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|               |                                                                                                                                                                                          |
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RESULTS OF THE MARCH 1990  
TRACE METAL CHARACTERIZATION  
STUDY ON UNIT 3 AT THE SHERBURNE  
COUNTY GENERATING STATION



**interpoll**

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COUNTY GENERATING STATION

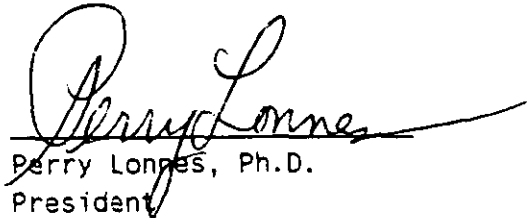
Submitted to:

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

Attention:

Bob Catron

Approved by:

  
Perry Lannes, Ph.D.  
President

Report Number 0-3005  
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PL/prp

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## ABBREVIATIONS

|                        |                                                     |
|------------------------|-----------------------------------------------------|
| ACFM                   | actual cubic feet per minute                        |
| cc (ml)                | cubic centimeter (milliliter)                       |
| DSCFM                  | standard cubic foot of dry gas per minute           |
| DSML                   | dry standard milliliter                             |
| DEG-F (°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 <sup>6</sup> BTU | 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 <sup>3</sup>     | milligrams per dry standard cubic meter             |
| ug/Nm <sup>3</sup>     | 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                                   |
| <                      | ≤ (when following a number)                         |

Standard conditions are defined as 68 °F (20 °C) and 29.92 IN. of mercury pressure.

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

Jj

## 1 INTRODUCTION

On March 27, 1990, Interpoll Laboratories personnel conducted a trace metal characterization study on the Unit 3 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 Hoeven, E. Trowbridge, M. Kaehler and S. Bainville. Coordination between testing activities and plant operation was provided by Bob Catron of NSP.

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

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<sup>P</sup> 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<sub>3</sub> and 10% H<sub>2</sub>O<sub>2</sub>. The third and fourth impingers contained 100 cc of a mixture of 4% KMnO<sub>4</sub> and 10% H<sub>2</sub>SO<sub>4</sub>. These impingers collect any elemental mercury which might penetrate the first two impingers.

The recovered four-part trace metal samples were returned to the laboratory where the probe rinse, filter and nitric acid impinger catch were combined, dissolved in acid (including the glass fiber filter) and analyzed by Inductively Coupled Argon Plasma Emission Spectrometry (ICP).

In some cases, the same were reanalyzed by graphite furnace atomic absorption spectrometry for greater sensitivity. The permanganate mercury catch and an aliquot of the front half catch were analyzed by cold vapor atomic absorption spectrometry (CV/AA). Two field-biased blanks were collected and recovered at each test site and analyzed with the field samples.

The Cr(VI) samples were collected with a Modified Method 13 sampling train without the optional filter. Pallflex<sup>2</sup> Type 2500 QAT ultrapure filters were used in the post-impinger filter holder in place of the normal glass fiber filters. The sampling train was rigorously cleaned prior to sampling to remove any traces of organic materials which react rapidly to reduce Cr(VI) to Cr(III). In addition, nonorganic vacuum grease was used to seal all ground glass joints in the sampling train.

A heated glass-lined sampling probe was used to extract the samples from the stack. The first two impingers were charged with 1 N NaOH which acts as a preservative. The samples were collected following Method 5 sampling protocol. After completion of sampling, the front half of the train was recovered using 0.5% Na<sub>2</sub>CO<sub>3</sub>. The backhalf was recovered using 0.1 N NaOH. The recovered samples were stored at 4 °C until analysis.

In the laboratory, the three-part field sample was combined, pH adjusted, APDC chelating agent added and extracted with MiBK. The MiBK was then evaporated and the residue taken up in nitric acid. The extract was then analyzed by ICP in accordance with EPA Method 6010.

An integrated flue gas sample was extracted simultaneously with each particulate and sulfur dioxide 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 on the SDA Inlet was conducted using a single point traverse. Each test run was 60 minutes in duration. Testing on the Stack was conducted from four test ports 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 metals and chromium VI 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 1 and 2. Chromium VI results are presented in Table 3.

The mass rates of trace metals determined at the Inlet sampling location, when stoichiometrically converted to the appropriate or actual species, and adjusted for unanalyzed species such  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{SO}_3$  and  $\text{P}_2\text{O}_5$  give total particulate mass flow rates which agree very well with the total inlet particulate mass flows measured on August 9, 1988 when a complete duct traverse (48-points) was performed as shown below:

| Run     | Particulate Mass Rate LB/HR |          |
|---------|-----------------------------|----------|
|         | Calculated from             | Measured |
|         | Trace Metals                |          |
|         | 3-27-90                     | 8-9-88   |
| 1       | 32300                       | 44300    |
| 2       | 47200                       | 44200    |
| 3       | 39500                       | -        |
| Average | 39700                       | 42300    |

The above calculated mass rates for 3-27-90 do not exhibit good agreement with the particulate mass rate calculated directly from a gravimetric analysis of the trace metal samples (71,000, 67000 and 38000 LB/HR). This is not surprising inasmuch as the 3-27-90 sampling consisted of a 3-point traverse. This data suggests a severe stratification of particulate mass flow in the inlet ducting.

Some contamination occurred of the Run 1 sample collected at the stack test site which invalidated the Run 1 results for arsenic, cadmium and zinc. This contamination did not effect other trace elements nor was it observed in the subsequent test runs.

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 1. Results of the March 27, 1990 Trace Metal Determinations on the Unit 3 Dry Scrubber System at the NSP Sherburne County Station in Becker, Minnesota.

| Element    | Concentration (ug/Nm <sup>3</sup> ) |       |                      |       |                      |       |
|------------|-------------------------------------|-------|----------------------|-------|----------------------|-------|
|            | Run 1                               |       | Run 2                |       | Run 3                |       |
|            | Inlet                               | Stack | Inlet                | Stack | Inlet                | Stack |
| Aluminum   | 1.39X10 <sup>5</sup>                | 261   | 2.56X10 <sup>5</sup> | 67    | 1.87X10 <sup>5</sup> | 101   |
| Arsenic    | 69.6                                | x     | 66.0                 | <.056 | 83.6                 | <.056 |
| Boron      | 8430                                | 2620  | 8270                 | 445   | 10900                | 1890  |
| Barium     | 10300                               | <6.5  | 15800                | <6.5  | 10300                | <6.5  |
| Beryllium  | 22.9                                | .0022 | 24.9                 | .0067 | 26.5                 | .0135 |
| Calcium    | 3.33X10 <sup>5</sup>                | 465?  | 6.06X10 <sup>5</sup> | 259   | 3.97X10 <sup>5</sup> | 251   |
| Cadmium    | 8.15                                | x     | 8.23                 | 11.8? | 7.34                 | 6.84  |
| Chromium   | 266                                 | 15.6  | 281                  | 9.26  | 316                  | 7.07  |
| Copper     | 587                                 | 108   | 555                  | 52.2  | 748                  | 25.6  |
| Iron       | 1.38X10 <sup>5</sup>                | 143   | 1.10X10 <sup>5</sup> | 103   | 1.58X10 <sup>5</sup> | 33.7  |
| Potassium  | 51700                               | 251   | 48500                | 84.8  | 58300                | 81.9  |
| Magnesium  | 20000                               | 40.3  | 30000                | 16.7  | 30400                | 29.2  |
| Manganese  | 5450                                | 7.73  | 5210                 | 51.9  | 6280                 | 5.16  |
| Molybdenum | 64.5                                | <4.4  | 47.2                 | <4.4  | 87.1                 | <4.4  |
| Sodium     | 84900                               | 949?  | 1.12X10 <sup>5</sup> | 627   | 1.19X10 <sup>5</sup> | 653   |
| Nickel     | 204                                 | 10.9? | 174                  | 10.0  | 237                  | 3.59  |
| Lead       | 640                                 | 16.9  | 494                  | 5.35  | 654                  | <4.5  |
| Selenium   | 45.4                                | <2.72 | 32.2                 | <2.79 | 58.5                 | <2.81 |
| Antimony   | 83.8                                | .97   | 58.1                 | 0.22  | 93.9                 | <.20  |
| Strontium  | 19900                               | 1.63  | 11700                | 5.58  | 24700                | 5.61  |
| Vanadium   | 704                                 | <.11  | 643                  | <.11  | 836                  | <.11  |
| Zinc       | 264                                 | x     | 258                  | 35.6  | 256                  | 23.4  |
| Mercury    | 7.88                                | 5.62  | 7.56                 | 2.34  | 4.95                 | 4.6   |
| Silver     | 2.29                                | <.33  | 2.31                 | <.33  | 2.30                 | <.34  |

x = Result invalidated due to field contamination

? = Result may be high due to field contamination

Table 2. Results of the March 27, 1990 Trace Metal Removal Efficiency Determinations (Based on Mass Flow) on the Unit 3 Dry Scrubber System at the NSP Sherburne County Station in Becker, Minnesota.

| Element    | Mass Flow (LB/HR) |                        |       |                        |       |                        | Avg. Removal Eff. (%w/w) |
|------------|-------------------|------------------------|-------|------------------------|-------|------------------------|--------------------------|
|            | Run 1             |                        | Run 2 |                        | Run 3 |                        |                          |
|            | Inlet             | Stack                  | Inlet | Stack                  | Inlet | Stack                  |                          |
| Aluminum   | 1168              | 1.91                   | 2119  | .493                   | 1553  | .742                   | 99.935                   |
| Arsenic    | .585              | x                      | .546  | <4.12X10 <sup>-4</sup> | .694  | <4.12X10 <sup>-4</sup> | >99.93                   |
| Boron      | 70.8              | 19.1                   | 68.5  | 3.28                   | 90.5  | 13.9                   | 84.2                     |
| Barium     | 86.6              | <.048                  | 131   | <.049                  | 85.5  | <.050                  | >99.950                  |
| Beryllium  | .192              | 1.61X10 <sup>-3</sup>  | .206  | 4.93X10 <sup>-3</sup>  | .220  | 9.92X10 <sup>-3</sup>  | 99.973                   |
| Calcium    | 2799              | 3.40?                  | 5017  | 1.91                   | 3296  | 1.85                   | 99.936                   |
| Cadmium    | .0684             | x                      | .0681 | .0869?                 | .0609 | .0503                  |                          |
| Chromium   | 2.24              | .114                   | 2.33  | .0682                  | 2.62  | .0520                  | 96.74                    |
| Copper     | 4.93              | .789                   | 4.59  | .384                   | 6.21  | .188                   | 91.35                    |
| Iron       | 1160              | 1.04                   | 911   | .759                   | 1312  | .248                   | 99.939                   |
| Potassium  | 434               | 1.83                   | 402   | .624                   | 484   | .602                   | 99.77                    |
| Magnesium  | 168               | .294                   | 248   | .123                   | 252   | .215                   | 99.91                    |
| Manganese  | 45.8              | .0565                  | 43.1  | .382                   | 52.1  | .0379                  | 99.66                    |
| Molybdenum | .542              | <.032                  | .391  | <.033                  | .723  | <.033                  | >93.63                   |
| Sodium     | 714               | 6.93?                  | 927   | 4.62                   | 988   | 4.80                   | 99.38                    |
| Nickel     | 1.71              | .0796?                 | 1.44  | .0736                  | 1.97  | .0264                  | 96.49                    |
| Lead       | 5.38              | .123                   | 4.09  | .0394                  | 5.43  | <.033                  | >98.71                   |
| Selenium   | .382              | <.0199                 | .267  | <.0205                 | .486  | <.0207                 | 94.62                    |
| Antimony   | .704              | 7.09X10 <sup>-3</sup>  | .481  | 1.62X10 <sup>-3</sup>  | .780  | <1.6X10 <sup>-3</sup>  | >99.48                   |
| Strontium  | 167               | .0119                  | 96.9  | .0411                  | 205   | .0412                  | 99.98                    |
| Vanadium   | 5.92              | <8.04X10 <sup>-4</sup> | 5.32  | <8.10X10 <sup>-4</sup> | 6.94  | <8.09X10 <sup>-4</sup> | 99.987                   |
| Zinc       | 2.22              | x                      | 2.14  | .262                   | 2.13  | .172                   | 89.97                    |
| Mercury    | .0662             | .0411                  | .0626 | .0172                  | .0411 | .0338                  | 42.85                    |
| Silver     | .0192             | 2.41X10 <sup>-3</sup>  | .0191 | <2.43X10 <sup>-3</sup> | .0191 | 2.50X10 <sup>-3</sup>  | >87                      |

x = Result invalidated due to field contamination  
 ? = Result may be high due to field contamination

Table 3      Summary of the Results of the March 27, 1990 Hexavalent Chromium (CrVI) Determinations on the Unit 3 Stack at the NSP Sherburne County Station.

| Test/Run | Concentration<br>(ug/Nm <sup>3</sup> ) | Emission Rate<br>(LB/HR) |
|----------|----------------------------------------|--------------------------|
| 2/1      | 1.30                                   | 0.0095                   |
| 2/2      | 0.39                                   | 0.0028                   |
| 2/3      | 1.41                                   | 0.0100                   |
| Avg      | 1.03                                   | 0.0074                   |

### 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 and chromium VI 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.

