanol and methylene chloride. Heat the rinsed silica gel at 50 °C for 10 minutes, then increase the temperature gradually to 180 °C over 25 minutes and maintain it at this temperature for 90 minutes. Cool at room temperature and store in a glass container with a Teflon-lined screw cap.

3.3.15 Silica Gel Impregnated with Sulfuric Acid. Combine 100 g of silica gel with 44 g of concentrated sulfuric acid in a screw capped glass bottle and agitate thoroughly. Disperse the solids with a stirring rod until a uniform mixture is obtained. Store the mixture in a glass container with a Teflon-lined screw cap.

3.3.16 Silica Gel Impregnated with Sodium Hydroxide. Combine 39 g of 1 N sodium hydroxide with 100 g of silica gel in a screw capped glass bottle and agitate thoroughly. Disperse solids with a stirring rod until a uniform mixture is obtained. Store the mixture in glass container with a Teflon-lined screw cap.

3.3.17 Carbon/Celite. Combine 10.7 g of AX-21 carbon with 124 g of Celite 545 in a 250-ml glass bottle with a Teflon-lined screw cap. Agitate the mixture thoroughly until a uniform mixture is obtained. Store in the glass container.

3.3.18 Nitrogen. Ultra high purity.

3.3.19 Hydrogen. Ultra high purity.

3.3.20 Internal Standard Solution. Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 1 under the heading "Internal Standards" in 10 ml of nonane.

3.3.21 Surrogate Standard Solution. Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 1 under the heading "Surrogate Standards" in 10 ml of nonane.

3.3.22 Recovery Standard Solution. Prepare a stock standard solution containing the isotopically labelled PCDD's and PCDF's at the concentrations shown in Table 1 under the heading "Recovery Standards" in 10 ml of nonane.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation.

4.1.1.1 Cleaning Glassware. All glass components of the train upstream of and including the adsorbent module, shall be cleaned as described in section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." Special care shall be devoted to the removal of residual silicone grease sealants on ground glass connections of used glassware. Any residue shall be removed by soaking the glassware for several hours in a chromic acid cleaning solution prior to cleaning as described above.

4.1.1.2 Adsorbent Trap. The traps must be loaded in a clean area to avoid contamination. They may not be loaded in the field. Fill a trap with 20 to 40 g of XAD-2. Follow the XAD-2 with glass wool and tightly cap both ends of the trap. Add 100 μl of the surrogate standard solution (section 3.3.21) to each trap.

4.1.1.3 Sample Train. It is suggested that all components be maintained according to the procedure described in APTD-0576.

4.1.1.4 Silica Gel. Weigh several 200 to 300 g portions of silica gel in an air tight container to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel may be weighed directly in its impinger or sampling holder just prior to sampling.

4.1.1.5 Filter. Check each filter against light for irregularities and flaws or pinhole leaks. Pack the filters flat in a clean glass container.

4.1.2 Preliminary Determinations. Same as section 4.1.2 of Method 5.

4.1.3 Preparation of Collection Train.

4.1.3.1 During preparation and assembly of the sampling train, keep all train openings where contamination can enter, sealed until just prior to assembly or until sampling is about to begin.

Note: Do not use sealant grease in assembling the train.

4.1.3.2 Place approximately 100 ml of water in the second and third impingers, leave the first and fourth impingers empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fifth impinger.

4.1.3.3 Place the silica gel 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.

4.1.3.4 Assemble the train as shown in Figure 23-1.

4.1.3.5 Turn on the adsorbent module and condenser coil recirculating pump and begin monitoring the adsorbent module gas entry temperature. Ensure proper sorbent temperature gas entry temperature before proceeding and before sampling is initiated. It is extremely important that the XAD-2 adsorbent resin temperature never exceed 50 °C because thermal decomposition will occur. During testing, the XAD-2 temperature must not exceed 20 °C for efficient capture of the PCDD's and PCDF's.

4.1.4 Leak-Check Procedure. Same as Method 5, section 4.1.4.

4.1.5 Sample Train Operation. Same as Method 5, section 4.1.5.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Seal the nozzle end of the sampling probe with Teflon tape or aluminum foil.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe. Remove the probe from the train and close off both ends with aluminum foil. Seal off the inlet to the train with Teflon tape, a ground glass cap, or aluminum foil.

Transfer the probe and impinger assembly to the cleanup area. This area shall be clean and enclosed so that the chances of losing or contaminating the sample are minimized. Smoking, which could contaminate the sample, shall not be allowed in the cleanup area.

Inspect the train prior to and during disassembly and note any abnormal conditions, e.g., broken filters, colored impinger liquid, etc. Treat the samples as follows:

4.2.1 Container No. 1. Either seal the filter holder or carefully remove the filter from the filter holder and place it in its identified container. Use a pair of cleaned tweezers to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the container any particulate matter and filter fibers which adhere to the filter holder gasket, by using a dry inert bristle brush and a sharp-edged blade. Seal the container.

4.2.2 Adsorbent Module. Remove the module from the train, tightly cap both ends, label it, cover with aluminum foil, and store it on ice for transport to the laboratory.

4.2.3 Container No. 2. Quantitatively recover material deposited in the nozzle, probe transfer lines, the front half of the filter holder, and the cyclone, if used, first, by brushing while rinsing three times each with acetone and then, by rinsing the probe three times with methylene chloride. Collect all the rinses in Container No. 2.

Rinse the back half of the filter holder three times with acetone. Rinse the connecting line between the filter and the condenser three times with acetone. Soak the connecting line with three separate portions of methylene chloride for 5 minutes each. If using a separate condenser and adsorbent trap, rinse the condenser in the same manner as the connecting line. Collect all the rinses in Container No. 2 and mark the level of the liquid on the container.

4.2.4 Container No. 3. Repeat the methylene chloride-rinsing described in Section 4.2.3 using toluene as the rinse solvent. Collect the rinses in Container No. 3 and mark the level of the liquid on the container.

4.2.5 Impinger Water. 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. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight.

4.2.7 Silica Gel. Note the color of the indicating silica gel to determine if it has been completely spent and make a mention of its condition. Transfer the silica gel from the fifth impinger to its original container and seal.

5. Analysis

All glassware shall be cleaned as described in section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." All samples must be extracted ithin 30 days of collection and analyzed ithin 45 days of extraction.

5.1 Sample Extraction.

5.1.1 Extraction System. Place an xtraction thimble (section 2.3.4), 1 g of silica el, and a plug of glass wool into the Soxhlet pparatus, charge the apparatus with toluene, nd reflux for a minimum of 3 hours. Remove e toluene and discard it, but retain the lica gel. Remove the extraction thimble om the extraction system and place it in a ass beaker to catch the solvent rinses.

5.1.2 Container No. 1 (Filter). Transfer the ontents directly to the glass thimble of the xtraction system and extract them multaneously with the XAD–2 resin.

5.1.3 Adsorbent Cartridge. Suspend the dsorbent module directly over the extraction imble in the beaker (See section 5.1.1). The lass frit of the module should be in the up osition. Using a Teflon squeeze bottle ontaining toluene, flush the XAD–2 into the imble onto the bed of cleaned silica gel. horoughly rinse the glass module catching he rinsings in the beaker containing the himble. If the resin is wet, effective xtraction can be accomplished by loosely acking the resin in the thimble. Add the AD–2 glass wool plug into the thimble.

5.1.4 Container No. 2 (Acetone and ethylene Chloride). Concentrate the sample o a volume of about 1–5 ml using the rotary vaporator apparatus, at a temperature of ess than 37 °C. Rinse the sample container hree times with small portions of methylene hloride and add these to the concentrated olution and concentrate further to near ryness. This residue contains particulate atter removed in the rinse of the train probe nd nozzle. Add the concentrate to the filter nd the XAD–2 resin in the Soxhlet apparatus escribed in section 5.1.1.

5.1.5 Extraction. Add 100 μl of the nternal standard solution (Section 3.3.20) to he extraction thimble containing the ontents of the adsorbent cartridge, the ontents of Container No. 1, and the oncentrate from section 5.1.4. Cover the ontents of the extraction thimble with the leaned glass wool plug to prevent the XAD–2 resin from floating into the solvent reservoir of the extractor. Place the thimble in the extractor, and add the toluene contained in the beaker to the solvent reservoir. Pour additional toluene to fill the reservoir approximately 2/3 full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle three times per hour. Extract the sample for 16 hours. After extraction, allow the Soxhlet to cool. Transfer the toluene extract and three 10-ml rinses to the rotary evaporator. Concentrate the extract to approximately 10 ml. At this point the analyst may choose to split the sample in half. If so, split the sample, store one half for future use, and analyze the other according to the procedures in sections 5.2 and 5.3. In either case, use a nitrogen evaporative concentrator to reduce the volume of the sample being analyzed to near dryness. Dissolve the residue in 5 ml of hexane.

5.1.6 Container No. 3 (Toluene Rinse). Add 100 μl of the Internal Standard solution (section 3.3.2) to the contents of the container. Concentrate the sample to a volume of about 1–5 ml using the rotary evaporator apparatus at a temperature of less than 37 °C. Rinse the sample container apparatus at a temperature of less than 37 °C. Rinse the sample container three times with small portions of toluene and add these to the concentrated solution and concentrate further to near dryness. Analyze the extract separately according to the procedures in sections 5.2 and 5.3, but concentrate the solution in a rotary evaporator apparatus rather than a nitrogen evaporative concentrator.

5.2 Sample Cleanup and Fractionation.

5.2.1 Silica Gel Column. Pack one end of a glass column, 20 mm x 230 mm, with glass wool. Add in sequence, 1 g silica gel, 2 g of sodium hydroxide impregnated silica gel, 1 g silica gel, 4 g of acid-modified silica gel, and 1 g of silica gel. Wash the column with 30 ml of hexane and discard it. Add the sample extract, dissolved in 5 ml of hexane to the column with two additional 5-ml rinses. Elute the column with an additional 90 ml of hexane and retain the entire eluate. Concentrate this solution to a volume of about 1 ml using the nitrogen evaporative concentrator (section 2.3.7).

5.2.2 Basic Alumina Column. Shorten a 25-ml disposable Pasteur pipette to about 16 ml. Pack the lower section with glass wool and 12 g of basic alumina. Transfer the concentrated extract from the silica gel column to the top of the basic alumina column and elute the column sequentially with 120 ml of 0.5 percent methylene chloride in hexane followed by 120 ml of 35 percent methylene chloride in hexane. Discard the first 120 ml of eluate. Collect the second 120 ml of eluate and concentrate it to about 0.5 ml using the nitrogen evaporative concentrator.

5.2.3 AX–21 Carbon/Celite 545 Column. Remove the botton 0.5 in. from the tip of a 9-ml disposable Pasteur pipette. Insert a glass fiber filter disk in the top of the pipette 2.5 cm from the constriction. Add sufficient carbon/celite mixture to form a 2 cm column. Top with a glass wool plug. In some cases AX–21 carbon fines may wash through the glass wool plug and enter the sample. This may be prevented by adding a celite plug to the exit end of the column. Rinse the column in sequence with 2 ml of 50 percent benzene in ethyl acetate, 1 ml of 50 percent methylene chloride in cyclohexane, and 2 ml of hexane. Discard these rinses. Transfer the concentrate in 1 ml of hexane from the basic alumina column to the carbon/celite column along with 1 ml of hexane rinse. Elute the column sequentially with 2 ml of 50 percent methylene chloride in hexane and 2 ml of 50 percent benzene in ethyl acetate and discard these eluates. Invert the column and elute in the reverse direction with 13 ml of toluene. Collect this eluate. Concentrate the eluate in a rotary evaporator at 50 °C to about 1 ml. Transfer the concentrate to a Reacti-vial using a toluene rinse and concentrate to a volume of 200 μl using a stream of N_2. Store extracts at room temperature, shielded from light, until the analysis is performed.

5.3 Analysis. Analyze the sample with a gas chromatograph coupled to a mass spectrometer (GC/MS) using the instrumental parameters in sections 5.3.1 and 5.3.2. Immediately prior to analysis, add a 20 μl aliquot of the Recovery Standard solution from Table 1 to each sample. A 2 μl aliquot of the extract is injected into the GC. Sample extracts are first analyzed using the DB–5 capillary column to determine the concentration of each isomer of PCDD's and PCDF's (tetra-through octa-). If tetra-chlorinated dibenzofurans are detected in this analysis, then analyze another aliquot of the sample in a separate run, using the DB–225 column to measure the 2,3,7,8 tetra-chloro dibenzofuran isomer. Other column systems may be used, provided that the user is able to demonstrate using calibration and performance checks that the column system is able to meet the specifications of section 6.1.2.2.

5.3.1 Gas Chromatograph Operating Conditions.

5.3.1.1 Injector. Configured for capillary column, splitless, 250°C.

5.3.1.2 Carrier Gas. Helium, 1–2 ml/min.

5.3.1.3 Oven. Initially at 150°C. Raise by at least 40°C/min to 190°C and then at 3°C/min up to 300°C.

5.3.2 High Resolution Mass Spectrometer.

5.3.2.1 Resolution. 10000 m/e.

5.3.2.2 Ionization Mode. Electron impact.

5.3.2.3 Source Temperature 250°C.

5.3.2.4 Monitoring Mode. Selected ion monitoring. A list of the various ions to be monitored is summarized in Table 3.

5.3.2.5 Identification Criteria. The following identification criteria shall be used for the characterization of polychlorinated dibenzodioxins and dibenzofurans.

1. The integrated ion-abundance ratio (M/M+2 or M+2/M+4) shall be within 15 percent of the theoretical value. The acceptable ion-abundance ratio ranges for the identification of chlorine-containing compounds are given in Table 4.

2. The retention time for the analytes must be within 3 seconds of the corresponding ^{13}C-labeled internal standard, surrogate or alternate standard.

3. The monitored ions, shown in Table 3 for a given analyte, shall reach their maximum within 2 seconds of each other.

4. The identification of specific isomers that do not have corresponding ^{13}C-labeled standards is done by comparison of the relative retention time (RRT) of the analyte to the nearest internal standard retention time with reference (i.e., within 0.005 RRT units) to the comparable RRT's found in the continuing calibration.

5. The signal to noise ratio for all monitored ions must be ~~greater than 2.5~~.

6. The confirmation of 2, 3, 7, 8–TCDD and 2, 3, 7, 8–TCDF shall satisfy all of the above identification criteria.

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7. For the identification of PCDF's, no signal may be found in the corresponding PCDPE channels.

5.3.2.6 Quantification. The peak areas for the two ions monitored for each analyte are summed to yield the total response for each analyte. Each internal standard is used to quantify the indigenous PCDD's or PCDF's in its homologous series. For example, the $^{13}C_{12}$–2,3,7,8-tetra chlorinated dibenzodioxin is used to calculate the concentrations of all other tetra chlorinated isomers. Recoveries of the tetra- and penta- internal standards are calculated using the $^{13}C_{12}$–1.2.3.4–TCDD. Recoveries of the hexa- through octa- internal standards are calculated using $^{13}C_{12}$–1.2.3.7.8.9–HxCDD. Recoveries of the surrogate standards are calculated using the corresponding homolog from the internal standard.

6. Calibration

Same as Method 5 with the following additions.

6.1 GC/MS System.

6.1.1 Initial Calibration. Calibrate the GC/MS system using the set of five standards shown in Table 2. The relative standard deviation for the mean response factor from each of the unlabeled analytes (Table 2) and of the internal, surrogate, and alternate standards shall be less than or equal to the values in Table 5. The signal to noise ratio for the GC signal present in every selected ion current profile shall be greater than or equal to 2.5. The ion abundance ratios shall be within the control limits in Table 4.

6.1.2 Daily Performance Check.

6.1.2.1 Calibration Check. Inject on μl of solution Number 3 from Table 2. Calculate the relative response factor (RRF) for each compound and compare each RRF to the corresponding mean RRF obtained during the initial calibration. The analyzer performance is acceptable if the measured RRF's for the labeled and unlabeled compounds for the daily run are within the limits of the mean values shown in Table 5. In addition, the ion-abundance ratios shall be within the allowable control limits shown in Table 4.

6.1.2.2 Column Separation Check. Inject a solution of a mixture of PCDD's and PCDF's that documents resolution between 2.3.7.8–TCDD and other TCDD isomers. Resolution is defined as a valley between peaks that is less than 25 percent of the lower of the two peaks. Identify and record the retention time windows for each homologous series.

Perform a similar resolution check on the confirmation column to document the resolution between 2.3.7.8 TCDF and other TCDF isomers.

6.2 Lock Channels. Set mass spectrometer lock channels as specified in Table 3. Monitor the quality control check channels specified in Table 3 to verify instrument stability during the analysis.

7. Quality Control

7.1 Sampling Train Collection Efficiency Check. Add 100 μl of the surrogate standards in Table 1 to the absorbent cartridge of each train before collecting the field samples.

7.2 Internal Standard Percent Recoveries. A group of nine carbon labeled PCDD's and PCDF's representing, the tetra-through octachlorinated homologues, is added to every sample prior to extraction. The role of the internal standards is to quantify the native PCDD's and PCDF's present in the sample as well as to determine the overall method efficiency. Recoveries of the internal standards must be between 40 to 130 percent for the tetra-through hexachlorinated compounds while the range is 25 to 130 percent for the higher hepta- and octachlorinated homologues.

7.3 Surrogate Recoveries. The five surrogate compounds in Table 2 are added to the resin in the adsorbent sampling cartridge before the sample is collected. The surrogate recoveries are measured relative to the internal standards and are a measure of collection efficiency. They are not used to measure native PCDD's and PCDF's. All recoveries shall be between 70 and 130 percent. Poor recoveries for all the surrogates may be an indication of breakthrough in the sampling train. If the recovery of all standards is below 70 percent, the sampling runs must be repeated. As an alternative, the sampling runs do not have to be repeated if the final results are divided by the fraction of surrogate recovery. Poor recoveries of isolated surrogate compounds should not be grounds for rejecting an entire set of the samples.

7.4 Toluene QA Rinse. Report the results of the toluene QA rinse separately from the total sample catch. Do not add it to the total sample.

8. Quality Assurance

8.1 Applicability. When the method is used to analyze samples to demonstrate compliance with a source emission regulation, an audit sample must be analyzed, subject to availability.

8.2 Audit Procedure. Analyze an audit sample with each set of compliance samples. The audit sample contains tetra through octa isomers of PCDD and PCDF. Concurrently, analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for the compliance samples and the EPA audit sample.

8.3 Audit Sample Availability. Audit samples will be supplied only to enforcement agencies for compliance tests. The availability of audit samples may be obtained by writing: Source Test Audit Coordinator (MD–77B), Quality Assurance Division, Atmospheric Research and Exposure Assessment Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, or by calling the Source Test Audit Coordinator (STAC) at (919) 541–7834. The request for the audit sample must be made at least 30 days prior to the scheduled compliance sample analysis.

8.4 Audit Results. Calculate the audit sample concentration according to the calculation procedure described in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

9. Calculations

Same as Method 5, section 6 with the following additions.

9.1 Nomenclature.

A_n = Integrated ion current of the noise at the retention time of the analyte.

A^*_{ci} = Integrated ion current of the two ions characteristic of the internal standard i in the calibration standard.

A_{cij} = Integrated ion current of the two ions characteristic of compound i in the jth calibration standard.

A^*_{cij} = Integrated ion current of the two ions characteristic of the internal standard i in the jth calibration standard.

A_{cs} = Integrated ion current of the two ions characteristic of surrogate compound i in the calibration standard.

A_i = Integrated ion current of the two ions characteristic of compound i in the sample.

A^*_i = Integrated ion current of the two ions characteristic of internal standard i in the sample.

A_{rs} = Integrated ion current of the two ions characteristic of the recovery standard.

A_s = Integrated ion current of the two ions characteristic of surrogate compound i in the sample.

C_i = Concentration of PCDD or PCDF i in the sample, pg/M^3.

C_T = Total concentration of PCDD's or PCDF's in the sample, pg/M^3.

m_{ci} = Mass of compound i in the calibration standard injected into the analyzer, pg.

m_{rs} = Mass of recovery standard in the calibration standard injected into the analyzer, pg.

m_s = Mass of surrogate compound i in the calibration standard, pg.

RRF_i = Relative response factor.

RRF_{rs} = Recovery standard response factor.

RRF_s = Surrogate compound response factor.

9.2 Average Relative Response Factor.

$$RRF_i = \frac{1}{n} \sum_{j=1}^{n} \frac{A_{cij}\, m^*_{ci}}{A^*_{cij}\, m_{ci}} \qquad \text{Eq. 23–1}$$

9.3 Concentration of the PCDD's and PCDF's.

$$C_i = \frac{m_i^*\, A_i}{A_i^*\, RRF_i\, V_{mstd}} \qquad \text{Eq. 23–2}$$

9.4 Recovery Standard Response Factor.

$$RRF_{rs} = \frac{A_{ci}^*\, m_{rs}}{A_{rs}\, m_{ci}^*} \qquad \text{Eq. 23–3}$$

9.5 Recovery of Internal Standards (R*).

$$R^* = \frac{A_i^*\, m_{rs}}{A_{rs}\, RF_{rs}\, m_i^*} \times 100\% \qquad \text{Eq. 23–4}$$

9.6 Surrogate Compound Response Factor.

$$RRF_s = \frac{A_{ci}^{*}\, m_s}{A_s\, m_{ci}^{*}} \qquad \text{Eq. 23-5}$$

9.7 Recovery of Surrogate Compounds (R_s).

$$R_s = \frac{A_s\, m_i^{*}}{A_i^{*}\, RRF_s\, m_s} \times 100\% \qquad \text{Eq. 23-6}$$

9.8 Minimum Detectable Limit (MDL).

$$MDL = \frac{2.5\, A_{si}\, m_i^{*}}{A_{ci}^{*}\, RRF_i} \qquad \text{Eq. 23-7}$$

9.9 Total Concentration of PCDD's and PCDF's in the Sample.

$$C_T = \sum_{i=1}^{n} C_i \qquad \text{Eq. 23-8}$$

Any PCDD's or PCDF's that are reported as nondetected (below the MDL) shall be counted as zero for the purpose of calculating the total concentration of PCDD's and PCDF's in the sample.

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TABLE 1.—COMPOSITION OF THE SAMPLE FORTIFICATION AND RECOVERY STANDARDS SOLUTIONS

Analyte	Concentration (pg/μl)
Internal Standards:	
$^{13}C_{12}$-2,3,7,8-TCDD	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100
$^{13}C_{12}$-OCDD	100
$^{13}C_{12}$-2,3,7,8-TCDF	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100
Surrogate Standards:	
$^{37}Cl_4$-2,3,7,8-TCDD	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100
Recovery Standards:	
$^{13}C_{12}$-1,2,3,4-TCDD	500
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	500

TABLE 2.—COMPOSITION OF THE INITIAL CALIBRATION SOLUTIONS

Compound	Concentrations (pg/μL)				
	Solution No.				
	1	2	3	4	5
Alternate Standard:					
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	2.5	5	25	250	500
Recovery Standards:					
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 3.—ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDD'S AND PCDF'S

Descriptor No.	Accurate mass	Ion type	Elemental composition	Analyte
2	292.9825	LOCK	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD (S)
	330.9792	QC	C_7F_{13}	PFK
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PECDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF (S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4 37ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCPDE
3	373.8208	M+2	$C_{12}H_2 35Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	LOCK	C_9F_{15}	PFK

TABLE 3.—ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDD'S AND PCDF's—Continued

Descriptor No.	Accurate mass	Ion type	Elemental composition	Analyte
	401.8559	M+2	$^{13}C_{12}H_{2}{}^{35}Cl_{5}{}^{37}ClO_{2}$	HxCDD (S)
	403.8529	M+4	$^{13}C_{12}H_{2}{}^{35}Cl_{4}{}^{37}Cl_{2}O$	HxCDD (S)
	445.7555	M+4	$C_{12}H_{2}{}^{35}Cl_{6}{}^{37}Cl_{2}O$	OCDPE
	430.9729	QC	$C_{9}F_{17}$	PFK
4	407.7818	M+2	$C_{12}H^{35}Cl_{6}{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_{5}{}^{37}Cl_{2}O$	HpCDF
	417.8253	M	$^{13}C_{12}H^{35}Cl_{7}O$	HpCDF (S)
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_{6}{}^{37}ClO$	HpCDF (S)
	423.7766	M+2	$C_{12}H^{35}Cl_{6}{}^{37}ClO_{2}$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_{5}{}^{37}Cl_{2}O_{2}$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_{6}{}^{37}ClO_{2}$	HpCDD (S)
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_{5}{}^{37}Cl_{2}O_{2}$	HpCDD (S)
	479.7165	M+4	$C_{12}H^{35}Cl_{7}{}^{37}Cl_{2}O$	NCPDE
	430.9729	LOCK	$C_{9}F_{17}$	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_{7}{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_{6}{}^{37}Cl_{2}O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_{7}{}^{37}ClO_{2}$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_{6}{}^{37}Cl_{2}O_{2}$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_{7}{}^{37}ClO_{2}$	OCDD (S)
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_{6}{}^{37}Cl_{2}O_{2}$	OCDD (S)
	513.6775	M+4	$C_{12}{}^{35}Cl_{8}{}^{37}Cl_{2}O_{2}$	DCDPE
	442.9728	QC	$C_{10}F_{17}$	PFK

(a) The following nuclidic masses were used:
H = 1.007825
C = 12.000000
^{13}C = 13.003355
F = 18.9984
O = 15.994915
^{35}Cl = 34.968853
^{37}Cl = 36.965903
S = Labeled Standard
QC = Ion selected for monitoring instrument stability during the GC/MS analysis.

TABLE 4.—ACCEPTABLE RANGES FOR ION-ABUNDANCE RATIOS OF PCDD'S AND PCDF's

No. of chlorine atoms	Ion type	Theoretical ratio	Control limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6[a]	M/M+2	0.51	0.43	0.59
7[b]	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF.
[b] Used only for ^{13}C-HpCDF.

TABLE 5.—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS

Compound	Relative response factors	
	Initial calibration RSD	Daily calibration % difference
Unlabeled Analytes:		
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDF	25	25
1,2,4,5,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25
OCDF	30	30
Internal Standards:		
$^{13}C_{12}$-2,3,7,8-TCDD	25	25
$^{13}C_{12}$-1,2,3,7,8-PeCDD	30	30
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	25	25
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	30	30
$^{13}C_{12}$-OCDD	30	30
$^{13}C_{12}$-2,3,7,8-TCDF	30	30
$^{13}C_{12}$-1,2,3,7,8-PeCDF	30	30
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	30	30
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	30	30
Surrogate Standards:		
$^{37}Cl_{4}$-2,3,7,8-TCDD	25	25
$^{13}C_{12}$-2,3,4,7,8-PeCDF	25	25
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	25	25
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	25	25
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	25	25

LE 5.—MINIMUM REQUIREMENTS FOR ITIAL AND DAILY CALIBRATION RESPONSE FACTORS—Continued

ompound	Relative response factors	
	Initial calibration RSD	Daily calibration % difference
rnate Standard: C_{12}-1,2,3,7,8,9-HxCDF	25	25

* * * * *

thod 26—Determination of Hydrogen oride Emissions From Stationary Sources

pplicability, Principle, Interferences, cision, Bias, and Stability

.1 Applicability. This method is licable for determining hydrogen chloride l) emissions from stationary sources.

.2 Principle. An integrated sample is racted from the stack and passed through ute sulfuric acid. In the dilute acid, the l gas is dissolved and forms chloride) ions. The Cl^- is analyzed by ion omatography (IC).

1.3 Interferences. Volatile materials which produce chloride ions upon dissolution during sampling are obvious interferences. Another likely interferent is diatomic chlorine (Cl_2) gas which reacts to form HCl and hypochlorous acid (HOCl) upon dissolving in water. However, Cl_2 gas exhibits a low solubility in water and the use of acidic, rather than neutral or basic collection solutions, greatly reduces the chance of dissolving any chlorine present. This method does not experience a significant bias when sampling a 400 ppm HCl gas stream containing 30 ppm Cl_2. Sampling a 220 ppm HCl gas stream containing 180 ppm Cl_2 results in a positive bias of 3.4 percent in the HCl measurement.

1.4 Precision and Bias. The within-laboratory relative standard deviations are 6.2 and 3.2 percent at HCl concentrations of 3.9 and 15.3 ppm, respectively. The method does not exhibit a bias to Cl_2 when sampling at concentrations less than 50 ppm.

1.5 Stability. The collected samples can be stored for up to 4 weeks before analysis.

1.6 Detection Limit. The analytical detection limit of the method is 0.1 μg/ml.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 26–1, and component parts are discussed below.

2.1.1 Probe. Borosilicate glass, approximately ⅜-in. (9-mm) I.D. with a heating system to prevent moisture condensation. A ⅜-in I.D. Teflon elbow should be attached to the inlet of the probe and a 1-in. (25-mm) length of ⅜-in. I.D. Teflon tubing should be attached to the open end of the elbow to permit the opening of the probe to be turned away from the gas stream. This reduces the amount of particulate entering the train. This probe configuration should be used when the concentration of particulate matter in the emissions is high. When high concentrations are not present, the Teflon elbow is not necessary, and the probe inlet may be perpendicular to the gas stream. A glass wool plug should *not* be used to remove particulate matter since a negative bias in the data could result. Instead, a Teflon filter (see Section 2.1.5) should be installed at the inlet (for stack temperatures <300 °F) or outlet (for stack temperatures >300 °F) of the probe.

2.1.2 Three-way Stopcock. A borosilicate, three-way glass stopcock with a heating system to prevent moisture condensation. The heated stopcock should connect directly to the outlet of the probe and the inlet of the first impinger. The heating system should be capable of preventing condensation up to the inlet of the first impinger. Silicone grease may be used, if necessary, to prevent leakage.

BILLING CODE 6560-50-M

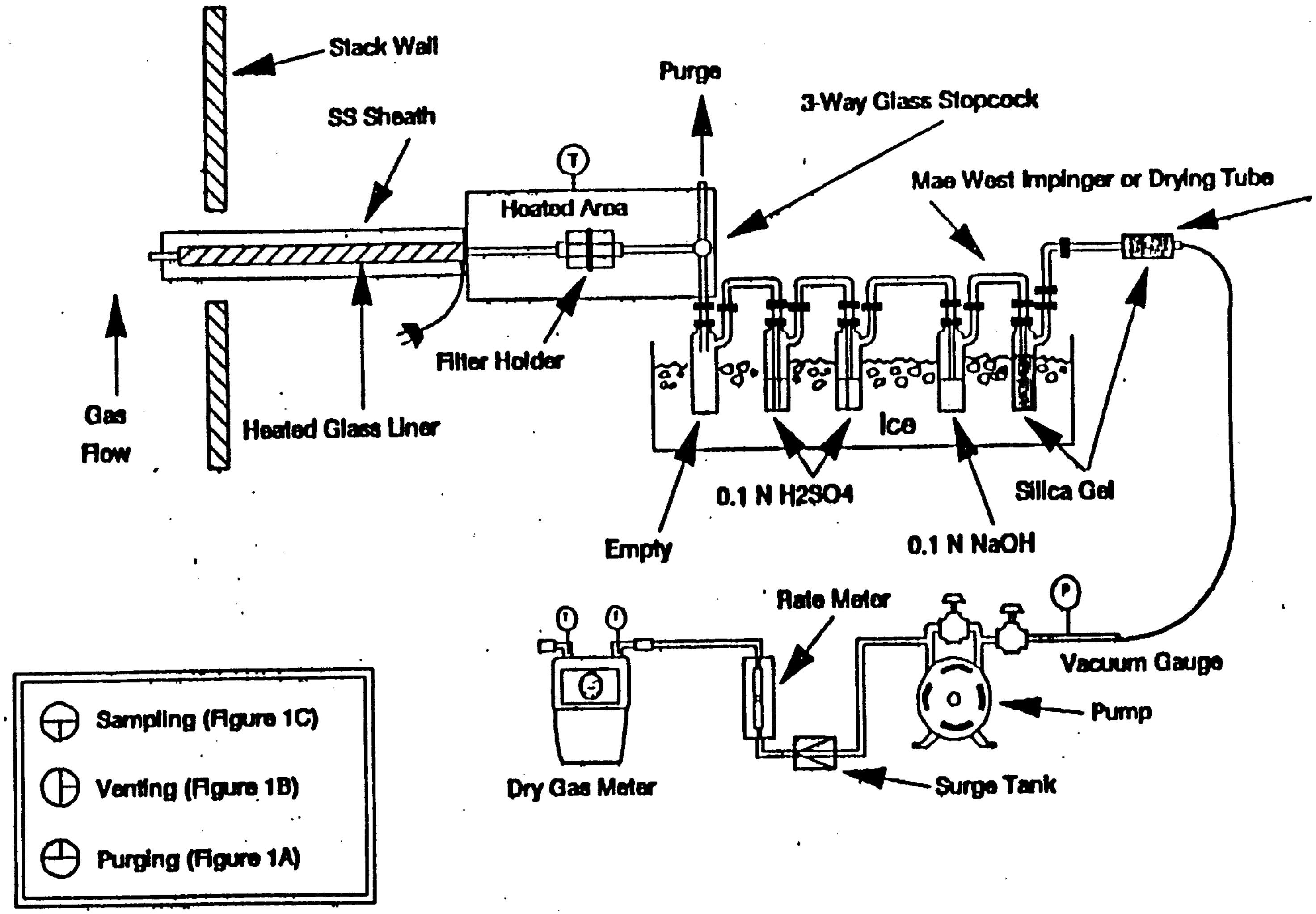

Figure 26-1. Sampling train.

BILLING CODE 6560-50-C

2.1.3 Impingers. Four 30-ml midget impingers with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage. For sampling at high moisture sources or for sampling times greater than 1 hour, a midget impinger with a shortened stem (such that the gas sample does not bubble through the collected condensate) should be used in front of the first impinger.

2.1.4 Drying Tube or Impinger. Tube or impinger, of Mae West design, filled with 6- to 16-mesh indicating type silica gel, or equivalent, to dry the gas sample and to protect the dry gas meter and pump. If the silica gel has been used previously, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

2.1.5 Filter. A 25-mm Teflon mat, Pallflex TX40H175 or equivalent. Locate between the probe liner and Teflon elbow in a glass or quartz filter holder in a filter box heated to 250 °F.

2.1.6 Sample Line. Leak-free, with compatible fittings to connect the last impinger to the needle valve.

2.1.7 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of 2 liters/min.

2.1.8 Purge Pump, Purge Line, Drying Tube, Needle Valve, and Rate Meter. Pump capable of purging the sampling probe at 2 liters/min, with drying tube, filled with silica gel or equivalent, to protect pump, and a rate meter capable of measuring 0 to 5 liters/min.

2.1.9 Stopcock Grease, Valve, Pump, Volume Meter, Barometer, and Vacuum Gauge. Same as in Method 6, Sections 2.1.4, 2.1.7, 2.1.8, 2.1.10, 2.1.11, and 2.1.12.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500-ml or larger, two.

2.2.2 Storage Bottles. 100-ml glass, with Teflon-lined lids, to store impinger samples (two per sampling run). During clean-up, the two front impinger contents (0.1 N H_2SO_4) should be combined. The contents of the two rear impinger (0.1 N NaOH) may be discarded, as these solutions are included only to absorb Cl_2, and thus protect the pump.

2.3 Sample Preparation and Analysis. The materials required for volumetric dilution and chromatographic analysis of samples are described below.

2.3.1 Volumetric Flasks. Class A, 100-ml size.

2.3.2 Volumetric Pipets. Class A, assortment. To dilute samples into the calibration range of the instrument.

2.3.3 Ion Chromatograph. Suppressed or nonsuppressed, with a conductivity detector and electronic integrator operating in the peak area mode. Other detectors, strip chart recorders, and peak height measurements may be used provided the 5 percent repeatability criteria for sample analysis and the linearity criteria for the calibration curve can be met.

3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS reagent grade). When such specifications are not available, the best available grade shall be used.

3.1. Sampling.

3.1.1 Water. Deionized, distilled water that conforms to ASTM Specification D 1193-77, Type 3.

3.1.2 Absorbing solution, 0.1 N Sulfuric Acid (H_2SO_4). To prepare 100 ml of the absorbing solution for the front impinger pair, slowly add 0.28 ml of concentrated H_2SO_4 to about 90 ml of water while stirring, and adjust the final volume to 100 ml using additional water. Shake well to mix the solution.

3.1.3. Chlorine Scrubber Solution, 0.1 N Sodium Hydroxide (NaOH). To prepare 100 ml of the scrubber solution for the back pair of impingers, dissolve 0.40 g of solid NaOH in about 90 ml of water, and adjust the final solution volume to 100 ml using additional water. Shake well to mix the solution.

3.2 Sample Preparation and Analysis.

3.2.1 Water. Same as in Section 3.1.1.

3.2.2 Blank Solution. A separate blank solution of the absorbing reagent should be prepared for analysis with the field samples. Dilute 30 ml of absorbing solution to 100 ml with water in a separate volumetric flask.

3.2.3 Sodium Chloride (NaCl) Stock Standard Solution. Solutions containing a nominal certified concentration of 1000 mg/l are commercially available as convenient stock solutions from which working standards can be made by appropriate volumetric dilution. Alternately, concentrated stock solutions may be produced from reagent grade NaCl. The NaCl should be dried at 100 °C for 2 or more hours and cooled to room temperature in a desiccator immediately before weighing. Accurately weigh 1.6 to 1.7 g of the dried NaCl to within 0.1 mg, dissolve in water, and dilute to 1 liter. The exact Cl concentration can be calculated using Eq. 26-1.

$$\mu g\ Cl^-/ml = g\ of\ NaCl \times 10^3 \times 35.453/58.44$$

Eq. 26-1

Refrigerate the stock standard solution and store no longer than 1 month.

3.2.4 Chromatographic Eluent. Effective eluents for nonsuppressed IC using a resin- or silica-based weak ion exchange column are a 4 mM potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mM 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH. An effective eluent for suppressed ion chromatography is a solution containing 3 mM sodium bicarbonate and 2.4 mM sodium carbonate. Other dilute solutions buffered to a similar pH and containing no interfering ions may be used. When using suppressed ion chromatography, if the "water dip" resulting from sample injection interferes with the chloride peak, use a 2 mM NaOH/2.4 mM sodium bicarbonate eluent.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the absorbing solution into each of the first two impingers, and add 15 ml of scrubber solution to the third and fourth impingers. Connect the impingers in series with the knockout impinger first, followed by the two impingers containing absorbing solution and the two containing the scrubber solution. Place a fresh charge of silica gel, or equivalent, in the drying tube or Mae West impinger.

4.1.2 Leak-Check Procedures. Leak-check the probe and three-way stopcock before inserting the probe into the stack. Connect the stopcock to the outlet of the probe, and connect the sample line to the needle valve. Plug the probe inlet, turn on the sample pump, and pull a vacuum of at least 250 mm Hg (10 in. Hg). Turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Place the probe in the stack at the sampling location, and adjust the probe and stopcock:

$$\frac{\mu g\ Cl^-}{ml} = \frac{g\ of\ NaCl \times 10^3 \times 35.453}{58.44}$$

Eq. 26-1

Refrigerate the stock standard solution and store no longer than 1 month.

3.2.4 Chromatographic Eluent. Effective eluents for nonsuppressed IC using a resin- or silica-based weak ion exchange column are a 4 mM potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mM 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH. An effective eluent for suppressed ion chromatography is a solution containing 3 mM sodium bicarbonate and 2.4 mM sodium

carbonate. Other dilute solutions buffered to a similar pH and containing no interfering ions may be used. When using suppressed ion chromatography, if the "water dip" resulting from sample injection interferes with the chloride peak, use a 2 mM NaOH/2.4 mM sodium bicarbonate eluent.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the aborbing solution into each of the first two impingers, and add 15 ml of scrubber solution to the third and fourth impingers. Connect the impingers in series with the knockout impinger first, followed by the two impingers containing absorbing solution and the two containing the scrubber solution. Place a fresh charge of silica gel, or equivalent, in the drying tube or Mae West impinger.

