AP42 Section:	1.1
Reference:	65
Title:	Results of the May 29, 1990 Trace Metal Characterization Study on Units 1 and 2 at the Sherburne County Generating Station in Becker, Minnesota. Interpoll Laboratories, Inc., Circle Pines, Minnesota. July, 1990.

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RESULTS OF THE MAY 29, 1990 TRACE METAL CHARACTERIZATION STUDY ON UNITS 1 AND 2 AT THE SHERBURNE COUNTY GENERATING STATION IN BECKER, MINNESOTA

interpoll

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

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

RESULTS OF THE MAY 29, 1990 TRACE METAL CHARACTERIZATION STUDY ON UNITS 1 AND 2 AT THE SHERBURNE COUNTY GENERATING STATION IN BECKER, MINNESOTA

Submitted to:

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

Attention:

Bob Catron

Approved by:

Perry Lonnes

Perry Lonnes, Ph.D.
President

Report Number 0-3053
JuLY 18, 1990
PL/prp

TABLE OF CONTENTS

ABBREVIATIONS

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

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

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

Jj

1 INTRODUCTION

On May 29, 1990, Interpoll Laboratories personnel conducted a trace metal characterization study on the Units 1 and 2 Dry Scrubbing System at the Northern States Power Company (NSP) Sherburne County Generating Station located in Becker, Minnesota. On-site testing was performed by D. Van Hoever, E. Trowbridge and S. Bainville. Coordination between testing activities and plant operation was provided by Bob Catron of NSP.

Units 1 and 2 are identical Combustion Engineering 750 megawatt boilers which came on line in 1976. During this test, both boilers were fired with 80% Rochelle and 20% Coalstrip pulverized subbituminous coal. Emissions are controlled by a wet limestone scrubbing system which consists of twelve individual rod venturi scrubber spray towers. Cleaned flue gas is exhausted to the atmosphere by a 600-foot radial steel-lined concrete stack common to Units 1 and 2.

A Multi-Metal Modified Method 5 (4M5) sampling train was used to isokinetically collect solid and vapor phase trace metals. The samples were collected and analyzed as per the EPA Draft Method "Methodology for the Determination of Metals Emissions in Exhaust Gases from Stationary Source Combustion Processes". The aerosol or solid phase trace metal samples were collected on high purity Pallflex® filters. The vapor phase trace metals were collected in an all glass impinger train. The first and second impingers each contained 100 cc of a mixture of 5% HNO_3 and 10% H_2O_2. The third impinger contained 100 cc of a mixture of 4% $KMnO_4$ and 10% H_2SO_4. This impinger collects any elemental mercury which might penetrate the first two impingers and is used only when mercury is being sampled. The recovered four-part trace metal samples were combined, dissolved in acid (including the glass fiber filter) and analyzed by Inductively Coupled Argon Plasma Emission Spectrometry (ICP). Mercury was analyzed by cold vapor atomic absorption. A field biased blank was collected and recovered at the test site and analyzed with the field samples.

An integrated flue gas sample was extracted simultaneously with each trace metal sample using a specially designed gas sampling system. Integrated flue gas samples were collected in 44-liter Tedlar bags housed in a protective aluminum housing. After sampling was complete, the bags were sealed and returned to the laboratory for Orsat analysis. Prior to sampling, the Tedlar bags are leak checked at 15 IN.HG. vacuum with an in-line rotameter. Bags with any detectable inleakage are discarded.

Testing was conducted from four test ports on the Stack oriented at 90 degrees. These test ports are located seven stack diameters downstream of the breeching inlet and eleven diameters upstream of the stack exit. A 16-point traverse was used to extract representative trace metal samples. Each traverse point was sampled four minutes to give a total sampling time of 64 minutes per run.

The important results of the test are summarized in Section 2. Detailed results are presented in Section 3. Field data and all other supporting information are presented in the appendices.

2 SUMMARY AND DISCUSSION

The results of the trace metals determinations are summarized in Tables 2.1 and 2.2.

Our review of the results suggest that the only anomolous result might be the high nickel concentration observed during the second run. This high value may be due to contamination.

No other difficulties were encountered in the field or in the laboratory evaluation of the samples. On the basis of this fact and a complete review of the entire data and results, it is our opinion, subject to the above qualifications, that the results reported herein are accurate and closely reflect the actual values which existed at the time the test was performed.

Table 2.1 Summary of the Results of the May 29, 1990 Trace Metals Engineering Test on Units 1 & 2 at the NSP - Sherco Plant Located in Becker, Minnesota.

