February 2007

Environmental Technology Verification Report

THERMO ELECTRON
MERCURY FREEDOM SYSTEM

Prepared by
Battelle

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ETV ETV ETV

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ETV Advanced Monitoring Systems Center

THERMO ELECTRON
MERCURY FREEDOM SYSTEM

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Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's air, water, and land resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and science support that can be used to solve environmental problems and to build the scientific knowledge base needed to manage our ecological resources wisely, to understand how pollutants affect our health, and to prevent or reduce environmental risks.

The Environmental Technology Verification (ETV) Program has been established by the EPA to verify the performance characteristics of innovative environmental technology across all media and to report this objective information to permitters, buyers, and users of the technology, thus substantially accelerating the entrance of new environmental technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of six environmental technology centers. Information about each of these centers can be found on the Internet at http://www.epa.gov/etv/.

Effective verifications of monitoring technologies are needed to assess environmental quality and to supply cost and performance data to select the most appropriate technology for that assessment. Under a cooperative agreement, Battelle has received EPA funding to plan, coordinate, and conduct such verification tests for "Advanced Monitoring Systems for Air, Water, and Soil" and report the results to the community at large. Information concerning this specific environmental technology area can be found on the Internet at http://www.epa.gov/etv/centers/center1.html.

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List of Abbreviations

AC	alternating current
agl	above ground level
AMS	Advanced Monitoring Systems
ASTM	American Society for Testing and Materials
°C	degrees Celsius
CEM	continuous emission monitor
CFR	Code of Federal Regulations
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
°F	degrees Fahrenheit
FGD	flue gas desulfurization
H_2O_2	hydrogen peroxide
H_2SO_4	sulfuric acid
Hg	mercury
$HgCl_2$	mercuric chloride
Hg^0	elemental mercury
Hg_{OX}	oxidized mercury
Hg_T	total mercury
HNO_3	nitric acid
ICCI	Illinois Clean Coal Institute
KCl	potassium chloride
klb/hr	thousands of pounds per hour
$KMnO_4$	potassium permanganate
LE	linearity error
L/min	liter per minute
µg/dscm	microgram per dry standard cubic meter
µg/L	microgram per liter
MFS	Mercury Freedom System
mL	milliliter
MW	megawatt
NIST	National Institute of Standards and Technology
NO_X	nitrogen oxides
OH	Ontario Hydro
ppm	parts per million
PE	performance evaluation
QA	quality assurance

QC	quality control
QMP	Quality Management Plan
RA	relative accuracy
%RD	percent relative deviation
SO_2	sulfur dioxide
TSA	technical systems audit
V	volt

Chapter 1
Background

The U.S. Environmental Protection Agency (EPA) supports the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized testing organizations; with stakeholder groups consisting of buyers, vendor organizations, and permitters; and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The EPA's National Exposure Research Laboratory and its verification organization partner, Battelle, operate the Advanced Monitoring Systems (AMS) Center under ETV. The AMS Center recently evaluated the performance of the Thermo Electron Mercury Freedom System (MFS), a continuous emissions monitor (CEM) for determining mercury in stack gas at a coal-fired power plant. This evaluation was carried out in collaboration with the Illinois Clean Coal Institute and with the assistance of the Northern Indiana Public Service Company.

Chapter 2
Technology Description

The objective of the ETV AMS Center is to verify the performance characteristics of environmental monitoring technologies for air, water, and soil. This verification report provides results for the verification testing of the MFS. Following is a description of the MFS, based on information provided by the vendor. The information provided below was not verified in this test.

Designed to meet the provisions of Chapter 40 of the Code of Federal Regulations (CFR) Parts 60 and 75 (40 CFR Parts 60 and 75), the MFS (Figure 2-1) can determine elemental (Hg^0), oxidized (Hg_{OX}), and total mercury (Hg_T) in exhaust stacks of coal-fired boilers. The system uses a direct measurement atomic fluorescence method that precludes the use of argon tanks and gold amalgamation. The system extracts a small sample flow from the flue gas stream and immediately dilutes it inside the probe. Any Hg_{OX} in the diluted sample is then converted to Hg^0 in a dry heated converter to obtain an Hg_T measurement. This diluted, converted sample is continuously transported to the mercury analyzer in the MFS rack where it is analyzed using atomic fluorescence technology developed specifically for measuring mercury vapor concentrations on a continuous, real-time basis. In this test, the continuous readings of the MFS were averaged and reported at one-minute intervals. The MFS determined only Hg_T for the purposes of this test.

Figure 2-1. Mercury Freedom System

The MFS consists of a sampling probe with an integrated converter, heated umbilical line, probe controller, saturated Hg^0 vapor calibrator, and an atomic fluorescence analyzer. The MFS can be audited by introduction of mercury calibration gas standards, which can be delivered directly to the probe inlet by the MFS umbilical. In its rack configuration, the system is 70 inches high by 36 inches deep by 24 inches wide. The probe box measures 34.5 inches long by 18.5 inches high by 10.5 inches wide and weighs 90 pounds. Onboard data storage capacity is

4 megabytes. Recording to a data acquisition system can be accomplished using analog output signals, digital (RS232/485), or modbus (via an industry standard Ethernet port). The list price of the system, as tested, excluding installation, training, and umbilical line, was $124,790.

Chapter 3
Test Design and Procedures

3.1 Introduction

This verification test was conducted according to procedures specified in the *Test/QA Plan for Verification of Continuous Emission Monitors (CEMs) and Sorbent-Based Samplers for Mercury at a Coal-Fired Power Plant*.(1) CEMs for mercury are designed to determine total and/or chemically speciated vapor phase mercury in combustion source emissions. Performance requirements for mercury CEMs are contained in 40 CFR Parts 60 and 75(2) and require assessment of the performance of newly installed mercury CEMs only for their determination of Hg_T. This total is the sum of vapor-phase mercury in all chemical forms in the combustion gas, including Hg^0 and Hg_{OX} (which is primarily mercuric chloride [$HgCl_2$]) vapors. In this test the MFS was verified for its measurement of Hg_T.

The MFS was verified by evaluating the following parameters:

- Relative accuracy (RA)
- Linearity
- Seven-day calibration error
- Cycle time
- Data completeness
- Operational factors such as ease of use, maintenance and data output needs, power and other consumables use, reliability, and operational costs.

Verification of the MFS was conducted in a field test that lasted from June 12 to July 25, 2006, and that included two separate four-day periods of reference mercury measurements carried out by ARCADIS Inc., under subcontract to Battelle, using American Society for Testing and Materials (ASTM) D 6784-02, the “Ontario Hydro” (OH) method.(3) RA was determined by comparing the MFS Hg_T results to simultaneous results from the OH method. Linearity was determined based on MFS responses to Hg^0 standards. Calibration error was evaluated by comparing MFS readings on mercury standard and zero gases performed once each day over a consecutive seven-day period. Cycle time was evaluated in terms of the response of the MFS when switching from a zero gas or upscale Hg^0 standard gas, supplied at the MFS probe inlet, to sampling of stack gas. Data completeness was assessed as the percentage of maximum data return achieved by the MFS over the test period. Operational factors were evaluated by means of observations during testing and records of needed maintenance, vendor activities, and expendables use.