4.1.2 Leak-Check Procedures. Leak-check the probe and three-way stopcock before inserting the probe into the stack. Connect the stopcock to the outlet of the probe, and connect the sample line to the needle valve. Plug the probe inlet, turn on the sample pump, and pull a vacuum of at least 250 mm Hg (10 in. Hg). Turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Place the probe in the stack at the sampling location, and adjust the probe and stopcock heating system to a temperature sufficient to prevent water condensation. Connect the first impinger to the stopcock, and connect the sample line to the last impinger and the needle valve. Upon completion of a sampling run, remove the probe from the stack and leak-check as described above. If a leak has occurred, the sampling run must be voided. Alternately, the portion of the train behind the probe may be leak-checked between multiple runs at the same site as follows: Close the stopcock to the first impinger (see Figure 1A of Figure 26–1), and turn on the sampling pump. Pull a vacuum of at least 250 mm Hg, turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Release the vacuum on the impinger train by turning the stopcock to the vent position to permit ambient air to enter (see Figure 1B of Figure 26–2). If this procedure is used, the full train leak-check described above must be conducted following the final run, and all preceding sampling runs must be voided if a leak has occurred.

4.1.3 Purge Procedure. Immediately before sampling, connect the purge line to the stopcock, and turn the stopcock to permit the purge pump to purge the probe (see Figure 1A of Figure 26–1). Turn on the purge pump, and adjust the purge rate to 2 liters/min. Purge for at least 5 minutes before sampling.

4.1.4 Sample Collection. Turn on the sampling pump, pull a slight vacuum of approximately 25 mm Hg (1 in. Hg) on the impinger train, and turn the stopcock to permit stack gas to be pulled through the impinger train (see Figure 1C of Figure 26–3). Adjust the sampling rate to 2 liters/min, as indicated by the rate meter, and maintain this rate to within 10 percent during the entire sampling run. Take readings of the dry gas meter volume and temperature, rate meter, and vacuum gauge at least once every 5 minutes during the run. A sampling time of 1 hour is recommended. Shorter sampling times may introduce a significant negative bias in the HCl concentration. At the conclusion of the sampling run, remove the train from the stack, cool, and perform a leak-check as described in section 4.1.2.

4.2 Sample Recovery. Disconnect the impingers after sampling. Quantitatively transfer the contents of the first three impingers (the knockout impinger and the two absorbing solution impingers) to a leak-free storage bottle. Add the water rinses of each of these impingers and connecting glassware to the storage bottle. The contents of the scrubber impingers and connecting glassware rinses may be discarded. The sample bottle should be sealed, shaken to mix, and labeled. The fluid level should be marked so that if any sample is lost during transport, a correction proportional to the lost volume can be applied.

4.3 Sample Preparation for Analysis. Check the liquid level in each sample, and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, the volume lost can be determined from the difference between the initial and final solution levels, and this value can be used to correct the analytical results. Quantitatively transfer the sample solution to a 100-ml volumetric flask, and dilute the solution to 100 ml with water.

4.4 Sample Analysis.

4.4.1 The IC conditions will depend upon analytical column type and whether suppressed or nonsuppressed IC is used. An example chromatogram from a nonsuppressed system using a 150-mm Hamilton PRP-X100 anion column, a * * * .2 l/min flow rate of a 4 mM 4-hydroxy benzoate solution adjusted to a pH of 8.6 using 1 N NaOH, a 50 μl sample loop, and a conductivity detector set on 1.0 μS full scale is shown in Figure 26–2.

4.4.2 Before sample analysis, establish a stable baseline. Next, inject a sample of water, and determine if any Cl^- appears in the chromatogram. If Cl^- is present, repeat the load/injection procedure until no Cl^- is present. At this point, the instrument is ready for use.

4.4.3 First, inject the calibration standards covering an appropriate concentration range, starting with the lowest concentration standard. Next, inject in duplicate, a QC sample followed by a water blank and the field samples. Finally, repeat the injection of calibration standards to allow compensation for any drift in the instrument during analysis of the field samples. Measure the Cl^- peak areas or heights of the samples. Use the average response from the duplicate injections to determine the field sample concentrations using a linear calibration curve generated from the standards.

4.5 Audit Analysis. An audit sample must be analyzed, subject to availability.

Figure 26-2. Example Chromatogram

5. Calibration

5.1 Dry Gas Metering System, Thermometers, Rate Meter, and Barometer. Same as in Method 6, sections 5.1, 5.2, 5.3, and 5.4.

5.2 Calibration Curve for Ion Chromatograph. To prepare calibration standards, dilute given volumes (1.0 ml or greater) of the stock standard solution, with 0.1 N H_2SO_4 (section 3.1.2) to convenient volumes. Prepare at least four standards that are within the linear range of the instrument and which cover the expected concentration range of the field samples. Analyze the standards as instructed in section 4.4.3, beginning with the lowest concentration standard. Determine the peak measurements, and plot individual values versus Cl^- concentration in μg/ml. Draw a smooth curve through the points. Use linear regression to lculate a formula describing the resulting near curve.

Quality Assurance

6.1 Applicability. When the method is ed to analyze samples to demonstrate ompliance with a source emission gulation, a set of two audit samples must be nalyzed.

6.2 Audit Procedure. The audit sample are loride solutions. Concurrently analyze the vo audit samples and a set of compliance amples in the same manner to evaluate the chnique of the analyst and the standards reparation. The same analyst, analytical agents, and analytical system shall be used oth for compliance samples and the EPA udit samples. If this condition is met, uditing the subsequent compliance analyses r the same enforcement agency within 30 ays is not required. An audit sample set may ot be used to validate different sets of ompliance samples under the jurisdiction of ifferent enforcement agencies, unless prior rrangements are made with both nforcement agencies.

6.3 Audit Sample Availability. The audit amples may be obtained by writing or alling the EPA Regional Office or the ppropriate enforcement agency. The request r the audit samples must be made at least days prior to the scheduled compliance ample analyses.

6.4 Audit Results. Calculate the oncentrations in mg/dscm using the ecified sample volume in the audit structions.

Note: Indication of acceptable results may e obtained immediately by reporting the udit results in mg/dscm and compliance sults in total µg HCl/ sample to the sponsible enforcement agency. Include the sults of both audit samples, their lentification numbers, and the analyst's ame with the results of the compliance etermination samples in appropriate reports the EPA Regional Office or the appropriate nforcement agency. Include this information ith subsequent analyses for the same nforcement agency during the 30-day period.

The concentrations of the audit samples btained by the analyst shall agree within 10 ercent of the actual concentrations. If the 10 ercent specification is not met, reanalyze he compliance samples and audit samples, nd include initial and reanalysis values in he test report.

Failure to meet the 10 percent specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance status of the affected facility.

7. Calculations

Retain at least one extra decimal figure beyond those contained in the available data in intermediate calculations, and round off only the final answer appropriately.

7.1 Sample Volume, Dry Basis, Corrected to Standard Conditions. Calculate the sample volume using Eq. 6–1 of Method 6.

7.2 Total µg HCl Per Sample.

$$m = \frac{(S-B)(100)(36.46)}{(35.453)} = (102.84)(S-B) \qquad \text{Eq. 26–2}$$

where:

m = Mass of Hcl in sample, µg.
S = Concentration of sample, µg Cl^-/ml.
B = Concentration of blank, µg Cl^-/ml.
100 = Volume of filtered and diluted sample, ml.
36.46 = Molecular weight of HCl, µg/µg-mole.
35.453 = Atomic weight of Cl, µg/µg-mole.

7.3 Concentration of HCl in the Flue Gas.

$$C = \frac{K_m}{V_{m(std)}} \qquad \text{Eq. 26–3}$$

where:

C = Concentration of HCl, dry basis, mg/dscm.
K = 10^{-3} mg/µg.
m = Mass of HCl in sample, µg.
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm.

8. Bibliography

1. Steinsberger, S.C. and J.H. Margeson. "Laboratory and Field Evaluation of a Methodology for Determination of Hydrogen Chloride Emissions from Municipal and Hazardous Waste Incinerators." U.S. Environmental Protection Agency, Office of Research and Development. Report No. ______ 1989.

2. State of California, Air Resources Board. Method 421. "Determination of Hydrochloric Acid Emissions from Stationary Sources." March 18, 1987.

3. Entropy Environmentalists Inc. "Laboratory Evaluation of a Sampling and Analysis Method for Hydrogen Chloride Emissions from Stationary Sources: Interim Report." EPA Contract No. 68–02–4442. Research Triangle Park, North Carolina. January 22, 1988.

* * * * *

Appendix A [Amended]

3. Method 19 of appendix A part 60 is amended by adding paragraphs 4.3 and 5.4 as follows: Method 19—Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide, and Nitrogen Oxides Emission Rates

* * * * *

4.3 Daily Geometric Average Pollutant Rates from Hourly Values. The geometric average pollutant rate (E_{go}) is computed using the following equation:

$$E_{go} = \mathrm{EXP}\left[(1/n) \sum_{j=1}^{n} [\ln(E_{hj})]\right] \qquad \text{Eq. 19–20a}$$

where:

E_{go} = daily geometric average pollutant rate, ng/J (lbs/million Btu) or ppm corrected to 7 percent O_2.
E_{hj} = hourly arithmetic average pollutant rate for hour "j," ng/J (lb/million Btu) or ppm corrected to 7 percent O_2.
n = total number of hourly averages for which pollutant rates are available within the 24 hr midnight to midnight daily period.
ln = natural log of indicated value.
EXP = the natural logarithmic base (2.718) raised to the value enclosed by brackets.

* * * * *

5.4 Daily Geometric Average Percent Reduction from Hourly Values. The geometric average percent reduction (%R_{go}) is computed using the following equation:

$$\%R_{ga} = 100 \left[1 - \mathrm{EXP}\left[(1/n) \sum_{j=1}^{n} \ln (E_{jo}/E_{ji})\right]\right]$$

Eq. 19–24a

where:

$\%R_{ga}$ = daily geometric average percent reduction.

E_{jo}, E_{ji} = matched pair hourly arithmetic average pollutant rate, outlet and inlet, respectively, ng/J (lb/million Btu) or ppm corrected to 7 percent O_2.

n = total number of hourly averages for which paired inlet and outlet pollutant rates are available within the 24-hr midnight to midnight daily period.

ln = natural log of indicated value.

EXP = the natural logarithmic base (2.718) raised to the value enclosed by brackets.

Note: The calculation includes only paired data sets (hourly average) for the inlet and outlet pollutant measurements.

* * * * *

[FR Doc. 91–1399 Filed 2–12–91; 8:45 am]

BILLING CODE 6560–50–M

DEPARTMENT OF COMMERCE

National Oceanic and Atmospheric Administration

50 CFR Parts 672 and 675

[Docket No. 90899–1019]

Groundfish of the Gulf of Alaska; Groundfish of the Bering Sea and Aleutian Islands

AGENCY: National Marine Fisheries Service (NMFS), NOAA, Commerce.

ACTION: Final rule, technical amendment.

SUMMARY: The Secretary of Commerce issues this final rule implementing a technical amendment to reinstate regulatory language that was unintentionally deleted by regulations implementing Amendment 13 to the Fishery Management Plan for the Bering Sea/Aleutian Islands Groundfish (Bering FMP) and Amendment 18 to the Fishery Management Plan for the Gulf of Alaska Groundfish Fishery (Gulf FMP) (54 FR 50386, December 6, 1989).

EFFECTIVE DATE: February 8, 1991.

FOR FURTHER INFORMATION CONTACT: Patricia Peacock (Fishery Management Specialist), NMFS, Plans and Regulations Division, 1335 East-West Highway, Silver Spring, Maryland 20910, telephone 301–427–2343.

SUPPLEMENTARY INFORMATION: Groundfish fisheries in the Exclusive Economic Zone off Alaska are governed by Federal regulations at 50 CFR parts 611, 620, 672, and 675 that implement the Bering and the Gulf FMPs. These FMPs were prepared by the North Pacific Fishery Management Council and approved by the Secretary of Commerce (Secretary) under provisions of the Magnuson Fishery Conservation and Management Act.

This final rule implements a technical amendment that (1) reinstates language unintentionally deleted by amendatory language in 54 FR 50383 (December 6, 1989) and (2) retains the amendatory language of 56 FR 492 (January 7, 1991).

On December 6, 1989, a final rule was published in the Federal Register (54 FR 50386) that was intended only to amend the introductory language of §§ 672.20(a)(2) and 675.20(a)(2). Item 11 of this amendatory language states that * * * paragraphs (a)(2) and (f)(1) are revised * * *; item 20 states that * * * paragraph (a)(2) is revised * * *. This amendatory language revised the introductory text of paragraph (a)(2) in these sections but deleted the remainder of the regulatory language in §§ 672.20(a)(2) and 675.20(a)(2). Paragraphs in § 672.20 that were unintentionally deleted were (a)(2)(i), (a)(2)(ii), (a)(2)(ii)(A), and (a)(2)(ii)(B). Table 1 in § 672.20 also was unintentionally deleted. Paragraphs that were unintentionally deleted in § 675.20 were as follows: (a)(2)(i), (a)(2)(i)(A), and (a)(2)(i)(B).

On January 1, 1991, final rules implementing Amendment 14 to the Bering FMP and Amendment 19 to the Gulf FMP also revised text in §§ 672.20(a)(2) and 675.20(a)(2) (56 FR 492, January 7, 1991). This rule retains the changes to these sections resulting from Amendments 14 and 19.

The Gulf and Bering FMPs were implemented using procedures specified by the Magnuson Act and the Administrative Procedure Act. However, as explained above, the language currently in §§ 672.20(a) and 675.20(a) does not correctly reflect the Gulf or Bering FMPs and subsequent amendments.

This final rule, technical amendment, is reinstating regulatory language needed to implement correctly the Bering and Gulf FMPs as amended.

Classification

This final rule, technical amendment, is issued under parts 672 and 675. Because this rule reinstates regulatory text that was inadvertently deleted during an earlier rulemaking proceeding and makes no substantive changes other than changes determined in rulemaking for Amendments 14 and 19, it is unnecessary under 5 U.S.C. 553(b)(B) to provide for prior public comment and there is good cause under 5 U.S.C. 553(d) not to delay for 30 days its effective date.

Because this rule is being issued without prior comment, a regulatory flexibility analysis is not required under the Regulatory Flexibility Act and none has been prepared.

This rule reinstates language that has been determined not to be a major rule under Executive Order 12291, does not contain policies with federalism implications sufficient to warrant preparation of a federalism assessment under E.O. 12612, and does not contain a collection-of-information requirement for the purposes of the Paperwork Reduction Act. There is no change in the regulatory impacts previously reviewed and analyzed.

List of Subjects in 50 CFR Parts 672 and 675

Fisheries, General limitations.

Dated: February 7, 1991.

Michael F. Tillman,

Acting Assistant Administrator for Fisheries, National Marine Fisheries Service.

For reasons set out in the preamble, 50 CFR parts 672 and 675 are amended as follows:

PART 672—GROUNDFISH OF THE GULF OF ALASKA

1\. The authority citation for part 672 continues to read as follows:

Authority: 16 U.S.C. 1801 *et seq.*

2\. Section 672.20(a)(2) is revised and Table 1 is added to the section to read as follows:

INTERPOLL LABORATORIES
4500 BALL ROAD N.E.
CIRCLE PINES, MN 55014-1819
(612) 786-6020

11/7/88

APPENDIX A

METHODOLOGY FOR THE DETERMINATION OF METALS EMISSIONS IN EXHAUST GASES FROM STATIONARY SOURCE COMBUSTION PROCESSES

1. Applicability and Principle

1.1 Applicability. This method is applicable for the determination of total chromium (Cr), cadmium (Cd), arsenic (As), nickel (Ni), manganese (Mn), beryllium (Be), copper (Cu), zinc (Zn), lead (Pb), selenium (Se), phosphorus (P), thallium (Tl), silver (Ag), antimony (Sb), barium (Ba), and mercury (Hg) emissions from municipal waste incinerators, sewage sludge incinerators, and hazardous waste incinerators. This method may also be used for the determination of particulate emissions following the additional procedures described. Modifications to the sample recovery and analysis procedures described in this protocol for the purpose of determining particulate emissions may potentially impact the front half mercury determination.*

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

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*Field tests to date have shown that of the total amount of mercury measured by the method, only 0 to <2% was measured in the front half. Therefore, it is tentatively concluded, based on the above data, that particulate emissions may be measured by this train, without significantly altering the mercury results.

This document is a preliminary draft. It has not been formally released by EPA and should not at this stage be construed to represent Agency policy. It is being circulated for comment on its technical accuracy and policy implications

remainder of the sampling train catches are analyzed for Cr, Cd, Ni, Mn, Be, Cu, Zn, Pb, Se, P, Tl, Ag, Sb, Ba, and As by inductively coupled argon plasma emission spectroscopy (ICAP) or atomic absorption spectroscopy (AAS). Graphite furnace atomic absorption spectroscopy (GFAAS) is used for analysis of antimony, arsenic, cadmium, lead, selenium, and thallium, if these elements require greater analytical sensitivity than can be obtained by ICAP. Additionally, if desired, the tester may use AAS for analyses of all metals if the detection limits meet the goal of the testing program. For convenience, aliquots of each digested sample fraction can be combined proportionally for a single analytical determination. The efficiency of the analytical procedure is quantified by the analysis of spiked quality control samples containing each of the target metals including actual sample matrix effects checks.

2. Range, Sensitivity, Precision, and Interferences

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

2.2 Analytical Sensitivity. ICAP detection limits in the analytical solution (based on <u>SW-846</u>, Method 6010) are approximately as follows: Sb (32 ng/ml), As (53 ng/ml), Ba (2 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml); Cu (6 ng/ml), Pb (42 ng/ml), Mn (2 ng/ml), Ni (15 ng/ml), P (75 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), Ti (40 ng/ml), and Zn (2 ng/ml). The actual method detection limits are sample dependent and may vary as the sample matrix may affect the limits. The detection limits for analysis by direct aspiration AAS (based on SW-846, Method 7000) are approximately as follows: Sb (200 ng/ml), As (2 ng/ml), Ba (100 ng/ml), Be (5 ng/ml), Cd (5 ng/ml), Cr (50 ng/ml), Cu (20 ng/ml), Pb (100 ng/ml), Mn (10 ng/ml), Ni (40 ng/ml), Se (2 ng/ml), Ag (10 ng/ml), Tl (100 ng/ml), and Zn (5 ng/ml). The detection limit for mercury by CVAAS is approximately 0.2 ng/ml. The use of GFAAS can give added sensitivity compared to the use of direct aspiration AAS for the following metals: Sb (3 ng/ml), As (1 ng/ml), Be (0.2 ng/ml), Cd (0.1 ng/ml), Cr (1 ng/ml), Pb (1 ng/ml), Se (2 ng/ml), and Tl (1 ng/ml). To ensure the possibility of optimum ease in obtaining accurate measurements, the concentration of target metals in samples should be at least ten times the detection limit. Under certain conditions, and with greater care in the analytical procedure, this concentration can be as low as approximately three times the detection limit. However, the scatter of such data may render them unacceptable or may require many analyses before the desired reliability of analytical data is obtained.

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

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

where: A = analytical detection limit, ug/ml.
B = volume of sample prior to aliquot for analysis, ml.
C = stack sample volume, dscm, m^3.
D = in-stack detection limit, ug/m^3.

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

Actual method in-stack detection limits are based on actual test values. If required, this method's in-stack detection limits listed can be improved for a specific test by using one or more of the following options:

- A normal 1-hour sampling run collects a stack gas sampling volume of about 1.25 m^3. If the sampling time is increased and 5 m^3 is collected, the in-stack method detection limits would be one fourth of the values shown above (this means that with this change, the method is four times more sensitive than normal).
- The in-stack detection limits assume that all of the sample is digested (with exception of the aliquot for mercury) and the final liquid volume for analysis is 300 ml for the front half and 150 ml for the back half sample. If the front half volume is reduced from 300 ml to 30 ml, the front half in-stack detection limits would be one tenth of the values shown above (ten times more sensitive). If the back half volume is reduced from 150 ml to 25 ml the in-stack detection limits would be one sixth of the above values. Matrix effects checks are necessary on

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TABLE A-1. IN-STACK METHOD DETECTION LIMITS (ug/m^3) FOR TRAIN FRACTIONS USING ICAP AND AAS

Metal	Front Half Fraction 1 Probe and Filter	Back Half$_1$ Fraction 2 Impingers 1-3	Back Half$_2$ Fraction 3 Impingers 4-5	Total Train
Antimony	7.7 (0.7)*	3.8 (0.4)*		11.5 (1.1)*
Arsenic	12.7 (0.3)*	6.4 (0.1)*		19.1 (0.4)*
Barium	0.5	0.3		0.8
Beryllium	0.07 (0.05)*	0.04 (0.03)*		0.11 (0.08)*
Cadmium	1.0 (0.02)*	0.5 (0.01)*		1.5 (0.3)*
Chromium	1.7 (0.2)*	0.8 (0.1)*		2.5 (0.3)*
Copper	1.4	0.7		2.1
Lead	10.1 (0.2)*	5.0 (0.1)*		15.1 (0.3)*
Manganese	0.5 (0.2)*	0.2 (0.1)*		0.7 (0.3)*
Mercury	0.05**	0.03**	0.03**	0.11**
Nickel	3.6	1.8		5.4
Phosphorus	18	9		27
Selenium	18 (0.5)*	9 (0.3)*		27 (0.8)*
Silver	1.7	0.9		2.6
Thallium	9.6 (0.2)*	4.8 (0.1)*		14.4 (0.3)*
Zinc	0.5	0.3		0.8

( )* Detection limit when analyzed by GFAAS.
** Detection limit when analyzed by CVAAS.
Actual method in-stack detection limits are based on actual test values.

analyses of samples and typically are of greater significance for samples that have been concentrated below the normal sample volume. A volume less than 25 ml may not allow resolubilization of the residue and may increase interference by other compounds.

- When both of the above two improvements are used on one sample at the same time, the resultant improvements are multiplicative. For example, where stack gas volume is increased by a factor of five and the total liquid sample digested volume of both the front and back halves is reduced by factor of six, the in-stack method detection limit is reduced by a factor of thirty (the method is thirty times more sensitive).
- Conversely, reducing stack gas sample volume and increasing sample liquid volume will increase limits. The front half and back half$_1$ samples (Fractions 1 and 2) can be combined prior to analysis. The resultant liquid volume (excluding Fraction 3 which must be analyzed separately) is recorded. Combining the sample as described does not allow determination (whether front or back half$_1$) of where in the train

the sample was captured. The in-stack method detection limit then becomes a single value for all metals, except mercury for which the contribution of Fraction 3 must be considered.

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

2.3 Precision. The precisions (relative standard deviation) for each metal detected in a method development test at a sewage sludge incinerator, are as follows: Sb (13.9%), As (13.5%), Ba (13.1%), Cd (11.5%), Cr (12.5%), Cu (11.9%), Pb (11.6%), Ni (7.7%), P (13.5%), Se (15.3%), Tl (12.3%), and Zn (11.8%). Beryllium, manganese and silver were not detected in the tests; however, based on the analytical sensitivity of the ICAP for these metals, it is assumed that their precisions should be similiar to those for the other metals.

2.4 Interferences. Iron can be a spectral interference during the analysis of arsenic, chromium, and cadmium by ICAP. Aluminum can be a spectral interference during the analysis of arsenic and lead by ICAP. Generally, these interferences can be reduced by diluting the sample, but this increases the method detection limit. Refer to EPA Method 6010 (SW-846) for details on potential interferences for this method. For all GFAAS analyses, matrix modifiers should be used to limit interferences, and standards should be matrix matched.

3. Apparatus

3.1 Sampling Train. A schematic of the sampling train is shown in Figure A-1. It is similar to the Method 5 train. The sampling train consists of the following components.

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

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

3.1.3 Filter Holder. Glass, same as Method 5, Section 2.1.5, except that a Teflon filter support may be used, if desired, to replace the glass frit.

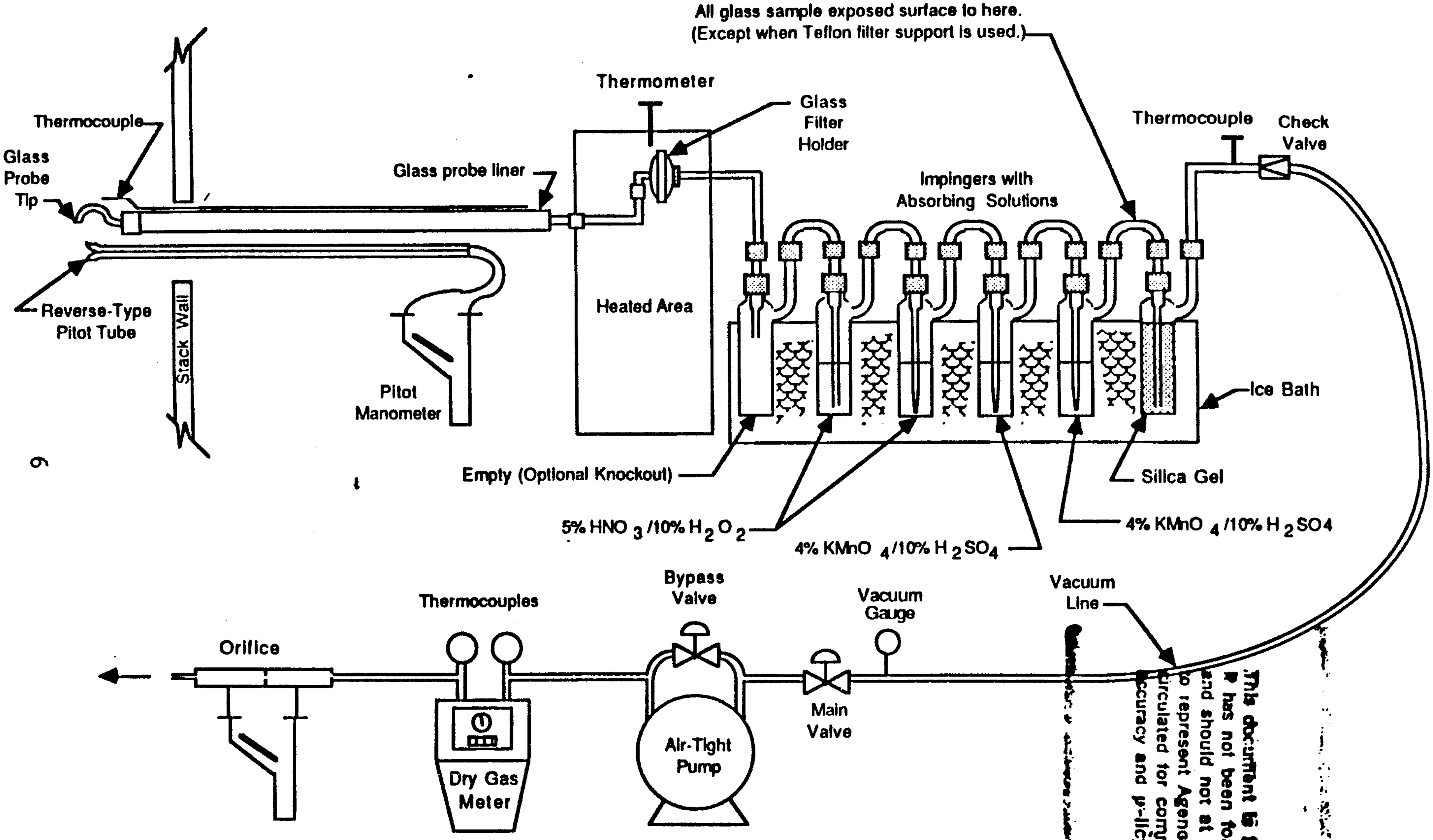

Figure A-1. Schematic of multiple metals sampling train.

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

3.1.5 Condenser. The following system shall be used for the condensation and collection of gaseous metals and for determining the moisture content of the stack gas. The condensing system should consist of four to six impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first impinger is optional and is recommended as a water knockout trap for use during test conditions which require such a trap. The impingers to be used in the metals train are now described. When the first impinger is used as a water knockout, it shall have a short stem. The second impinger (or the first HNO_3/H_2O_2 impinger) shall be as described for the first impinger in Method 5, Paragraph 2.1.7. The third impinger (or the impinger used as the second HNO_3/H_2O_2 impinger in any case) is the same as the Greenburg Smith impinger with the standard tip described as the second impinger in Method 5, Paragraph 2.1.7. All other impingers used in the metals train are the same as the second impinger previously described. The first impinger should be empty, the second and third shall contain known quantities of a nitric acid/hydrogen peroxide solution (Section 4.2.1), the fourth (and fifth, if required) shall contain a known quantity of acidic potassium permanganate solution (Section 4.2.2), and the last impinger shall contain a known quantity of silica gel or equivalent desiccant. A thermometer capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger. When the water knockout impinger is not needed, it is removed from the train and the other impingers remain the same. If mercury analysis is not needed, the potassium permanganate impingers are removed.

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

3.2 Sample Recovery. The following items are needed for sample recovery:

3.2.1 Nonmetallic Probe Liner and Probe Nozzle Brushes, Wash Bottles, Sample Storage Containers, Petri Dishes, Graduated Cylinders, Plastic Storage Containers, Funnel and Rubber Policeman, and Glass Funnel. Same as Method 5, Sections 2.2.1 through 2.2.8, respectively.

3.2.2 Labels. For identification of samples.

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

3.2.4 Nonmetallic Bristle Brush. For quantitative recovery of materials collected in the front half of the sampling train.

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

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

3.3.2 Graduated Cylinders. For preparation of reagents.

3.3.3 Parr^{R} Bombs or Microwave Pressure Relief Vessels with Capping Station (CEM Corporation model or equivalent).

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

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

3.3.6 Filter Funnels. For holding filter paper.

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

3.3.8 Disposable Pasteur Pipets and Bulbs.

3.3.9 Volumetric Pipets.

3.3.10 Analytical Balance. Accurate to within 0.1 mg.

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

3.3.12 Hot Plates.

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

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

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

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

4. Reagents

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

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

4.1.1 Filters. The filters shall contain less than 1.25 ug of each of the metals to be measured. Analytical results provided by filter manufacturers are acceptable. However, if no such results are available, filter blanks must be analyzed for each target metal prior to emission testing. Quartz fiber or glass fiber filters without organic binders shall be used. The filters should exhibit at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D2986-71 (incorporated by reference). For particulate determination, the filter should have a surface alkalinity less than 7.5 pH. Again, analytical results provided by filter manufacturers are acceptable. Pallflex^{R} type 2500 QAT-UP Ultra Pure Filters have been found to meet these limits.

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

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

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

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

4.1.6 Potassium Permanganate.

4.1.7 Sulfuric Acid. Concentrated.

4.1.8 Silica Gel and Crushed Ice. Same as Method 5, Sections 3.1.2 and 3.1.3, respectively.

4.2 Pretest Preparation for Sampling Reagents.

4.2.1 Nitric Acid/Hydrogen Peroxide Absorbing Solution. Add 50 ml of concentrated nitric acid and 333 ml of 30 percent hydrogen peroxide to a 1000 ml volumetric flask or graduated cylinder containing approximately 500 ml of water. Dilute to volume with water. The reagent shall contain less than 2 ng/ml of each target metal.

4.2.2 Acidic Potassium Permanganate Solution. Fill a 1 liter volumetric flask or graduated cylinder with approximately 800 ml of water. Weigh out 40.0 g of potassium permanganate and dissolve it in the water. Add 100 ml of concentrated sulfuric acid and mix well. Let the solution cool and dilute to volume with water. The reagent shall contain less than 2 ng/ml of each target metal.

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

4.2.3 Nitric Acid, 0.1 N. Add 6.3 ml of concentrated nitric acid (70 percent) to a graduated cylinder containing approximately 900 ml of water. Dilute to 1000 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of each target metal.

4.2.4 Hydrochloric Acid, 8 N. Add 690 ml of concentrated hydrochloric acid to a graduated cylinder containing 250 ml of water. Dilute to 100 ml with water. Mix well. The reagent shall contain less than 2 ng/ml of each the target metals.

4.3 Glassware Cleaning Reagents.

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

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

4.3.3 Nitric Acid, 10 Percent (V/V). Add 500 ml of concentrated nitric acid to a graduated cylinder containing approximately 4000 ml of water. Dilute to 5000 ml with water.

4.4 Sample Digestion and Analysis Reagents.

4.4.1 Hydrochloric Acid, Concentrated.

4.4.2 Hydrofluoric Acid, Concentrated.

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

4.4.4 Nitric Acid, 10 Percent (V/V). Add 100 ml of concentrated nitric acid to 800 ml of water. Dilute to 1000 ml with water. Mix well. Reagent shall contain less than 2 ng/ml of each target metal.

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4.4.5 Nitric Acid, 5 Percent (V/V). Add 50 ml of concentrated nitric acid to 800 ml of water. Dilute to 1000 ml with water. Reagent shall contain less than 2 ng/ml of each target metal.

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

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

4.4.8 Stannous Chloride.

4.4.9 Potassium Permanganate, 5 Percent (W/V).

4.4.10 Sulfuric Acid, Concentrated.

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

4.4.12 Potassium Persulfate, 5 Percent (W/V).

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

4.4.14 Lanthanum Oxide, La_2O_3.

4.4.15 AAS Grade Hg Standard, 1000 ug/ml.

4.4.16 AAS Grade Pb Standard, 1000 ug/ml.

4.4.17 AAS Grade As Standard, 1000 ug/ml.

4.4.18 AAS Grade Cd Standard, 1000 ug/ml.

4.4.19 AAS Grade Cr Standard, 1000 ug/ml.

4.4.20 AAS Grade Sb Standard, 1000 ug/ml.

4.4.21 AAS Grade Ba Standard, 1000 ug/ml.

4.4.22 AAS Grade Be Standard, 1000 ug/ml.

4.4.23 AAS Grade Cu Standard, 1000 ug/ml.

4.4.24 AAS Grade Mn Standard, 1000 ug/ml.

4.4.25 AAS Grade Ni Standard, 1000 ug/ml.

4.4.26 AAS Grade P Standard, 1000 ug/ml.

4.4.27 AAS Grade Se Standard, 1000 ug/ml.

4.4.28 AAS Grade Ag Standard, 1000 ug/ml.

4.4.29 AAS Grade Tl Standard, 1000 ug/ml.

4.4.30 AAS Grade Zn Standard, 1000 ug/ml.

4.4.31 AAS Grade Al Standard, 1000 ug/ml.

4.4.32 AAS Grade Fe Standard, 1000 ug/ml.

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

4.4.34 Mercury Standards and Quality Control Samples. Prepare a 10 ug/ml mercury standard by adding 5 ml of 1000 ug/ml mercury standard to a 500 ml volumetric flask. Dilute to 500 ml with 20 ml of 15 percent nitric acid and then water. Prepare a 200 ng/ml standard by adding 5 ml of the 10 ug/ml standard to a 250 ml volumetric flask and dilute to 250 ml with 5 ml of 4% $KMnO_4$, 5 ml 15 percent nitric acid, and then water. Other standards should be prepared by dilution of the 200 ng/ml mercury standard. At least four standards should be used to prepare the standard curve. Standards containing 0.25, 0.50, 1.00, and 2.00 total ng are suggested. Quality control samples should be prepared by making a separate 10 ng/ml standard and diluting until it is in the range of the samples.

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

MIXED STANDARD SOLUTIONS FOR ICAP ANALYSIS

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

Prepare these standards by combining and diluting the appropriate volumes of the 1000 ug/ml solutions with 5 percent nitric acid. A minimum of one standard and a blank can be used to form each calibration curve. However, a separate quality control sample spiked with known amounts of the target metals in quantities expected to be in the midrange of the calibration curve should be prepared. Suggested standard levels are 50 ug/ml for Al, 25 ug/ml for Cr and Pb, 15 ug/ml for Fe, and 10 ug/ml for the remaining elements. Standards containing less than 1 ug/ml of metal should be prepared daily. Standards containing greater than 1 ug/ml of metal should be stable for a minimum of 1 to 2 weeks.

4.4.36 Graphite Furnace AAS Standards for Antimony, Arsenic, Cadmium, Lead, Selenium, and Thallium. Prepare a 10 ug/ml standard by adding 1 ml of 1000 ug/ml standard to a 100 ml volumetric flask. Dilute to 100 ml with 10 percent nitric acid. For graphite furnace AAS, the standards must be matrix matched; e.g., if the samples contain 6 percent nitric acid and 4 percent hydrofluoric acid, the standards should also be made up with 6 percent nitric

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acid and 4 percent hydrofluoric acid. Prepare a 100 ng/ml standard by adding 1 ml of the 10 ug/ml standard to a 100 ml volumetric flask and dilute to 100 ml with the appropriate matrix solution. Other standards should be prepared by dilution of the 100 ng/ml standards. At least four standards should be used to make up the standard curve. Suggested levels are 10, 50, 75, and 100 ng/ml. Quality control samples should be prepared by making a separate 10 ug/ml standard and diluting until it is in the range of the samples. Standards containing less than 1 ug/ml of metal should be prepared daily. Standards containing greater than 1 ug/ml of metal should be stable for a minimum of 1 to 2 weeks.

4.4.37 Matrix Modifiers.

4.4.37.1 Nickel Nitrate, 1 Percent (V/V). Dissolve 4.956 g of $Ni(NO_3)_2 \cdot 6H2O$ in approximately 50 ml of water in a 100 ml volumetric flask. Dilute to 100 ml with water.

4.4.37.2 Nickel Nitrate, One-tenth Percent (V/V). Dilute 10 ml of 1 percent nickel nitrate solution to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for As.

4.4.37.3 Lanthanum. Dissolve 0.5864 g of La_2O_3 in 10 ml of concentrated HNO_3 and dilute to 100 ml with water. Inject an equal amount of sample and this modifier into the graphite furnace during AAS analysis for Pb.

5. Procedure

5.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

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

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

5.1.3 Preparation of Sampling Train. Follow the same general procedures given in Method 5, Section 4.1.3, except place 100 ml of the nitric acid/hydrogen peroxide solution (Section 4.2.1) in the two HNO_3/H_2O_2 impingers (normally the second and third impingers), place 100 ml of the acidic potassium permanganate solution (Section 4.2.2) in the fourth and fifth impinger, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the last impinger. Alternatively, the silica gel may be weighed directly in the impinger just prior to train assembly.

Several options are available to the tester based on the sampling conditions. The use of an empty first impinger can be eliminated if the moisture to be collected in the impingers is calculated or determined to be less than 150 ml. The tester shall include two impingers containing the acidic potassium permanganate solution for the first test run, unless past testing experience at the same or similar sources has shown that only one is necessary. The last permanganate impinger may be discarded if both permanganate impingers have retained their original deep purple permanganate color. A maximum of 200 ml in each permanganate impinger (or a maximum of three permanganate impingers) may be used, if necessary, to maintain the desired color in the last permanganate impinger.

Retain for reagent blanks, 100 ml of the nitric acid/hydrogen peroxide solution and 100 ml of the acidic potassium permanganate solution. These solutions should be labeled and treated as described in Section 7. Set up the sampling train as shown in Figure A-1.