Item	Run 1	Run 2	Run 3
(Concentration ug/Nm^3)			
Aluminum	725.0	1870.3	662.6
Antimony	0.7	0.3	0.8
Arsenic	2.5	3.5	2.8
Barium	267.5	514.8	226.4
Beryllium	0.3	0.3	0.3
Boron	332.1	6900.3	3723.9
Cadmium	1.7	1.1	0.8<
Calcium	5431.7	11327.4	6224.2
Chromium	16.5	14.3	7.6
Copper	10.5	13.5	11.4
Iron	837.7	1102.6	821.6
Lead	9.0	7.5	8.3
Magnesium	571.7	1481.2	569.4
Manganese	24.8	26.3	52.2
Mercury	0.8	1.6	1.2
Molybdenum	2.3<	3.8	2.3<
Nickel	1.5<	21.1	1.6
Potassium	127.7	165.6	159.0
Selenium	6.6	9.0	10.6
Silver	0.9<	0.9<	1.0<
Sodium	383.1	549.4	469.5
Strontium	203.6	367.3	212.0
Vanadium	21.0	26.3	25.0
Zinc	21.8	24.8	20.4

A trailing '<' indicates that the true value is below the detection limit.

Table 2.2 Summary of the Results of the May 29, 1990 Trace Metals Engineering Test on Units 1 & 2 at the NSP - Sherco Plant Located in Becker, Minnesota.

Item	Run 1	Run 2	Run 3
(Emission rate 10^{-3}LB/HR)			
Aluminum	8972.5	23387.7	7705.2
Antimony	8.4	4.1	9.2
Arsenic	30.4	43.3	32.6
Barium	3310.1	6437.5	2633.0
Beryllium	3.3	3.6	3.5
Boron	4109.7	86285.2	43307.7
Cadmium	20.5	13.2	9.7<
Calcium	67224.1	141643.9	72385.1
Chromium	204.6	178.8	88.1
Copper	130.2	169.4	132.1
Iron	10367.2	13787.9	9554.5
Lead	111.6	94.1	96.9
Magnesium	7075.7	18521.9	6622.1
Manganese	306.8	329.4	607.6
Mercury	9.3	19.6	14.1
Molybdenum	27.9<	47.1	26.4<
Nickel	18.6<	263.5	18.5
Potassium	1580.6	2070.5	1849.3
Selenium	81.8	112.9	123.3
Silver	11.2<	11.3<	11.4<
Sodium	4741.9	6870.4	5459.7
Strontium	2519.7	4592.8	2465.7
Vanadium	260.3	329.4	290.6
Zinc	269.6	310.6	237.8

A trailing '<' indicates that the true value is below the detection limit.

3 RESULTS

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

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

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

Test No. 1
Units 1 & 2 Stack

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

	Run 1	Run 2	Run 3
Date of run	05-29-90	05-29-90	05-29-90
Dry basis (orsat)			
carbon dioxide.....	13.50	13.50	13.40
oxygen.....	6.60	6.50	6.60
carbon monoxide.....	0.00	0.00	0.00
nitrogen.....	79.90	80.00	80.00
Wet basis (orsat)			
carbon dioxide.....	11.33	11.37	11.34
oxygen.....	5.54	5.48	5.58
carbon monoxide.....	0.00	0.00	0.00
nitrogen.....	67.06	67.41	67.69
water vapor.....	16.07	15.74	15.39
Dry molecular weight.....	30.42	30.42	30.41
Wet molecular weight.....	28.43	28.46	28.50
Specific gravity.....	0.982	0.983	0.984
Water mass flow.....(LB/HR)	0.00	0.00	0.00
FO	1.059	1.067	1.067

