BECHTEL JACOBS
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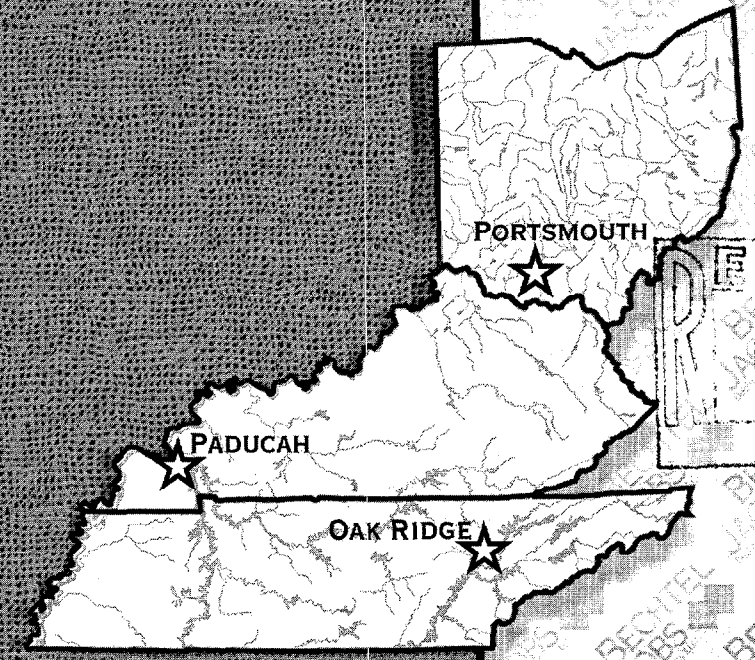
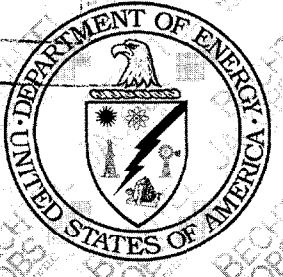
ENVIRONMENTAL MANAGEMENT
& ENRICHMENT FACILITIES
MANAGEMENT AND INTEGRATION CONTRACT

Field Evaluation of MERCEM Mercury Emission Analyzer System at the Oak Ridge TSCA Incinerator

East Tennessee Technology Park
Oak Ridge, Tennessee





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BECHTEL JACOBS COMPANY LLC
FOR THE UNITED STATES
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East Tennessee Technology Park Oak Ridge, Tennessee

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BECHTEL JACOBS COMPANY LLC
managing the
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East Tennessee Technology Park
Oak Ridge Y-12 Plant Oak Ridge National Laboratory
Paducah Gaseous Diffusion Plant Portsmouth Gaseous Diffusion Plant
under contract DE-AC05-98OR22700
for the
U.S. DEPARTMENT OF ENERGY

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ABBREVATIONS

A	ampere
AC	alternating current
APC	air pollution control
Btu	British thermal unit
°C	degrees Celsius
C	orifice meter discharge coefficient
CC	confidence coefficient
CD	calibration drift
CE	calibration error
CEM	continuous emissions monitor
CEMS	continuous emissions monitoring system
Cl_2	chlorine
cm	centimeter
CMST	Characterization, Monitoring and Sensor Technology
CMT	candidate monitoring technique
CNF	Central Neutralization Facility
CO	carbon monoxide
CO_2	carbon dioxide
cP	Centipoise
CVAAS	Cold Vapor Atomic Absorption Spectrometry
d	difference
dcf	dry cubic foot
dd	date number format of day
DOE	U.S. Department of Energy
dscf	dry standard cubic foot (usually at conditions of 68°F and 29.92 in. Hg)
dscm	dry standard cubic meter (usually at conditions of 68°F and 29.92 in. Hg)
EERC	Energy & Environmental Research Center (of the University of North Dakota)
EPA	U.S. Environmental Protection Agency
ETTP	East Tennessee Technology Park
°F	degrees Fahrenheit
F_{crit}	statistical F-test critical value
F_{exp}	statistical F-test observed value
ft	foot
ft/s	foot per second
g	gram
gpm	gallon per minute
h	hour
H_2O	water
H_2SO_4	sulfuric acid
HAP	hazardous air pollutant
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	hydrochloric acid
Hg	mercury
$HgCl_2$	mercuric chloride
hh	time number format for hour
HNO_3	nitric acid

HP	health physics
ID	inner diameter
IH	industrial hygiene
in. H_2O	inches of water
in. Hg	inches of mercury
IWS	ionizing wet scrubber
$KMnO_4$	potassium permanganate
kV	kilovolt
L	liter
L/h	liters per hour
L/min	liters per minute
lb	pound
lb-mol	pound mole
LCD	liquid crystal display
LMES	Lockheed Martin Energy Systems, Inc.
m	meter
µg	microgram
µm	micron
mA	milliampere
MACT	Maximum Achievable Control Technology
MD	mean deviation
min	minute
mL	milliliter
mm	date number format for month
MWFA	Mixed Waste Focus Area
n	number of data points
N	normality of a solution
N_2	nitrogen
NESHAP	National Emission Standards for Hazardous Air Pollutants
ng	nanogram
nm	nanometer
NO_2	nitrogen dioxide
O_2	oxygen
OD	outside diameter
% O_2	oxygen concentration, percent by volume, dry basis
P_c	corrected concentration
PC	personal computer
PCB	polychlorinated biphenyl
pCi	picocurie
PLC	programmable logic controller
P_m	measured concentration
PM	particulate matter
ppbv	parts per billion by volume
ppm	parts per million
ppmv	parts per million by volume
PS12	Performance Specification 12
psig	pounds per square inch, gage
°R	degrees Rankine
RA	relative accuracy

RATA	relative accuracy test audit
R_{CEM}	CEM response
RCRA	Resource Conservation and Recovery Act
R_{EM}	emission limit value
rf	response factor
R_{HL}	value of high-level calibration standard
RM	reference method
RM_{avg}	average of the reference method data set, or alternatively, value of emission standard
RSD	relative standard deviation
R_V	reference value
SCC	secondary combustion chamber
scf	standard cubic foot (usually at conditions of 68°F and 29.92 in. Hg)
scfh	standard cubic foot per hour (usually at conditions of 68°F and 29.92 in. Hg)
SD	standard deviation
sec	second
$SnCl_2$	stannous chloride
SO_2	sulfur dioxide
t	t-statistic
TSCA	Toxic Substances Control Act
µmho	unit of conductance, 10^{-6} reciprocal ohm
V	volt
vol %	volume percent
VOST	volatile organic sampling train
V/V	ratio, volume-to-volume
wt %	weight percent
Y	dry gas meter calibration factor
yy	date number format of year
ZD	zero drift

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The project team was led by the following individuals:

J. E. Dunn, Jr., Bechtel Jacobs Company LLC
L. V. Gibson, Jr., Lockheed Martin Energy Systems, Inc.
R. L. Baker, Aldora Technologies
I. Skegg and W. Sigl, SICK UPA GmbH (formerly Perkin Elmer Bodenseewerk Environmental and Process Control Division), Meersburg, Germany

David Dillehay, president of Aldora Technologies, made a prompt and substantial participation commitment without which the project could not have proceeded.

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Technical editing of the report was accomplished by Susan Fisher, whose patience never seems to wear out while waiting for final resolution of report details.

EXECUTIVE SUMMARY

The U.S. Department of Energy (DOE) and the Environmental Protection Agency (EPA) previously conducted testing of extended duration on three mercury continuous emissions monitors (CEMs) in the 1996–97 timeframe at a commercial cement kiln burning hazardous wastes at Holly Hill, South Carolina. The testing was in conjunction with a proposal on Maximum Achievable Control Technology (MACT) standards for hazardous waste combustors. The emission characteristics of the kiln, specifically the combination of high particulate matter, moisture, and acid gases, were believed to have contributed to the failure of the tested CEMs.

The MERCEM[1] mercury CEM analyzer for stack gases was selected for further evaluation on a DOE mixed waste incinerator at Oak Ridge, Tennessee, expected to present less adverse conditions. The MERCEM is manufactured by Perkin Elmer[2] in Germany and is represented in the United States by Aldora Technologies of League City, Texas. The overall scope of the evaluation was carried out over a seven-week period from September through October 1998. The performance of the MERCEM was evaluated according to proposed EPA Performance Specification 12 (PS12), as were alternative methods of calibration with reference concentrations of mercury and a qualitative assessment of long-term endurance under wet stack conditions. A total of two relative accuracy test series—one at each of two operating configurations—was conducted. A Phase I test was conducted while feeding liquid wastes to the incinerator, while a Phase II test evaluated the MERCEM during simultaneous liquid and solid waste feed operations. A summary of the MERCEM performance compared with the proposed specifications in PS12 is presented in Table ES.1.

The MERCEM exhibited potential at the mixed waste incinerator to meet proposed requirements under conditions of operation with liquid feeds only at stack mercury concentrations in the range of proposed MACT standards. Reliable performance under conditions of incinerating liquids and solids simultaneously was not demonstrated for the operating conditions and configuration of the host facility. The reliability of available reference materials, particularly mercury calibration gas in cylinders, was not adequately demonstrated without further evaluating their incorporation into routine operating procedures performed by facility personnel.

It was possible to conduct the demonstration safely at a facility incinerating radioactively contaminated wastes and to release the equipment for later unrestricted use elsewhere. Experience gained by this testing revealed a number of operation and maintenance and quality assurance issues related to the use of a total mercury CEM for compliance monitoring of emissions from a hazardous waste incinerator.

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[1] Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors.

[2] Perkin Elmer in Germany has been purchased by SICK UPA GmbH since the test was conducted.

Table ES.1. MERCEM performance with PS12 criteria at 130 μg/dscm emission limit

PS12 Criteria	Specification	Results
	Phase I	
Calibration & Zero Drift CD[a] ZD	 ≤10% of emission standard ≤5% of emission standard	 Pass[b] Pass
Calibration Error[c,d]	≤15% of reference concentration	Pass
Response Time	Sampling time ≤1/3 of averaging period for the applicable standard **and** delay time for reporting analysis no greater than 1 h (Batch CEMS)	Pass
Interference Test	≤10% of emission standard	Test not done in Phase I
Relative Accuracy	≤20% of mean value of reference method test data **or** ≤10% of emission standard	Pass Pass
	Phase II	
Calibration & Zero Drift CD[e] ZD	 ≤10% of emission standard ≤5% of emission standard	 Pass[b] Pass
Calibration Error[d]	≤15% of reference concentration	Pass
Response Time	Sampling time ≤1/3 of averaging period for the applicable standard **and** delay time for reporting analysis no greater than 1 h (Batch CEMS)	Pass
Interference Test	≤10% of emission standard	Pass
Relative Accuracy	≤20% of mean value of reference method test data **or** ≤10% of emission standard	Fail[b] Fail[b]

[a] One mid-test span value not conducted – cylinder at stack.
[b] Results include site-specific response factor (rf).
[c] Mid-level calibration error not assessed due to calibration gas supply limitations.
[d] Only Hg(0) calibration gas utilized.
[e] Drift period duration 6 consecutive days, not 7 days.

1. INTRODUCTION

1.1 BACKGROUND

The U.S. Environmental Protection Agency (EPA) proposed revised regulations for the burning of hazardous wastes in incinerators and boilers and industrial furnaces on April 19, 1996.[1] The proposed regulations outlined the eventual requirement of advanced continuous emissions monitors (CEMs) for some hazardous air pollutants (HAPs) or surrogates for HAPs and encouraged the use of CEMs for other HAPs or HAP surrogates. In support of these monitoring requirements, the EPA and the U.S. Department of Energy (DOE) formed a joint program to identify and test commercially available CEMs to meet the proposed requirements. As a result of a request for proposals, an extended duration test of three total mercury CEMs was conducted at a commercial hazardous waste burning cement kiln at Holly Hill, South Carolina, in 1996–97.[2] It is believed that failure of the CEMs was due to emission characteristics of the kiln, specifically the combination of high-particulate matter, moisture, and acid gases.[3] Furthermore, the monitors tested were designed to meet regulatory monitoring requirements for the German market and typically operate downstream of air pollution control devices, which control the amount of particulate matter, acid gases, and mercury in the flue gas to levels found on a hazardous waste or commercial incinerator with a wet scrubber. Thus, the test venue chosen was not a reasonable site for evaluating the mercury monitors.

Because of the uncharacteristically high levels of mercury in wastes stored throughout the DOE complex and the need to treat that waste, a follow-up test to demonstrate the feasibility of using mercury monitors at DOE mixed waste incinerators was planned. It was expected that a DOE mixed waste incinerator utilizing a wet scrubbing system would present less adverse conditions and thus allow a total mercury CEM to operate successfully. To demonstrate this point, the Mercury CEM System (MERCEM), manufactured by Perkin Elmer[1] in Meersburg, Germany, and represented in the United States by Aldora Technologies, was selected for further evaluation at the DOE Toxic Substances Control Act (TSCA) Incinerator located at the East Tennessee Technology Park (ETTP, formerly the K-25 Site) in Oak Ridge, Tennessee.

The overall scope of the evaluation was carried out within approximately a two-month period from September through October 1998. Perkin Elmer supplied a newly factory conditioned MERCEM mercury CEM system. Within the first 9 days on-site, the MERCEM was installed, commissioned, and underwent performance testing including a calibration error test and a one-week zero and calibration drift test, as well as a relative accuracy (RA) test with comparison to reference method measurements. The performance test was repeated after four weeks of instrument operation and data collection under normal operating conditions with interference response testing conducted as part of the second performance test. This period also allowed for the reference method wet chemistry results to be analyzed from the initial test effort.

The DOE Office of Science and Technology Characterization, Monitoring and Sensor Technology Crosscutting (CMST) Program and the Mixed Waste Focus Area (MWFA) funded the project. The mercury monitor and its related vendor support services and calibration gas cylinders were provided at no cost.

[1] Perkin Elmer in Germany has been purchased by SICK UPA GmbH since the test was conducted.

1.2 PROJECT OBJECTIVES

The objectives of the evaluation were as follows:

- evaluate performance of the MERCEM emissions monitoring system under wet (saturated flue gas) stack conditions of a mixed waste incinerator,
- evaluate methods for calibration with reference concentrations of mercury, and
- assess qualitatively the longer-term endurance of the MERCEM.

1.3 PROJECT OVERVIEW

To meet the project objectives within budgetary constraints, a technical approach and restrictive, but manageable, scope were developed. Site modifications to accommodate proposed testing were determined to be minimal with logistics and services to support the project readily available.

Available data from the TSCA Incinerator indicated that most of the previously measured mercury emissions may be vapor phase elemental mercury as is generally the case downstream of a wet scrubber. For this reason, a performance evaluation addressing speciated mercury was determined to be beyond the scope and budget of the project. Accordingly, U.S. EPA Method 101A [4] for total mercury was selected as a cost-effective alternative to Method 101B [5] for speciated mercury or to Method 29 [6] for metals including mercury. A sampling location in the stack was selected with the probe of the reference method train co-located with the MERCEM probe and remaining fixed (i.e., not traversed).

Performance testing attempted to address elements of EPA Draft Performance Specification 12 (PS12) [1], specifically:

- calibration error (CE) test,
- 7-day calibration and zero drift (ZD) test,
- RA test, and
- interference response test.

The MERCEM remained installed and collected data during the four weeks between each performance test to provide a qualitative assessment of longer-term operational issues.

1.4 PROJECT ORGANIZATION

The following organizations participated directly in the project:

- Lockheed Martin Energy Systems, Inc. (LMES, Oak Ridge, Tennessee);
- Bechtel Jacobs Company LLC (Oak Ridge, Tennessee);
- Aldora Technologies (League City, Texas and Hartsdale, New York);
- Perkin Elmer, Bodenseewerk Perkin-Elmer GmbH, Division Environmental and Process Control (Meersburg, Germany); and
- Spectra Gases (Alpha, New Jersey).

1.5 REPORT FORMAT

This report is presented in one volume with appendices. Chapter 1 provides an introduction and background. Chapter 2 provides a description of the host facility, preparation of the site and test logistics, and a description of the MERCEM analyzer. Chapter 3 discusses the technical approach to plan and execute the performance test and to the complete the field activities. Chapter 4 provides details of the specific technical procedures and mercury standards. Chapter 5 presents and discusses the results obtained during the evaluation. Conclusions are given in Chapter 6 and Chapter 7 lists recommendations. Chapter 8 provides references. Separate appendices are included for field test photographs (Appendix A), responsibilities of participating organizations (Appendix B), TSCA Incinerator Facility site access training requirements (Appendix C), a copy of a report on Germany Equivalency by TUV Rhineland Sicherheit und Umweltschutz GmbH for the MERCEM (Appendix D), reference method data (Appendix E), and raw field data (Appendix F).

2. DESCRIPTION OF HOST FACILITY AND MERCEM

2.1 GENERAL FACILITY DESCRIPTION

The TSCA Incinerator is the only operational incinerator in the United States that can process hazardous and radioactively contaminated polychlorinated biphenyl (PCB) waste. The facility is regulated by nine federal and state issued permits and approvals.

The TSCA Incinerator consists of a rotary kiln (7.5-ft OD, 6-ft ID x 25-ft long) and a secondary combustion chamber (SCC), which is 2702 ft^3 with 4-sec residence time. In addition to liquid and solid feed systems, there is an off-gas system as well as a kiln ash removal system and tanks and sumps for management of feeds and liquid effluents. A schematic of the TSCA Incinerator is shown in Fig. 2.1.

Organic liquids, aqueous, and solid wastes can be fed into the rotary kiln. Only high-heat organic liquid wastes are permitted to the SCC. The rotary kiln and SCC each have an auxiliary burner that utilizes natural gas to control incineration temperatures.

The off-gas cleaning system consists of a quench chamber, venturi scrubber, packed bed scrubber, two ionizing wet scrubbers (IWS) in series, an induced draft fan, and the exhaust stack. The off-gas cleaning system cools and saturates the combustion gases, neutralizes the acid gas components such as hydrochloric acid (HCl), and removes particulate matter (PM) from the off-gas.

In the quench chamber, combustion gases are cooled from the SCC exit temperature of approximately 2200°F to approximately 180°F by contact with fresh water and recycled scrubber liquids. Excess water collects in the recycle tank at the base of the quench chamber while the saturated gas stream is routed to the inlet of the venturi scrubber.

The automatic variable-throat venturi scrubber is between the quench chamber and the packed bed scrubber and removes some acid gases and particulate matter (>1μm). The venturi assembly consists of converging and diverging cones with an adjustable throat to allow the pressure drop to be varied. Venturi pressure drop normally is controlled between the range of 8.5 to 12 in. of water. Scrubber solution is injected through a nozzle upstream of the throat. The venturi scrubber has a demister on the outlet section to remove entrained liquid droplets, which are then drained to the quench recycle tank.

Additional HCl and other acid gases are removed from the gas phase by cross-flow contact with recycled scrubber solution in the packed bed scrubber.

Two IWS units are included for removal of submicron PM. The IWS units provide cross-flow contact of the flue gases with recycled scrubber solution. Each of the IWS units consists of an ionizer module followed by a packed bed section. Removal of particles from the gas stream in the IWS occurs in one of two ways: particles are electrically charged by energized wires in the ionizing section and migrate toward oppositely charged collection plates where they are collected, or they become attached to the wetted surfaces of the scrubber packing.

Downstream of the IWS units is the induced draft fan. The fan maintains a slightly negative pressure in the rotary kiln by pulling gases from the combustion chambers through the off-gas cleaning system. Treated combustion gases are exhausted to atmosphere through a 100-ft-high stack.