### 3.1 Results of Orsat and Moisture Analyses

Interpoll Report No. 0-3005  
Northern States Power - Sherco  
Becker, Minnesota

Test No. 1  
Unit 3 SDA Inlet

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

| Date of run                 | Run 1<br>03-27-90 | Run 2<br>03-27-90 | Run 3<br>03-27-90 |
|-----------------------------|-------------------|-------------------|-------------------|
| Dry basis (orsat)           |                   |                   |                   |
| carbon dioxide.....         | 13.30             | 13.40             | 13.50             |
| oxygen.....                 | 6.70              | 6.50              | 6.40              |
| carbon monoxide.....        | 0.00              | 0.00              | 0.00              |
| nitrogen.....               | 80.00             | 80.10             | 80.10             |
| Wet basis (orsat)           |                   |                   |                   |
| carbon dioxide.....         | 12.03             | 12.05             | 12.06             |
| oxygen.....                 | 6.06              | 5.84              | 5.72              |
| carbon monoxide.....        | 0.00              | 0.00              | 0.00              |
| nitrogen.....               | 72.38             | 72.02             | 71.54             |
| water vapor.....            | 9.53              | 10.09             | 10.68             |
| Dry molecular weight.....   | 30.40             | 30.40             | 30.42             |
| Wet molecular weight.....   | 29.21             | 29.15             | 29.09             |
| Specific gravity.....       | 1.009             | 1.007             | 1.005             |
| Water mass flow.....(LB/HR) | 662829            | 695254            | 743527            |

Test No. 1  
Unit 3 Stack

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

|             | Run 1    | Run 2    | Run 3    |
|-------------|----------|----------|----------|
| Date of run | 03-27-90 | 03-27-90 | 03-27-90 |

Dry basis (orsat)

|                      |       |       |       |
|----------------------|-------|-------|-------|
| carbon dioxide.....  | 13.50 | 13.80 | 13.90 |
| oxygen.....          | 6.50  | 6.20  | 6.10  |
| carbon monoxide..... | 0.00  | 0.00  | 0.00  |
| nitrogen.....        | 80.00 | 80.00 | 80.00 |

Wet basis (orsat)

|                      |       |       |       |
|----------------------|-------|-------|-------|
| carbon dioxide.....  | 11.43 | 11.75 | 11.89 |
| oxygen.....          | 5.50  | 5.28  | 5.22  |
| carbon monoxide..... | 0.00  | 0.00  | 0.00  |
| nitrogen.....        | 67.70 | 68.13 | 68.42 |
| water vapor.....     | 15.37 | 14.84 | 14.47 |

|                             |       |       |       |
|-----------------------------|-------|-------|-------|
| Dry molecular weight.....   | 30.42 | 30.46 | 30.47 |
| Wet molecular weight.....   | 28.51 | 28.61 | 28.66 |
| Specific gravity.....       | 0.985 | 0.988 | 0.990 |
| Water mass flow.....(LB/HR) | 0.00  | 0.00  | 0.00  |

|    |       |       |       |
|----|-------|-------|-------|
| FD | 1.067 | 1.065 | 1.065 |
|----|-------|-------|-------|

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Test No. 2  
Unit 3 Stack

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

| Date of run                 | Run 1<br>03-27-90 | Run 2<br>03-27-90 | Run 3<br>03-27-90 |
|-----------------------------|-------------------|-------------------|-------------------|
| <b>Dry basis (orsat)</b>    |                   |                   |                   |
| carbon dioxide.....         | 13.60             | 13.60             | 13.60             |
| oxygen.....                 | 6.10              | 6.10              | 6.00              |
| carbon monoxide.....        | 0.00              | 0.00              | 0.00              |
| nitrogen.....               | 80.30             | 80.30             | 80.40             |
| <b>Wet basis (orsat)</b>    |                   |                   |                   |
| carbon dioxide.....         | 11.68             | 11.61             | 11.58             |
| oxygen.....                 | 5.24              | 5.21              | 5.11              |
| carbon monoxide.....        | 0.00              | 0.00              | 0.00              |
| nitrogen.....               | 68.98             | 68.55             | 68.46             |
| water vapor.....            | 14.10             | 14.63             | 14.85             |
| Dry molecular weight.....   | 30.42             | 30.42             | 30.42             |
| Wet molecular weight.....   | 28.67             | 28.60             | 28.57             |
| Specific gravity.....       | 0.990             | 0.988             | 0.987             |
| Water mass flow.....(LB/HR) | 0.00              | 0.00              | 0.00              |

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

Interpoll Report No. 0-3005  
Northern States Power - Sherco  
Becker, Minnesota

Test No. 1  
Unit 3 SDA Inlet

Results of the Multi Metal Modified Method 5 (4M5) Sampling

|                               | Run 1    | Run 2     | Run 3     |
|-------------------------------|----------|-----------|-----------|
| Date of run                   | 03-27-90 | 03-27-90  | 03-27-90  |
| Time run start/end.....(HRS)  | 840/ 940 | 1025/1125 | 1210/1310 |
| Static pressure.....(IN.WC)   | -13.20   | -13.20    | -13.20    |
| Cross sectional area (SQ.FT)  | 1090.00  | 1090.00   | 1090.00   |
| Pitot tube coefficient.....   | .840     | .840      | .840      |
| Water in sample gas           |          |           |           |
| condenser.....(ML)            | 0.0      | 0.0       | 0.0       |
| impingers.....(GRAMS)         | 65.0     | 82.0      | 90.0      |
| desiccant.....(GRAMS)         | 28.0     | 16.0      | 15.0      |
| total.....(GRAMS)             | 93.0     | 98.0      | 105.0     |
| Gas meter coefficient.....    | 0.9992   | 0.9992    | 0.9992    |
| Barometric pressure...(IN.HG) | 29.12    | 29.12     | 29.12     |
| Avg. orif.pres.drop...(IN.WC) | 1.69     | 1.67      | 1.70      |
| Avg. gas meter temp...(DEF-F) | 81.7     | 87.5      | 90.4      |
| Volume through gas meter....  |          |           |           |
| at meter conditions...(CF)    | 43.75    | 43.76     | 44.20     |
| standard conditions.(DSCF)    | 41.63    | 41.20     | 41.39     |
| Total sampling time....(MIN)  | 60.00    | 60.00     | 60.00     |
| Nozzle diameter.....(IN)      | .249     | .249      | .249      |
| Avg.stack gas temp ..(DEG-F)  | 280      | 280       | 280       |
| Volumetric flow rate.....     |          |           |           |
| actual.....(ACFM)             | 3691895  | 3660354   | 3697315   |
| dry standard.....(DSCFM)      | 2243426  | 2209850   | 2216340   |
| Isokinetic variation.....(%)  | 99.7     | 100.2     | 100.4     |

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Northern States Power - Sherco  
Becker, Minnesota

Test No. 1  
Unit 3 Stack

Results of the Multi Metal Modified Method 5 (4M5) Sampling

|                              | Run 1    | Run 2     | Run 3     |
|------------------------------|----------|-----------|-----------|
| Date of run                  | 03-27-90 | 03-27-90  | 03-27-90  |
| Time run start/end.....(HRS) | 840/ 954 | 1025/1139 | 1210/1324 |
| Static pressure.....(IN.WC)  | -0.72    | -0.72     | -0.72     |
| Cross sectional area (SQ.FT) | 683.49   | 683.49    | 683.49    |
| Pitot tube coefficient.....  | .840     | .840      | .840      |
| Water in sample gas          |          |           |           |
| condenser.....(ML)           | 0.0      | 0.0       | 0.0       |
| impingers.....(GRAMS)        | 115.0    | 110.0     | 109.0     |
| desiccant.....(GRAMS)        | 10.0     | 7.0       | 4.0       |
| total.....(GRAMS)            | 125.0    | 117.0     | 113.0     |
| Gas meter coefficient.....   | 0.9980   | 0.9980    | 0.9980    |
| Barometric pressure..(IN.HG) | 28.98    | 28.98     | 28.98     |
| Avg. orif.pres.drop..(IN.WC) | 0.88     | 0.83      | 0.83      |
| Avg. gas meter temp..(DEF-F) | 63.4     | 62.8      | 64.0      |
| Volume through gas meter.... |          |           |           |
| at meter conditions...(CF)   | 33.22    | 32.38     | 32.27     |
| standard conditions.(DSCF)   | 32.45    | 31.67     | 31.49     |
| Total sampling time....(MIN) | 64.00    | 64.00     | 64.00     |
| Nozzle diameter.....(IN)     | .178     | .178      | .178      |
| Avg.stack gas temp..(DEG-F)  | 167      | 166       | 166       |
| Volumetric flow rate.....    |          |           |           |
| actual.....(ACFM)            | 2829194  | 2829323   | 2811269   |
| dry standard.....(DSCFM)     | 1950168  | 1965867   | 1962255   |
| Isokinetic variation.....(%) | 102.9    | 99.6      | 99.2      |

### 3.3 Results of the Hexavalent Chromium (Cr VI) Sampling

Interpoll Report No. 0-3005  
Northern States Power - Sherco  
Becker, Minnesota

Test No. 2  
Unit 3 Stack

Results of Hexavalent Chromium (Cr VI) Sampling

|                              | Run 1     | Run 2     | Run 3     |
|------------------------------|-----------|-----------|-----------|
| Date of run                  | 03-27-90  | 03-27-90  | 03-27-90  |
| Time run start/end.....(HRS) | 1430/1540 | 1550/1659 | 1710/1820 |
| Static pressure.....(IN.WC)  | -0.72     | -0.72     | -0.72     |
| Cross sectional area (SQ.FT) | 683.49    | 683.49    | 683.49    |
| Pitot tube coefficient.....  | .840      | .840      | .840      |
| Water in sample gas          |           |           |           |
| condenser.....(ML)           | 0.0       | 0.0       | 108.0     |
| impingers.....(GRAMS)        | 104.0     | 106.0     | 8.0       |
| desiccant.....(GRAMS)        | 5.0       | 8.0       | 0.0       |
| total.....(GRAMS)            | 109.0     | 114.0     | 116.0     |
| Gas meter coefficient.....   | 0.9980    | 0.9980    | 0.9980    |
| Barometric pressure..(IN.HG) | 28.80     | 28.80     | 28.80     |
| Avg. orif.pres.drop..(IN.WC) | 0.83      | 0.83      | 0.84      |
| Avg. gas meter temp..(DEF-F) | 70.1      | 67.2      | 74.1      |
| Volume through gas meter.... |           |           |           |
| at meter conditions...(CF)   | 32.66     | 32.55     | 32.92     |
| standard conditions.(DSCF)   | 31.30     | 31.37     | 31.32     |
| Total sampling time....(MIN) | 64.00     | 64.00     | 64.00     |
| Nozzle diameter.....(IN)     | .178      | .178      | .178      |
| Avg.stack gas temp ..(DEG-F) | 164       | 164       | 163       |
| Volumetric flow rate.....    |           |           |           |
| actual.....(ACFM)            | 2801458   | 2808571   | 2804604   |
| dry standard.....(DSCFM)     | 1957528   | 1950487   | 1944863   |
| Isokinetic variation.....(%) | 98.9      | 99.5      | 99.6      |

**APPENDIX A**

**SAMPLING TRAIN CALIBRATION DATA**

Interpoll Laboratories, Inc.  
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: June 13, 1990

Meter Box No. : 4 (Rockwell Dry Test Meter Serial No. 964551)

Date of Last Calibration: March 5, 1990

Calibration Technician: D. Van Hoever

Wet Test Meter No.: American Meter AL-20

| Date of Use    | Report No. | Initial Meter<br>Reading | Final Meter<br>Reading | Volume/Job<br>(cu. ft.) | Total Volume*<br>(cu. ft.) |
|----------------|------------|--------------------------|------------------------|-------------------------|----------------------------|
|                |            |                          |                        |                         |                            |
| March 6, 1990  | 0-2967     | 865.30                   | 1022.55                | 157.25                  | 157.25                     |
| March 27, 1990 | 0-3005     | 25.00                    | 166.50                 | 141.50                  | 298.75                     |

\* Total volume through meter since last calibration.

Bar. Press: 29.36 in. Hg

**INTERPOLL LABORATORIES, INC.**  
**METER CALIBRATION SHEET**

# 900 LEM V43

Control Module No. 24  
Serial No. DTM 964553  
Net Test Water No. AL-20  
Technician [Signature]

[illegible]

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

Approved by Verity Jones Date 3-3-10

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

Interpoll Laboratories, Inc.  
(612) 786-6020

**Nozzle Calibration  
Data Sheet**

Date of Calibration: March 27, 1990  
Technician: E. Trowbridge

Nozzle Number 5-4

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

| Position | Diameter<br>(inches) |
|----------|----------------------|
| 1        | 0.249                |
| 2        | 0.250                |
| 3        | 0.249                |
| Average: | 0.249                |

Interpoll Laboratories  
(612)786-6020

S-Type Pitot Tube Inspection Sheet

Pitobe No. 16-17

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 ( $P_B$ ) .461 IN.

Alignment:

4.  $\alpha_1 < 10^\circ$  0
5.  $\alpha_2 < 10^\circ$  0
6.  $B_1 < 5^\circ$  1
7.  $B_2 < 5^\circ$  1
8.  $Z < .125"$  .02
9.  $W < .0625"$  .02

Distance from Pitot to Probe Components:

10. Pitot to 0.500 IN. nozzle .758 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) .760 IN.

Date of Inspection:

Inspected by:

12-8-89

E. Fawcett

Interpoll Laboratories, Inc.  
(612) 786-6020

Meter Box Calibration and Usage Status

Date of Report: June 13, 1990

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

Date of Last Calibration: March 7, 1990

Calibration Technician: D. Van Hoever

Wet Test Meter No.: American Meter AL-20

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

\* Total volume through meter since last calibration.

3/7/90

**INTERPOL LABORATORIES, INC.**  
**METER CALIBRATION SHEET**

**900131 VJ3**

Control Module No.

Serial No. DTM 964.552

Wat Test Histon No. AL-20

Technician David Lane

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

Approved by Servatomo Date 3-15-90

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

Interpoll Laboratories, Inc.  
(612) 786-6020

Nozzle Calibration  
Data Sheet

Date of Calibration: March 27, 1990  
Technician: D. Van Hoever

Nozzle Number 4-3

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

| Position | Diameter<br>(inches) |
|----------|----------------------|
| 1        | 0.175                |
| 2        | 0.177                |
| 3        | 0.180                |
| Average: | 0.178                |

Interpoll Laboratories  
(612)786-6020

S-Type Pitot Tube Inspection Sheet

Pitobe No. 8-14

Pitot tube dimensions:

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

Alignment:

4.  $\alpha_1 < 10^\circ$  0
5.  $\alpha_2 < 10^\circ$  0
6.  $B_1 < 5^\circ$  1
7.  $B_2 < 5^\circ$  1
8.  $Z < .125"$  .03
9.  $W < .0625"$  .02

Distance from Pitot to Probe Components:

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

Date of Inspection:

12-8-89

Inspected by:

\_\_\_\_\_

Interpoll Laboratories, Inc.  
(612) 786-6060

EPA Method 5 Gas Metering System  
Quality Control Check Data Sheet

Job NSI/SH200

Date 3-26-90

Operator E. T. Trawick

Module No. 4

Instructions: Operate the control module at a flow rate equal to Q<sub>He</sub> for 10 minutes before attaching the umbilical. Record the following data:

Bar press 29.30 in. Hg.  $\tau =$  .9992 Q<sub>He</sub> 1.88 in. W.C.

| Time<br>(min)   | Volume<br>(CF)        | Meter Temp. (°F)               |        |
|-----------------|-----------------------|--------------------------------|--------|
|                 |                       | Inlet                          | Outlet |
| <del>0.0</del>  | (26.00)               | <del>71 66</del>               |        |
| 2.5             | 27.86                 | 71                             | 66     |
| 5.0             | 29.72                 | 75                             | 68     |
| 7.5             | 31.60                 | 78                             | 70     |
| 10              | 33.46                 | 80                             | 71     |
| <del>10.0</del> | V <sub>m</sub> = 7.46 | Avg (t <sub>m</sub> ) = 72.3°F |        |

Calculate Y<sub>en</sub> as follows:

$$Y_{en} = \frac{1.786}{\tau V_m} \left[ \frac{(t_m + 460)}{P_b} \right]^{0.5}$$

$$Y_{en} = \frac{1.786}{(.9992)(7.46)} \left[ \frac{(72.3 + 460)}{(29.30)} \right]^{0.5}$$

$$Y_{en} = \underline{1.02}$$

If Y<sub>en</sub> 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

S-432R

EPA Method 5 Gas Metering System  
Quality Control Check Data Sheet

Job NSP / SHERCO

Date 3-26-90

Operator S. BAINVILLE

Module No. 8

Instructions: Operate the control module at a flow rate equal to  $\Delta H_0$  for 10 minutes before attaching the umbilical. Record the following data:

Bar press 29.03 in. Hg.  $\tau =$  .9980  $\Delta H_0$  1.88 in. W.C.

| Time<br>(min)   | Volume<br>(CF) | Meter Temp. ( $^{\circ}$ F)              |        |
|-----------------|----------------|------------------------------------------|--------|
|                 |                | Inlet                                    | Outlet |
| <del>1.0</del>  | (511.57)       | <del>54 55</del>                         |        |
| 2.5             | 513.46         | 56                                       | 55     |
| 5.0             | 515.33         | 58                                       | 56     |
| 7.5             | 517.21         | 60                                       | 56     |
| 10              | 519.11         | 62                                       | 57     |
| <del>15.0</del> | $V_m = 7.54$   | Avg ( $t_m$ ) = <u>57.5</u> $^{\circ}$ F |        |