Test No. 1
Units 1 & 2 Stack

3.2 Results of Multi-Metal Modified Method 5 (4M5) Sampling.....

	Run 1	Run 2	Run 3
Date of run	05-29-90	05-29-90	05-29-90
Time run start/end.....(HRS)	0845-1001	1035-1152	1225-1342
Static pressure.....(IN.WC)	-1.20	-1.20	-1.20
Cross sectional area (SQ.FT)	829.58	829.58	829.58
Pitot tube coefficient.....	0.840	0.840	0.840
Water in sample gas			
condenser.....(ML)	0.0	0.0	0.0
impingers.....(GRAMS)	177.0	172.0	167.0
desiccant.....(GRAMS)	14.0	14.0	13.0
total.....(GRAMS)	191.0	185.0	180.0
Gas meter coefficient.....	1.0013	1.0013	1.0013
Barometric pressure..(IN.HG)	28.47	28.47	28.47
Avg. orif.pres.drop..(IN.WC)	1.88	1.85	1.84
Avg. gas meter temp..(DEF-F)	75.7	71.8	75.0
Volume through gas meter....			
at meter conditions...(CF)	49.85	49.40	49.40
standard conditions.(DSCF)	47.02	46.94	46.65
standard conditions..(NM^3)	1.332	1.329	1.321
Total sampling time....(MIN)	64.00	64.00	64.00
Nozzle diameter.....(IN)	.183	.183	.183
Avg.stack gas temp ..(DEG-F)	185	185	291
Volumetric flow rate.....			
actual.....(ACFM)	5068854	5104263	5505374
dry standard.....(DSCFM)	3305953	3340203	3106503
Isokinetic variation.....(%)	101.0	99.8	106.6

APPENDIX A

RESULTS OF VOLUMETRIC FLOW RATE DETERMINATIONS

Interpoll Report No. 0-3053
Northern States Power - Sherco
Becker, Minnesota

Test No. 1
Units 1 & 2 Stack

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

Date of Determination.....	05-29-90
Time of Determination.....(HRS)	810
Barometric pressure.....(IN.HG)	28.47
Pitot tube coefficient.....	.84
Number of sampling ports.....	4
Total number of points.....	16
Shape of duct.....	Round
Stack diameter.....(IN)	390
Duct area.....(SQ.FT)	829.58
Direction of flow.....	UP
Static pressure.....(IN.WC)	-1.2
Avg. gas temp.....(DEG-F)	185
Moisture content.....(% V/V)	16.07
Avg. linear velocity.....(FT/SEC)	102.0
Gas density.....(LB/ACF)	.05732
Molecular weight.....(LB/LBMOLE)	30.42
Mass flow of gas.....(LB/HR)	1.746359E+07
Volumetric flow rate.....	
actual.....(ACFM)	5077454
dry standard.....(DSCFM)	3308995

APPENDIX B

LOCATION OF TEST PORTS

EPA Method 1 Specification

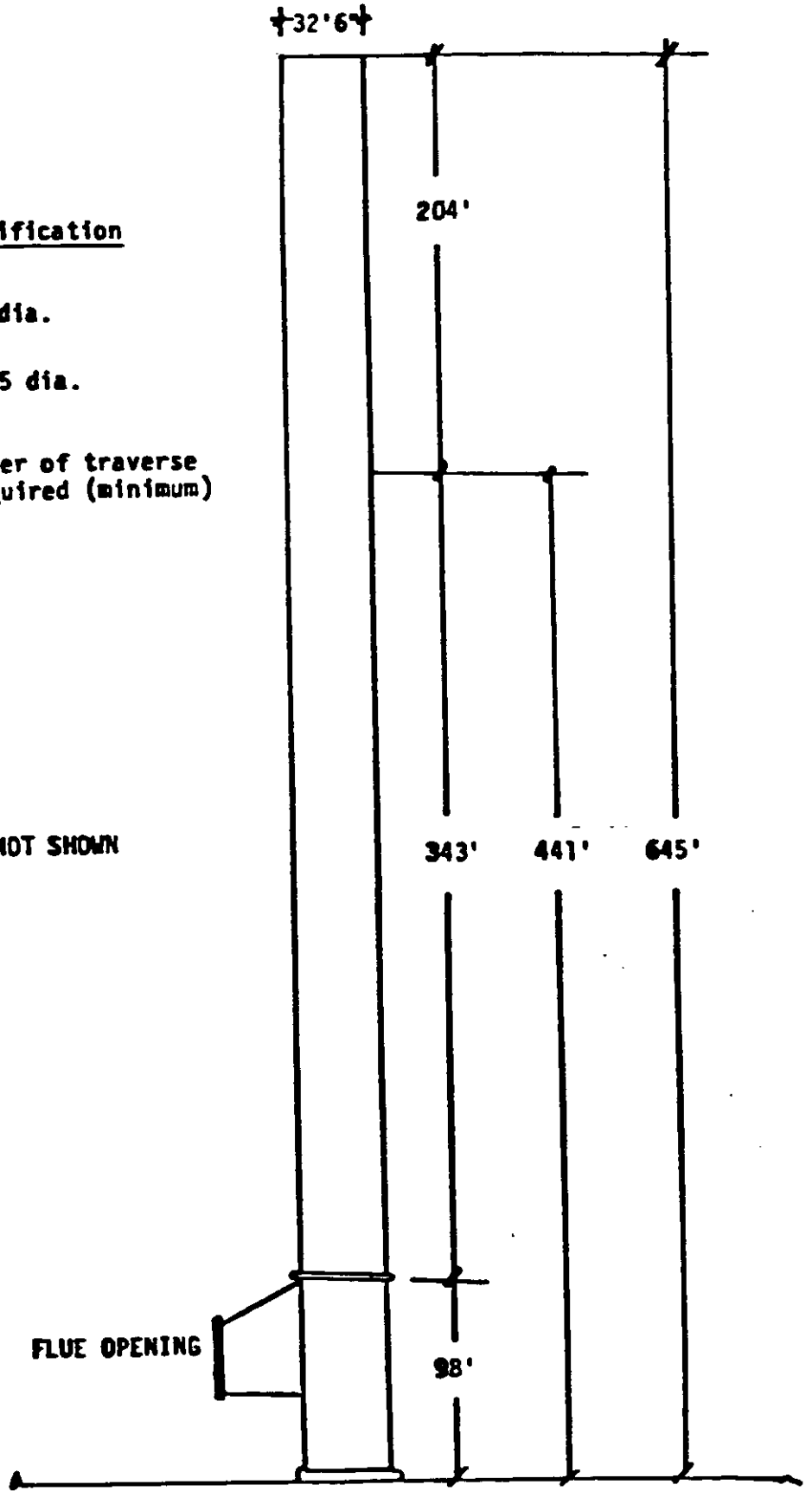
$A = \frac{204}{32.5} = 6.28$ dia.