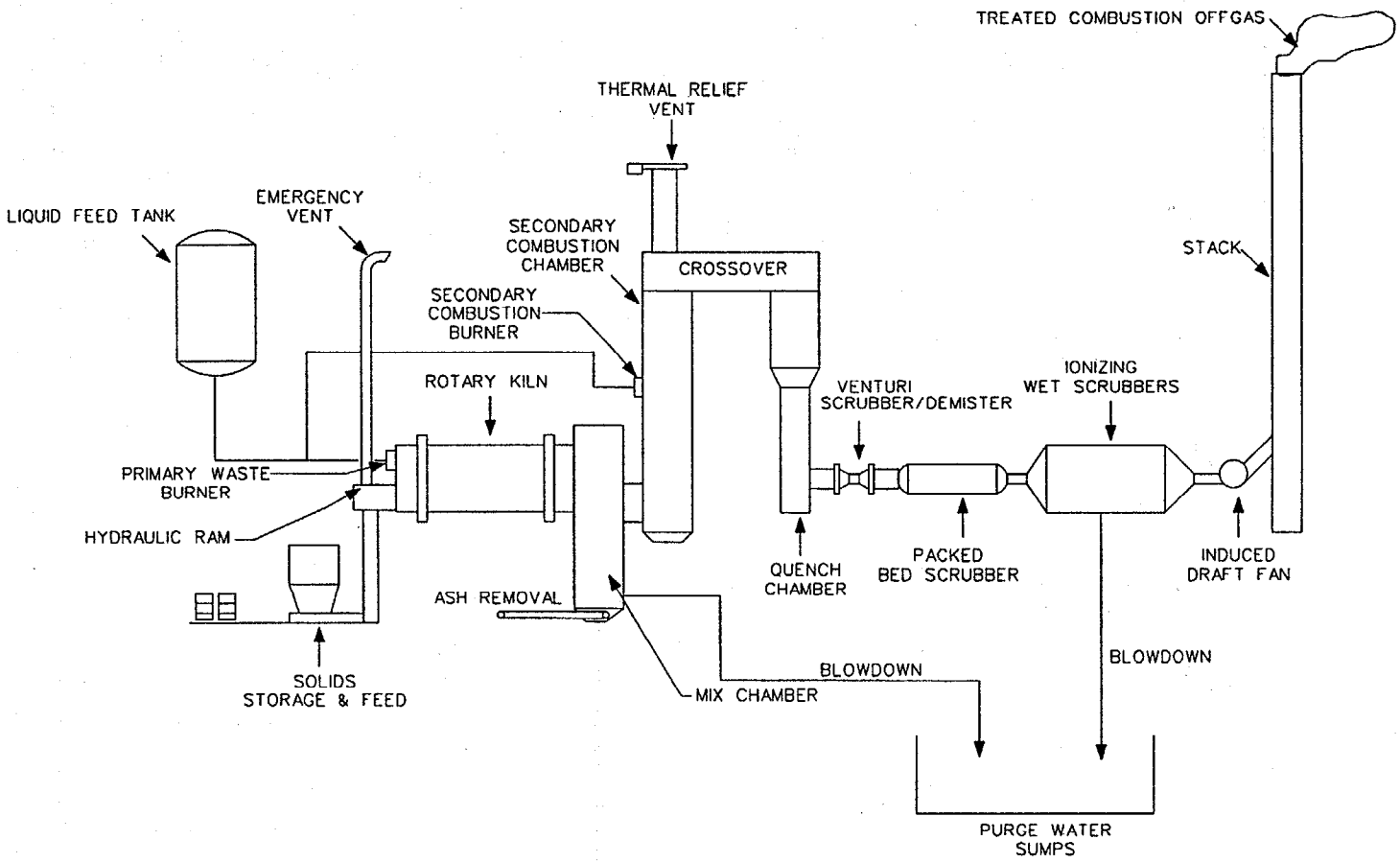

Fig. 2.1. Schematic of the TSCA Incinerator and off-gas cleaning system.

Additionally, to protect the air pollution control (APC) system from possible damage caused by high-inlet temperature excursions, exhaust gases from the SCC can be bypassed to atmosphere through a thermal relief vent. When the bypass occurs, the waste feeds are automatically stopped, auxiliary fuel is used to maintain the temperature in the SCC, and an alarm sounds to notify the operator. Bypass of the APC system is an infrequent emergency operation to protect the APC.

2.2 GENERAL FACILITY OPERATING CONDITIONS

Typical stack gas characteristics of the TSCA Incinerator are summarized in Table 2.1.

Table 2.1. TSCA Incinerator stack gas characteristics

Parameter	Value
Temperature	175–185°F
Moisture	50%
CO_2	5–8%
O_2	9–11%
CO	<10 ppmv
Pressure	-0.25 in. H_2O
Velocity	18–20 ft/s

Waste feed and operating temperature constraints for the TSCA Incinerator are summarized in Table 2.2.

Table 2.2. Waste feed and operating temperature constraints for the TSCA Incinerator

Parameter	Units	Primary organic	Secondary organic	Aqueous	Bulk solids	Total
Feed rate min.	lb/h	170	130	-	-	-
Feed rate max.	lb/h	826	630	380	650[a]	-
Heat content min.	Btu/lb	7000	10000	-	-	-
Btu feed rate max.	Btu/h	8,800,000	8,800,000	-	8,900,000	-
PCB feed rate max.	lb/h	450	450	450	300	450
Viscosity max.	cP	100	100	100	-	-
Ash liquid total	lb/h	-	-	-	-	44
Chlorine total	lb/h	-	-	-	-	260
Fluorine total	lb/h	-	-	-	-	20
Sulfur total	lb/h	-	-	-	-	88
Antimony total	lb/h	-	-	-	-	168
Arsenic total	lb/h	-	-	-	-	0.322
Barium total	lb/h	-	-	-	-	168
Beryllium total	lb/h	-	-	-	-	0.00175
Cadmium total	lb/h	-	-	-	-	0.78
Chromium total	lb/h	-	-	-	-	0.118
Lead total	lb/h	-	-	-	-	2.625
Mercury total	lb/h	-	-	-	-	0.02

Table 2.2 (continued)

Parameter	Units	Primary organic	Secondary organic	Aqueous	Bulk solids	Total
Nickel total	lb/h	-	-	-	-	168
Selenium total	lb/h	-	-	-	-	168
Silver total	lb/h	-	-	-	-	168
Thallium total	lb/h	-	-	-	-	280
Parameter			**Units**	**Kiln**	**SCC**	-
Min. Temperature, RCRA[b] wastes			°F	1572	2200	-
Min. Temperature, TSCA wastes			°F	1800	2200	-

[a] 950 lb/h with no liquid feed.
[b] RCRA = Resource Conservation and Recovery Act

2.3 SITE PREPARATION

2.3.1 Test Setup

Testing for this program occurred at the TSCA Incinerator owned by the U.S. DOE and managed by Bechtel Jacobs Company LLC for the U.S. DOE under contract DE-AC05-98OR22700. Lockheed Martin Energy Systems, Inc., is a DOE prime contractor that formerly managed the incinerator and currently manages the Oak Ridge Y-12 Plant under contract DE-AC05-84OR21400.

Field activities were supported by a mobile technology laboratory procured to support the establishment of a test bed for continuous emissions monitors.[7] The mobile technology laboratory consists of a pull-behind trailer approximately 48 ft long by 8 ft wide with commercial-grade construction throughout. It complies with all applicable codes and standards, including those of the Department of Transportation. The interior is finished with no exposed conduits or ductwork. Electrical service of 200 A is available, and the interior is air-conditioned, heated, and humidity controlled. Countertop workspace and storage cabinets are provided with countertops on one side capable of being folded down to accommodate rack-mounted instrument cabinets. A laboratory fume hood with a flush surface sink and cup drain is provided. There are side windows with separate portholes through which sample and instrument lines may be passed. An office area is separate from the laboratory.

The MERCEM was placed inside the mobile laboratory trailer for the duration of the test program. Electrical power, nitrogen, and plant air were supplied to the analyzer. A telephone line was installed in the laboratory. A personal computer (PC) and programmable logic controller (PLC) interface were also installed in the trailer for acquisition and storage of MERCEM data and for viewing the status of the incineration process. The PC had the capability to display the incinerator process, store data from the MERCEM's on-board PLC, trend the process and the MERCEM data, and correct MERCEM data for oxygen (O_2) concentration as measured by the facility O_2 CEMs.

Additional logistical considerations are outlined in Appendix B, which summarizes responsibilities assigned to the participating organizations.

2.3.2 Sampling Locations

The TSCA Incinerator stack (53.75 in. ID) has two stack-sampling platforms used for sampling and monitoring emissions from the APC system. Both are accessible by ladders from the ground. The lower platform is approximately 30 ft from the ground. One port at this location is dedicated to a probe that extracts stack gas analyzed for carbon monoxide (CO), carbon dioxide (CO_2), and O_2 by the facility CEMs. Other ports at this level are used for experimental CEM testing and compliance testing for gaseous pollutants. The upper platform is approximately 50 ft from ground level and contains ports for a continuous radionuclide sampling system, a continuous metals sampling system, reference methods requiring traverses, and experimental CEM testing.

Ports on the upper platform meet ideal regulatory sampling location requirements for particulate traverses: at least eight equivalent stack or duct diameters downstream and two equivalent diameters upstream from any flow disturbance.

The sampling probe for the MERCEM analyzer was installed in a port on the lower sampling platform as shown in Fig. 2.2. A heated sample line transported the gas sample to the analyzer in the trailer. The probe of the reference method sampling train was co-located with the MERCEM probe and remained fixed (i.e., not traversed). A sketch of the co-located probe arrangement inside the stack is shown in Fig. 2.3.

2.3.3 Incinerator Feed Operation

Incinerator feed operation during testing is discussed in Sects. 5.4, 5.5, and 5.6. Testing was conducted during normal incinerator operating conditions, and no feed streams were spiked. Performance data were obtained while incinerating liquids only and while incinerating liquids and containerized solids simultaneously.

2.3.4 Radiological Control

A unique concern associated with testing any type of equipment that comes in contact with TSCA Incinerator process streams is that of radiological contamination. The TSCA Incinerator thermally treats low-level radioactively contaminated hazardous waste containing PCBs and is categorized as a "Radiological Facility" per DOE guidelines. The radiological activity level of air emissions is low because the radiological activity in the feed material is low and most of the radiological activity in the off-gas stream is associated with PM removed by the APC system.

Although the likelihood of contaminating monitoring system hardware to the extent that release could not be allowed was considered to be remote, potential for radiological contamination was addressed. To ensure that the MERCEM left the TSCA Incinerator site in a radiologically clean state, HP technicians directly surveyed the probe and filter in the filter housing. In addition, the sample line was rinsed with nitric acid, and the rinse solution was sampled and analyzed for total activity. The condensate removed from the gas processed by the analyzer was also sampled and analyzed for total activity. As deemed necessary based on judgment by HP technicians, tubing connections inside the analyzer cabinet were disconnected to survey analyzer internal surfaces.

Other radiological contamination control measures included return of that portion of stack gas sample not processed for analysis back to the stack, filtering the exhaust of the processed gas sample, and return of liquid condensates to a sump for treatment.

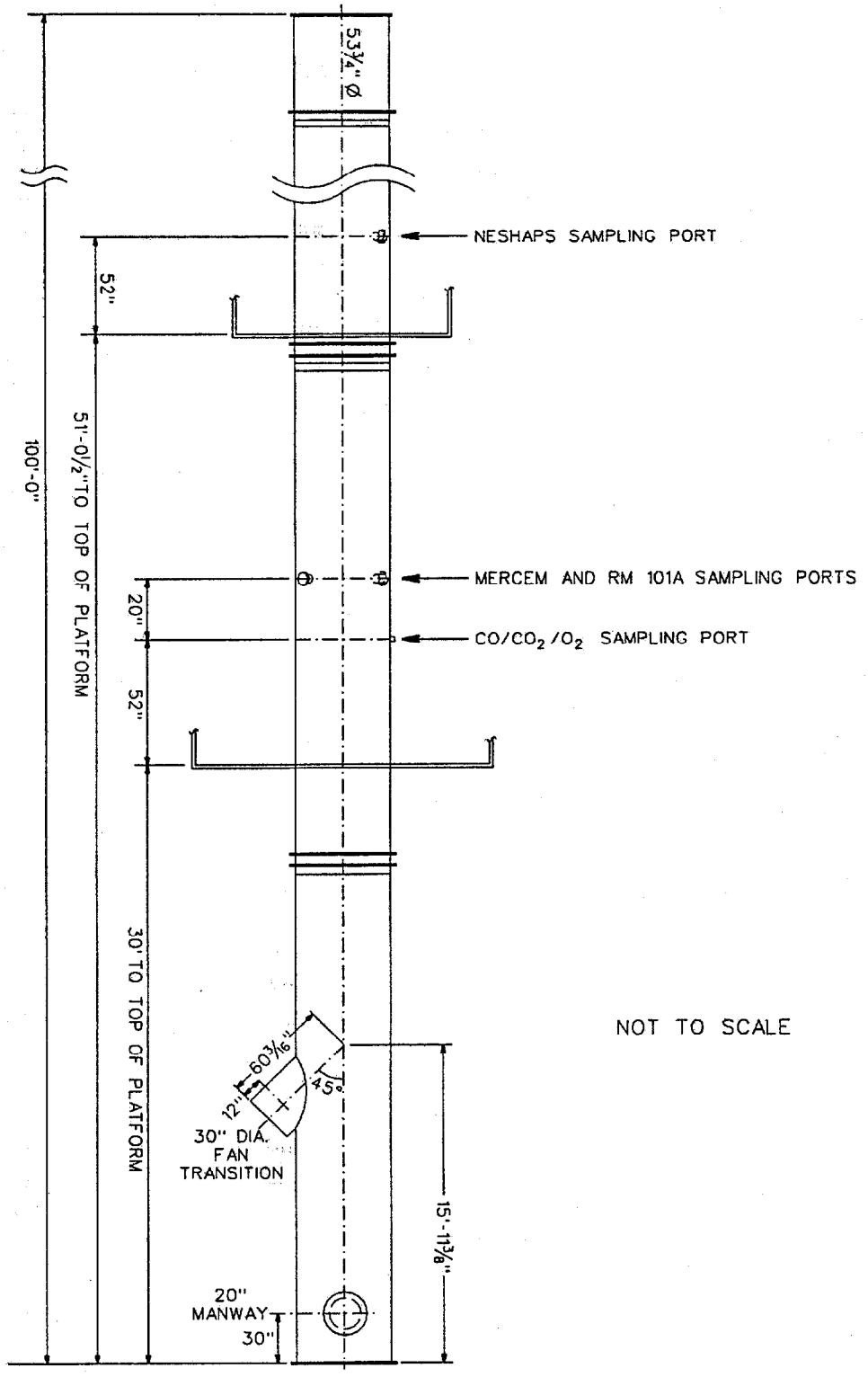

Fig. 2.2. Elevation view of MERCEM and reference method sampling ports.

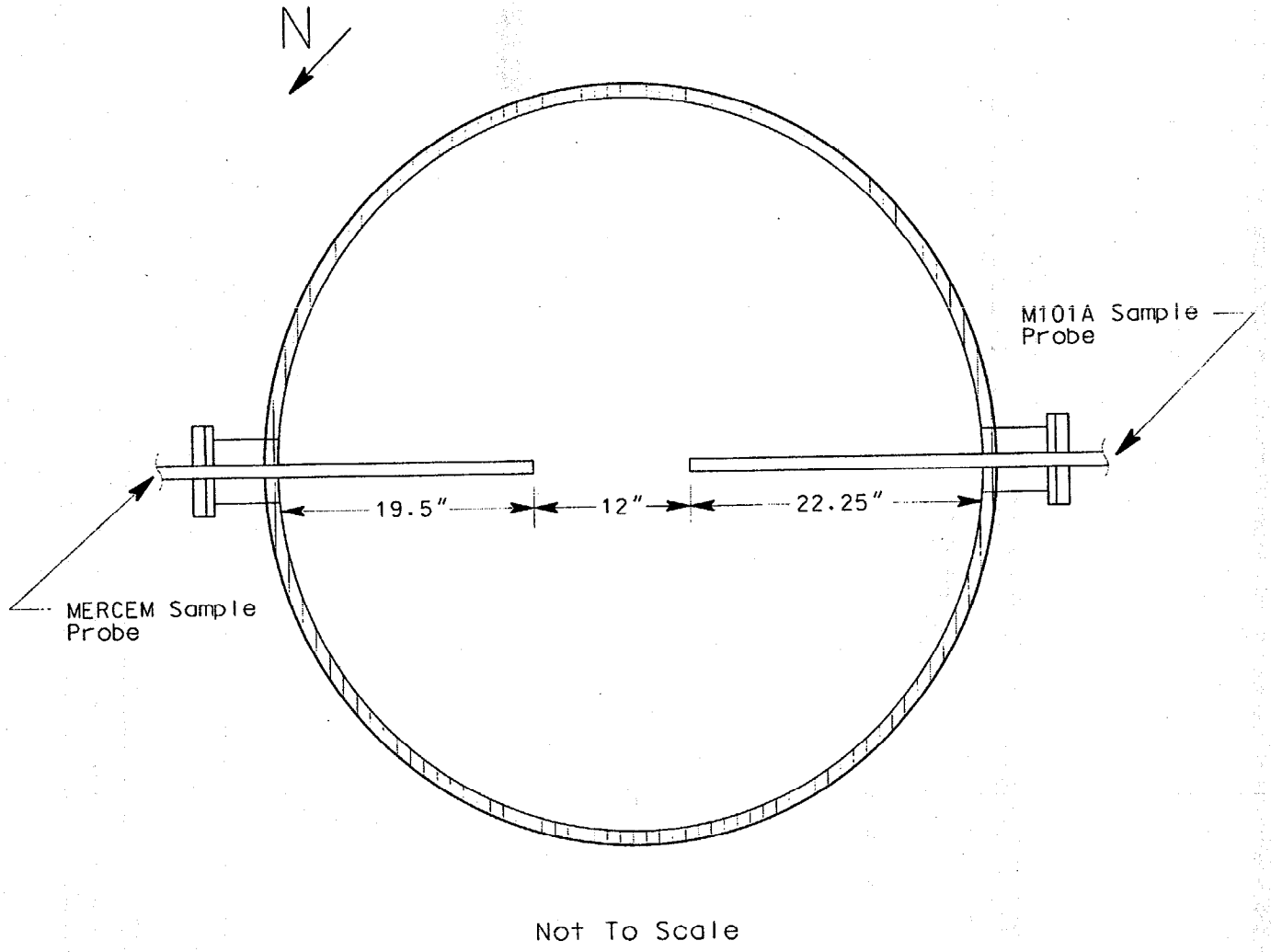

Fig. 2.3. Co-located arrangement of MERCEM and reference method sampling probes.

2.3.5 Health and Safety

Ensuring the safety and health of individuals participating in the project was of the utmost importance and of top priority during test planning. This project was conducted following the guidelines embodied by the DOE Integrated Safety Management System. All activities were planned in advance allowing all participants a chance to voice any concern or suggestion for improving the safety of an activity.

A safe and healthy environment was maintained by understanding the hazards involved in the work area and by being trained to deal properly with these hazards. All personnel participating in the test were required to meet the training requirements specified in Appendix C, based on their level of participation in the test and the work activities required of them.

Clear two-way communications were vital to the success of the project. To ensure clear communications regarding health and safety issues associated with carrying out this project, the actions listed below were taken.

- A "Take-Two" meeting was conducted prior to setting up the monitoring system to instruct test participants on the site safety program.
- Pre-test and post-test meetings were held to discuss any health and safety concerns or technical questions regarding the test.
- Two-way radios were issued to each organization participating in the field evaluation. The primary function of the radios was to keep everyone informed of the status of the test and facility conditions; however; the radios could also have been used to convey health and safety information in a rapid manner.

2.4 MERCEM ANALYZER DESCRIPTION

2.4.1 Measuring Principle

The MERCEM is designed to measure total mercury emissions from hazardous waste combustors. A gas sample is extracted from the stack through a probe with a coarse inlet filter at constant sample rate (not isokinetically) of 17 liters per minute (L/min) and transported to the analyzer through a Teflon-lined probe and sample system heated to 185°C to avoid condensation and corrosion.

At the CEM cabinet, a small portion of the sample flow (approximately 0.5 L/min) enters a reactor in which ionic mercury is reduced to elemental mercury by a stannous chloride ($SnCl_2$) solution. The sample gas containing vapor phase elemental mercury is separated from the liquid and enters an amalgamation unit. In the amalgamation unit, mercury is collected on a gold and platinum trap. At the end of the collection time period, the trap is purged with nitrogen and a photometric baseline is established. Then the trap is heated to drive off the mercury, which is measured by cold vapor atomic absorption spectrometry (CVAAS). The sensitivity of the instrument can be varied by changing the collection time, which is about 10 sec for a 0–100 micrograms per dry standard cubic meter (μg/dscm) measuring range. The entire cycle time is approximately 3 minutes. A flow chart for illustrating the sample path is shown in Fig. 2.4.