Calculate  $Y_{en}$  as follows:

$$Y_{en} = \frac{1.786}{\tau V_m} \left[ \frac{(t_m + 460)}{P_b} \right]^{0.5}$$

$$Y_{en} = \frac{1.786}{(.9980)(7.54)} \left[ \frac{(57.5) + 460}{(29.03)} \right]^{0.5}$$

$$Y_{en} = \underline{1.0019}$$

If  $Y_{en}$  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

S-432R

EPA Method 5 Gas Metering System  
Quality Control Check Data Sheet

Job N.S.P. SHERCO

Date 3/28/90

Operator Duane Van Hoever

Module No. 8

Instructions: Operate the control module at a flow rate equal to  $\Delta H_e$  for 10 minutes before attaching the umbilical. Record the following data:

Bar press 28.83 in. Hg.  $\tau =$  .9980  $\Delta H_e$  1.90 in. W.C.

| Time<br>(min)   | Volume<br>(CF) | Meter Temp. (°F)                |               |
|-----------------|----------------|---------------------------------|---------------|
|                 |                | Inlet                           | Outlet        |
| <del>0.0</del>  | ( 856 )        | <del>80</del>                   | <del>82</del> |
| 2.8             | 857.53         | 90                              | 82            |
| 4.0             | 859.13         | 91                              | 83            |
| 6.8             | 860.64         | 92                              | 84            |
| 10              | 863.79         | 92                              | 84            |
| <del>12.0</del> | $V_m = 7.79$   | Avg ( $t_m$ ) = <u>87.25</u> °F |               |

Calculate  $Y_{en}$  as follows:

$$Y_{en} = \frac{1.786}{\tau V_m} \left[ \frac{(t_m + 460)}{P_b} \right]^{0.5}$$

$$Y_{en} = \frac{1.786}{(.998)(7.79)} \left[ \frac{( ) + 460}{( )} \right]^{0.5}$$

$$Y_{en} = \underline{1.0008}$$

If  $Y_{en}$  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

S-432R

## Interpoll Laboratories, Inc.

Temperature Measurement Device  
Calibration Sheet

Unit under test:

Vendor ATKINS  
 Model 39658  
 Range -112.0°F to +1990°F °F  
 Date of Calibration 2-5-90  
 Serial Number PDT-1  
 Thermocouple Type   
 Technician E. T. LOW BRIDGE

## Method of Calibration:

- ☐ Comparison against ASTM mercury in glass thermometer using a thermostatted and insulated aluminum block design 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  $\pm 0.1\%$  of span (2100 °F)  $\pm 1$  degree (for negative temperatures add  $\pm 2$  degrees. The CL-300 simulates exactly the millivoltage of a Type K thermocouple at the indicated temperature.

| Desired Temp (°F)<br>Nominal | Temperature of Standard or Simulated Temp (°F) | Response of Unit Under Test (°F) | Deviation       |     |
|------------------------------|------------------------------------------------|----------------------------------|-----------------|-----|
|                              |                                                |                                  | $\Delta t$ (°F) | (%) |
| 0                            | 0                                              | 2.9                              | 2.9             | .63 |
| 100                          | 100                                            | 100.0                            | —               | —   |
| 200                          | 200                                            | 203                              | 3               | .45 |
| 300                          | 300                                            | 301                              | 1               | .13 |
| 400                          | 400                                            | 398                              | 2               | .23 |
| 500                          | 500                                            | 499                              | 3               | .31 |
| 600                          | 600                                            | 599                              | 1               | .09 |
| 700                          | 700                                            | 699                              | 1               | .08 |
| 800                          | 800                                            | 803                              | 3               | .23 |
| 900                          | 900                                            | 905                              | 5               | .36 |
| 1000                         | 1000                                           | 1008                             | 8               | .54 |
| 1100                         | 1100                                           | 1111                             | 11              | .70 |
| 1200                         | 1200                                           | 1216                             | 16              | .96 |
| 1300                         | 1300                                           | 1315                             | 15              | .85 |
| 1400                         | 1400                                           | 1418                             | 18              | .96 |
| 1500                         | 1500                                           | 1514                             | 14              | .71 |
| 1600                         | 1600                                           | 1614                             | 14              | .67 |
| 1700                         | 1700                                           | 1708                             | 8               | .37 |
| 1800                         | 1800                                           | 1804                             | 4               | .17 |
| 1900                         | 1900                                           | 1895                             | 5               | .21 |
| 2000                         | 2000                                           | 1988                             | 12              | .48 |
| 2100                         |                                                |                                  |                 |     |
|                              |                                                | Averages:                        | 6.4             | .43 |

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



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 ATKINS  
Model 39658-K Serial Number PDT-4  
Range -112.0°F to +1899°F °F Thermocouple Type CL-300  
Date of Calibration 2-2-90 Technician E. T. [Signature]

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  $\pm 0.1\%$  of span ( $2100^{\circ}\text{F}$ )  $\pm 1$  degree (for negative temperatures add  $\pm 2$  degrees. The CL-300 simulates exactly the millivoltage of a Type K thermocouple at the indicated temperature.

| Desired Temp (°F)<br>Nominal | Temperature of Standard or Simulated Temp (°F) | Response of Unit Under Test (°F) | Deviation       |     |
|------------------------------|------------------------------------------------|----------------------------------|-----------------|-----|
|                              |                                                |                                  | $\Delta t$ (°F) | (%) |
| 0                            | 0                                              | -2.7                             | 2.7             | .58 |
| 100                          | 100                                            | 95                               | 5               | .89 |
| 200                          | 200                                            | 148                              | 2               | .50 |
| 300                          | 300                                            | 296                              | 4               | .52 |
| 400                          | 400                                            | 392                              | 8               | .93 |
| 500                          | 500                                            | 481                              | 9               | .93 |
| 600                          | 600                                            | 592                              | 8               | .75 |
| 700                          | 700                                            | 692                              | 8               | .68 |
| 800                          | 800                                            | 796                              | 4               | .51 |
| 900                          | 900                                            | 897                              | 3               | .22 |
| 1000                         | 1000                                           | 1000                             | —               | —   |
| 1100                         | 1100                                           | 1102                             | 2               | .12 |
| 1200                         | 1200                                           | 1207                             | 7               | .42 |
| 1300                         | 1300                                           | 1305                             | 5               | .28 |
| 1400                         | 1400                                           | 1408                             | 8               | .43 |
| 1500                         | 1500                                           | 1515                             | 5               | .25 |
| 1600                         | 1600                                           | 1609                             | 9               | .14 |
| 1700                         | 1700                                           | 1697                             | 3               | .13 |
| 1800                         | 1800                                           | 1793                             | 7               | .30 |
| 1900                         | 1900                                           | 1883                             | 17              | .72 |
| 2000                         | 2000                                           | 1977                             | 23              | .93 |
| 2100                         |                                                |                                  |                 |     |
| Averages:                    |                                                |                                  | 6.36            | .46 |

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



Interpoll Laboratories, Inc.

Temperature Measurement Device  
Calibration Sheet

Unit under test:

Vendor GORDON  
Model 5310 K Serial Number PDT-10  
Range -112°F to 1999°F Thermocouple Type K  
Date of Calibration 3-5-90 Technician S. BAINVILLE

Method of Calibration:

- ☐ Comparison against ASTM mercury in glass thermometer using a thermostatted and insulated aluminum block designed to provide uniform temperature. The temperature is adjusted by adjusting the voltage on the block heater cartridge.
- ☒ Omega Model CL-300 Type K Thermocouple Simulator which provides 22 precise temperature equivalent millivolt signals. The CL-300 is cold junction compensated. Calibration accuracy is  $\pm 0.1\%$  of span ( $2100^{\circ}\text{F}$ )  $\pm 1$  degree (for negative temperatures add  $\pm 2$  degrees. The CL-300 simulates exactly the millivoltage of a Type K thermocouple at the indicated temperature.

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

OF = off scale response by unit under test ( $^{\circ}\text{F}$ )  
% dev =  $100 \Delta t / (460 + t)$



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

DIFFERENTIAL INCHES H<sub>2</sub>O

A-15

PULSATION RANGE

PROOF

101  
100  
99

CORRECT VOLUME = INDEX READING x PROOF + 100

20 40 60 80 100 120

FLOW RATE - CUBIC FEET OF AIR PER HOUR

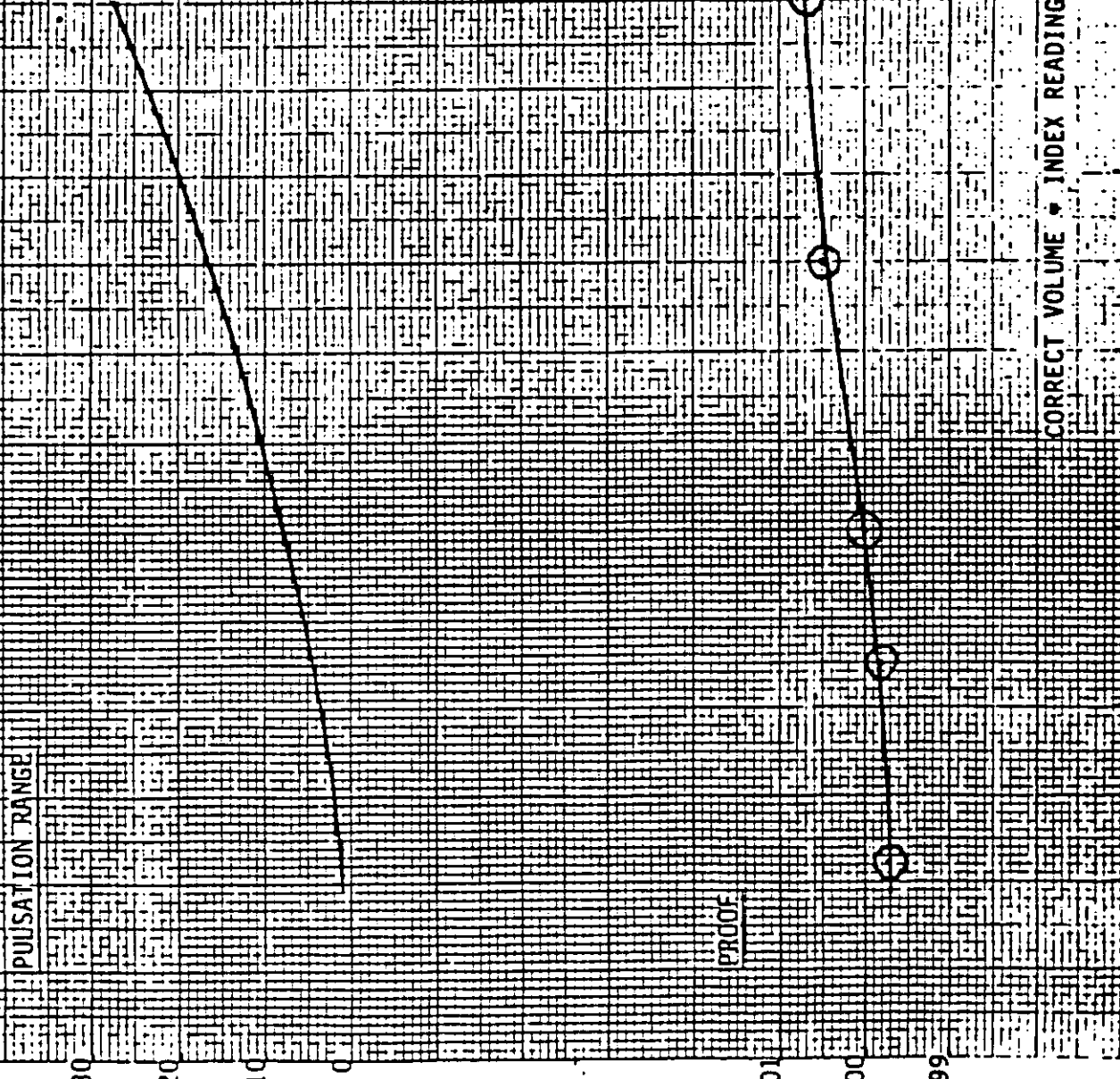
DAVID BANKS  
AUGUST, 1989

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

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

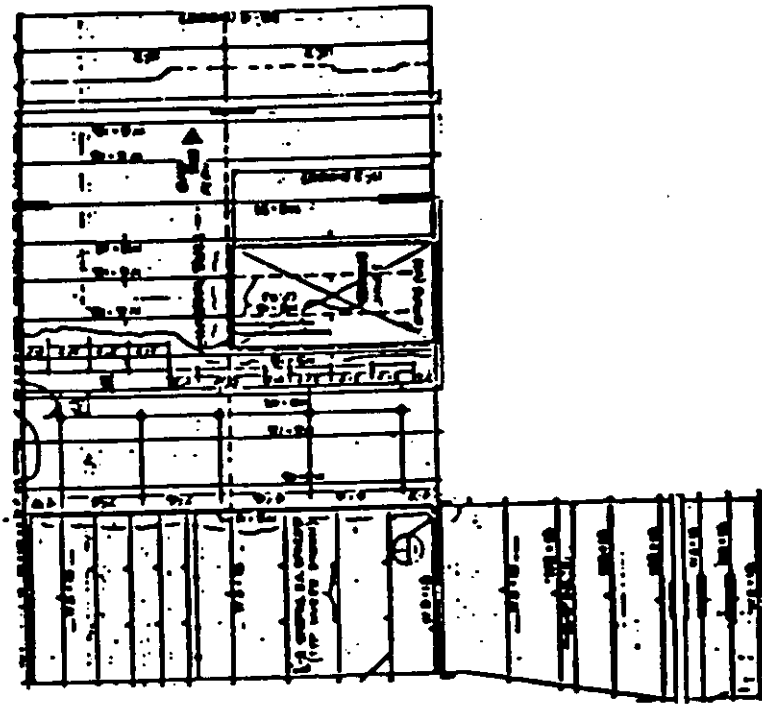
DIFFERENTIAL PRESSURE AND PROOF CALIBRATION CURVES