$B = \frac{343}{32.5} = 10.55$ dia.

MN = Total number of traverse points required (minimum)

MN = 12

OUTER COLUMN NOT SHOWN



NSP SHERBURNE COUNTY GENERATING PLANT
UNITS 1&2 STACK

NOT TO SCALE

APPENDIX C

4M5 FIELD DATA SHEETS

INTERPOLL LABORATORIES EPA METHOD 2 FIELD DATA SHEET

Job NSP/SHERCO

Source UNITS 1&2 STACK

Test 1 Run 1 Date 5-29-90

Stack dimen. 390 IN.

Dry bulb ____ °F Wet bulb ____ °F

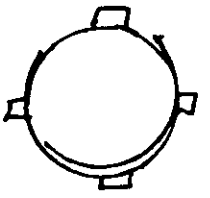
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.

Barometric pressure 28.47 in Hg

Static pressure -1.2 in WC

Operators E. TROWBRIDGE

Pitot No. 23- Cp .840



Schematic of Cross Section

Traverse Point No.	Fraction of Diameter	Distance from Stack Wall (in)	Distance from End of Port (in)	Velocity Pressure (in WC)	Temperature of gas (°F)
		Port length:	in.	Time start: 8:10	hrs
A	1	.032	12	2.4	185
	2	.105		2.5	
	3	.194		2.6	
	4	.323		2.6	
B	1			2.4	185
	2			2.6	
	3			2.6	
	4			2.6	
C	1			2.5	185
	2			2.6	
	3			2.6	
	4			2.6	
D	1			2.4	185
	2			2.5	
	3			2.5	
	4			2.6	

Temp. meas. tool & S/N: PDT-4

Time end: hrs

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

CF-011

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job NSP / SHERCO
Source UNIT #1 & 2 BOILER STACK
Date 5-29-90 Test 1 Run 1