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

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

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

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

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

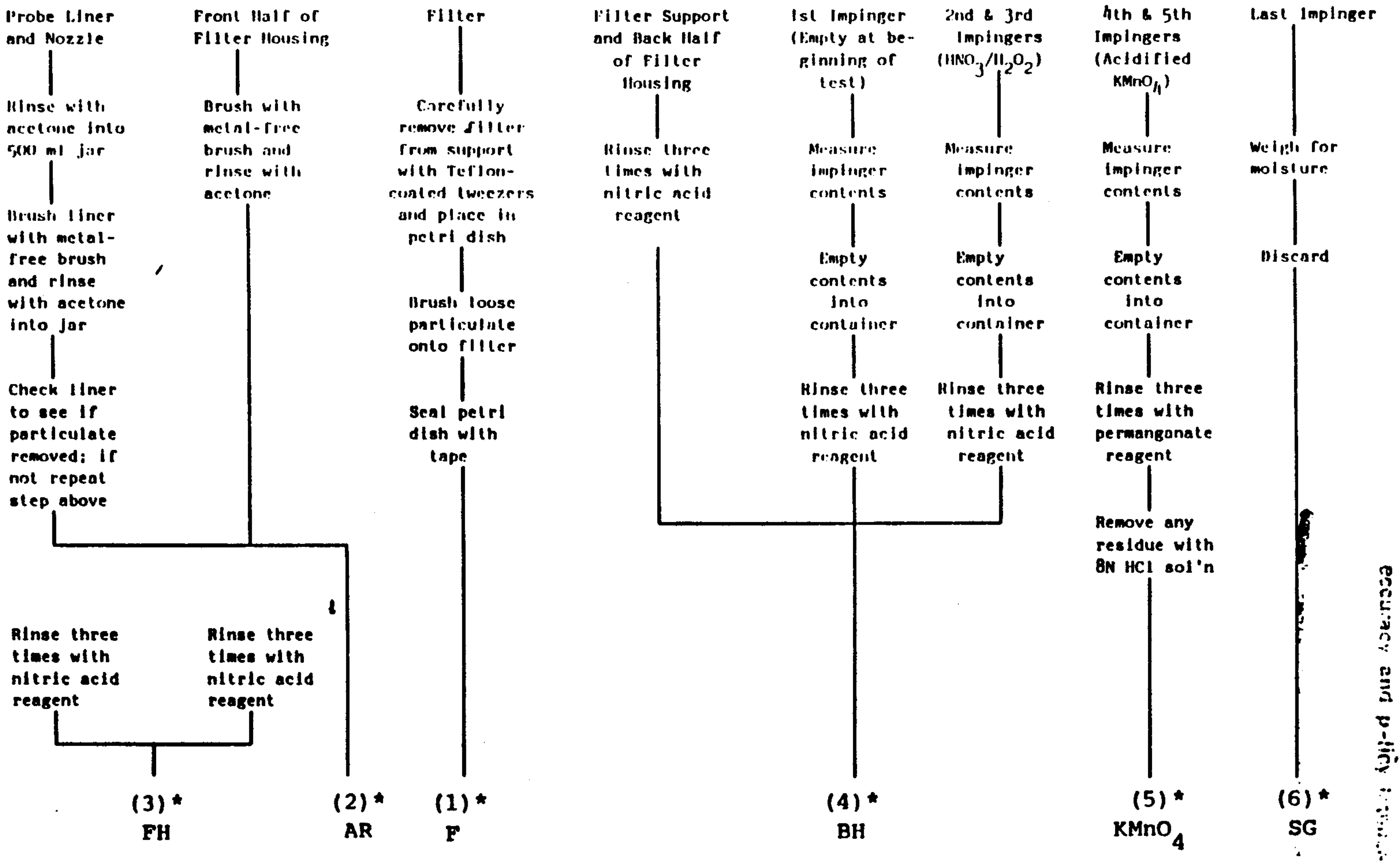
The probe should be allowed to cool prior to sample recovery. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a rinsed, non-comtaminating cap over the probe nozzle to prevent losing or gaining particulate matter. Do not cap the probe tip tightly while the sampling train is cooling. This normally causes a vacuum to form in the filter holder, thus causing the undesired result of drawing liquid from the impingers into the filter.

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

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

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

5.2.2 Container No. 2 (Probe). <u>Note</u>: Container No. 2 can be omitted if the testing does not include the determination of particulate concentration. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate



* Indicates container numbers.

Figure A-2. Sample recovery scheme.

matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a metal-free Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

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

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid on transferring liquid washings to the container. Follow the acetone rinse with a metal-free probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

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

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a metal-free nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to

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the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container clearly to identify its contents.

5.2.3 Container No. 3 (Probe Rinse). Rinse the probe liner, probe nozzle, and front half of the filter holder by rinsing these components thoroughly with 0.1 N nitric acid and placing the wash into a sample storage container. Perform the rinses as described in Method 12, Section 5.2.2. Measure and record the volume of the combined rinses, and place the sample into a storage container. Mark the height of the fluid level on the outside of the container and use this mark to determine if leakage occurs during transport. Seal the container and clearly label the contents.

5.2.4 Container No. 4 (Impingers 1 through 3, Contents and Rinses). Due to the large quantity of liquid involved, the tester may place the impinger solutions in more than one container. Measure the liquid in the first three impingers volumetrically to within 0.5 ml using a graduated cylinder. Record the volume of liquid present. This information is required to calculate the moisture content of the sampled flue gas. Clean each of the first three impingers and connecting glassware by thoroughly rinsing with 0.1 N nitric acid as described in Method 12, Section 5.2.4. Combine the rinses and impinger solutions, measure and record the volume. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport. Seal the container and clearly label the contents.

5.2.5 Container No. 5 (Acidified Potassium Permanganate Solution and Rinses, Impingers No. 4 & 5). Pour all the liquid from the permanganate impingers (fourth and fifth, if two permanganate impingers are used) into a graduated cylinder and measure the volume to within 0.5 ml. This information is required to calculate the moisture content of the sampled flue gas. Rinse the permanganate impinger(s) and connecting glass pieces a minimum of three times with acidified potassium permanganate solution. Combine the rinses with the permanganate impinger solution in a graduated cylinder and measure the total volume within 0.5 ml. Place the combined rinses and impinger contents in a labeled container. Mark the height of the fluid level on the outside of the container to determine if leakage occurs during transport. See the following note and the Precaution in Paragraph 4.2.2 and properly seal the container and clearly label the contents.

Note: Due to the potential reaction of the potassium permanganate with the acid, there may be pressure buildup in the sample storage bottles. These bottles should not be filled full and should be vented to relieve excess pressure. Venting is highly recommended. A No. 70-72 hole drilled in the container cap and Teflon liner has been found to allow adequate venting without loss of sample.

5.2.6 Container No. 6 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. The small amount of particles that may adhere to the impinger wall need not be removed. Do not use water or other liquids to transfer the silica gel since weight gained in the silica gel impinger is used for moisture calculations. Alternatively, if a balance is available in the field, record the weight of the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

5.2.7 Container No. 7 (Acetone Blank). Once during each field test, place 100 ml of the acetone used in the sample recovery process into a labeled container for use as a recovery solvent blank. Seal the container.

5.2.8 Container No. 8 (0.1 N Nitric Acid Blank). Once during each field test, place 100 ml of the 0.1 N nitric acid solution used in the sample recovery process into a labeled container for use as a recovery solvent blank. Seal the container.

5.2.9 Container No. 9 (5% Nitric Acid/10% Hydrogen Peroxide Blank). Once during each field test, place 100 ml of the 5% nitric acid/10% hydrogen peroxide solution used as the nitric acid impinger reagent into a labeled container for use as a blank. Seal the container.

5.2.10 Container No. 10 (Acidified Potassium Permanganate Blank). Once during each field test, place 100 ml of the acidified potassium permanganate solution used as the impinger solution and in the sample recovery process into a labeled container for use as a blank. Seal the container.

Note: This container should be vented, as described in Section 5.2.4, to relieve excess pressure.

5.2.11 Container No. 11 (Filter Blank). Once during each field test, place an unused filter from the same lot as the sampling filters in a labeled petri dish. Seal the petri dish.

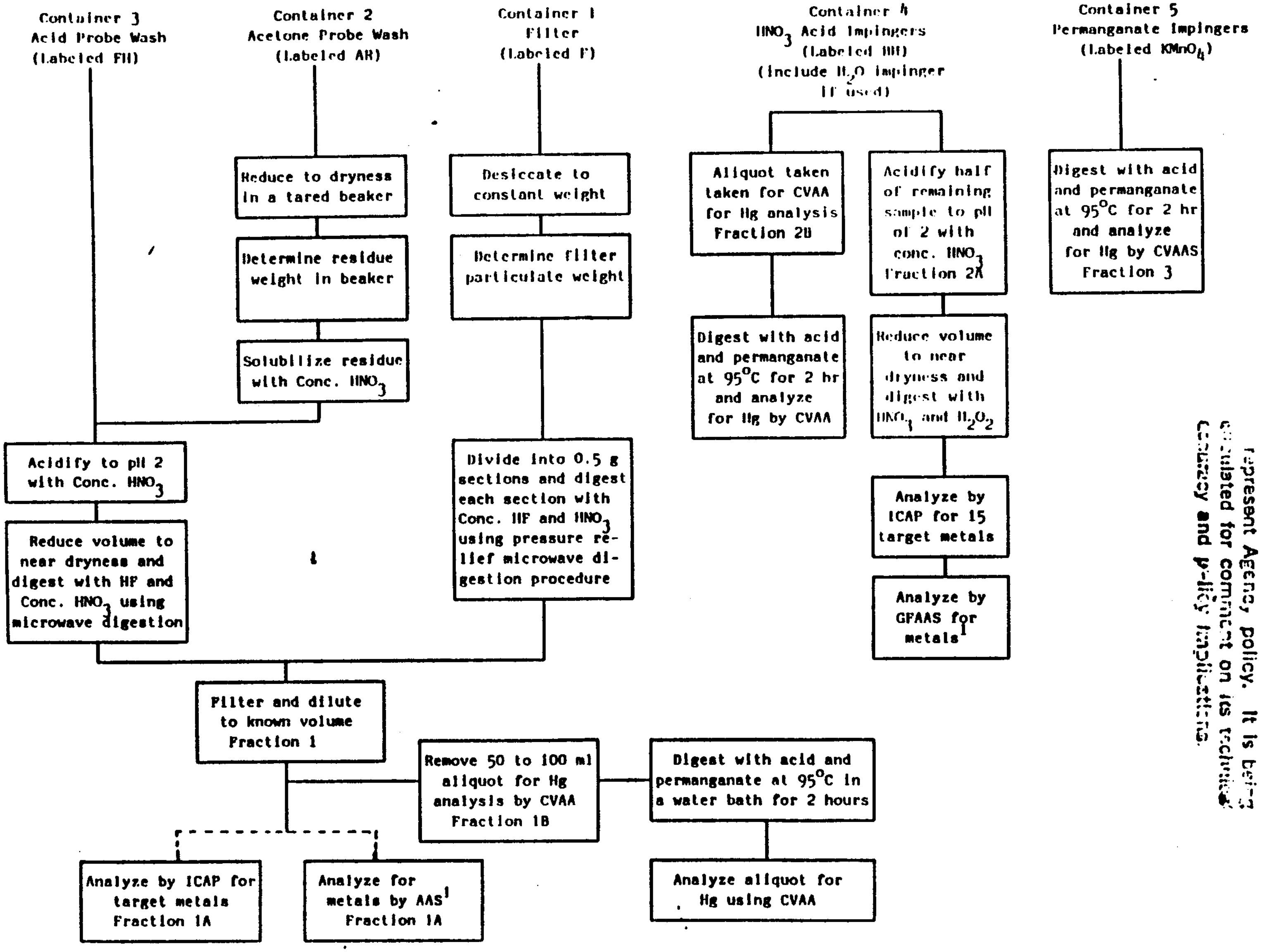
5.3 Sample Preparation. Note the level of the liquid in each of the containers and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. A diagram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

5.3.1 Container No. 1 (Filter). The filter with its filter catch should be divided into portions containing approximately 0.5 g each and placed into the analyst's choice of either individual microwave pressure relief vessels or Parr^{R} Bombs. Six ml of concentrated nitric acid and 4 ml of concentrated hydrofluoric acid should be added to each vessel. For microwave heating, microwave the sample vessels for approximately 12-15 minutes in intervals of 1 to 2 minutes at 600 Watts. For conventional heating, heat the Parr Bombs at 140°C (285°F) for 6 hours. Then cool the samples to room temperature and combine with the acid digested probe rinse as required in Section 5.3.3, below.

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

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

5.3.2 Container No. 2 (Probe). Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to an acid-cleaned tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight according to the procedures described in Section 4.3 of Method 5. Report the results to the nearest 0.1 mg. Resolubilize the residue with concentrated nitric acid and combine the resultant sample including all liquid and any particulate matter with Container No. 3 prior to beginning the following step 5.3.3.



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

Figure A-3. Sample preparation and analysis scheme.

5.3.3 Container No. 3 (Probe Rinse). The pH of this sample shall be 2 or lower. If the pH is higher, the sample should be acidified with concentrated nitric acid to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 50 ml by heating on a hot plate at a temperature just below boiling. Inspect the sample for visible particulate matter, and depending on the results of the inspection, perform one of the following. If no particulate matter is observed, combine the sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1. If particulate matter is observed, digest the sample in microwave vessels or Parr^{R} Bombs following the procedures described in Section 5.3.1; then combine the resultant sample directly with the acid digested portions of the filter prepared previously in Section 5.3.1. The resultant combined sample is referred to as Fraction 1. Filter the combined solution of the acid digested filter and probe rinse samples using Whatman 541 filter paper. Dilute to 300 ml (or the appropriate volume for the expected metals concentration) with water. Measure and record the combined volume of the Fraction 1 solution to within 0.1 ml. Quantitatively remove a 50 ml aliquot and label as Fraction 1B. Label the remaining 250 ml portion as Fraction 1A. Fraction 1A is used for ICAP or AAS analysis. Fraction 1B is used for the determination of front half mercury.

5.3.4 Container No. 4 (Impingers 1-3). Measure and record the total volume of this sample to within 0.5 ml. Remove a 50 ml aliquot for mercury analysis and label as Fraction 2B. The Fraction 2B aliquot should be prepared and analyzed as described in Section 5.4.3. The remaining portion of Container No. 3 should be labeled Fraction 2A and shall be pH 2 or lower. If necessary, use concentrated nitric acid to lower this fraction to pH 2. The sample should be rinsed into a beaker with water and the beaker should be covered with a ribbed watchglass. The sample volume should be reduced to approximately 20 ml by heating on a hot plate at a temperature just below boiling. Then follow either of the digestion procedures described in Sections 5.3.4.1 and 5.3.4.2, below.

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

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

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

5.3.5 Container No. 5 (Impingers 4 & 5). Measure and record the total volume of this sample to within 0.5 ml. This sample is referred to as Fraction 3. Follow the analysis procedures described in Section 5.4.3.

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

5.4 Sample Analysis. For each sampling train, five individual samples are generated for analysis. A schematic identifying each sample and the prescribed sample preparation and analysis scheme is shown in Figure A-3. The first two samples, labeled Fractions 1A and 1B, consist of the digested samples from the front half of the train. Fraction 1A is for ICAP or AAS analysis as described in Sections 5.4.1 and/or 5.4.2. Fraction 1B is for determination of front half mercury as described in Section 5.4.3.

The back half of the train was used to prepare the third through fifth samples. The third and fourth samples, labeled Fractions 2A and 2B, contain the digested samples from the H_2O and NHO_3/H_2O_2 Impingers 1 through 3. Fraction 2A is for ICAP or AAS analysis. Fraction 2B will be analyzed for mercury.

The fifth sample, labeled Fraction 3, consists of the impinger contents and rinses from the permanganate Impingers 4 and 5. This sample is analyzed for mercury as described in Section 5.4.3. The total back half mercury catch is determined from the sum of Fraction 2B and Fraction 3.

5.4.1 ICAP Analysis. Fraction 1A and Fraction 2A are analyzed by ICAP using EPA Method 200.7 (40 CFR 136, Appendix C). Calibrate the ICAP and set up an analysis program as described in Method 200.7. The quality control procedures described in Section 7.3.1 of this method shall be followed.

Recommended wavelengths for use in the analysis are listed below.

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

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

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

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

5.4.2 AAS by Direct Aspiration and/or Graphite Furnace. If analysis of metals in Fraction 1A and Fraction 2A using graphite furnace or direct aspiration AAS is desired, Table A-2 should be used to determine which techniques and methods should be applied for each target metal. Table A-2 should also be consulted to determine possible interferences and techniques to be followed for their minimization. Calibrate the instrument according to Section 6.3 and follow the quality control procedures specified in Section 7.3.2.

5.4.3 Cold Vapor AAS Mercury Analysis. Fraction 1B, Fraction 3, and Fraction 2B should be analyzed for mercury using cold vapor atomic absorption spectroscopy following the method outlined in EPA Method 7470 or in Standard

TABLE A-2. APPLICABLE TECHNIQUES, METHODS, AND MINIMIZATION OF INTEFERENCE FOR AAS ANALYSIS

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

(continued)

TABLE A-2 (CONTINUED)

Metal	Technique	Method No.	Wavelength (nm)	Interference Cause	Interference Minimization
Cu	Aspiration	7210	324.7	Absorpt & scatter	Consult manufacturer's manual
Fe	Aspiration	7380	248.3	Contamination	Great care taken to aviod contamination
Pb	Aspiration	7420	283.3	217.0 nm alternat	Background correction required
Pb	Furnace	7421	283.3	Poor recoveries	Matrix modifier, add 10 uL of phosphorus acid to 1-mL of prepared sample in sampler cup
Mn	Aspiration	7460	279.5	403.1 nm alternat	Background correction required
Ni	Aspiration	7520	232.0	352.4 nm alternat	Background correction required
				Fe, Co, & Cr	Matrix matching or a nitrous-oxide/acety flame
				Nonlinear respons	Sample dilution or use 352.4 nm line
Se	Furnace	7740	196.0	Volitality	Spike samples & reference materials & add nickel nitrate to minimize volatilization
				Adsorpt & scatter	Background correction is required & Zeeman background correction can be useful
Ag	Aspiration	7760	328.1	Absorpt & scatter	Background correction is required
				AgCl insoluble	Avoid hydrochloric acid unless silver is in solution as a chloride complex
				Viscosity	Sample & standards monitored for apiration rate
Tl	Aspiration	7840	276.8		Background correction is required Hydrochloric acid should not be used
Tl	Furnace	7841	276.8		Background correction is required
				Hydrochloric acid or chloride	Verify that losses are not occuring for volitization by spiked samples or standad addt Palladium is a suitable matrix modifier
Zn	Aspiration	7950	213.9	High Si, Cu & P	Strontium removes Cu and phosphate
				Contamination	Care should be taken to avid contamination

Methods for Water and Wastewater Analysis, 15th Edition, Method 303F. Set up the calibration curve as described in Section 7.3 of Method 303F. Add approximately 5 ml of each sample to BOD bottles. Record the amount of sample added. The amount used is dependent upon the expected levels of mercury. Dilute to approximately 120 ml with mercury-free water. Add approximately 15 ml of 5 percent potassium permanganate solution to the Fraction 2B and Fraction 3 samples. Add 5 percent potassium permanganate solution to the Fraction 1B sample as needed to produce a purple solution lasting at least 15 minutes. A minimum of 25 ml is suggested. Add 5 ml of 50 percent nitric acid, 5 ml of concentrated sulfuric acid, and 9 ml of 5 percent potassium persulfate to each sample and each standard. Digest the solution in the capped BOD bottle at 95°C (205°F) in a convection oven or water bath for 2 hours. Cool. Add 5 ml of hydroxylamine hydrochloride solution and mix the sample. Then add 7 ml of stannous chloride to each sample and analyze immediately.

6. Calibration

Maintain a laboratory log of all calibrations.

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

6.2 Inductively Coupled Argon Plasma Spectrometer Calibration. Prepare standards as outlined in Section 4.6.4. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures using the above standards. The instrument calibration should be checked once per hour. If the instrument does not reproduce the concentrations of the standard within 10 percent, the complete calibration procedures should be performed.

6.3 Atomic Absorption Spectrometer - Direct Aspiration, Graphite Furnace and Cold Vapor Mercury Analyses. Prepare the standards as outlined in Section 4.6.5. Calibrate the spectrometer using these prepared standards. Calibration procedures are also outlined in the EPA methods referred to in Table A-2 and in Standard Methods for Water and Wastewater, 15th Edition, Method 303F (for mercury). Each standard curve should be run in duplicate and the mean values used to calculate the calibration line. The instrument should be recalibrated approximately once every 10 to 12 samples.

7. Quality Control

7.1 Sampling. Field Reagent Blanks. The blank samples in Container Numbers 7 through 11 produced previously in Sections 5.2.7 through 5.2.11, respectively, shall be processed, digested, and analyzed as follows. Digest and process Container No. 11 contents per Section 5.3.1, Container No. 7 per Section 5.3.2, and Container No. 8 per Section 5.3.3. This produces Fraction Blank 1A and Fraction Blank 1B. Digest and process Container No. 9 contents per Section 5.3.4. This produces Fraction Blank 2A and Fraction Blank 2B. Container No. 11 contents are Fraction Blank 3. Analyze Fraction Blank 1A and Fraction Blank 2A per Section 5.4.1 and/or 5.4.2. Analyze Fraction Blank 1B, Fraction Blank 2B, and Fraction Blank 3 per Section 5.4.3. The maximum correction allowed to the field source sample value is the lesser of the following: (1) the actual blank value, or (2) the maximum blank correction allowed per the Note in Section 8.4.3 and the Note in Section 8.5.2.

7.2 With prior approval by the Administrator, an attempt may be made to determine if the reagents used in Section 5.3 caused contamination. They should be analyzed by the procedures in Section 5.4. Then the Administrator will determine whether or not the laboratory blank values can be used in the calculation of the stationary source test results.

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

7.3.1 ICAP Analysis. Follow the quality control shown in Section 8 of Method 6010. For the purposes of a three run test series, these requirements have been modified to include the following: two instrument check standard runs, two calibration blank runs, one interference check sample at the beginning of the analysis (must be within 25% or analyze by standard addition), one quality control sample to check the accuracy of the calibration standards (must be within 25% of calibration), and one duplicate analysis (must be within 5% of average or repeat all analysis).

7.3.2 Direct Aspiration and/or Graphite Furnace AAS Analysis for Antimony, Arsenic, Barium, Beryllium, Cadmium, Copper, Chromium, Lead, Nickel, Manganese, Mercury, Phosphorus, Selenium, Silver, Thallium, and Zinc. All samples should be analyzed in duplicate. Perform a matrix spike on one front half sample and one back half sample or one combined sample. If recoveries of less than 75 percent or greater than 125 percent are obtained for the matrix spike, analyze each sample by the method of additions. A quality control sample should be analyzed to check the accuracy of the calibration standards. The results must be within 10% or the calibration repeated.

7.3.3 Cold Vapor AAS Analysis for Mercury. All samples should be analyzed in duplicate. A quality control sample should be analyzed to check the accuracy of the calibration standards (within 10% or repeat calibration). Perform a matrix spike on one sample from the nitric impinger portion (must be within 25% or samples must be analyzed by the method of standard additions). Additional information on quality control can be obtained from EPA Method 7470 or in Standard Methods for Water and Wastewater, 15th Edition, Method 303F.

8. Calculations

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

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

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

8.4 Metals (Except Mercury) in Source Sample.

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

$$M_{fh} = C_a F_{dm} V_{soln} \qquad \text{Eq. 1}$$

where:

M_{fh} = total mass of each metal (except Hg) collected in the front half of the sampling train (Fraction 1), ug.

C_a = concentration of metal in sample Fraction 1A as read from the standard curve (ug/ml).

F_{dm} = dilution factor (F_{dm} = the inverse of the fractional portion V_{conc-p} in V_{conc}. V_{conc} is the solution actually used in the instrument to produce the reading which is C_a. V_{conc} is either a pure or a diluted solution of Fraction 1A. When V_{conc} has been diluted to bring it into the analytical range based on the calibration of the instrument, V_{conc} will have the following two portions: V_{conc-d}, which is the diluent solution, and V_{conc-p}, which is the original Fraction 1A solution. For example, when the dilution of Fraction 1A is from 2 to 10 ml, the fractional portion V_{conc-p} (2 ml) in V_{conc} (10 ml) is 1/5, and F_{dm} = 5).

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

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

$$M_{bh} = C_a \, F_a \, V_a \qquad \text{Eq. 2}$$

where:

M_{bh} = total mass of metal (except Hg) collected in the back half of the sampling train (Fraction 2), ug.
C_a = concentration of metal in sample Fraction 2A, as read from the standard curve (ug/ml).
F_a = aliquot factor, volume of Fraction 2 divided by volume of aliquot Fraction 2A.
V_a = volume of sample aliquot analyzed (concentrated Fraction 2A), ml.

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

$$M_t = (M_{fh} - M_{fb}) + (M_{bh} - M_{bb}) \qquad \text{Eq. 3}$$

where:

M_t = total mass of each metal (separately stated for each metal) collected in the sampling train, ug.
M_{fb} = blank correction value of mass of metal detected in front half of field reagent blank, ug.
M_{bb} = blank correction value of mass of metal detected in back half of field reagent blank, ug.

Note: The maximum value of field reagent blank that may be subtracted is 2 ug for the front half and 1 ug for the back half, or 3 ug for an analysis of the solution resulting from combining Fraction 1A and Fraction 2A;* or 5% of the average mass for the correponding fraction of the sampling train, whichever is greater.

8.5 Mercury in Source Sample.

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

$$Hg_{fh} = \left(\frac{C}{V_{bf}}\right) \times V_{soln} \qquad \text{Eq. 4}$$

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

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circulated for comment on its technical
accuracy and policy implications

where:

Hg_{fh} = total mass of mercury collected in the front half of the sampling train (Fraction 1), ug.
C = quantity of mercury in analyzed sample, ug.
V_{soln} = total volume of digested sample solution (Fraction 1), ml.
V_{bf} = volume of Fraction 1B analyzed, ml. See the following Note.

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

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

$$Hg_{(F2B)} = \left(\frac{C}{V_{bb2}}\right) \times V_{soln} \qquad \text{Eq. 5}$$

where:

$Hg_{(F2B)}$ = total mass of mercury collected in Fraction 2, ug.
C = quantity of mercury in analyzed sample, ug.
V_{bb2} = volume of Fraction 2B analyzed, ml (see Note in Section 8.5.1).
V_{soln} = total volume of Fraction 2, ml.

$$Hg_{(F3)} = \left(\frac{C}{V_{bb3}}\right) x\ V_{soln} \qquad \text{Eq. 6}$$

where:

$Hg_{(F3)}$ = total mass of mercury collected in Fraction 3, ug.
C = quantity of mercury in analyzed sample, ug.
V_{bb3} = volume of Fraction 3 analyzed, ml (see Note in Section 8.5.1).
V_{soln} = total volume of Fraction 3, ml.

$$Hg_{bh} = Hg_{(F2B)} + Hg_{(F3)} \qquad \text{Eq. 7}$$

where:

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

formally released by EPA and should not at this stage be construed to represent Agency policy. It is being circulated for comment on its technical accuracy and policy implications.

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

$$M_t = (Hg_{fh} - Hg_{fb}) + (Hg_{bh} - Hg_{bb}) \qquad \text{Eq. 8}$$

where:

M_t = total mass of mercury collected in the sampling train, ug.

Hg_{fb} = blank correction value of mass of mercury detected in the front half of the field reagent blank, ug.

Hg_{bb} = blank correction value of mass of mercury detected in the back half of the field reagent blank, ug.

Note: The maximum value of field reagent blank that may be subtracted is 1 ug for the front half and 2 ug for the back half, or 3 ug for a combined front half/back half analysis; or 5% of the average mass of mercury for the correponding fraction of the sampling train; whichever is greater.

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

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

where:

C_s = concentration of each metal in the stack gas, mg/dscm.

K_4 = 10^{-3} mg/ug.

M_t = total mass of each metal collected in the sampling train, ug.

$V_{m(std)}$ = volume of gas sample as measured by the dry gas meter, corrected to dry standard conditions, dscm.

8.7 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively. To calculate the average stack gas velocity, use Equation 2-9 of Method 2 and the data from this field test.

9. Bibliography

9.1 Ward, T.E., S.C. Steinsberger, W.G. DeWees, R.R. Segall, W.S. Smith, K.M. Luk, and P.M. Grohse. "Laboratory and Field Evaluation of Methodology for Measuring Emissions of Trace Metals for Stationary Sources," Draft report prepared for U.S. Environmental Protection Agency, Environmental Monitoring

Systems Laboratory, Research Traingle Park, NC, under Contract No. 68-02-4442, September 1988.

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

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

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

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

TABLE 4.—ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HIGH RESOLUTION MASS SPECTROMETRY FOR PCDDS AND PCDFS—Continued

Descriptor No.	Accurate mass	Ion type	Elemental composition	Analyte
	513.6775 442.9728	M+4 LOCK	$C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	DCDPE PFK

(a) The following nuclidic masses were used: H = 1.007825 C = 12.000000 ^{13}C = 13.003355 F = 18.9984 O = 15.994915 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903 S = Labeled Standard

TABLE 5.—ACCEPTABLE RANGES FOR ION-ABUNDANCE RATIOS OF PCDDS AND PCDFS

Number of chlorine atoms	Ion type	Theoretical ratio	Control limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6[1]	M/M+2	0.51	0.43	0.59
7[2]	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

[1] Used only for ^{13}C-HxCDF.

[2] Used only for ^{13}C-HpCDF.

TABLE 6.—MINIMUM REQUIREMENTS FOR INITIAL AND DAILY CALIBRATION RESPONSE FACTORS

Compound	Relative Response Factors	
	Initial calibration RSD	Daily calibration percentage difference
Unlabeled Analytes:		
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDF	25	25
1,2,4,5,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25
OCDF	30	30
Internal standards:		
$^{13}C_{12}$-2,3,7,8-TCDD	25	25
$^{13}C_{12}$-1,2,3,7,-PeCDD	25	25
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	25	25
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	30	30
$^{13}C_{12}$-OCDD	30	30
$^{13}C_{12}$-2,3,7,8-TCDF	30	30
$^{13}C_{12}$-1,2,3,7,8-PeCDF	30	30
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	30	30
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	30	30
Surrogate standards:		
$^{37}Cl_4$-2,3,7,8-TCDD	25	25
$^{13}C_{12}$-2,3,4,7,8-PeCDF	25	25
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	25	25
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	25	25
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	25	25
Alternate Standard:		
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	25	25

* * * * *

Method 26—Determination of Hydrogen Chloride Emissions From Stationary Sources

1. Applicability, Principle, Interferences, Precision, Bias, and Stability

1.1 Applicability. This method is applicable for determining hydrogen chloride (HCl) emissions from stationary sources.

1.2 Principle. An integrated sample is extracted from the stack and passed through dilute sulfuric acid. In the dilute acid, the HCl gas is dissolved and forms chloride (Cl^-) ions. The Cl^- is analyzed by ion chromatography (IC).

1.3 Interferences. Volatile materials which produce chloride ions upon dissolution during sampling are obvious interferences. Another likely interferent is diatomic chlorine (Cl_2) gas which reacts to form HCl and hypochlorous acid (HOCl) upon dissolving in water. However, Cl_2 gas exhibits a low solubility in water and the use of acidic, rather than neutral or basic collection solutions, greatly reduces the chance of dissolving any chlorine present. This method does not experience a significant bias when sampling a 400 ppm HCl gas stream containing 50 ppm Cl_2. Sampling a 220 ppm HCl gas stream containing 180 ppm Cl_2 results in a positive bias of 3.4 percent in the HCl measurement.

1.4 Precision and Bias. The within-laboratory relative standard deviations are 6.2 and 3.2 percent at HCl concentrations of 3.9 and 15.3 ppm, respectively. The method does not exhibit a bias to Cl_2 when sampling at concentrations less than 50 ppm.

1.5 Stability. The collected samples can be stored for up to 4 weeks before analysis.

1.6 Detection Limit. The analytical detection limit of the method is 0.1 μg/ml.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 26-1, and component parts are discussed below.

2.1.1 Probe. Borosilicate glass, approximately ⅜-in. (9-mm) I.D. with a heating system to prevent moisture condensation. A ⅜-in. I.D. Teflon elbow should be attached to the inlet of the probe and a 1-in. (25-mm) length of ⅜-in. I.D. Teflon tubing should be attached to the open end of the elbow to permit the opening of the probe to be turned away from the gas stream. This reduces the amount of particulate entering the train. This probe configuration should be used when the concentration of particulate matter in the emissions is high. When high concentrations are not present, the Teflon elbow is not necessary, and the probe inlet may be perpendicular to the gas stream. A glass wool plug should *not* be used to remove particulate matter since a negative bias in the data could result. Instead, a Teflon filter (see § 2.1.5) should be installed at the inlet (for stack temperatures <300 °F) or outlet (for stack temperatures >300 °F) of the probe.

2.1.2 Three-way Stopcock. A borosilicate, three-way glass stopcock with a heating system to prevent moisture condensation. The heated stopcock should connect directly to the outlet of the probe and the inlet of the first impinger. The heating system should be capable of preventing condensation up to the inlet of the first impinger. Silicone grease may be used, if necessary, to prevent leakage.

2.1.3 Impingers. Four 30-ml midget impingers with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage.

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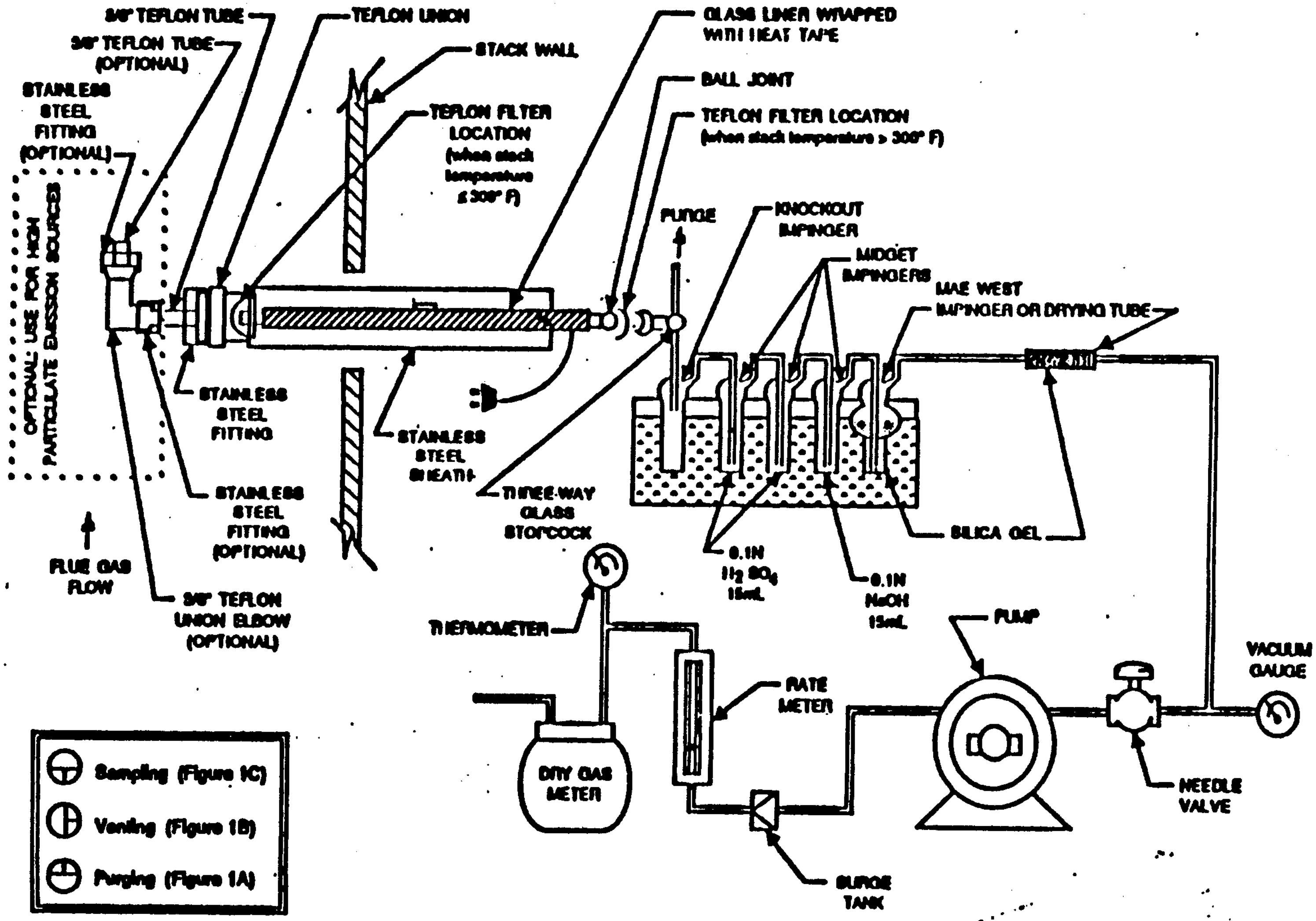

Figure 26-1. Sampling Train

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For sampling at high moisture sources or for sampling times greater than one hour, a midget impinger with a shortened stem (such that the gas sample does not bubble through the collected condensate) should be used in front of the first impinger.

2.1.4 Drying Tube or Impinger. Tube or impinger, of Mae West design, filled with 6- to 16-mesh indicating type silica gel, or equivalent, to dry the gas sample and to protect the dry gas meter and pump. If the silica gel has been used previously, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used.

2.1.5 Filter. A 25-mm Teflon mat, Pallflex TX40HI75 or equivalent. Locate between the probe liner and Teflon elbow in a glass or quartz filter holder in a filter box heated to 250 °F.

2.1.6 Sample Line. Leak-free, with compatible fittings to connect the last impinger to the needle valve.

2.1.7 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of 2 liters/min.

2.1.8 Purge Pump, Purge Line, Drying Tube, Needle Valve, and Rate Meter. Pump capable of purging the sampling probe at 2 liters/min, with drying tube, filled with silica gel or equivalent, to protect pump, and a rate meter capable of measuring 0 to 5 liters/min.

2.1.9 Stopcock Grease, Valve, Pump, Volume Meter, Barometer, and Vacuum Gauge. Same as in Method 6, §§ 2.1.4, 2.1.7, 2.1.8, 2.1.10, 2.1.11, and 2.1.12.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500-ml or larger, two.

2.2.2 Storage Bottles. 100-ml glass, with Teflon-lined lids, to store impinger samples (two per sampling run). During clean-up, the two front impinger contents (0.1 N H_2SO_4) should be combined. The contents of the two rear impingers (0.1 N NaOH) may be discarded, as these solutions are included only to absorb Cl_2, and thus protect the pump.

2.3 Sample Preparation and Analysis. The materials required for volumetric dilution and chromatographic analysis of samples are described below.

2.3.1 Volumetric Flasks. Class A, 100-ml size.

2.3.2 Volumetric Pipets, Class A, assortment. To dilute samples into the calibration range of the instrument.

2.3.3 Ion Chromatograph. Suppressed or non-suppressed, with a conductivity detector and electronic integrator operating in the peak area mode. Other detectors, strip chart recorders, and peak height measurements may be used provided the 5 percent repeatability criteria for sample analysis and the linearity criteria for the calibration curve can be met.

3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS reagent grade). When such specifications are not available, the best available grade shall be used.

3.1 Sampling.

3.1.1 Water. Deionized, distilled water that conforms to ASTM Specification D 1193-77, Type 3.

3.1.2 Absorbing Solution, 0.1 N Sulfuric Acid (H_2SO_4). To prepare 100 ml of the absorbing solution for the front impinger pair, slowly add 0.28 ml of concentrated H_2SO_4 to about 90 ml of water while stirring, and adjust the final volume to 100 ml using additional water. Shake well to mix the solution.

3.1.3 Chlorine Scrubber Solution, 0.1 N Sodium Hydroxide (NaOH). To prepare 100 ml of the scrubber solution for the back pair of impingers, dissolve 0.40 g of solid NaOH in about 90 ml of water, and adjust the final solution volume to 100 ml using additional water. Shake well to mix the solution.

3.2 Sample Preparation and Analysis.

3.2.1 Water. Same as in Section 3.1.1.

3.2.2 Blank Solution. A separate blank solution of the absorbing reagent should be prepared for analysis with the field samples. Dilute 30 ml of absorbing solution to 100 ml with water in a separate volumetric flask.

3.2.3 Sodium Chloride (NaCl) Stock Standard Solution. Solutions containing a nominal certified concentration of 1000 mg/l are commercially available as convenient stock solutions from which working standards can be made by appropriate volumetric dilution. Alternately, concentrated stock solutions may be produced from reagent grade NaCl. The NaCl should be dried at 110 °C for 2 or more hours and cooled to room temperature in a desiccator immediately before weighing. Accurately weigh 1.6 to 1.7 g of the dried NaCl to within 0.1 mg, dissolve in water, and dilute to 1 liter. The exact Cl^- concentration can be calculated using Eq. 26-1.

$$\mu g\ Cl^-/ml = g\ of\ NaCl \times 10^3 \times 35.453/58.44$$

Eq. 26-1

Refrigerate the stock standard solution and store no longer than one month.

3.2.4 Chromatographic Eluent. Effective eluents for non-suppressed IC using a resin- or silica-based weak ion exchange column are a 4 mm potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mm 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH. An effective eluent for suppressed ion chromatography is a solution containing 3 mm sodium bicarbonate and 2.4 mm sodium carbonate. Other dilute solutions buffered to a similar pH and containing no interfering ions may be used. When using suppressed ion chromatography, if the "wafer dip" resulting from sample injection interferes with the chloride peak, use a 2 mm NaOH/2.4 mm sodium bicarbonate eluent.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection Train. Prepare the sampling train as follows: Pour 15 ml of the absorbing solution into each of the first two impingers, and add 15 ml of scrubber solution to the third and fourth impingers. Connect the impingers in series with the knockout impinger first, followed by the two impingers containing absorbing solution and the two containing the scrubber solution. Place a fresh charge of silica gel, or equivalent, in the drying tube or Mae West impinger.