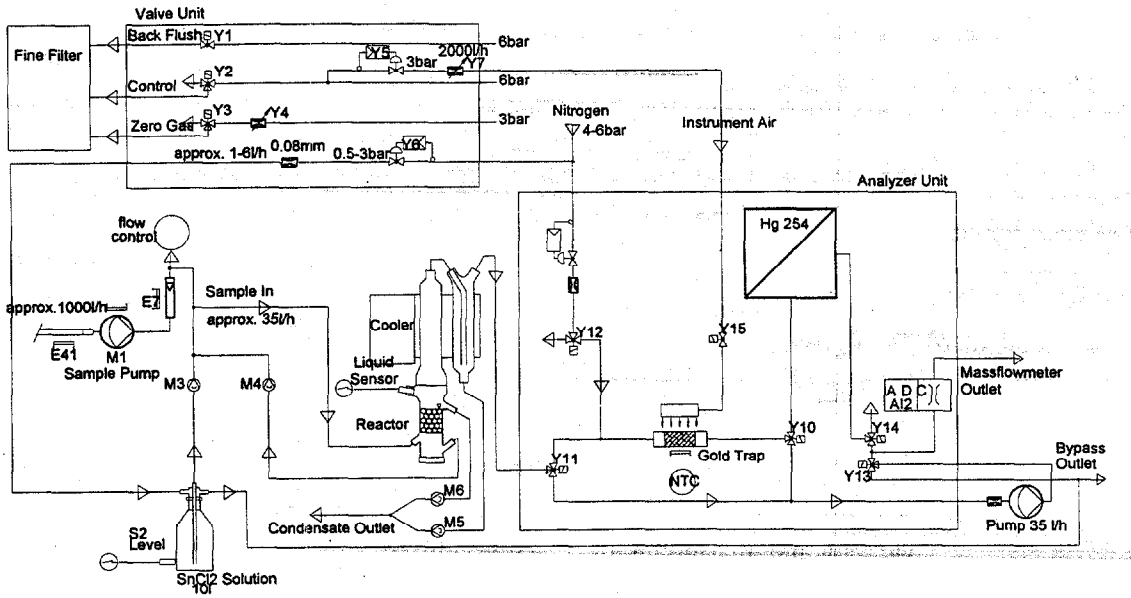

Fig. 2.4. MERCEM flow chart.

Because the flow through the photometer consists of only nitrogen and elemental mercury, optical interferences are eliminated. Finally, the remaining flue gas sample is dried and the volume flow rate measured so that instrument output can be reported on a dry basis. The reagents used in the analyzer are stored in the CEM enclosure and pumped continuously into the reactors. Replenishment of reagents and removal of waste solutions are required about every three months. The MERCEM uses an internal chiller to maintain the required instrument temperature. The MERCEM has been successfully approved in Germany by the TUV Rheinland Sicherheit und Umweltschultz GmbH. A copy of the comprehensive aptitude test report is attached as Appendix D.

2.4.2 Reduction Principle

Mercuric chloride ($HgCl_2$) is reduced to elemental mercury by means of the $SnCl_2$ reaction solution. The reaction starts immediately behind the gas diaphragm vacuum pump at the point where the stack gas is extracted from the bypass stream. The reaction is completed in the reactor and the gas is separated from the liquid. To prevent the reagent from being diluted by the stack gas, its concentration is maintained by refilling the reaction solution cyclically from a reservoir with a peristaltic pump. A second pump removes a slipstream of the solution. After having passed a two-stage Peltier cooler, the sample enters the gold trap for amalgamation at a dew point of approximately 5°C.

2.4.3 Amalgamation Principle

For a defined time, the stack gas is transported through a temperature-controlled gold trap whereby the mercury forms an amalgam with the gold. The sample flow is kept constant and recorded exactly by an electronic mass flow meter. Once the collecting period has elapsed, the gold trap is purged with nitrogen and the baseline of the analyzer is determined. Thus drift is eliminated. Thereafter the gold trap is electrically heated by means of resistor wire to purge the mercury from the gold. The nitrogen stream transports the mercury to the CVAAS photometer. An advantage of the amalgamation principle is that there are virtually no interferences with other stack gas components, and the sensitivity of the system can be adapted to the desired measuring range by using different sample collecting periods.

2.4.4 Photometric Concentration Determination

Together with the nitrogen, the mercury is conducted into the cell; the absorbance of the peak range is determined by photometric measurement. After calculation of the sample gas volume and the absorbance, the concentration is displayed on a liquid crystal display (LCD) panel. The concentration is also available as a 4- to 20-mA signal. Once the gold trap has been purged and cooled down by a strong air stream, the gold trap is prepared for the next sample.

2.4.5 Cycle Time

As a standard, the cycle time of a measurement with a measuring range of either 0–100 $\mu g/m^3$ or 0–300 $\mu g/m^3$ is 180 sec, as shown in Fig. 2.5. The measurement cycle is comprised of the following:

Cooling:	Approximately 30 sec
Collecting period:	10 sec
Baseline:	Approximately 50 sec
Heating and measurement:	Approximately 90 sec

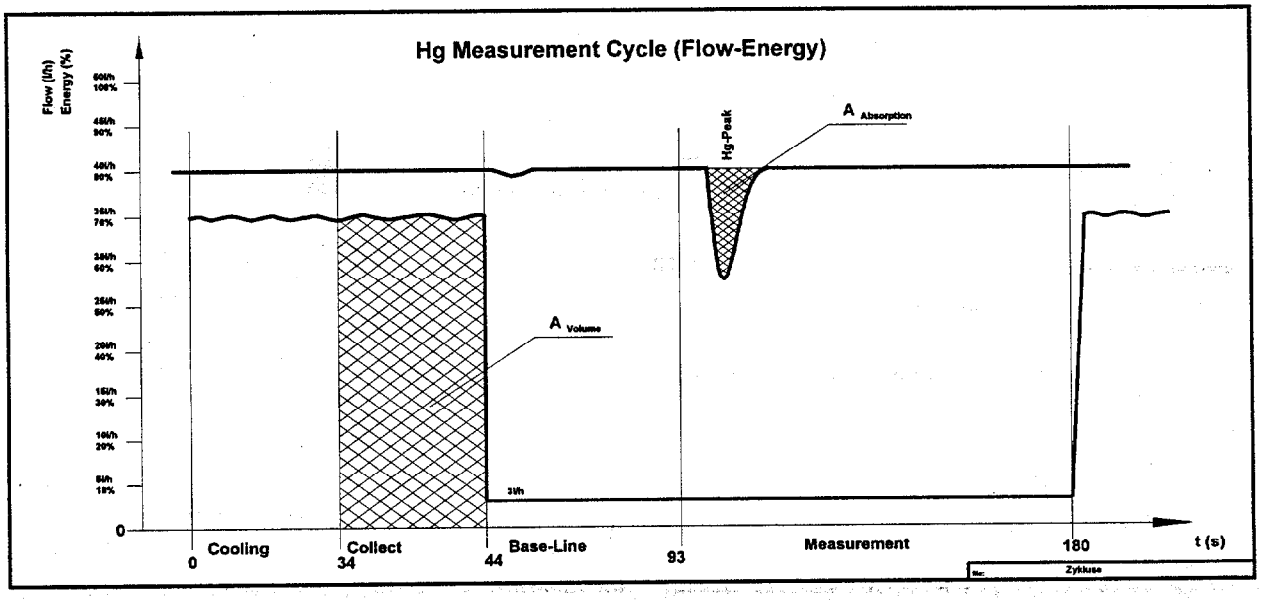

Fig. 2.5. Profile of a measurement cycle.

2.4.6 Optimizing the Sensitivity

By setting different sample collecting times, the sensitivity of the system can be adapted to the desired measuring range. For a measuring range of 0–100 $\mu g/m^3$, a sample flow rate of about 35 L/h and a collecting period of 10 sec are required. Figure 2.6 is an example curve of the photometer absorbance vs. the gold trap load. The collecting time for the gold trap must be reduced, if the energy decreases and there is a very high Hg concentration. A maximum of 20 ng of Hg may be collected on the trap.

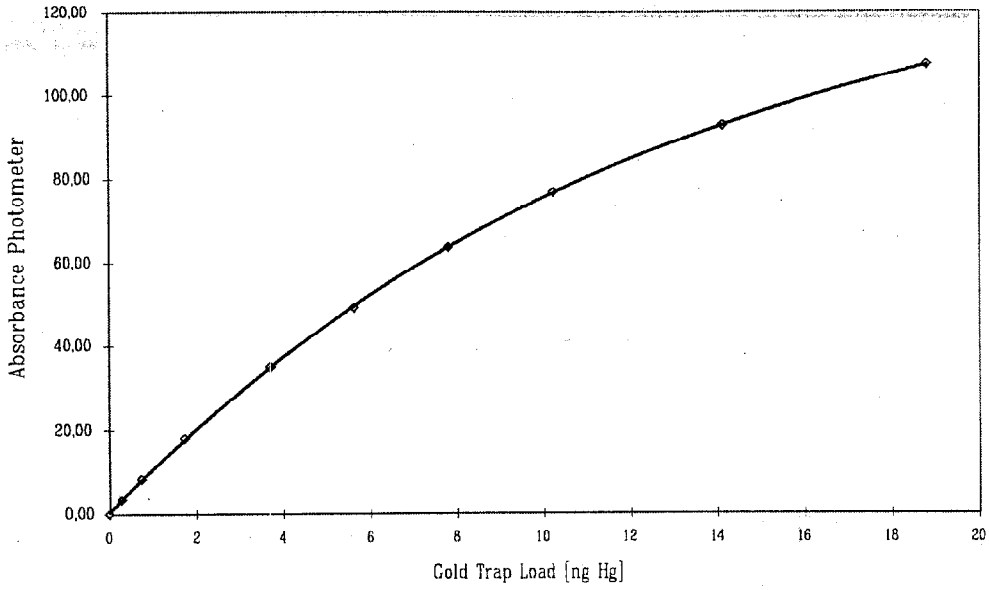

Fig. 2.6. Example curve representing absorbance vs. gold trap load.

Calculation of gold trap load

Quantity (ng) = Concentration ($\mu g/m^3$) * Volume (mL) * (1/1000)
Volume (mL) = Collecting period (sec) * Flow rate (L/h) * (1000/3600)

Example

Collecting period:	10 sec
Flow rate:	35 L/h
Concentration:	90 $\mu g/m^3$

Volume = 10 * 35 * 1000/3600 = 97.2 mL
Quantity = 90 * 97.2 * 1/1000 = 8.748 ng

Design

The MERCEM consists of the following major components:

- ACE 100 PLC control electronics,
- MFU 100 heating control,
- reactor,
- thermoelectric cooler,
- CVAAS analyzer unit,
- gas diaphragm vacuum pumps,
- peristaltic pumps,
- reservoir for $SnCl_2$ reduction solution, and
- opto boxes (signal transfer).

These components are mounted in a 19-in. steel Rittal cabinet as a stand-alone system with its own sampling system. The system cabinet is equipped with a safety shut-off feature that disconnects the electrical supply, if the main fuse is activated, or if the incorporated temperature safety switch detects a temperature inside the cabinet exceeding 63°C.

3. TECHNICAL APPROACH

3.1 PERFORMANCE TESTING

Performance testing was based on Draft PS12. Available data on mercury emissions from the TSCA Incinerator suggested most of the mercury to be in the vapor phase. Because of funding and schedule constraints, a performance evaluation on speciated mercury was deemed to be outside of feasible project scope. Additionally, Method 101A was selected as an alternate approved reference method and was more cost-effective compared to either Method 101B, for speciated mercury, or Method 29, for metals including mercury.

3.1.1 Preliminary Reference Method Testing

Although the MERCEM system was calibrated at the factory, the need was considered to conduct an initial series of reference method tests to develop a site-specific response factor to adjust MERCEM output readings before any measurements to determine relative accuracy. Such a measure was recognized to have merit particularly in cases in which reference materials are unavailable or unreliable. However, due to the short-term schedule of the project, no preliminary reference method testing was performed prior to the initial RA test. It was discussed that this type of site-specific response factor could be evaluated once test results from the first RATA series were available, which would be a three- to four-week turnaround. The MERCEM span setting remained at the factory setting of 1.0 throughout the entire test period. No span adjustment to either the gas cylinders or the permeation device was made.

The use of calibration correction factors is not defined in PS12. However, there has been discussion of the validity of this pragmatic approach. When mercury CEMs are used for applications throughout Germany, the CEMs are factory calibrated before installation, then compared against a series of wet chemistry tests similar to EPA's current reference method. All three commercially available Hg CEMs use a fixed gain factor to adjust the CEM output to the results of the wet chemistry tests. Because of reliance on comparison to a wet chemistry test, the MERCEM at the time of this evaluation did not have a certified internal reference standard. The MERCEM can use a supplemental permeation tube or calibration gas as a reference value for the purpose of daily calibration checks, but it does not adjust the output concentration on the basis of the response to these daily span checks. The CEMs' responses to the initial RA test during the six-month evaluation testing at Holly Hill, South Carolina, were used to derive a calibration adjustment factor for each CEM.[8] This response factor was applied to adjust the output of each CEM for all subsequent testing. A similar approach was used for portions of this data presentation (primarily calibration drift data) utilizing initial RA test data.

The response factor is defined as a simple ratio of the average reference method response over the average CEM response. This approach assumes linear response across the entire measurement range. The response of the CEM then assumes the form of a linear equation $\mathbf{y = mx + b}$. For the purpose of adjusting the data, the zero offset, $b = 0$; y = the adjusted data point, the response factor (rf) represents the slope (m) of the linear equation, and x = the actual CEM output.

3.1.2 Calibration Error Test

Calibration error (CE) is the difference between the concentration indicated by the CEMS and the known concentration generated by a calibration source when the entire CEMS, including the sampling interface, is challenged. PS12 prescribes that the CEMS be challenged three non-consecutive times at each measurement point and that the responses be recorded. The measurement levels are specified as a zero level (0–20% of the emission limit), mid-level (40–60% of the emission limit), and high-level (80–120% of the emission limit). According to PS12, calibration error is to be assessed using standards for both elemental mercury and mercuric chloride.

Calibration error was assessed only for elemental mercury.

The calibration error at each measurement point is calculated by

$$CE = \left| \frac{d}{R_v} \right| \times 100 \ ,$$

where

d = difference.
R_V = reference concentration value.

According to PS12, the mean difference between the indicated CEMS concentration and the reference concentration value at all test levels shall be no greater than ±15% of the reference concentration at each level.

3.1.3 Interference Response Test

Interference response testing was conducted during the CE test at the high concentration level. After the CE measurements were made, the interference test gases were substituted for a portion of the nitrogen dilution gas flow. The response of the MERCEM was recorded and compared three times alternately to that with the elemental mercury challenge alone. Each interference test gas was introduced singly. The interference test gases were injected into the sampling system in a manner that would allow for all the conditioning of an actual sample.

Table 3.1 summarizes the interference test gas concentration targets.

Table 3.1. Interference test gas concentrations in nitrogen

Gas	Concentration
Carbon monoxide	500 ± 50 ppm
Carbon dioxide	10 ± 1%
Oxygen	20.9 ± 1%
Sulfur dioxide	500 ± 50 ppm
Nitrogen dioxide	250 ± 25 ppm
Hydrogen chloride (HCl)	50 ± 5 ppm
Chlorine (Cl_2)	10 ± 1 ppm

Water vapor at 25 ± 5% was provided as an interference test gas from a steam generator calibrator unit furnished by Aldora Technologies.

Percent interference is calculated by

$$I = \left| \frac{d}{R_{HL}} \right| \times 100 ,$$

where

d = difference,
R_{HL} = value of the high-level calibration standard.

According to PS12, the sum of the interferences must be < 10% of the emission limit value.

3.1.4 Calibration and Zero Drift Test

Calibration drift (CD) is defined in PS12 as the difference in CEMS output readings from the established reference value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place. Zero drift (ZD) is defined as the difference in CEMS output readings for zero input after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place. PS12 is designed to allow calibration of the CEMS by use of a calibration standard that challenges the pollutant analyzer part of the CEMS (and as much of the whole system as possible), but which does not necessarily challenge the entire CEMS.

CD and ZD were evaluated daily for a 7-day period. ZD was determined by exposing the CEMS to zero gas. Calibration drift was determined by challenging the CEMS with elemental mercury only as specified in PS12.

CD is calculated by

$$CD = \frac{R_{CEM} - R_v}{R_v} \times 100 ,$$

where

R_{CEM} = the CEMS response,
R_V = the reference value of the high-level calibration standard (80–120% of the applicable emission standard).

ZD is calculated by

$$ZD = \frac{R_{CEM} - R_v}{R_{EM}} \times 100 ,$$

where

R_{EM} = the emission limit value.

According to PS12, the CEMS calibration may not drift or deviate from the reference value (R_V)of the calibration standard by more than 10% of the emission limit, and the zero point shall not drift by more than 5% of the emission standard. This raises a potential ambiguity in presentation of results when the calibration drift is also calculated as a percent of the reference value as instructed in PS12.

The April 19, 1996, EPA proposal provided for an emission standard for mercury of 50 μg/dscm corrected to 7% O_2. A subsequent notice of data availability and request for comment on May 2, 1997, changed the proposed emission limit for mercury to 40 μg/dscm corrected to 7% O_2.[9] An emission standard of 100 $\mu g/m^3$, based on understood potential direction of evolving regulatory decisions, was initially assumed for purposes of evaluating data from this field test. Final regulatory requirements that will become effective became available before publication of this report and are factored into results and discussion presented in Chapter 5.

3.1.5 Relative Accuracy Test

Relative accuracy (RA) is defined in PS12 as the absolute mean difference between the pollutant concentration determined by the CEMS and the value determined by the reference method (RM) plus the 2.5% error confidence coefficient of a series of tests divided by the mean of the RM tests or the applicable emission limit.

The RA test was conducted by comparing simultaneous MERCEM and RM measurements. The RM used was EPA Method 101A, "Determination of Particulate and Gaseous Mercury Emissions from Stationary Sources." The RM measurements were performed with a sampling probe co-located with the MERCEM probe at a fixed point (i.e., not traversed). Nine 1-h runs were conducted during each performance test period. The runs were scheduled during a consistent set of operating conditions for the incinerator to the extent possible. No additional waste feed characterization was performed beyond that required for operation under applicable regulatory permits and approvals.

RA is calculated by

$$RA = \frac{|MD| + |CC|}{RM_{avg}}$$

and

$$CC = t_{0.975}\frac{SD}{\sqrt{n}}$$

where

RA = relative accuracy,
|MD| = absolute mean difference between the CEMS and RM values,
|CC| = 2.5% error confidence coefficient (one-tailed),
RM_{avg} = average of the RM data set or the value of the emission standard,
SD = standard deviation of the differences between the CEMS and RM values,
$t_{0.975}$ = t statistic, 2.306 for nine runs,
n = number of sample pairs.

According to PS12, the RA of the CEMS must be no greater than 20% of the mean value of the RM test data in terms of units of the emission standard, or 10% of the applicable standard, whichever benchmark is greater.

3.2 ADDITIONAL PERFORMANCE CRITERIA

If a candidate monitoring technique (CMT) meets the RA criterion of PS12, it can be considered to have acceptable precision and accuracy. Failure to meet this criterion, however, could be caused by inaccuracies and imprecision in the reference method measurements. Additional criteria may be applied to assess the precision and bias of the monitoring technology.[10]

3.2.1 Precision

3.2.1.1 Relative Standard Deviation Ratio

The relative standard deviation (RSD) ratio can be used to evaluate whether a monitoring technique is more precise than the reference method:

$$\frac{RSD_{CMT}}{RSD_{RM}} < 1 \ .$$

The test is detailed in EPA Method 301.[11] A ratio of < 1 indicates that the CMT has a lower RSD than the reference method and is, therefore, more precise.

3.2.1.2 Achievable Precision

Based on the achievable precision for the reference method and consideration of potentially applicable standards, the following criterion may be applied to assess precision:

$$RSD_{CMT} \leq 50\% \text{ and } SD_{CMT} \leq 50 \ \mu g/m^3 \ .$$

Passing either the RSD ratio in the previous section or the 50% criterion described above may be considered acceptable. If the CMT cannot pass either of these criteria, one final precision criterion may be applied.

3.2.1.3 F-Test

The F-test (detailed in Method 301 and in 40 CFR 75, Subpart E) [12] can be used to evaluate the ratio of variances of the CMT and reference method at a statistically significant level. The following equation can be used for the F-test:

$$F_{exp} = \frac{SD^2_{CMT}}{SD^2_{RM}} \ ,$$

where

F_{exp} = F ratio,
SD_{CMT} = standard deviation of the CMT,
SD_{RM} = corresponding standard deviation of the RM.

Passing the F-test requires that F_{exp} be less than the critical value, F_{crit}, for that particular number of samples. For 9 runs, $F_{crit} = 3.438$. Note that the F-test is the most lenient of all the precision tests.