Operators E.T & D.V.H
Meter Box No. 6 ΔH 1.84 IN WC
Coeaster coeff. 1.0013

Pitot No. ____ Cp .840
Bar. Press. 28.07 inHg H2O x
Nozzle No. 6-3 Nozzle Dia .183 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dec. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas In	Gas Out	Oxygen (x v/v)
	0845	980.30											
A - 4	4	983.47	2.60	1.94	3.41	9.0	185	240	252	40	72	71	6.2
3	8	986.65	2.60	1.92	6.62	9.5	185	245	255	40	74	71	6.3
2	12	989.66	2.50	1.85	9.70	9.5	184	244	253	41	76	72	6.3
1	16	992.73	2.40	1.78	2.74	9.5	184	250	253	42	76	72	6.4
B - 4	20	995.77	2.50	1.86	5.84	9.5	184	252	252	41	76	72	6.4
3	24	998.97	2.60	1.93	9.00	9.5	184	251	253	41	76	72	6.1
2	28	1002.10	2.60	1.93	2.15	9.7	184	250	252	42	76	72	6.2
1	32	1005.10	2.30	1.71	5.13	9.5	184	253	255	42	77	73	6.3
C - 4	36	1008.30	2.70	2.01	8.35	10.2	184	250	252	43	78	74	6.4
3	40	1011.50	2.60	1.94	1.52	10.2	184	250	255	43	80	75	6.2
2	44	1014.08	2.60	1.94	4.70	10.2	185	252	252	43	80	75	6.2
1	48	1017.66	2.30	1.72	7.69	10.0	185	253	254	44	80	75	6.2
D - 4	52	1020.74	2.60	1.94	0.86	10.5	185	250	249	44	80	75	6.3
3	56	1023.96	2.50	1.87	3.98	10.5	185	251	252	44	80	75	6.4
2	60	1027.10	2.50	1.87	7.09	10.5	185	253	250	45	82	77	6.2
1	64	1030.15	2.40	1.80	0.16	10.4	185	250	252	45	82	77	6.3
	1001												
	θ = 64	Vm = 49.85		ΔH = 1.88							Avg. = 75.7		

S-0037R

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job NGP/SHERCO
Source UNIT 1 & 2 STACK
Date 5-29-80 Test 1 Run 2

Operators E. Thornburg T D Y H
Meter Box No. 6 ΔH 1.84 IN WC
Barometer coeff. 1.0013

Pitot No. Cp .84
Bar. Press. 28.07 inHg H2O x
Nozzle No. 6-3 Nozzle Dia. .183 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dec. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Gas/In	Gas/Out	Oxygen (xx/v)
	1035	30.50											
D 4	4	33.68	2.40	1.90	3.63	10.0	185	240	246	40	70	68	6.5
3	8	36.70	2.50	1.80	6.67	9.0	185	245	250	41	72	68	6.4
2	12	39.75	2.50	1.81	9.71	9.0	185	247	260	41	74	69	6.3
1	16	42.71	2.40	1.74	12.70	8.9	185	246	252	41	74	69	6.1
C 4	20	45.90	2.70	1.95	15.87	9.7	185	248	260	41	74	69	6.1
3	24	48.95	2.60	1.88	18.98	9.6	185	250	255	42	74	69	6.2
2	28	52.05	2.60	1.88	22.09	9.6	185	249	256	42	74	69	6.3
1	32	55.00	2.30	1.67	25.02		184	250	254	42	74	69	6.3
B 4	36	58.12	2.60	1.88	28.13	9.6	185	252	252	43	74	69	6.5
3	40	61.20	2.60	1.88	31.25	9.6	185	252	250	43	74	69	6.4
2	44	64.30	2.60	1.88	34.36	9.6	185	250	248	43	76	70	6.4
1	48	67.35	2.40	1.74	37.36	9.5	185	250	250	43	76	70	6.4
A 4	52	70.55	2.80	2.03	40.59	9.8	185	253	248	43	76	70	6.1
3	56	73.77	2.70	1.96	43.77	9.7	185	252	250	43	76	70	6.2
2	60	76.88	2.60	1.89	46.89	9.7	185	250	253	43	76	70	6.5
1	64	79.90	2.40	1.75	49.89	9.6	184	252	260	43	76	70	6.2
	(1152)												
	θ = 64	Vm = 49.40		ΔH = 1.85							Avg. = 71.8		

C-5

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

Job NSP/SHERCO
Source UNITS 1+2 - STACK
Date 5-29-90 Test 1 Run 3

Operators ET - DVM
Meter Box No. 6 ΔH 1.84 ΔH@ 1.84 IN WC
Calibrator coeff. 1.0013

Pitot No. Cp .840
Bar. Press. 28.97 inHg H2O X
Nozzle No. 6-3 Nozzle Dia. .385 IN.