WET TEST METER

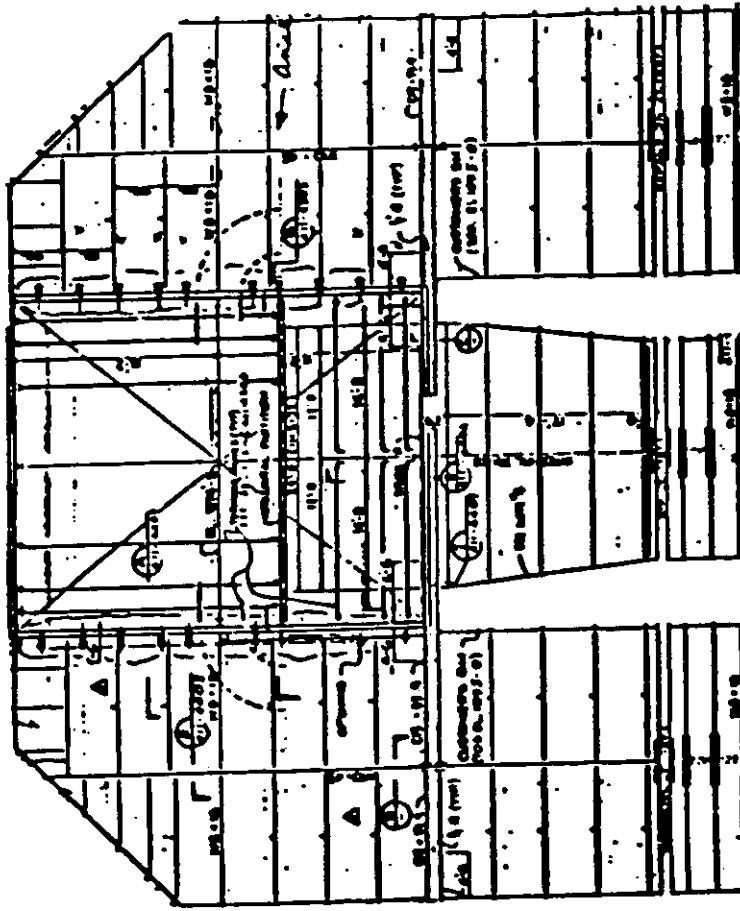


**APPENDIX B**

**LOCATION OF TEST PORTS**



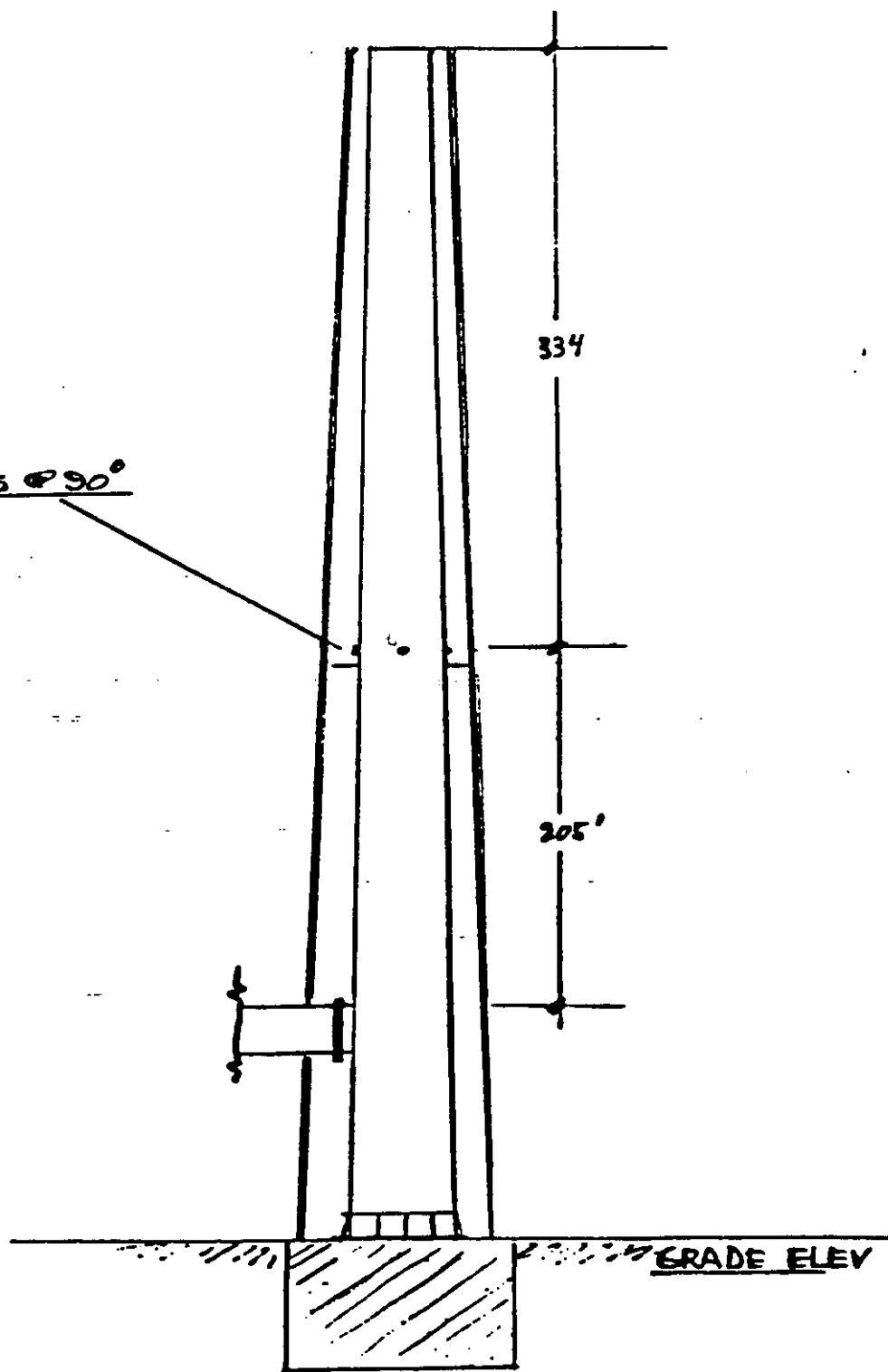
SIDE ELEVATION



FRONT ELEVATION

NSP SHERCO UNIT 3 AIR HEATER OUTLET TEST SITE

FOUR PORTS @ 90°  
D<sub>s</sub> = 29'-6"



LP 11/87  
REV 3/88

NOT TO SCALE

**APPENDIX C**

**4M5 FIELD DATA SHEETS - SDA INLET**

Job NSP-SHERO  
Source WATT 3 SDA INLET  
Test 1 Run 1 Date 3-27-90  
Stack dimen. 360 X 436 IN.  
Dry bulb \_\_\_\_\_ °F Wet bulb \_\_\_\_\_ °F  
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.  
Barometric pressure 29.12 in Hg  
Static pressure -13.2 in WD  
Operators E. T. Rowbridge - M. Kucner  
Pitot No. 17-16 V Cp 840

[illegible]

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

## INTERPOL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job NSP/GHRCO  
Source UNIT 3 GDA INLET  
Method YMS Filter holder: Am-STACK SS

Date 3-27-90 Test 1 Run 1  
No. of traverse points 3  
Filter type: SS INSTACK

### Sample Train Leak Check:

Pretest:  $\leq 0.02$  cfm at 15 in. Hg. (vac) ☒  
Posttest:  $\leq 0$  cfm at 15 in. Hg. (vac) ☒

**Particulate Catch Data:**

No. of filters used:      Recovery solvent(s)

SS # 20

~~A~~ acetone

other (s)

No. of probe wash bottles:

Sample recovered by: ET

**Condensate Data:**

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | -          | 200  | 65         |
| Impinger No. 2 |            | 100  |            |
| Impinger No. 3 |            | 200  |            |
| Condenser      |            |      |            |
| Desiccant      | 1391       | 1363 | 28         |
|                |            |      |            |
| Total          |            |      | 93         |

**Integrated Gas Sampling Data:**

Bag Pump No. 39 Box No. 20 Bag No. 1

**Bag Material:** 5-layer Aluminized Tedlar **Size:** 44 L

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

Time start: 0841 (HRS) Time end: 0939 (HRS)

Sampling rate: 400 cc/min Operator: ET

S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 7

CF-023

## INTERPOL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

405 NSP/5NCO

Source SDA INLOS  
Date 3-27-80

Run

**46**

**Deputy**

20

**FOR JOURNALISTS**

5. TPW 1050 - 1250000

~~20 MI 327 HY 2~~

9912

**4-1-10-10.**

1998

# MISSION

058.17

# THE

# Unit 1

1

1

**29 IN.**

[illegible]

# INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job NSP/SHARCO Date 3-27-90 Test 1 Run 2  
 Source UNIT 3 SDA INLET No. of traverse points 3  
 Method 4MS Filter holder: IN STACK-SS Filter type: SS

## Sample Train Leak Check:

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

## Particulate Catch Data:

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

SS# 23 ☒ acetone                       
☐ other(s)                     

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

## Condensate Data:

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | 80         | 100  | 80         |
| Impinger No. 2 |            | 100  | 2          |
| Impinger No. 3 | 2          | 200  |            |
| Condenser      |            |      |            |
| Desiccant      | 1411       | 1395 | 16         |
|                |            |      |            |
| Total          |            |      | 98         |

## Integrated Gas Sampling Data:

Bag Pump No. 39 Box No. 20 Bag No. 2  
 Bag Material: 5-layer Aluminized Tedlar Size: 44 L  
 Pretest leak check: 0 cc/min at 15 in. Hg.  
 Time start: 1026 (HRS) Time end: 1124 (HRS)  
 Sampling rate: 400 cc/min Operator: ET  
 S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 7

CF-023

JUB NSP/5HRCO  
 Source CHART 3 5 PA TAPET  
 Date 8-27-60 RUN 2  
 1961 1 RUN 2  
 Operators ET-MK  
 Meter Box No. 2 HV 1.88 IN UC  
 Generator count. 1,002

Pitot No. 12-16V CP 840  
 Bar. Press. 28.2 IN H<sub>2</sub>O  
 Nozzle No. 5-2 Nozzle Dia. 2.52 IN.

[illegible]

# INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job NSP/SHORO Date 3-27-80 Test 1 Run 3  
 Source Unit 3 SDA INLET No. of traverse points 3  
 Method 4/MS Filter holder: INSTACK-SS Filter type: SS

## Sample Train Leak Check:

Pretest: ( 0.02 cfm at 15 in. Hg. (vac) ~~1~~  
 Posttest: 0 cfm at 14 in. Hg. (vac) ~~1~~

## Particulate Catch Data:

No.s of filters used: SS #15 Recovery solvent(s):  
☒ acetone                       
☐ other(s)                       
 No. of probe wash bottles: 1  
 Sample recovered by: ET

## Condensate Data:

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | -          |      |            |
| Impinger No. 2 |            |      | 88         |
| Impinger No. 3 |            |      | 2          |
| Condenser      |            |      |            |
| Desiccant      | 1419       | 1404 | 15         |
|                |            |      |            |
| Total          |            |      | 105        |

## Integrated Gas Sampling Data:

Bag Pump No. 159 Box No. 20 Bag No. 3  
 Bag Material: 5-layer Aluminized Tedlar Size: 44 L  
 Pretest leak check: 0 cc/min at 15 in. Hg.  
 Time start: 1211 (HRS) Time end: 1309 (HRS)  
 Sampling rate: 400 cc/min Operator: ET  
 S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 7

CF-023

INTERPOLL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

JMB NSP/SPN  
 Source 44173  
 Date 4-2-80  
 Run E

Operator ET-MAX  
 Motor Box No. 2  
 Generator 1.999X

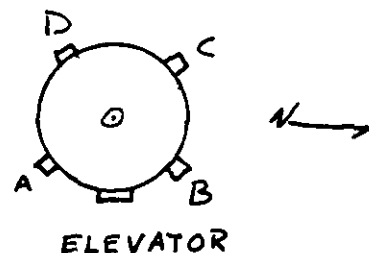
12-16V CP .540  
 Bar. Press. 2412 IN HG H2O  
 Nozzle No. 5-2 Nozzle Dia. .242 IN.

[illegible]

**APPENDIX D**

**4M5 FIELD DATA SHEETS - STACK**

Job NSD / SHERCO  
Source UNIT # 3 STACK  
Test 1 Run 0 Date 3-27-90  
Stack dimen. 354 IN.  
Dry bulb        °F Wet bulb        °F  
Manometer: ☒ Reg. ☐ Exp. ☐ Elec.  
Barometric pressure 28.98 in Hg  
Static pressure -.72 in WC  
Operators DBH + SLB  
Fitot No. 14-8 Cp 840



### Schematic of Cross Section

[illegible]

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

S-392.1

INTERPOL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job NSP / SHERCO  
 Source UNIT #3  
 Method 4MS Filter holder: SS

Date 3-27-90 Test 1 Run 1  
 No. of traverse points 10  
 Filter type: glass fiber-pall

**Sample Train Leak Check:**

Pretest: { 0.02 cfm at 15 in. Hg. (vac) ☒  
 Posttest: { 0 cfm at 5 in. Hg. (vac) ☒

**Particulate Catch Data:**

No.s of filters used: 4MS-0109 Recovery solvent(s):  
☒ acetone  
☒ other(s) Nitric  
 No. of probe wash bottles: 1  
 Sample recovered by: [Signature]

**Condensate Data:**

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 |            |      |            |
| Impinger No. 2 | 515        | 400  | 115        |
| Impinger No. 3 |            |      |            |
| Condenser      |            |      |            |
| Desiccant      | 1498       | 1438 | 10         |
|                |            |      |            |
| Total          |            |      | 125        |

**Integrated Gas Sampling Data:**

Bag Pump No. B3 Box No. 17 Bag No. 1  
 Bag Material: 5-layer Aluminized Tedlar Size: 44 L  
 Pretest leak check: 0 cc/min at 14 in. Hg.  
 Time start: 0846 (HRS) Time end: 0953 (HRS)  
 Sampling rate: 400 cc/min Operator: [Signature]  
 S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 8

CF-023



INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job N.S.P. SHERCO III  
 Source UNIT # 3 Stack  
 Method 4ms Filter holder: SS

Date 3/27/90 Test 1 Run 2  
 No. of traverse points 16  
 Filter type: glass fiber - pall

**Sample Train Leak Check:**

Pretest: ( 0.02 cfm at 15 in. Hg. (vac) ☒  
 Posttest: .005 cfm at 6 in. Hg. (vac) ☒

**Particulate Catch Data:**

No.s of filters used: \_\_\_\_\_ Recovery solvent(s)  
CAIO ☒ acetone  
☒ other(s) NITRIC  
 No. of probe wash bottles: 1  
 Sample recovered by: JEH & S.L.B.