4.1.2 Leak-Check Procedures. Leak-check the probe and three-way stopcock before inserting the probe into the stack. Connect the stopcock to the outlet of the probe, and connect the sample line to the needle valve. Plug the probe inlet, turn on the sample pump, and pull a vacuum of at least 250 mm Hg (10 in. Hg). Turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Place the probe in the stack at the sampling location, and adjust the probe and stopcock heating system to a temperature sufficient to prevent water condensation. Connect the first impinger to the stopcock, and connect the sample line to the last impinger and the needle valve. Upon completion of a sampling run, remove the probe from the stack and leak-check as described above. If a leak has occurred, the sampling run must be voided. Alternately, the portion of the train behind the probe may be leak-checked between multiple runs at the same site as follows: Close the stopcock to the first impinger (see Figure 1A of Figure 26-1), and turn on the sampling pump. Pull a vacuum of at least 250 mm Hg, turn off the needle valve, and note the vacuum gauge reading. The vacuum should remain stable for at least 30 seconds. Release the vacuum on the impinger train by turning the stopcock to the vent position to permit ambient air to enter (see Figure 1B of Figure 26-2). If this procedure is used, the full train leak-check described above must be conducted following the final run, and all preceding sampling runs must be voided if a leak has occurred.

4.1.3 Purge Procedure. Immediately before sampling, connect the purge line to the stopcock, and turn the stopcock to permit the purge pump to purge the probe (see Figure 1A of Figure 26-1). Turn on the purge pump, and adjust the purge rate to 2 liters/min. Purge for at least 5 minutes before sampling.

4.1.4 Sample Collection. Turn on the sampling pump, pull a slight vacuum of approximately 25 mm Hg (1 in. Hg) on the impinger train, and turn the stopcock to permit stack gas to be pulled through the impinger train (see Figure 1C of Figure 26-3). Adjust the sampling rate to 2 liters/min, as indicated by the rate meter, and maintain this rate to within 10 percent during the entire sampling run. Take readings of the dry gas meter volume and temperature, rate meter, and vacuum gauge at least once every five minutes during the run. A sampling time of one hour is recommended. Shorter sampling times may introduce a significant negative bias in the HCl concentration. At the conclusion of the sampling run, remove the train from the stack, cool, and perform a leak-check as described in Section 4.1.2.

4.2 Sample Recovery. Disconnect the impingers after sampling. Quantitatively transfer the contents of the first three impingers (the knockout impinger and the two absorbing solution impingers) to a leak-free storage bottle. Add the water rinses of each of these impingers and connecting glassware to the storage bottle. The contents of the scrubber impingers and connecting glassware rinses may be discarded. The

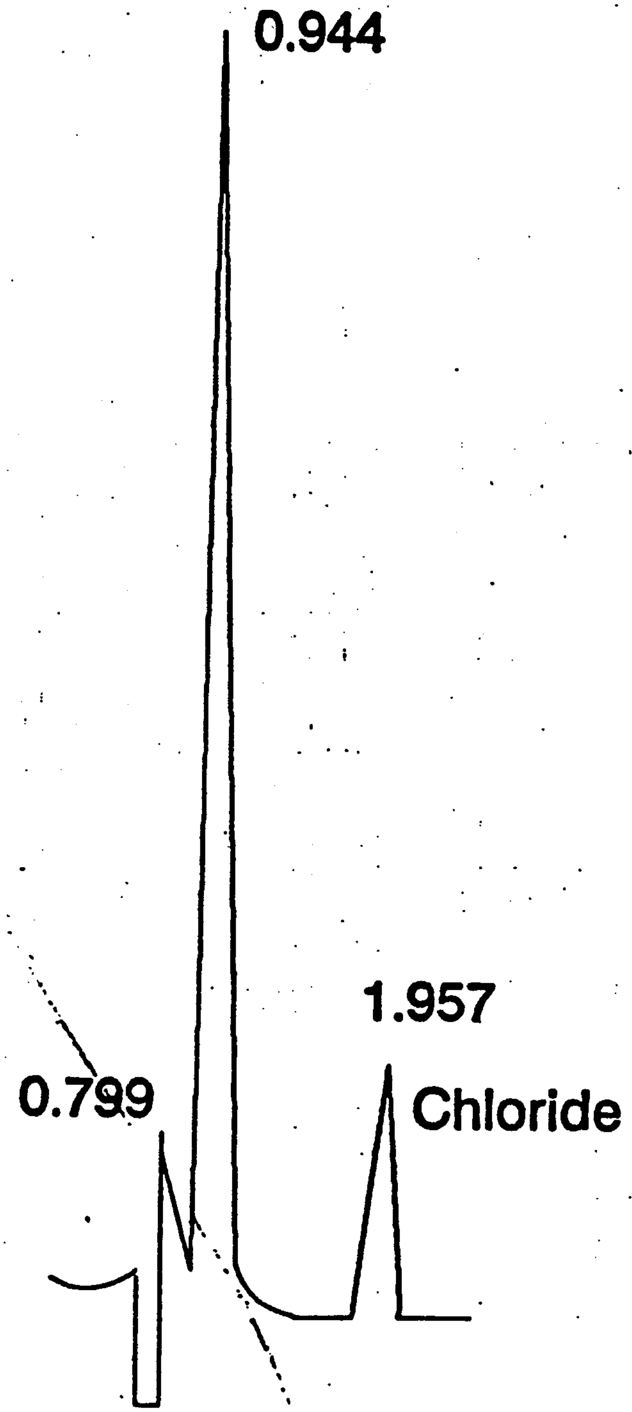

Figure 26-2. Example Chromatogram

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sample bottle should be sealed, shaken to mix, and labeled. The fluid level should be marked so that if any sample is lost during transport, a correction proportional to the lost volume can be applied.

4.3 Sample Preparation for Analysis. Check the liquid level in each sample, and determine if any sample was lost during shipment. If a noticeable amount of leakage has occurred, the volume lost can be determined from the difference between the initial and final solution levels, and this value can be used to correct the analytical results. Quantitatively transfer the sample solution to a 100-ml volumetric flask, and dilute the solution to 100 ml with water.

4.4 Sample Analysis.

4.4.1 The IC conditions will depend upon analytical column type and whether suppressed or non-suppressed IC is used. An example chromatogram from a non-suppressed system using a 150-mm Hamilton PRP-X100 anion column, a 2 ml/min flow rate of a 4 mM 4-hydroxy benzoate solution adjusted to a pH of 8.6 using 1 N NaOH, a 50 µl sample loop, and a conductivity detector set on 1.0 µS full scale is shown in Figure 26-2.

BILLING CODE 6560-50-M

4.4.2 Before sample analysis, establish a stable baseline. Next, inject a sample of water, and determine if any Cl^- appears in the chromatogram. If Cl^- is present, repeat the load/injection procedure until no Cl^- is present. At this point, the instrument is ready for use.

4.4.3 First, inject the calibration standards covering an appropriate concentration range, starting with the lowest concentration standard. Next, inject in duplicate, a QC sample followed by a water blank and the field samples. Finally, repeat the injection of calibration standards to allow compensation for any drift in the instrument during analysis of the field samples. Measure the Cl^- peak areas or heights of the samples. Use the average response from the duplicate injections to determine the field sample concentrations using a linear calibration curve generated from the standards.

4.5 Audit Analysis. There is currently no validated audit sample for this method. It is recommended to analyze a QC sample along with the field samples as described above.

5. Calibration

5.1 Dry Gas Metering System, Thermometers, Rate Meter, and Barometer. Same as in Method 6, sections 5.1, 5.2, 5.3, and 5.4.

5.2 Calibration Curve for Ion Chromatograph. To prepare calibration standards, dilute given volumes (1.0 ml or greater) of the stock standard solution, with 0.1 N H_2SO_4 (section 3.1.2) to convenient volumes. Prepare at least four standards that are within the linear range of the instrument and which cover the expected concentration range of the field samples. Analyze the standards as instructed in section 4.4.3, beginning with the lowest concentration standard. Determine the peak measurements, and plot individual values versus Cl^- concentration in μg/ml. Draw a smooth curve through the points. Use linear regression to calculate a formula describing the resulting linear curve.

6. Quality Assurance

6.1 Applicability. When the method is used to analyze samples to demonstrate compliance with a source emission regulation, a set of two audit samples must be analyzed.

6.2 Audit Procedure. The audit samples are chloride solutions. Concurrently analyze the two audit samples and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit samples. If this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

6.3 Audit Sample Availability. The audit samples may be obtained by writing or calling the EPA regional office or the appropriate enforcement agency. The request for the audit samples must be made at least 30 days prior to the scheduled compliance sample analyses.

6.4 Audit Results. Calculate the concentrations in mg/dscm using the specified sample volume in the audit instructions.

Note: Indication of acceptable results may be obtained immediately by reporting the audit results in mg/dscm and compliance results in total μg HCl/sample to the responsible enforcement agency.

Include the results of both audit samples, their identification numbers, and the analyst's name with the results of the compliance determination samples in appropriate reports to the EPA regional office or the appropriate enforcement agency. Include this information with subsequent analyses for the same enforcement agency during the 30-day period.

The concentrations of the audit samples obtained by the analyst shall agree within 10 percent of the actual concentrations. If the 10 percent specification is not met, reanalyze the compliance samples and audit samples, and include initial and reanalysis values in the test report.

Failure to meet the 10 percent specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance status of the affected facility.

7. Calculations

Retain at least one extra decimal figure beyond those contained in the available data in intermediate calculations, and round off only the final answer appropriately.

7.1 Sample Volume, Dry Basis, Corrected to Standard Conditions. Calculate the sample volume using Eq. 6-1 of Method 6.

7.2 Total μg HCl Per Sample.

$$m=(S-B)(100)(36.46)/(35.453)=(102.84)(S-B) \qquad \text{Eq. 26-2}$$

Where:

m = Mass of HCl in sample, μg.
S = Concentration of sample, μg Cl^-/ml.
B = Concentration of blank, μg Cl^-/ml.
100 = Volume of filtered and diluted sample, ml.
36.46 = Molecular weight of HCl, μg/μg-mole.
35.453 = Atomic weight of Cl, μg/μg-mole.

7.3 Concentration of HCl in the Flue Gas.

$$C=K\,m/V_{m(std)} \qquad \text{Eq. 26-3}$$

Where:

C = Concentration of HCl, dry basis, mg/dscm.
$K=10^{-3}$ mg/μg.
m = Mass of HCl in sample, μg.
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm.

8. Bibliography

1. Steinsberger, S.C. and J.H. Margeson. "Laboratory and Field Evaluation of a Methodology for Determination of Hydrogen Chloride Emissions from Municipal and Hazardous Waste Incinerators." U.S. Environmental Protection Agency, Office of Research and Development. Report No. 600/3-89/064, April 1989.

2. State of California, Air Resources Board. Method 421, "Determination of Hydrochloric Acid Emissions from Stationary Sources." March 18, 1987.

3. Entropy Environmentalists Inc., "Laboratory Evaluation of a Sampling and Analysis Method for Hydrogen Chloride Emissions from Stationary Sources: Interim Report." EPA Contract No. 68-02-4442, Research Triangle Park, North Carolina, January 22, 1988.

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[FR Doc. 89-28723 Filed 12-19-89; 8:45 am]

BILLING CODE 6560-50-M

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40 CFR Part 60

[AD-FRL-3683-6]

Standards of Performance for new Stationary Sources; Revision of Procedure 1 of Appendix F and Addition of Performance Specification 4A to Appendix B

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule and notice of public hearing.

SUMMARY: The purpose of this proposed rule is to revise Procedure 1 of Appendix F to make it applicable without further revision for all regulated pollutants, as was intended originally. In addition, Performance Specification 4A is being added to Appendix B to allow the performance testing of carbon monoxide continuous emissions monitoring systems (CEMS) at municipal wastes combustion facilities.

A public hearing will be held, if requested, to provide interested persons an opportunity for oral presentation of data, views, or arguments concerning the proposed rule.

DATES: *Comments*. Comments must be received on or before March 12, 1990.

Public Hearing. If anyone contacts EPA requesting to speak at a public hearing by January 16, 1990, a public hearing will be held February 5, 1990 beginning at 10:00 a.m. Persons interested in attending the hearing should call the contact mentioned under ADDRESSES to verify that a hearing will be held.

Request to Speak at Hearing. Persons wishing to present oral testimony must contact EPA by January 16, 1990.

Appendix K: Calibration Equations

CALCULATION EQUATIONS

METHOD 2

$$\overline{V}_s = 85.48\ C_p\ (\sqrt{\Delta p})_{avg} \sqrt{\frac{T_{s(avg)}}{P_s\ M_s}}$$

$$Q_{s,d} = 60(1 - B_{ws})\ \overline{V}_s\ A\ \left(\frac{528}{T_{s(avg)}}\right)\ \left(\frac{P_s}{29.92}\right)$$

$$Q_a = 60\ \overline{V}_s\ A$$

$$\dot{m}_g = \frac{4.995\ Q_{s,d}\ G_d}{1 - B_{ws}}$$

$$RH^* = 100\ (vp_{twb}\ 0.0003641\ P_s\ (T_{db} - T_{wb}))/vp_{tdb}$$

$$B^*_{ws} = RH(vp_{tdb})/P_s$$

$$= \frac{4.585 \times 10^{-2}\ P_s M_s}{T_s\ (avg)}$$

*Alternate equations for calculating moisture content from wet bulb and dry buld data.

SYMBOLS

A = Cross sectional area of stack, SQ. FT.

A_n = Cross sectional area of nozzle, SQ. FT.

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

C_p = Pitot tube coefficient, dimensionless

C_a = Concentration of particulate matter in stack gas, wet basis, GR/ACF

C_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, GR/DSCF

EA = Excess air, percent by volume

Y = Dry test meter correction factor, dimensionless

G_d = Specific gravity (relative to air), dimensionless

I = Isokinetic variation, percent by volume

M_d = Molecular weight of stack gas, dry basis, g/g - mole.

$\dot{m}_g$ = Mass flow of wet flue gas, LB/HR

$\dot{m}_p$ = Particulate mass flow, LB/HR

M_s = Molecular weight of stack gas, wet basis, g/g, mole.

M_p = Total amount of particulate matter collected, g

P_{bar} = Atmospheric pressure, IN. HG. (uncompensated)

P_g = Stack static gas pressure, IN. WC.

P_s = Absolute pressure of stack gas, IN.HG.

P_{std} = Standard absolute pressure, 29.92 IN. HG.

A_a = Actual volumetric stack gas flow rate, ACFM

$Q_{s,d}$ = Dry volumetric stack gas flow rate corrected to standard conditions, DSCFM

RH = Relative humidity, %

T_{db} = Dry bulb temperature of stack gas, °F

T_{wb} = Wet bulb temperature of stack gas, °F

$T_{m(avg)}$ = Absolute average dry gas meter temperature, °R

$T_s(avg)$ = Absolute average stack temperature, °F

T_{std} = Standard absolute temperature, 528 °F (68 °F)

θ = Total sampling time, min.

V_{lc} = Total volume of liquid collected in impingers and silica gel, ml

V_m = Volume of gas sample as measured by dry gas meter, CF

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, DSCF

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, SCF

$\overline{V}_s$ = Average actual stack gas velocity, FT/SEC

vP_{tdb} = Vapor pressure at T_{db}, IN. HG.

vp_{twb} = Vapor pressure at T_{wb}, IN. HG

$\overline{\Delta H}$ = Average pressure differential across the orifice meter, IN. WC.

ΔP = Velocity pressure of stack gas, IN. WC.

γ = Dry test meter correction coefficient, dimensionless

ρ = Actual gas density, LB/ACF

CALCULATION EQUATIONS

METHOD 3

$$\%EA = \frac{100(\%O_2 -).5\%\ CO)}{0.264\%\ N_2 - \%O_2 + 0.5\%\ CO}$$

$$M_d = 0.44(\%CO_2) + 0.32\ (\%O_2) + 0.28\ (\%N_2 + \%CO)$$

$$M_s = M_d\ (I - B_{ws}) + 0.18\ B_{ws}$$

$$B_{ws} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}}$$

CALCULATION EQUATIONS

METHOD 5

$$V_{m(std)} = 17.65\ V_m\ \gamma\ \left(\frac{P_{bar} + \overline{\Delta H}/13.6}{T_{m(avg)}}\right)$$

$$V_{w(std)} = 0.0472\ V_{Is}$$

$$B_{ws} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}}$$

$$I = 0.0944\ \left(\frac{T_{s(avg)}\ V_{m(std)}}{P_s\ V_s\ A_n\ \theta\ (1 - B_{ws})}\right)$$

$$C_s = \frac{15.43\ M_p}{V_{m(std)}}$$

$$C_a = \frac{272.3\ M_p\ P_s}{T_{s(avg)}\ (V_{w(std)} + V_{m(std)})}$$

$$(\dot{m}_p)_1 = 8.5714 \times 10^{-3}\ C_s\ Q_{s,d}$$

$$(\dot{m}_p)_2 = \frac{1.3228 \times 10^{-1}\ M_p\ \ A}{0\ A_n}$$

$$\dot{m}_p = \frac{(\dot{m}_p)_1 + (\dot{m}_p)_2}{2}$$

SYMBOLS

A = Cross sectional area of stack, SQ. FT.

A_n = Cross sectional area of nozzle, SQ. FT.

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

C_p = Pitot tube coefficient, dimensionless

C_a = Concentration of particulate matter in stack gas, wet basis, GR/ACF

C_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, GR/DSCF

EA = Excess air, percent by volume

γ = Dry test meter correction factor, dimensionless

G_d = Specific gravity (relative to air), dimensionless

I = Isokinetic variation, percent by volume

M_d = Molecular weight of stack gas, dry basis, g/g --mole.

$\dot{m}_g$ = Mass flow of wet flue gas, LB/HR

$\dot{m}_p$ = Particulate mass flow, LB/HR

M_s = Molecular weight of stack gas, wet basis, g/g, mole.

M_p = Total amount of particulate matter collected, g

P_{bar} = Atmospheric pressure, IN. HG. (uncompensated)

P_g = Stack static gas pressure, IN. WC.

P_s = Absolute pressure of stack gas, IN.HG.

P_{std} = Standard absolute pressure, 29.92 IN. HG.

A_a = Actual volumetric stack gas flow rate, ACFM

$Q_{s,d}$ = Dry volumetric stack gas flow rate corrected to standard conditions, DSCFM

RH = Relative humidity, %

T_{db} = Dry bulb temperature of stack gas, ^{o}F

T_{wb} = Wet bulb temperature of stack gas, ^{o}F

$T_{m(avg)}$ = Absolute average dry gas meter temperature, ^{o}R

$T_s(avg)$ = Absolute average stack temperature, ^{o}F

T_{std} = Standard absolute temperature, 528 ^{o}F (68 ^{o}F)

θ = Total sampling time, min.

V_{lc} = Total volume of liquid collected in impingers and silica gel, ml

V_m = Volume of gas sample as measured by dry gas meter, CF

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, DSCF

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, SCF

$\overline{V}_s$ = Average actual stack gas velocity, FT/SEC

vP_{tdb} = Vapor pressure at T_{db}, IN. HG.

vp_{twb} = Vapor pressure at T_{wb}, IN. HG

$\overline{\Delta H}$ = Average pressure differential across the orifice meter, IN. WC.

ΔP = Velocity pressure of stack gas, IN. WC.

γ = Dry test meter correction coefficient, dimensionless

ρ = Actual gas density, LB/ACF

CALCULATION EQUATIONS

Calculation Equations for Gas Concentrations

Dry Standard Volume (V_{std})

$$V_{std} = \frac{17.65\ V_m \gamma P_b}{(\bar{t}_m + 460)}$$

where; V_m = volume of gas as recorded on dry test meter in CF

γ = correction coefficient of dry test meter derived from calibration against standard wet test meter, dimensionless

P_b = barometric pressure in IN. HG.

$\bar{t}_m$ = average temperature of gas in dry test meter, in °F

Gas Concentration of Component i (C_i)

$$C_i = \frac{.03531\ m_i K_i}{V_{std}}$$

where; C_i = concentration of gas in MG/DSCM

m_i = total amount of corresponding ion in the collected samples as reported by laboratory in µg.

K_i = stoichiometric conversion from ion to gas for component i ($K_{i=HF}$ = 1.05306, $K_{i=HCl}$ = 1.02843, $K_{i=H_2SO_4}$ = 1.02099, $K_{i=NH_3}$ = 0.94412)

Gas Concentration in GR/DSCF

$$GR/DSCF = 4.3705 \times 10^{-4}\ C_i$$

Gas Concentration in PPM-DRY

$$PPM\text{-}DRY = \frac{24.04\ C_i}{M_i}$$

where: M_i = molecular weight of component or gas i
($M_{i=HF}$ = 20.006, $M_{i=HCl}$ = 36.461, $M_{i=H_2SO_4}$ = 98.076, $M_{i=NH_3}$ = 17.031)

Gas Emission or Mass Rate ($\dot{m}_i$)

$$\dot{m}_i = 8.5714 \times 10^{-3}\ (GR/DSCF)(Q_{s,d})$$

where: $\dot{m}_i$ = mass or emission rate of gas i in LB/HR

GR/DSCF = concentration of gas i in GR/DSCF, and

$Q_{s,d}$ = dry standard volumetric flow rate at point of concentration measurement in DSCFM

Method 25A

Total Gaseous Organics Calculation Equations

$$GR\ C/SCF = 2.180 \times 10^{-4}\ (ppm,w)$$

$$GR\ C/DSCF = 2.180 \times 10^{-4}\ (ppm,w)/(1-MC/100)$$

$$LB\ C/HR = 8.5714 \times 10^{-3}\ (GR/DSCF)\ (DSCFM)$$

where:

GR C/SCF = grains of total gaseous organics as carbon per actual (wet) standard cubic foot

GR C/DSCF = grains of total gaseous organics as carbon per dry standard cubic foot

LB C/HR = pounds of total gaseous organics as carbon emitted hour

Note 1: The Ratfisch Model RS 55 Heated FID Analyzer as normally operated with a heated filter, sample line and heated detector oven gives ppm,w.

Note 2: ppm,C = ppm as carbon = 3(ppm propane)

CALCULATION OF PAH EMISSION RATES

in g/sec and 10^{-6}g/sec

$$\dot{m}\ (\text{g/sec}) = 4.716 \times 10^{-10}\ C_s(\text{ug/m}^3)\ Q_{s,d}(\text{DSCFM})$$

$$\dot{m}\ (10^{-3}\text{g/sec}) = 4.716 \times 10^{-7}\ \text{Ca}(\text{ug/m}^3)\ Q_{s,d}(\text{DSCFM})$$

$$\dot{m}\ (10^{-6}\text{g/sec}) = 4.716 \times 10^{-4}\ C_s(\text{ug/m}^3)\ Q_{s.d}(\text{DSCFM})$$

where:

Q_s = ug/dsm^3

$Q_{s.d}$ = DSCF/MIN

CALCULATION OF DIOXIN EMISSION RATES

in g/sec and 10^{-8}g/sec

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

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

where:

Q_s = ng/dsm^3

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

CALCULATIONS FOR METALS

1. CONCENTRATION (ug/Nm^3)

$$\text{ug/Nm}^3 = \frac{35.31\ \text{ft}^3}{\text{m}^3} \times \frac{\text{ug in sample}}{V_m\ (\text{DSCF})}$$

where V_m = Exhaust gas volume through meter (DSCF)

2. EMISSION RATE (lb/hr)

$$(\text{lb/hr}) = \frac{\text{ug}}{\text{Nm}^3} \times \frac{\text{lb}}{454\text{g}} \times \frac{\text{g}}{1000\text{mg}} \times \frac{\text{mg}}{1000\text{ug}} \times \frac{\text{DSCF}}{\text{min}} \times \frac{60\ \text{min}}{\text{hr}} \times \frac{0.0283\ \text{Nm}^3}{\text{DSCF}}$$

$$(\text{lb/hr}) = (3.74 \times 10^{-9}) \times \text{DSCFM} \times \frac{\text{ug}}{\text{Nm}^3}$$

where DSCFM = Volumetric flow rate in the source

Appendix L: Sampling Train Calibration Data

INTERPOLL LABORATORIES

EPA Method 5 Gas Metering System
Quality Control Check Data Sheet

Job ANL/OTP B.q Stone Date 10/26/92

Operator D. Van Hoever Module No. 3

Instructions: Operate the control module at a flow rate equal to ^H@ for 10 minutes before attaching the umbilical. Record the following data:

Bar press 28.71 in. Hg. T = .9984 ^H@ 1.81 in. W.C.

Time (min)	Volume (CF)	Meter Temp. (°F)	
		Inlet	Outlet
	(342.80)		
2.5	344.71	65	58
5.0	346.62	66	59
7.5	348.53	68	60
10	350.44	69	60
	Vm= 7.64	Avg(tm)= 63.125 °F	

Calculate Y_{cn} as follows:

$$Y_{cn} = \frac{1.786}{T\ V_m}\left[\frac{(t_m + 460)}{P_b}\right]^{0.5}$$

$$Y_{cn} = \frac{1.786}{(.9984)(7.64)}\left[\frac{(63.13) + 460}{(28.71)}\right]^{0.5}$$

Y_{cn} = .9995

If Y_{cn} is not within the range of 0.97 to 1.03, "the volume metering system should be investigated before beginning."

CFR Title 40, Part 60, Appendix A, Method 5, Section 4.4.1

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Interpoll Laboratories, Inc.
(612) 786-6060

EPA Method 5 Gas Metering System
Quality Control Check Data Sheet

Job ANL / ETP – Big STONE Date 10-26-92

Operator R.R. Module No. 3

Instructions: Operate the control module at a flow rate equal to $\Delta H@$ for 10 minutes before attaching the umbilical. Record the following data:

Bar press 28.71 in. Hg. γ = .9984 $\Delta H@$ 1.81 in. W.C.

Time (min)	Volume (CF)	Meter Temp. (°F)	
		Inlet	Outlet
[redacted]	674.90	[redacted]	
2.5	676.86	73	72
5.0	678.85	75	73
7.5	680.79	77	73
10	682.74	78	74
[redacted]	V_m = 7.84	Avg(t_m) = 74.38 °F	

Calculate Y_{cm} as follows:

$$Y_{cm} = \frac{1.786}{\gamma\ V_m}\left[\frac{(t_m + 460)}{P_b}\right]^{0.5}$$

4314

$$Y_{cm} = \frac{1.786}{(\quad)(\quad)}\left[\frac{(\quad) + 460}{(\quad)}\right]^{0.5}$$

Y_{cm} = .9840

If Y_{cm} is not within the range of 0.97 to 1.03, "the volume metering system should be investigated before beginning."

CFR Title 40, Part 60, Appendix A, Method 5, Section 4.4.1

INTERPOLL LABORATORIES
EPA Method 5 Gas Metering System
Quality Control Check Data Sheet

Job ANL/OTR-Bigstone Date 10-26-92

Operator R. Rosenthal Module No. 7

Instructions: Operate the control module at a flow rate equal to ^H@ for 10 minutes before attaching the umbilical. Record the following data:

Bar press 28.71 in. Hg. T = 1.0001 ^H@ 1.77 in. W.C.

Time (min)	Volume (CF)	Meter Temp. (°F)	
		Inlet	Outlet
	(769.10)		
2.5	771.02	57	53
5.0	772.91	59	54
7.5	774.83	60	54
10	776.77	62	55
	V_m= 7.67	Avg(t_m)= 56.75°F	

Calculate Y_{cn} as follows:

$$Y_{cn} = \frac{1.786}{T\ V_m}\left[\frac{(t_m + 460)}{P_b}\right]^{0.5}$$

7.67 4.243

$$Y_{cn} = \frac{1.786}{(\quad)(\quad)}\left[\frac{(\quad) + 460}{(\quad)}\right]^{0.5}$$

Y_{cn} = .9880

If Y_{cn} is not within the range of 0.97 to 1.03, "the volume metering system should be investigated before beginning."

CFR Title 40, Part 60, Appendix A, Method 5, Section 4.4.1

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INTERPOLL LABORATORIES
EPA Method 5 Gas Metering System
Quality Control Check Data Sheet

Job ANL / OTP - Bigstone

Date 10-27-92

Operator R. R.

Module No. 9

Instructions: Operate the control module at a flow rate equal to ^H@ for 10 minutes before attaching the umbilical. Record the following data:

Bar press 28.71 in. Hg. T = 1.0001 ^H@ 1.77 in. W.C.

Time (min)	Volume (CF)	Meter Temp. (°F)	
		Inlet	Outlet
	(86.60)		
2.5	88.54	73	72
5.0	90.52	74	72
7.5		75	72
10	94.43	76	73
	V_m= 7.83	Avg(t_m)=	73.38 °F

Calculate Y_{cn} as follows:

$$Y_{cn} = \frac{1.786}{T\ V_m}\left[\frac{(t_m + 460)}{P_b}\right]^{0.5}$$

2.281

$$Y_{cn} = \frac{1.786}{(\quad)(\quad)}\left[\frac{(\quad) + 460}{(\quad)}\right]^{0.5}$$

Y_{cn} = .9832

If Y_{cn} is not within the range of 0.97 to 1.03, "the volume metering system should be investigated before beginning."

CFR Title 40, Part 60, Appendix A, Method 5, Section 4.4.1

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Interpoll Laboratories, Inc.
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: October 30, 1992

Meter Box No. : 3 (Rockwell Dry Test Meter Serial No. 712852)

Date of Last Calibration: October 2, 1992
Calibration Technician: D. Brennan
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.)
October 14, 1992	2-7191	852.30	1020.36	168.06	168.06
October 21, 1992	2-7249	1022.00	1341.62	319.62	487.68
October 26, 1992	2-7328	1342.80	1927.40	584.60	1072.28

* Total volume through meter since last calibration.

Interpoll Laboratories, Inc.
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: October 30, 1992

Meter Box No. : 4 (Rockwell Dry Test Meter Serial No. 964553)

Date of Last Calibration: September 23, 1992
Calibration Technician: D. Brennan
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.)
September 30, 1992	2-3652	944.00	1448.85	504.85	504.85
October 20, 1992	2-7250	1450.20	1817.67	367.47	872.32
October 26, 1992	2-7328	1820.70	2159.45	388.75	1211.07

* Total volume through meter since last calibration.

Interpoll Laboratories, Inc.
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: October 30, 1992

Meter Box No. : 9 (Rockwell Dry Test Meter Serial No. 949231)

Date of Last Calibration: September 17, 1992
Calibration Technician: D. Brennan
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.)
September 22, 1992	2-3644	975.60	1497.45	521.85	521.85
October 7, 1992	2-3650	1499.70	1644.20	144.50	666.35
October 14, 1992	2-7191	1644.44	1767.32	122.88	789.23
October 26, 1992	2-7328	1769.10	2404.38	635.28	1424.51

* Total volume through meter since last calibration.

Interpoll Laboratories, Inc.
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: November 2, 1992

Meter Box No. : 2-S (Rockwell Dry Test Meter Serial No. 69185)

Date of Last Calibration: August 19, 1992
Calibration Technician: D. Van Hoever
Wet Test Meter No.: American Meter AL-17 (0.05 CF/REV)

Date of Use	Report No.	Initial Meter Reading	Final Meter Reading	Volume/Job (cu. ft.)	Total Volume* (cu. ft.)
September 21, 1992	2-3642	72.800	77.497	4.697	4.697
October 13, 1992	2-7190	94.265	101.825	7.560	12.257
October 21, 1992	2-7249	101.950	108.475	6.575	18.832
October 26, 1992	2-7328	108.670	123.184	14.514	33.346

* Total volume through meter since last calibration.

Interpoll Laboratories, Inc.
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: November 2, 1992

Meter Box No. : 5-S (Rockwell Dry Test Meter Serial No. 64711)
Date of Last Calibration: January 21, 1992
Calibration Technician: E. Trowbridge

Wet Test Meter No.: American Meter AL-17 (0.05 CF/REV)

Date of Use	Report No.	Initial Meter Reading	Final Meter Reading	Volume/Job (cu. ft.)	Total Volume* (cu. ft.)
February 4, 1992	2-3498	145.687	153.198	7.511	7.511
May 5, 1992	2-3549	156.140	165.143	9.003	16.514
August 4, 1992	2-3611	168.562	179.777	11.215	27.729
October 14, 1992	2-7189	179.900	188.813	8.913	36.642
October 16, 1992	2-7190	188.995	196.245	7.250	43.892
October 26, 1992	2-7328	196.255	210.155	13.900	57.792

* Total volume through meter since last calibration.

Date 10-2-92

Bar. Press. 28.95 in. Hg

INTERPOLL LABORATORIES, INC.
METER CALIBRATION SHEET
EPA METHOD 5

Control Module No. ________
Serial No. DTM 712 852
Wet Test Meter No. AL-20
Technician D. BRENNAN

ΔH (in. WC)		Gas Volume Wet Test Meter (ft³)	* Cal. Index φ (%)	* Diff. Wet Test Meter ΔP_w (in.WC)	Gas Volume Dry test meter (ft³)		Gas temperatures			Time θ (min/sec)	Meter Coeff. Y	Orifice Const. ΔH@	C_f
							Wet Test	Dry Test					
nominal	actual				V_{di}	V_{df}	t_w (°F)	t_{di} (°F)	t_{do} (°F)				
0.5	.50	2	99.85	0.01	846.500	848.560	66.0	84	76	5.02	.9939	1.78	
1.2	1.2	3	99.91	0.025	849.000	852.077	66.0	87	77	4.50	1.0006	1.74	
2.0	2.0	3	99.93	0.055	832.000	835.070	66.0	85	75	3.49	.9973	1.82	
3.3	3.3	5	100.00	0.09	835.500	840.602	66.0	87	76	5.00	1.0003	1.85	
4.7	4.7	5	100.02	0.12	841.000	846.110	66.0	90	78	4.14	.9999	1.88	
	2	3	4	5	6	7	8	9	10	11/12 Avg	.9984	1.81	

Positive leak check performed by D. BRENNAN
Meter was in tolerance ☒
Meter was not in tolerance ☐**; readjusted linkage**
Meter was not in tolerance ☐**; changed dry test meters**

Approved by Daniel **Date** 10/8/92

* Based on AL-20 wet test meter calibration in Nov. 1991 against Bell Prover (NBS Traceable) - Carl Poe Co.

Date 9/23/92

Bar. Press. 29.30 in. Hg

INTERPOLL LABORATORIES, INC.
METER CALIBRATION SHEET
EPA METHOD 5

Control Module No. 4
Serial No. DTM 964 553
Wet Test Meter No. AL-20
Technician D. BRENNAN

ΔH (in. WC) nominal	ΔH (in. WC) actual	Gas Volume Wet Test Meter (ft³)	* Cal. Index ϕ (%)	* Diff. Wet Test Meter ΔP_w (in.WC)	Gas Volume Dry test meter (ft³) V_{di}	Gas Volume Dry test meter (ft³) 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. ΔH@	C_f
0.6	.50	2	99.85	0.01	936.500	938.545	63.0	77	72	5:12	.9967	1.88	
1.2	1.2	3	99.91	0.025	939.000	942.064	63.0	79	73	5:05	.9947	1.91	
2.0	2.0	3	99.93	0.055	922.000	925.052	63.0	77	71	3.58	.9978	1.94	
3.3	3.3	5	100.00	0.09	925.500	930.563	63.0	80	72	5:13	1.0036	1.98	
4.7	4.7	5	100.02	0.12	931.000	936.043	63.0	82	73	4:23	1.0070	1.99	
	2	3	4	5	6	7	8	9	10	11/12 Avg.	1.0000	1.94	

Positive leak check performed by D. BRENNAN
Meter was in tolerance ☒
Meter was not in tolerance ☐**; readjusted linkage**
Meter was not in tolerance ☐**; changed dry test meters**

Approved by Daniel J. Dey **Date** 9/24/92

✓ replaced orifice (9/23/92)

* Based on AL-20 wet test meter calibration in Nov. 1991 against Bell Prover (NBS Traceable) - Carl Poe C

S0102RR 11/91

Date 9/17/92

Bar. Press. 28.93 in. Hg

INTERPOLL LABORATORIES, INC.
METER CALIBRATION SHEET
EPA METHOD 5

Serial No. DTM 949 231
Wet Test Meter No. AL-20
Technician D. BRENNAN

ΔH (in. WC)		Gas Volume Wet Test Meter (ft³)	* Cal. Index φ (%)	* Diff. Wet Test Meter ΔP_w (in.WC)	Gas Volume Dry test meter (ft³)		Gas temperatures			Time θ (min/sec)	Meter Coeff. Y	Orifice Const. ΔH@	C_f
nominal	actual				V_{di}	V_{df}	Wet Test t_w (°F)	Dry Test t_{di} (°F)	Dry Test t_{do} (°F)				
0.6	.50	2	99.85	0.01	967.000	969.025	66.5	80	74	4.57	1.0045	1.74	
1.2	1.2	3	99.91	0.025	970.000	973.035	66.5	82	75	4.48	1.0069	1.74	
* 2.0	2.0	3	99.93	0.055	951.000	954.047	66.5	79	72	3.44	.9955	1.76	
3.3	3.3	5	100.00	0.09	955.000	960.071	66.5	81	73	4.55	.9971	1.80	
4.7	4.7	5	100.02	0.12	961.000	966.058	66.5	81	75	4.07	1.0009	1.79	
	2	3	4	5	6	7	8	9	10	[illegible] AVG	1.0001	1.77	

Positive leak check performed by D. BRENNAN
Meter was in tolerance ☒
Meter was not in tolerance ☐; readjusted linkage
Meter was not in tolerance ☐; changed dry test meters

Approved by Date Sept 18, 92

* Based on AL-20 wet test meter calibration in Nov. 1991 against Bell Prover (NBS Traceable) - Carl Poe Co.

Date 8/19/92

Bar. Press. 29.22 in. Hg

INTERPOLL LABORATORIES
MINIMODULE CALIBRATION SHEET
Flow Rate Range 0-0.13 CFM
(0-4 LPM)

Meter Box No. 25

Serial No. DTM 69185

Wet Test Meter No. AL-17 (.05 CF/REV)

Technician

					Gas Volume Dry test meter (ft³)		Gas temperatures					
								Dry Test				
Rotameter Reading (cc/min)	ΔP_d (IN/WC)	Gas Volume Wet Test Meter (ft³)	** Cal. Index ϕ (%)	Diff. Wet Test Meter ΔP_w (in.WC)	V_{di}	V_{df}	Wet Test t_w (°F)	t_{di} (°F)		Time θ (min/sec)	Meter Coeff. Y	Rotameter Coeff. Z
1000	.001	.275	99.55	0.04	72055	72.338	61.6	83			.9990	
1000	.001	.15	99.55	0.04	72.388	72.541	61.6	84			1.0098	
1000	.001	.20	99.55	0.04	72.541	72.747	61.6	87			1.0055	
1	2	3	4	5	6	7	8	9		10/11 Average:	1.0047	

Meter was in tolerance ☒

Meter was not in tolerance ☐; readjusted linkage

Meter was not in tolerance ☐; changed dry test meters

Gas Meter Bimetallic Therm. Calibration/Verification*

☐ ≥ ± 5°F (Out of control) ☐ Recalibrated / /

☐ ≤ ± 5°F (in control) ☐ No action required

Approved by Date Aug 20, 1992

* Verified against mercury in glass thermometer or calibrated platinum resistance thermometer.