3.2 Test Facility

The host facility for the MFS verification was the R. M. Schahfer Generating Station, located near Wheatfield, Indiana, approximately 20 miles south of Valparaiso, Indiana. The Schahfer plant consists of four units (designated 14, 15, 17, and 18), with a total rated capacity of about 1,800 megawatts (MW). The MFS verification was conducted at Unit 17, which burns pulverized Illinois sub-bituminous coal and has an electrostatic precipitator and a wet flue gas desulfurization (FGD) unit. Unit 17 has a typical capacity of about 380 MW. The unit was operated near this capacity for most of the test period, although the typical daily pattern of operation was to reduce load substantially for a few hours between late evening and early morning.

Flue gas from Unit 17 feeds into a free-standing concrete chimney with an internal liner. The top of the stack is 499 feet above ground level (agl). Emission test ports are located at a platform approximately 8 feet wide that encircles the outside of the stack at 370 feet agl. The stack diameter at the platform level is 22 feet 6 inches, so the total flow area is 397.6 square feet. The last flow disturbance is at the FGD connection to the stack liner at 128 feet agl. Thus, the emission test ports were over 10 stack diameters downstream from the last flow disturbance and nearly six diameters upstream from the stack exit. Four emission test ports were located at 90º intervals around the circumference of the stack about 4 feet above the platform at 370 feet agl and were standard 4-inch ports with #125 flanges. No traversing was done during sampling; both the OH method and the MFS CEM sampled from a single fixed point inside the inner liner of the stack at their respective port locations. This arrangement is justified by the absence of stratification observed for sulfur dioxide (SO_2) and nitrogen oxides (NO_X) at this sampling location.

Table 3-1 summarizes key operating and stack gas conditions that characterize Schahfer Unit 17 during the field period, showing the range and average values of key parameters and constituents. Stack gas pressure was slightly positive at the sampling location.

3.3 Test Procedures

Following are the test procedures used to evaluate the MFS CEM.

3.3.1 Relative Accuracy

The RA of the MFS was evaluated by comparing its Hg_T results to simultaneous results obtained by sampling stack gas with the OH reference method. The OH method is the currently accepted reference method for mercury measurements in stack gas and employs dual impinger trains sampling in parallel through a common probe to determine oxidized and elemental vapor-phase mercury by means of appropriate chemical reagents.(3) In each of two separate weeks of the field test period, ARCADIS conducted a series of 12 OH runs, each two hours in duration, as described in Sections 3.5 and 4.1. The MFS was in operation at the Unit 17 test site for the second of those OH sampling periods. The Hg_T concentrations in stack gas determined by the OH reference method were compared to corresponding results from the MFS, by averaging the successive MFS readings over the period of each OH run. A Thermo Electron vendor representative operated the MFS during the OH sampling; however, Schahfer facility staff operated the MFS during other periods.

Table 3-1. Operating and Stack Gas Conditions at Schahfer Station Unit 17

Parameter	Average	Range
Unit 17 Load[a]	334 MW	140–391
Coal Feed Rate[a]	297 klb/hr[c]	140–374
Temperature[a]	130°F	118–140
Moisture[b]	15.5%	13.3–16.7
NO_x[a]	97 ppm[d]	61–165
SO_2[a]	193 ppm	104–316
Hg_T vapor[b]	0.91 µg/dscm[e]	0.73–1.22

a: Values calculated from hourly data recorded routinely at the R.M. Schahfer facility, June 12 to July 25, 2006.
b: Values based on measurements made during OH reference sampling periods June 12–15 and July 10–13, 2006.
c: klb/hr = thousands of pounds per hour.
d: ppm = parts per million.
e: µg/dscm = micrograms per dry standard cubic meter.

The OH trains were dismantled for sample recovery in the field by ARCADIS staff, and all collected sample fractions were logged and stored for transfer to the ARCADIS analytical laboratory. All sample handling, quality assurance/quality control (QA/QC) activities, and mercury analyses were conducted by ARCADIS. Subsequent to mercury analysis, ARCADIS reviewed the data and reported final mercury results from all trains in units of µg/dscm. The results from the paired OH trains were checked relative to the duplicate precision requirement for use of the OH data,(3,4) and qualified OH results were averaged to produce the final reference data used for comparison to the MFS results. RA was calculated as described in Section 5.1 for Hg_T based on the OH reference data, and the average of the MFS results for Hg_T was compared to the corresponding average from the OH reference method.

3.3.2 Linearity

Linearity was evaluated by challenging the MFS with three concentrations of Hg^0 standard gases using a calibration source built into the MFS. These standards were supplied to the MFS in non-repetitive triplicate through the MFS's inlet filter at a rate that exceeded the MFS's inlet flow rate. Each challenge was maintained long enough to achieve a stable response before moving to the next challenge gas. The triplicate responses of the MFS at each challenge concentration were averaged, and the average values were then compared to the known mercury level of the standards. The three challenge concentrations were 3.0, 6.0, and 9.0 µg/dscm.

3.3.3 Seven-Day Calibration Error

At programmed 24-hour intervals over the period of July 17 to July 23, the MFS was challenged with zero gas and an Hg^0 standard concentration of 10 µg/dscm, using the MFS calibration source. These challenge gases were supplied through the MFS's inlet filter at a rate that exceeded the MFS's inlet flow rate. Each such challenge was maintained long enough to achieve a stable response. Deviation of the MFS zero and calibration readings from the expected zero or calibration value was assessed to determine calibration error in the readings.

3.3.4 Cycle Time

Cycle time was determined by monitoring the MFS readings while switching from sampling zero gas to sampling stack gas, and from sampling an Hg^0 standard to sampling stack gas. The former procedure determined the upscale response (or rise) time, and the latter the downscale response (or fall) time. In each case, the response time was determined as the time needed to achieve 95% of the change from one stable reading to the next.

3.3.5 Data Completeness

No additional test procedures were carried out specifically to address data completeness. This parameter was assessed based on the overall data return relative to the total amount of data return possible for the technology being tested.

3.3.6 Operational Factors

Operational factors such as maintenance needs, data output, consumables use, and ease of use were evaluated based on observations by Battelle and Schahfer facility staff. A laboratory record book was maintained at the host facility and was used to enter daily observations on these factors. Examples of information recorded in the record books are the daily status of diagnostic indicators for the MFS, use or replacement of any consumables, the effort or cost associated with maintenance or repair, vendor effort (e.g., time on site) for repair or maintenance, the duration and causes of any down time or data acquisition failure, and operator observations about ease of use of the MFS.