**Condensate Data:**

| Item           | Weight(g) |      |            |
|----------------|-----------|------|------------|
|                | Final     | Tare | Difference |
| Impinger No. 1 | -         | 400  |            |
| Impinger No. 2 | 510       |      | 110        |
| Impinger No. 3 |           |      |            |
| Condenser      |           |      |            |
| Desiccant      | 1341      | 1334 | 7          |
|                |           |      |            |
| Total          |           |      | 117        |

**Integrated Gas Sampling Data:**

Bag Pump No. B3 Box No. 17 Bag No. 1  
 Bag Material: 5-layer Aluminized Tedlar Size: 44 L  
 Pretest leak check: 0 cc/min at 14 in. Hg.  
 Time start: 10<sup>30</sup> (HRS) Time end: 11<sup>39</sup> (HRS)  
 Sampling rate: 400 cc/min Operator: JEH  
 S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 8

CF-023

INTERPOL LABDRATDRIES EPA METHOD 5 FIELD DATA SHEET

U.S.P. SHERCO

Source UNIT # 3 Stack  
Date 3/27/90 1001, HUN 2

**D** **M** **C**

Part 2-5-63

Pilot No. 1114-8 CP 94  
Bar. Press. 28.48 in Hg  
Nautic No. 4-3 Nautic Dia

[illegible]

INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job N.S.P. SHERO  
 Source UNIT # 3 Stack  
 Method 4m2 Filter holder: 55

Date 3/27/90 Test 1 Run 3  
 No. of traverse points 16  
 Filter type: glass fiber - pall

**Sample Train Leak Check:**

Pretest: ( 0.02 cfm at 15 in. Hg. (vac) ☒  
 Posttest: 0.00 cfm at 5 in. Hg. (vac) ☒

**Particulate Catch Data:**

No.s of filters used: 0208 Recovery solvent(s)  
☒ acetone  
☒ other(s) NITRIL  
 No. of probe wash bottles: 1  
 Sample recovered by: DOH # 9.03

**Condensate Data:**

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | -          | 400  |            |
| Impinger No. 2 | 509        |      | 109        |
| Impinger No. 3 |            |      |            |
| Condenser      |            |      |            |
| Desiccant      | 1452       | 1448 | 4          |
|                |            |      |            |
| Total          |            |      | 113        |

**Integrated Gas Sampling Data:**

Bag Pump No. B3 Box No. 17 Bag No. 3  
 Bag Material: 5-layer Aluminized Tedlar Size: 44 L  
 Pretest leak check: 0 cc/min at 14 in. Hg.  
 Time start: 1210 (HRS) Time end: 1324 (HRS)  
 Sampling rate: 400 cc/min Operator: DOH  
 S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 8

CF-023

100 N. S. P. SHERCO

Job \_\_\_\_\_ N.S.P. SHERCO  
Source \_\_\_\_\_ UNIT #3 STACK  
Date 3/27/90 1001 / NO

Deputy Director  
Deputy Director  
Deputy Director

0867-0980  
11-2-87  
05-05-83

**25. 11. 2017**

**Pittor No.  
Bar. Pr. N.  
Nozzio**

$\frac{89}{\text{CP} - \text{H}_2}$

10/3 11

[illegible]

APPENDIX E

CHROMIUM SAMPLING TRAIN FIELD DATA SHEETS



INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job N.S.P. SHERGO Date 3/27/90 Test 2 Run 1  
 Source UNIT # 3 Stack No. of traverse points 16  
 Method Filter holder: S.S. Filter type: teflon

**Sample Train Leak Check:**

Pretest: ( 0.02 cfm at 15 in. Hg. (vac) ☒  
 Posttest: 0.00 cfm at 5 in. Hg. (vac) ☒

**Particulate Catch Data:**

No.s of filters used: \_\_\_\_\_ Recovery solvent(s) \_\_\_\_\_  
 \_\_\_\_\_ ☐ acetone  
 \_\_\_\_\_ ☒ other(s) 1 N NaOH  
 No. of probe wash bottles: 1  
 Sample recovered by: DUP

**Condensate Data:**

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | -          | 300  | 104        |
| Impinger No. 2 |            |      |            |
| Impinger No. 3 |            |      |            |
| Condenser      |            |      |            |
| Desiccant      | 1348       | 1343 | 5          |
|                |            |      |            |
| Total          |            |      | 109        |

**Integrated Gas Sampling Data:**

Bag Pump No. B3 Box No. 22 Bag No. 1  
 Bag Material: 5-layer Aluminized Tedlar Size: 44 L  
 Pretest leak check: 0 cc/min at 14 in. Hg.  
 Time start: 1430 (HRS) Time end: 1540 (HRS)  
 Sampling rate: 460 cc/min Operator DUP  
 S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 8

CF-023

405 N.S.P. SWEDS

N.S.P. SHIELD

SOURCE UNIT #3 Stack

3/27/92 1981 2 JUN 7

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# H

**Departments**

Water, Box

4444

875 No 4th St

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**Abstract**

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[illegible]

# INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job N.S.P. SHERCO Date 3/27/90 Test 2 Run 2  
 Source Unit #3 Stack No. of traverse points 16  
 Method Filter holder: S.S. Filter type: teflon

## Sample Train Leak Check:

Pretest: ( 0.02 cfm at 15 in. Hg. (vac) ☒  
 Posttest: 0.00 cfm at 5 in. Hg. (vac) ☒

## Particulate Catch Data:

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

☐ acetone  
☒ other(s) CH<sub>2</sub>Cl<sub>2</sub>

No. of probe wash bottles: 1

Sample recovered by: [Signature]

## Condensate Data:

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | -          |      |            |
| Impinger No. 2 | 406        | 300  | 106        |
| Impinger No. 3 |            |      |            |
| Condenser      |            |      |            |
| Desiccant      | 1460       | 1452 | 8          |
|                |            |      |            |
| Total          |            |      | 114        |

## Integrated Gas Sampling Data:

Bag Pump No. B3 Box No. 22 Bag No. 2

Bag Material: 5-layer Aluminized Tedlar Size: 44 L

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

Time start: 1550 (HRS) Time end: 1659 (HRS)

Sampling rate: 400 cc/min Operator: [Signature]

S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 8

CF-023

106 N.S.P. SHERCO

JOB \_\_\_\_\_ N.S.P. SHERCO  
SOURCES UNIT B 3 Stack  
DB1 3127190 1961 2 HUN 2

Operator: TH 9513

Operator ~~744~~ 9513  
Meter Box No. 8 AM 1.88 IN DE  
Cassette cost. — 9.80

Pilot No. V14-8 CP 88  
Bar. Press. 23.80 in Hg H2O 15 x  
Naze 10 No. 9-3 Naze 10 Dia 1.8 in.

| Traverse Point No. | Sampling Time (min) | Sample Volume (cf) | Velocity Head (inWC) | Drifts Meter (inWC) | VAC. inHg | Temperatures (°F) |       |     |      |        | Oxygen (xv/v) |         |     |
|--------------------|---------------------|--------------------|----------------------|---------------------|-----------|-------------------|-------|-----|------|--------|---------------|---------|-----|
|                    |                     |                    |                      |                     |           | Stack             | Probe | Dyn | Leq. | Gas In |               | Gas Out |     |
| 1550               |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
| 4                  | 4                   | 653.50             | 1.20                 | .83                 | 5.54      | 25                | 164   |     |      | 40     | 68            | 66      | 6.5 |
| 3                  | 8                   | 655.58             | 1.20                 | .83                 | 7.57      | 2.5               | 164   |     |      |        | 68            | 72      | 6.4 |
| 2                  | 12                  | 657.62             | 1.30                 | .9                  | 9.70      | 2.5               | 164   |     |      |        | 74            | 68      | 6.3 |
| 1                  | 16                  | 659.74             | 1.20                 | .83                 | 1.75      | 2.5               | 164   |     |      | 39     | 75            | 69      | 6.2 |
| 4                  | 20                  | 661.77             | 1.20                 | .84                 | 3.81      | 2.5               | 163   |     |      |        | 72            | 68      | 6.4 |
| 3                  | 24                  | 663.90             | 1.15                 | .80                 | 5.81      | 2.5               | 163   |     |      |        | 66            | 66      | 6.3 |
| 2                  | 28                  | 665.93             | 1.20                 | .83                 | 7.85      | 3.0               | 163   |     |      | 38     | 66            | 63      | 6.3 |
| 1                  | 32                  | 667.90             | 1.10                 | .76                 | 9.78      | 3.0               | 163   |     |      |        | 64            | 61      | 6.4 |
| 4                  | 36                  | 669.79             | 1.25                 | .86                 | 1.84      | 3.0               | 163   |     |      |        | 64            | 61      | 6.3 |
| 3                  | 40                  | 671.80             | 1.20                 | .82                 | 3.86      | 3.0               | 164   |     |      | 38     | 65            | 61      | 6.3 |
| 2                  | 44                  | 673.89             | 1.20                 | .82                 | 5.88      | 3.0               | 164   |     |      |        | 67            | 62      | 6.4 |
| 1                  | 48                  | 675.93             | 1.10                 | .76                 | 7.82      | 3.0               | 164   |     |      |        | 70            | 64      | 6.4 |
| 4                  | 52                  | 677.82             | 1.25                 | .86                 | 9.89      | 3.0               | 164   |     |      | 38     | 70            | 64      | 6.4 |
| 3                  | 56                  | 679.88             | 1.30                 | .90                 | 2.01      | 3.0               | 164   |     |      |        | 73            | 65      | 6.3 |
| 2                  | 60                  | 682.00             | 1.25                 | .86                 | 4.08      | 3.0               | 164   |     |      |        | 75            | 66      | 6.4 |
| 1                  | 64                  | 684.09             | 1.10                 | .76                 | 6.04      | 3.0               | 164   |     |      | 39     | 75            | 67      | 6.3 |
|                    | (1654)              |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
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|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
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|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
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|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |
|                    |                     |                    |                      |                     |           |                   |       |     |      |        |               |         |     |

# INTERPOLL LABORATORIES EPA METHOD 5/17 SAMPLE LOG SHEET

Job N.S.P. SHERLO Date 3/27/90 Test 2 Run 3  
 Source UNIT # 3 Stack No. of traverse points       
 Method      Filter holder: SS Filter type: teflon

## Sample Train Leak Check:

Pretest: { 0.02 cfm at 15 in. Hg. (vac) ☒  
 Posttest: { 0.00 cfm at 5 in. Hg. (vac) ☒

## Particulate Catch Data:

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

     ☐ acetone  
     ☒ other(s) 1 N. NaOH

No. of probe wash bottles:     

Sample recovered by: DW

## Condensate Data:

| Item           | Weight (g) |      |            |
|----------------|------------|------|------------|
|                | Final      | Tare | Difference |
| Impinger No. 1 | -          | 300  |            |
| Impinger No. 2 | 408        |      | 108        |
| Impinger No. 3 |            |      |            |
| Condenser      |            |      |            |
| Desiccant      | 1357       | 1349 | 8          |
|                |            |      |            |
| Total          |            |      | 116        |

## Integrated Gas Sampling Data:

Bag Pump No. B3 Box No. 22 Bag No. 3

Bag Material: 5-layer Aluminized Tedlar Size: 44 L

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

Time start: 1710 (HRS) Time end: 1820 (HRS)

Sampling rate: 400 cc/min Operator: DW

S/N of O<sub>2</sub> Analyzer used to monitor train outlet: 8

CF-023

INTERPOL LABORATORIES EPA METHOD 5 FIELD DATA SHEET

N.S.P. SHERO.

JOB \_\_\_\_\_  
SOURCE \_\_\_\_\_  
DATE 3/27/90 \_\_\_\_\_  
N.S.P. SHERID  
Unit 23 - Sick  
1981 2 Nov 3

22  
 23  
 24  
 25  
 26  
 27  
 28  
 29  
 30  
 31  
 32

2855-8  
37 HX  
DVM + HAD

NAME

Pistol No.  
Bar. Press.  
Nozzle No.

018.

LP 1000  
1000 H2O 15 X  
1000 DIB 172 IN.

[illegible]

**APPENDIX F**

**LABORATORY DATA SHEETS FOR METHOD 3**

Interpoll Laboratories  
(612) 786-6020

Chain of Custody  
Sample Deposition Sheet

Job NSP / SHERCO  
Team Leader DVH  
Date Submitted 3-27-90  
Test No. 1

Source UNIT #3  
Test Site STACK  
Date of Test 3-27-90  
No. of Runs Completed 3

| No. of Samples | Type of Sample                                                                                                                                                                                                                                                                                              | Analysis Required                                                                                                                                                                                                                                                                                          | Comments                       |
|----------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------|
| 4              | Probe Wash:<br><input checked="" type="checkbox"/> Acetone<br><input type="checkbox"/> D.I. Water                                                                                                                                                                                                           | <input type="checkbox"/> As per EPA M-5<br><input checked="" type="checkbox"/> Other <u>Metals</u>                                                                                                                                                                                                         | + Blank                        |
| 4              | Filter:<br><input checked="" type="checkbox"/> 4" G.F.<br><input type="checkbox"/> S.S. Thimble<br><input type="checkbox"/> 2.5" G.F.<br><input type="checkbox"/> 47 mm G.F.                                                                                                                                | <input type="checkbox"/> As per EPA M-5<br><input type="checkbox"/> As per EPA M-17<br><input checked="" type="checkbox"/> Other <u>Metals</u>                                                                                                                                                             | + BLANK                        |
| 4 pairs        | Impinger Catch:<br><input type="checkbox"/> D.I. Water<br><input type="checkbox"/> 3% H <sub>2</sub> O <sub>2</sub><br><input type="checkbox"/> 4MS Hg Only<br><input checked="" type="checkbox"/> 4MS Metals<br><input type="checkbox"/> 1.0 N NaOH<br><input checked="" type="checkbox"/> Other <u>Hg</u> | <input type="checkbox"/> MN Protocol<br><input type="checkbox"/> WI Protocol<br><input type="checkbox"/> EPA M-6 or 8<br><input type="checkbox"/> Acid Gases<br><input type="checkbox"/> Formaldehyde<br><input checked="" type="checkbox"/> Metals<br><input checked="" type="checkbox"/> Other <u>Hg</u> | 11                             |
| 3              | Integrated Gas sample                                                                                                                                                                                                                                                                                       | <input checked="" type="checkbox"/> As per EPA M-3<br><input type="checkbox"/> As per EPA M-10<br><input type="checkbox"/> Other                                                                                                                                                                           |                                |
|                | Oxides of Nitrogen (NO <sub>x</sub> )                                                                                                                                                                                                                                                                       | <input type="checkbox"/> As per EPA M-7A<br><input type="checkbox"/> Other                                                                                                                                                                                                                                 | Date _____<br>Time (HRS) _____ |
|                | <input type="checkbox"/> Fuel Sample<br><input type="checkbox"/> Aggregate                                                                                                                                                                                                                                  | <input type="checkbox"/> Attached fuel Form #S-0163RRR                                                                                                                                                                                                                                                     |                                |
|                | Particle Size                                                                                                                                                                                                                                                                                               | <input type="checkbox"/> X-Ray Sedigraph<br><input type="checkbox"/> Bahco Method<br><input type="checkbox"/> Other                                                                                                                                                                                        |                                |
|                | Audit Samples<br><input type="checkbox"/> Sulfur Dioxide<br><input type="checkbox"/> Oxides of Nit.<br><input type="checkbox"/> Other                                                                                                                                                                       | <input type="checkbox"/> As per EPA M-6<br><input type="checkbox"/> As per EPA M-7A<br><input type="checkbox"/> 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

EPA Method 3 Data Reporting Sheet  
Orsat Analysis

Job NSP / Shercc Source Unit #3  
Team Leader DVH Test Site Stack  
Date Submitted 3-27-90 Date of Test 3-27-90  
Test No. 1 No. of Runs Completed 3  
Date of Analysis 3-28-90 Technician RL

| Test/<br>Run | Sample<br>Log Number<br>and Type | No.<br>of<br>An. | Buret Readings (ml) |                       |                      | Conc.<br>CO <sub>2</sub><br>%v/v Dry | Conc.<br>O <sub>2</sub><br>%v/v Dry | F <sub>0</sub> |
|--------------|----------------------------------|------------------|---------------------|-----------------------|----------------------|--------------------------------------|-------------------------------------|----------------|
|              |                                  |                  | Zero Pt.            | After CO <sub>2</sub> | After O <sub>2</sub> |                                      |                                     |                |
| 1/1          | 9242-36<br>□ B □ F               | 1                | 0.00                | 13.50                 | 20.00                | 13.50                                | 6.50                                | 1.07           |
|              |                                  | 2                | 0.00                | 13.50                 | 20.00                | 13.50                                | 6.50                                | 1.07           |
|              |                                  | Avg              |                     |                       |                      | 13.50                                | 6.50                                |                |
| 1/2          | 9242-37<br>□ B □ F               | 1                | 0.00                | 13.80                 | 20.00                | 13.80                                | 6.20                                | 1.07           |
|              |                                  | 2                | 0.00                | 13.80                 | 20.00                | 13.80                                | 6.20                                | 1.07           |
|              |                                  | Avg              |                     |                       |                      | 13.80                                | 6.20                                |                |
| 1/3          | 9242-38<br>□ B □ F               | 1                | 0.00                | 13.90                 | 20.00                | 13.90                                | 6.10                                | 1.07           |
|              |                                  | 2                | 0.00                | 13.90                 | 20.00                | 13.90                                | 6.10                                | 1.07           |
|              |                                  | Avg              |                     |                       |                      | 13.90                                | 6.10                                |                |
|              | □ B □ F                          | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | □ B □ F                          | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | □ B □ F                          | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | □ B □ F                          | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | □ B □ F                          | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | □ B □ F                          | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |

- ☒ Ambient Air QA Check  
☐ Orsat Analyzer System Leak Check  
☐ F<sub>0</sub> Within EPA M-3 Guidelines  
for fuel type.