** Based on AL-17 wet test meter calibration in August 1989 against Bell Prover (NBS Traceable) - Carl Poe Co.

S0102R 8/89

Date 1-21-92

Bar. Press. 29.61 in. Hg

INTERPOLL LABORATORIES
MINIMODULE CALIBRATION SHEET
Flow Rate Range 0-0.13 CFM
(0-4 LPM)

Meter Box No. 55
Serial No. DTM 64711
Wet Test Meter No. AL-17 (.05 CF/REV)
Technician- E. TROWBRIDGE

		Gas Volume Wet Test Meter (ft³)	** Cal. Index ϕ (%)	Diff. Wet Test Meter ΔP_w (in.WC)	Gas Volume Dry test meter (ft³)		Gas temperatures			Time θ (min/sec)	Meter Coeff. Y	Rota-meter Coeff. Z
Rota-meter Reading (cc/min)	ΔP_d (IN/WC)				V_{di}	V_{df}	Wet Test t_w (°F)	Dry Test t_{di} (°F)				
1000	.001	.2	99.55	0.04	143.450	143.648	66	60		5/25	.9975	
1000	.001	.2	99.55	0.04	143.700	143.900	66	63		5/27	.9935	
1000	.001	.2	99.55	0.04	143.950	144.153	66	70		5/29	.9917	
1	2	3	4	5	6	7	8	9		10/11 Average:	.9941	

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

Gas Meter Bimetallic Therm. Calibration/Verification*
☐ ≥ ± 5°F (Out of control) ☐ Recalibrated / /
☒ ≤ ± 5°F (in control) ☒ No action required

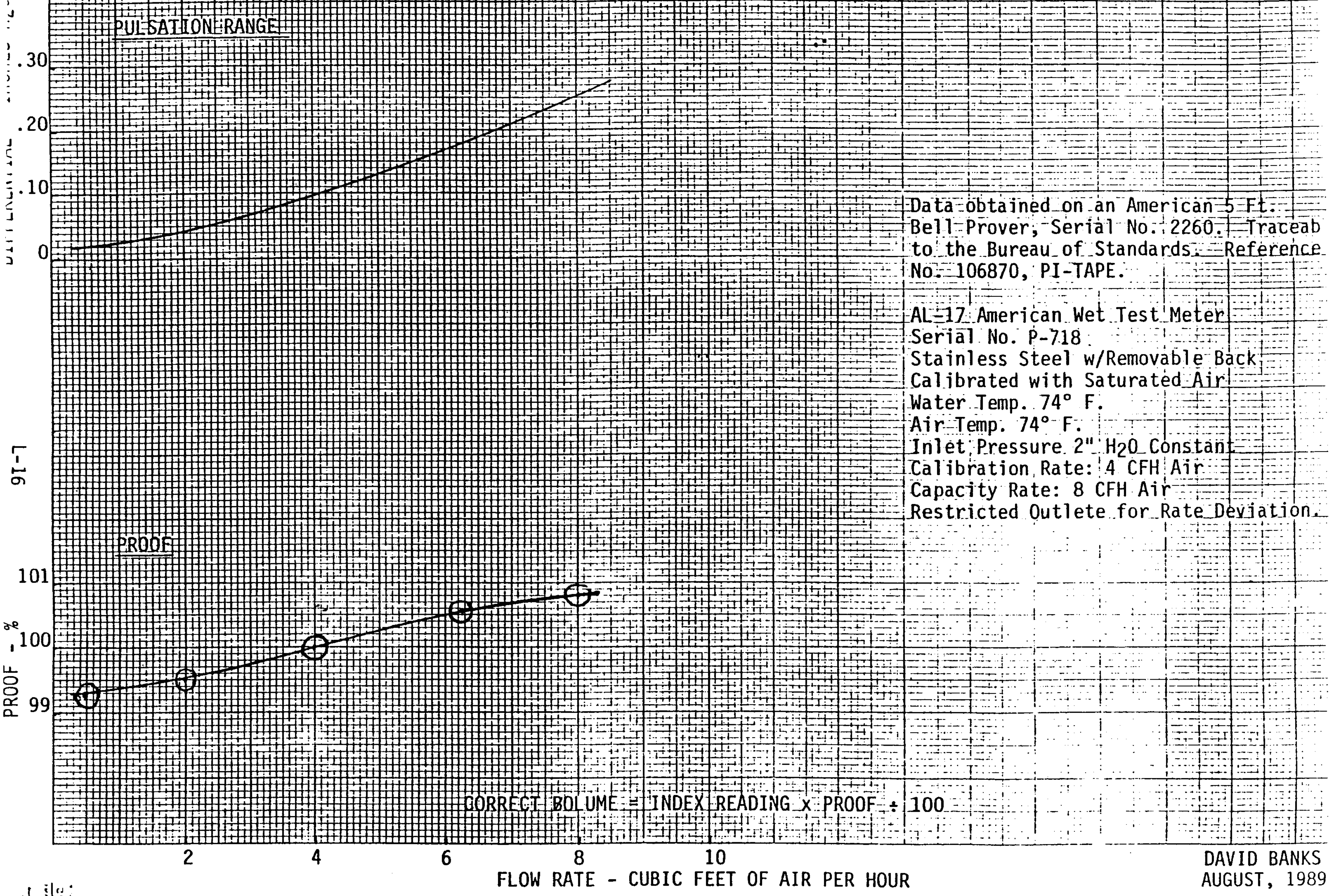
Approved by Daniel J [illegible] Date 1/27/92

* Verified against mercury in glass thermometer or calibrated platinum resistance thermometer.

** Based on AL-17 wet test meter calibration in August 1989 against Bell Prover (NBS Traceable) - Carl Poe Co.

DIFFERENTIAL PRESSUR PROOF CALIBRATION CURVE

WET TEST METER

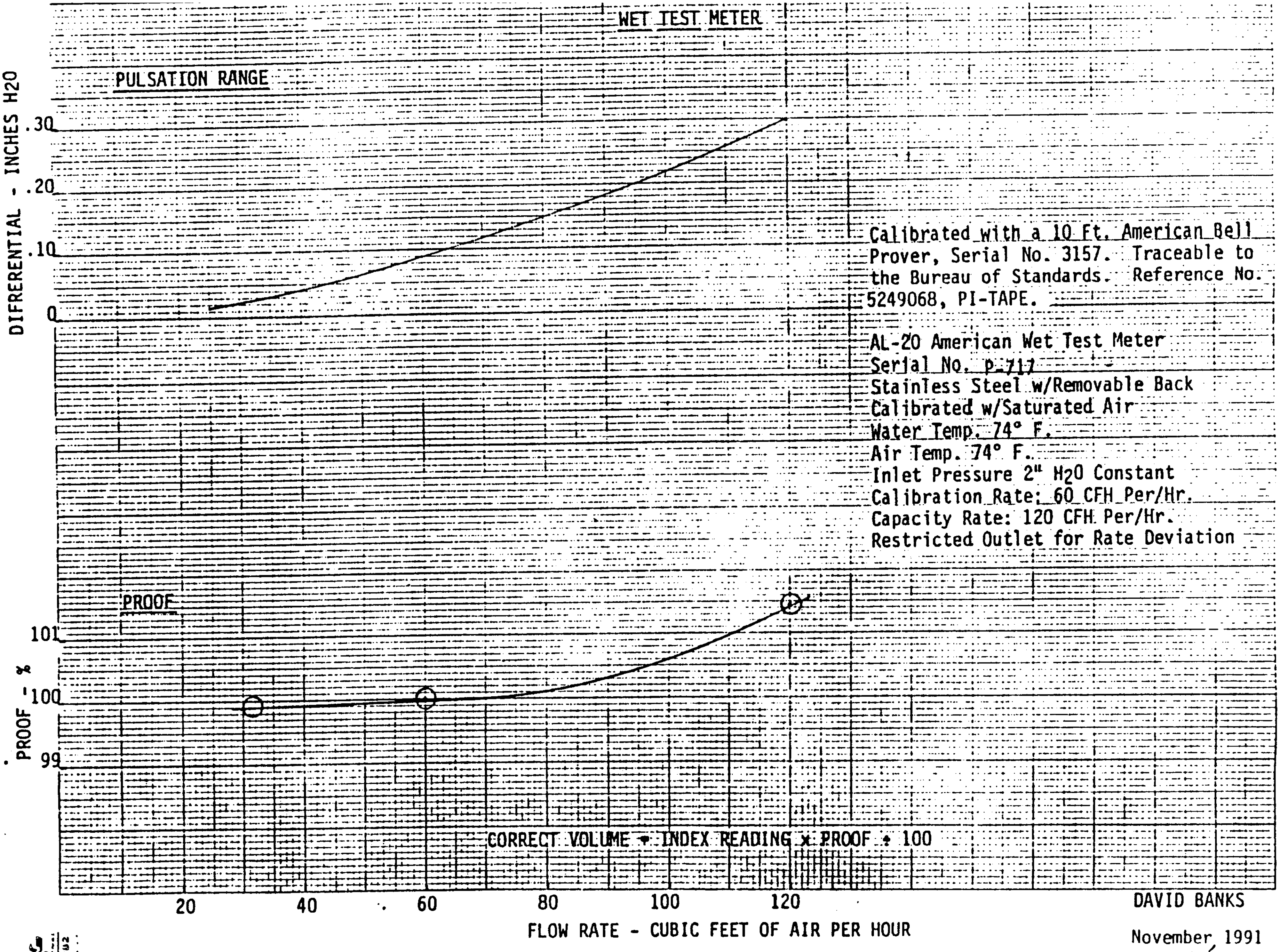


Data obtained on an American 5 Ft. Bell Prover, Serial No. 2260. Traceab to the Bureau of Standards. Reference No. 106870, PI-TAPE.

AL-17 American Wet Test Meter
Serial No. P-718
Stainless Steel w/Removable Back
Calibrated with Saturated Air
Water Temp. 74° F.
Air Temp. 74° F.
Inlet Pressure 2" H_2O Constant
Calibration Rate: 4 CFH Air
Capacity Rate: 8 CFH Air
Restricted Outlete for Rate Deviation.

CORRECT BOLUME = INDEX READING x PROOF ÷ 100

DAVID BANKS
AUGUST, 1989



Calibrated with a 10 Ft. American Bell Prover, Serial No. 3157. Traceable to the Bureau of Standards. Reference No. 5249068, 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

DAVID BANKS

November, 1991

Interpoll Laboratories, Inc.
(612) 786-6020

Nozzle Calibration
Data Sheet

Date of Calibration: 10-26-92

Technician: Duane Van Hoever

Nozzle : Glass

The nozzle is rotated in 60 degree increments and the diameter at each point is measured to the nearest 0.001 inch. The observed readings and average are shown below.

Position	Diameter (inches)
1	.238
2	.238
3	.240
Average:	.239

Interpoll Laboratories, Inc.
(612) 786-6020

Nozzle Calibration
Data Sheet

Date of Calibration: 10-26-92 Nozzle Number mm5-4

Technician: Ron Rosenthal

The nozzle is rotated in 60 degree increments and the diameter at each point is measured to the nearest 0.001 inch. The observed readings and average are shown below.

Position	Diameter (inches)
1	.234
2	.234
3	.235
Average:	.234

Interpoll Laboratories, Inc.
(612) 786-6020

Nozzle Calibration
Data Sheet

Date of Calibration:	10-26-92	Nozzle Number 22-8
Technician:	Gary Hove	

The nozzle is rotated in 60 degree increments and the diameter at each point is measured to the nearest 0.001 inch. The observed readings and average are shown below.

Position	Diameter (inches)
1	.244
2	.245
3	.243
Average:	.244

Interpoll Laboratories, Inc.

Temperature Measurement Device
Calibration Sheet

Unit under test:

Vendor Beckman
Model HD 110T
Serial Number 18
Range 0-1999 °F
Thermocouple Type K
Date of Calibration 9/9/92
Technician Dutt

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	0	0	0
100	100	96	4	.71
200	200	199	1	.15
300	300	298	2	.26
400	400	395	5	58
500	500	494	6	.63
600	600	597	3	.28
700	700	698	2	.17
800	800	803	3	.24
900	900	905	5	.37
1000	1000	1010	10	.68
1100	1100	1114	14	.90
1200	1200	1220	20	1.20
1300	1300	1321	21	1.19
1400	1400	1425	25	1.34
1500	1500	1523	23	1.17
1600	1600	1624	24	1.16
1700	1700	1720	20	.92
1800	1800	1818	18	.80
1900	1900	1911	11	.46
2000		—		
2100		—		
		Averages:		.696

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.

Interpoll Laboratories, Inc.

Temperature Measurement Device
Calibration Sheet

Unit under test:

Vendor Beckman Industrial

Model HDi10T

Serial Number #20

Range -4 - 1999 °F

Thermocouple Type K

Date of Calibration 9/9/92

Technician Dual

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)	Deviation (%)
0		-3	3	.65
100		93	7	1.25
200		196	4	.61
300		295	5	.66
400		392	8	.93
500		491	9	.94
600		594	6	.57
700		695	5	.43
800		800	0	0
900		902	2	.15
1000		1007	7	.48
1100		1110	10	.64
1200		1216	16	.96
1300		1317	17	.96
1400		1421	21	1.13
1500		1519	19	.97
1600		1620	20	.97
1700		1716	16	.74
1800		1814	14	.62
1900		1907	7	.30
2000				
2100				
		Averages:		.70

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

GARY HOVE

Unit under test:
Vendor OMEGA
Model HA21
Serial Number 24
Range 0-2000 °F
Thermocouple Type K
Date of Calibration 7-20-92
Technician

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)	Deviation (%)
0	0	.7	.7	.15
100	100	98.8	1.2	.21
200	200	200.9	.9	.13
300	300	299.5	.5	.07
400	400	398.9	1.1	.12
500	500	498.8	1.2	.125
600	600	601.4	1.4	.13
700	700	700.2	.2	.02
800	800	801.7	1.7	.13
900	900	900.1	.1	.007
1000	1000	1001.0	1.0	.07
1100	1100	1100	0	0
1200	1200	1202	2.0	.12
1300	1300	1300.1	.1	.006
1400	1400	1402.5	2.5	.134
1500	1500	1500.3	.3	.015
1600	1600	1602.1	2.1	.102
1700	1700	1699.6	.4	.019
1800	1800	1801.4	1.6	.07
1900	1900	1801.6	1.6	.068
2000	2000	2000	0	0
2100		2098.6	1.4	.07
		Averages:	1.0	.080

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

(612)786-6020

S-Type Pitot Tube Inspection Sheet

Pitobe No. 23-8

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

6. $B_1 < 5^0$ 2
7. $B_2 < 5^0$ 2

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.

☒ Meets all EPA design criteria thus $C_p = 0.84$

☐ Does not meet EPA design criteria - thus calibrate in wind tunnel $C_p =$ ________

Date of Inspection: 3-2-92

Inspected by:

CFR Title 40 Part 60 Appendix A Method 2

(612)786-6020

S-Type Pitot Tube Inspection Sheet

Pitobe No. MM5-8

Pitot tube dimensions:

1. External tubing diameter (D_t) .316 IN.
2. Base to Side A opening plane (P_A) .460 IN.
3. Base to Side B opening plane (P_B) .462 IN.

Alignment:

4. $\alpha_1 < 10^\circ$ 0
5. $\alpha_2 < 10^\circ$ 0
6. $B_1 < 5^\circ$ 2
7. $B_2 < 5^\circ$ 2
8. Z <.125" .02
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.

☒ Meets all EPA design criteria thus C_p = 0.84

☐ Does not meet EPA design criteria - thus calibrate in wind tunnel C_p = ________

Date of Inspection: 2-29-92

Inspected by:

CFR Title 40 Part 60 Appendix A Method 2

INTERPOLL LABORATORIES
(612)786-6020

Stack Sampling Department - QA
Aneroid Barometer Calibration Sheet

Date 7-31-92

Technician GARY HOVE

Mercury Column Barometer No. NOVA-1

Aneroid Barometer No. 636235

Actual Mercury Barometer Read	Ambient Temp.	Temperature Correction Factor	Adjsted Mercury Barometer Read	Initial Aneriod Barometer Read	Difference (P_{ba}-P_{bm})
29.30	74°F	.121	29.179	29.18	0
LADITUTE		.001	29.178		

Has this barometer shown any consistent problems with calibration? [✓ Yes]/No. If yes, explain. LAST OWNER

Has problem been alleviated? [✓ Yes]/No. How? READJUSTED

*Note

Aneroid barometers will be calibrated periodically against a mercury column barometer. The aneroid barometer to be calibrated should be placed in close proximity to the mercury barometer and left to equilibrate for 20-30 minutes before calibrating. Aneriod barometer will be calibrated to the adjusted mercury barometer readings.

S-312

INTERPOLL LABORATORIES
(612)786-6020

Stack Sampling Department - QA
Aneroid Barometer Calibration Sheet

Date 7-1-92

Technician R. Rosenthal

Mercury Column Barometer No. Nova -1

Aneroid Barometer No. S/N 560209

Actual Mercury Barometer Read	Ambient Temp.	Temperature Correction Factor	Adjsted Mercury Barometer Read	Initial Aneriod Barometer Read	Difference (P_{ba}-P_{bm})
28.91	72	.109	28.801		
ALTITUDE		-.001	28.80	28.83	-.03

Has this barometer shown any consistent problems with calibration? Yes/[✓ No.] If yes, explain.

Has problem been alleviated? Yes/No. How? —

*Note

Aneroid barometers will be calibrated periodically against a mercury column barometer. The aneroid barometer to be calibrated should be placed in close proximity to the mercury barometer and left to equilibrate for 20-30 minutes before calibrating. Aneriod barometer will be calibrated to the adjusted mercury barometer readings.

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INTERPOLL LABORATORIES
(612)786-6020

Stack Sampling Department - QA
Aneroid Barometer Calibration Sheet

Date 9/9/92

Technician D. Van Hoever

Mercury Column Barometer No. 1

Aneroid Barometer No. D44

Actual Mercury Barometer Read	Ambient Temp.	Temperature Correction Factor	Adjsted Mercury Barometer Read	Initial Aneriod Barometer Read	Difference (P_{ba}-P_{bm})
28.85	68	-.105	28.75	28.80	.+05

Has this barometer shown any consistent problems with calibration? Yes/No. If yes, explain. NO

Has problem been alleviated? Yes/No. How?

*Note

Aneroid barometers will be calibrated periodically against a mercury column barometer. The aneroid barometer to be calibrated should be placed in close proximity to the mercury barometer and left to equilibrate for 20-30 minutes before calibrating. Aneriod barometer will be calibrated to the adjusted mercury barometer readings.

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Appendix M: Statement of Work for Field Testing and Analytical Services

September 30, 1992
Attachment to PR B84855

STATEMENT OF WORK

FOR

FIELD TESTING AND ANALYTICAL SERVICES IN SUPPORT OF THE BINDER ENHANCED dRDF (b-dRDF)/COAL TEST PROGRAM AT THE BIG STONE, SD PLANT

1. INTRODUCTION

1.1 BACKGROUND

The National Renewable Energy Laboratory (NREL) and Argonne National Laboratory (ANL) under the sponsorship of the U.S. Department of Energy will conduct, with the participation of the private sector, co-combustion testing of b-dRDF and coal blends in a commercial-scale cyclone fired boiler.

These tests are planned to be conducted at Otter Tail Power Company's 440 MWe power generation facility located in Big Stone City, South Dakota during the October 19-30, 1992 time period.

The combustion test will consist of two different operating conditions to be conducted over a two day period. Each test day will be of at least 10 hours duration. During each test day, Interpoll Laboratories, Inc. shall sample and analyze the flue gas emissions, and analyze the ash residue and feedstock samples. The ash residue and feedstock samples will be collected and delivered to Interpoll Laboratories test personnel by others.

Only one (1) mobilization will be required since the two-day test program will be conducted on consecutive days.

The principal product of the test program will be a report containing test data from flue gas emission tests, the ash residue analyses and the feedstock composition analyses. This report will be submitted by Interpoll Laboratories to NREL/ANL for review and approval consistent with the schedule shown in Section 3. Testing requirements are given in Sections 2.1.1, 2.1.2 and 2.1.3.

1.2 STUDY OBJECTIVE

The objective of this test program is to conduct air emission, ash residue and feedstock tests and analyses of co-fired blends of b-dRDF and coal in a commercial-scale cyclone boiler. These test results will allow private industry and/or the electric utility industry to develop operating, technical and financial data which will enhance and expand the productive commercial use of b-dRDF as an alternative fuel product. These test data will also allow state regulatory agencies to evaluate methods for permitting and monitoring such units in the future and will provide a data base for equipment manufacturers.

2. TESTING REQUIREMENTS

2.1 SAMPLING AND ANALYSIS METHODOLOGY

The manual and instrumental emission determinations shall be conducted according to the provisions of standard EPA methods cited in CFR Title 40, Part 60, Appendix A (revised July 1, 1990) and other special purpose EPA methodologies reported elsewhere in the literature. All data shall be calculated and reported in units as specified in the above-cited references.

The sampling and emission determinations will be conducted jointly by Otter Tail Power Company/ANL personnel and Interpoll Laboratories. The flue gas sampling and emission determinations to be conducted by Interpoll Laboratories is described in Section 2.1.1. The ash residue and feedstock samples will be collected by Otter Tail Power and ANL personnel and delivered to Interpoll Laboratories for analysis. The ash and feedstock analysis is given in Section 2.1.2 and 2.1.3, respectively.

2.1.1 Stack Gas Sampling and Analysis

The following determinations shall be performed on each of the two (2) consecutive 10-hour test days.

All emission levels shall be reported as 7% 0_2, dry basis. Emission rates will be reported in units of lb/hr and lb/MMBTU. Dioxins/furans shall be measured as total tetra through Octa-chlorinated dibenzo-p-dioxins and dibenzofurans with units of ng/dscm. Where not specifically noted, otherwise a minimum of three (3) determinations shall be made for each item in Section 2.1.1a through 2.1.1o. For those elements not detected, detection levels shall be calculated and reported.

a. CO_2 and O_2 concentrations and emission rate (for CO_2 only)- EPA Method 3A.

b. CO concentration and emission rate - EPA Method 10.

c. Gas moisture content (nine determinations) - EPA Method 4.

d. Particulate (including condensables) concentration and emission rate (three (3) one-hour determinations) - EPA Method 5.

e. SO_2 concentration and emission rate - EPA Method 6C.

f. Oxides of nitrogen (NO_x) concentration and emission rate - EPA Method 7E.

g. Dioxins and furans (PCDD and PCDF) concentration and emission rate (three (3) two-hour determinations (ca.2m^3) using an EPA MM5 semi-volatile sampling train with purified XAD-2 resin shall be made. One (1) field-biased sampling train blank will also be recovered. Confirmation of 2, 3, 7, 8-TCDD isomer shall be made - EPA Method 23.

h. Polynuclear aromatic hydrocarbons (PAH) concentration and emission rate. Each sample shall be analyzed for the following PAHs:

- Acenapthene
- Acenapthylene
- Anthracene

- Benzo-a-anthracene
- Benzo-a-pyrene
- Benzo-b, k-fluoranthene
- Benzo-g, hi, i-perylene
- Chrysene
- Dibenzo-a, h-anthracene
- Fluoranthene
- Fluorence
- Indeno - 1,2,3-g, d-pyrene
- Naphthalene
- Phenanthrene
- Pyrene

EPA Method 8270 will be used for analyzing the PAH aliquots.

i. Polychlorinated Biphenyls (PCBs) concentrations and emission rates. Each sample (minimum of three) shall be analyzed for the congeners Mono through Deca (10 congeners).

j. Total chlorine (Cl) and hydrogen chloride (HCl), hydrogen bromide (HBr) and hydrogen fluoride (HF) concentration and emission rate (three (3) one-hour determinators) - EPA Method 26.

k. Benzene, Toluene and Xylene (BTX) concentrations and emission rates (minimum of three samples) - EPA Method 18, Section 7.4.

l. Total hydrocarbons (THC) concentration and emission rates - EPA Method 25A.

m. Trace metals concentration and emission rates (three (3) two-hour determinations) using a Multi-Metal Modified Method 5 (4M5) sampling train. One field-biased blank will be collected, recovered and analyzed with the field samples - EPA Method 29.

Metals to be analyzed are:

- Arsenic
- Beryllium
- Cadmium
- Chromium
- Lead
- Mercury
- Nickel
- Antimony
- Barium
- Copper
- Selenium
- Silver
- Thallium
- Zinc

n. Opacity in percent. Three (3) 6-minute determinations by an EPA-certified reader - EPA Method 9.

o. Stack gas velocity and volumetric flow rate shall be determined - EPA Method 2.

p. A drawing shall be prepared showing the following:

- location and number of test ports
- number of traverse points
- number of traverse points per traverse
- duct diameter

q. Continuous emission testing shall be conducted for the following emissions: SO_2, NO_x, THC, CO, CO_2 and O_2.

2.1.2 Ash Analysis

Bottom ash and flyash samples will be collected during the two (2) test periods by Otter Tail Power Company/ANL personnel. These samples will be delivered to Interpoll Laboratories test personnel for analysis. The ash samples shall be analyzed for total composition, leaching potential (TCLP) and physical characteristics.

2.1.2.1 Compositional Analysis

Two (2) samples of bottom ash and three (3) samples of flyash for each test day (a total of ten (10) samples) will be submitted to Interpoll Laboratories for analyses. The following determinations shall be made:

- Arsenic
- Beryllium
- Cadmium
- Chromium
- Lead
- Mercury
- Nickel
- Antimony
- Barium
- Copper
- Selenium
- Silver
- Thallium
- Zinc
- Chlorides
- Sulfates
- PCDD/PCDF

  Interpoll Laboratories shall analyze the three (3) flyash samples submitted each test day (a total of six (6) samples) for total tetra-through octa-chlorinated dibenzo-p-dioxins and dibenzofurans.

- Mineral Analysis

  Interpoll Laboratories shall analyze the two (2) bottom ash samples and three (3) flyash samples submitted each test day (a total of ten (10) samples for the following: SiO_2, Al_2O_3, K_2O, TiO_2, MgO, Fe_2O_3, Fe_3O_4, Na_2O, ZnO, SO_3, P_2O_5, BaO, and CaO. This analysis shall be expressed as a weight percentage on an "as received" basis and "dry" basis.

2.1.2.2 Leaching Potential (using TCLP test method)

Two (2) samples of bottom ash and three (3) samples of flyash for each test day (a total of ten (10) samples) will be submitted to Interpoll Laboratories for analyses. The following determination shall be performed on the TCLP leachates for:

- Arsenic
- Beryllium
- Cadmium
- Chromium
- Lead
- Mercury
- Nickel
- Antimony
- Barium
- Copper
- Selenium
- Silver
- Thallium
- Zinc
- pH of ash and pH of leachate produced by the leach test
- Alkalinity
- Chlorides
- Sulfates
- Total dissolved solids (TDS)

2.1.2.3 Ash Physical Characteristics

Two samples of bottom ash and three (3) samples of flyash for each test day (a total of ten (10) samples) will be submitted to Interpoll Laboratories for analyses. The following determinations shall be performed:

- Moisture content
- Percent combustibles

2.1.3 Feedstock Analysis

For the coal only test day coal samples will be collected from the conveyer belt at one-hour intervals by Otter Tail Power/ANL personnel. These samples will be composited into three samples and delivered to Interpoll Laboratories for analysis.

For the blended fuel, test samples of each fuel type (b-dRDF and coal) will be collected individually from their respective conveyor belts at one-hour intervals by Otter Tail Power/ANL personnel. These samples will be composited into three samples for each fuel type and delivered to Interpoll Laboratories for analysis.

A total of nine (9) feedstock samples will be delivered to Interpoll Laboratories for analysis. The following determinations will be performed on each of the fuel samples submitted:

- Ultimate and proximate analysis. All elements expressed as percentage by weight on "as received" basis and "dry basis".
- High heating value (HHV) and bulk density. HHV in BTU/lb and bulk density in lb/ft.

- Total chlorine (presented as an element of the ultimate analysis)
- Ash fusion temperature (8-point)
- Trace metals content. These metals shall include those metals given in Section 2.1.1m.

3. REPORTING REQUIREMENTS

Interpoll Laboratories shall prepare a detailed report summarizing the results of the air emission, ash analyses and feedstock analyses. A draft copy of the report shall be submitted two (2) months after completion of the on-site tests. Five (5) copies of the final report shall be submitted by Interpoll Laboratories one (1) month after receipt of the approved or corrected draft report from NREL/ANL.

The report shall consist of the following elements:

a. Executive Summary
b. Tests Results Summary
c. Results of Individual Air Emission Determination
d. Results of Ash Residue Determinations
e. Results of Feedstock Determinations
f. Appendices:
    1. Sampling and analytical procedures used (including detection levels)
    2. Field data sheets and notes
    3. Laboratory data and reports
    4. Calculation equations and report nomenclature
    5. Quality assurance data (claim of custody, etc.)

4. ITEMS TO BE FURNISHED BY BIG STONE, SD PLANT

Interpoll Laboratories will not be responsible for facility modifications necessary to conduct the air emissions sampling tests. However, Interpoll shall indicate in writing to NREL/ANL, at least one-month prior to the scheduled tests what modifications are needed to facilitate testing activities.

5. OTHER DOCUMENTATION REQUIRED TO BE FURNISHED BY INTERPOLL LABORATORIES

Interpoll Laboratories shall submit to NREL/ANL at least one-month prior to the scheduled tests the following documentation:

- A copy of the corporate Health and Safety Plan consistent with the requirements of EPA's Occupational Health and Safety Manual and other applicable EPA safety guidance.

- A copy of the corporate Quality Assurance/Quality Control Plan

Appendix N: Quality Assurance/Quality Control Plan

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

Telephone (612)786-6020

INTERPOLL LABORATORIES

QUALITY ASSURANCE/
QUALITY CONTROL PLAN

Version 5.01
January 2, 1992

Total Number of Revisions 3
Date of last revision January 2, 1992

QA Plan approved by:

Ben A. Addie

Ben A. Addie,
Quality Control Officer

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1 INTRODUCTION

Interpoll Laboratories, when originally founded in 1973, was dedicated to providing comprehensive air pollution testing services. Initial capabilities in stack testing and ambient air quality and meteorological monitoring expanded rapidly due to strong support from local industry. Particle sizing, atmospheric dispersion modeling and fuel analysis were soon added to the growing list of available services. Continued expansion of our client base and the need of individual clients for more and more sophisticated chemical analysis led to ever expanding analytical capabilities. In 1980 the laboratory went commercial and began providing analytical services directly to clients.

The Chemistry Department's primary mission is to perform accurate and precise determinations of a wide variety of analytes in support of Interpoll Laboratories' field testing activities and as a commercial service to industry and government at large. This is accomplished by the continuing purchase of state-of-the-art instrumentation, employment of knowledgeable and experienced chemists and technicians, training, responsible project and client management, and a comprehensive independent internal quality assurance and control program. Because of the wide variety of analytical services provided and the differences in equipment and analytical skills required to perform many of the analyses, three different laboratories with different areas of specialization have evolved within Interpoll Laboratories: Ambient Air Quality/Stack Testing Laboratory, Inorganic Laboratory, Organic Laboratory. All three laboratories are monitored and internally audited by the Quality Assurance Program.

Interpoll Laboratories is an environmental testing laboratory and, as such, has recognized the need for recycling and the conservation of our natural resources. Our document control system was specifically designed to reduce the amount of paper generated when changes are made to existing documents. All pages of Interpoll Laboratories documents (e.g., manuals, SOP's, methods, etc.) are labeled as follows:

Manual (Method)____________
Version No.________________
Revision No._______________
Date______________________
Page_________of___________

The Version Number will increase by one only when major changes or rewrites occur. When minor changes are made, the Version Number will remain the same and the Revision Number will increase by one on the revised page(s). Only the revised pages will be printed and redistributed, which will significantly reduce the amount of paper generated.

2 QUALITY ASSURANCE POLICY STATEMENT

A Quality Assurance Program is necessary to evaluate and control the factors which are important in establishing and maintaining the production of reliable data. This program is essential because the results obtained by laboratory analyses are often the basis on which important decisions are made.

The Quality Assurance Program has two important functions. The first is "control of quality." The portion of the program which fulfills this function considers all of the factors influencing the reliability of results. Facilities, instrumentation and personnel are included in this portion.

The second function is "measurement of quality" which is accomplished by collecting data from duplicate analyses, spiked samples and performance standards. Statistical methods are applied to evaluate the precision and accuracy of the analytical method, thus monitoring the reliability of the results.

The ultimate goal of the Quality Assurance Program is to consider, evaluate and control all of the variables which can affect the final results produced in the laboratory. Frequent review and revision of the Quality Assurance Program is an integral part of quality control in the laboratory.

3 QUALITY ASSURANCE RESPONSIBILITY

Responsibility for the Quality Assurance Program is assigned to the Quality Assurance Coordinator who works with the Department Managers to continually review all aspects of the program and make revisions as needed.

However, the laboratory stresses the fact that the effectiveness of the Quality Assurance Program is dependent on the cooperation of all laboratory personnel. Each staff member is expected to frequently review the techniques employed and monitor data quality through the analysis of duplicate and spiked samples, independent check standards, and through participation in numerous external audits sponsored by the EPA, as well as State Certification Programs. Interpoll Laboratories list of certifications is given in Figure 3-1.

Figure 3-1. List of Certifications

Department of Defense

Interpoll Laboratories is certified by the Department of Defense, United States Army Toxic and Hazardous Materials Agency (USATHAMA) for numerous organic and inorganic parameters.

State of Minnesota

Interpoll Laboratories is certified for all analytes available for certification through the Minnesota Department of Health, including bacteriological analysis, a number of inorganic parameters and lead for both the Clean and Safe Water programs.

The State of Minnesota has announced it will be certifying environmental laboratories for an extended lists of metals. As soon as the State initiates the certification process, Interpoll Laboratories will apply for certification of all metals that will be applicable.

State of Wisconsin

Interpoll Laboratories is certified for all analytes available for certification through the Wisconsin Department of Natural Resources, including numerous inorganic and organic parameters.

Figure 3-1 Continued

Performance Audit Programs

Interpoll Laboratories participates in the following audit programs:

- EPA Water Pollution Performance Studies
- EPA Water Supply Performance Studies
- EPA Acid Rain Audits
- EPA Filter Audits (Lead, Nitrate, Sulfate)
- Twin City Round Robin Interlaboratory Group
- Analytical Products Group Quarterly Audits

4 PERSONNEL

4.1 Organization

The laboratory staff is divided into two groups; Organic and Inorganic. The activities of each group are guided by a Department Manager. Communication between the Department Managers is encouraged in an effort to share common problems and solutions. In addition, many samples require work in both areas, therefore, the most appropriate approach to the analysis effort can best be worked out together. An organizational chart is included as Figure 4-1.

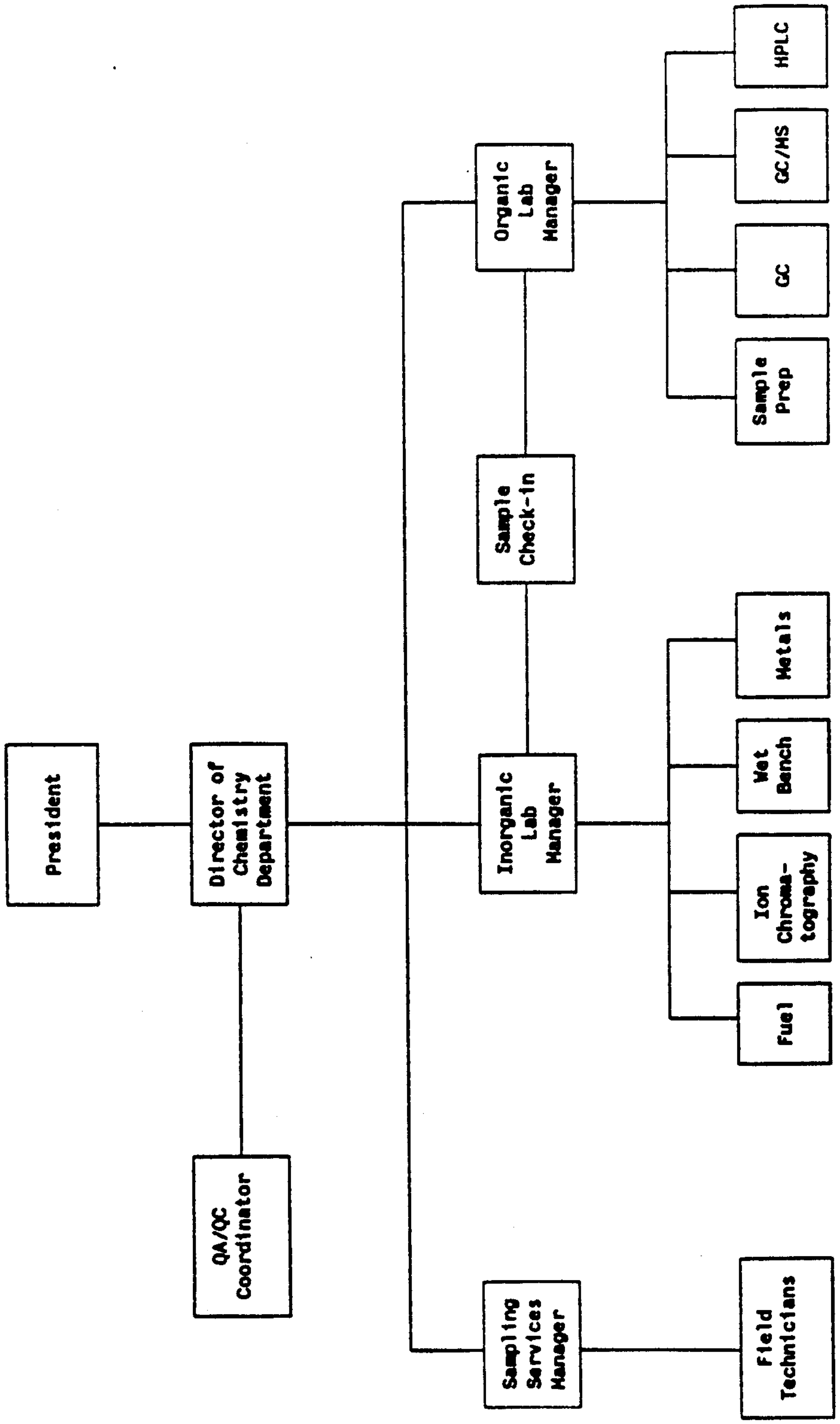
4.2 Requirements

A trained, professional staff is necessary for the production of reliable data. At Interpoll Laboratories, there are certain minimum educational and experience requirements necessary for each position in the laboratory. For each position, a minimum level of training is prescribed.

4.2.1 Laboratory Director

The Laboratory Director is the technical leader for the entire laboratory. Therefore, this position requires familiarity with all of the analyses currently being performed in the laboratory. The responsibilities include review of data generated and, in many cases, acting as an interface between clients and analysts. The minimum requirement for this position is a bachelor's degree and ten years of laboratory experience. An advanced degree is desirable.

Figure 4-1. Interpoll Laboratories Organizational Chart



4.2.2 Quality Assurance Coordinator

The Quality Assurance Coordinator is responsible for implementation and maintenance of the Quality Assurance/Quality Control Plan, including monitoring laboratory performance by keeping QA charts for each method and by periodically submitting blind samples for analysis. This individual is not part of the analytical staff, but works with staff members to ensure that the best possible data is generated. The minimum requirement is a bachelor's degree and two years of laboratory experience with an emphasis in the area of quality assurance.

4.2.3 Department Manager

The Department Manager is responsible for all analytical work performed by a group of analysts; therefore, technical expertise is essential for this position. This individual must be capable of scheduling the workload to maximize the use of equipment and personnel and must be able to deal effectively with analysts and clients. The minimum requirement is a bachelor's degree and five years experience with an emphasis in the area being managed. An advanced degree is desirable.

4.2.4 Chemist III

The Chemist III position requires a bachelor's degree and seven years of experience and/or a PHD in a related field. A Chemist III is responsible for one or more laboratories. Responsibilities include organization of the workload, equipment maintenance, preparation of standards and reagents, preparation of samples, and the proper documentation of all procedures utilized and the analytical results. This position may supervise Technicians I, II and III and the Chemist I positions. This position is responsible for method development and research projects.

4.2.5 Chemist II

The Chemist II position requires a bachelor's degree and three years of experience and/or a master's degree in a related field. A Chemist II may be responsible for one or more laboratories. Responsibilities include organization of the workload, equipment maintenance, preparation of standards and reagents, preparation of samples, and the proper documentation of all procedures utilized and the analytical results. This position may supervise Technicians I and II. This position may be responsible for method development and research projects.

4.2.6 Chemist I

The Chemist I position requires a bachelor's degree in a related field of science. A Chemist I is responsible for a specific laboratory. Responsibilities include organization of the workload, equipment maintenance, preparation of standards and reagents, preparation of samples, and the proper documentation of all procedures utilized and the analytical results. This position may be supervised by the Chemist III.

4.2.7 Technician III

The Technician III position requires an associate degree and /or seven years of laboratory experience. A laboratory Technician III has demonstrated competence in a given area and often works independently. The Technician III is responsible for organization of the workload, performing the required analytical procedures and documentation of results and procedures on the required forms. The Technician III position can be considered equal to a Chemist I position after competence in three or more areas has been proven. The Technician III may be supervised by the Chemist III position.

4.2.8 Technician II

The Technician II position requires an associates degree and/or five years of related laboratory experience. A laboratory Technician II works under the wupervision of a Chemist II or III. Responsibilities include the preparation of reagents to performing wet bench analytical proceedures.