Traverse Point No.	Sampling Time (min)	Sample Volume (cf)	Velocity Head (inWC)	Orifice Meter (inWC)	Dev. Vol. (cf)	VAC. inHg	Temperatures (°F) Stack	Probe	Oven	Impg.	Cyc/In	Cyc/Out	Oxygen (x v/v)
	1225	80.30											
A 4	4	83.50	2.70	1.95	3.48	9.5	185	243	247	42	73	71	6.2
3	8	86.40	2.60	1.86	6.58	9.4	185	246	250	42	75	71	6.3
2	12	89.64	2.60	1.86	9.66	9.4	185	248	250	42	75	71	6.1
1	16	92.64	2.40	1.72	2.66		184	251	254	43	76	71	6.4
B 4	20	95.80	2.65	1.90	5.79	9.8	185	250	255	43	77	72	6.2
3	24	98.98	2.60	1.87	8.90	9.8	185	253	254	43	77	72	6.2
2	28	102.00	2.60	1.87	2.01	9.8	185	250	252	43	77	72	6.2
1	32	105.02	2.40	1.73	4.99	9.5	184	251	255	44	78	72	6.3
C 4	36	108.18	2.70	1.94	8.16	10.0	185	253	252	44	79	73	6.2
3	40	111.30	2.60	1.87	1.29	9.9	185	252	256	44	79	73	6.0
2	44	114.45	2.60	1.87	4.40	9.9	185	250	249	44	79	73	6.2
1	48	117.36	2.30	1.66	7.33	9.5	184	250	252	44	79	73	6.3
D 4	52	120.50	2.60	1.87	0.45	9.9	185	252	254	44	80	73	6.1
3	56	123.60	2.60	1.88	3.57	9.9	185	253	257	44	80	73	6.3
2	60	126.67	2.55	1.84	6.66	9.8	185	252	255	45	80	73	6.2
1	64	129.70	2.40	1.73	9.66	9.5	184	250	252	45	80	73	6.3
	(1342)												
	θ = 64	Vm = 49.40		ΔH 1.84							Avg. = 75.0		

APPENDIX D

LABORATORY DATA SHEETS FOR METHOD 3

Interpoll Laboratories
(612) 786-6020

Chain of Custody
Sample Deposition Sheet

Job NSP/SHERCO
Team Leader E.T ROWBRIDGE
Date Submitted 5-29-90
Test No. 1

Source UNITS 1 & 2
Test Site STACK
Date of Test 5-29-90
No. of Runs Completed 3

No. of Samples	Type of Sample	Analysis Required	Comments
5	Probe Wash: ☒ Acetone ☐ D.I. Water	☐ As per EPA M-5 ☒ Other 4M5	
5	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 M5	2 BLANKS #4M5-7009 #4M5-7010
10	Impinger Catch: ☐ D.I. Water ☐ 3% H_2O_2 ☒ M5 Hg Only ☒ M5 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)
1	☒ Fuel Sample ☐ Aggregate	☒ Attached fuel Form #S-0163RRR	mineral ash FWT.
	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

APPENDIX E

RESULTS OF TRACE METAL ANALYSIS

INTERPOLL LABORATORIES, INC.
(612)786-6020

NSP/Sherco
Laboratory Log No. 9639

Results of Trace Metal Analysis

Test: 1
Source: Units No. 1 & 2 Stack
Sample Type: 4M5 Train Catch

		Total Mass of Trace Metal in Sample (ug)				
Trace Metal	EPA Method	Field Blank 1	Field Blank 2	Run 1	Run 2	Run 3
(Log No.)		(9639-21)	(9639-22)	(9639-23)	(9639-24)	(9639-25)
Aluminum	SW-846, 6010	330	350	1340	2860	1250
Arsenic	SW-846, 7060	< 0.05	< 0.05	3.27	4.58	3.67
Boron	SW-846, 6010	15	29	464	9190	4940
Barium	SW-846, 6010	8	8.8	364	692	307
Beryllium	SW-846, 7091	0.022	0.034	0.37	0.40	0.42
Calcium	SW-846, 6010	256	237	7480	15300	8470
Cadmium	SW-846, 6010	< 1	< 1	2.2	1.4	< 1.1
Chromium	SW-846, 6010	4.4	4.2	26	23	14
Copper	SW-846, 6010	4	3	17	21	18
Iron	SW-846, 6010	73	76	1190	1540	1160
Potassium	SW-846, 6010	90	80	250	300	290
Magnesium	SW-846, 6010	32	33	793	2000	784
Manganese	SW-846, 6010	67	6.8	38	40	74
Molybdenum	SW-846, 6010	26	28	28	31	29
Sodium	SW-846, 6010	596	674	1110	1330	1220
Nickel	SW-846, 6010	5.0	4	5.8	32	6.1
Lead	SW-846, 6010	9.8	12	22	20	21
Selenium	SW-846, 7740	< 0.5	< 0.5	8.79	12.2	13.8
Antimony	SW-846, 7041	0.2	0.05	0.95	0.49	1.1
Strontium	3500-Sr. B[1]	10	< 5	276	493	285
Vanadium	SW-846, 6010	< 1	< 1	28	35	33
Zinc	SW-846, 6010	14	15	43	47	41
Mercury	SW-846, 7470	0.2	0.2	0.40	1.60	0.88
Silver	SW-846, 6010	< 1.2	< 1.2	< 1.2	< 1.2	< 1.3

[1]Standard Methods, 17th Edition.