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

EPA Method 3 Guidelines  
Fuel Type F<sub>0</sub> 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            | 1.434-1.596 |
| Butane             | 1.405-1.553 |
| Wood/Wood Bark     | 1.000-1.100 |

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

Interpoll Laboratories  
(612) 786-6020

Chain of Custody  
Sample Deposition Sheet

Job NSP/SHelco  
Team Leader E. T. Newthel  
Date Submitted 3-27-90  
Test No. 1

Source Unit 3  
Test Site SDA Unit 3  
Date of Test 3-27-90  
No. of Runs Completed 3

| No. of Samples | Type of Sample                                                                                                                                                                                                                                                                         | Analysis Required                                                                                                                                                                                                                                                          | Comments                       |
|----------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------|
| <u>1</u>       | Probe Wash:<br><input checked="" type="checkbox"/> Acetone<br><input type="checkbox"/> D.I. Water                                                                                                                                                                                      | <input checked="" type="checkbox"/> As per EPA M-5<br><input checked="" type="checkbox"/> Other <u>metals</u>                                                                                                                                                              |                                |
| <u>2</u>       | Filter:<br><input type="checkbox"/> 4" G.F.<br><input checked="" type="checkbox"/> 6.5" Thimble<br><input type="checkbox"/> 2.5" G.F.<br><input type="checkbox"/> 47 mm G.F.                                                                                                           | <input type="checkbox"/> As per EPA M-5<br><input type="checkbox"/> As per EPA M-17<br><input checked="" type="checkbox"/> Other                                                                                                                                           |                                |
| <u>8</u>       | Impinger Catch:<br><input type="checkbox"/> D.I. Water<br><input type="checkbox"/> 3% H <sub>2</sub> O <sub>2</sub><br><input type="checkbox"/> 4M5 Hg Only<br><input checked="" type="checkbox"/> 4M5 Metals<br><input type="checkbox"/> 1.0 N NaOH<br><input type="checkbox"/> Other | <input type="checkbox"/> MN Protocol<br><input type="checkbox"/> WI Protocol<br><input type="checkbox"/> EPA M-6 or 8<br><input type="checkbox"/> Acid Gases<br><input type="checkbox"/> Formaldehyde<br><input type="checkbox"/> Metals<br><input type="checkbox"/> Other |                                |
| <u>3</u>       | Integrated Gas sample                                                                                                                                                                                                                                                                  | <input checked="" type="checkbox"/> As per EPA M-3<br><input type="checkbox"/> As per EPA M-10<br><input type="checkbox"/> Other                                                                                                                                           |                                |
|                | Oxides of Nitrogen (NO <sub>x</sub> )                                                                                                                                                                                                                                                  | <input type="checkbox"/> As per EPA M-7A<br><input type="checkbox"/> Other                                                                                                                                                                                                 | Date _____<br>Time (HRS) _____ |
|                | <input type="checkbox"/> Fuel Sample<br><input type="checkbox"/> Aggregate                                                                                                                                                                                                             | <input type="checkbox"/> Attached fuel Form #S-0163RRR                                                                                                                                                                                                                     |                                |
|                | Particle Size                                                                                                                                                                                                                                                                          | <input type="checkbox"/> X-Ray Sedigraph<br><input type="checkbox"/> Bahco Method<br><input type="checkbox"/> Other                                                                                                                                                        |                                |
|                | Audit Samples<br><input type="checkbox"/> Sulfur Dioxide<br><input type="checkbox"/> Oxides of Nit.<br><input type="checkbox"/> Other                                                                                                                                                  | <input type="checkbox"/> As per EPA M-6<br><input type="checkbox"/> As per EPA M-7A<br><input type="checkbox"/> 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

EPA Method 3 Data Reporting Sheet  
Orsat Analysis

Job NSP / Shenco Source Unit #3  
Team Leader ET Test Site Unit #3  
Date Submitted 3-27-96 Date of Test 3-27-96  
Test No. 1 No. of Runs Completed 3  
Date of Analysis 3-28-96 Technician RL

| Test/<br>Run | Sample<br>Log Number<br>and Type | No.<br>of<br>An. | Buret Readings (ml) |                       |                      | Conc.<br>CO <sub>2</sub><br>%v/v Dry | Conc.<br>O <sub>2</sub><br>%v/v Dry | F <sub>o</sub> |
|--------------|----------------------------------|------------------|---------------------|-----------------------|----------------------|--------------------------------------|-------------------------------------|----------------|
|              |                                  |                  | Zero Pt.            | After CO <sub>2</sub> | After O <sub>2</sub> |                                      |                                     |                |
| 1/1          | 4242-17<br>B O F                 | 1                | 0.00                | 13.30                 | 20.00                | 13.30                                | 6.70                                | 1.07           |
|              |                                  | 2                | 0.00                | 13.30                 | 20.00                | 13.30                                | 6.70                                | 1.07           |
|              |                                  | Avg              |                     |                       |                      | 13.30                                | 6.70                                |                |
| 1/2          | 4242-18<br>B O F                 | 1                | 0.00                | 13.40                 | 19.90                | 13.40                                | 6.50                                | 1.07           |
|              |                                  | 2                | 0.00                | 13.40                 | 19.90                | 13.40                                | 6.50                                | 1.07           |
|              |                                  | Avg              |                     |                       |                      | 13.40                                | 6.50                                |                |
| 1/3          | 4242-19<br>B O F                 | 1                | 0.00                | 13.50                 | 19.90                | 13.50                                | 6.40                                | 1.07           |
|              |                                  | 2                | 0.00                | 13.50                 | 19.90                | 13.50                                | 6.40                                | 1.07           |
|              |                                  | Avg              |                     |                       |                      | 13.50                                | 6.40                                |                |
|              | B O F                            | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B O F                            | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B O F                            | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B O F                            | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B O F                            | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B O F                            | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |

- ☒ Ambient Air QA Check  
☒ Orsat Analyzer System Leak Check  
☒ F<sub>o</sub> Within EPA M-3 Guidelines  
for fuel type.

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

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

EPA Method 3 Guidelines

| Fuel Type          | F <sub>o</sub> 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  
(612) 796-6020

Chain of Custody  
Sample Deposition Sheet

Job N.S.P. SHERCO Source Unit #3  
Team Leader Don Test Site Stack  
Date Submitted 3/27/90 Date of Test 3/27/90  
Test No. 2 No. of Runs Completed 3

| No. of Samples | Type of Sample                                                                                                                                                                                                                                                                                   | Analysis Required                                                                                                                                                                                                                                                                | Comments                       |
|----------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------|
|                | Probe Wash:<br><input type="checkbox"/> Acetone<br><input type="checkbox"/> D.I. Water                                                                                                                                                                                                           | <input type="checkbox"/> As per EPA M-5<br><input type="checkbox"/> Other _____                                                                                                                                                                                                  |                                |
|                | Filter:<br><input type="checkbox"/> 4" G.F.<br><input type="checkbox"/> S.S. Thimble<br><input type="checkbox"/> 2.5" G.F.<br><input type="checkbox"/> 47 mm G.F.                                                                                                                                | <input type="checkbox"/> As per EPA M-5<br><input type="checkbox"/> As per EPA M-17<br><input type="checkbox"/> Other _____                                                                                                                                                      |                                |
| 4              | Impinger Catch:<br><input type="checkbox"/> D.I. Water<br><input type="checkbox"/> 3% H <sub>2</sub> O <sub>2</sub><br><input type="checkbox"/> 4M5 Hg Only<br><input type="checkbox"/> 4M5 Metals<br><input type="checkbox"/> 1.0 N NaOH<br><input checked="" type="checkbox"/> Other <u>CR</u> | <input type="checkbox"/> MN Protocol<br><input type="checkbox"/> WI Protocol<br><input type="checkbox"/> EPA M-6 or 8<br><input type="checkbox"/> Acid Gases<br><input type="checkbox"/> Formaldehyde<br><input type="checkbox"/> Metals<br><input type="checkbox"/> Other _____ |                                |
| 3              | Integrated Gas sample                                                                                                                                                                                                                                                                            | <input checked="" type="checkbox"/> As per EPA M-3<br><input checked="" type="checkbox"/> As per EPA M-10<br><input type="checkbox"/> Other _____                                                                                                                                |                                |
|                | Oxides of Nitrogen (NO <sub>x</sub> )                                                                                                                                                                                                                                                            | <input type="checkbox"/> As per EPA M-7A<br><input type="checkbox"/> Other _____                                                                                                                                                                                                 | Date _____<br>Time (HRS) _____ |
|                | <input type="checkbox"/> Fuel Sample<br><input type="checkbox"/> Aggregate                                                                                                                                                                                                                       | <input type="checkbox"/> Attached fuel Form #S-0163RRR                                                                                                                                                                                                                           |                                |
|                | Particle Size                                                                                                                                                                                                                                                                                    | <input type="checkbox"/> X-Ray Sedigraph<br><input type="checkbox"/> Bahco Method<br><input type="checkbox"/> Other _____                                                                                                                                                        |                                |
|                | Audit Samples<br><input type="checkbox"/> Sulfur Dioxide<br><input type="checkbox"/> Oxides of Nit.<br><input type="checkbox"/> Other _____                                                                                                                                                      | <input type="checkbox"/> As per EPA M-6<br><input type="checkbox"/> As per EPA M-7A<br><input type="checkbox"/> 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 \_\_\_\_\_

EPA Method 3 Data Reporting Sheet  
Orsat Analysis

Job NSP / Sherco

Source Unit #3

Team Leader DVH

Test Site Stack

Date Submitted 3-27-90

Date of Test 3-27-90

Test No. 2

No. of Runs Completed 3

Date of Analysis 3-28-90

Technician RL

| Test/<br>Run | Sample<br>Log Number<br>and Type | No.<br>of<br>An. | Buret Readings (ml) |                       |                      | Conc.<br>CO <sub>2</sub><br>%v/v Dry | Conc.<br>O <sub>2</sub><br>%v/v Dry | F <sub>e</sub> |
|--------------|----------------------------------|------------------|---------------------|-----------------------|----------------------|--------------------------------------|-------------------------------------|----------------|
|              |                                  |                  | Zero Pt.            | After CO <sub>2</sub> | After O <sub>2</sub> |                                      |                                     |                |
| 2-1          | 9242-47<br>B F                   | 1                | 0.00                | 13.60                 | 19.70                | 13.60                                | 6.10                                | 1.09           |
|              |                                  | 2                | 0.00                | 13.60                 | 19.70                | 13.60                                | 6.10                                | 1.09           |
|              |                                  | Avg              |                     |                       |                      | 13.60                                | 6.10                                |                |
| 2-2          | 9242-48<br>B F                   | 1                | 0.00                | 13.60                 | 19.70                | 13.60                                | 6.10                                | 1.09           |
|              |                                  | 2                | 0.00                | 13.60                 | 19.70                | 13.60                                | 6.10                                | 1.09           |
|              |                                  | Avg              |                     |                       |                      | 13.60                                | 6.10                                |                |
| 2-3          | 9242-49<br>B F                   | 1                | 0.00                | 13.60                 | 19.60                | 13.60                                | 6.00                                | 1.10           |
|              |                                  | 2                | 0.00                | 13.60                 | 19.60                | 13.60                                | 6.00                                | 1.10           |
|              |                                  | Avg              |                     |                       |                      | 13.60                                | 6.00                                |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |
|              | B F                              | 1                |                     |                       |                      |                                      |                                     |                |
|              |                                  | 2                |                     |                       |                      |                                      |                                     |                |
|              |                                  | Avg              |                     |                       |                      |                                      |                                     |                |

- ☒ Ambient Air QA Check  
☒ Orsat Analyzer System Leak Check  
☒ F<sub>e</sub> Within EPA M-3 Guidelines  
for fuel type.

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

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

EPA Method 3 Guidelines

| Fuel Type          | F <sub>e</sub> 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          |

**APPENDIX G**

**RESULTS OF TRACE METAL ANALYSIS**

INTERPOLL LABORATORIES, INC.  
(612) 786-6020

NSP/Sherco  
Laboratory Log No. 9242

Results of Trace Metal Analysis

Test: 1  
Source: Unit No. 3 Stack  
Sample Type: 4M5 Train Catch

| Trace Metal | EPA Method              | Total Mass of Trace Metal in Sample (ug) |           |           |           |
|-------------|-------------------------|------------------------------------------|-----------|-----------|-----------|
|             |                         | Field Blank                              | Run 1     | Run 2     | Run 3     |
| (Log No.)   |                         | (9242-39)                                | (9242-40) | (9242-41) | (9242-42) |
| Aluminum    | SW-846, 6010            | 270                                      | 510       | 330       | 360       |
| Arsenic     | SW-846, 7060            | 0.17                                     | 0.10      | < 0.05    | < 0.05    |
| Boron       | SW-846, 6010            | 33                                       | 2420      | 408       | 1690      |
| Barium      | SW-846, 6010            | 8.1                                      | 12.5      | 10.6      | 9.5       |
| Beryllium   | SW-846, 7091            | 0.018                                    | 0.020     | 0.024     | 0.030     |
| Calcium     | SW-846, 6010            | 247                                      | 608       | 413       | 405       |
| Cadmium     | SW-846, 6010            | 237                                      | 27        | 11        | 6.5       |
| Chromium    | SW-846, 6010            | 3.9                                      | 15        | 19        | 7.0       |
| Copper      | SW-846, 6010            | 40                                       | 102       | 50        | 26        |
| Iron        | SW-846, 6010            | 60                                       | 169       | 130       | 68        |
| Potassium   | SW-846, 6010            | 110                                      | 295       | 140       | 137       |
| Magnesium   | SW-846, 6010            | 25                                       | 62        | 40        | 51        |
| Manganese   | SW-846, 6010            | 15                                       | 7.6       | 47        | 5.1       |
| Molybdenum  | SW-846, 6010            | 26                                       | 32        | 28        | 30        |
| Sodium      | SW-846, 6010            | 540                                      | 1030      | 720       | 740       |
| Nickel      | SW-846, 6010            | 29                                       | 11        | 10        | 4.2       |
| Lead        | SW-846, 6010            | 5.2                                      | 19.5      | 8.8       | 5.9       |
| Selenium    | SW-846, 7740            | < 2.5                                    | < 2.5     | < 2.5     | < 2.5     |
| Antimony    | SW-846, 7041            | 0.21                                     | 1.1       | 0.41      | 0.17      |
| Strontium   | 3500-Sr. B <sup>1</sup> | < 5                                      | 6.5       | < 5       | < 5       |
| Vanadium    | SW-846, 6010            | < 0.1                                    | < 0.1     | < 0.1     | < 0.1     |
| Zinc        | SW-846, 6010            | 180                                      | 117       | 41        | 30        |
| Mercury     | SW-846, 7470            | < 0.1                                    | 0.96      | < 0.4     | < 0.2     |
| Silver      | SW-846, 6010            | < 0.3                                    | < 0.3     | < 0.3     | < 0.3     |

<sup>1</sup>Standard Methods, 17th Edition.