3.2.2 Bias

3.2.2.1 t-Test

The t-statistic at the 95% confidence level may be used to determine whether the difference between the CMT and the RM is statistically significant. Guidance for this criterion can be taken from EPA Performance Specification 2. [13] The following t-test equation can be used:

$$\frac{|MD|}{CC} \leq 1 \ ,$$

where

|MD| = absolute mean difference between the CMT and the RM,
CC = 2.5% error confidence coefficient (one-tailed),
SD = standard deviation of the differences between the CMT and RM,
n = number of sample pairs.

If the CMT fails to meet the above bias test, an alternative test can be applied.

3.2.2.2 Absolute Difference Test

An absolute difference test can be derived based on logic similar to the achievable precision test of Sect. 3.2.1.2 and expressed as follows:

$$|MD| \leq 50\% \text{ and } |MD| \leq 50 \ \mu g/m^3 \ .$$

3.3 OPERATIONAL TESTING

The MERCEM was installed and allowed to collect data for four weeks between scheduled performance tests to assess long-term operational issues in a qualitative sense. During the endurance testing, performance was documented in terms of data availability and maintenance requirements. This documentation consisted of the CEMS electronic data record and notations maintained by personnel checking the monitor.

3.4 VERIFICATION OF MERCURY VAPOR CALIBRATION GAS CONCENTRATIONS

Only gas phase elemental mercury was used to assess calibration error and calibration drift. Aldora Technologies investigated both calibration gases of known mercury vapor concentrations supplied in gas cylinders and the generation of a gas phase mercury standard using a permeation tube device.

Verification of the calibration gas concentration was attempted using an Arizona Instrument Corporation Jerome 431-X gold film mercury vapor analyzer, a direct reading field instrument used extensively in the workplace environment and for location of mercury spills with a range of 1–999 $\mu g/m^3$. Additionally, a midget impinger train based on the reference method, minus the probe and filter, was used to sample known volumes of the different challenge gases with analysis by wet chemical procedures.

4. REFERENCE METHOD SAMPLING, ANALYTICAL, AND CALIBRATION GAS VERIFICATION PROCEDURES

The objective of this field test was to determine whether the MERCEM was capable of meeting the draft performance specifications and data quality objectives proposed by the EPA in the draft Maximum Achievable Control Technology (MACT) rule for hazardous waste combustors under operating conditions of a U.S. DOE mixed waste incinerator. A major part of this evaluation involved comparison of the MERCEM measurements to simultaneous reference method measurements and calibration challenges to the MERCEM with reference concentrations of elemental mercury. A level of quality was assured for all of the activities with calibrations of sampling and analytical equipment performed according to reference method guidelines, and the work was performed under an organizational quality assurance plan with procedures in place for chain-of-custody, internal quality control, sample tracking, and reporting of results.

4.1 EPA METHOD 1: SAMPLING POINT DETERMINATION

EPA Method 1 [14] was used as criteria for sample and velocity measurement locations. With EPA Method 1, the duct or stack cross section is divided into equal areas. A traverse point is located in each of the resulting areas. The minimum number of traverse points depends on the equivalent duct diameter and distance in equivalent diameters to flow disturbances upstream and downstream of the sample location. The sample location is checked for cyclonic flow.

4.2 EPA METHOD 2: STACK GAS VELOCITY

EPA Method 2 [15] was used to determine the stack gas temperature, velocity, and volumetric discharge rate. The velocity of the gas is determined from the density of the gas and the measurement of static pressure and the average velocity head. A stainless-steel-sheathed Type K thermocouple is used to measure stack temperature while a stainless steel Type S pitot tube and an incline manometer are used to measure stack gas velocity. To minimize aerodynamic interference, the thermocouple and pitot tube are assembled according to the method specifications, including specifications when constructed with a sampling probe or nozzle as part of an assembly. Pre-test and post-test leak checks are conducted to ensure the accuracy of the velocity measurements.

4.3 EPA METHOD 3: STACK GAS ANALYSIS

During reference method measurements for RA determination, EPA Method 3 [16] was used to determine the stack gas O_2 and CO_2 concentrations and the dry molecular weight. In Method 3, an integrated stack gas sample is extracted from the stack over the duration of the reference method sampling run and analyzed with a classical Orsat analyzer. Data from the TSCA Incinerator facility O_2, CO_2, and CO CEMS were also available.

4.4 EPA METHOD 4: STACK GAS MOISTURE CONTENT

EPA Method 4 [17] as a reference method was conducted simultaneously with the pollutant measurement reference method run to determine moisture content of the stack gas. By Method 4, if the gravimetrically measured moisture content exceeds the saturation level at the measured stack gas temperature and pressure, the saturation level will be assigned for determining the stack gas wet molecular weight.

4.5 EPA METHOD 101A: PARTICULATE AND GASEOUS MERCURY EMISSIONS

EPA Method 101A was selected as the reference method for determining RA of the MERCEM as defined by PS12. In Method 101A, particulate and gaseous mercury emissions are withdrawn isokinetically from the source and collected in acidic potassium permanganate ($KMnO_4$) solution. During sample analysis, the Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by CVAAS.

4.5.1 Sample Train Configuration

The nozzle and the probe liner were constructed of quartz glass. The optional filter was omitted.

4.5.2 Sample Media

The impinger train was prepared as outlined in Table 4.1.

Table 4.1. Method 101A impinger contents

Impinger No.	Impinger type	Contents	Initial volume (mL)
1	Greenburg-Smith or modified Greenburg-Smith	4% $KMnO_4$/10% H_2SO_4	50
2	Greenburg-Smith	4% $KMnO_4$/10% H_2SO_4	100
3	Greenburg-Smith or modified Greenburg-Smith	4% $KMnO_4$/10% H_2SO_4	100
4		Initially empty	0
5	Greenburg-Smith or modified Greenburg-Smith	Silica gel, indicating	~200 g

4.5.3 Glassware Preparation

Glassware preparation followed the procedure of EPA Method 29. All sampling train glassware was first rinsed with hot tap water and then washed in hot soapy water. Next, glassware was rinsed three times with tap water, followed by three additional rinses with deionized water. All glassware was then soaked in a 10% (V/V) nitric acid solution for a minimum of 4 h, rinsed three times with deionized water, rinsed a final time with acetone, and allowed to air dry. All glassware openings where contamination could occur were covered until the sampling train was assembled for sampling.

4.5.4 Sampling Procedures

The probe of the reference method sampling train was co-located with the MERCEM probe and remained fixed (i.e., not traversed). Otherwise, the sampling train was generally operated according to protocol of an isokinetic reference method determination based on EPA Method 5. For each reference method determination, a sampling period of 60 minutes was established with a target gas sample volume of 30 dscf. Sampling parameter data were recorded every 5 minutes. In the event of process upsets causing automatic incinerator waste feed cutoffs, sampling was halted if normal incinerator operation could not be resumed within 5 minutes.

4.5.5 Recovery Procedures

The steps below were performed during sample recovery. If the characteristic purple color of the permanganate remained in the third impinger, the run was deemed as potentially good. Because of the potential reaction of $KMnO_4$ with acid, sample bottles were not completely filled and were vented to relieve pressure.

1. The weight of each impinger, which had also been pre-weighed, was measured within 0.1 g and recorded.

2. The contents of the liquid impingers were poured into a glass sample bottle (Container No. 1).

3. Taking care that material on the outside of the probe or other exterior surfaces did not get into the sample, mercury was quantitatively recovered from the probe nozzle, probe fitting, probe liner, impingers, and connecting glassware by rinsing with fresh 4% $KMnO_4$/10% H_2SO_4, carefully assuring removal of all loose particulate matter. The probe was rinsed with 100 mL of fresh acidic permanganate solution and the remaining glassware with another 100 mL.

4. To remove any residual brown deposits on the glassware following the permanganate rinse, the impingers were then rinsed with water and this rinse added to Container No. 1.

5. After the water rinse, the walls and stems of the impingers were rinsed with 50 mL of 8 N HCl, turning and shaking the impingers so that the 8 N HCl contacted the inside surfaces. The HCl wash was not placed into the acidified permanganate but into a separate container labeled Container No. 1A, to which 150 mL of water had been previously added. The HCl wash was then followed by rinsing the impinger walls and stems with a total of 50 mL of deionized water and placing that rinse in Container No. 1A.

4.5.6 Blank Corrections

For the RA assessment, emission sample values were corrected by simple subtraction of the corresponding measured field blank train values. A value of zero was assigned to analytical laboratory results lower than detection or reporting limits.

4.6 MERCURY VAPOR CALIBRATION GAS STANDARDS AND VERIFICATION

4.6.1 Reference Materials

4.6.1.1 Calibration Gas Cylinders

Spectra Gases (Alpha, New Jersey) generously offered to supply the project with cylinders of elemental mercury calibration gas. Table 4.2 provides a summary of the nominal prepared concentrations. The project initially requested 50% and 90% bottles for the calibration error test and 80% for the daily calibration drift test based upon a MERCEM range of 0–100 $\mu g/m^3$. Later in the project, two more gas bottles were requested. These included a higher concentration gas targeted for 80% of a 0–300 $\mu g/m^3$ range that was needed after transient spikes of mercury were detected in the flue gas. The other bottle requested was a lower concentration gas at 30% of a 0–100 $\mu g/m^3$ range to test the low emission end. Spectra responded by providing two pairs of cylinders for the Phase I testing in September that were made at the same time with target concentrations of 6 parts per billion by volume (ppbv) (CC90909 and CC90913) and 8 ppbv (CC90843 and CC90848). Cylinders CC94705 and CC94785 were identified as 3 ppbv and 30 ppbv, respectively, based upon Spectra's coarse measurement method. They were shipped to the test site in October and arrived just in time for Phase II testing.

Table 4.2. Mercury calibration gas cylinders provided by Spectra Gases

Cylinder No.	Nominal prepared Hg concentration (ppbv)
CC90843	8
CC90848	8
CC90909	6
CC90913	6
CC94705	3
CC94785	30

4.6.1.2 Elemental Mercury Permeation Tube Calibration Apparatus

The project procured from Aldora Technologies an elemental mercury permeation tube calibration device controlled by an isothermal bath with nominal range of 0–150 $\mu g/m^3$. The permeation tube apparatus consisted of a Fisher Scientific isothermal water bath maintained at 50°C. A VICI Metronics elemental mercury capillary was used as the mercury source. The carrier gas was plant nitrogen. The VICI capillary was placed in a quartz glass U-tube and connected to the inlet and outlet ¼-in.Teflon tube lines with silicon rubber tubing couplings. The flowrate through the capillary was monitored utilizing a rotameter. The manual set point flow of the rotameter was 60 liters per hour (L/h). The gas exiting the permeation tube apparatus was delivered to the MERCEM analyzer by manually disconnecting the tubing from the cooler outlet (cold vapor atomic absorption unit inlet). The flowrate required by the CVAAS unit at this point is approximately 35 L/h. The surplus sample flue gas was vented to atmosphere (via a tee at the permeation tube outlet) to ensure adequate supply and not to create any pressure effects. It was found that it was necessary to "purge" the permeation tube device for several hours to obtain repeatable results. In some of the initial tests utilizing the permeation tube prior to a uniform purging protocol, the results were less consistent. It was further noticed that the water bath level could influence the reading and had to be maintained at a consistent level. The VICI mercury capillary was identified as being "calibrated" at 50 ng/min.

4.6.2 Verification of Calibration Gas Concentrations

4.6.2.1 Jerome 431-X Mercury Vapor Analyzer

The test plan also called for comparison readings of the MERCEM and a Jerome 431-X gold film mercury vapor analyzer manufactured by Arizona Instrument Corporation. The Jerome 431-X is an ambient analyzer designed for analysis of mercury vapor in the workplace and for the location of mercury spills. The analyzer has a range of 0–1 mg/m^3 mercury. The principle of operation involves the use of a thin gold film, which exhibits an increase in electrical resistance in the presence of mercury vapor proportional to the mass of mercury in the sample. Activating the sample mode starts an internal pump, which draws air through a scrubber filter and into the flow system of the instrument. After 2 sec, the sample solenoid bypass opens, closing off the scrubber filter from the flow system. The sample air passes through an acid gas filter and is drawn over the gold film sensor. The sensor adsorbs and integrates the mercury vapor. After 7 sec, the sample solenoid bypass closes and the remainder of the sample is drawn through the scrubber filter and the flow system. The measured concentration is then displayed on the digital meter. After a large number of samples, accumulated mercury is thermally desorbed manually from the sensor.[18]

4.6.2.2 Method 101A Midget Impinger Train

A modified Method 101A impinger train, minus the probe and filter, with the midget impingers arranged in the same configuration as Method 101A, was also used by LMES personnel for calibration gas verification. This was based on a modified Method 101A impinger train previously used for calibration gas verification during the joint EPA/DOE demonstration.[8] Table 4.3 outlines the impinger contents. A "VOST" (volatile organic sampling train) pump and dry gas meter were used to draw a known gas volume through the train.

Table 4.3. Modified Method 101A impinger contents

Impinger No.	Impinger type	Contents	Initial volume (mL)
1	Modified Greenburg-Smith	Empty	
2	Modified Greenburg-Smith	4% $KMnO_4$/10% H_2SO_4	10
3	Modified Greenburg-Smith	4% $KMnO_4$/10% H_2SO_4	10
4	Modified Greenburg-Smith	Empty	
5	Modified Greenburg-Smith	Silica gel, indicating	~20 g

The verification train was recovered in a similar fashion to the reference method source testing train. The liquid impingers were collected in a sample bottle labeled "Container No. 1." The impingers were rinsed with 10 mL of fresh acidic permanganate solution followed by 10 mL of water with these two rinses added to Container No. 1. Then the impingers were rinsed with 10 mL of 8 N HCl, which was placed in a separated container labeled "Container No. 1A" prior to which 80 mL of water had been added. Train glassware was rinsed with deionized water prior to reuse and initially cleaned with the glassware preparation procedure of EPA Method 29 using 10% HNO_3, as noted in Sect. 4.5.3.

4.6.2.3 Calibration Gas Cylinder Analysis by the Energy & Environmental Research Center

One calibration gas cylinder each of the 6 ppbv and 8 ppbv concentrations (CC90843 and CC90913, respectively) was sent to the Energy & Environmental Research Center (EERC) of the University of North Dakota in Grand Forks, North Dakota, for laboratory analysis prior to the start of field test activities. The two original cylinders sent to EERC for analysis were returned along with cylinder CC94705 for post-test wet chemical verification analysis.

4.7 ANALYTICAL PROCEDURES

Analytical procedures primarily involved the measurement of mercury from reference method source testing associated with RA testing and the measurement of radiological total activity to support release of equipment free of contamination.

4.7.1 Mercury Analysis

Mercury analysis of acidic permanganate impinger solutions and 8 N HCl rinses was performed separately by SW-846 Method 7470.[19] Method 7470 is a cold-vapor atomic absorption technique based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

4.7.2 Radiological Total Activity

The procedure for total activity screening involved counting on a Tri-Carb scintillation analyzer. Aqueous samples that require no preparation are added directly to the scintillation cocktail. Solid and semi-solid sample aliquots are digested in nitric acid on a hot plate, cooled, filtered, and diluted to a specified volume. Oil sample aliquots are weighed directly into a tared counting vial. A specified volume of liquid scintillation cocktail is added to each vial and mixed with the sample aliquot prior to counting.

5. DISCUSSION OF RESULTS

5.1 PROJECT CHRONOLOGY

Table 5.1 provides a chronology of key events during execution of the project.

Table 5.1. Key events of MERCEM field test

Date	Key events
Installation and Commissioning	
September 8	Equipment arrival on-site
September 9–10	MERCEM analyzer commissioning
September 11–14	Analyzer optimization and fine-tuning
Phase I Performance Testing	
September 11–18	First 7-day calibration and zero drift test
September 15	First calibration error test
September 16–17	First RA test
September 18–October 18	Operational testing
October 8	Mid-operational testing period inspection
Phase II Performance Testing	
October 19–23	Second 7-day calibration and zero drift test
October 20	Second calibration error test
October 22–23	Second RA test
October 24	Interference response testing
October 25	Decontamination and decommissioning
November 4	MERCEM analyzer shipped from host facility

5.2 MERCEM INSTALLATION AND COMMISSIONING

The MERCEM monitor was delivered to the site by representatives from Aldora Technologies and Perkin Elmer and lifted into the mobile laboratory trailer located adjacent to the TSCA Incinerator. Connections to utilities, the extractive sampling line, and the return exhaust line, and startup of the monitor were easily accomplished in a two-day period. The analyzer system was drawing stack gas and measuring mercury concentrations by the end of the second day.

A major issue that arose during installation and commissioning was the potential for transient spikes of mercury to be released into the flue gas during the feeding of containerized solids in discrete charges introduced by the ram feeder system for the rotary kiln. Although the feed operating conditions were within hourly and daily limits established in regulatory approvals, the temporarily elevated stack concentrations on occasion over-ranged the MERCEM and required manual intervention for recovery and resumption of reliable unattended operation.

The first over-range incident occurred on Friday, September 11, 1998. A mercury spike from a containerized box of solid waste fed to the rotary kiln exceeded the set range of 300 $\mu g/m^3$. The effects from this incident were minimal, however, and the monitor was left in automatic sampling mode overnight. Another over-range event occurred on the morning of Monday, September 14, at approximately 07:45. The monitor was found in standby after the over-range incident, and the monitor and sampling system were saturated with mercury. Residual mercury raised the background level, requiring flushing of the system in purge mode and manual disassembling and cleaning of as many internal parts as possible. Residual mercury contamination in the instrumentation located inside the analyzer cabinet was successfully reduced to normal background levels by 22:00 that day when the daily calibration and ZD tests were conducted. A decision was made to operate only with liquid waste during the Phase I RA test.

5.3 INTERPRETATION OF TEST RESULTS

The interpretation of test results requiring comparison of the MERCEM response with reference calibration standards was complicated by uncertainty in reference values for mercury calibration gas. Compressed mercury vapor from six gas cylinders was used as reference material during the test program. Although the concept of using a compressed mercury vapor calibration standard appears feasible, the methodology for manufacturing mercury calibration gas standards is still under development and has not yet been perfected. Evaluation of the test results was further complicated by a limited number of gas cylinders of known concentration. Of the six cylinders available for the test, only two underwent a pre-test wet chemistry verification analysis to determine the actual mercury vapor concentration in the cylinder. A post-test verification was performed on the original two cylinders plus a third cylinder that was not available during Phase I testing. Thus, some tests were conducted using non-verified gas cylinders to preserve sufficient quantities in previously verified cylinders to allow for post-test verification analysis. Where verification analysis data were available, the reference value used for comparison with the MERCEM was the average of the pre- and post-test verification analytical results. Otherwise, the average of all MERCEM measurements made on a particular cylinder during either the Phase I or Phase II test was used as the reference value for a non-verified mercury calibration gas cylinder. Verification results for the calibration gas cylinders are presented in Sect. 5.8.

Results from the TSCA Incinerator field evaluation of the MERCEM have been reported elsewhere prior to the publication of this report.[20–22] When the original data analysis was performed on the field test data, the EPA-proposed emission limit for mercury was 40 µg/dscm corrected to 7% O_2.[9] With the uncertainty in the final MACT emission standard for mercury and comments from federal regulators hinting that the final standard could be higher than previously proposed, an emission standard of 100 µg/dscm was assumed for purposes of evaluating data. Since the test results were previously reported, EPA issued the MACT standards and finalized the mercury emission standard at 130 µg/dscm for existing incinerators.[23] The data presented here have not been corrected to the new emission standard of 130 µg/dscm. Note that a higher emission standard would be less stringent for the MERCEM to pass.

5.4 PHASE I PERFORMANCE TEST RESULTS

5.4.1 Calibration Error Test

The Phase I calibration error test was conducted on September 15, 1998, by introducing the reference material—either ambient air or compressed mercury gas—into the sample line between the

probe outlet and the sample line inlet. This allowed the sampling system downstream of the probe to be challenged with the reference material. Measurements were made at two rather than three measurement levels, as prescribed by PS12, due to volume of calibration gas availability. Two or three MERCEM measurements were made at each condition, and the results were averaged to provide the MERCEM response. Results of the first calibration error test are presented in Table 5.2.

Table 5.2. Phase I calibration error test results

Run No.	Condition	Reference value ($\mu g/m^3$)	MERCEM response ($\mu g/m^3$)	Difference ($\mu g/m^3$)	% of criterion (absolute)
1	Hg – zero	0	4.03	4.03	4.03
3	Hg – zero	0	5.23	5.23	5.23
5	Hg – zero	0	5.01	5.01	5.01
2	Hg – high	78.1	77.0	-1.1	-1.4
4	Hg – high	78.1	78.5	0.4	0.5
6	Hg – high	78.1	78.8	0.7	0.9

The difference between the reference value and the MERCEM for the zero response was calculated assuming an emission limit value for mercury of 100 µg/dscm. The reference value for ambient air was assumed to be zero. In absence of a verified concentration for cylinder CC90848, the reference value used for the gas cylinder was the average value of the MERCEM responses from the calibration error test at the high condition. A more defensible assignment of the non-verified reference gas cylinder concentration would have been to measure the mercury cylinder concentration locally at the MERCEM analyzer and use the local measurement as the reference value. (Note: This was done in the Phase II calibration error test.)