4.2.8 Technician I

The Technician I position requires some vocational training or one years of related laboratory experience. A laboratory Technician I works under the supervision of a Chemist II or III. Responsibilities range from record keeping to the preparation of reagents.

4.2.9 Laboratory Aide

The Laboratory Aide position requires a high school diploma. A laboratory Technician I works under the direct supervision of a Chemist II or III. Responsibilities range from glassware cleaning to simple sample preparation proceedures.

4.3 Staff Development

New developments in methods and instrumentation are being made continually. In order to produce the most accurate, reliable data possible, it is necessary for the staff to keep up to date with state-of-the-art methods and instruments. Recognizing this fact, Interpoll Laboratories encourages employees to continue to develop their area of expertise by reviewing current literature, attending conferences, seminars and training sessions when available, and seeking additional academic training when it would be beneficial. Every effort is made to arrange workloads to facilitate these activities.

5 LABORATORY FACILITIES, EQUIPMENT AND SERVICES

5.1 Space

The laboratory occupies 16000 square feet of space which is divided so that similar analyses are performed in the same area.

5.1.1 The Sample Receiving Area is where incoming samples are logged in and preserved if necessary. This area is locked at all times for secure storage of samples and reagents. This area is also used for washing and preparing bottles for sample collection, as well as the washing and drying of all glassware used in the laboratories.

5.1.2 The Liquid Chromatography Lab includes two ion chromatographs (IC) and two high performance liquid chromatographs (HPLC). This area also includes the water purification system which produces laboratory grade water for analyses.

5.1.3 The Extraction Lab is where all TCLP, ASTM and EP Toxicity extractions are performed. This area houses six TCLP zero headspace extractors (ZHE), three EP Toxicity extractors, and all relevant equipment necessary to perform these extractions.

5.1.4 The Inorganic laboratory is the area where all inorganic analyses are performed. All titration and distillation equipment, as well as microbiological equipment is found in this laboratory. This laboratory has enough bench space to perform many inorganic analyses simultaneously to provide quicker turnaround to our clients.

5.1.5 Three laboratories are designated for metals analysis; one laboratory is strictly for sample preparation and the other two for instrumentation. The sample preparation lab has ample hood space for hot plate sample digestion and also has the capabilities for microwave digestion. Of the two instrumentation labs, one houses two atomic absorption units and one graphite furnace, the other houses an inductively coupled plasma unit.

5.1.6 The organics analysis area includes the GC/MS laboratory, the general organics instrumentation laboratory and two separate sample preparation laboratories. The GC/MS laboratory houses three gas chromatograph/mass spectrometers (GC/MS). The general organics instrumentation laboratory contains eight gas chromatographs with various detectors, auto samplers, and personal computers. The two sample preparation laboratories contain all necessary glassware and equipment needed to perform liquid-liquid, soxhlet and sonication extractions, as well as the equipment required for evaporation and concentration of sample extracts. One of the preparation laboratories houses the Automated Gel Permeation Chromatography (GPC) extract clean up apparatus and other column chromatography clean-up equipment.

5.1.7 Fuel analyses are performed in a laboratory specifically equipped for this purpose. This ASTM laboratory is capable of performing all ASTM analyses on coal, coke, and ash samples. This laboratory also has the capabilities to test paint, wood, sludge, and many different matrices by ASTM methodologies.

5.1.8 The particle size and morphology laboratory is equipped with an X-ray Sedigraph, flow clean benches, polarizing/phase contrast microscopy, sieving equipment, and a stereo zoom microscope.

5.1.9 The balance laboratory houses three analytical balances and is used primarily by the inorganics area and the metals area to weigh samples prior to analysis.

5.2 Electrical

Each laboratory is wired to accommodate the instruments it houses. Many instruments require relatively constant voltage and have built-in voltage regulators to maintain stable, drift-free operation. However, some instruments require an external voltage regulator to serve this purpose.

5.3 Ventilation

The laboratory has six 96-inch fume hoods with roof-mounted exhaust blowers and two 48-inch fume hoods with a ceiling-mounted exhaust blower. A make-up air system is installed throughout the entire laboratory. This make-up air system provides the building with positive or negative air pressure where needed, conditions the temperature of incoming air, provides for zone control over four separate units, and filters all incoming air. The consequences of this system are: better temperature control; reduced intralaboratory contamination; and the ability to provided both positively and negatively pressured air where necessary.

5.4 General Cleaning

A clean, well organized laboratory reduces the probability for mistakes and minimizes the number of interferences present in the laboratory. A janitorial service performs the general cleaning, however, each analyst is responsible for keeping lab benches free of clutter and making certain potential contaminants are not kept in the area.

5.5 Glassware

5.5.1 Specifications

The majority of the glassware used in the laboratory is borosilicate glass. Materials such as Teflon, polyethylene and polypropylene are employed in special situations when appropriate.

Sources of error which are associated with calibrated glassware for precise measurements are carefully considered. All volumetric glassware purchased for the laboratory meets Class A Federal Specifications as shown in Table 5-1. Upon receipt, laboratory glassware and pipets are given to the Quality Assurance Coordinator for calibration. All calibration results are logged in a notebook labeled for this purpose. After passing the specifications mentioned above, the glassware and/or pipets are allowed to be used in the laboratory, and the supplier is notified of the deficiency. Automatic micropipets are used when measuring extremely small volumes. These pipets are calibrated monthly by weighing the volume of water delivered. The calibration is recorded in the same notebook.

The method of calibration ("to contain" or "to deliver") and the temperature at which the glassware was calibrated are considered when selecting glassware for a particular application. Glass pipets are inspected for cracks or chips in the tips that may affect the volume they deliver. When new pipets are purchased, 10% of them are verified by weighing the water they deliver. If they do not meet the specifications given in Table 5-1, the pipets are not used and the supplier is notified. Automatic micropipets are used when measuring extremely small volumes. These pipets are calibrated monthly by weighing the volume of water delivered. The calibration is recorded in a notebook kept for this purpose.

5.5.2 Cleaning Procedures

Several cleaning procedures are used at Interpoll Laboratories. The analytical method for which the glassware is to be used determines which procedure is followed.

5.5.2.1 Inorganic

5.5.2.1.1 Wash all glassware with hot, soapy water.[1] Use the "S/P Brand Laboratory Detergent Concentrate." Be sure to scrub all glassware with brushes. Gloves and a lab coat should be work at all times.

5.5.2.1.2 Rinse the glassware three (3) times with hot tap water.

5.5.2.1.3 Rinse the glassware with distilled water.

5.5.2.1.4 Rinse the glassware 4-5 times with Milli-Q water.

5.5.2.1.5 Dry glassware in oven and return to inorganics laboratory.[2] Put glassware away in the appropriate area.

5.5.2.2 Organic

5.5.2.2.1 Prepare the laboratory detergent solution by following the instructions given on the detergent container.

5.5.2.2.2 Immerse the glassware into the detergent solution and allow it to soak before scrubbing it with nylon brushes. Wash the glassware until all visible contamination has been removed. Rinse it with hot tap water and set it aside to thoroughly drain.

---

[1] Phosphorus glassware should be washed without detergent. Ammonia nitrogen glassware should be rinsed with ammonia-free water.

[2] Do not oven dry volumetric flasks; these must be air dried.

5.5.2.2.3 Volumetric flasks are not to be rinsed with chromic-sulfuric acid. Instead, repeatedly rinse them with deionized water and then, finally, with methanol.

5.5.2.2.4 Dry the flasks in a drying oven set at 230°F for at least 20 minutes, or longer, until all of the methanol has evaporated. Remove the flasks and allow them to cool to room temperature. Store the volumetric flasks, with stoppers inserted, in a clean, segregated area and reserve them for organic analyses.

5.5.2.2.5 Prepare the chromic-sulfuric acid solution (Chromerge) by following the instructions given on the chromium trioxide container. Rinse the well drained, detergent washed glassware with the Chromerge so that all interior surfaces have been wetted with the acid. Pour off the Chromerge and recover it in a beaker. This Chromerge can be reused until it has been chemically depleted (green in color as opposed to orange-brown). Allow the acid to thoroughly drain from the glassware. This insures that the acid has had a sufficient amount of contact time to oxidize any residues remaining after detergent washing.

5.5.2.2.6 Rinse the glassware with tap water and then with deionized water. Collect the washings for neutralization and proper disposal. Set the glassware aside to drain.

5.5.2.2.7 Solvent rinse the glassware, in order, with methanol, acetone and then methylene chloride. Collect the non-halogenated and halogenated solvents separately for proper disposal. After the solvent has completely evaporated from the glassware, store it in a clean, segregated area and reserve it for organic analyses.

5.5.2.3 Metals

5.5.2.3.1 Wash all glassware with hot, soapy water using the laboratory detergent. Be sure to scrub all glassware using brushes. Gloves and a lab coat should be worn at all times.

5.5.2.3.2 Rinse glassware with hot tap water.

5.5.2.3.3 Soak the glassware in a 1:1 nitric acid/water mixture for 15 minutes. Wear gloves, a face shield, and a full-length apron!

5.5.2.3.4 Thoroughly rinse the glassware with tap water.

5.5.2.3.5 Rinse the glassware with successive portions of Milli-Q water. Rinse the glassware 4-5 with Milli-Q water to avoid trace metal contamination.

5.5.2.3.6 Glassware can be brought back to the Metals area without being dried.

Table 5-1. Tolerances for Volumetric Glassware (Abridged from National Bureau of Standards Data, 1941).

Capacity (mL) less than and including	Limit of error (mL)
Graduated Flasks	
25	0.03
50	0.05
100	0.08
200	0.10
250	0.11
300	0.12
500	0.15
1,000	0.30
2,000	0.50
Transfer Pipets	
2	0.006
5	0.01
10	0.02
25	0.025
30	0.03
50	0.05
100	0.08
200	0.10
Burets[1]	
5	0.01
10	0.02
30	0.03
50	0.05
100	0.10

---

[1] Limits of error are of total or partial capacity. Customary practice is to test the capacity at five intervals.

5.6 Reagents, Solvents and Gases

5.6.1 Reagent Quality

Chemicals are available in various grades of purity. Primary Standard Chemicals are specially manufactured analytical reagents of exceptional purity. They are used in critical work such as standardizing volumetric solutions and preparing reference standards. Analytical Reagent grade chemicals (sometimes referred to as Reagent grade) are suitable for general analytical work. Some manufacturers use an American Chemical Society (ACS) designation on certain chemicals, meaning they meet standards set by the ACS Committee on Analytical Reagents. Technical and Practical grade chemicals are of limited value and are seldom used in an analytical laboratory.

5.6.2 Selection of Proper Grade of Reagent

The selection of the proper grade of reagent quality is determined by the parameter of interest and the method of analysis. Analytical reagent grade is normally utilized unless a specific purity is designated in the procedure.

5.6.3 Date Coding of Reagents

The date a reagent is received is recorded on the container label, and it is properly discarded when the shelf life expires. The laboratory policy is to order reagents in quantities that will be used in less than one year, unless a shorter shelf life is specified.

5.6.4 Preparation of Reagent Solutions

Traditional analytical techniques are used in the laboratory preparation of reagent solutions and standards. All solutions are labeled including the initials of the preparer and the date of preparation. A data sheet is filled out for every solution prepared. This data sheet is kept in a logbook and can stand to prove the quality of standards and spiking solutions should a potential problem arise. All solutions are numbered to be sure all data sheets are in the logbook, and that all solutions have the appropriate paperwork. All solutions are stored in the appropriate manner. Stock solutions are restandardized as required by the methodology.

5.6.5 Laboratory Water

Laboratory grade water is produced by an ion exchange system incorporating in series strong acid, strong base, and mixed bed ion exchange tanks producing less than 10 megaohm water. Eighteen megaohm water is produced by feeding less than 10 megaohm water through a Millipore Millique® ion exchange-activated carbon system. This laboratory water conforms to ASTM Type II specifications.

5.6.6 Gases

The required purity of a gas is determined by the intended use, and is specified by the instrument manufacturer or given in the analytical method. When ordering a gas for a new application, special attention is given to selecting the appropriate purity.

Gas cylinders are moved by means of a cart equipped with a securing device. Cylinder caps are left on the cylinder when transporting. Cylinders are secured at all times.

6 ANALYTICAL INSTRUMENTATION

A key element in the generation of accurate and precise results is the use of high quality, well maintained instruments. Instruments must be selective for the analyte to be measured and have a stable response in order to generate reproducible results.

6.1 Laboratory Instruments

The principle instruments currently in use at Interpoll Laboratories are listed below according to the analytical area which is the primary user. The variety of instruments available gives the analyst the opportunity to choose the method best suited to the sample in question. Frequently, a problem is approached using two different instruments so that a comparison of results is possible.

6.1.1 Metals

		Year Acquired
6.1.1.1	Perkin Elmer 560 atomic absorption spectrophotometer.	1982
6.1.1.2	Perkin Elmer HGA 400 graphite furnace with an AS-1.	1982
6.1.1.3	Perkin Elmer Zeeman 5100 PC atomic absorption spectrophotometer with an AS-60 furnace autosampler and an IBM compatible data station.	1988
6.1.1.4	Thermo Jarrell Ash ICAP 61 inductively coupled plasma spectrophotometer with 30 channel fixed polychrometer, and, in addition to the regular nebulizer, a ultrasonic nebulizer manufactured by CETAC Technologies.	1989

6.1.2 Liquid Chromatography

		Year Acquired
6.1.2.1	Dionex System 10 ion chromatograph with an IBM LC-9540 data system.	1982
6.1.2.2	Ion chromatography system consisting of an IBM LC pump, a Wescan conductivity detector and an IBM LC-9540 data system.	1983
6.1.2.3	IBM LC/9533-2A liquid chromatograph with Ternary gradient capability and equipped with fluorescence and UV/VIS detectors.	1983
6.1.2.4	Dionex System Model 4000i HPLC-IC with autosampler; dual channel with conductivity and UV detectors.	1987
6.1.2.5	Club AT 386 IBM compatible personal computer with 640K RAM and a 60 MB hard drive equipped with the Maxima 820 Version 3.3 chromatography software.	1988
6.1.2.6	Club AT 386 IBM compatible personal computer with 640K RAM and a 60 MB hard drive equipped with the Maxima 820 Version 3.3 chromatography software.	1988
6.1.2.7	Waters HPLC/Carbamate analysis system consisting of: Model 600E powerline gradient controller; Model U6K injector; Model 470 scanning fluorescence detector; carbamate analysis module; temperature control module; water satellite WISP; Model 991 photodiode array detector.	1991

		Year Acquired
6.1.2.8	NEC Powermate 386/25 IBM compatible personal computer with 2.0 MB RAM, 110 MB hard drive, 1.2 MB floppy disk drive, equipped with Foreground/Background software, Waters 5200 thermal printer/plotter.	1991
6.1.3	General Inorganic	
6.1.3.1	Bausch & Lomb spectrophotometer Model Spectronic 20.	1976
6.1.3.2	Varian Aerograph Series 200 gas chromatograph with FID detector and gas sampling valve.	1979
6.1.3.3	Turner fluorometer.	1979
6.1.3.4	Two Orsat analyzers.	1979
6.1.3.5	Trident pH meter Model 68.	1980
6.1.3.6	Scott Model 216 total hydrocarbon analyzer with an FID detector.	1981
6.1.3.7	Turner Spectro fluorometer Model 430.	1981
6.1.3.8	Precision Scientific low temperature incubator Model 815.	1982
6.1.3.9	YSI Model 32 conductance meter.	1983
6.1.3.10	Radiometer pH M84 pH meter with an ABU 80 auto burette and a TTT 80 auto-titrator.	1983
6.1.3.11	Orion 901 ionanalyzer with a variety of ion selective electrodes.	1983
6.1.3.12	3 EP toxicity tumbler extractors	1983
6.1.3.13	Hach Model 2100 A turbidimeter.	1984
6.1.3.14	Milton Roy Spectronic 501 UV-VIS spectrophotometer with flow cell.	1986
6.1.3.15	Six TCLP zero headspace extractors (ZHE).	1989/90

		Year Acquired
6.1.3.16	YSI Model 3500 pH meter.	1990
6.1.4	Sample Preparation	
6.1.4.1	IEC International Centrifuge model CS.	1973
6.1.4.2	Fisher electrically heated waterbath with controller.	1983
6.1.4.3	ACE Glass mega soxhlet extractor model 6810-H including mantle, tripod and controller.	1983
6.1.4.4	Burrell wrist-action shaker Model 75.	1987
6.1.4.5	Tekmar sonic disruptor Model TM 500.	1987
6.1.4.6	Fisher electrically heated waterbath with controller.	1988
6.1.4.7	ACE Glass mega soxhlet extractor model 6810-H including mantle, tripod and controller.	1990
6.1.4.8	Waters Gel Permeation Chromatography Clean-up apparatus consisting of: Model 501 HPLC pump; Model 486 Tunable absorbance detector; Model 712 WISP; Waters fraction collector.	1991

6.1.5 Gas Chromatography

		Year Acquired
6.1.5.1	Hewlett-Packard Model 5880A dual column research gas chromatograph with data acquisition system including TC, FID and EC detectors, capillary column capability with flow splitter, gas sampling valve and Model 7671A auto injector.	1982
6.1.5.2	Tracor Model 565 mega bore capillary gas chromatograph with Hall conductivity, H.Nu photoionization, and flame ionization detectors.	1982
6.1.5.3	Chemical Data Systems 320-011 microprocessor controlled dual trap concentrator (purge and trap, and air and solids analysis for volatile organic compounds).	1983
6.1.5.4	HP Model 5840 capillary gas chromatograph with flame ionization and electron capture detectors; Model 7671A automatic liquid sample injector; Model 7675A purge and trap sampler.	1983
6.1.5.5	Tekmar LSC-2 and ALS microprocessor controlled ten station auto purge and trap.	1986
6.1.5.6	Shimadzu gas chromatograph Model GC-9A with dual FID; autosampler AOC-9A.	1987
6.1.5.7	Shimadzu capillary gas chromatograph Model GC-14A with electron capture and flame ionization detectors; AOC-9A autosampler.	1988

		Year Acquired
6.1.5.8	Tracor Model 540 mega-bore capillary GC with a Hall electroconductivity detector and a H.Nu photoionization detector; Tekmar LSC 2000 purge and trap; Dynatech PTA-30 autosampler.	1988
6.1.5.9	Club AT 386 IBM compatible personal computer with 2000K RAM and a 60 MB hard drive equipped with the Maxima 820 Version 3.3 chromatography software.	1988
6.1.5.10	Shimadzu mega-bore capillary gas chromatograph Model GC-14A with H.Nu photo ionization and flame ionization detectors, Tekmar LSC 2000 purge and trap, and ALS 2016 autosampler.	1990
6.1.5.11	NEC Powermate XS Plus IBM compatible personal computer with 2000K RAM and a 45 MB hard drive equipped with the Maxima 820 Version 3.3 chromatography software.	1990
6.1.5.12	NEC Powermate 286 IBM compatible personal computer with 2000K RAM and 30 MB hard drive equipped with the Maxima 820 Version 3.3 chromatography software as a process only station.	1990
6.1.5.13	Varian Model 3400 packed column gas chromatograph with flame ionization detector, oxidation/reduction catalyst system and Model 4400 integrator.	1991

		Year Acquired
6.1.5.14	Varian Model 3300 packed column gas chromatograph with flame ionization and flame photometric detectors and Model 4400 integrator.	1991
6.1.5.15	Shimadzu capillary gas chromatograph Model GC-14A with flame ionization detector; CR501 data processor with FDD-1A disk drive.	1991

Year
Acquired

6.1.6 Gas Chromatography/Mass Spectrometry

This laboratory is equipped with three GC/MS systems.

6.1.6.1 Finnigan MAT Incos 50 quadrupole MS/DS (Mass Spectrometer/Data Systems) with the following features: 1987

Heated electron ionization (EI) ion source, quadrupole mass analyzer, SuperIncos data system with 71 MB Winchester-type disk drive, 5 1/4" 360 Kb floppy disk drive, 20 MB streamer tape drive, NBS/EPA library, SuperIncos software AutoQuan™ automated target compound analysis software, and Form Master™ software. Printronix MVP printer.

Factory interfaced, data system controlled Varian Model 3400 gas chromatograph with Grob-type split/splitless capillary injector.

Subambient CO_2 cooling for Varian GC.

Varian 8035 liquid autosampler.

		Year Acquired
6.1.6.2	Finnigan MAT Incos 50 quadrupole MS/DS (Mass Spectrometer/Data Systems) with the following features:	1988

Heated electron ionization (EI) ion source, quadrupole mass analyzer, SuperIncos data system with 71 MB Winchester-type disk drive, 5 1/4" 360 Kb floppy disk drive, 20 MB streamer tape drive, NBS/EPA library, SuperIncos software AutoQuan™ automated target compound analysis software, and Form Master™ software. Printronix MVP printer.

Factory interfaced, data system controlled Varian Model 3400 gas chromatograph with Grob-type split/splitless capillary injector.

Subambient CO_2 cooling for Varian GC.

Tekmar 4200 automatic heated sample module.
Tekmar 4000 Dynamic Headspace concentrator.

		Year Acquired
6.1.6.2	Finnigan ITS40 MS/DS (Mass Spectrometer/Data Systems) with the following features:	1991

Heated electron ionization (EI) ion source, ion trap mass analyzer, ITS 40 data system on Compaq 386/20e computer, VGA monitor, 3 1/2" 1.4 MB floppy disk drive, 110 MB hardrive, NIST library, Epson FX850 printer.

Factory interfaced, data system controlled Varian Model 3400 gas chromatograph with Grob-type split/splitless capillary injector.

Subambient N_2 cooling for Varian GC.

Tekmar ALS2016 sample module.
Tekmar LSC2000 concentrator.

		Year Acquired
6.1.7	Fuel	
6.1.7.1	Preiser-Mineco residual moisture oven.	1981
6.1.7.2	Precision Scientific flash point apparatus.	1981
6.1.7.3	Sartorius 1303MP top loading balance.	1981
6.1.7.4	Parr oxygen bombs.	1981
6.1.7.5	Adiabatic calorimeter.	1981
6.1.7.6	Lindberg carbon hydrogen analyzer.	1982
6.1.7.7	Preiser-Mineco ash fusion furnace.	1982
6.1.7.8	Preiser-Mineco volatile matter analyzer.	1982
6.1.7.9	Leco sulfur analyzer.	1983
6.1.7.10	Gilson sieve analyzer.	1983
6.1.7.11	Mettler AE160 electronic analytical balance.	1985
6.1.7.12	Size reduction equipment.	1988
6.1.7.13	Dohrmann DX-20B halides analyzer.	1988
6.1.7.14	Dohrmann DC180 organic carbon analyzer.	1989

		Year Acquired
6.1.8	Balances	
6.1.8.1	Mettler H51 analytical balance	1978
6.1.8.2	Mettler H10 analytical balance	1978
6.1.8.3	Cahn instruments microbalance	1979
6.1.8.4	Sartorius analytical balance	1980
6.1.8.5	O'Haus triple beam balance	1981
6.1.8.6	Sartorius 1203MP analytical balance	1981
6.1.8.7	Pelouze 0100 100lb balance	1986
6.1.8.8	Mettler AE160 analytical balance	1986
6.1.8.9	Mettler AE200 analytical balance	1987
6.1.8.10	Sargent-Welch TL12000 analytical balance	1991
6.1.9	Microbiology	
6.1.9.1	Precision GCA Model 2EG incubator.	1986
6.1.9.2	American Sterilizer Model 576A.	1986
6.1.9.3	Precision coliform incubator bath.	1986
6.1.9.4	Quebec dark field colony counter.	1986

6.2 Instrument Maintenance

Each instrument has a suggested maintenance schedule. Preventative maintenance is practiced on all instrumentation at Interpoll Laboratories. An active preventative maintenance program will minimize degradation of the quality of analytical results or instrument failure. All instrumentation is on a yearly maintenance schedule unless more frequent servicing is required. Maintenance schedules are found in the front of each instrument log. Instrument maintenance and repair activities are documented in the instrument log kept with each instrument. Balances are serviced by an outside vendor on an annual basis.

6.3 Instrument Calibration

In addition to the proper maintenance, calibration of an instrument is essential if accurate data are to be generated. In some cases, the instrument must be tuned to optimize sensitivity. In others, it is necessary to analyze a set of known standards to produce a response curve. The frequency with which these procedures must be carried out is dictated by the instrument manual and/or the analytical method employed. In the case of instrument tuning that is not done on a daily basis, a label is placed on the instrument indicating when the procedure was last done and when it is due to be repeated. Records of calibrations done in conjunction with the analysis of a batch of samples are filed as part of the data associated with that batch.

7 DATA GENERATION

7.1 Sample Collection

7.1.1 Results obtained are only as valid as the sample which is submitted to the laboratory for analysis, therefore, it is essential that all individuals who collect samples be familiar with proper sampling techniques. All sampling is done in accordance with Interpoll Laboratories Sampling Protocol SOP. When samples are to be collected by the client, sampling requirements are discussed with the client before samples are collected to ensure that adequate sample is collected and appropriate containers are used. At that time, also, arrangements are made for proper preservation and storage to maintain the integrity of the sample.

7.1.2 Interpoll Laboratories Sampling Services division has the capabilities to perform many monitoring and sampling services.

7.1.2.1 Groundwater monitoring is performed in accordance with standard operating procedures developed by Interpoll Laboratories. These procedures are based on the latest MPCA/EPA protocols and are routinely updated by our staff. Complete documentation of all field procedures along with the analysis of trip blanks, field blanks, and duplicated well samplings is an integral part of Interpoll Laboratories quality assurance program. Groundwater monitoring includes groundwater sampling, leachate sampling, vadose zone (Lysimeter) sampling, drinking water and residential well sampling, and surface water sampling.

7.1.2.2 Wastewater monitoring includes wastewater sampling and flow monitoring. Interpoll Laboratories field technicians provide wastewater sampling and flow monitoring in any type of manhole or open channel flow setting. Interpoll Laboratories confined-space entry teams are trained and equipped to provide a professional approach to all safety-related issues. Our teams are equipped with remote gas sensing equipment, SCBA, rescue lifts, and lifelines at all times.

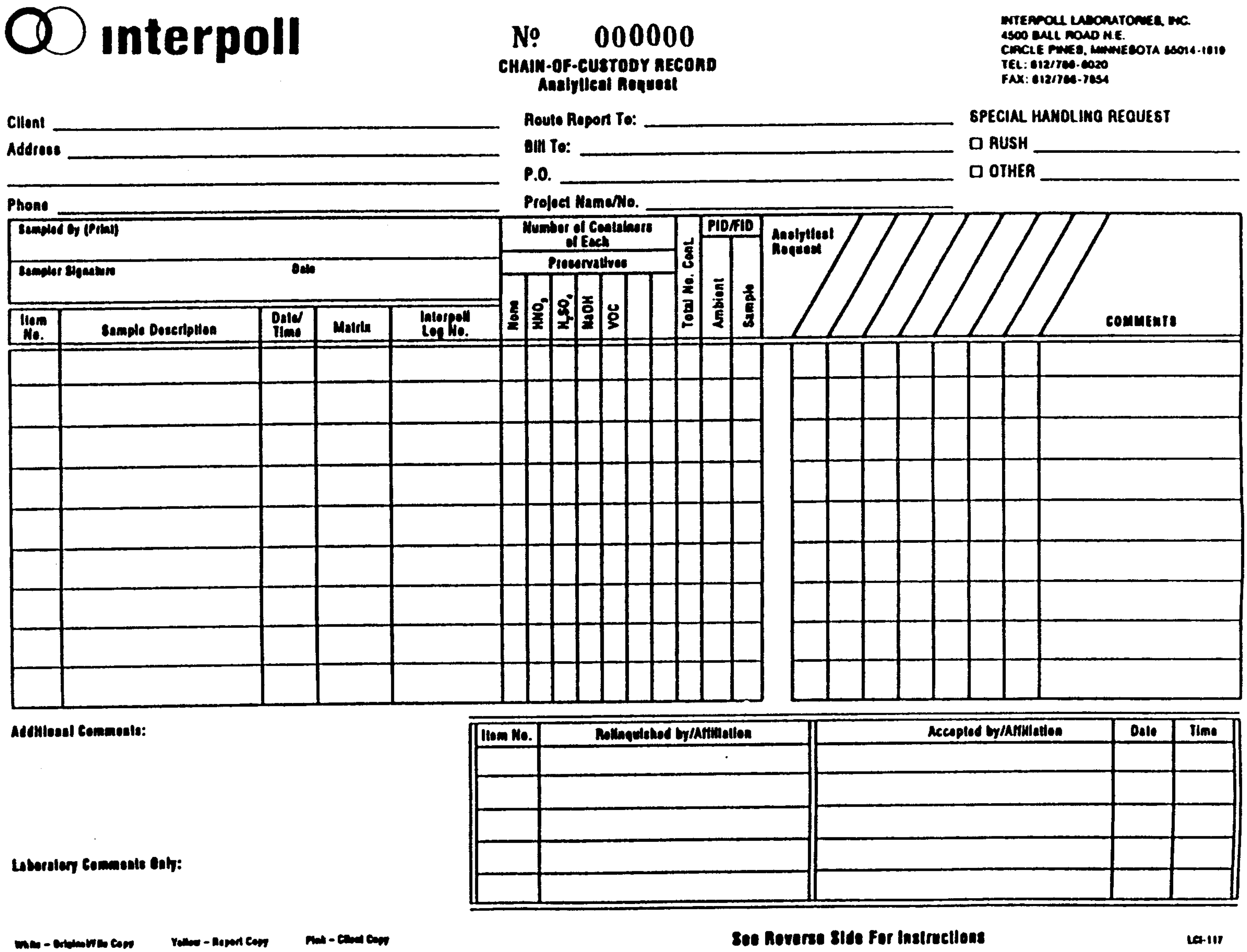
7.1.2.3 Additional field services include soil sampling, PCB contact swipe sampling, air and gas sampling, hazardous waste collection and waste composition studies.

7.2 Chain of Custody

Chain of custody procedures create a paper trail for the sample, documenting the life-span from collection until the generation of the analytical report.

Samples are collected, labeled and secured in the field. A chain of custody form is completed by the client or the sampling staff and forwarded, along with the samples, to the laboratory for analysis. Samples are accepted from sampling staff or courier and signed over to Interpoll Laboratories personnel in sample receiving. The sample receiving area is locked when unattended. When samples are relinquished to the analyst, the analyst signs the "Internal Chain-of-Custody Sample Log" acknowledging that he has received the sample. Interpoll Laboratories' chain-of-custody forms follow (Figures 7-1, 7-2 and 7-3).

The analyst's laboratory notebook and bench work sheets serve as the continuation of the paper trail. As such, each page of these media are initialed and dated by the analyst. At any point where the analysis of the sample is interrupted, the analyst will note the date and the storage location of the sample. This storage location will be the sample receiving area or another secured area of the laboratory.



interpoll

№ 000000

CHAIN-OF-CUSTODY RECORD
Analytical Request

INTERPOLL LABORATORIES, INC.
4500 BALL ROAD N.E.
CIRCLE PINES, MINNESOTA 55014-1819
TEL: 612/786-6020
FAX: 612/786-7854

Client ______________________

Address ______________________

____

Phone ______________________

Route Report To: ______________________

Bill To: ______________________

P.O. ______________________

Project Name/No. ______________________

SPECIAL HANDLING REQUEST

☐ RUSH ______________________

☐ OTHER ______________________

Sampled By (Print)

Sampler Signature Date

					Number of Containers of Each								PID/FID		Analytical Request							
					Preservatives																	
Item No.	Sample Description	Date/Time	Matrix	Interpoll Log No.	None	HNO_3	H_2SO_4	NaOH	VOC			Total No. Cont.	Ambient	Sample								COMMENTS

Additional Comments:

Laboratory Comments Only:

Item No.	Relinquished by/Affiliation	Accepted by/Affiliation	Date	Time

White – Original/File Copy Yellow – Report Copy Pink – Client Copy

See Reverse Side For Instructions

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Figure 7-1. Chain of Custody Record

Figure 7-2. Sample Custody Transfer Form

Interpoll Laboratories
(612)786-6020

Sample Custody Transfer Form

Job__________ Date__________

Interlaboratory transfer:
Person in custody of samples__________ Init._____
Person to receive custody __________ Init._____

Out of laboratory transfer:
Date shipped __________ Date received__________
Method of shipment__________ Carrier__________
Person releasing samples__________ Init._____
Person receiving custody__________ Init._____

	Sample Log Number	Initials of Person in Custody of Samples	Initials of Person Receiving Samples
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
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.

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Figure 7-3. Internal Chain-of-Custody Sample Log

INTERPOLL LABORATORIES
(612) 786-6020

INTERNAL CHAIN-OF-CUSTODY SAMPLE LOG

RECEIVED						RETURNED					
Client	Log No.	Aliquot	By	Date	Time	Sample	Extract	Other	Date	Time	Comments

Aliquot: Metals=M IC cations=IC-C IC anions=IC-A Fuel=F VOC vials=VOC General organic=O
Inorganic unpreserved=I Other inorganic splits =specify parameter

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7.3 Sample Preservation

The correct preservation technique is important in maintaining the integrity of the sample. The purposes and applicability of various preservation methods are described in Table 7-1. Tables 7-2 through 7-8 list Environmental Protection Agency Recommendations for sampling and sample preservation for various parameters.

7.4 Sample Documentation

Upon receipt at the laboratory, the sample description is entered into the Sample Log Notebook. Each sample batch is given a sequential batch number (0001 to 9999), and each sample within that batch acquires a sequence number (starting with 01); resulting in a unique number for each sample, i.e., XXXX-XX. This number is entered into the Sample Log Notebook next to the appropriate description. The sample number is placed on each container from that sampling location.

After sample numbers have been assigned, a data packet is prepared for the sample set. Analytical request/report sheets are selected for the required analytical work, and the parameters requested are indicated. An example is given as Figure 7-4. A "Forms Distribution Summary" (Figure 7-5) is prepared indicating which request sheets have been distributed. Chain of custody forms and invoicing information are included in the sample packet.

The request/report sheets are sent to the appropriate analytical area, and the remainder of the packet is filed in the sample receiving area. The request/report sheet is returned to the receiving area when the analysis is complete. When all data from a given group has been returned to the receiving area, that portion of the packet is sent to the appropriate Department Manager and the Quality Assurance Coordinator for review.

Table 7-1. Purpose and Applicability of Preservation Methods.

Preservative	Action	Applicable to:
Acid (HNO_3)	Metals solvent, prevents precipitation	Metals
Acid (H_2SO_4)	Bacterial inhibitor	Organic samples (COD, oil & grease organic carbon), Nitrogen-phosphorus forms
	Salt formation with organic bases	Ammonia, amines
Alkali (NaOH)	Salt formation with volatile compounds	Cyanides, organic acids
Refrigeration	Bacterial inhibitor, retards chemical reaction rates	Acidity-alkalinity organic materials, BOD, color, odor, organic P, organic N, carbon, etc., biological organism (coliform, etc.)

In summary, refrigeration at temperatures near 4 degrees celsius is the best preservation technique available, but it is not applicable to all types of samples.

Table 7-2. Required Containers, Sample Sizes, Preservation Techniques, Holding Times, Methodologies, and Detection Limits for Interpoll Laboratories General Inorganic Parameters.

Parameter	Container'	Minimum Sample Volume Required Soil	Minimum Sample Volume Required Water	Maximum Holding Time	Preservation	EPA Method	Detection Limits Soil	Detection Limits Water	Sampling Code'
Acidity	P,G	10 g	100 mL	14 days	cool, 4°C	305.1	1 mg/Kg	10 mg/L	A
Alkalinity	P,G	10 g	100 mL	14 days	cool, 4°C	310.1	1 mg/Kg	0.1 mg/L	A
Bromide	P,G	10 g	100 mL	28 days	none required	300.0	1 mg/Kg	0.02 mg/L	A
Chloride	P,G	10 g	50 mL	28 days	none required	300.0	0.5 mg/Kg	0.01 mg/L	A
Chlorine, residual	P,G	NA	100 mL	analyze immediately	none required	Hach	NA	0.7 mg/L	A
Chlorophyll "A"	P,G	NA	100 mL	filter within 24 HRS, can be held for 3 WKS	freeze after filtration	10200H2'	NA	2.0 ug/L	A
Color	P,G	NA	100 mL	48 hours	cool, 4°C	110.3	NA	1 CU'	A
						Hach		5 CU'	A
Cyanide	P,G	20 g	100 mL	14 days'	cool 4°C, NaOH to pH >12 0.6 g ascorbic acid'	9010/335.2	0.4 mg/Kg	0.02 mg/L	B
Fluoride	P	10 g	50 mL	28 days	none required	300.0	0.75 mg/Kg	0.015 mg/L	C
						340.2	0.5 mg/Kg	0.1 mg/L	C
Hardness	P,G	NA	100 mL	6 months	HNO_3 to pH < 2	130.2	NA	1 mg/L	D
Iodide	P,G	NA	100 mL	24 hours	cool, 4°C	345.1	NA	2 mg/L	A
Metals	P,G	10 g	100 mL	6 months	HNO_3 to pH <2	(See Table 7-3)			D

Table 7-2. Continued

Parameter	Container[1]	Minimum Sample Volume Required		Maximum Holding Time	Preservation	EPA Method	Detection Limits		Sampling Code[1]
		Soil	Water				Soil	Water	
Microbiology									
Fecal coliform	P,G	10 g	100 mL	6 hours	cool 4°C, 0.008% $Na_2S_2O_3$[3]	9222D[1]	100 col/g	1 col/100 mL	E
Total coliform	P,G	10 g	100 mL	6 hours	cool 4°C, 0.008% $Na_2S_2O_3$[3]	9222B[1]	100 col/g	1 col/100 mL	E
Plate count, total	P,G	NA	100 mL	6 hours	cool 4°C,	9215B[1]	NA	1 col/mL	E
Nitrogen									
Ammonia	P,G	20 g	100 mL	28 days	cool 4°C, H_2SO_4 to pH <2	350.2	2.5 mg/Kg	0.05 mg/L	F
Nitrate	P,G	20 g	100 mL	48 hours	cool 4°C	300.0 353.3	0.3 mg/Kg 2.50 mg/Kg	0.006 mg/L 0.05 mg/L	A A
Nitrate-nitrite	P,G	20 g	100 mL	28 days	cool 4°C, H_2SO_4 to pH <2	300.0	0.5 mg/Kg	0.01 mg/L	F
Nitrite	P,G	20 g	100 mL	48 hours	cool 4°C	300.0 354.1	0.15 mg/Kg 2.5 mg/Kg	0.003 mg/L 0.01 mg/L	A A
Organic	P,G	20 g	250 mL	28 days	cool 4°C, H_2SO_4 to pH <2			calculation	F
Total kjeldahl	P,G	20 g	250 mL	28 days	cool 4°C, H_2SO_4 to pH <2	351.3	5.0 mg/Kg	0.05 mg/L	F

Table 7-2. Continued

Parameter	Container[1]	Minimum Sample Volume Required Soil	 Water	Maximum Holding Time	Preservation	EPA Method	Detection Limits Soil	 Water	Sampling Code[1]
Oil & Grease	G	20 g	250 mL	28 days	cool 4°C, H_2SO_4 to pH <2	9071/413.1	5 mg/Kg	1 mg/L	G
Organic carbon	P,G	10 g	100 mL	28 days	cool 4°C, H_2SO_4 to pH <2	415.1	500 mg/Kg	0.1 mg/L	H
Oxygen demand									
Biological	P,G	NA	100 mL	48 hours	cool 4°C	507[1]	NA	4 mg/L	A
Biological carbonaceous	P,G	NA	100 mL	48 hours	cool 4°C	507 (5.E.6)[1]	NA	4 mg/L	A
Chemical (low)	P,G	20 g	100 mL	28 days	cool 4°C H_2SO_4 to pH <2	410.2	5 mg/Kg	0.4 mg/L	F
Chemical (medium)	P,G	20 g	100 mL	28 days	cool 4°C H_2SO_4 to pH <2	410.3	10 mg/Kg	2 mg/L	F
Chemical (high)	P,G	20 g	100 mL	28 days	cool 4°C H_2SO_4 to pH <2	410.1	25 mg/Kg	5 mg/L	F
Oxygen, dissolved									
Probe	G bottle and top	NA	300 mL	analyze immediately	none required	421F[1]	NA	0.1 mg/L	J
Winkler	G bottle and top	NA	300 mL	8 hours	none required	421F[1]	NA	0.1 mg/L	J
pH	P,G	10 g	25 mL	analyze immediately	none required	9045/150.1	0.01 pH units	0.01 pH units	A
Phenols	G only	20 g	250 mL	28 days	cool 4°C, H_2SO_4 to pH <2	9065/420.1	0.5 mg/Kg	0.005 mg/L	G
Phosphate	P,G	10 g	100 mL	28 days	cool 4°C	300.0	2.5 mg/Kg	0.05 mg/L	A

Table 7-2. Continued

Parameter	Container[1]	Minimum Sample Volume Required: Soil	Minimum Sample Volume Required: Water	Maximum Holding Time	Preservation	EPA Method	Detection Limits: Soil	Detection Limits: Water	Sampling Code[1]
Phosphorus, ortho	P,G	10 g	100 mL	48 hours	filter immediately, cool 4°C	365.2	1 mg/Kg	0.01 mg/L	A
						300.0	0.81 mg/Kg	0.016 mg/L	A
Phosphorus, total	P,G	20 g	50 mL	28 days	cool 4°C, H_2SO_4 to pH <2	365.2	1 mg/Kg	0.01 mg/L	F
Residue									
Filterable	P,G	NA	100 mL	7 days	cool 4°C	160.2	NA	10 mg/L	A
Non-filterable	P,G	NA	100 mL	7 days	cool 4°C	160.1	1 mg/Kg	4 mg/L	A
Settleable	P,G	NA	100 mL	48 hours	cool 4°C	160.5	NA	4 mg/L	A
Total	P,G	20 g	100 mL	7 days	cool 4°C	160.3	1 mg/Kg	10 mg/L	A
Total volatile	P,G	20 g	100 mL	7 days	cool 4°C	160.4	1 mg/Kg	10 mg/L	A
Silica	P	10 g	100 mL	28 days	cool 4°C	200.7	5 mg/Kg	0.020 mg/L	C
Specific conductance	P,G	NA	100 mL	28 days	cool 4°C	9050/120.1	NA	0.1 umho/cm	A
Sulfate	P,G	10 g	100 mL	28 days	cool 4°C	300.0	1.25 mg/Kg	0.025 mg/L	A
						9038/375.4	5 mg/Kg	1 mg/L	A
Sulfide	P,G	10 g	100 mL	7 days	cool 4°C, add zinc acetate plus sodium hydroxide to pH >9	9030/376.1	5 mg/Kg	1 mg/L	K
Sulfite	P,G	10 g	100 mL	analyze immediately	cool 4°C	300.0	25 mg/Kg	0.5 mg/L	A
Surfactant	P,G	NA	100 mL	48 hours	cool 4°C	425.1	NA	0.025 mg/L	A
Temperature	P,G	NA	100 mL	analyze immediately	none required	Interpoll	NA	0.1°C	A
Turbidity	P,G	NA	50 mL	48 hours	cool 4°C	180.1	NA	0.1 NTU	A

Table 7-2. Notes

1. Polyethylene (P) or Glass (G).
2. Standard Methods, 17th Edition.
3. Should only be used in the presence of residual chlorine.
4. Color units.
5. Maximum holding time is 24 hours when sulfide is present.
6. Standard Methods, 16th Edition.
7. pH units.
8. Use the "Sampling Codes" to determine the total volume of water needed to perform the analyses of interest. To arrive at this volume, simply total the "Minimum Volume" given for each parameter of a particular Sampling Code. Note that the sample preservation required changes from one code to another. Therefore, separate samples must be collected for each code and cannot be shared between codes. Example: Alkalinity requires 100 mL of unpreserved sample (code "A") as does color (also code "A"); therefore, the total sample volume required for these two analyses is 200 mL. If cyanide also was required (code "B"), a separate bottle containing 100 mL would have been needed because of its specific preservation requirements.