3.4 CEM Installation

The MFS rack system was installed in an air-conditioned laboratory trailer placed at the base of the Unit 17 stack. The rack components drew electrical power from two 120V/15A circuits inside the trailer. Compressed air (110 pounds per square inch gauge) was supplied from a compressor located near the trailer to a wall-mountable air purification panel provided by Thermo Electron and located inside the trailer. The rack system was connected to the sampling probe on the stack by a heated umbilical over 450 feet in length. Three 120V/30A circuits provided power for the sampling probe, the mercury converter in the probe assembly, and the heating of the umbilical line, respectively. Installation of the MFS was conducted by one Thermo Electron field engineer, who trained Battelle and Schahfer facility staff in routine operation of the MFS. That field engineer also operated the MFS during the period of OH reference method sampling.

The MFS umbilical and needed utility supplies were in place by June 7, 2006, and the MFS rack system arrived at the Schahfer field site on June 19. The MFS was first connected to the stack on June 21. The slightly recessed position of the flange on the sampling port was found to prevent the opening of doors on the CEM's sampling probe, so a port extension was installed that allowed the doors to clear the port opening on the side of the stack. The use of this extension caused the sampling point for the MFS probe to be 2.45 feet from the inner wall of the stack, rather than 3.28 feet (1 meter) as prescribed in the test/QA plan.(1) Thus, the MFS sampled stack gas from a point 10 inches closer to the stack wall than did the OH reference method. This difference is not expected to affect the comparison of CEM and OH data in Section 6.1 because of the lack of stratification observed in the Unit 17 stack for other gases (SO_2 and NO_x).

Several problems were encountered with the MFS once it was installed at Schahfer Unit 17, including rapidly dropping mercury readings that were ascribed by the vendor to inlet contamination; improper connection of the rack system computer to the MFS; improper orientation of valves in the probe and MFS rack system; sampling flow rate set excessively high for the Unit 17 stack conditions; inadequate performance of inertial filter material in the sampling probe; and failure of a probe heater control board in the MFS rack system. Thermo Environmental representatives worked with the assistance of Schahfer facility staff to address these problems; however, proper operation of the MFS was not achieved until July 9.

As noted below, the field verification began with collection of a series of 12 OH samples from June 12 to15. The MFS system was not on site for that set of OH runs and was not fully operational until July 9, shortly before a second set of 12 OH sampling runs was conducted on July 10 to 13. The MFS then continued to monitor stack gas continuously until the end of the field test on July 25.

3.5 Verification Schedule

The MFS was verified between July 9 and July 25, 2006, in a portion of a field test that lasted from June 12 to July 25 and in which two sorbent-based mercury sampling systems and one other mercury CEM were also evaluated. The MFS became fully operational for sampling stack gas at Unit 17 on July 9 and was shut down on July 25, 2006. Table 3-2 shows the weekly activities relevant to the MFS verification that were conducted prior to and during the field period.

Table 3-3 shows the actual schedule of OH reference method sampling completed by ARCADIS in the week of July 10. The OH sampling proceeded efficiently, with three runs conducted on each of four successive days. In all cases, Thermo Electron personnel and other participating vendors were informed of the planned start time of each OH run and, in few instances, the start time of a run was delayed slightly to assure that the technologies being tested were fully ready to obtain data during the OH run. All OH runs were of exactly two hours duration, and Thermo Electron personnel were notified as the ending time of each run approached.

Table 3-2. Weekly Test Activities During the Field Period

Week of	Test Activity
May 15	Battelle trailer arrived at Schahfer facility
May 22	Electric power and other utilities established at Schahfer facility
May 29	No activity related to MFS
June 5	MFS umbilical installed at Unit 17 stack
June 12	First OH reference method sampling period
June 19	MFS rack components and probe arrive at test site
June 26	MFS trial operation and troubleshooting
July 3	MFS trial operation and troubleshooting
July 10	MFS fully operational; Second OH reference method sampling period
July 17	Routine operation
July 24	Routine operation concluded; MFS shut down and removed from Battelle trailer

Table 3-3. Schedule of OH Method Sampling in the Week of July 10, 2006

Run Number	Date	Start Time	End Time
1	7/10/06	9:00	11:00
2	7/10/06	11:50	13:50
3	7/10/06	14:55	16:55
4	7/11/06	8:30	10:30
5	7/11/06	11:15	13:15
6	7/11/06	14:00	16:00
7	7/12/06	8:30	10:30
8	7/12/06	11:40	13:40
9	7/12/06	14:15	16:15
10	7/13/06	8:20	10:20
11	7/13/06	11:10	13:10
12	7/13/06	13:45	15:45

Chapter 4
Quality Assurance/Quality Control

QA/QC procedures were performed in accordance with the quality management plan (QMP) for the AMS Center(5) and the test/QA plan for this verification test.(1) QA/QC procedures and results are described below.

One deviation from the test/QA plan occurred due to the inability to position the MFS sampling point at 1 meter inside the inner wall of the stack (see Section 3.4). A deviation form was prepared and approved noting this occurrence.

4.1 OH Reference Method

This verification test included a comparison of the MFS results to those of the OH reference method for flue gas mercury.(3,4) The quality of the reference measurements was assured by adherence to the requirements of the OH method, including requirements for solution and field blanks, spiked samples, and continuing calibration standards. All OH reference measurements were made with paired trains, and the percent relative deviation (%RD = the difference between the paired train results divided by the sum of those results, expressed as a percentage) of each data pair was required to be $\leq$ 10% (at mercury levels >1.0 µg/dscm) or $\leq$ 20% (at mercury levels $\leq$ 1.0 µg/dscm).(4) Data not meeting this criterion were excluded from comparison with the MFS results. The following sections present key data quality results from the OH method.

4.1.1 OH Reproducibility

The mercury results of the OH stack gas samples are shown in Table 4-1, for the July 10–13 period of OH method sampling. This table indicates the OH run number and lists the average vapor phase Hg_{OX}, Hg^0, and Hg_T results from the paired OH trains in each run and the %RD of each pair of results. All mercury results are in µg/dscm, i.e., adjusted to 20°C (68°F) and one atmosphere pressure.

Inspection of Table 4-1 shows that Hg_T in the Unit 17 stack ranged from 0.787 to 1.215 µg/dscm in the OH runs conducted in the July 10–13 period. The average Hg_T value in this period was 1.008 µg/dscm (note that one OH result for Hg_T is excluded from this average because of inadequate dual train precision, as described below). Hg^0 comprised the great majority of the Hg_T, consistent with the scrubbing of the Schahfer Unit 17 flue gas. Hg_{OX} never exceeded about 0.09 µg/dscm, and was typically about 5% of the Hg_T.