The MERCEM response, while measuring ambient concentrations of mercury, were unusually high: between 4 and 5 $\mu g/m^3$, as compared to ambient measurements made for the ZD test. The cause of the higher readings is thought to be due primarily to residual mercury contamination in the sample line from the saturation event that morning. Zero points during the calibration error test were made on ambient air drawn from the stack sampling platform through the sample line, whereas ZD test measurements were made while sampling "local" ambient air from inside the mobile laboratory trailer where the analyzer was located. During the course of the testing, the analyzer was zeroed on both local ambient air and nitrogen with no measurable difference. Another explanation of the elevated zero readings during the calibration error test could be that the ambient mercury concentration at the stack sampling platform may have been above detectable levels.

The first calibration error test was done in part as a "shake-out" test and also to establish the mechanics and timing requirements of regulator changes and response times and to monitor the analyzer's recovery from the saturation event. Limiting problems resulted when sustained lower-temperature ambient air was run through the sample line for more than 30 minutes at a time. This caused sample line temperatures to drop below the set point of 185°C, at which time testing was paused with no flow for the line to recover temperature.

5.4.2 Calibration and Zero Drift (ZD) Test

Results from the first calibration and ZD tests are summarized in Table 5.3, both without and with a response factor of 0.87 applied to the MERCEM output. Missing results were not reported. The MERCEM results were derived from one, two, or three measurements of the reference material where multiple measurements were reported as average values. The reference material included local ambient air from inside the laboratory trailer for the ZD calculation and compressed mercury vapor in cylinders for the mid- and high-range drift tests. The gas cylinder concentrations in cylinders CC90913 and CC90843 were verified by pre- and post-test analysis at 47.2 $\mu g/m^3$ and 72.9 $\mu g/m^3$, respectively.

Table 5.3. Phase I calibration and ZD test results using compressed mercury gas cylinders

Date	R_{CEM} ($\mu g/m^3$)	R_V ($\mu g/m^3$)	Diff ($\mu g/m^3$)	R_{CEM}*rf ($\mu g/m^3$)	Diff ($\mu g/m^3$)
09/11/98	0.17	0.0	0.17	0.15	0.15
	53.6	47.2	6.4	46.6	-0.6
	85.8	72.9	12.9	74.6	1.7
09/12/98	0.2	0.0	0.2	0.17	0.17
	53.7	47.2	6.5	46.7	-0.5
	85.0	72.9	12.1	74.0	1.1
09/13/98	0.4	0.0	0.4	0.35	0.35
	56.2	47.2	9.0	48.9	1.7
	87.5	72.9	14.6	76.1	3.2
09/14/98	0.1	0.0	0.1	0.09	0.09
	52.9	47.2	5.7	46.0	-1.2
	82.2	72.9	9.3	71.5	-1.4
09/15/98	-0.1	0.0	-0.1	-0.09	-0.09
	NA[a]	47.2	NA	NA	NA
	NA	72.9	NA	NA	NA
09/16/98	0.6	0.0	0.6	0.52	0.52
	52.0	47.2	4.8	45.2	-2.0
	81.4	72.9	8.5	70.8	-2.1
09/17/98	1.5	0.0	1.5	1.3	1.3
	53.2	47.2	6.0	46.3	-0.9
	82.1	72.9	9.2	71.4	-1.5
09/18/98	NA	0.0	NA	NA	NA
	NA	47.2	NA	NA	NA
	85.0	72.9	12.1	74.0	1.1

[a] NA = Measurements were not made.

The ZD results easily meet the PS12 criterion at the assumed emission standard of 100 $\mu g/m^3$ or at the less stringent MACT standard of 130 $\mu g/m^3$ without applying the response factor to the MERCEM measurements. The high-level calibration drift is outside of the 10% of emission limit (+/-10 $\mu g/m^3$) at the assumed limit of 100 $\mu g/m^3$ in several cases, and is also outside of the acceptable tolerance limits in one case at 10% or the MACT emission limit (+/-13 $\mu g/m^3$). If the response factor is applied to the data, however, the calibration drift results all fall well within either tolerance range.

The permeation tube test results for the same period are contained in Table 5.4. Without exception, the MERCEM mercury concentration readings were from 1 to 11 $\mu g/m^3$, higher than the calculated permeation tube calculated values.

Table 5.4. Phase I calibration and ZD test results using mercury permeation tube apparatus

Date	R_{CEM} ($\mu g/m^3$)	R_V ($\mu g/m^3$)	Diff ($\mu g/m^3$)	R_{CEM}*rf ($\mu g/m^3$)	Diff ($\mu g/m^3$)
09/11/98	72.5	61.6	10.9	63.1	1.5
09/12/98	66.9	60.4	6.5	58.2	-2.2
09/13/98	110.6	99.3	11.3	96.2	-3.1
09/14/98	99.6	97.8	1.8	86.7	-11.1
09/15/98	106.8	103.5	3.3	92.9	-10.6
09/16/98	100.9	98.3	2.6	87.8	-10.5
09/17/98	104.1	98.5	5.8	90.6	-7.7
09/18/98	94.3	97.2	2.9	84.6	-9.7

The reference mercury concentration generated by the permeation tube apparatus took a leap from ~60 $\mu g/m^3$ to 100 $\mu g/m^3$ between September 12 and 13, 1998. This may be explained by the method used here to arrive at the permeation tube concentrations. These calculated values are the result of using the VICI certified vial diffusion rate of 50 ng/min at 50°C, the MERCEM discrete sample volume from the integrated mass flow meter, and the selected loading time constant of 10 sec. The MERCEM discrete sample volume was observed to decrease from ~130 mL to 80 mL. A small gas diaphragm pump, which draws a slip stream of ~35 L/h from the larger sampling pump flow rate of 1000 L/h, also influences the volume measurement. The diaphragm pump was rebuilt in the course of the saturation incident and was replaced between Phase I and Phase II.

5.4.3 Relative Accuracy (RA) Test

Test dates and times for the Phase I RA test are presented in Table 5.5.

Table 5.5. Test dates and times for Phase I RA test

Day	Run No.	Start time	Stop time
09/16/98	1	13:00 14:20 14:50	13:05 14:30 15:35
	2	16:12	17:12
	3	17:56	18:56
09/17/98	4	08:31	09:31
	5	09:52	10:52
	6	11:18	12:18
	7	12:45	13:45
	8	14:54	15:54
	9	16:54	17:54

During the Phase I RA test, the incinerator was operated only with liquid wastes being fed to the secondary and aqueous waste feed systems. An analysis of the waste feeds is presented in Table 5.6.

Table 5.6. Waste feed analysis for Phase I RA test

Parameter	Units	SCC organic liquid	Aqueous feed
Density	g/mL	0.899	1.05
Viscosity	cP	27.1	1.45
Heating value	Btu/lb	14256	172
Ash content	wt %	0.556	3.87
PCB	µg/g	2400	267
Organic chlorine	wt %	0.892	0.376
Organic fluorine	wt %	0.0861	0.0618
Sulfur	wt %	0.126	0.317
Mercury	µg/g	2.70	1.79
Alpha activity	pCi/g	330 ± 28	139 ± 29
Ba-133	pCi/g	NR[a]	1.08 ± 0.20
Am-241	pCi/g	0.186 ± 0.057	20.1 ± 1.6
Beta activity	pCi/g	327 ± 23	151 ± 30
C-14	pCi/g	0.115 ± 3.497	10.2 ± 25.0
Cs-134	pCi/g	-0.035 ± 0.071	-1.15 ± 0.24
Cs-137	pCi/g	0.023 ± 0.151	4.33 ± 0.35
Co-57	pCi/g	0.01 ± 0.03	0.33 ± 0.08
Co-60	pCi/g	0.011 ± 0.101	0.084 ± 0.210
Np-237	pCi/g	0.306 ± 0.136	0.219 ± 0.150
Nb-95	pCi/g	0.084 ± 0.079	NR
Pu-238	pCi/g	0.073 ± 0.134	0.392 ± 0.200
Pu-239	pCi/g	0.067 ± 0.090	0.884 ± 0.250
K-40	pCi/g	NR	19.4 ± 4.3
Pa-234m	pCi/g	135 ± 23	162 ± 22
Sr-90	pCi/g	-2.00 ± 5.02	8.51 ± 12.00
Tc-99	pCi/g	67.1 ± 17.8	29.7 ± 22.0
Th-228	pCi/g	1.09 ± 0.31	0.36 ± 0.21
Th-230	pCi/g	0.49 ± 0.16	3.73 ± 0.68
Th-232	pCi/g	0.034 ± 0.045	0.031 ± 0.063
Th-234	pCi/g	93.6 ± 1.57	38.5 ± 2.1
Total activity	pCi/g	554 ± 8	372 ± 13
H-3	pCi/g	211 ± 72	709 ±100
Uranium alpha activity	pCi/g	274 ± 5	68.4 ± 3.0
Uranium concentration	µg/g	773	116

[a] NR = Not reported.

Incinerator operating conditions for the Phase I RA test are summarized in Table 5.7.

Table 5.7. Incinerator process parameters during Phase I RA test

Parameter	Units	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	Run 8	Run 9
Kiln temperature	°F	1835	1836	1907	1851	1850	1850	1851	1850	1851
SCC temperature	°F	2235	2237	2237	2237	2237	2238	2237	2236	2237
Aqueous waste feed rate	lb/h	318	331	331	351	351	351	351	351	351
SCC waste feed rate	lb/h	301	309	315	340	332	330	329	308	39
Kiln auxiliary natural gas	Scfh	10685	10250	11693	9770	9651	9787	9410	9760	9479
SCC auxiliary natural gas	Scfh	6870	7078	6263	6650	6514	6458	6445	6557	6041
Kiln face pressure	in H_2O	-0.76	-0.73	-0.75	-0.65	-0.60	-0.60	-0.63	-0.62	-0.61
Quench temperature	°F	184	184	184	185	184	184	184	184	184
Quench recycle flow	gpm	136	142	135	145	137	131	127	132	129
Venturi pressure differential	in H_2O	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0
Venturi recycle flow	gpm	154	153	154	152	153	155	156	155	155
Packed bed recycle flow	gpm	180	181	181	181	180	181	180	180	180
Packed bed pH	PH	6.7	6.7	6.8	6.7	6.8	6.7	6.7	6.7	6.8
IWS #1 recycle flow	gpm	470	471	470	471	470	470	470	470	469
IWS #2 recycle flow	gpm	491	491	492	490	493	489	488	489	489
IWS #1 voltage	kV	27.5	27.6	28.0	29.3	29.4	26.2	28.3	28.4	28.1
IWS #2 voltage	kV	24.9	25.9	24.1	26.4	22.1	25.1	26.2	24.5	25.8
Quench blowdown	gpm	18.8	20.1	20.3	20.3	21.1	21.0	21.8	21.6	23.5
Quench blowdown conductivity	µmho/cm	1018	1720	1796	1775	1839	1750	1515	1479	1552
IWS blowdown	gpm	1.9	1.3	1.7	0.4	2.4	1.7	1.8	2.0	2.0
IWS blowdown conductivity	µmho/cm	8158	8859	8878	9963	10000	10000	10000	9957	10000
Combustion gas velocity	ft/s	18.3	18.1	18.1	17.6	17.3	17.3	17.1	17.2	16.7
Stack gas CO CEM	ppmv	3.9	3.2	3.3	3.5	2.6	3.3	3.0	3.1	3.0
Stack gas O_2 CEM	vol %	8.6	8.5	8.1	8.5	8.4	8.4	8.5	8.5	8.6
Stack gas CO_2 CEM	vol %	6.9	6.9	7.1	7.0	7.0	7.0	6.9	6.8	6.9

Results from the Phase I RA test are summarized in Table 5.8. The results were corrected to 7% O_2 using Orsat data from the reference method by adjustment using the following relationship:

$$P_c = P_m \times \frac{14}{21 - \%O_2},$$

where

P_c = corrected concentration,
P_m = measured concentration,
% O_2 = oxygen concentration, % by volume, dry basis.

Run 1 was started and stopped twice before finishing, and Run 9 was also known to have unsteady feed operation. The O_2 concentrations determined by Orsat analysis by the stack testers in Runs 1 and 9 approached 11% vs. 8–9% for the other seven runs. The Orsat analysis appeared higher for O_2 in Run 9 than the facility O_2 CEM.

Table 5.8. Phase I RA test results

Run No.	RM 101A (µg/dscm)	MERCEM (µg/dscm)		Difference (µg/dscm)
1	59.1	64.6		5.5
2	46.9	53.0		6.1
3	51.5	54.6		3.1
4	61.2	69.0		7.8
5	63.4	70.6		7.2
6	62.0	70.5		8.5
7	49.1	60.2		11.1
8	48.0	60.1		12.1
9	66.8	82.6		15.8
Statistical Analysis				
Mean	56.44444	65.02222	**|MD|**	8.577778
SD	7.55763	9.265093	**SD**	3.857712
% RSD	13.3895	14.24912	**CC**	2.965302
% RSD ratio	1.064201		**|MD| + CC**	11.54308
F_{exp}	1.502894		**|MD|/CC**	2.892716
MD/RM 101A	15.19685		**n**	9
N	9	9	**t**	2.306006
F_{crit}	3.438103			
Response Factor	0.87			
% RA compared to RM mean				20.45034
%RA compare to MACT emission standard				8.879292

For the Phase I RA test, the MERCEM demonstrated an RA of 20% compared to the reference method and met the RA criterion without applying any correction factor to the factory calibration. PS12 also defines an alternative RA criterion where RA must be no greater than 10% when the applicable emission standard is substituted for the average RM value. If the final mercury emission standard from the MACT rule of 130 μg/dscm is applied, then the Phase I RATA would pass the 10% criterion at 8.9% RA, again without the use of a correction factor.

5.4.4 Additional Precision and Bias Criteria

For precision, the relative standard deviation ratio of the candidate technology to that of the reference method was used to evaluate if the candidate was more precise. Here the MERCEM has a ratio of 1.06 vs. 1.0.

As an additional precision criteria, the MERCEM data set displays a percent relative standard deviation <50% (14%) and a standard deviation <50 $\mu g/m^3$ (actually 9.3).

An F-test reveals that the MERCEM is not statistically more imprecise than the reference method.

For bias, the mean difference between the MERCEM and reference method paired data points is greater than the confidence coefficient, but the absolute difference is an acceptable percentage (50%) of the average reference method data and <50 $\mu g/ m^3$ (actually 15% and 8.6 $\mu g/ m^3$).

5.5 OPERATIONAL TEST RESULTS

The MERCEM remained installed and was collecting data intermittently during the four weeks between each performance test to provide a qualitative assessment of long-term operational issues.

To minimize the effects of an over-ranging incident and possible saturation of the MERCEM from a transient mercury spike, both programmable controls and administrative controls were enacted to switch the MERCEM to standby mode. The programmed threshold value of 300 $\mu g/m^3$, which automatically placed the MERCEM in standby in the event of a significant excursion, was activated several times during the operational test period. Administrative controls were also enacted to prevent feeding mercury at a rate that might exceed the 300 $\mu g/m^3$ threshold in the stack during operation of the MERCEM. Each burn sheet, containing constituent concentrations and feed rate limits for each waste stream, was reviewed to determine the possible impact on mercury emissions. Conservative control limits for the mercury concentration in solid waste and the collective mercury feed rate for all liquid waste streams were established at ~1 μg/g and 0.01 lb/h, respectively, to prevent operation of the MERCEM during possible momentary excursions exceeding 300 $\mu g/m^3$. Prior to initiating waste feeds on a new burn sheet, the Shift Supervisor placed the MERCEM in either operating mode or standby mode based on the established mercury limits. There were significant periods during which the concentration of mercury in solid or liquid wastes being fed to the incinerator was greater than the conservatively established control points, and the MERCEM was manually placed in standby mode.

In the process of optimizing MERCEM operation, intervention by the TSCA Incinerator staff was required on several occasions to troubleshoot problems encountered with the monitor. The first intervention came on Tuesday, September 22, 1998, when a "Liquid Flow Alarm (D122)" was encountered the week after the Phase I RA test. Upon inspection, the problem was identified in the analyzer cabinet as a failure to reconnect the power strip to the peristaltic pump rack, which controls

flow of new reagent into and condensate out of the reactor chamber. At the conclusion of the Phase I testing, the MERCEM underwent light maintenance in preparation for interim operation of the system, and the power strip to the peristaltic pump rack was inadvertently not reconnected. Over the weekend, condensate from the stack sample gas slowly increased the liquid level in the reactor until the liquid alarm sensor detected the presence of liquid and placed the monitor in standby mode. The liquid alarm sensor was removed and cleaned, the peristaltic pump power strip was reconnected, and the monitor was returned to operating mode.

Adjustments to the sampling probe blowback function with instrument air were refined as a result of the site-specific operating experience. On Wednesday, September 23, 1998, the monitor was found in standby mode with a text alarm "Flow Caution During Loading (R8)" engaged and time-stamped on the MERCEM LCD display screen. Upon consultation with Aldora Technologies, the decision was made to replace the M2 sampling pump with a new identical pump on hand shipped from Perkin Elmer for use as a spare. Replacement of the M2 pump; however, this action did not alleviate the alarm condition. Upon further investigation, the bypass flowmeter was found to be reading low and blockage of the coarse sintered metal filter in the sampling probe due to buildup of particulate matter was suspected. This observation was corroborated by an unusually high particulate buildup that occurred during the same time period on the filter in the plant National Emission Standards for Hazardous Air Pollutants (NESHAPS) continuous sampling system for radionuclides. The control logic of the MERCEM PLC was modified to enable the blowback function to flush the sampling probe with instrument air. This cleared the blockage and the bypass flow returned to normal. After the sampling probe was flushed, a gradual decrease in the bypass line flow was observed again over the next 24 hours. This time the blowback function was set to automatically flush the sampling probe at a 1-h frequency. After the automatic blowback function was enabled, no further reductions in bypass flow were observed during the test program.

The Aldora Technologies field engineer made a one-day visit during the operational test period on October 8, 1998, to inspect the monitor and run calibrations with the calibration gas cylinders. Table 5.9 summarizes the results of the measurements.

After the system was optimized during this first week, operational testing and controls were established to prevent over-ranging the monitor while feeding containerized solid waste, the MERCEM ran smoothly without any other unexplained problems or interruptions. The most significant system weakness observed during the operational test period was the monitor's inability to deal with the irregular nature, both in terms of frequency and magnitude of mercury emissions, while feeding heterogeneous solid wastes. Although the MERCEM is a continuous sampling device, the actual collection period is only 10 sec out of a 3-minute measurement cycle. As a result, the monitor "sees" just over 5% of the total emissions and reports the emission profile based on a partial sample. This mode of operation is sufficient when feeding a homogeneous waste stream that produces a fairly steady stack concentration as demonstrated in the Phase I RA test. The problem of characterizing the flue gas emissions, however, becomes more challenging for a monitor when transients of varying magnitudes are introduced by way of a heterogeneous waste mixture. In the time allotted for this test program, there were not adequate resources to identify a permanent solution for dealing with the transient spikes of mercury from the solid waste charges. This issue must be addressed in the future, however, if the MERCEM is to be considered for deployment on a thermal treatment unit processing heterogeneous waste. A possible design modification to the MERCEM for handling transient swings in mercury emissions was tested during the Phase II test and is discussed in Section 5.6.4.

Table 5.9. Operational test calibration check results

Reference material	Reference value ($\mu g/m^3$)	MERCEM response ($\mu g/m^3$)
Ambient air	0.0	-0.19
Ambient air	0.0	-0.13
CC90848	NA[a]	84.0
CC90848	NA	87.2
CC90909	NA	58.8
CC90909	NA	59.4
CC90843	72.9	82.2
CC90843	72.9	90.5
CC90843	72.9	91.9
CC90913	47.2	60.3
CC90913	47.2	59.7
Ambient air	0.0	0.2
Ambient air	0.0	0.1

a NA = Measurements were not made.