INTERPOLL LABORATORIES, INC.  
(612) 786-6020

NSP/Sherco  
Laboratory Log No. 9242

Results of Trace Metal Analysis

Test: 1  
Source: Unit No. 3 Stack  
Sample Type: Permanganate Impinger Catch

| Trace Metal | EPA Method   | Total Mass of Trace Metal in Sample (ug) |           |           |           |
|-------------|--------------|------------------------------------------|-----------|-----------|-----------|
|             |              | Field Blank                              | Run 1     | Run 2     | Run 3     |
| (Log No.)   |              | (9242-23)                                | (9242-27) | (9242-31) | (9242-35) |
| Mercury     | SW-846, 7470 | 0.80                                     | 5.0       | 2.9       | 4.9       |

INTERPOLL LABORATORIES, INC.  
(612)786-6020

NSP/Sherco  
Laboratory Log No. 9242

Results of Trace Metal Analysis

Test: 1  
Source: Unit No. 3 SDA Inlet  
Sample Type: Inlet Train Catch

|             |              | Total Mass of Trace Metal in Sample (ug) |           |           |
|-------------|--------------|------------------------------------------|-----------|-----------|
| Trace Metal | EPA Method   | Run 1                                    | Run 2     | Run 3     |
| (Log No.)   |              | (9242-06)                                | (9242-10) | (9242-14) |
| Aluminum    | SW-846, 6010 | 164000                                   | 299000    | 219000    |
| Arsenic     | SW-846, 6010 | 82                                       | 77        | 98        |
| Boron       | SW-846, 6010 | 9940                                     | 9640      | 12820     |
| Barium      | SW-846, 6010 | 12150                                    | 18440     | 12100     |
| Beryllium   | SW-846, 6010 | 27                                       | 29        | 31        |
| Calcium     | SW-846, 6010 | 392000                                   | 707000    | 465000    |
| Cadmium     | SW-846, 6010 | 10                                       | 10        | 9         |
| Chromium    | SW-846, 6010 | 313                                      | 328       | 370       |
| Copper      | SW-846, 6010 | 695                                      | 650       | 880       |
| Iron        | SW-846, 6010 | 162000                                   | 128000    | 185000    |
| Potassium   | SW-846, 6010 | 60900                                    | 56600     | 68300     |
| Magnesium   | SW-846, 6010 | 23600                                    | 35100     | 35600     |
| Manganese   | SW-846, 6010 | 6420                                     | 6080      | 7360      |
| Molybdenum  | SW-846, 6010 | 109                                      | 88        | 135       |
| Sodium      | SW-846, 6010 | 100000                                   | 130000    | 139000    |
| Nickel      | SW-846, 6010 | 242                                      | 204       | 279       |
| Lead        | SW-846, 6010 | 758                                      | 580       | 770       |
| Selenium    | SW-846, 6010 | 56                                       | 40        | 71        |
| Antimony    | SW-846, 6010 | 99                                       | 68        | 110       |
| Strontium   | SW-846, 6010 | 23400                                    | 13700     | 28900     |
| Vanadium    | SW-846, 6010 | 830                                      | 750       | 980       |
| Zinc        | SW-846, 6010 | 320                                      | 310       | 320       |
| Mercury     | SW-846, 7470 | 0.29                                     | 0.32      | < 0.25    |
| Silver      | SW-846, 6010 | < 3.0                                    | < 3.0     | < 3.0     |

INTERPOLL LABORATORIES, INC.  
(612)786-6020

NSP/Sherco  
Laboratory Log No. 9242

Results of Trace Metal Analysis

Test: 1  
Source: Unit No. 3 SDA Inlet  
Sample Type: Permanganate Impinger Catch

| Trace Metal | EPA Method   | Total Mass of Trace Metal in Sample (ug) |           |           |           |
|-------------|--------------|------------------------------------------|-----------|-----------|-----------|
|             |              | Field Blank                              | Run 1     | Run 2     | Run 3     |
| (Log No.)   |              | (9242-50)                                | (9242-51) | (9242-52) | (9242-53) |
| Mercury     | SW-846, 7470 | 0.22                                     | 9.8       | 9.3       | 6.6       |

## NSP Sherco Unit 3

March 90

| All ICP     |            |    |          |      |       |       |       |
|-------------|------------|----|----------|------|-------|-------|-------|
| Method      | Historical |    | Measured | Used | Run 1 | Run 3 | Run 3 |
| ICP         | 375        | Al | 270      | 270  | 510   | 330   | 360   |
| GF/AA       | <5         | As | 0.17     | .05  | x     | <.05  | <.05  |
| ICP         | 9          | B  | 33       | 9    | 2420  | 408   | 1690  |
| ICP         | 12         | Ba | 8.1      | 8.1  | 12.5  | 10.6  | 9.5   |
| GF/AA       | <.1        | Be | .018     | .018 | .020  | .024  | .030  |
| ICP         | 181        | Ca | 247      | 181  | 608?  | 413   | 405   |
| ICP         | <.4        | Cd | 237      | <.4  | x     | 11?   | 6.5   |
| ICP         | <.7        | Cr | 3.9      | <.7  | 15    | 9     | 7     |
| ICP         | 3.2        | Cu | 40       | 3.2  | 102   | 50    | 26    |
| ICP         | 38         | Fe | 60       | 38   | 169   | 130   | 68    |
| ICP         | 64         | K  | 110      | 64   | 295   | 140   | 137   |
| ICP         | 25         | Mg | 25       | 25   | 62    | 40    | 51    |
| ICP         | <.5        | Mn | 15       | <.5  | 7.6   | 47    | 5.1   |
| ICP         | 33         | Mo | 26       | 33   | 32    | 28    | 30    |
| ICP         | 158        | Na | 540      | 158  | 1030? | 720   | 740   |
| ICP         | <1         | Ni | 29       | <1   | 11?   | 10    | 4.2   |
| ICP         | <4         | Pb | 5.2      | <4   | 19.5  | 8.8   | 5.9   |
| GF/AA       | <5         | Se | <2.5     | <2.5 | <2.5  | <2.5  | <2.5  |
| GF/AA       | <.5        | Sb | .21      | .21  | 1.1   | 0.41  | 0.17  |
| GF/AA       | <5         | Sr | <5       | <5   | 6.5   | <5    | <5    |
| ICP         | <.1        | V  | <.1      | <.1  | <.1   | <.1   | <.1   |
| ICP         | 9.1        | Zn | 180      | 9.1  | x     | 41    | 30    |
| Cold Vapor  | <.4        | Hg | <.1      | <.4  | .96   | <.4   | <.2   |
| ICP         | <.6        | Ag | <.3      | <.3  | <.3   | <.3   | <.3   |
| Cold Vapor  |            | Hg | .80      | .8   | 5.0   | 2.9   | 4.9   |
| Total Train |            | Hg | <.9      | .8   | 5.96  | 2.9   | 4.9   |

x = Invalidated due to field contamination (3)

? = Result may be high due to field contamination (3)

\*Prepared by P. Lonnes

May 1990

Total analyses = 72

**APPENDIX H**

**RESULTS OF LIME ANALYSIS**

INTERPOLL LABORATORIES, INC.  
(612) 786-6020

NSP/Sherco  
Laboratory Log No. 9242

Results of Trace Metals Analysis

Source: Unit No. 3

Sample Type: Lime

| Concentration of Trace Metal |              |                  |
|------------------------------|--------------|------------------|
| Metal                        | EPA Method   | in Sample (ug/g) |
| (Log No.)                    |              | (9242-73)        |
| Arsenic                      | SW-846, 6010 | < 5.0            |
| Beryllium                    | SW-846, 6010 | < 0.1            |
| Cadmium                      | SW-846, 6010 | < 0.2            |
| Chromium                     | SW-846, 6010 | 15.3             |
| Nickel                       | SW-846, 6010 | 3.2              |
| Lead                         | SW-846, 6010 | < 2.5            |
| Selenium                     | SW-846, 6010 | < 5.0            |
| Mercury                      | SW-846, 7470 | 0.07             |

**APPENDIX I**

**RESULTS OF TRACE METALS ANALYSES ON COAL SAMPLE**

INTERPOLL LABORATORIES, INC.  
(612)786-6020

NSP/Sherco  
Laboratory Log No. 9242

Results of Trace Metals Analysis

Source: Unit 3  
Sample Type: Coal

| Concentration of Trace Metal |                     |                  |
|------------------------------|---------------------|------------------|
| Metal                        | Method <sup>1</sup> | in Sample (ug/g) |
| (Log No.)                    |                     | (9242-74)        |
| Arsenic                      | SW-846, 7060        | 0.70             |
| Beryllium                    | SW-846, 7091        | 0.32             |
| Cadmium                      | SW-846, 7131        | 0.06             |
| Chromium                     | SW-846, 6010        | 4.4              |
| Nickel                       | SW-846, 6010        | 2.3              |
| Lead                         | SW-846, 6010        | 9.1              |
| Selenium                     | SW-846, 7740        | < 1.6            |
| Mercury                      | ASTM D3684          | < 0.02           |

<sup>1</sup>Sample prepped by ASTM Method D3683.

**APPENDIX J**

**PROCEDURES**

**Trace Metals (4M5) Procedures**

11/7/89

## 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<sup>R</sup> 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.

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APR 14 1989

INTERPOL LABORATORY

1

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and 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 SW-846, Method 6010) are approximately as follows: Sb (32 ng/ml), As (53 ng/ml), Ba (2 ng/ml), Be (0.3 ng/ml), Cd (4 ng/ml), Cr (7 ng/ml), Cu (6 ng/ml), Pb (42 ng/ml), Mn (2 ng/ml), Ni (15 ng/ml), P (75 ng/ml), Se (75 ng/ml), Ag (7 ng/ml), 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<sup>3</sup>, 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<sup>3</sup>.  
 D = in-stack detection limit, ug/m<sup>3</sup>.

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:

- o A normal 1-hour sampling run collects a stack gas sampling volume of about 1.25 m<sup>3</sup>. If the sampling time is increased and 5 m<sup>3</sup> 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).
- o 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

TABLE A-1. IN-STOCK METHOD DETECTION LIMITS ( $\mu\text{g}/\text{m}^3$ )  
FOR TRAIN FRACTIONS USING ICAP AND AAS

| Metal      | Front Half<br>Fraction 1<br>Probe and Filter | Back Half <sub>1</sub><br>Fraction 2<br>Impingers 1-3 | Back Half <sub>2</sub><br>Fraction 3<br>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.

- o 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).
- o Conversely, reducing stack gas sample volume and increasing sample liquid volume will increase limits. The front half and back half<sub>1</sub> 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<sub>1</sub>) of where in the train

4

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.

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.

- o 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 similar 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.

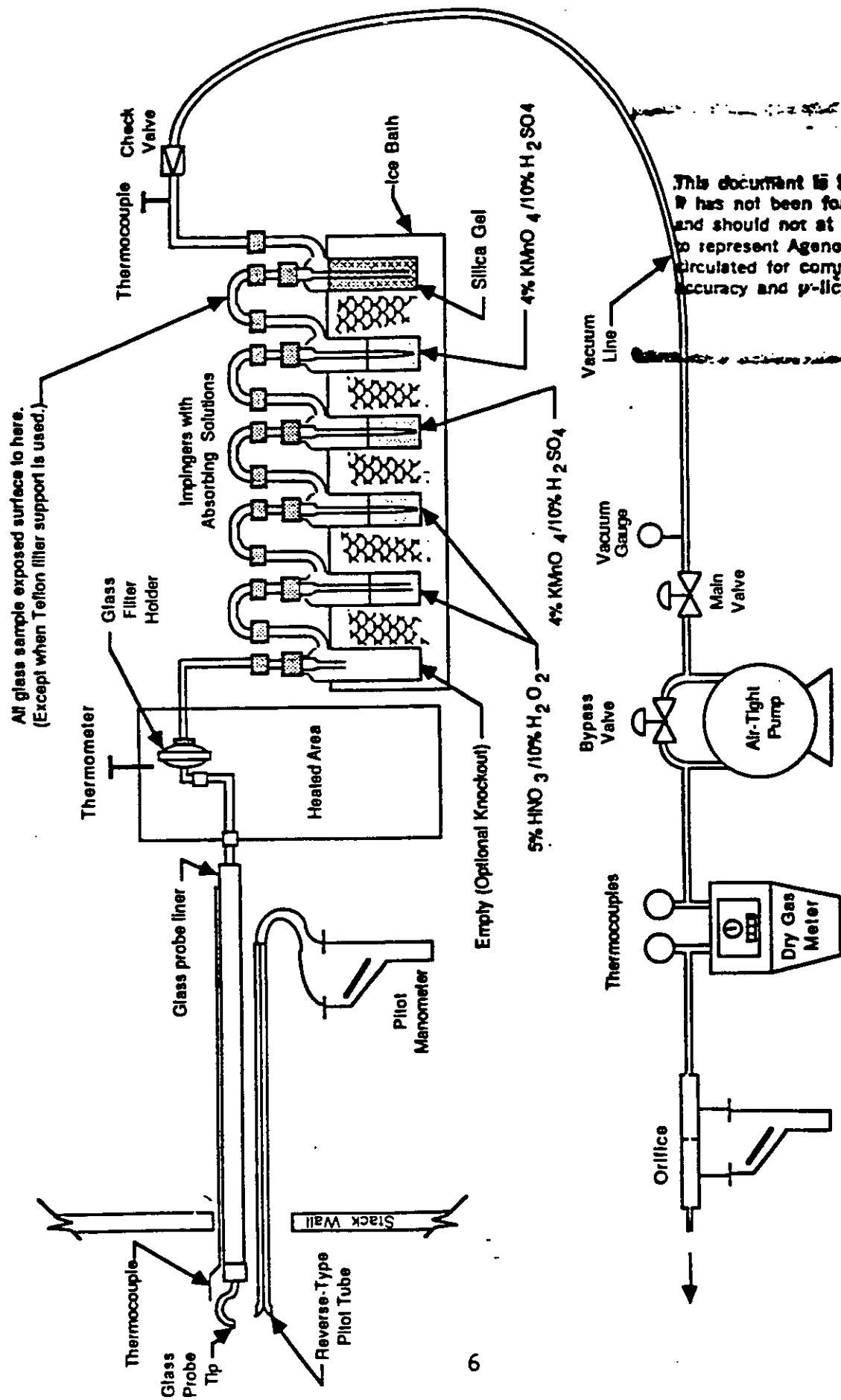


Figure A-1. Schematic of multiple metals sampling train.

Interpoll Laboratories, Inc.  
(612)786-6020

**Determination of Hexavalent Chromium  
in Sampling Train Samples**

Interpoll Method #: 6010-MM5  
EPA Reference Method #: SW-846, 6010

Version 1.0  
June 20, 1990

# 1 APPLICATION

This method is applicable to the determination of hexavalent chromium in stationary source samples collected by a sampling train.

Method No. 6010-MMS  
Revision No. 0  
Date June 20, 1990

## 2 RANGE

The range of this method extends from a detection limit of 0.05 ug to 50 ug. The upper range can be greatly extended through dilutions.

Method No. 6010-MM5  
Revision No. 0  
Date June 20, 1990

2-1

### 3 SAMPLE HANDLING AND PRESERVATION

3.1 Samples should be analyzed as soon as possible.

3.2 To retard the chemical activity of hexavalent chromium, the samples should be transported and stored until the time of analysis at 4°C.

Method No. 6010-MM5  
Revision No. 0  
Date June 20, 1990

#### 4 SUMMARY OF METHOD

The probe wash and filter of the sampling train are combined, digested, and filtered. The filtrate is combined with the impinger catch, the pH of this solution is adjusted to 2.4, and chelated using APDC. Adjust the solution pH again to 2.8 and extract with MIBK. This isolates the Cr(VI) from the other Cr species. The MIBK is evaporated and the residue taken up in nitric acid. The solution is then analyzed by ICP or GF/AA.