Table 4-1. OH Results from July 10–13, 2006, Sampling Period

OH Run	Mercury Concentration (μg/dscm) and %RD of Paired Train Results[a]					
	Hg_{OX}	%RD	Hg^0	%RD	Hg_T	%RD
1	0.033	10.1	0.902	0.8	0.935	0.4
2	0.037	2.9	0.823	1.4	0.860	1.2
3	0.040	3.7	0.929	1.1	0.969	0.9
4	0.066	52.3	0.886	1.4	0.952	4.9
5	0.029	11.6	0.757	0.3	0.787	0.1
6	0.038	2.0	1.018	6.5	1.056	6.4
7	0.028	5.7	1.055	1.2	1.083	1.3
8	0.084	7.2	0.997	12.6	1.081[b]	11.0
9	0.090	6.3	1.126	0.7	1.215	0.2
10	0.093	0.6	0.982	0.1	1.074	0.1
11	0.092	0.9	1.014	2.0	1.107	1.8
12	0.037	22.7	1.015	0.6	1.053	0.2

a: %RD = difference between paired train results divided by sum of paired train results.
b: This data point excluded from calculation of RA because %RD value exceeds acceptance criterion.

The %RD values in Table 4-1 show generally close agreement between the paired OH train results for all three mercury fractions. The %RD values are less than about 6.5% in almost all runs for both Hg^0 and Hg_T. The only exceptions were the results for OH Run #8. The Hg_T result from that run is excluded from calculations of RA because the %RD value is outside the 10% criterion for values >1.0 μg/dscm. The %RD values for Hg_{OX} are slightly higher than those for Hg_T and Hg^0, presumably due to the low Hg_{OX} concentrations, with two %RD values exceeding 20%.

4.1.2 OH Blank and Spike Results

Analyses were conducted on 10 total samples collected at the Schahfer site from the blank reagents used in the OH method in the July 10–13 period. Only two of those samples showed detectable mercury, with concentrations of 0.003 and 0.006 microgram per liter (μg/L), respectively. This blank reagent concentration is negligible in comparison to the mercury in impinger solutions recovered from trains after stack sampling. Those recovered sample concentrations were typically about 0.1 μg/L, 0.2 μg/L, and 4 μg/L in potassium chloride (KCl) solution, hydrogen peroxide (H_2O_2) solution, and potassium permanganate ($KMnO_4$) solution, respectively.

Blank OH sampling trains were prepared and taken to the sampling location on the Unit 17 stack on three occasions in each week of OH sampling, and were then returned for sample recovery without exposure to stack gas. These blank OH trains provide additional assurance of the quality of the train preparation and recovery steps. For the July 10–13 sampling period, the total amounts of mercury recovered from the three blank trains range from 0.193 to 0.250 μg, equivalent to less than 10% of the typical total amount of mercury recovered from a train after stack sampling at the Schahfer plant. Those blank train results correspond to stack gas mercury concentrations of less than 0.1 µg/dscm under typical sampling conditions in this verification.

All initial and continuing blank and calibration values from laboratory analysis of the OH samples met the requirements of the OH method. The recovery of mercury spiked into each reagent solution recovered from blank and sampled OH trains was also evaluated during laboratory analysis. Those spike recoveries ranged from 88 to 117% and averaged 100%. The recovery of mercury spiked into blank train samples as part of the performance evaluation (PE) audit also met the prescribed criteria, as described in Section 4.2.1.

4.2 Audits

Three types of audits were performed during the verification test: a PE audit of the reference method, a technical systems audit (TSA) of the verification test procedures, and a data quality audit. Audit procedures are described further below.

4.2.1 Performance Evaluation Audits

PE audits of the OH method were carried out through procedures implemented at the Schahfer plant during the field period. Table 4-2 summarizes the procedures and results of the PE audits of the OH reference method, showing the parameter audited, the date of the audit, the OH and reference values, the observed agreement, and the target agreement. The OH method incorporates dual sampling trains, and the equipment used by ARCADIS to carry out the OH sampling included dual Model 522 Source Sampler meter boxes (Apex Instruments, Fuquay-Varina, North Carolina) designated by their serial numbers as #2007 and #2008. As a result, for some parameters, Table 4-2 includes results for both meter boxes or for both of the dual OH trains.

Four PE audits were conducted:

- A Fluke Model 52 II digital thermometer (Serial No. 80730162) was used to audit the probe temperature measurements made by the #2007 meter box and the stack temperature measurements made by the #2008 meter box. For this comparison, the appropriate thermocouple was disconnected from the meter box and connected to the Fluke thermometer.

- A BIOS International Corporation DryCal National Institute of Standards and Technology-(NIST)-traceable flow measurement standard (Model DC2-B, Serial No. 103777, vendor-calibrated on May 9, 2006) was used to audit the sample gas flow rate with each of the two OH meter boxes.

- A set of weights (Rice Lake Weight Set, Serial No. 1JXA) calibrated to ASTM Class 3 standards was used to audit the electronic balance (AND FP-6000, Serial No. 6402118) used for weighing the OH method impingers.

- Recovery of mercury from OH trains was audited by spiking impingers containing KCl, H_2O_2/nitric acid (HNO_3), and $KMnO_4$/sulfuric acid (H_2SO_4) reagents in two blank OH impinger trains, with 1 milliliter (mL) of a prepared mercury solution, in each of the two separate periods of OH sampling. The mercury spiking solution was 2.5 µg/mL Hg in 1%

HNO_3 and was prepared by dilution of a NIST-traceable 1,000-ppm (i.e., 1,000-μg/mL) standard (Aa34n-1, Accustandards, Inc.). In the first week of OH sampling, Impingers 2, 4, and 5 of Blank Trains 8L and 8R were spiked; and, in the final week of OH sampling, Impingers 2, 4, and 6 of Blank Trains 7L and 7R were spiked.

Table 4-2 shows that all the PE audit results were within the target tolerances set in the test/QA plan.(1)

Table 4-2. Summary of PE Audit Results

Parameter	Date	OH Result	Reference Value	Agreement with Standard	Target Agreement
OH temperature measurement	6/14/06				
	probe T	228°F[a]	230°F	0.29%	2% absolute T
	stack T	127°F[b]	129°F	0.31%	
OH sample flow measurement	7/11/06	15.02 L/min[a]	14.56 L/min	3.2%	5%
		14.58 L/min[b]	14.35 L/min	1.6%	
Impinger weighing	6/14/06	199.72	200 grams	0.14%	Greater of 1% or 0.5 gram
		499.27	500 grams	0.15%	
Mercury spike recovery	6/14/06				
	train 8L				
	imp 2	2.48 μg	2.5 μg	0.8%	25%
	imp 4	2.02 μg	2.5 μg	19.2%	25%
	imp 5	2.08 μg	2.5 μg	16.8%	25%
	train 8R				
	imp 2	2.47 μg	2.5 μg	1.2%	25%
	imp 4	1.97 μg	2.5 μg	21.2%	25%
	imp 5	2.10 μg	2.5 μg	16.0%	25%
	7/12/06				
	train 7L				
	imp 2	2.24 μg	2.5 μg	10.4%	25%
	imp 4	2.12 μg	2.5 μg	15.2%	25%
	imp 6	2.38 μg	2.5 μg	4.8%	25%
	train 7R				
	imp 2	2.27 μg	2.5 μg	9.2%	25%
	imp 4	2.33 μg	2.5 μg	6.8%	25%
	imp 6	2.39 μg	2.5 μg	4.4%	25%

a: #2007 meter box.
b: #2008 meter box.
L/min = liters per minute; T = temperature; imp = impinger.