5.6 PHASE II PERFORMANCE TEST RESULTS

5.6.1 Interference Response Test

According to PS12, percent interference is calculated as a difference in response while measuring a reference concentration with and without the interference gas present relative to an emission limit value. With uncertainty in establishing a reference value, it may be more convenient to redefine percent interference more simply as a percent change in response while measuring a level of mercury not intentionally varied. This approach is consistent with reporting of data from the EPA Hg CEMS demonstration.[24]

To meet the draft PS12, the required concentrations of interference gases, including water, must be introduced through the CEM with and without a nominal high-level concentration of mercury calibration gas to determine any positive or negative interferences. To accomplish this combining of cylinder gases, a factory Perkin Elmer gas blending apparatus was used. This device with an integrated moisture steamer consisted of three selectable mass flow meters (1200, 300, 100 L/h), isolation valves, and temperature controllers to accomplish the blending of dry and moist (blended with stream) calibration gases. The interconnecting tubing and fittings are all 316 stainless steel throughout the unit. This unit is used to field calibrate Perkin Elmer "hot wet" HCl CEMs and is equipped with a 3-m section of sample line that is connected from the field calibrator unit to the MERCEM sampling pump and controlled to 185°C. The MERCEM M1 sample pump operates at a nominal 1000 L/h of which only 35 L/h is passed through the divider capillary and drawn by the M2 pump through the reactor, cooler and ultimately the CVAAS unit.

During the interference response test, the M1 pump was switched off and the field calibrator combined flowrate of 300 L/h was delivered to the MERCEM. The blending of the mercury and interference gases was at a 50:50 ratio with each mass flow controller set at 150 L/h. For this reason, the interference gas cylinders contained twice the specified concentrations called for in the PS12 protocol. Cylinder CC90848 of elemental mercury calibration gas was used as the source for mercury. This cylinder was a non-certified cylinder with an indicated concentration of 8 ppbv and nominally read 88 $\mu g/m^3$ on the MERCEM over the two-month test program.

To begin the test, the analyzer zero was verified on plant nitrogen. The mercury calibration gas was then connected and the 50:50 blend of nitrogen zero gas and mercury calibration gas was tested. A baseline reading of 44.4 $\mu g/m^3$ was recorded over three MERCEM sample cycles (9 minutes total). Alternatively, interference gases with the mercury gas and nitrogen with mercury gas were delivered to the MERCEM and the analyzer responses recorded upon stabilizing. Due to the limitation of mercury calibration gas, single iterations for each gas were conducted. The water interference test was conducted through the steamer unit with resulting moisture content of 24.5%.

Interference response testing was conducted on October 24, 1998. Results of the interference response testing of the MERCEM are presented in Table 5.10. The sum of the interferences totaled 7.04% and met the interference response test criterion of <10%.

Table 5.10. Interference response test results

Carrier gas	MERCEM response ($\mu g/m^3$)	Difference ($\mu g/m^3$)	Difference (%)	Absolute difference (%)
N_2/CO_2	44.4/44.5	0.1	0.225	0.225
N_2/CO	44.4/44.2	-0.2	-0.450	0.450
N_2/O_2	44.4/44.2	-0.2	-0.450	0.450
N_2/SO_2	44.4/44.3	-0.1	-0.225	0.225
N_2/NO_2	45.4/45.7	0.3	0.661	0.661
N_2/Cl_2	45.4/44.9	-0.5	-1.101	1.101
N_2/HCl	45.4/44.4	-1.0	-2.203	2.203
N_2/H_2O	46.4/47.2	0.8	1.724	1.724
Total response				**7.040**

5.6.2 Calibration Error Test

The Phase II calibration error test was performed on October 20, 1998. The results, presented in Table 5.11, assume an emission limit value for mercury of 100 $\mu g/m^3$ for comparison of zero gas response. Calibration gas cylinders CC90848 and CC94785 were used, respectively, to challenge the entire sampling and analyzer system at the mid- and high-ranges. As the concentration of gas in these cylinders was not verified, the reference values used were the average of the MERCEM measurements made locally at the analyzer on the day of the calibration error test. Local measurements were made in the morning at approximately 9:00 a.m. by sampling from the cylinders at the analyzer interface, and later in the day the cylinders were relocated to the sampling platform for the calibration error test. The calibration error test measurements were made between 4:30 p.m. and 6:30 p.m. The test confirmed the integrity of the entire sampling system. All readings were within the PS12 criteria of ±15% absolute.

Table 5.11. Phase II calibration error test results

Run No.	Condition	Reference value (μg/m³)	MERCEM response (μg/m³)	Difference (μg/m³)	% of criterion (absolute)
3	Hg – zero	0.0	2.2	2.2	2.20
6	Hg – zero	0.0	1.8	1.8	1.80
8	Hg – zero	0.0	2.45	2.45	2.45
2	Hg – mid	89.9	91.1	1.2	1.33
4	Hg – mid	89.9	91.6	1.7	1.89
9	Hg – mid	89.9	90.5	0.6	0.67
1	Hg – high	317	310	-7	-2.21
5	Hg – high	317	317	0	0.00
7	Hg – high	317	313	-4	-1.26

5.6.3 Calibration and ZD Test

The calibration and ZD test using calibration gas cylinders CC90913 and CC90843, respectively, for the mid- and high-ranges was repeated in Phase II. Cylinder CC94705 that was post-certified at a concentration of 26.1 μg/m^3, was also used as a low-range reference. Because of limited quantities of calibration gases, however, a full set of calibration and ZD tests was not conducted to preserve sufficient quantities in the previously used cylinders in Phase I for post-test wet chemistry verification analysis. ZD testing was done while sampling local ambient air in the laboratory trailer. The MERCEM response is the average of two measurements made of the mercury vapor concentration in the reference gas. Results of the calibration and ZD test are presented in Table 5.12.

Table 5.12. Phase II calibration and ZD test results using compressed mercury gas cylinders

Date	R_{CEM} (μg/m³)	R_V (μg/m³)	Diff (μg/m³)	R_{CEM}*rf (μg/m³)	Diff (μg/m³)
10/18/98	-0.16	0.0	-0.16	-0.14	-0.14
	NA[a]	26.1	NA	NA	NA
	60.0	47.2	12.8	52.2	5.0
	91.2	72.9	18.3	79.3	6.4
10/19/98	-0.42	0.0	-0.42	-0.37	-0.37
	30.2	26.1	4.1	26.3	0.2
	58.0	47.2	10.8	50.5	3.3
	89.4	72.9	16.5	77.8	4.9
10/20/98	0.13	0.0	0.13	0.11	0.11
	31.0	26.1	4.9	27.0	0.9
	60.4	47.2	13.2	52.6	5.4
	93.5	72.9	20.6	81.3	8.4
10/21/98	-0.2	0.0	-0.2	-0.17	-0.17
	32.2	26.1	6.1	28.0	1.9
	60.2	47.2	13.0	52.4	5.2
	92.8	72.9	19.9	80.7	7.8

Table 5.12 (continued)

Date	R_{CEM} ($\mu g/m^3$)	R_V ($\mu g/m^3$)	Diff ($\mu g/m^3$)	R_{CEM}*rf ($\mu g/m^3$)	Diff ($\mu g/m^3$)
10/22/98	-0.15	0.0	-0.15	-0.13	-0.13
	31.8	26.1	5.7	27.7	1.6
	59.2	47.2	12.0	51.5	4.3
	93.1	72.9	20.2	81.0	8.1
10/23/98	0.02	0.0	0.02	0.01	0.01
	31.8	26.1	5.7	27.7	1.6
	61.1	47.2	13.9	53.2	6.0
	90.8	72.9	17.9	79.0	6.1

[a] NA = Measurements were not made.

As in Phase I, the Phase II ZD results easily meet the PS12 criterion at either the assumed emission standard of 100 $\mu g/m^3$ or at the less stringent MACT standard of 130 $\mu g/m^3$ without application of the response factor. The majority of the mid- and high-level calibration drift measurements, however, are outside of the 10% of the assumed emission limit (+/-10 $\mu g/m^3$) and in several cases are beyond 10% of the MACT emission limit (+/-13 $\mu g/m^3$). Applying the response factor to the MERCEM data shifts all of the calibration drift results to an acceptable difference at either emission standard.

The calibration drift test results with the permeation tube apparatus are presented in Table 5.13.

Table 5.13. Phase II calibration and ZD test results using permeation tube apparatus

Date	R_{CEM} ($\mu g/m^3$)	R_v ($\mu g/m^3$)	Diff ($\mu g/m^3$)	R_{CEM}*rf ($\mu g/m^3$)	Diff ($\mu g/m^3$)
10/18/98	86.0	75.2	10.8	74.8	-0.4
10/19/98	77.9	67.1	10.8	67.8	0.7
10/20/98	76.2	67.1	9.1	66.3	-0.8
10/21/98	77.2	68.0	9.2	67.2	-0.8
10/22/98	74.8	66.4	8.4	65.1	-1.3
10/23/98	76.0	64.5	11.5	66.1	1.6

5.6.4 Relative Accuracy Test

Test dates and times for the Phase II RA test are presented in Table 5.14.

Table 5.14. Test dates and times for Phase II RA test

Day	Run No.	Start time	Stop time
10/22/98	1	10:12 &11:33	10:52 & 11:53
10/22/98	2	12:20	13:20
10/22/98	3	14:01	15:01
10/22/98	4	15:32	16:32
10/22/98	5	17:53	18:53
10/22/98	6	19:23	20:23
10/23/98	7	08:23	09:23
10/23/98	8	09:49 & 11:00	10:25 & 11:25
10/23/98	9	13:03	14:03

During the Phase II RA test, the incinerator was operated with organic liquid waste and containerized solids being fed to the rotary kiln. An analysis of the waste feeds is presented in Table 5.15.

Table 5.15. Waste feed analysis for Phase II RA test

Parameter	Units	Primary combustion chamber organic liquid	Bulk solids
Density	g/mL	0.964	0.733
Viscosity	cP	2.73	NA[a]
Heating value	Btu/lb	7759	4179
Ash content	wt %	1.50	64.3
PCB	µg/g	8413	273
Organic chlorine	wt %	0.0096	0.019
Organic fluorine	wt %	0.299	0.012
Sulfur	wt %	0.0416	0.274
Mercury	µg/g	0.985	0.471
Alpha activity	pCi/g	212 ± 6	143 ± 15
Beta activity	pCi/g	268 ± 5	173 ± 7
C-14	pCi/g	-0.88 ± 8.25	NR[b]
Cs-134	pCi/g	-0.421 ± 0.391	NR
Cs-137	pCi/g	0.246 ± 0.384	0.27 ± 5.64
Pb-212	pCi/g	NR	3.6 ± 0.9
Np-237	pCi/g	0.089 ± 0.103	0.80 ± 1.09
Pu-238	pCi/g	0.061 ± 0.106	0.08 ± 0.07
Pu-239	pCi/g	0.058 ± 0.067	0.005 ± 0.028
Pa-234m	pCi/g	246 ± 50	245 ± 46
Sr-90	pCi/g	2.93 ± 0.52	NR
Tc-99	pCi/g	30.7 ± 15.3	0.264 ± 0.418

Table 5.15 (continued)

Parameter	Units	Primary combustion chamber organic liquid	Bulk solids
Tl-208	pCi/g	NR[a]	1.29 ± 0.06
Th-228	pCi/g	0.864 ± 0.241	0.023 ± 0.029
Th-230	pCi/g	0.473 ± 0.164	0.002 ± 0.014
Th-232	pCi/g	0.102 ± 0.074	0.008 ± 0.018
Th-234	pCi/g	135 ± 6	245 ± 46
Total activity	pCi/g	561 ± 9	604 ± 23
H-3	pCi/g	-62.2 ± 34.1	NR
Uranium alpha activity	pCi/g	206 ± 5	296 ± 48
Uranium concentration	µg/g	873	242

[a] NA = Not applicable.
[b] NR = Not reported.

Incinerator operating conditions for the Phase II RA test are summarized in Table 5.16.

Table 5.16. Incinerator process parameters during Phase II RA test

Parameter	Units	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	Run 8	Run 9
Kiln temperature	°F	1844	1848	1847	1918	1837	1837	1887	1858	1868
SCC temperature	°F	2232	2221	2222	2226	2222	2222	2222	2221	2228
PCC organic waste feed rate	lb/h	377	357	297	383	371	415	482	448	515
Bulk solids waste feed rate	lb/h	159	106	90	80	80	80	112	78	80
Kiln auxiliary natural gas	scfh	9878	11287	11619	10576	10653	11013	10803	9882	10230
SCC auxiliary natural gas	scfh	9077	9020	9665	8319	9633	9606	8885	9721	10085
Kiln face pressure	in H_2O	-0.63	-0.52	-0.49	-0.50	-0.63	-0.63	-0.61	-0.62	-0.59
Quench temperature	°F	185	185	185	185	185	185	185	185	185
Quench recycle flow	gpm	126	122	151	140	132	119	126	134	124
Venturi pressure differential	in H_2O	8.9	8.9	8.9	8.9	8.9	9.0	8.9	8.9	8.9
Venturi recycle flow	gpm	158	160	154	155	158	160	158	156	158
Packed bed recycle flow	gpm	175	174	174	174	174	174	174	174	174
Packed bed pH	pH	6.6	6.6	6.6	6.6	6.7	6.6	6.6	6.7	6.5
IWS #1 recycle flow	gpm	635	636	636	637	636	637	637	636	636
IWS #2 recycle flow	gpm	570	581	572	574	573	574	580	573	580
IWS #1 voltage	kV	26.3	27.5	27.1	29.4	27.5	29.8	29.4	23.3	29.4
IWS #2 voltage	kV	27.8	22.0	25.5	26.1	24.5	27.0	24.0	25.2	23.5
Quench blowdown	gpm	16.4	10.9	10.3	19.9	12.6	13.2	16.8	17.3	14.7
Quench blowdown conductivity	µmho/cm	4492	6605	6489	6953	4129	5964	3652	4110	3370
IWS blowdown	gpm	7.1	9.0	9.9	8.8	10.9	11.3	7.7	6.9	4.3
IWS blowdown conductivity	µmho/cm	2016	3189	4471	5050	4352	4681	2533	3010	2879
Combustion gas velocity	ft/s	19.0	20.2	20.2	17.6	20.0	19.9	19.0	18.5	19.2
Stack gas CO CEM	ppmv	83.4	14.4	1.6	1.6	47.5	0	22.8	88.0	5.9
Stack gas O_2 CEM	vol %	8.5	9.0	8.9	7.8	8.9	8.8	8.3	8.5	8.6
Stack gas CO_2 CEM	vol %	7.3	7.0	7.0	7.6	6.7	6.9	7.2	7.0	7.0

To minimize the potential for interruptions during the Phase II RA test, a 1.5-L vessel acting as a buffering accumulator was installed to buffer the effects of a mercury spike while feeding solid wastes. The mercury concentration in the solid waste feeds was much lower during the Phase II RA test than during previous testing with the MERCEM when over-ranging was observed. The effectiveness of the accumulator in buffering mercury spikes or, conversely, any impact on biasing the results could not be ascertained in this test. The concept is one that should be investigated, however, as a possible solution for dealing with momentary spikes of mercury when feeding heterogeneous wastes.

Results from the Phase II RA test are summarized in Table 5.17. Again, the results were corrected to 7% O_2 using Orsat data from the reference method.

Table 5.17. Phase II RA test results

Run No.	RM 101A (µg/dscm)	MERCEM (µg/dcsm)		Difference (µg/dscm)
1	6.7	24.3		17.6
2	9.8	22.0		12.2
3	4.5	20.4		15.9
4	2.8	19.2		16.4
5	5.7	20.7		15.0
6	3.6	19.9		16.3
7	1.9	19.9		18.0
8	4.4	21.5		17.1
9	6.4	20.5		14.1
Statistical analysis				
Mean	5.088889	20.93333	**|MD|**	15.84444
SD	2.380359	1.519046	**SD**	1.832424
% RSD	46.77562	7.256588	**CC**	1.408527
% RSD ratio	0.155136		**|MD| + CC**	17.25297
F_{exp}	0.407246		**|MD|/CC**	11.24895
MD/RM 101A	311.3537		**n**	9
n	9	9	**t**	2.306006
F_{crit}	3.438103			
% RA compared to RM mean				339.0322
% RA compared to MACT emission standard				13.27152
% RA compared to mean RM mean with response factor applied				284.9316
% RA compared to MACT emission standard with response factor applied				11.15373

For the Phase II RA test, the MERCEM demonstrated an RA of 339% compared to the reference method. If an emission standard for mercury as high as 173 µg/ dscm were proposed, the results of the second test would then meet the alternative criterion of 10% based on an emission standard as defined in PS12.

Considering the final MACT mercury standard of 130 µg/dscm and applying the response factor determined in the Phase I testing, the Phase II results look more promising but still do not pass the alternative 10% RA criteria by posting a value of 11.2%.

5.6.5 Additional Precision and Bias Criteria

For the second RA test, the MERCEM exhibited favorable performance against all of the alternative precision criteria but not the bias criteria.

5.7 MERCEM PERFORMANCE WITH PS12 CRITERIA AT MACT EMISSION LIMIT

With the finalization of the mercury emission limit for existing incinerators at 130 µg/dscm in the MACT rule, an assessment of the MERCEM performance with the PS12 criteria was done assuming the final MACT emission limit for mercury. The results of this assessment are summarized in Table 5.18.

Table 5.18. MERCEM performance with PS12 criteria at 130 µg/dscm emission limit

PS12 Criteria	Specification	Results
Phase I		
Calibration & Zero Drift CD[a] ZD	 ≤10% of emission standard ≤5% of emission standard	 Pass[b] Pass
Calibration Error[c,d]	≤15% of reference concentration	Pass
Response Time	Sampling time ≤1/3 of averaging period for the applicable standard **and** delay time for reporting analysis no greater than 1 h (Batch CEMS)	Pass
Interference Test	≤10% of emission standard	Test not done in Phase I
Relative Accuracy	≤20% of mean value of reference method test data **or** ≤10% of emission standard	Pass Pass
Phase II		
Calibration & Zero Drift CD[e] ZD	 ≤10% of emission standard ≤5% of emission standard	 Pass[b] Pass
Calibration Error[d]	≤15% of reference concentration	Pass
Response Time	Sampling time ≤1/3 of averaging period for the applicable standard **and** delay time for reporting analysis no greater than 1 h (Batch CEMS)	Pass

Table 5.18 (continued)		
PS12 Criteria	**Specification**	**Results**
Interference Test	≤10% of emission standard	Pass
Relative Accuracy	≤20% of mean value of reference method test data **or** ≤10% of emission standard	Fail[b] Fail[b]

[a] One mid-test span value not conducted – cylinder at stack.
[b] Results include site-specific response factor (rf).
[c] Mid-level calibration error not assessed due to calibration gas supply limitations.
[d] Only Hg(0) calibration gas utilized.
[e] Drift period duration 6 consecutive days, not 7 days.

Because of the limited quantities and concentrations of mercury calibration gases that were readily available at the time of the test, there were some portions of PS12 that were not completely evaluated verbatim with the performance specification as previously outlined in the test report. The majority of the specifications, however, were evaluated with satisfactory results. The MERCEM passed all elements of PS12 during the Phase I test, although the response factor had to be applied to the MERCEM calibration drift results to pass the calibration drift criterion.

The Phase II testing was more challenging than the Phase I test in terms of having to measure lower mercury concentrations present in the stack and as a result of perturbations in stack mercury concentrations caused by simultaneously feeding liquid and solid wastes to the incinerator. The MERCEM passed all of the PS12 criteria during Phase II except relative accuracy. Even after applying the response factor, the calculated relative accuracy of 11.2% fell just short of the 10% of emission standard criterion.

The results from this comparison of the MERCEM with PS12 show that the MERCEM demonstrates favorable performance against the PS12 criteria. The data do support the feasibility of monitoring mercury emissions with the MERCEM at a hazardous waste burning incinerator utilizing a wet gas cleaning system. At the same time, however, the results also point to the need for additional longer-term monitor testing and possible modifications to the monitor for handling transient emissions from treating heterogeneous waste streams, further testing of the stability of calibration gas standards at concentrations required by the MACT mercury emission limit, and agreement on the application of site-specific response factors to monitor measurements.