Method No. 6010-MM5  
Revision No. 0  
Date June 20, 1990

## 5 INTERFERENCES

Cr(VI) reacts with most organic compounds and surfaces.

Method No. 6010-MM5

Revision No. 0

Date June 20, 1990

5-1

## 6 APPARATUS AND GLASSWARE

### 6.1 For GF/AA analysis:

6.1.1 Chromium hollow cathode lamp.

6.1.2 Wavelength: 357.9 nm.

#### 6.1.3 Furnace Program:

|                 |         |
|-----------------|---------|
| Drying time     | 30 sec. |
| Drying temp.    | 125°C   |
| Ashing time     | 30 sec. |
| Ashing temp.    | 1000°C  |
| Atomizing time  | 10 sec. |
| Atomizing temp. | 2700°C  |

### 6.2 For ICP analysis:

6.2.1 Thermo Jarrell Ash ICAP 61 Plasma Spectrometer.

6.2.2 Optical system: 0.75 meter Paschen-Runge mount with a 25 micron entrance slit and 50 micron exit slits.

6.2.3 Detectors: Hamamatsu Side Window 1/2 inch (1.3 cm) diameter; R300, R427, R306, R889.

6.2.4 Computer: IBM PC/AT 640K RAM, 30 MByte Winchester hard drive, 1.44 MByte floppy drive, enhanced color display.

### 6.3 Hot plate.

Method No. 6010-MM5

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6.4 Volumetric flasks: 100 mL, 250 mL, and 1000 mL, for preparation of standards and sample dilution.

6.5 Graduated cylinders: For preparation of reagents.

6.6 Microwave digester: CEM MDS-81D, Teflon PFA vessels (120 mL size) with pressure relief valve, digestion turntable, capping station.

6.7 Beakers and watch glasses: 250 mL beakers for sample digestion with watchglasses to cover the tops.

6.8 Ring stands and clamps: For securing equipment.

6.9 Filter funnels.

6.10 Glass fiber filters.

6.11 Disposable pasteur pipets and bulbs.

6.12 Volumetric pipets.

6.13 Separatory funnels.

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## 7 REAGENTS

7.1 Ammonium pyrrolidine dithiocarbamate (APDC) solution. Dissolve 1.0 gram APDC in demineralized water and dilute to 100 mL. Prepare fresh daily.

7.2 Potassium dichromate stock solution: Dissolve 141.4 mg of dried potassium dichromate,  $K_2Cr_2O_7$  (analytical reagent grade), in Type II water and dilute to 1 liter (1 mL = 50 ug Cr).

7.3 Potassium dichromate standard solution: Dilute 10.00 mL potassium dichromate stock solution to 100 mL (1 mL = 5 ug Cr).

7.4 Sulfuric acid, 10 % (v/v): Dilute 10 mL of distilled reagent grade sulfuric acid,  $H_2SO_4$ , to 100 mL with Type I water.

7.5 Methyl isobutyl ketone (MIBK)

7.6 Sulfuric acid, 0.12M: Slowly add 6.5 mL concentrated  $H_2SO_4$  to demineralized water and dilute to 1 liter.

7.7 Nitric acid, concentrated

7.8 Sodium Hydroxide solution, 1 M: Dissolve 40 grams NaOH in demineralized water and dilute to 1 liter.

7.9 Digestion Solution: Mix 20 grams NaOH and 30 grams  $Na_2CO_3$  and dilute to 1 liter with demineralized water.

Method No. 6010-MM5

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## 8 SAFETY

Normal, accepted laboratory safety practices should be followed. No known carcinogenic materials are used in this method.

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8-1

## 9 PROCEDURE

9.1 Combine the probe rinse and the filter from the sample train in a beaker.

9.2 Add 40 mL of the digestion solution.

9.3 Heat beaker on hot plate for 30 minutes. The solution should not be taken to dryness.

9.4 After cooling, the mixture is filtered using a distilled-water prewashed glass fiber filter using vacuum filtration.

9.5 Combine the filtrate (which contains the CrVI) with the impinger catch and adjust the pH to 2.4 using  $\text{H}_2\text{SO}_4$ .

9.6 Add 5 mL of APDC per 100 mL of solution.

9.7 Transfer to separatory funnel and extract with 10 mL of MIBK per 100 mL of solution. Shake vigorously for 5 minutes. Allow layers to separate (10 - 15 minutes). Remove MIBK layer into a flask. Repeat the above two more times and put MIBK into the same flask.

9.8 Evaporate the MIBK on a hot plate almost to dryness.

9.9 Add 25 mL of 5%  $\text{HNO}_3$  and heat for 45 minutes.

9.10 Transfer to a 25 mL volumetric flask and bring to volume with 5%  $\text{HNO}_3$ .

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9.11 Analyze by ICP. (If samples are below the ICP detection limit, reanalyze by GF/AA with Zeeman background correction if required).

9.12 Sample Analysis: The prepared sample is analyzed by ICP for Hexavalent Chromium. The wavelength is 267.716 nm.

9.12.1 Instrument Set-up: The instrument is set-up and profiled as described in the ICP Standard Operating Procedure (Interpoll Method Number ICP-IL1).

9.12.2 Instrumental Calibration: Following set-up, the instrument is calibrated using a blank and a solution containing 100 mg/L of chromium.

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## 10 CALCULATION

To calculate results in total ug, use the following equation:

$$\text{ug/mL Cr} \times (\text{Dilution Factor}) \times \text{Volume of Sample (mL)} = \text{total ug}$$

where:

ug/mL Cr = read off ICP printout

Volume of Sample = final volume of sample after preparation  
(Liters)

Dilution Factor = any dilutions required to bring the  
concentration of Cr within the calibration  
range or to remove interferences

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## 11 QUALITY CONTROL PROCEDURES

11.1 To verify the absence of matrix interferences, at least one matrix spike must be performed. If not enough sample was submitted, an instrument spike must be run. Percent recovery must meet control limits for the method.

11.2 One spike must be performed in order to determine extraction efficiency.

11.3 One duplicate must be run per batch. The Percent Relative Difference must meet the control limits for the method. If there isn't enough sample, an instrument duplicate will be run.

11.4 A method blank must be run to verify that the method is performing to specifications.

11.5 Any sample above the linear range of the instrument must be diluted and reanalyzed.

11.6 Two calibration blanks are analyzed with each analytical batch. One is analyzed at the beginning of the run and the other at the end.

11.7 Two instrument check standards should be analyzed during an analytical run. One at the beginning and another at the end. These standards verify the accuracy of the instrument calibration.

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## 12 ACCURACY AND PRECISION

12.1 The accuracy limits for this method are from 80 to 120 percent recovery.

12.2 The precision limit for this method is 10 relative percent difference.

12.3 Check standards must be within  $\pm 10\%$  of their true value or the data associated with them is considered invalid.

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### 13 REFERENCES

13.1 EPA Method 6010; Test Methods for Evaluating Solid Waste (SW-846); U.S. EPA Office of Solid Waste and Emergency Response; November 1986.

13.2 EPA Method 7191; Test Methods for Evaluating Solid Waste (SW-846); U.S. EPA Office of Solid Waste and Emergency Response; November 1986.

13.3 EPA Method 7197; Test Methods for Evaluating Solid Waste (SW-846); U.S. EPA Office of Solid Waste and Emergency Response; November 1986.

13.4 Draft Method "Methodology for the Determination of Metals Emissions in Exhaust Gases from Stationary Sources of Combustion Processes"; received April, 1989 from Source Methods Standardization Branch, US EPA Atmospheric Research and Exposure Assessment Laboratory; Research Triangle Park, NC 27711.

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Revision No. 0  
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APPENDIX K

CALCULATION EQUATIONS

## CALCULATION EQUATIONS

### METHOD 2

$$\bar{V}_s = 95.48 C_p (\sqrt{\Delta p})_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}}$$

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

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

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

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

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

$$= \frac{4.595 \times 10^{-2} P_s M_s}{T_s (avg)}$$

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

## 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

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

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

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

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

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

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

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

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

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

### SYMBOLS

|                  |                                                                                                          |
|------------------|----------------------------------------------------------------------------------------------------------|
| A                | = Cross sectional area of stack, SQ. FT.                                                                 |
| A <sub>n</sub>   | = Cross sectional area of nozzle, SQ. FT.                                                                |
| B <sub>ws</sub>  | = Water vapor in gas stream, proportion by volume                                                        |
| C <sub>p</sub>   | = Pitot tube coefficient, dimensionless                                                                  |
| C <sub>a</sub>   | = Concentration of particulate matter in stack gas, wet basis, GR/ACF                                    |
| C <sub>s</sub>   | = 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 <sub>d</sub>   | = Specific gravity (relative to air), dimensionless                                                      |
| I                | = Isokinetic variation, percent by volume                                                                |
| M <sub>d</sub>   | = 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 <sub>s</sub>   | = Molecular weight of stack gas, wet basis, g/g, mole.                                                   |
| M <sub>p</sub>   | = Total amount of particulate matter collected, g                                                        |
| P <sub>bar</sub> | = Atmospheric pressure, IN. HG. (uncompensated)                                                          |
| P <sub>g</sub>   | = 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)  
 $\theta$  = 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  
 $v_{p_{tdb}}$  = Vapor pressure at  $T_{db}$ , IN. HG.

- $v_{p_{twb}}$  = Vapor pressure at  $T_{wb}$ , IN. HG
- $\overline{\Delta H}$  = Average pressure differential across the orifice meter, IN. WC.
- $\Delta P$  = Velocity pressure of stack gas, IN. WC.
- $\gamma$  = Dry test meter correction coefficient, dimensionless
- $\rho$  = Actual gas density, LB/ACF

Job NSP Sherco

Source UNIT 3 STACK

Date of Test 8-27-78

| Element    | GF/AS | H/D   | MICROGRAMS   |      |       | Analytical Detection Limit | Minimum Detectable Mass | Best Estimate of Field Blank | Total Mass in Sample (ug) |       |       | Total Mass in Sample (ug) |       |  |
|------------|-------|-------|--------------|------|-------|----------------------------|-------------------------|------------------------------|---------------------------|-------|-------|---------------------------|-------|--|
|            |       |       | Field Blanks |      | Run 1 |                            |                         |                              | Run 2                     | Run 3 | Run 1 | Run 2                     | Run 3 |  |
|            |       |       | 4            | 1 2  |       |                            |                         |                              |                           |       |       |                           |       |  |
| Aluminum   |       | X     | 375          | 270  | 2.5   | ≤ 24                       | 270                     | 570                          | 330                       | 360   | 240   | 60                        | 70    |  |
| Arsenic    | X     |       | 45           | 0.17 | .05   | ≤ .1                       | .05                     | .10                          | ≤ .05                     | ≤ .05 | .10   | ≤ .1                      | ≤ .1  |  |
| Baron      | X     |       | 9            | 33   | .6    | ≤ 4                        | 9                       | 2420                         | 408                       | 1690  | 2411  | 379                       | 1631  |  |
| Barium     | X     |       | 12           | 81   | .1    | ≤ 6                        | 81                      | 12.5                         | 10.6                      | 9.5   | ≤ 6   | ≤ 6                       | ≤ 6   |  |
| Beryllium  | X     |       | ≤ 1          | 0.18 | .004  | ≤ .02                      | .018                    | .020                         | .024                      | .030  | .002  | .006                      | .012  |  |
| Calcium    | X     |       | 181          | 247  | 1     | ≤ 44                       | 181                     | 608.3                        | 413                       | 405   | 457   | 232                       | 224   |  |
| Cadmium    | X     |       | ≤ 4          | 237  | .4    | ≤ 1                        | .4                      | 27                           | 11                        | 6.5   | 26.6  | 10.6                      | 6.1   |  |
| Chromium   | X     |       | ≤ 7          | 39   | .7    | ≤ 1.4                      | .7                      | 15                           | 9                         | 7     | 14.3  | 2.3                       | 6.3   |  |
| Copper     | X     |       | 32           | 40   | 5     | ≤ 1.2                      | 32                      | 102                          | 50                        | 26    | 98.8  | 46.3                      | 228   |  |
| Iron       | X     |       | 38           | 60   | .5    | ≤ 24                       | 38                      | 169                          | 130                       | 68    | 131   | 72                        | 30    |  |
| Potassium  | X     |       | 64           | 110  | 40    | ≤ 12                       | 64                      | 295                          | 140                       | 137   | 231   | 74                        | 75    |  |
| Magnesium  | X     |       | 85           | 25   | 1     | ≤ 1.2                      | 25                      | 62                           | 40                        | 51    | 37    | 15                        | 26    |  |
| Manganese  | X     |       | ≤ 5          | 15   | .5    | ≤ 1                        | .5                      | 7.6                          | 47                        | 5.1   | 7.1   | 46.5                      | 46    |  |
| Molybdenum | X     |       | 33           | 26   | .5    | ≤ 4                        | 33                      | 32                           | 28                        | 30    | ≤ 4   | ≤ 4                       | ≤ 4   |  |
| Sodium     | X     |       | 153          | 540  | 1     | ≤ 32                       | 153                     | 1030.3                       | 720                       | 740   | 870   | 510                       | 522   |  |
| Nickel     | X     |       | ≤ 1          | 29   | 1     | ≤ 2                        | 1                       | 11.2                         | 10                        | 4.2   | 10    | 7                         | 5.2   |  |
| Lead       | X     |       | ≤ 4          | 5.2  | 2.5   | ≤ 4                        | 4                       | 19.5                         | 8.8                       | 5.9   | 15.5  | 4.8                       | ≤ 4   |  |
| Selenium   | X     |       | ≤ 5          | 2.5  | .05   | ≤ 2.5                      | 2.5                     | ≤ 2.5                        | ≤ 2.5                     | ≤ 2.5 | ≤ 2.5 | ≤ 2.5                     | ≤ 2.5 |  |
| Antimony   | X     |       | ≤ 5          | 2.1  | .5    | ≤ .2                       | .21                     | 1.1                          | 0.41                      | 0.17  | .89   | .2                        | ≤ .2  |  |
| Strontium  | X     |       | ≤ 5          | ≤ 3  | .1    | ≤ 5                        | 5                       | 6.5                          | ≤ 5                       | ≤ 5   | 1.5   | ≤ 5                       | ≤ 5   |  |
| Vanadium   | X     |       | ≤ 1          | ≤ 1  | .1    | ≤ .1                       | .1                      | ≤ .1                         | ≤ .1                      | ≤ .1  | ≤ .1  | ≤ .1                      | ≤ .1  |  |
| Zinc       | X     |       | 9.1          | 180  | .4    | ≤ 5                        | 9.1                     | 117                          | 41                        | 30    | 107.7 | 31.7                      | 30.9  |  |
| Mercury    | Front | CY/AA | ≤ 4          | ≤ 1  | .1    | ≤ 1                        | .4                      | .96                          | ≤ .4                      | ≤ .2  |       |                           |       |  |
|            | KMnO4 |       | ≤ 4          | .80  | .1    | ≤ 1                        | .8                      | 5.0                          | 2.9                       | 4.9   |       |                           |       |  |
|            | Total |       | ≤ 8          | ≤ 9  | .1    | ≤ .2                       | .8                      | 5.96                         | 2.9                       | 4.9   | 5.16  | 2.1                       | 4.1   |  |
| Silver     | X     |       | ≤ 6          | ≤ 3  | .3    | ≤ .3                       | .3                      | ≤ .3                         | ≤ .3                      | ≤ .3  | ≤ .3  | ≤ .3                      | ≤ .3  |  |

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

X = ... Gold contamination