APPENDIX F

RESULTS OF MINERAL ASH ANALYSIS

INTERPOLL LABORATORIES, INC.
(612)786-6020

NSP/Sherco
Sample Log No. 9639-26

Results of the Mineral Ash Analysis[1]

Test: 1

Sample Type: Coal

Parameter	Ignited Basis, % by weight	As Received (Dry Basis) % by weight
Silica (SiO_2)	35.31	2.03
Alumina (Al_2O_3)	16.79	0.97
Titania (TiO_2)	1.07	0.061
Ferric oxide (Fe_2O_3)	6.93	0.39
Lime (CaO)	19.45	1.12
Magnesia (MgO)	4.45	0.26
Potassium oxide (K_2O)	0.40	0.023
Sodium oxide (Na_2O)	1.40	0.081
Sulfur trioxide (SO_3)	12.75	0.73
Phos. pentoxide (P_2O_5)	0.64	0.038
Manganese oxide (Mn_3O_4)	0.11	0.006
Barium oxide (BaO)	0.53	0.03
Under(over)determined[2]	0.17	
Loss on ignition		94.25

[1]Analyses performed in accordance with ASTM D3682 and D1757 by ICP emission spectrometry.

[2]Under(over)determined in the case of the ignited basis results corresponds to the sum of the determinant errors in the individual analyses of the various reported analyte plus the percent by weight of any minerals in the sample for which an analysis was not performed (a positive value corresponds to underdetermined and a negative value to overdetermined).

APPENDIX G

RESULTS OF FUEL ANALYSIS

INTERPOLL LABORATORIES INC.
Fuel Laboratory
(612) 786-6020

06-18-1990

Client: NSP Sherco

Laboratory Log Number: 9639-26-7942

Sample Identification: Coal Test 1 5-29-90

Ultimate Analysis WT %

Parameter	Moisture & Ash Free	Moisture Free	As Received
Moisture, Total			28.06
Ash		8.00	5.75
Carbon	74.90	68.91	49.58
Hydrogen	5.43	4.99	3.59
Nitrogen	0.89	0.82	0.59
Oxygen (calculated)	18.15	16.70	12.01
Sulfur	0.63	0.58	0.42
Heating Value, BTU/LB.	12912	11880	8547

Respectfully submitted,

Gregg W. Holman

Gregg W. Holman
Senior Scientist
Inorganic Chemistry Department

9796

APPENDIX H

PROCEDURES

Flow. Flow determinations were carried out in accordance with EPA Method 2, CFR Title 40, Part 60, Appendix A (Revised July 1, 1988). A type S pitot was used to sense velocity pressure and an inclined manometer was used to measure velocity pressures. Gas temperatures were measured using a calibrated Type K thermocouple and digital temperature meter. Gas density (i.e. molecular weight) was calculated from the composition of the gas which was determined by Orsat.

INTERPOLL LABS
4500 BALL ROAD N.E.
CIRCLE PINES, MN 55014-1819
(612) 786-6020

11/7/88

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

*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

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

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.

APPENDIX I

CALCULATION EQUATIONS

CALCULATION EQUATIONS

METHOD 2

$$\bar{V}_s = 85.48\ C_p\ (\sqrt{\Delta p})_{avg}\ \sqrt{\frac{T_{s(avg)}}{P_s\ M_s}}$$

$$Q_{s,d} = 60(1 - B_{ws})\ \bar{V}_s\ A\ (\frac{528}{T_{s(avg)}})\ (\frac{P_s}{29.92})$$

$$Q_a = 60\ \bar{V}_s\ A$$

$$\dot{m}_g = \frac{4.995\ Q_{s,d}\ G_d}{1 - B_{ws}}$$

$$RH^* = 100\ (vp_{twb}\ 0.0003641\ P_s\ (T_{db} - T_{wb}))/vp_{tdb}$$

$$B^*_{ws} = RH(vp_{tdb})/P_s$$

$$= \frac{4.585 \times 10^{-2}\ P_s M_s}{T_s\ (avg)}$$

*Alternate equations for calculating moisture content from wet bulb and dry buld data.