4.2.2 Technical Systems Audit

A Battelle Quality Management representative conducted a TSA at the Schahfer test site on June 14 to ensure that the verification test was being conducted in accordance with the test/QA plan(1) and the AMS Center QMP.(5) As part of the TSA, test procedures were compared to those specified in the test/QA plan,(1) and data acquisition and handling procedures, as well as the reference standards and method, were reviewed. The Quality Management representative observed OH method sampling and sample recovery processes, interviewed ARCADIS personnel, and observed the PE audit procedures noted above, except for the OH sample flow and second OH train spiking audits, which were conducted at a later date. Observations and findings from the TSA were documented and submitted to the Battelle Verification Test Coordinator for response. None of the findings of the TSA at the Schahfer site required corrective action. In addition, an internal TSA was conducted in the laboratory charged with analyzing the OH samples. This TSA was conducted by the ARCADIS independent QA Officer in the laboratory on-site at EPA in Research Triangle Park, North Carolina, on July 19 and July 27, 2006. None of the findings of this laboratory TSA required corrective action. Records from both TSA efforts are permanently stored with the Battelle Quality Manager.

4.2.3 Data Quality Audit

At least 10% of the data acquired during the verification test were audited. Battelle's Quality Manager traced the data from the initial acquisition, through reduction and statistical analysis, to final reporting to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked.

4.3 QA/QC Reporting

Each audit was documented in accordance with Sections 3.3.4 and 3.3.5 of the QMP for the ETV AMS Center.(5) Once the audit reports were prepared, the Battelle Verification Test Coordinator ensured that a response was provided for each adverse finding or potential problem and implemented any necessary follow-up corrective action. The Battelle Quality Manager ensured that follow-up corrective action was taken. The results of the TSA were submitted to the EPA.

4.4 Data Review

Records generated in the verification test received a one-over-one review before these records were used to calculate, evaluate, or report verification results. Data were reviewed by a Battelle technical staff member involved in the verification test. The person performing the review added his/her initials and the date to a hard copy of the record being reviewed.

Chapter 5
Statistical Methods

The statistical methods used to evaluate the performance factors listed in Section 3.1 are presented in this chapter. Qualitative observations were also used to evaluate verification test data.

5.1 Relative Accuracy

The RA of the MFS for Hg_T determination with respect to the OH reference method results was assessed as a percentage, using Equation 1:

$$RA = \frac{\left|\overline{d}\right| + t^{\alpha}_{n-1}\dfrac{S_d}{\sqrt{n}}}{\overline{x}} \times 100\% \qquad (1)$$

where *d* refers to the difference between the OH reference mercury concentration and the average MFS reading over the OH sampling period, and *x* corresponds to the OH reference mercury concentration. S_d denotes the sample standard deviation of the differences, while t^{α}_{n-1} is the *t* value for the 100(1 - α)th percentile of the distribution with n-1 degrees of freedom. The RA was determined for an α value of 0.025 (i.e., 97.5% confidence level, one-tailed). All paired OH data meeting the method quality criteria were eligible for inclusion in the calculation of RA. An RA of less than 20% is considered acceptable.(2) Alternatively, when the mean reference mercury level is less than 5.0 µg/dscm (as in this test), agreement of the overall mean MFS value within 1.0 µg/dscm of the mean OH value is also considered acceptable.(2)

5.2 Linearity

The linearity of the MFS response was assessed by comparing its responses to the Hg^0 standard concentrations, using Equation 2:

$$LE = \frac{\left|R - A\right|}{R} \times 100 \qquad (2)$$

where *LE* is the linearity error at each concentration, *R* is the reference mercury concentration supplied to the MFS, and *A* is the average of the triplicate readings at each concentration. LE within 10% is acceptable.(2)

5.3 Seven-Day Calibration Error

The assessment of calibration error was based on the difference between the MFS responses and the known mercury content of the zero or standard gas. Calibration error was calculated from the MFS responses to both the zero and calibration gases for each of the seven consecutive days of this test. Specifically, calibration error was calculated using Equation 3:

$$CE = \frac{|R - A|}{S} \times 100 \tag{3}$$

where *CE* is the calibration error as a percentage of the MFS span value, *R* is the reference mercury concentration supplied to the CEM, *A* is the MFS response to the reference gas, and *S* is the span value of the instrument. Acceptable calibration error is within 5%.(2) However, for this verification, a span value of 10 µg/dscm was assumed and, therefore, the secondary acceptance criterion of 1.0 µg/dscm (10% of span) applies.(2) The absolute value of the differences ($R - A$) were also reported.

5.4 Cycle Time

The upscale and downscale cycle times (essentially the rise and fall times) of the MFS response were determined as the elapsed time needed to achieve 95% of the final stable reading after switching from zero gas to stack gas and from a high mercury standard to stack gas, respectively. The slower (i.e., longer) of the two response times was reported as the cycle time of the MFS. Cycle times not exceeding 15 minutes are acceptable under Part 75.(2)

5.5 Data Completeness

Data completeness was calculated as the percentage of the total possible data return that was achieved by the MFS over the entire field period. This calculation used the total hours of data recorded divided by the total hours of data in the entire field period. The field period began at the start of the first OH method run on June 12 and ended at the shutdown of the CEM on July 25. For this calculation, no distinction was made between data recorded during stack gas monitoring and that recorded during calibration or zeroing, or in performance of linearity, cycle time, and seven-day calibration error testing. The causes of any substantial incompleteness of data were established from operator observations or vendor records.

Chapter 6
Test Results

The results of the verification tests of the Thermo Electron MFS are presented below for each of the performance parameters. To illustrate the overall results for this CEM, Figure 6-1 shows all of the MFS CEM's stack gas Hg_T readings for its period of operation, which spanned approximately 16 days, from July 9 (designated as Day 27) to July 25 (Day 44) of the field period. The x-axis label in Figure 6-1 defines the July 10 to 13 OH sampling period as days 28 to 31. Figure 6-1 shows that the Hg_T readings of the MFS were usually between about 0.8 and 1.7 µg/dscm. A frequent daily pattern of Hg_T readings is evident, in that lower Hg_T values were reported in the early morning hours when the load was reduced on Unit 17. The gap in data on Days 31 through 34 is apparently caused by a failure to record those data, as no data from that time period were found in the data file downloaded from the MFS at the end of the field period.