   In the case of soil samples, no preservation is required. As a result, a single sample can be taken, but the weight of soil required must again be a total of the minimum amounts needed for each individual parameter.

   Also note that certain parameters specifically require only plastic or only glass containers as indicated by either a "P" or "G." In other cases, either plastic or glass may be used as indicated by a "P,G."

Table 7-3. Interpoll Laboratories Metals Detection Limits and Analytical Methods.

A polyethylene (P) or glass (G) container may be used for the collection of samples requiring the determination of the metals listed below. A minimum sample volume of 200 mL is required for water and 20 grams for soil. Preservation for all metals (unless otherwise specified) is HNO_3 to pH <2 with a holding time of six months for both water and soil samples.

	Flame/AA			Inductively Coupled Plasma			Graphite Furnace/AA		
	EPA Method/	Detection Limit		EPA Method/	Detection Limit		EPA Method/	Detection Limit	
Parameter	SW-846	mg/Kg	mg/L	SW-846	mg/Kg	ug/L	SW-846	mg/Kg	ug/L
Aluminum	202.1/7020	200	1.0	200.7/6010	5.0	25	202.2	0.2	1.0
Antimony	204.1/7040	100	0.5	200.7/6010	1.0	5.0	204.2/7041	0.1	0.5
Arsenic	NA	NA	NA	200.7/6010	10	50	206.2/7060	0.1	0.5
Barium	208.1/7080	80	0.4	200.7/6010	0.2	1.0	200.2	0.02	0.1
Beryllium	210.1/7090	6.0	0.03	200.7/6010	0.2	1.0	210.2/7091	0.005	0.01
Boron	NA	NA	NA	200.7	1.2	6.0	NA	NA	NA
Cadmium	213.1/7130	6.0	0.03	200.7/6010	0.4	2.0	213.2/7131	0.005	0.01
Calcium	215.1/7140	18	0.09	200.7/6010	2.0	10	NA	NA	NA
Chromium	218.1/7190	10	0.05	200.7/6010	1.0	5.0	218.2/7191	0.2	1.0
Chromium VI[1]	7196[2]	0.5	0.005	NA	NA	NA	NA	NA	NA
Cobalt	219.1/7200	20	0.1	200.7/6010	0.6	3.0	219.2/7201	0.02	0.1
Copper	220.1/7210	10	0.05	200.7/6010	2.0	10	220.2	0.02	0.08
Iron	236.1/7380	20	0.1	200.7/6010	1.0	5.0	236.2	0.02	0.1
Lead	239.1/7420	40	0.2	200.7/6010	5.0	25	239.2/7421	0.04	0.2

[1] The maximum holding time for hexavalent chromium is 24 hours. Samples must be cooled to 4°C for preservation.

[2] Colorimetric method.

NA = no approved methodology exists for analysis by this instrument.

Table 7-3. Continued

	Flame/AA			Inductively Coupled Plasma			Graphite Furnace/AA		
	EPA Method/	Detection Limit		EPA Method/	Detection Limit		EPA Method/	Detection Limit	
Parameter	SW-846	mg/Kg	mg/L	SW-846	mg/Kg	ug/L	SW-846	mg/Kg	ug/L
Magnesium	242.1/7450	1.0	0.005	200.7/6010	0.2	1.0	NA	NA	NA
Manganese	243.1/7460	10	0.05	200.7/6010	0.2	1.0	243.2	0.008	0.04
Mercury[1]	NA	NA	NA	NA	NA	NA	245.2/7470	0.01	0.2
Molybdenum	246.1/7480	80	0.4	200.7/6010	1.0	5.0	246.2/7481	0.04	0.2
Nickel	249.1/7520	20	0.1	200.7/6010	0.4	2.0	249.2	0.06	0.3
Potassium	250.1/7610	8.0	0.04	200.7/6010	80	400	NA	NA	NA
Selenium	NA	NA	NA	200.7/6010	10	50	270.2/7740	0.1	0.5
Silicon	370.1	400	2.0	200.7/6010	2.0	10	NA	NA	NA
Silver	272.1/7760	12	0.06	200.7/6010	0.6	3.0	272.2	0.006	0.03
Sodium	273.1/7770	4.0	0.02	200.7/6010	2.0	10	273.2	0.004	0.02
Strontium	326A[2]	10	0.05	NA	NA	NA	NA	NA	NA
Thallium	279.1/7840	100	0.5	200.7/6010	10	50	279.2/7841	0.1	0.5
Tin	282.1/7870	800	4.0	200.7/6010	3.0	15	282.2	0.04	0.2
Titanium	283.1	400	2.0	NA	NA	NA	283.2	0.2	1.0
Vanadium	286.1/7910	160	0.8	200.7/6010	0.2	1.0	286.2/7911	0.12	0.6
Zinc	289.1/7950	2.0	0.01	200.7/6010	0.8	4.0	289.2	0.0004	0.002

[1] Mercury is analyzed by Cold Vapor Atomic Absorption (CVAA). The maximum holding time is 28 days. Samples must be preserved with HNO_3 to pH <2.

[2] Standard Methods, 16th Edition.

NA = no approved methodology exists for analysis by this instrument.

Table 7-4. Interpoll Laboratories Fuel Laboratory Analytical Methods.

No preservation or sampling container requirements are specified in ASTM methodology.

Parameter	Method	Detect. Limit
1. Proximate Analysis of Coal (Solid Fuels)	ASTM D3172	-
1.1 Preparation (part of moisture	ASTM D2013	-
1.2 Moisture	ASTM D3173, D3303 · Drying Oven	0.05%
1.3 Ash	ASTM D3174 Muffle Furnace	0.05%
1.4 Calorific Value (BTU)	ASTM D2015 Adiabatic Bomb Calorimeter	% BTU/LB
1.5 Volatile Matter	ASTM D3175 Heat at Controlled Conditions	0.1%
1.6 Fixed Carbon	By subtraction (100-(% moisture, ash, volatile matter))	
2. Short Proximate	(A condensed, combined analysis for II-8231 moisture, ash, BTU, sulfur.)	
3. Ultimate Analysis of Coal		
3.1 Moisture, Ash, Calorific Value	As in proximate analysis	
3.2 Carbon and Hydrogen	ASTM D3178 Hi temp furnace	0.05%
3.3 Sulfur	Fritz and Freeland, Anal. Chem 26(10), 1593-1595, (1954) Bomb wash titration	0.05%

Table 7-4. Continued

Parameter		Method	Detect. Limit
	3.4 Sulfur (alternate)	ASTM D1552 Hi temp furnace	
	3.5 Nitrogen	ASTM D3179 Kjeldahl-Gunning	0.03%
	3.6 Oxygen	By subtraction (100-(% C, H, S, N, Ash, Moisture))	
4.	Miscellaneous Coal (Solid Fuel) Procedures		
	4.1 Calculation of Coal Analyses to Different Bases	ASTM D3180	
	4.2 Free Swelling Index	ASTM D720 Flame Heat	-
	4.3 Sieve Analysis	ASTM D410 Sieve Shaker	-
	4.4 Fusibility of Ash	ASTM D1587 Ash Cone	20°F
	4.5 Loss-On-Ignition	ASTM D3174, II-823 Muffle Furnace	0.05%
5.	Analysis of Tar and Liquid Petroleum Products		
	5.1 Specific Gravity	ASTM D1298, II-8225 Hydrometer	0.0005
	5.2 Flashpoint (closed cup)	ASTM D93, II-8221 Pensky-Martens	2°F + 200° 5°MT 200°
	5.3 Heat of Combination	ASTM D240 Bomb Calorimeter	5 BTU/LB
	5.4 Sulfur	Fritz and Freeland, Anal. Chem 26(10), 1593-1595, (1954) Bomb wash titration	0.05%

Table 7-4. Continued

Parameter	Method	Detect. Limit
5.5 Sulfur (alternate)	ASTM D1552 Hi Temp Furnace	
5.6 Prep. for CL⁻	Analysis ASTM D808, II-8223 Bomb Wash	-
5.7 Prep. for Pb Analysis	ASTM D2788, II-8222 Bomb Wash	-
5.8 Ash	ASTM D482, II-8220 (Mod.) Muffle	0.05%
5.9 Sulfates Ash	ASTM D874, II-8220 Muffle	0.05%
5.10 Water in Petroleum	ASTM D95, II-8227 Distillation	0.1%
5.11 Acidity of Oils	ASTM D1093, II-8224 Centrifuge	0.1
5.12 Sediment in Oils	ASTM D473 Extract with Toluene	0.05%
5.13 PCB	(Done by Organic Lab)	
5.14 Viscosity	ASTM D445 (Sent to Commercial Testing)	-
5.15 Pour Point	ASTM D97 (Sent to Commercial Testing)	-

Table 7-4. Continued

Parameter	Method	Detect. Limit
6. Miscellaneous Procedures		
6.1 Carbonate	II-8212 Acid Evaluation	0.05%
6.2 Equilibrium Moisture	ASTM D1412, II-8228 Humidity Bath	0.05%
6.3 Four-point Moisture	ASTM C25, II-8230	0.05%

Table 7-5. Organic methodologies, detection limits, container types, sample volumes required and preservation techniques.

Water samples for volatiles analyses are to be collected in I-Chem 300 series (or equivalent) 40 mL amber glass VOA screw cap containers equipped with teflon-faced septa. A minimum of two VOA containers completely filled without headspace or air bubbles are required for each analysis. VOA samples cannot be shared between analysis, i.e., two vials are needed for 465-C and two additional vials are needed for 8240, etc. Four drops of concentrated HCl are added to each vial as preservative. Samples are stored at 4°C.

Solid samples for volatile analyses are to be collected in I-Chem 300 series (or equivalent) 125 mL or larger amber glass wide mouth bottles equipped with teflon-faced screw caps. A single bottle filled as completely as possible to minimize voids and headspace is required for analysis. No preservative is added to the bottle. Samples are stored at 4°C. The maximum holding time prior to analysis is 14 days.

	Volatiles Detection Limits									
	601/602	8010/8020		MDH 465-C		502.2		624	8240	
	(GC/Hall PID)	(GC/Hall PID)		(GC/Hall PID)		(GC/Hall PID)		(GC/MS)	(GC/MS)	
Parameter	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L
Acetone	N	N	N	2.8	22	N	N	N	5.0	50
Acrolein	N	N	N	N	N	N	N	N	1.3	13
Acrylonitrile	N	N	N	N	N	N	N	N	0.48	4.8
Allyl chloride	N	N	N	0.044	0.35	N	N	N	N	N
Benzene	0.47	0.59	0.47	0.059	0.47	0.059	0.47	1.0	0.10	1.0
Bromobenzene	N	N	N	N	N	0.052	0.42	N	N	N
Bromochloromethane	N	N	N	N	N	0.039	0.31	N	N	N
Bromodichloromethane	0.56	0.070	0.56	0.070	0.56	0.070	0.56	1.6	0.16	1.6
Bromoform	0.39	0.049	0.39	0.049	0.39	0.049	0.39	2.4	0.24	2.4
Bromomethane	0.23	0.029	0.23	0.029	0.23	0.029	0.23	2.1	0.21	2.1
2-Butanone (MEK)	N	N	N	0.49	3.9	N	N	N	0.24	2.4

N - Not listed as an analyte for this particular method.

Table 7-5. Continued

	Volatiles Detection Limits									
	601/602	8010/8020		MDH 465-C		502.2		624	8240	
	(GC/Hall PID)	(GC/Hall PID)		(GC/Hall PID)		(GC/Hall PID)		(GC/MS)	(GC/MS)	
Parameter	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L
n-Butylbenzene	N	N	N	N	N	0.034	0.27	N	N	N
sec-Butylbenzene	N	N	N	N	N	0.029	0.23	N	N	N
tert-Butylbenzene	N	N	N	N	N	0.051	0.41	N	N	N
Carbon disulfide	N	N	N	N	N	N	N	N	0.17	1.7
Carbon tetrachloride	0.44	0.055	0.44	0.055	0.44	0.055	0.44	1.7	0.17	1.7
Chlorobenzene	0.23	0.029	0.23	0.029	0.23	0.029	0.23	1.0	0.10	1.0
Chloroethane	0.26	0.032	0.26	0.032	0.26	0.032	0.26	1.5	0.15	1.5
2-Chloroethyl vinyl ether	0.70	0.088	0.70	0.088	0.70	N	N	2.3	0.23	2.3
Chloroform	0.33	0.041	0.33	0.041	0.33	0.041	0.33	1.0	0.10	1.0
Chloromethane	1.4	0.17	1.4	0.17	1.4	0.17	1.4	3.2	0.32	3.2
2-Chlorotoluene	N	N	N	N	N	0.036	0.29	N	N	N
4-Chlorotoluene	N	N	N	N	N	0.025	0.20	N	N	N
Dibromochloromethane	1.1	0.14	1.1	0.14	1.1	0.14	1.1	7.5	0.75	7.5
1,2-Dibromo-3-chloropropane	N	N	N	N	N	0.042	0.34	N	N	N
Dibromomethane	N	N	N	0.062	0.50	0.062	0.50	1.0	0.10	1.0
1,2-Dibromoethane	N	N	N	0.032	0.26	0.032	0.26	N	N	N
1,2-Dichlorobenzene	0.49	0.061	0.49	0.061	0.49	0.061	0.49	N	0.10	1.0
1,3-Dichlorobenzene	0.46	0.058	0.46	0.058	0.46	0.058	0.46	N	0.10	1.0
1,4-Dichlorobenzene	0.69	0.086	0.69	0.086	0.69	0.086	0.69	N	0.10	1.0
1,4-Dichloro-2-butane	N	N	N	N	N	N	N	N	0.16	1.6

N = Not listed as an analyte for this particular method.

Table 7-5. Continued

Parameter	Volatiles Detection Limits									
	601/602 (GC/Hall PID)	8010/8020 (GC/Hall PID)		MDH 465-C (GC/Hall PID)		502.2 (GC/Hall PID)		624 (GC/MS)	8240 (GC/MS)	
	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L
Dichlorodifluoromethane	0.91	0.11	0.91	0.11	0.91	0.11	0.91	N	0.32	3.2
1,1-Dichloroethane	0.16	0.020	0.16	0.020	0.16	0.020	0.16	1.7	0.17	1.7
1,2-Dichloroethane	0.47	0.059	0.47	0.059	0.47	0.059	0.47	1.3	0.13	1.3
1,1-Dichloroethene	0.66	0.082	0.66	0.082	0.66	0.082	0.66	1.6	0.16	1.6
cis-1,2-Dichloroethene	0.28	0.035	0.28	0.035	0.28	0.035	0.28	N	N	N
trans-1,2-Dichloroethene	0.28	0.035	0.28	0.035	0.28	0.035	0.28	1.3	0.13	1.3
1,2-Dichloropropane	0.35	0.044	0.35	0.044	0.35	0.044	0.35	1.3	0.13	1.3
1,3-Dichloropropane	N	N	N	0.048	0.38	0.048	0.38	N	N	N
2,2-Dichloropropane	N	N	N	N	N	0.062	0.50	N	N	N
1,1-Dichloro-1-propene	N	N	N	0.022	0.18	0.022	0.18	N	N	N
2,3-Dichloro-1-propene	N	N	N	0.044	0.35	N	N	N	N	N
cis-1,3-Dichloropropene	0.19	0.024	0.19	0.024	0.19	N	N	4.0	0.40	4.0
trans-1,3-Dichloropropene	0.18	0.022	0.18	0.022	0.18	N	N	1.2	0.12	1.2
Ethanol	N	N	N	N	N	N	N	N	5.0	50
Ethyl ether	N	N	N	0.14	1.1	N	N	N	N	N
Ethylbenzene	0.42	0.052	0.42	0.052	0.42	0.052	0.42	1.0	0.10	1.0
Ethylmethacrylate	N	N	N	N	N	N	N	N	0.30	3.0
Hexachlorobutadiene	N	N	N	N	N	0.018	0.14	N	N	N
2-Hexanone	N	N	N	N	N	N	N	N	1.0	10

N = Not listed as an analyte for this particular method.

Table 7-5. Continued

Volatiles Detection Limits

	601/602	8010/8020		MDH 465-C		502.2		624	8240	
	(GC/Hall PID)	(GC/Hall PID)		(GC/Hall PID)		(GC/Hall PID)		(GC/MS)	(GC/MS)	
Parameter	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L
Iodomethane	N	N	N	N	N	N	N	N	0.20	2.0
Isopropylbenzene (cumene)	N	N	N	0.22	1.8	0.22	1.8	N	N	N
p-Isopropyltoluene	N	N	N	N	N	0.021	0.17	N	N	N
Methylene chloride	3.0	0.38	3.0	0.38	3.0	0.38	3.0	4.8	0.48	4.8
4-Methyl-2-pentanone (MIBK)	N	N	N	0.20	1.6	N	N	N	0.91	9.1
Naphthalene	N	N	N	N	N	0.070	0.56	N	N	N
Pentachloroethane	N	N	N	0.21	1.7	N	N	N	N	N
n-Propylbenzene	N	N	N	N	N	0.030	0.24	N	N	N
Styrene	N	N	N	N	N	0.044	0.35	N	0.10	1.0
Tetrachloroethene	0.45	0.056	0.45	0.056	0.45	0.056	0.45	8.0	0.80	8.0
Tetrahydrofuran	N	N	N	1.1	8.7	N	N	N	N	N
Toluene	0.92	0.11	0.92	0.11	0.92	0.11	0.92	1.0	0.10	1.0
1,1,1,2-Tetrachloroethane	N	N	N	0.038	0.30	0.038	0.30	N	N	N
1,1,2,2-Tetrachloroethane	2.1	0.27	2.1	0.27	2.1	0.27	2.1	1.5	0.15	1.5
1,2,3-Trichlorobenzene	N	N	N	N	N	0.038	0.30	N	0.53	5.3
1,2,4-Trichlorobenzene	N	N	N	N	N	0.031	0.25	N	N	N
1,1,1-Trichloroethane	1.4	0.18	1.4	0.18	1.4	0.18	1.4	2.6	0.26	2.6
1,1,2-Trichloroethane	1.0	0.13	1.0	0.13	1.0	0.13	1.0	6.0	0.60	6.0
Trichloroethene	0.58	0.072	0.58	0.072	0.58	0.072	0.58	2.1	0.21	2.1
Trichlorofluoromethane	0.87	0.11	0.87	0.11	0.87	0.11	0.87	1.2	0.12	1.2

N = Not listed as an analyte for this particular method.

Table 7-5. Continued

	Volatiles Detection Limits									
	601/602 (GC/Hall PID)	8010/8020 (GC/Hall PID)		MDH 465-C (GC/Hall PID)		502.2 (GC/Hall PID)		624 (GC/MS)	8240 (GC/MS)	
Parameter	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L
1,2,3-Trichloropropane	N	N	N	0.072	0.58	0.072	0.58	N	0.53	5.3
1,1,2-Trichloro-2,2,1-trifluoroethane	N	N	N	0.11	0.90	N	N	N	N	N
1,2,4-Trimethylbenzene	N	N	N	N	N	0.038	0.30	N	N	N
1,3,5-Trimethylbenzene	N	N	N	N	N	0.026	0.21	N	N	N
Vinyl acetate	N	N	N	N	N	N	N	N	0.58	5.8
Vinyl chloride	0.30	0.038	0.30	0.038	0.30	0.038	0.30	1.6	0.16	1.6
Total xylenes	2.2	0.28	2.2	0.28	2.2	0.28	2.2	N	0.10	1.0

N = Not listed as an analyte for this particular method.

Table 7-6. Organic methodologies, detection limits, container types, sample volumes required and preservation techniques.

Water samples for semi-volatile analyses are to be collected in I-Chem 300 series (or equivalent) 1000 mL amber glass bottles equippped with teflon-faced screw caps. At least one full bottle is needed for each analysis. Semi-volatile samples cannot be shared between analyses, i.e., one full bottle is needed for Method 608 and another full bottle is needed for Method 610. No preservative is added to the bottle. Samples are stored at 4°C.

Solid samples for semi-volatile analyses are to be collected in I-Chem 300 series (or equivalent) 125 mL or larger amber glass wide mouth bottles equipped with teflon-faced screw caps. A single full bottle is requied for analysis. No preservative is added to the bottle. Samples are stored at 4°C. The maximum holding times are seven days until extraction, forty days after extraction.

Semivolatile Detection Limits

Parameter	625 (GC/MS)	8270 (GC/MS)		608 (GC/ECD)	8080 (GC/ECD)		610 (HPLC)	8310 (HPLC)	
	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg
Acenaphthene	1.0	1.0	0.033	N	N	N	0.18	0.18	0.0060
Acenaphthylene	0.87	0.87	0.029	N	N	N	0.21	0.21	0.0070
Acetophenone	N	1.6	0.053	N	N	N	N	N	N
Aldrin	4.0	4.0	0.13	0.0040	0.0040	0.00013	N	N	N
4-Aminobiphenyl	N	13	0.43	N	N	N	N	N	N
Aniline	N	1.3	0.043	N	N	N	N	N	N
Anthracene	0.20	0.20	0.0067	N	N	N	0.0059	0.0059	0.00020
alpha-BHC	5.3	5.3	0.18	0.033	0.033	0.0011	N	N	N
beta-BHC	9.4	9.4	0.31	0.024	0.024	0.0080	N	N	N
delta-BHC	5.9	5.9	0.20	0.038	0.038	0.0013	N	N	N
gamma-BHC (Lindane)	8.1	8.1	0.27	0.030	0.030	0.0010	N	N	N

N = Not listed as an analyte for this particular method.

Table 7-6. Continued

	Semivolatile Detection Limits								
	625	8270		608	8080		610	8310	
	(GC/MS)	(GC/MS)		(GC/ECD)	(GC/ECD)		(HPLC)	(HPLC)	
Parameter	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg
Benzidine	14	14	0.47	N	N	N	N	N	N
Benzo(a)anthracene	1.8	1.8	0.060	N	N	N	0.0089	0.0089	0.00030
Benzo(a)pyrene	0.39	0.39	0.013	N	N	N	0.00054	0.00054	0.000018
Benzo(b)fluoranthene	0.99	0.99	0.033	N	N	N	0.00040	0.00040	0.000013
Benzo(g,h,i)perylene	1.2	1.2	0.040	N	N	N	0.015	0.015	0.00050
Benzo(k)fluoranthene	1.7	1.7	0.057	N	N	N	0.0020	0.0020	0.000067
Benzoic acid	N	75	2.5	N	N	N	N	N	N
Benzyl alcohol	N	2.5	0.083	N	N	N	N	N	N
Bis(2-chloroethoxy)methane	1.8	1.8	0.060	N	N	N	N	N	N
Bis(2-chloroethyl)ether	0.40	0.40	0.013	N	N	N	N	N	N
Bis(2-chloroisopropyl)ether	1.4	1.4	0.047	N	N	N	N	N	N
Bis(2-ethylhexyl)phthalate	2.5	2.5	0.083	N	N	N	N	N	N
4-Bromophenyl phenyl ether	2.6	2.6	0.087	N	N	N	N	N	N
Butylbenzyl phthalate	0.98	0.98	0.033	N	N	N	N	N	N
Chlordane	10	10	0.33	0.084	0.084	0.0028	N	N	N
4-Chloroaniline	N	3.9	0.13	N	N	N	N	N	N
4-Chloro-3-methylphenol	2.4	2.4	0.080	N	N	N	N	N	N
1-Chloronaphthalene	N	4.2	0.14	N	N	N	N	N	N
2-Chloronaphthalene	0.51	0.51	0.017	N	N	N	N	N	N

N = Not listed as an analyte for this particular method.

Table 7-6. Continued

Parameter	625 (GC/MS) ug/L	8270 (GC/MS) ug/L	8270 (GC/MS) mg/Kg	608 (GC/ECD) ug/L	8080 (GC/ECD) ug/L	8080 (GC/ECD) mg/Kg	610 (HPLC) ug/L	8310 (HPLC) ug/L	8310 (HPLC) mg/Kg
	Semivolatile Detection Limits								
2-Chlorophenol	1.2	1.2	0.040	N	N	N	N	N	N
4-Chlorophenyl phenyl ether	0.40	0.40	0.013	N	N	N	N	N	N
Chrysene	0.33	0.33	0.011	N	N	N	0.020	0.020	0.00067
4,4'-DDD	2.4	2.4	0.080	0.21	0.21	0.0070	N	N	N
4,4'-DDE	4.2	4.2	0.14	0.070	0.070	0.0023	N	N	N
4,4'-DDT	2.8	2.8	0.093	0.25	0.25	0.0083	N	N	N
Di-n-butyl phthalate	1.4	1.4	0.047	N	N	N	N	N	N
Di-n-octyl phthalate	2.8	2.8	0.093	N	N	N	N	N	N
Dibenz(a,j)acridine	N	30	1.0	N	N	N	N	N	N
Dibenzo(a,h)anthracene	0.88	0.88	0.029	N	N	N	0.0021	0.0021	0.000070
Dibenzofuran	N	2.5	0.083	N	N	N	N	N	N
1,2-Dichlorobenzene	2.1	2.1	0.070	N	N	N	N	N	N
1,3-Dichlorobenzene	1.8	1.8	0.060	N	N	N	N	N	N
1,4-Dichlorobenzene	2.5	2.5	0.083	N	N	N	N	N	N
3,3'-Dichlorobenzidine	6.2	6.2	0.21	N	N	N	N	N	N
2,4-Dichlorophenol	2.6	2.6	0.087	N	N	N	N	N	N
2,6-Dichlorophenol	N	20	0.67	N	N	N	N	N	N
Dieldrin	3.3	3.3	0.11	0.080	0.080	0.0027	N	N	N
Diethyl phthalate	0.57	0.57	0.019	N	N	N	N	N	N

N = Not listed as an analyte for this particular method.

Table 7-6. Continued

	Semivolatile Detection Limits								
	625 (GC/MS)	8270 (GC/MS)		608 (GC/ECD)	8080 (GC/ECD)		610 (HPLC)	8310 (HPLC)	
Parameter	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg
P-Dimethylaminoazobenzene	N	5.1	0.17	N	N	N	N	N	N
7,12-Dimethylbenz(a)anthracene	N	2.0	0.067	N	N	N	N	N	N
R,R'-Dimethylphenethylamine	N	53	1.8	N	N	N	N	N	N
Dimethyl phthalate	1.8	1.8	0.060	N	N	N	N	N	N
2,4-Dimethylphenol	3.5	2.4	0.080	N	N	N	N	N	N
4,6-Dinitro-2-methyl phenol	2.8	2.8	0.093	N	N	N	N	N	N
2,4-Dinitrophenol	2.3	2.3	0.077	N	N	N	N	N	N
2,4-Dinitrotoluene	2.6	2.6	0.087	N	N	N	N	N	N
2,6-Dinitrotoluene	1.8	1.8	0.060	N	N	N	N	N	N
Diphenylamine	N	2.5	0.083	N	N	N	N	N	N
1,2-Diphenylhydrazine	2.4	2.4	0.080	N	N	N	N	N	N
Endosulfan I	27	27	0.90	0.047	0.047	0.0016	N	N	N
Endosulfan II	26	26	0.87	0.062	0.062	0.0021	N	N	N
Endosulfan sulfate	15	15	0.50	0.21	0.21	0.0070	N	N	N
Endrin aldehyde	8.6	8.6	0.29	0.18	0.18	0.0060	N	N	N
Endrin	21	21	0.70	0.065	0.065	0.0022	N	N	N
Ethyl methanesulfonate	N	4.2	0.14	N	N	N	N	N	N
Fluoranthene	1.2	1.2	0.040	N	N	N	0.015	0.015	0.00050
Fluorene	1.0	1.0	0.033	N	N	N	0.013	0.013	0.00043

N = Not listed as an analyte for this particular method.

Table 7-6. Continued

	Semivolatile Detection Limits								
	625 (GC/MS)	8270 (GC/MS)		608 (GC/ECD)	8080 (GC/ECD)		610 (HPLC)	8310 (HPLC)	
Parameter	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg
Heptachlor	3.6	3.6	0.12	0.0070	0.0070	0.00023	N	N	N
Heptachlor epoxide	35	35	1.2	0.019	0.019	0.00063	N	N	N
Hexachlorobenzene	1.9	1.9	0.063	N	N	N	N	N	N
Hexachlorobutadiene	2.9	2.9	0.097	N	N	N	N	N	N
Hexachlorocyclopentadiene	2.8	2.8	0.093	N	N	N	N	N	N
Hexachloroethane	1.2	1.2	0.040	N	N	N	N	N	N
Indeno(1,2,3-cd)pyrene	0.90	0.90	0.030	N	N	N	0.021	0.021	0.00070
Isophorone	1.1	1.1	0.037	N	N	N	N	N	N
Methoxychlor	10	10	0.33	0.22	0.22	0.0073	N	N	N
3-Methylcholanthrene	N	8.2	0.27	N	N	N	N	N	N
Methyl methanesulfonate	N	2.7	0.090	N	N	N	N	N	N
2-Methylnaphthalene	N	8.3	0.28	N	N	N	N	N	N
4-Methyl phenol	N	0.90	0.030	N	N	N	N	N	N
2-Methyl phenol	N	0.90	0.030	N	N	N	N	N	N
Naphthalene	1.5	1.5	0.050	N	N	N	0.18	0.18	0.0060
1-Naphthylamine	N	6.4	0.21	N	N	N	N	N	N
2-Naphthylamine	N	6.2	0.21	N	N	N	N	N	N
2-Nitroaniline	N	4.8	0.16	N	N	N	N	N	N
3-Nitroaniline	N	10	0.33	N	N	N	N	N	N
4-Nitroaniline	N	10	0.33	N	N	N	N	N	N

N = Not listed as an analyte for this particular method.

Table 7-6. Continued

	Semivolatile Detection Limits								
	625	8270		608	8080		610	8310	
	(GC/MS)	(GC/MS)		(GC/ECD)	(GC/ECD)		(HPLC)	(HPLC)	
Parameter	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg
Nitrobenzene	1.4	1.4	0.047	N	N	N	N	N	N
2-Nitrophenol	1.1	1.1	0.037	N	N	N	N	N	N
4-Nitrophenol	2.1	2.1	0.070	N	N	N	N	N	N
n-Nitroso-di-n-propylamine	2.9	2.9	0.097	N	N	N	N	N	N
N-Nitroso-dibutylamine	N	1.8	0.060	N	N	N	N	N	N
n-Nitroso-dimethylamine	14	14	0.47	N	N	N	N	N	N
n-Nitroso-diphenylamine	0.78	0.78	0.026	N	N	N	N	N	N
N-Nitrosopiperidine	N	2.2	0.073	N	N	N	N	N	N
PCB-1016	17	17	0.57	0.10	0.10	0.0033	N	N	N
PCB-1221	6.8	6.8	0.23	0.10	0.10	0.0033	N	N	N
PCB-1232	12	12	0.40	0.10	0.10	0.0033	N	N	N
PCB-1242	23	23	0.77	0.10	0.10	0.0033	N	N	N
PCB-1248	38	38	1.3	0.10	0.10	0.0033	N	N	N
PCB-1254	50	50	1.7	0.10	0.10	0.0033	N	N	N
PCB-1260	51	51	1.7	0.10	0.10	0.0033	N	N	N
Pentachlorobenzene	N	3.6	0.12	N	N	N	N	N	N
Pentachloronitrobenzene	N	3.7	0.12	N	N	N	N	N	N
Pentachlorophenol	2.8	2.8	0.093	N	N	N	N	N	N
Phenacetin	N	7.2	0.24	N	N	N	N	N	N
Phenanthrene	0.40	0.40	0.013	N	N	N	0.0051	0.0051	0.00017

N = Not listed as an analyte for this particular method.

Table 7-6. Continued

	Semivolatile Detection Limits								
	625 (GC/MS)	8270 (GC/MS)		608 (GC/ECD)	8080 (GC/ECD)		610 (HPLC)	8310 (HPLC)	
Parameter	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg	ug/L	ug/L	mg/Kg
Phenol	4.2	4.2	0.14	N	N	N	N	N	N
2-Picoline	N	6.0	0.20	N	N	N	N	N	N
Pronamide	N	3.6	0.12	N	N	N	N	N	N
Pyrene	1.5	1.5	0.050	N	N	N	0.094	0.094	0.0031
1,2,4,5-Tetrachlorobenzene	N	13	0.43	N	N	N	N	N	N
2,3,4,6-Tetrachlorophenol	N	4.5	0.15	N	N	N	N	N	N
Toxaphene	270	270	9.0	0.56	0.56	0.019	N	N	N
1,2,4-Trichlorobenzene	2.4	2.4	0.080	N	N	N	N	N	N
2,4,5-Trichlorophenol	N	4.4	0.15	N	N	N	N	N	N
2,4,6-Trichlorophenol	1.9	1.9	0.063	N	N	N	N	N	N

N = Not listed as an analyte for this particular method.

Table 7-7. Organic methodologies, detection limits, container types, sample volumes required and preservation techniques.

Water samples for herbicides analyses are to be collected in I-Chem 300 series (or equivalent) 1000 mL amber glass bottles equipped with teflon-faced screw caps. At least one full bottle is needed for each analysis. Semi-volatile samples cannot be shared between analyses, i.e., one full bottle is needed for Method 608 and another full bottle is needed for Method 610. No preservative is added to the bottle. Samples are stored at 4^0C.

Solid samples for herbicides analyses are to be collected in I-Chem 300 series (or equivalent) 125 mL or larger amber glass wide mouth bottles equipped with teflon-faced screw caps. A single full bottle is required for analysis. No preservative is added to the bottle. Samples are stored at 4^0C.

The maximum holding times for all Table 7-7 analysis are seven days to extraction, forty days after extraction.

	Herbicide Detection Limits		
	615 (GC/ECD)	8150 (GC/ECD)	
Parameter	ug/L	ug/L	mg/Kg
2,4-D	0.33	0.33	0.0041
2,4-DB	2.0	2.0	0.024
2,4,5-T	0.30	0.30	0.0038
Dalapon	5.8	5.8	0.072
Dicamba	19	19	0.24
Dichloroprop	0.53	0.53	0.0066
Dinoseb	58	58	0.72
MCPA	25	25	0.31
MCPP	80	80	1.0
Picloram	6.3	6.3	0.078
Silvex	0.33	0.33	0.0041

N = Not listed as an analyte for this particular method.

Table 7-8. Organic methodologies, detection limits, container types, sample volumes required and preservation techniques.

See Table 7-5 for container types, sample volumes required, preservation techniques, and holding times needed for Total Hydrocarbons as Gasoline. See Table 7-6 for container types, sample volumes required, preservation techniques, and holding times needed for Total Hydrocarbons as Fuel Oil.

	Total Hydrocarbons Detection Limits			
	5030/8015 (P&T GC/FID)		3510/3550/8015 (GC/FID)	
Parameter	ug/L	mg/Kg	ug/L	mg/Kg
As Fuel Oil	N	N	43	1.4
As Gasoline	10	1.2	N	N

N = Not listed as an analyte for this particular method.