5.8 COMPARISON OF MERCEM AND RM 101A RESULTS WITH EMISSION CALCULATIONS

Mercury emissions during RA testing were estimated assuming zero system removal efficiency, average feed rates from the process data, and arithmetic mean concentrations of mercury from feed analytical data. For the Phase I RA test, three samples of the SCC feed contained 2.42, 3.31, and 2.38 µg/g of mercury. The aqueous feed sample mercury concentrations were 1.9, 1.75, and 1.71 µg/g. For the Phase II RA test, the rotary kiln organic liquid feed had results of 0.734, 1.33, and 0.89 µg/g mercury. Solids are fed based on the actual composition of representative samples obtained using an approved sampling plan and acceptance criteria. The concentration of mercury in the solids feed during the Phase II RA test was 0.471 µg/g. An estimate of the total stack flow had to be calculated since the reference method results were from a fixed single point not necessarily at the point of average stack

velocity. The calculated mercury emissions based on the assumptions listed above during the Phase I and Phase II RA tests are given in Tables 5.19 and 5.20. Also shown are the RM 101A and MERCEM results and the apparent mercury removal efficiency based on the RM 101A and the MERCEM data. The RM 101A Orsat results were used to correct the calculated emissions values to 7% O_2.

Table 5.19. Comparison of Phase I RA test results with calculated emissions

Run No.	RM 101A (µg/dscm)	MERCEM (µg/dcsm)	Calculated emissions (µg/dscm)	Apparent removal efficiency based on RM 101A (%)	Apparent removal efficiency based on MERCEM (%)
1	59.1	64.6	73.6	19.7	12.2
2	46.9	53.0	62.9	25.4	15.7
3	51.5	54.6	63.0	18.3	13.3
4	61.2	69.0	66.9	8.5	-3.1
5	63.4	70.6	71.4	11.2	1.1
6	62.0	70.5	75.1	17.4	6.1
7	49.1	60.2	65.8	25.4	8.5
8	48.0	60.1	66.7	28.0	9.9
9	66.8	82.6	83.1	19.6	0.6

Table 5.20. Comparison of Phase II RA test results with calculated emissions

Run No.	RM 101A (µg/dscm)	MERCEM (µg/dcsm)	Calculated emissions (µg/dscm)	Apparent removal efficiency based on RM 101A (%)	Apparent removal efficiency based on MERCEM (%)
1	6.7	24.3	19.5	65.6	-24.6
2	9.8	22.0	16.5	40.6	-33.3
3	4.5	20.4	16.6	72.9	-22.9
4	2.8	19.2	17.9	85.8	-7.3
5	5.7	20.7	17.0	66.5	-21.8
6	3.6	19.9	19.3	81.3	-3.1
7	1.9	19.9	21.4	91.1	7.0
8	4.4	21.5	20.4	78.4	-5.4
9	6.4	20.5	22.8	71.9	10.1

These results are indicative of the difficulty of quantifying the behavior of mercury through a combustion and gas scrubbing process. Looking at only the calculated emissions assuming total partitioning of mercury in the feed to the off-gas and the RM 101A results, the Phase I test predicts a mercury removal efficiency for the system between 8 and 28%. These results are more or less in agreement with the general assumption for mercury behavior in the TSCA Incinerator (i.e., zero removal efficiency for regulatory reporting purposes.) Data from the most recent state air test conducted in 1995 corroborate the zero removal efficiency assumption. Two test conditions—one with organic liquid and aqueous waste feeds to the kiln and the second with organic liquid, aqueous, and solid waste feeds to the kiln—were demonstrated in the state air test. In both conditions, the aqueous waste feed was spiked with a beryllium solution, a mercury solution, and lead nitrate to ensure that sufficient quantities of the metals were fed to the incinerator for determining removal efficiencies. Mercury feed concentrations in the organic liquid and aqueous waste feeds were 1.43 and 3.79 μg/g, respectively, for the liquids-feed-only condition. The liquids and solids feed condition saw mercury concentrations of 1.12, 4.40, and 0.08 μg/g, respectively, in the organic liquid, aqueous, and solid waste feeds. Three stack sampling runs were conducted for each of the two test conditions. Removal efficiencies for the six runs were 14.7% for the first liquid-feed-only run and 0% for the other five runs.[25]

The Phase II RA test results, on the other hand, tend to indicate a much higher removal efficiency for mercury between 40 and 90%. A non-regulatory test performed in the same year as the 1995 state air test, also reported elevated mercury removal efficiencies. This test was conducted while feeding organic waste to both the primary and secondary combustion chambers and solids all containing about 0.2 μg/g of mercury and aqueous waste with non-detectable mercury. The reported mercury concentrations were 2–3 $\mu g/m^3$, but the mercury removal efficiencies were reported as 68.3% and 84.7%.[26] If these and the Phase II RA reference method results are not accurate, then they are at least precise.

Including the MERCEM results in a discussion of the mercury emissions does not, unfortunately, greatly substantiate one position more than the other. It is interesting, however, that the calculated mercury emission values are amazingly close to the MERCEM results from the Phase II RA test. If the MERCEM values are correct, then the Phase II data may point to a low bias in RM 101A at low mercury concentrations. Other studies have found that results from Method 29, which uses the same permanganate reagent as Method 101A, were lower than anticipated for mercury when attempting to spike a flue gas with mercuric chloride in the range of 4–24 $\mu g/m^3$. Elemental mercury spiked into the same flue gas was recovered completely.[27] Perhaps mercury emissions in the few $\mu g/m^3$ range (<20), as observed during the Phase II RA test, are mercuric chloride and the RM 101A results are biased low. This would help to explain the large removal efficiencies calculated for Phase II RA as opposed to those calculated for Phase I. The presence of small amounts of mercuric chloride in the flue gas would have a greater effect on mercury removal calculations at lower total emissions than at higher emissions. Mercuric chloride, on the other hand, is not an expected constituent of the TSCA Incinerator flue gas because it would be scrubbed out of the gas in the wet scrubbers preceding the stack. No reference method data exists to quantify the presence of mercuric chloride in the TSCA Incinerator emissions.

From the data presented here, it is impossible to make any conclusions regarding the fate of mercury in the TSCA Incinerator flue gas. More data, including speciation, is needed to fully understand the behavior of mercury and the performance of manual methods and instrument methods for measuring mercury.

5.9 MERCURY VAPOR CALIBRATION GAS VERIFICATION RESULTS

Cylinders CC90843 and CC90913 were sent by Spectra Gases to EERC for sampling and analysis prior to use in the field evaluation in Oak Ridge. The pre-test measurements for cylinders CC90843 and CC90913 were made in duplicate using a full-scale EPA Method 101A (absorbing the mercury in a potassium permanganate solution) and using a Semtech continuous emission mercury analyzer (a CEM). The post-test measurements for the two previously verified cylinders and for cylinder CC94705 were done with a midget impinger set to ensure that there was enough gas in the cylinders to complete the measurement.[28] Results are summarized in Table 5.21. For each cylinder, the average of the four wet chemistry results was used to establish the reference concentration of that cylinder. Note that only the August 1998 verification results were available for decisions during the field testing.

Table 5.21. Calibration gas cylinder analyses by EERC

Cylinder No.	Target prepared conc. (ppbv)	Date	Measured Hg (µg/L)	Sample volume (mL)	Gas sampled (ft^3)	Time sampled (min)	Hg conc. ($\mu g/m^3$)	Hg conc. (ppbv)
CC90843	8	08/28/98	59.0	500	11.765	30	88.5	10.44
		08/31/98	40.2	500	10.904	30	65.1	7.67
		08/31/98	Semtech CEM	NA[a]	0.706	8	72.2	8.51
		02/16/99	60.92	100	3.0	60	70.7	8.33
		02/18/99	28.60	200	3.0	60	67.1	7.91
		02/18/99	Semtech CEM	NA	2.0	19	64.2	7.57
CC90913	6	08/28/98	37.5	500	12.606	30	52.5	6.19
		08/31/98	28.6	500	11.417	30	44.2	5.21
		08/31/98	Semtech CEM	NA	0.441	5	47.1	5.55
		02/16/99	42.2	100	3.1	60	48.7	5.73
		02/18/99	19.2	200	3.1	60	43.7	5.15
		02/18/99	Semtech CEM	NA	1.2	11	40.8	4.81
CC94705	3	02/16/99	21.7	100	3.1	60	25.1	2.96
		02/18/99	22.8	100	3.0	60	27.1	3.20
		02/18/99	Semtech CEM	NA	2.4	23	20.5	2.42

[a] NA = Measurements were not made.

A comparison of the target concentrations as provided by Spectra Gases and the verified results reported by EERC show very good agreement for all three bottles tested.

The more recent analytical results of the two cylinders previously analyzed do not significantly vary from the earlier results. There was a 5-month time difference between the two sets of analyses. Thus, the mercury concentration in the cylinders appears to be fairly stable over time.

The Jerome 431-X analyzer readings taken on calibration gas cylinders of known concentration and the permeation tube are presented in Table 5.22 for the period September 14–18, 1999. The average MERCEM measurements of the same reference standards are also shown for comparison. As seen from the table, readings from the Jerome 431-X analyzer were found to be sporadic and not useful for verification purposes, and the attempted verification effort was discontinued at the end of the Phase I test period.

Table 5.22. Jerome 431-X verification of mercury vapor reference materials

Reference material	Date	Reference material concentration ($\mu g/m^3$)	Average MERCEM reading ($\mu g/m^3$)	Jerome 431-X reading ($\mu g/m^3$)
CC90913	9/14/98	47.2	52.9	25
				25
	9/16/98		52.0	21
				22
				23
				21
	9/17/98		53.2	16
				17
CC90843	9/14/98	72.9	82.2	41
				41
	9/16/98		81.4	32
				32
				32
				32
	9/17/98		82.1	27
				26
				27
	9/18/98		85.0	66
				64
				66
Permeation tube	9/14/98	99.6	97.8	39
				39
				34
				56
				51
	9/16/98	100.9	98.3	70
				41
				39
				21
				22
				22
	9/17/98	104.1	98.5	12
				12
				13
	9/18/98	94.3	97.2	37
				37
				36
				36

Cylinders supplied by Spectra Gases as well as output from the permeation tube device were sampled and analyzed by LMES personnel after the second RA test. The results are shown in Table 5.23. The initial analytical results from the laboratory were all below reporting limits. A review of the data showed that each impinger train was analyzed with the permanganate and HCl submitted and analyzed as separate samples. A lower reporting limit of 0.04 μg was reported for the permanganate, but the reporting limit for the HCl was 1.2 μg. The permanganate had a dilution factor of 1, but the HCl had a dilution factor of 25. Reanalysis of the samples at a lower dilution factor produced the results in Table 5.23. These results reveal potential inaccuracy and imprecision from the verification procedure itself or the manner in which it was implemented with the small mercury levels and the small sample volumes probably a contributing factor. Note that the results in Table 5.23 were not used to adjust any measurements.

Table 5.23. Mercury calibration gas analyses by LMES

Source	Date	Measured Hg in sample (μg)	Gas sampled (m^3)	Time sampled (min)	Hg concentration ($\mu g/m^3$)
Perm tube	10/22/98	0.748	0.015319	29.7	48.8
CC90843	10/28/98	0.595	0.015128	33.7	39.3
CC90848	10/28/98	0.908	0.014995	30.8	60.6
CC90913	10/28/98	0.908	0.014956	30.2	60.7
CC94785	10/28/98	3.238	0.015362	32.7	210.8

6. CONCLUSIONS

1. The two-month evaluation of the MERCEM total mercury monitor from Perkin Elmer provided a useful venue in determining the feasibility of using a CEM to measure total mercury in a saturated flue gas.

2. The MERCEM exhibited potential at a mixed waste incinerator to meet requirements proposed in PS12 under conditions of operation with liquid feeds only at stack mercury concentrations in the range of proposed MACT standards.

3. Performance of the MERCEM under conditions of incinerating solid and liquid wastes simultaneously was less reliable than while feeding liquid feeds only for the operating conditions and configuration of the host facility.

4. The permeation tube calibration method used in this test relied on the CEM internal volumetric and time constants to relate back to a concentration, whereas a compressed gas cylinder concentration is totally independent of the analyzer mass flowmeter and flowrates.

5. Mercury concentration in the compressed gas cylinders was fairly stable over a 5-month period.

6. The reliability of available reference materials was not fully demonstrated without further evaluation of their incorporation into routine operating procedures performed by facility personnel.

7. The degree of mercury control occurring in the TSCA Incinerator off-gas cleaning system could not be quantified from the data collected in this study.

8. It was possible to conduct the demonstration at a facility incinerating radioactively contaminated wastes and to release the equipment for later unrestricted use elsewhere.

9. Experience gained by this testing answered additional site-specific and general questions regarding the operation and maintenance of CEMs and their use in compliance monitoring of total mercury emissions from hazardous waste incinerators.

7. RECOMMENDATIONS

1. Do more work on preparation and certification of mercury vapor calibration gas in cylinders.

2. In parallel, develop a permeation tube based reference standard that produces a reference concentration independent of any measurements made by the CEM.

3. After further work is done on compressed mercury gas cylinders and permeation tube calibration devices, a down-selection should be made based on a comparison of the two methods.

4. Evaluate methods for mitigating the effects of transient mercury spikes on the monitor response while feeding discrete charges of heterogeneous solid waste.

5. Conduct additional reference method testing at the TSCA Incinerator to determine the relative contribution of various mercury species in the flue gas.

6. Conduct longer term testing of a mercury monitor(s) to evaluate monitor performance and availability over an extended operating period.

7. Use a mercury monitor to evaluate process chemistry changes for improving control of mercury emissions from the TSCA Incinerator.

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28. Dennis L. Laudal, University of North Dakota, letter report to William G. Miller, Spectra Gases, "Spectra Gases, Inc., PO No. 68973 Analysis of Mercury Calibration Gas Cylinders," February 22, 1999.

APPENDIX A

FIELD TEST PHOTOGRAPHS

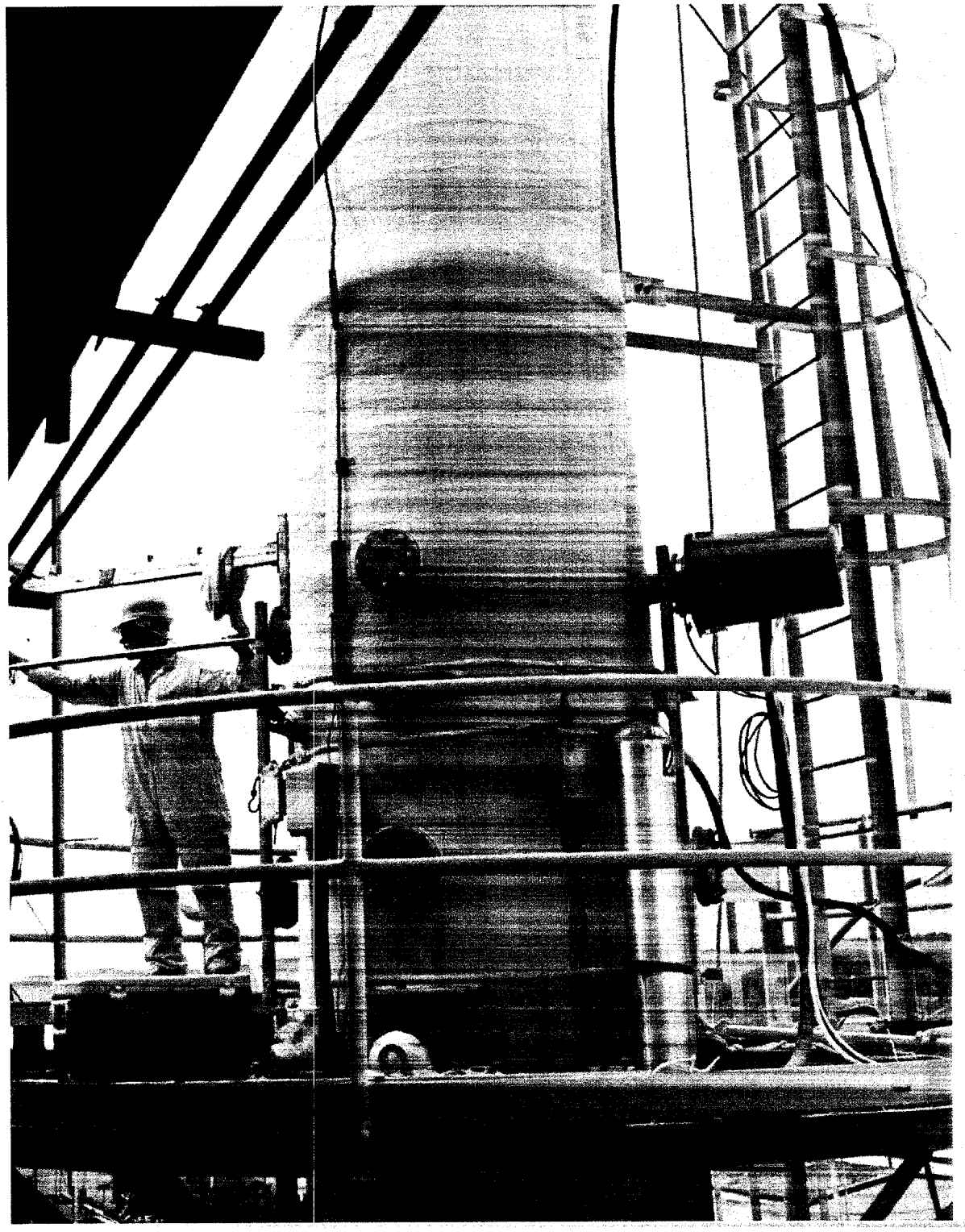

Fig. A.1. Reference method sampling train probe (left) and MERCEM sampling probe (right) installed on lower stack sampling platform.

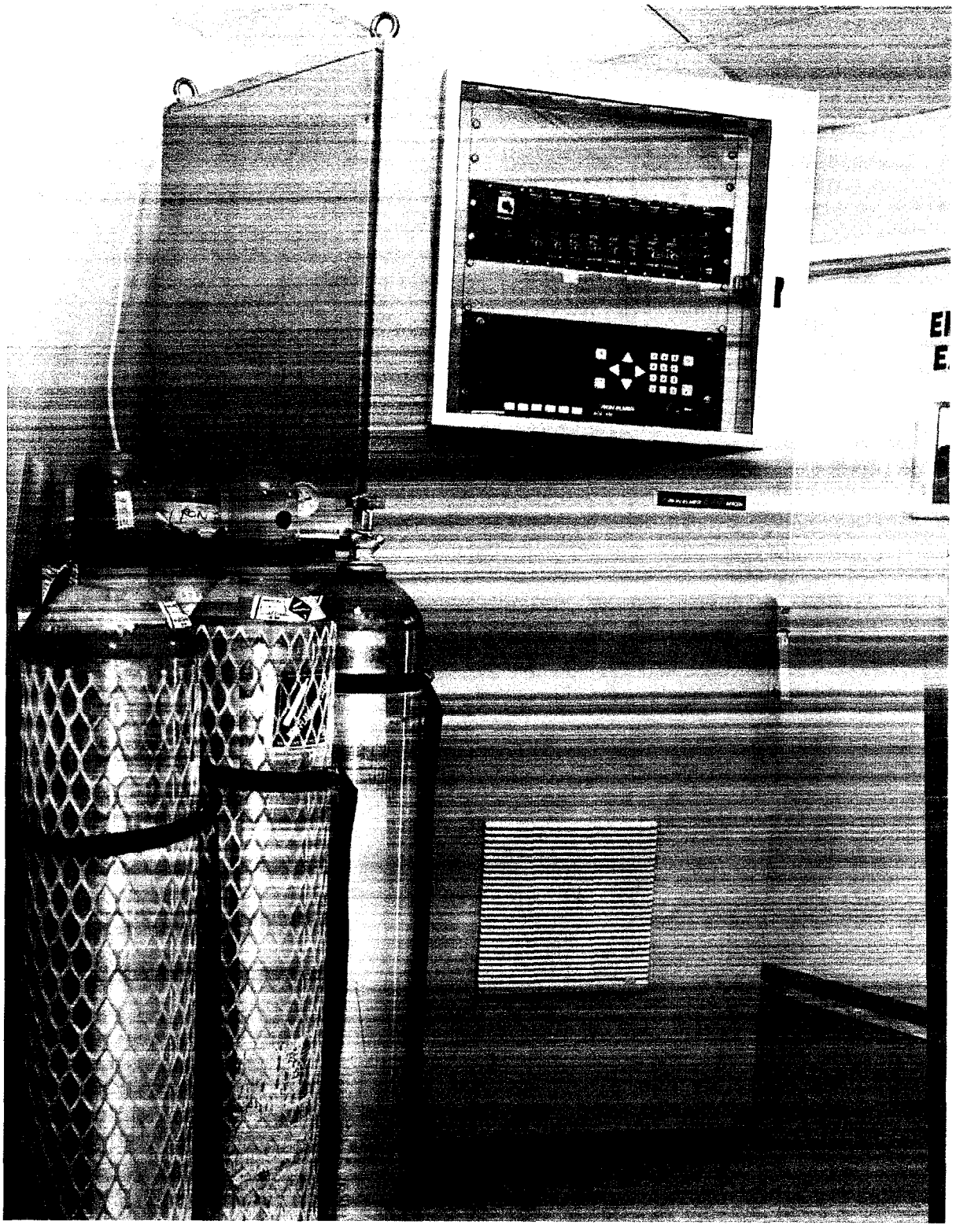

Fig. A.2. External view of MERCEM monitor cabinet (background) and mercury vapor calibration gas bottles (foreground).