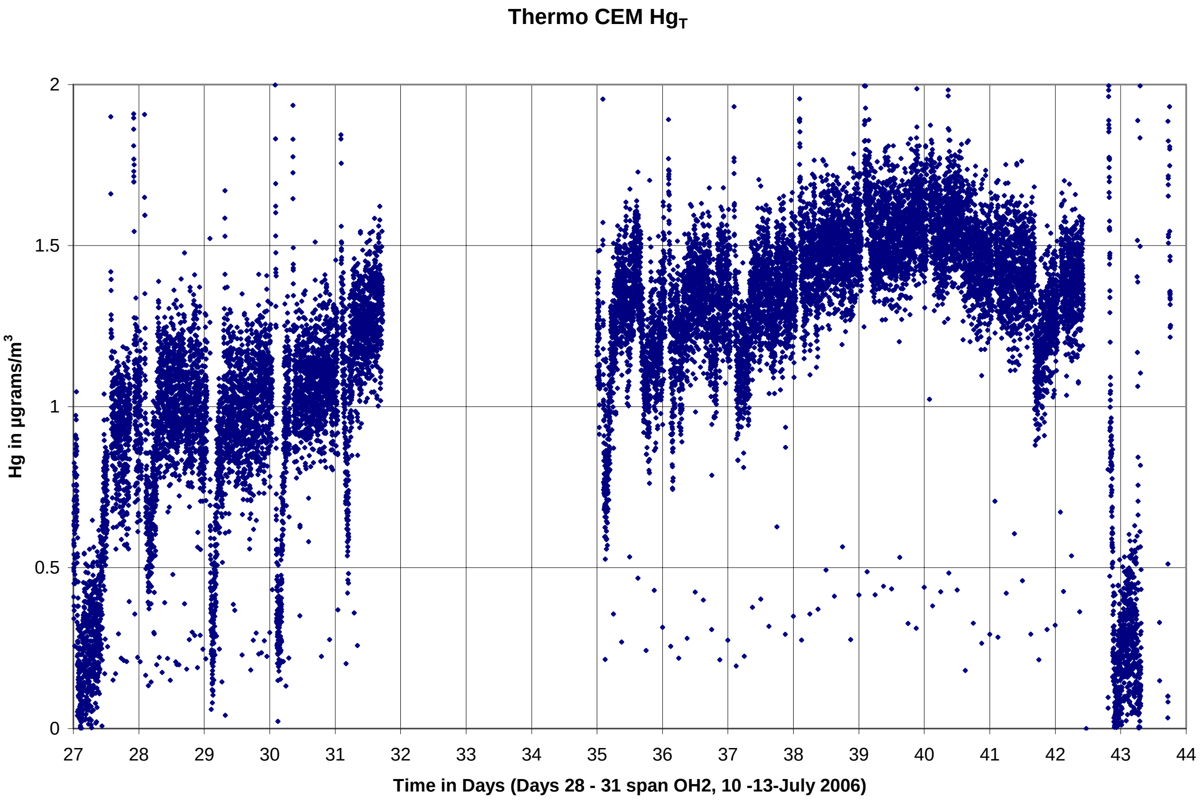

Figure 6-1. Hg_T Readings from Thermo Electron MFS During the Field Test

6.1 Relative Accuracy

The RA of the MFS with respect to the OH results for Hg_T was calculated using Equation 1 in Chapter 5. Table 6-1 lists the MFS results for those time periods corresponding to each of the OH sampling runs (see Table 4-1). The MFS Hg_T results in Table 6-1 are each the average of 120 one-minute average readings, obtained in continuous monitoring over the 2-hour period of each OH run. The MFS and OH results for Hg_T are shown graphically in Figure 6-2 for the July 10–13 sample set. Note that the OH result for Hg_T from run #8 in the July 10–13 sampling period was excluded from the calculation of RA (and from Figure 6-2) because the %RD value exceeded the acceptance criterion (see Section 4.1.1).

Table 6-1. Results from Thermo Electron MFS for Each OH Sampling Run

Date	OH Run	MFS Hg_T (µg/dscm)
7/10/2006	1	1.023
7/10/2006	2	1.038
7/10/2006	3	1.071
7/11/2006	4	1.004
7/11/2006	5	0.955
7/11/2006	6	0.971
7/12/2006	7	1.074
7/12/2006	8	1.071
7/12/2006	9	1.052
7/13/2006	10	1.236
7/13/2006	11	1.273
7/13/2006	12	1.294

Based on the Hg_T data in Tables 4-1 and 6-1, and shown in Figure 6-2, the RA of the MFS for Hg_T determination was calculated to be 16.4%.

In addition to the calculation of RA, the mean values of Hg_T from the OH method and the MFS CEM were compared. The data from run #8 were excluded from this comparison as well. The mean Hg_T value from the other 11 OH results was 1.008 µg/dscm, and the mean MFS Hg_T result for those same 11 runs was 1.090 µg/dscm, a difference of 0.082 µg/dscm. This difference is well within the 1.0 µg/dscm acceptable difference from the mean OH value.(2)