CALCULATION EQUATIONS

METHOD 5

$$V_{m(std)} = 17.65\ V_m\ \gamma\ \left(\frac{P_{bar} + \overline{\Delta H}/13.6}{T_{m(avg)}}\right)$$

$$V_{w(std)} = 0.0472\ V_{Is}$$

$$B_{ws} = \frac{V_{w(std)}}{V_{w(std)} + V_{m(std)}}$$

$$I = 0.0944\ \left(\frac{T_{s(avg)}\ V_{m(std)}}{P_s\ V_s\ A_n\ \theta\ (1 - B_{ws})}\right)$$

$$C_s = \frac{15.43\ M_p}{V_{m(std)}}$$

$$C_a = \frac{272.3\ M_p\ P_s}{T_{s(avg)}\ (V_{w(std)} + V_{m(std)})}$$

$$(\dot{m}_p)_1 = 8.5714 \times 10^{-3}\ C_s\ Q_{s,d}$$

$$(\dot{m}_p)_2 = \frac{1.3228 \times 10^{-1}\ M_p\ A}{\theta\ A_n}$$

$$\dot{m}_p = \frac{(\dot{m}_p)_1 + (\dot{m}_p)_2}{2}$$

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

$\bar{V}_s$ = Average actual stack gas velocity, FT/SEC

vp_{tdb} = Vapor pressure at T_{db}, IN. HG.

Job NSP Sherco Source Units 1&2 Date of Test 5-29-90

Element	GF/AA	HCD	MICROGRAMS Field Blanks H	1	2	Analytical Detection Limit	Minimum Detectable Mass	Best Estimate of Field Blank	Total Mass in Sample (µg) (Not corrected for blank) Run 1	Run 2	Run 3	Total Mass in Sample (µg) (Corrected for blank) Run 1	Run 2	Run 3
Aluminum		X	375	330	350	2.5	≤24	375	1340	2860	1250	965	2485	875
Arsenic	X		-	<.05	<.05	.05	≤.05	0	3.27	4.6	3.7	3.27	4.6	3.7
Boron		X	9	15	29	1	≤20	22	414	9190	4940	442	9168	4918
Barium		X	12	8	8.8	1	≤6	8	364	692	307	356	684	279
Beryllium	X		-	.022	.034	.001	≤.04	.02	.37	.40	.42	.35	.38	.40
Calcium		X	181	256	257	10	≤43	250	7430	15300	8470	7230	15050	8220
Cadmium		X	<.4	<1	<1	1	≤1	0	2.2	1.4	<1.1	2.2	1.4	<1.1
Chromium		X	<.7	4.4	4.2	1	≤1	4	26	23	14	22	19	10
Copper		X	3.2	4	3	1	≤1.2	3	17	21	18	14	18	15
Iron		X	38	73	76	1	≤24	75	1190	1540	1160	1115	1465	1085
Potassium		X	64	90	80	80	≤12	80	250	300	290	170	220	210
Magnesium		X	25	32	33	1	≤12	32	793	2000	784	761	1968	752
Manganese		X	<.5		6.8	.5	≤1	5	38	40	74	33	35	69
Molyhdenum		X	33	26	28	1	≤3	26	28	31	29	≤3	5	≤3
Sodium		X	158	396	674	2	≤100	600	1110	1330	1320	510	730	620
Nickel		X	<1	5	4	1	≤2	4	58	32	6.1	≤2	28	21
Lead		X	<4	9.8	12	5	≤4	10	22	20	21	12	10	11
Selenium	X		-	<.5	<.5	0.5	≤.5	0	8.8	12	14	8.8	12	14
Antimony	X		-	.2	.05	.05	≤.1	.05	.95	.49	1.1	.90	.44	1.05
Strontium	X		-	10	<5	5	≤5	5	276	493	285	271	488	280
Vanadium		X	<.6	<1	<1	1	≤1	0	28	35	33	28	35	33
Zinc		X	9	14	15	1	≤5	14	43	47	41	29	33	27
Mercury Front	CV/AA		<1	.2	.2	.04	≤.1	.20	.40	1.6	.88			
Mercury KMnO4			<1	.16	<.04	.04	≤.1	<.04	.80	.68	.92			
Mercury Total			<1	.36	<.24	.08	≤.2	.20	1.20	2.28	1.80	1.00	2.08	1.60
Silver		X	<.6	<1.2	<1.2	1.2	≤1.2	1.2	<1.2	<1.2	<1.3	<1.2	<1.2	<1.3

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