Figure 7-4. Inorganic Area Data Reporting Sheet

Due Date: ____________________

Interpoll Laboratories
(612)786-6020

Inorganic Area Data Reporting Sheet

CLIENT: ____________________
JOB: ____________________
CLIENT NO: ____________________
P.O. NO: ____________________
PROJECT MGR: ____________________

PHONE: ____________________
DATE: ____________________

CONTACT: ____________________

LABORATORY REPORT #: ____________________
SAMPLES COLLECTED: ____________________
SAMPLES RECEIVED: ____________________

	Invoicing	Signature	Report Routing
PL	☐	☐	☐
Lab Mgr	☐	☐	☐
Ino Mgr	☐	☐	☐
Org Mgr	☐	☐	☐

SAMPLE ID:				
SAMPLE TYPE:				
LOG NO:				

PARAMETER	UNITS	DETECT LIMIT	ANALYSIS DATE & INITIALS	METHOD				
Acidity								
Alkalinity								
Cyanide								
Conductance								
Color								
COD								
Hardness								
Ammonia Nitrogen								
Organic Nitrogen								
Kjeldahl Nitrogen								
Nitrate Nitrogen								
Nitrite Nitrogen								
Oil & Grease								
pH								
Phenol								
Ortho Phosphorus								
Total Phosphorus								
Suspended Solids								
Total Solids								
Volatile Solids								
Sulfide								
Turbidity								

Footnotes: ____________________

In-House Comments:

LCI-17RR

Figure 7-5. Forms Distribution Summary

Due Date: ____________

Interpoll Laboratories, Inc.
(612)786-6020

Forms Distribution Summary

CLIENT: ______________________
____
____
____

PHONE: ______________________

CONTACT: ______________________

LABORATORY REPORT #: ____________
SAMPLES COLLECTED: ____________
SAMPLES RECEIVED: ____________

(DRS = "Data Reporting Sheet")

JOB: ______________________
CLIENT NO: ______________________
P.O. NO: ______________________
PROJECT MGR: ______________________

DATE: ______________________

	Invoicing	Signature	Report Routing
PL	☐	☐	☐
Lab Mgr	☐	☐	☐
InO Mgr	☐	☐	☐
Org Mgr	☐	☐	☐

Inorganic

___ ______________________
___ LCI-17R, Inorganic Area DRS (Fill out or add to corresponding form LCI-19R)

Metals

___ ______________________
___ LCI-04R, Metals DRS
___ LCI-07R, E.P. Toxicity DRS (Also fill out form LCI-37)
___ LCI-08R(3), EPA Waste Oil Profile DRS
___ LCI-09R(1), ASTM Leach DRS
___ LCI-15R, Mineral Ash DRS
___ LCI-34R(4), MPCA Waste Oil Profile DRS
___ LCI-36(1), Incineration Param. DRS

Fuel

___ ______________________
___ LCI-02R, Fuel DRS
___ LCI-08R(1), EPA Waste Oil Profile DRS
___ LCI-20, Fusion Temperature of Ash DRS
___ LCI-34R(1), MPCA Waste Oil Profile DRS
___ LCI-36(2), Incineration Param. DRS

Ion Chromatography

___ ______________________
___ LCI-08R(2), EPA Waste Oil Profile DRS
___ LCI-09R(2), ASTM Leach DRS
___ LCI-21R, Ion Chromatography DRS
___ LCI-34R(2), MPCA Waste Oil Profile DRS
___ LCI-36(3), Incineration Param. DRS

Organic

___ ______________________
___ LCI-05RR(1-2), VOA (Water) DRS
___ LCI-61R(1-2), VOA (Soil) DRS
___ LCI-06R, PAH DRS
___ LCI-18R, Phenols DRS
___ LCI-28, PCB DRS
___ LCI-29, Pesticide/Herbicide DRS
___ LCI-34R(3), MPCA Waste Oil Profile DRS
___ LCI-35R(1-5), Method 625 (Water) DRS
___ LCI-68(1-5), Method 625 (Soil) DRS
___ LCI-13, Request for GC/MS Analysis
___ LCI-57(1-3), MDH 465-C (Water) DRS
___ LCI-58(1-3), MDH 465-C (Soil) DRS
___ LCI-59(1-4), Method 502.2 (Water) DRS
___ LCI-60(1-4), Method 502.2 (Soil) DRS
___ LCI-62, BTX/Total Hydrocarbons (Water) DRS
___ LCI-63, BTX/Total Hydrocarbons (Soil) DRS
___ LCI-64(1-2), Method 624 (Water) DRS
___ LCI-65(1-2), Method 624 (Soil) DRS
___ LCI-66(1-3), Method 8240 (Water) DRS
___ LCI-67(1-3), Method 8240 (Soil) DRS
___ LCI-69, Phthalate DRS

Particle Sizing

___ ______________________
___ LCI-25, Request for Particle Size Analysis
___ LCI-26, Particle Size Distribution Analysis
___ LCI-27, Combining Particle Size Results

Comments:

LCI-30RR

After approval by the appropriate Department Manager and the Quality Assurance Coordinator, the packet is given to the Lab Secretary for typing. She then gives the report to the appropriate Department Managers for signatures. The signed report is sent to the appropriate individual(s). A copy of the report is kept in the client file, along with the data reporting sheets and all QC data associated with the project.

7.5 Analytical Methods

The use of recognized analytical methods is an important part of quality control. Use of a reliable and well documented method supports the validity of the results. At Interpoll Laboratories, EPA-approved methodologies are used whenever possible. Analyses for which no EPA method is available are completed following recognized methods which have been validated by other agencies or groups, such as American Society for Testing and Materials (ASTM), Association of Official Analytical Chemists (AOAC), American Public Health Association (APHA) and United States Geological Survey (USGS). Tables 7-2 through 7-8 list the methods currently in routine use at Interpoll Laboratories.

Each analytical area maintains a Standard Operating Procedures (SOP) manual which specifies all of the equipment, reagents and procedures associated with each analysis. These procedures are written in a standard format and are specific for the equipment and reagents at Interpoll Laboratories. An outline of the standard format is included as Figure 7-6.

The SOP manual is used to train new staff members and is also helpful to experienced analysts when a procedure is not performed frequently. The analysis is completed exactly as specified in the SOP. Any deviation, such as volume changes, are recorded on the lab work sheets so there will be no question about how a particular sample was handled.

Figure 7-6. Interpoll Laboratories Standard Operating Procedure General Outline.

1. APPLICATION

   A statement of the type of samples for which the method is applicable.

2. RANGE

   Detection limits and concentration range for which the method is useable.

3. SAMPLE HANDLING & PRESERVATION

   Description of how the samples are to be collected and stored prior to analysis.

4. SUMMARY OF METHOD

   Short explanation of the method.

5. INTERFERENCES

   List of interferences and how they are eliminated or compensated for.

6. APPARATUS AND GLASSWARE

   A list of the equipment the analyst will need.

7. REAGENTS

   Detailed instructions for the preparation and standardization of all reagents used in the method.

8. SAFETY

   List of any potential hazards associated with the reagents or procedures.

9. PROCEDURE

   A step-by-step description of how the method is carried out.

Figure 7-6 Continued

10. CALCULATION

Equations for processing raw data to obtain final results.

11. QUALITY CONTROL PROCEDURES

Description of when standards, spikes and duplicates should be analyzed. Also includes any special QC needed.

12. ACCURACY AND PRECISION

Recovery and relative difference control limits.

13. DATA PACKAGES

Description of data deliverables.

14. REFERENCES

7.6 Documentation of Analysis

The relevant information and results of all analyses are recorded on the appropriate laboratory work sheet. The work sheets are designed to fit the recording needs of individual types of analysis, but they all include the date of analysis, sample identification number, results and initials of the analyst. Results obtained for standards and blanks are also recorded on the work sheets. All laboratory work sheets are filed for future reference. All analysts are required to follow Interpoll labs standard operating proceedure for data deliverables to ensure data documentation is complete. The analyst will bring a completed data package to the appropriate laboratory manager for review and approval. Once approved, results can be reported to clients.

8 DATA PROCESSING

8.1 Expression of Results

Specific guidelines for the expression of laboratory results are followed in reporting analytical data. These guidelines facilitate the proper interpretation of results with reference to the accuracy of a test and standardize the reporting of data.

8.1.1 Units

Results are expressed in units appropriate for the sample submitted. For liquid samples, this is most often mg/L or ug/L. In the case of solid samples, mg/kg or % w/w are frequently used.

8.1.2 Significant Figures

Only significant figures are used for the reporting of laboratory results. The proper use of significant figures is an indication of the reliability and limits of the analytical method used. A value is reported in significant figures when it contains all digits known to be true and one last digit in doubt. For example, if a value is reported as 13.5 mg/L, the "13" is known to be true, while the "5" is uncertain and may be a "4" or "6."

Zeros may or may not be significant according to the following rules:

a. Final zeros after a decimal point are always significant figures. For example, the value 9.800 grams implies that the weight is known to the nearest milligram.

b. Zeros before a decimal point preceded by one or more digits are significant. For example, in the value 300.01, all zeros are significant.

c. Zeros positioned between at least two non-zero digits are always significant. The value 30.07 contains four significant figures.

8.1.3 Rounding Off Numbers

Analytical results are rounded off by dropping digits which are not significant according to the following rules:

a. If the digit 6, 7, 8 or 9 is dropped, the preceding digit is increased by one unit.

b. If the digit 0, 1, 2, 3 or 4 is dropped, the preceding digit is not altered.

c. If the digit 5 is dropped, the preceding number is rounded off to the nearest even number.

8.1.4 Calculations

The results of mathematical calculations involved in the expression of results are rounded off in significant figures according to the following rules:

a. When numbers are multiplied or divided, the results are rounded off to as few significant figures as are present in the factor with the fewest significant figures.

Example: $$\frac{23 \times 6.034 \times 4.3214}{1.734} = 345.87$$

This number would be rounded to 350.

b. When numbers are subtracted or added, the number that has the fewest decimal places limits the number of places carried in the sum or difference.

Example:

```
 0.0324
17.01
24.003
 0.01
-------
41.0554
```

This number would be rounded to 41.06

8.2 Data Reporting

Each sample that is received by the laboratory is given an identification number which is entered into the master Sample Log Notebook along with the sample description. When the analysis of a sample is completed and all of the data have been entered on the Data Reporting Sheet, results are forwarded to the clerical staff for typing. A copy of the Data Reporting Sheet is kept on file in the laboratory.

9 DATA QUALITY ASSESSMENT

The evaluation of daily performance is a vital link in the Quality Assurance Program. Systematic daily checks are required to demonstrate that reproducible results are being generated and that the method is measuring the actual concentration of sample analyte.

All quality control results must be approved before sample results are reported. The analysts must complete a "QC Data Report" form (Figures 9-1 and 9-2) containing the Method QC Checklist and all precision and recovery data for each batch of samples. If there are any out-of-control situations or special concerns, they must be noted on this form.

9.1 Quality Control Definitions

Interpoll Laboratories, Inc. provides a quality assurance/quality control (QA/QC) package designed to provide analytically sound and defensible data to our clients. This QA/QC package includes the method-monitoring samples which are used to indicate acceptable performance. These QC samples, along with their acceptance criteria, are defined below.

9.1.1 Method Blank

The method blank is an artificial sample containing all the reagents and compounds added to a real sample which measures the background contamination levels associated with each method.

9.1.1.1 Inorganic Analysis

If an inorganic method blank contains an analyte concentration which exceeds the detection limit, the source of contamination must be identified and corrective action taken before sample analysis is begun.

Figure 9-1. Analytical Method QC Checklist

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Analytical Method QC Checklist

Project Name: ____________________ Date: ____________________

Category: ____________________ Analyst: ____________________

Matrix: ____________________ Batch Numbers:

Prep Method #: ____________________ __________ __________

Analytical Method #: ____________________ __________ __________

Total Number of Samples Analyzed: __________ __________ __________

1. Values for all instrument blanks below detection limit: Yes ____ No ____

2. Reference standard analysis: Source ____________________

Analyte	Theoretical Value	Observed Value	Percent Recovery

3. Matrix Spikes

Number of spiked samples analyzed: __________ Average % Recovery: __________

Range of % Recovery: __________

Recovery of each spike within control limits: Yes ____ No ____

4. Duplicates

Number of duplicate samples analyzed: __________

Average Relative Percent Difference: __________

Range of Relative Percent Difference: __________

Precision for each set of duplicates within control limits: Yes ____ No ____

5. Method Blank

Number of method blanks analyzed: __________

All below detection limit: Yes ____ No ____

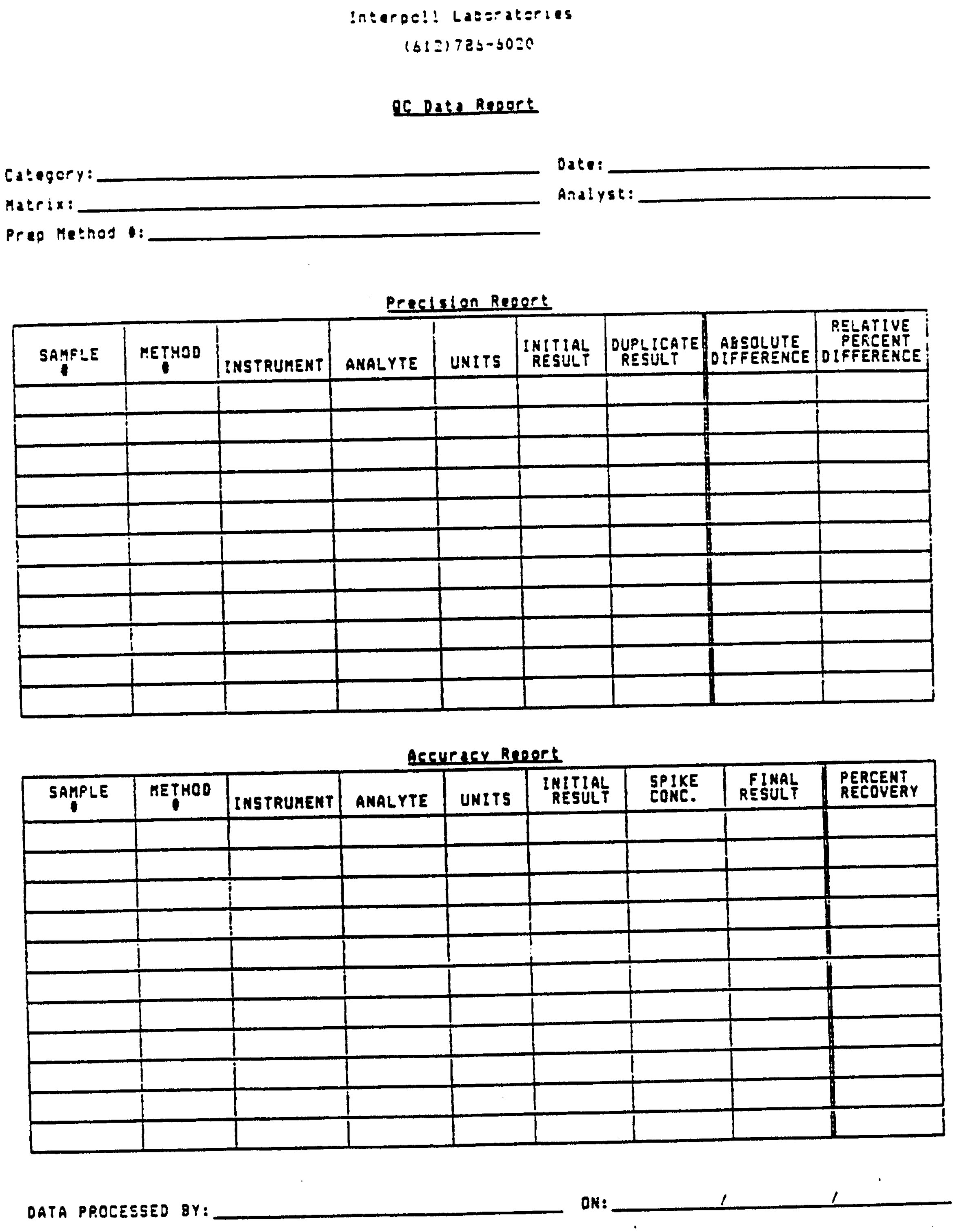
6. Calibration verified every ________ samples.

Comments: ______________________________

____

LQA-26RRR (b)

Figure 9-2. QC Data Report



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QC Data Report

Category: ____________________

Matrix: ____________________

Prep Method #: ____________________

Date: ____________________

Analyst: ____________________

Precision Report

SAMPLE #	METHOD #	INSTRUMENT	ANALYTE	UNITS	INITIAL RESULT	DUPLICATE RESULT	ABSOLUTE DIFFERENCE	RELATIVE PERCENT DIFFERENCE

Accuracy Report

SAMPLE #	METHOD #	INSTRUMENT	ANALYTE	UNITS	INITIAL RESULT	SPIKE CONC.	FINAL RESULT	PERCENT RECOVERY

DATA PROCESSED BY: ____________________ ON: ____/____/____

LQA-26RPR (a)

9.1.1.2 Organic Analysis

All analytes must be below the method detection limits except the following compounds:

Volatiles	Semivolatiles
Acetone	Bis (2-ethylhexyl) phthalate
MEK (2-Butanone)	Butyl benzyl phthalate
Methylene Chloride	Diethyl phthalate
Toluene	Dimethyl phthalate
	Di-n-butyl phthalate
	Di-n-octyl phthalate

The above commonly-detected laboratory contaminants may be present in the method blank at up to five times the detection limit. All results for samples prepared on the same day as the method blank are corrected for the contaminants found in the method blank.

9.1.2 Check Sample

A check sample is prepared and analyzed with each batch of samples to monitor the performance of the method and to validate the calibration curve. Known amounts of the analytes of interest are added to a matrix blank before it is taken through the analytical process with the samples. The analytes used to spike the check sample are obtained from a source other than those used to calibrate the instrument. If available, check samples may be purchased from a commercial source.

Intralaboratory recovery limits are developed for each method and are used to determine the efficiency of the analysis. EPA limits are used until intralaboratory limits can be calculated. If a check sample fails to meet the established criteria, the source of failure must be determined and the appropriate corrective action taken before sample analysis is begun.

9.1.3 Matrix Spike/Matrix Spike Duplicate

9.1.3.1 Inorganic Analysis

Ten percent of samples submitted are split into the original sample, duplicate sample, and a matrix spike. The matrix spike is spiked with a known amount of analyte and all three samples are prepared and analyzed according to the method.

9.1.3.2 Organic Analysis

Ten percent of samples submitted are split into the original sample, a matrix spike, and a matrix spike duplicate. The matrix spike and the matrix spike duplicate are spiked with a known amount of analyte and all three samples are prepared and analyzed according to the method.

The results from the spike and duplicate analysis are used to indicate matrix effects, as well as to gather precision and accuracy data for each parameter. Intralaboratory recovery limits are developed for each method and are used to determine the efficiency of the analysis. EPA limits are used until intralaboratory limits can be calculated. If the percent recovery or relative percent difference fails to meet the established criteria, the source of failure must be identified and corrective action taken. All samples analyzed in association with the failed matrix spike must be reanalyzed.

9.1.4 Surrogate Standards

Surrogate standards are compounds chemically similar to those being analyzed by GC/MS which are added to each sample, method blank, and matrix spike/matrix spike duplicate to monitor the efficiency of preparation and analysis. Table 9-1 summarizes the surrogates by analysis and lists the action criteria for each group of surrogates.

Table 9-1. GC/MS Surrogate Standards

INTERPOLL LABORATORIES, INC.

GC/MS SURROGATE STANDARDS

ANALYSIS	SURROGATE	ACTION CRITERIA	ACTION CRITERIA SOURCE
Volatiles	1,2-Dichloroethane-d_4 Toluene-d_8 Bromofluorobenzene	All surrogates must meet the recovery limits.	Intralaboratory Limits USEPA Program Statement of Work
Base/Neutrals	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14}	Two of the three surrogates must meet recovery limits. The third must have a recovery of 10% or greater.	Intralaboratory Limits USEPA Program Statement of Work
Acid	2-Fluorophenol Phenol-d_5 2,4,6-Tribromophenol	Two of the three surrogates must meet recovery limits. The third must have a recovery of 10% or greater.	Intralaboratory Limits USEPA Program Statement of Work

9.2 Precision

Precision is the reproducibility of a method when it is repeated on a homogeneous sample under controlled laboratory conditions. Precision does not take into account the fact that the observed values may be widely displaced from the true value as a result of systematic or constant errors.

Ten percent of the samples are analyzed in duplicate to document that the laboratory is generating reproducible results. The results of the duplicate samples are recorded on a "QC Data Report" form (Figure 9-2). Precision data are added to the Lotus 1-2-3 Precision database. Once a sufficient number of duplicate samples have been determined (a minimum of ten) over an extended period of laboratory time, the precision control limit is calculated for each parameter and the data are represented graphically as well as in tabular form.

At Interpoll Laboratories, precision is expressed either as absolute difference or as relative percent difference.

Absolute difference (AD) is reported when the sample result is less than ten times the method detection limit. Absolute difference is calculated as follows:

$$[\mathit{Sample\ Result} - \mathit{Replicate}\ ] = AD$$

The control limit is established by calculating the mean absolute difference and multiplying by a Shewart factor based on the number of observations in the sub-group. The control limit is recalculated quarterly using the most recent 36 data points.

Precision is expressed as relative percent difference for sample results greater than or equal to ten times the detection limit and is calculated as follows:

$$RPD = \frac{\mathit{Absolute\ Difference}}{\mathit{Average\ Value}} \times 100$$

The control limit is established by calculating the mean percent recovery and multiplying by a Shewart factor based on the number of observations in the sub-group, which in this case is two.

$$\overline{RPD} = \frac{\Sigma\ RPD}{N}$$

$$Control\ Limit = 3.27\ (\overline{RPD})$$

The control limit is recalculated quarterly using the most recent 36 data points.

9.3 Accuracy

Accuracy is the agreement between the amount of a component measured by the test method and the amount of that component actually present in the sample. A method may have excellent precision, but recover only a part of the element being determined because of poorly standardized solutions, inexact dilution techniques, inaccurate balance weights or improperly calibrated equipment. Conversely, a method may be accurate but lack precision because of poor instrument sensitivity or other factors beyond the control of the analyst.

A spiked sample is analyzed at a minimum of 10% to verify that accurate results are being generated in the laboratory. The results are recorded on a "QC Data Report" form (Figures 9-1 and 9-2). The accuracy data are entered into the Accuracy database. Once an adequate number of spiked samples have been analyzed (a minimum of ten), over an extended period of time, the upper and lower control limits for accuracy are determined for each parameter and control charts are generated.

Accuracy, expressed as percent recovery (PR), is calculated by the following equation:

$$PR = \frac{(Final\ Result) - (Initial\ Result)}{Spike\ Concentration} \times 100$$

where "Final Result" is the value obtained when the spiked sample was analyzed, "Initial Result" is the value obtained for the unspiked sample, and "Spike Concentration" is the known quantity which was added to the sample.

Control limits are based on the mean percent recovery and the standard deviation of the percent recovery over time. Mean percent recovery and standard deviation are calculated according to the following equations:

$$\text{Mean Percent Recovery } (\overline{PR}) = \frac{\Sigma\ PR}{N}$$

where N is the number of spiked samples analyzed.

$$\text{Standard Deviation} = \sqrt{\frac{\Sigma\ (PR - \overline{PR})^2}{N-1}}$$

The lower control limit for inorganic parameters is set at the mean percent recovery minus three standard deviations, and the upper control limit is set at the mean percent recovery plus three standard deviations.

$$LCL = \overline{PR} - 3(SD)$$

$$UCL = \overline{PR} + 3(SD)$$

The lower control limit for organic parameters is set at the mean percent recovery minus two standard deviations, and the upper control limit is set at the mean percent recovery plus two standard deviations.

$$LCL = \overline{PR} - 2(SD)$$

$$UCL = \overline{PR} + 2(SD)$$

These control limits are recalculated quarterly using the most recent 36 data points.

9.4 Sensitivity

The detection limits that are necessary for a given project are generally delineated at the outset. The method detection limit is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. Upon completion of the analytical work, the data is reviewed to ascertain that these detection limits have been met.

The method detection limit (MDL) is determined by the following procedure:

a. An estimate of the detection limit is made.

b. A laboratory standard is prepared at one to five times the estimated MDL.

c. A minimum of seven aliquots of this standard are processed through the entire analytical method. Results are calculated as described in the method and reported in the method reporting units.

d. Calculate the standard deviation of the results:

$$\textit{Standard Deviation} = \sqrt{\frac{\Sigma\ (X_i - \bar{X})^2}{N-1}}$$

where X_i is the analytical result, $\bar{x}$ is the mean value, and N is the number of aliquots analyzed.

e. Compute the MDL:

$$MDL = t \times (SD)$$

where t = the student's t value appropriate for a 99% confidence level and a standard deviation estimate for N-1 degrees of freedom (see Table 9-2) and SD is the standard deviation of replicate analyses.

Table 9-2. Table of Students' *t* Values at the 99 Percent Confidence Level

Number of Replicates	Degrees of Freedom (N-1)	*t*
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
~	~	2.326

9.5 Completeness

Completeness is calculated by the following equation:

$$Completeness\ (\%) = (S_R/S_L) \times 100$$

where S_R is the number of samples for which valid results are reported and S_L is the number of samples logged in for the project.

The QA goal for completeness is 100%. If this goal is not met, all deficiencies will be explained in the project report.

9.6 Standard Reference Materials

Reference samples are used as check samples. They are of known concentrations and are available from EPA and other sources. These check samples are analyzed periodically as a measure of the quality of the data produced by the analysts and the methods employed. They are also useful to verify the accuracy of the calibration standards. Recoveries of reference samples analyzed in conjunction with samples are used as an aid to data review.

10 CORRECTIVE ACTION

An out of control situation is defined as any value that falls outside the control limits. When an analysis is found to be out of control, the Department Manager will be notified and further analyses will not be performed until the problem is resolved. The problem and its solution are documented on Interpoll Laboratories Form LQA-70 "Corrective Action Form" (Figure 10-1). This form must be completed by the analyst and approved by the inorganic or organic Department Manager and the Quality Assurance Coordinator prior to the release of sample results. Depending on the nature of the situation, the Department Manager and/or Quality Assurance Coordinator may request that the samples involved be reanalyzed. This proceedure is outlined in the Data Package SOP given to each analyst to follow.

Figure 10-1. Out of Control Events Log

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CORRECTIVE ACTION FORM

ANALYST: ______________________ DATE: ____________

SUMMARY OF DISCREPANCY:

CORRECTIVE ACTION TAKEN:

LABORATORY MANAGER: ______________________ DATE: ____________

QA/QC MANAGER: ______________________ DATE: ____________

LQA-70

11 REFERENCES

11.1 Handbook for Sampling and Sample Preservation of Water and Wastewater, EPA 600/4-82-029.

11.2 Quality Assurance Handbook for Air Pollution Measurement Systems, Volume 1, EPA-600/9-76-0005.

11.3 "Methods for the Organics Analysis of Municipal and Industrial Wastewater", EPA-600/4-82-057.

11.4 Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019.

Appendix O: Safety Plan

Interpoll Laboratories
(612)786-6020

Safety Policy Statement

It is the policy of Interpoll Inc. that every operation shall be performed in the safest possible manner, so as to prevent injuries to persons and damaged property. To accomplish this, every reasonable effort will be made in the interest of accident prevention, fire prevention and health protection.

To implement this policy, Interpoll will continue all efforts to maintain a safe and healthful place to work. To the maximum extent possible, we will continue to provide safe working equipment, necessary safety protection devices, and first aid equipment. The control of accidents will continue to be considered as an integral part of our operations and the responsibility of every employee.

Since we have a large number of projects at any time which usually have only individual Interpoll employees or small teams of Interpoll employees on the project site, it is absolutely imperative that every Interpoll employee regard his safety and the safety of those in his area as part of his/her responsibilities. It is, however, particularly important that all supervisors, including lead technicians, pay special attention to the safety of those employees working under their supervision.

All work related accidents must be reported to your supervisor. It is the responsibility of the department head in charge of the operational area in which an accident happened to investigate the accident and report his findings to the safety committee of his office. It is then the responsibility of the department head, supervisor, and employee to make sure the necessary action to prevent similar accidents has been taken.

Each employee must review the safety requirements for his/her job. There are available manuals and guidelines relating to specific safety problem areas such as handling of hazardous materials. If you have any questions regarding the safe practices for any assigned task, check with your supervisor. Repeated failure to follow accepted safe practices or safety regulations may lead to termination of your employment with Interpoll.

Use of Intoxicants or Drugs

No Interpoll Employee is permitted to use intoxicants or to be under the influence of any intoxicant or drug while on the job. This includes anytime when operating a company vehicle. Failure to obey this policy may result in immediate termination of your employment with Interpoll.

General Safe Practices

Safety is the responsibility of everyone employed by Interpoll Inc. Our policy is to provide an environment for employees which is as healthful and safe as possible, establish and maintain effective safety procedures and practices in all company activities, and comply with applicable federal, state and local laws and standards.

The information in this statement about safe practices is for your protection. Read, understand and observe the procedures.

Accidents don't just happen; they are caused. Most are caused by the unsafe actions of people as a result of lack of knowledge, inattention and thoughtlessness. Lack of knowledge of these safe practices will not justify noncompliance.

1. Accidents or injuries must be reported immediately to your supervisor, no matter how minor they seem.
2. Know how to do your job. Check your work area to determine what problems or hazards may exist. Review the

safety requirements of each assigned job with your supervisor. Your activity may endanger other persons or nearby equipment or property. Take necessary steps to safeguard them.

3. Report unsafe equipment, hazardous conditions and unsafe acts to your supervisor.
4. Use the safety equipment specified for the job.
5. Practice good housekeeping in your work area.
6. For your protection, obey all warning signs such as "Keep Out", "No Smoking", "Eye Protection Required", and "Authorized Personnel Only".
7. Do not take shortcuts. Use ladders, ramps, stairways, and designated paths.
8. Do not engage in horseplay or roughhousing at any time. To do so may lead to injury and/or be cause for discharge.
9. No Interpoll employee is permitted to use intoxicants or to be under the influence of any intoxicant or drug while on the job. This includes anytime when operating a company vehicle. Failure to obey this policy may result in immediate termination of your employment with Interpoll.
10. When in doubt about safety equipment or procedures required to do your job, ask your supervisor of Department Head.
11. Failure to follow practices relating to your safety or that of fellow employees or failure to properly safeguard equipment, tools, or materials may lead to your discharge.

Housekeeping

It is the responsibility of all employees and supervisors to practice good housekeeping.

1. Place all rubbish in trash containers or if you are working at a job site away from your office, collect rubbish and dispose of it at the shop.
2. Return unused supplies to the shop at the completion of your job.
3. Do not leave tools and supplies where they will create a hazard to others. Put them in the proper tool box or return them to the shop.
4. Wipe up spilled liquids immediately.
5. Place oily rage in approved metal containers.
6. Keep change areas, toilets, wash-up facilities , and drinking fountains clean. They are provided for your convenience and comfort.

Personal Protective Equipment

Personal protective equipment can and is a life saver! When equipment has been specified for certain work assignments or areas, you must use it.

1. Hard hats must be worn on all work sites at all times by employees, subcontractor's personnel and visitors where there is any exposure to head injuries. When in doubt, ask your supervisor.
2. Wear clothing suitable for the work you are doing. Minimum attire is long pants and a T-shirt.
3. Wear sturdy work shoes. Safety shoes are desirable and are mandatory in areas where there are hazards to feet. Sneakers and sandals are not permitted. The cost of safety shoes will be reimbursed to you within certain restrictions. For details, contact your supervisor.
4. Wear proper eye protection when exposed to flying objects, dust, chemicals, or harmful rays.

5. Ear protection (muffs or plugs) must be worn al all high-noise level jobs. Cotton or other related material is not acceptable as an ear plug. When in doubt, ask your supervisor.
6. Respiratory equipment may be required in areas where health hazards exist due to accumulations of dust, fumes, mists, or vapors, particularly when sampling or handling samples containing hazardous materials.
7. Safety belts and lifelines must be work when other safeguards, such as nets, planking, or scaffolding, cannot be used. Be sure safety lines are independent of other rigging.
8. Wear gloves when handling material which could cut, tear, or burn your hands.
9. You must wear rubber boots for work in concrete or water.

Fire Protection and Prevention

Since Interpoll employees often may be working at a client's job site or plant, it is important to learn the procedures for reporting fires and local fire safety from the client's safety personnel.

At your own Interpoll office, become familiar with local procedures, exist, fire hazards and the location of fire fighting equipment.

1. Familiarize yourself with the location of all fire fighting equipment in your work area.
2. Tampering with fire fighting equipment is grounds for discharge.
3. Learn the classification of fires:

   Class A - Ordinary combustible materials such as wood, coal, paper, or fabrics where wetting and cooling is the method to extinguish the fire.

Class B - Flammable petroleum products or other flammable liquids where oxygen must be excluded to extinguish the fire.

Class C - Fires in or near energized electrical equipment where, because use of water would be hazardous, a "nonconducting" extinguishing agent must be used.

4. Only approved solvents should be used for cleaning and degreasing. The use of gasoline and similar flammable products for this purpose is prohibited.
5. Keep the work area neat. An orderly work area reduces the fire and accident hazard.
6. Where fire hazards are prevalent, get additional fire extinguishers and/or post a fire watch.
7. When you must weld or burn near combustible materials, move them, cover them with fire-resistant fabric, or wet them down. When in doubt, consult your supervisor.
8. Flammable and combustible liquids must be handled only in properly labeled safety cans.
9. Place oily rags in appropriate covered metal containers.
10. Do not attempt any work involving a source of ignition near a pit, sewer, drain, manhole, trench, or enclosed space where flammable gases may be present. Wait until tests have been made with a combustible gas indicator and the area has been declared safe for hot work.
11. Do not weld or cut on a tank or in an enclosure that has contained gasoline or other flammable gas or liquid unless it has been purged, and proved to be safe.
12. The use of open fires is prohibited unless specifically authorized by the responsible supervisor.

<u>Motor Vehicles</u>

1. Proof of valid motor vehicle license of the proper classification is required before you may operate a vehicle while employed by Interpoll.

2. Interpoll will obtain a motor vehicle report on all new employees. In addition, each employee who operates Interpoll vehicles as part of his/her job will have a motor vehicle report check annually.
3. No Interpoll employee is permitted to operate any motor vehicle on the job while under the influence of any intoxicant or drug.
4. You must obey all traffic laws and regulations while operating a motor vehicle on the job.
5. The driver of any Interpoll vehicle is responsible for properly securing any load and for periodically checking that the load has remained secure.
6. Do not ride in the bed of a truck containing materials which are not properly secured to prevent movement.
7. You are prohibited from riding on loads, fenders, running boards, or tailgates, or with legs or arms dangling over the sides.
8. Do not back up any vehicle or equipment when the view to the rear is obstructed unless a "spotter" signals that it is safe to do so.

First Aid

Interpoll employees often work on job sites where first aid equipment and facilities are furnished by the client. In those cases, be sure you are familiar with procedures and personnel necessary to obtain treatment.

When working in an isolated area, it is the Lead Technician's responsibility to be sure that a first aid kit is available. He/she should also familiarize himself/herself with the route to the closest emergency service.

All Interpoll offices have first aid equipment and are located near emergency facilities. Become familiar with the location of equipment and with the available emergency facilities.

Tell you supervisor if you have a physical restriction, such as diabetes, impiared eyesight or hearing, back or heart trouble, hernia or aversions to height. We do not expect you to do a job which might result in injury to yourself or others.

Never move an injured or seriously ill person unless necessary to prevent further injury.

If you are injured on the job, follow these procedures:

1. GET APPROPRIATE FIRST AID/OR EMERGENCY MEDICAL ATTENTION!
2. Contact your supervisor and our Insurance Claims Administrator as soon as possible.
3. If you receive emergency medical attention beyond first aid, the following procedures apply:
    a. Inform the doctor/hospital that it is a job related injury. If the medical facility needs additional information, have them contact Interpoll.
    b. Make arrangements for a doctor's report to be sent to Interpoll for use by the insurance company.
4. Before returning to work, you must present a doctor's permit. Your supervisor will not allow you back without one.
5. As soon after your initial treatment as you are able, contact your supervisor so that he/she may complete the accident report and we can report to the insurance company and appropriate state and federal agencies.

Electrical

Electrical hazards are a leading cause of accidents. To help avoid electrical accidents, you must obey the following practices:

1. All electrical tools and equipment must be grounded.
2. Power cords and tools should be inspected and repaired if faulty.
3. Turn in damaged or defective electrical tools immediately for repair or replacement.
4. Temporary electrical cords must be covered or elevated. They must be kept clear of walkways or other locations where they may be exposed to damage or create a tripping hazard.
5. Be alert for hazardous areas which are not properly marked.

Hand and Portable Power Tools

Only tools in safe working condition may be used. Comply with all of the manufacturers' instructions and observe the following safe practices:

1. Inspect tools daily and turn in damaged or defective tools for repairs or replacement.
2. Power saws, grinders, and other power tools must be operated with the proper guards in place. Removing guards or rendering them inoperative may be grounds for your discharge.
3. Cords and hoses must be kept out of walkways and off stairs and ladders.
4. When using tools listed below or working near others using such tools, you must use the additional personal protective equipment specified. If you have questions about the protective equipment, ask your supervisor.

Chipping Hammers, and Impact Wrenches	Eye Protection and Hearing Protection
Cutting Torches, and Arc Welders	Eye Protection and Hand Protection
Power-actuated Tools, Grinders Hand-held Chipping Hammers, Saws and Drills	Eye Protection

5. All portable electric tools must be grounded except Underwriters Laboratory approved, double-insulated tools.
6. Power cords and portable power tool cords must be inspected frequently for defects, and repaired or discarded.
7. Shut down all fuel-powered tools while refueling.
8. Smoking is prohibited during refueling operations, and other nearby sources of ignition must be halted during refueling.

Material Handling, Storage and Disposal

All material must be properly stacked and secured to prevent sliding, falling or collapse. Aisles, stairs and passageways must be kept clear to provide for the safe movement of employees and equipment and to provide access in emergencies.

1. Use proper lifting techniques when handling materials:
    a. Get down close to the load.
    b. Keep your back straight.
    c. Lift gradually, using your legs. Do not jerk or twist.
    d. Get help for bulky or heavy loads.
2. Stored materials must not block any exit from a building.
3. Pipe, casing, drill rod and similar supplies should be stored in racks or stacked and blocked to prevent movement.
4. Protruding nails must be bent or pulled when uncrating materials.

Ladders

Particularly when you are on a construction site, you may be required to use all types of ladders for access to your work area. Do not use unsafe or makeshift ladders. The following guidelines apply to the use of ladders provided by Interpoll and serve as general criteria to help evaluate the safety of ladders provided by the client or contractor.

1. Job-made ladders must be constructed to conform with the established standards.
2. Manufactured ladders used on the job will be the industrial grade type of heavy-duty ladder.
3. Broken or damaged ladders must not be used. Repair or destroy them immediately. Ladders to be repaired must be tagged "Do Not Use".
4. Do not splice together short ladders to make a longer ladder.
5. All straight ladders must be tied off at the top.
6. Ladders should not be placed against movable objects.
7. The base of the ladder must be set back a safe distance from the vertical approximately one-fourth of the working length of the ladder.
8. Ladders used for access to a floor or platform must extend at least three feet above the landing.
9. The areas around the top and base of ladders must be free of tripping hazards such as loose materials, trash and electric cords.
10. Ladders which project into passageways or doorways, where they could be struck by personnel, moving equipment, or materials being handled, must be protected by barricades or guards.
11. You must face the ladder at all times when ascending or descending.

12. Be sure that your shoes are free of mud, grease, or other substances which could cause a slip or fall.
13. Do not carry materials up a ladder. Use a hand line.
14. Always more the ladder to avail overreaching.
15. Step ladders must be fully opened to permit the spreader to lock.
16. You are prohibited from standing on the top two steps of a stepladder.
17. Metal ladders must not be used in areas where they could contact energized wiring.

Appendix P: Additional Analyses Conducted on Samples Collected on October 26 and October 27, 1992

1. Results of Trace Metals Analysis Conducted by Interpoll Laboratories, Sample log numbers 7328-114 and 7328-139.

Sample Identification: 10/26/92, 1300-1600 hours
Sample Type: Coal

Metal	Method	Concentration (mg/kg) as Received
Cadmium	SW-846,6010	<1
Lead	SW-846,6010	7
Mercury	Fluorescence	0.18

Sample Identification: 10/26/92, 0700-1000 hours
Sample Type: Coal

Metal	Method	Concentration (mg/kg) as Received
Cadmium	SW-846,6010	<1
Lead	SW-846,6010	<2.5
Mercury	Fluorescence	0.16

2. Results of Bulk Density Analysis Conducted by Interpoll Laboratories

Sample Type: Coal

Log No.	Sample Identification	Bulk Density[a] LB/FT^3
7328-113	10/26/92, 1000-1300 hours	44.65
7328-115	10/27/92, 0700-1000 hours	46.67
7328-116	10/27/92, 1000-1300 hours	44.81
7328-117	10/27/92, 1300-1600 hours	46.59
7328-118	10/27/92, 6:00 p.m.	45.01

Sample Type: b-dRDF Pellets

Log No.	Sample Identification	Bulk Density[a] LB/FT^3
7328-119	10/27/92, 0700-1000 hours	20.87
7328-120	10/27/92, 1000-1300 hours	21.46
7328-121	10/27/92, 6:00 p.m.	19.86

[a] Analysis performed gravimetrically

3. Results of Sulfur Analysis Conducted by Argonne National Laboratory

Sample Type: Coal

Log No.	Sample Identification	Sulfur[a] - %
2263-01	10/26/92, 0700-1000 hours	0.73
2263-02	10/26/92, 1300-1600 hours	0.76
2263-03	10/26/92, 6:00 p.m.	0.71

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[a] Analysis performed by ASTM Method D3177

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13. ABSTRACT *(Maximum 200 words)*

This research project characterized the effects of burning a combination of binder-enhanced densified refuse-derived fuel (b-dRDF) pellets and coal in a commercial-scale electrical generating facility. Pelletized b-dRDF has been burned in coal combustors, but only in quantities of less than 3% in large utility systems. This research investigated using b-dRDF in quantities up to 20%. Through this research, project participants gained operating, technical, and economic viability data that will be critical for the near-term commercial use of b-dRDF.

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