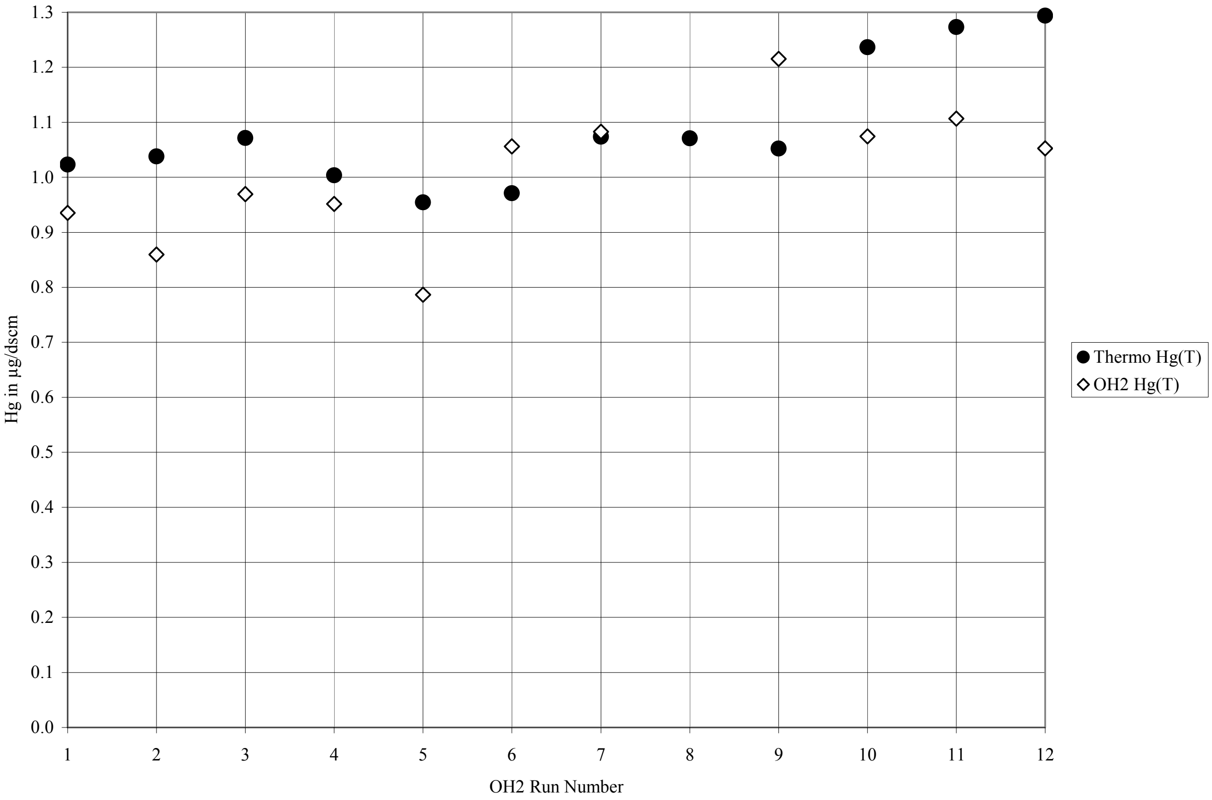

Figure 6-2. Thermo Electron MFS and OH Hg_T Results, July 10–13, 2006

6.2 Linearity

The linearity of the Thermo Electron MFS was evaluated over a concentration range of 3 to 9 µg/dscm. Table 6-2 shows the results of the linearity test. Shown in the table are the date of the test, the Hg^0 standard concentrations, the triplicate MFS responses to each mercury standard, the mean of the triplicate sets of responses, the difference between that mean and the standard value, and the resulting LE, calculated using Equation 2 (Section 5.2). As shown in Table 6-2, the LE of the MFS CEM was within 7.2% at the lowest concentration point and within 2.7% at the higher two points.

Table 6-2. Thermo Electron MFS Linearity Test Results

Linearity Test Date	Hg^0 Standard (µg/dscm)	MFS Responses (µg/dscm)	MFS Mean (µg/dscm)	Difference (µg/dscm)	LE (%)
July 25	3.00	3.24 3.17 3.24	3.22	0.22	7.2
	6.00	6.20 6.13 6.15	6.16	0.16	2.7
	9.00	9.20 9.24 9.25	9.23	0.23	2.6

6.3 Seven-Day Calibration Error

Calibration error of the Thermo Electron MFS CEM was determined based on zero and calibration responses obtained on each of seven consecutive days. Table 6-3 summarizes the results, listing the zero and calibration responses and the resulting calibration error, calculated according to Equation 3 (Section 5.3) and expressed as a percentage of the 10 µg/dscm span value. Table 6-3 shows that the MFS exhibited zero readings ranging from 0.024 to 0.027 µg/dscm (up to 0.27% of span), and differences from the 10 µg/dscm standard of up to 0.13 µg/dscm (up to 1.3% of span). All the MFS calibration results are well within the 5% of span acceptance criterion and the alternate 1 µg/dscm acceptance criterion for a span range of 10 µg/dscm.(2)

Table 6-3. Results of Zero/Calibration Stability Tests for Thermo Electron MFS

Date	Zero Readings (µg/dscm)	Difference from Standard[a] (µg/dscm)	Zero Error (%)[b]	Calibration Readings (µg/dscm)	Difference from Standard[c] (µg/dscm)	Calibration Error (%)[b]
July 17	0.025	0.025	0.25	10.06	0.06	0.6
July 18	0.025	0.025	0.25	10.00	0.00	0.0
July 19	0.024	0.024	0.24	10.04	0.04	0.4
July 20	0.026	0.026	0.26	10.01	0.01	0.1
July 21	0.027	0.027	0.27	10.07	0.07	0.7
July 22	0.026	0.026	0.26	10.09	0.09	0.9
July 23	0.027	0.027	0.27	10.13	0.13	1.3

a: Relative to standard concentration of zero.
b: Relative to span value of 10 µg/dscm.
c: Relative to standard concentration of 10.00 µg/dscm.

6.4 Cycle Time

The cycle time of the Thermo Electron MFS CEM was assessed by switching the CEM between sampling calibration or zero gas and sampling stack gas. This assessment was somewhat complicated by the integrated sampling mode of the MFS, which produced new mercury readings at one-minute intervals, and by noise in the CEM readings at the low stack gas mercury levels observed. However, data were sufficient to allow an estimate of cycle time. Table 6-4 presents the data from periods used to assess the cycle time of the MFS, showing the date, time, and value of the MFS readings in µg/dscm; the readings chosen as the initial and final readings, and readings near the 95% change level; and the estimate of cycle time (either rise time from zero gas to stack gas or fall time from calibration gas to stack gas).

Table 6-4. Assessment of Cycle Time of the Thermo Electron MFS

Date	Time	MFS Reading (µg/dscm)	Comments	Cycle Time Estimate
July 25	17:17	0.10		Rise time 5 to 6 minutes
	17:18	0.10		
	17:19	0.08	Initial reading	
	17:20	0.51		
	17:21	1.89		
	17:22	2.30		
	17:23	1.82		
	17:24	1.71	99.4% increase	
	17:25	1.64	95% increase	
	17:26	1.72	Final reading	
July 25	17:50	10.70		Fall time 3 to 4 minutes
	17:51	10.64	Initial reading	
	17:52	6.49		
	17:53	3.17		
	17:54	2.29	94.5% decrease	
	17:55	1.93	98.5% decrease	
	17:56	1.81		
	17:57	1.80	Final reading	

Table 6-4 shows that the MFS rise time was estimated to be 5 to 6 minutes, and the fall time as 3 to 4 minutes. The MFS reading initially overshot the stack gas Hg_T level upon switching from zero gas to stack gas (see readings in Table 6-4 at 17:17 to 17:26), so the readings actually settled toward the stable stack gas value rather than increasing to it. This behavior is apparently an artifact of the switching of gases supplied to the MFS probe. The longer of the two response times, i.e., 5 to 6 minutes, is the cycle time of the MFS.

6.5 Data Completeness

The total duration of the field test was from the start of the first OH sampling run on June 12 to the shutdown of the CEMs on July 25, a total of 43.4 days. The Thermo Electron MFS was operational in the field for only 16.8 days, from July 9 to July 25, or 38.7% of the entire field period. Table 6-5 shows a breakdown of the operating activities of the MFS over those 16.8 operational days.

Table 6-5 shows that the 16.8 days of operation of the Thermo Electron MFS CEM consisted of approximately 1.2 days of calibration, zeroing, and other programmed QC procedures; 0.1 days conducting or re-stabilizing after programmed filter blowback; and 15.4 days of routine monitoring of stack gas mercury. Unfortunately, for 3.3 days of that routine monitoring (19.7% of the Thermo Electron MFS operational time), data were apparently not recorded by the MFS CEM, so only 12.1 days of actual stack gas data were recovered from the MFS CEM. The great majority of this data loss occurred from July 13 to 17, as shown in Figure 6-1.

Table 6-5. Thermo Electron MFS Operational Activities July 9 to 25, 2006

Activity	Number of Measurement Intervals[a]	Days	Percent of Time
Stack Gas Monitoring	17,401	12.1	72.1%
Monitoring Data Not Recorded	4,757	3.3	19.7%
Filter Blowback	191	0.1	0.8%
Calibration/Zeroing/Other Checks	1,782	1.2	7.4%
Totals	24,131	16.8	100%

a: Each measurement was a one-minute average.

6.6 Operational Factors

The Thermo Electron MFS used only electrical power and facility compressed air as consumables. The electrical power needs consisted of two 15A circuits to run the rack system in the trailer, and three other 30A circuits for the umbilical, the stack sampling probe, and the mercury converter, respectively. The MFS used software that controlled all monitoring, calibration, and data acquisition functions and displayed the current mercury reading on the front panel of the mercury analyzer. The software is fully accessible by means of an analog phone line through a modem built into the MFS's computer, and Thermo Electron staff frequently used this means of access to the MFS. The software automatically saved data records, but did not automatically save a data file over daily or other time periods. Instead, the operator entered the range of records (date, time) to be downloaded, and the software compiled those records into a data file. This operation was carried out twice during the field period, on June 13 and July 25. Data recovery was incomplete in the second download, as about three days of data were apparently unrecorded by the Thermo Electron MFS. The data file produced by this download process was also hard to interpret, as MFS operations such as zeroes, calibrations, filter blowback, etc., were not clearly identified. Instead, Battelle staff were provided with a small Excel® file showing the alphanumeric code associated with each such operation, so that the data file could be interpreted. Battelle staff wrote a program to automatically decipher the codes in the MFS data file and insert identifiers for the operations. The Thermo Electron field engineer indicated that an update to the MFS software will be made to automate identification of each MFS reading.

The Thermo Electron MFS did not arrive at the Schahfer Unit 17 site until June 19, due to problems found in the vendor's pre-test checkout process, and continued to have problems once installed in the field. On June 22 the Thermo Electron field engineer suspected that the inertial particle filter in the sampling probe was removing mercury from the stack gas sample, so the probe was brought to ground level for cleaning. However, thorough cleaning of the probe on June 22 and 23 caused no improvement in MFS performance. Over the next several days (until a Thermo Electron engineer arrived on July 6), Schahfer facility staff spent extensive time with Thermo Electron representatives by telephone troubleshooting several alarms in the MFS, including low dilution flow; low filter dilution factor; pressure, flow, and span failure; high probe temperature; and low eductor pressure. During this period, a valve was found to be in the wrong position in the MFS sampling probe, and an improper connection was found between the MFS and the laptop computer in the rack. On July 6 there was no communication with the MFS probe controller, and over the next two days the Thermo Electron engineer worked with Schahfer staff to rework the probe, check for plugging, install a new circuit board, and reestablish communication with the probe. These staff cleaned the probe and found that the stack gas sample flow rate was set at 20 L/min when a flow of about 2 L/min would be appropriate for a wet stack such as at Unit 17. The sample flow was reduced to 2 L/min, and three critical orifices in the probe were cleaned and replaced. At this time, the Thermo Electron engineer also replaced a control board in the probe controller in the rack system and corrected a check valve in the rack system that was installed backwards. By July 9 the MFS was operating correctly; on the morning of July 10, the MFS was declared ready by Thermo Electron for monitoring in comparison with the OH method, and for the next several days the CEM operated apparently without problems and produced consistent readings of stack gas mercury. However, the CEM probe temperature and converter temperature were found to be out of normal range by the early morning of July 20, presumably due to a bad control board. The Thermo Electron engineer also reported difficulties during final calibration checks at the end of the test, on July 25, and suspected problems in the filter or probe.

Thermo Electron representatives spent about 11 man-days at the Schahfer Unit 17 test site during the field test and controlled the MFS remotely via modem in other periods. In addition, several hours were spent over multiple days in telephone conversations between Thermo Electron and Schahfer facility staff, troubleshooting the behavior of the CEM.

Chapter 7
Performance Summary

The RA of the Thermo Electron MFS was 16.4% for Hg_T, based on comparison to 11 OH reference results. The overall average value from that set of OH data was 1.008 µg/dscm, respectively, whereas that from the MFS was 1.090 µg/dscm, a difference of 0.082 µg/dscm.

The LE of the MFS was 2.6 to 7.2% when tested over the range of 3 to 9 µg/dscm.

The seven-day calibration error of the MFS was evaluated with zero gas and with a calibration gas of 10 µg/dscm Hg^0. Error in zero readings ranged from 0.24 to 0.27% of span, and error in calibration gas readings from 0.0 to 1.3% of span, in both cases relative to an assumed 10 µg/dscm span value.

Cycle time of the MFS was estimated to be 5 to 6 minutes, based on readings during switching from zero gas to sampling of stack gas. The MFS recorded a mercury reading every minute, so the cycle time was estimated as a multiple of this integration time.

Data completeness for the MFS was 38.7%, based on its operation for 16.8 days over the approximately six-week field test. Considering only those 16.8 days on which the MFS was fully operational, 12.1 days of stack gas data were recovered, 1.2 days were spent in calibration/zeroing/other instrument checks, and 0.1 day was spent in conducting or recovering from filter blowback. Another 3.3 days of routine stack gas monitoring proceeded without apparent problems but produced no recorded mercury data.

The MFS required 120V AC power and connection to facility compressed air. The MFS is controlled by software that can be accessed locally or remotely and that provides rapid control of all instrument operations and information on mercury results and instrument functions. Zeroing, calibrations, and other operations were not clearly identified in the data files that resulted from this software. It was necessary to decipher the data files, by means of a separate code file provided by Thermo Electron, to identify such operations in the data. The MFS suffered from several problems that delayed its arrival in the field and limited its operational time once there. Problems included inadequate performance of the inertial probe material, failure of probe heating and control circuit boards, improperly installed valves, excessively high sample flow for the stack conditions, and failure of communication with the MFS on-board computer.

The cost of the Thermo Electron MFS as tested was approximately $124,790, excluding the umbilical line, installation, and training.

Chapter 8
References

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4. *Performance Specification 12A – Specifications and Test Procedures for Total Vapor Phase Mercury Continuous Emission Monitoring Systems in Stationary Sources*, 40 CFR Part 60 Appendix B, July 2005.

5. *Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center*, Version 6.0, U.S. EPA Environmental Technology Verification Program, Battelle, Columbus, Ohio, November 2005.