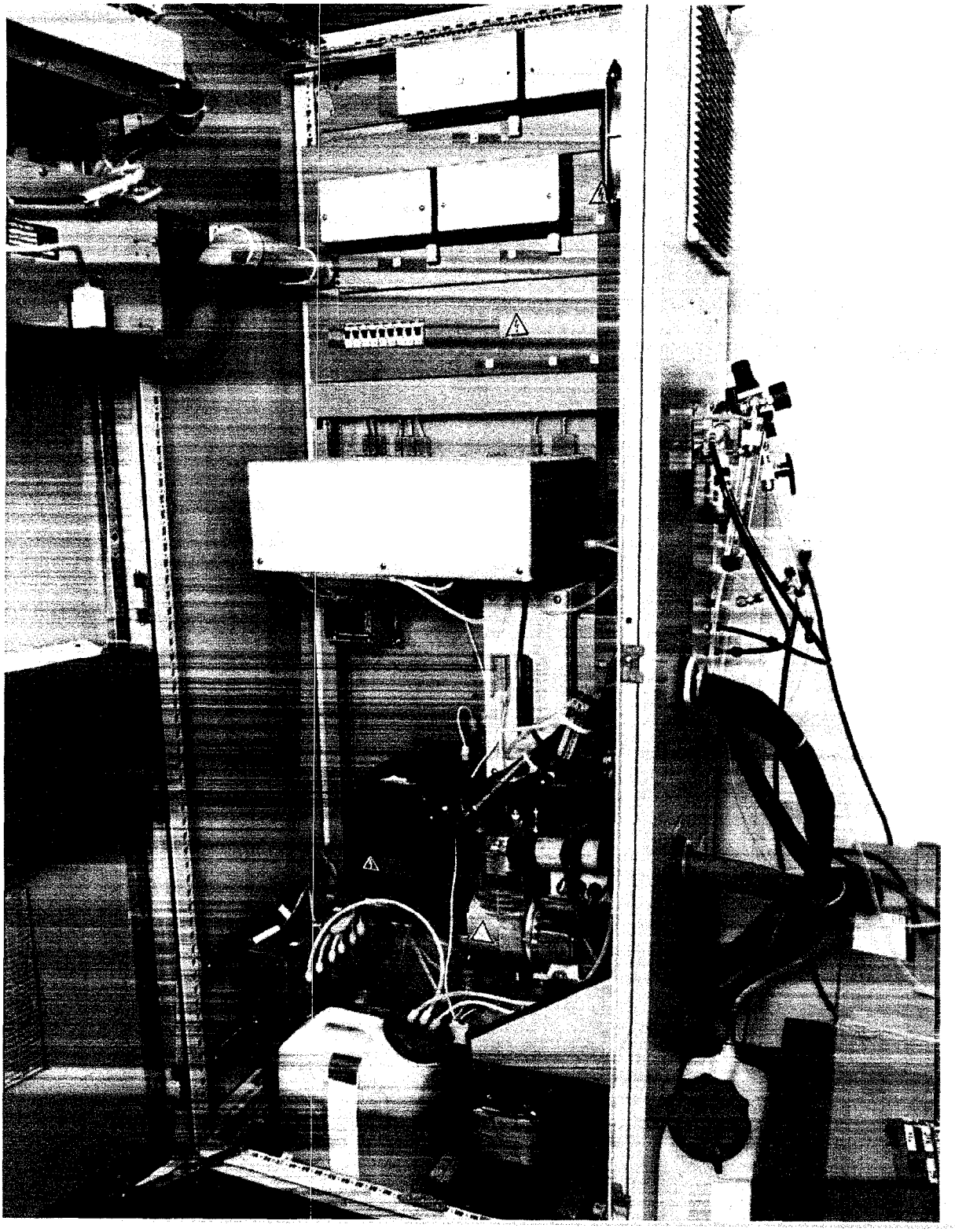

Fig. A.3. Internal view of MERCEM monitor cabinet.

Fig. A.4. Mercury vapor calibration gas bottles and blending apparatus (lower left) for introducing interference test gases.

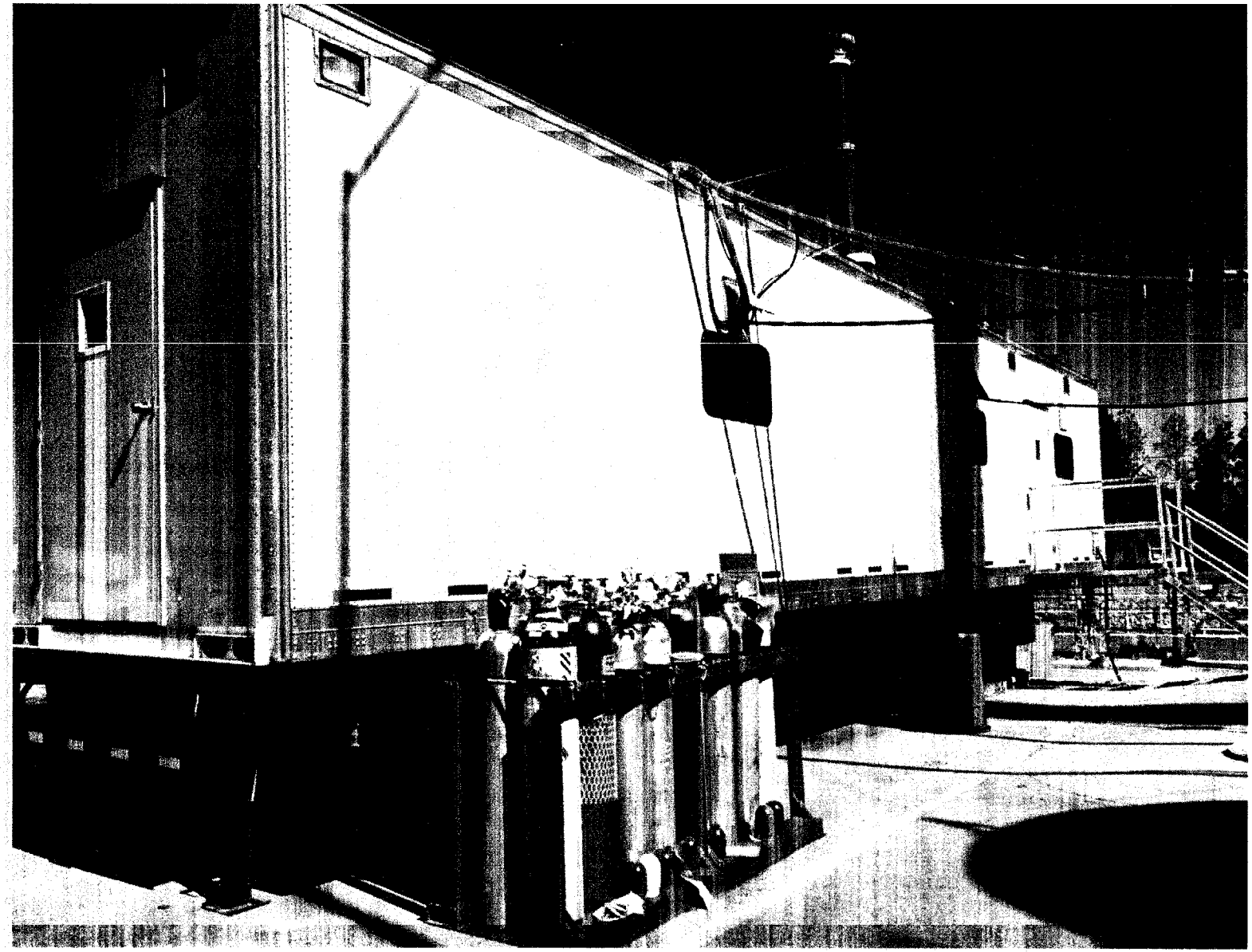

Fig. A.5. Mobile laboratory trailer used to house MERCEM monitor cabinet with interference test gas bottles in the foreground.

Fig. A.6. Researchers conducting test with MERCEM monitor.

Fig. A.7. Researchers reviewing data from MERCEM monitor.

APPENDIX B

RESPONSIBILITIES OF PARTICIAPTING ORGANIZATIONS

Table B.1. Responsibilities of participating organizations

Organization	Responsibility
Aldora	Provide a minimum 3-day notice of the arrival of the MERCEM to TSCA staff.
Aldora	Provide a condensate collection bottle to collect liquid and spent reagent condensed from the gas sample stream.
Aldora	Confirm the concentration of stannous chloride in the condensate and inform TSCA staff.
Aldora	Provide recipe for preparation of stannous chloride reagent.
Aldora	Provide a temperature-controlled stainless steel probe, heated filter housing, and 100-ft heated sample line to extract stack gas and transport it to the analyzer.
Aldora	Provide a regulator and polishing unit for the air line.
Aldora	Provide a laptop PC for storing and trending MERCEM CEMS data.
Aldora	Contact Spectra Gases to determine willingness to provide compressed gas cylinders of Hg to be used as the calibration gas. The target concentration of Hg is ~70μg/m^3. Investigate whether a pre- or post-test of the calibration gas will be provided by Spectra.
Aldora	Investigate the size of the calibration gas cylinders to determine if they can be placed inside the test bed trailer hood, which will accommodate 40-in. cylinders.
Aldora	Investigate the availability and cost of using a permeation tube-type calibration device. Provide necessary information to TSCA staff for ordering the permeation tube device. Determine if vendor can provide a pre- and post-calibration of the permeation rate.
Aldora	Provide a steam generator calibrator unit for the H_2O vapor interference test.
LMES	Install existing TSCA heated sample line and pump to return extracted bypass sample gas to the stack.
LMES	Provide and install a filter on the exhaust of the processed gas stream with little or no pressure drop imposed by the filter.
LMES	Sample condensate from the analyzer, submit sample for total activity analysis, and dispose of the condensate in site collection sump.
LMES	Prepare stannous chloride reagent based on recipe provided by Aldora.
LMES	Provide a 6-in. to 4-in. transition piece to interface the probe to the stack port or install the probe on a 4-in. port. In either case, the probe will be braced to support the weight of the probe and filter housing.
LMES	Install the sample gas extraction equipment. The probe for the analyzer will be installed in a port on the lower sampling platform. The sample line will be strung from the stack to the test bed trailer in such a manner to prevent sags and dips in the line.
LMES	Rinse the sample line with nitric acid, sample, and submit the rinse solution for total activity analysis.
LMES	Install the hood chimney on the test bed trailer and have the hood inspected.
LMES	Investigate the feasibility of using an industrial hygiene (IH) ambient air mercury (Hg) monitor that could be used for verifying the CEMS input gas stream during calibration. Determine if instrument can be sent to factory for calibration prior to start of testing. Provide copies of vendor literature to other test participants.
LMES	Investigate the incremental project cost of setting up and running a midget impinger train for verification of mercury calibration gas concentrations.

Table B.1 (continued)

Organization	Responsibility
LMES	Conduct 1-h Reference Method 101A RA tests and perform sample analysis. Method 101A probe will be a fixed, single-point probe installed in a port on the lower sampling platform and co-located with the MERCEM probe. The number of Method 101A runs conducted each day will be 3-3-3 or 5-4, depending on supply of waste feed selected for testing.
LMES	Prepare test plan based on PS12, Holly Hill test documentation, and technical discussions.
TSCA	Provide assistance with arranging customs broker in Knoxville for direct shipment of analyzer to Oak Ridge.
TSCA	Arrange to have riggers lift and place the MERCEM analyzer instrument cabinet in the test bed trailer.
TSCA	Check with Central Neutralization Facility (CNF), the on-site wastewater treatment plant, to determine if condensate can be placed directly in sump.
TSCA	Provide Aldora with a list containing typical stack pollutant concentrations.
TSCA	Provide electrical power, nitrogen, and plant air to the analyzer. The sample line requires 208-V AC single phase (with one neutral and one ground). The MERCEM and the sample line will draw approximately 30 A. A nitrogen line will be run inside the trailer and it will be provided with a regulator to regulate the nitrogen pressure at 10 psig. An air line will be run inside the trailer.
TSCA	Provide a telephone line to the trailer.
TSCA	Install a PC/PLC in the test bed trailer for acquisition and storage of MERCEM CEMS data and for viewing the status of the incineration process. This PC will have capability to view the incineration process, store data from the Hg monitor, trend process and MERCEM CEMS data, and correct the Hg CEMS data for O_2 concentration based on measurements by the facility O_2 CEMS.
TSCA	Arrange to have health physics (HP) technicians survey the probe and filter in the filter housing. HP technicians may request that tubing connections inside the analyzer cabinet be disconnected to survey analyzer internal surfaces.
TSCA	Investigate possibility of having coverage 1 h/day by IH technician during monitor testing to operate IH ambient Hg monitor for verifying calibration gas concentrations.
TSCA	Procure and provide the interference test gases [except water (H_2O)] for the interference response test.
TSCA	Investigate with the EPA Office of Solid Waste the current thinking with regard to the proposed MACT limit for Hg emissions.
TSCA	Investigate the availability of waste material to determine if a single homogeneous waste can be fed during two to three days of testing to coincide with a RA test audit (RATA). The waste streams may vary from the first to the second RATA.

APPENDIX C

TSCA INCINERATOR FACILITY SITE ACCESS TRAINING REQUIREMENTS

Table C.1. TSCA Incinerator facility site access training requirements

Activity	Training Requirements
Access to TSCA Incinerator site (40 h per year or less)	TSCA Site Access Training Video
Access to TSCA Incinerator site (more than 40 h per year)	TSCA Site Access Training Video Park Worker Training
Escorted access into radiological areas	TSCA Site Access Training Video Park Worker Training Permission of Facility Manager
Unescorted access into radiological areas	TSCA Site Access Training Video Park Worker Training 24-h Hazardous Waste Operations and Emergency Response (HAZWOPER) Training Rad-Worker II Training Permission of Facility Manager
Hands-on work in radiological areas (escorted or unescorted)	TSCA Site Access Training Video Park Worker Training 24-h HAZWOPER Training Rad-Worker II Training Permission of Facility Manager
Hands-on work in non-radiological treatment/ storage/disposal areas (K-1425, K-1435-B, K-1435-C)	TSCA Site Access Training Video Park Worker Training 24-h HAZWOPER Training Permission of Facility Manager

APPENDIX D

TUV REPORT

INSTITUT FÜR UMWELTSCHUTZ
UND ENERGIETECHNIK

REPORT ON THE APTITUDE TEST OF THE TOTAL MERCURY MEASURING DEVICE "MERCEM"

BODENSEEWERK PERKIN-ELMER GMBH, MEERSBURG

TÜV-Report No.: 936/805012
Cologne, April 25, 1996

Report on the Aptitude Test of the MERCEM Mercury Measuring Device
from Bodenseewerk Perkin Elmer, Meersburg

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1 OVERVIEW

Bodenseewerk Perkin-Elmer GmbH commissioned the TÜV Rheinland Sicherheit und Umweltschutz GmbH to conduct a pilot aptitude test of the MERCEM measuring device according to the standards for continuous emission measuring devices [1]. The sample gas component gaseous mercury (Hg) and its compounds were determined.

The measuring principle of this extractively operating measuring device is based on the wet-chemical reduction of the total content of mercury in the sample gas into its elemental form and - following an amalgamation procedure - the subsequent analysis by UV photometry.

The tests included laboratory tests and a field test of three months duration in the purified waste gas of a communal refuse incineration plant. The smallest measuring range tested was 0 to 100 $\mu g/m^3$.

The pilot aptitude test was based on minimum performance requirements, all of which were fulfilled.

Therefore, the TÜV Rheinland Sicherheit und Umweltschutz GmbH has proposed that the tested device be published as an aptitude-tested measuring device for the continuous determination of gaseous mercury and its compounds in plant emissions, pursuant to the 13th and 17th BImSchV and TA Luft (German environmental regulations on the prevention of air pollution).

2 OBJECTIVES

Bodenseewerk Perkin-Elmer GmbH commissioned the TÜV Rheinland Sicherheit und Umweltschutz GmbH to conduct a pilot aptitude test of the MERCEM measuring device within the measuring range of 0 to 100 µg/m^3. The measuring system is an extractively operating instrument with wet-chemical sample preparation procedure followed by an amalgamation step and subsequent UV photometrical analysis.

The examinations included laboratory tests and a three months lasting long-duration test of two measuring devices in the purified waste gas of a communal refuse incineration plant.

The measuring results of these examinations were compared to the minimum requirements according to the

> "Guidelines concerning the aptitude test, installation, calibration and maintenance of measuring devices for continuous emission measurements" published by the Federal Environmental Agency ("BMU" = Federal Minister for the Environment, Environmental Protection and Reactor Safety [1].

3 DESCRIPTION OF THE MEASURING DEVICE

3.1 Measuring Principle and Setup of the Measuring Device

The main components of the MERCEM measuring device are (see fig. 3.1)

- the sample gas line
- the wet-chemical sample preparation unit
- the analyzer including the amalgamation unit
- the electronic evaluation and control unit

Sample extraction is carried out extractively via a sampling probe, a sampling tube, a gas diaphragm vaccuum pump and a flow meter. The sample gas flow is approx. 1000 l/h to minimize adsorption effects at the walls.

After the flow meter and the gas diaphragm vaccuum pump a partial gas stream of approx 30 l/h is conducted through a second gas membrane vaccuum pump. From here, the waste gas is conducted to the reaction solution, the rest is led to the flue gas outlet.

The sampling probe, the gas membrane vaccuum pump (1000 l/h) and the sample gas tube are heated to approx. 185 °C.

The mercury compounds are reduced to elementary mercury by means of a $SnCl_2$ reaction/reduction solution. For this purpose, the reaction solution is led behind the gas diaphragm vaccuum pump (1000 l/h) and the reaction starts immediately at the point where the reaction solution comes into contact with the waste gas. The reaction is finished inside the reactor and the separation of gas/liquid takes place. To prevent that the reagent is diluted by waste gas, reaction solution is cyclically refilled from a reservoir by means of a peristaltic pump. A second pump removes excess solution. After having passed a two-step cooler with a constant dew point of 5 °C the sample gas enters the gold trap for amalgamation.

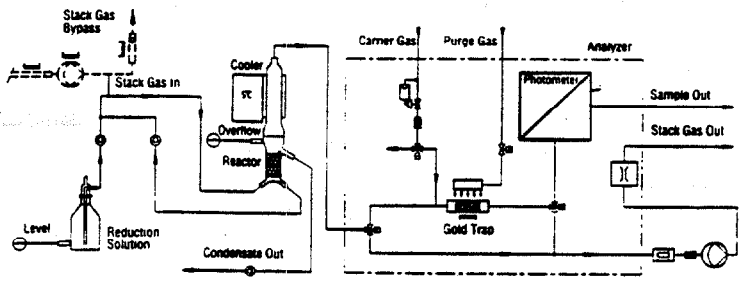

Fig 3.1: Schematic Setup of the MERCEM Measuring Device

The waste gas is collected on the gold trap for a defined period. The sample flow is kept constant and exactly registered by an electronic mass flow meter. At the end of the collection period the gold trap is purged with zero gas (nitrogen or instrument air) and the base line of the analyzer is determined.

Thereafter, the gold trap is heated electrically - by means of resistor wire - to remove the mercury from the gold.

The zero gas stream (nitrogen, exclusively) carries the mercury into the cell for UV-photometrical measurement.

After purging and cooling by means of a strong air stream the gold trap is ready for the next sample.

The cycle time of one measurement is 180 s and consists of:

Collection period	:	approx. 10 s
Baseline	:	approx. 50 s
Heating and measurement	:	approx. 90 s
Cooling	:	approx. 30 s

The sensitivity of the system can be adapted to the desired measuring range by adjusting different sample collection times. A sample flow of approx. 30 l/h and a sample collection period of approx. 10 s are to be used for a measuring range of 0 to 100 $\mu g/m^3$.

3.2 Technical Data

The most important technical data are shown in table 3.1. For further details on the measuring device please refer to the operating instructions included in annex 10.5.

Table 3.1: Technical Data of the MERCEM Analyzer System

Number of measuring ranges	programmable
Automatic switch-over of measuring ranges	yes
Output signal	0 ... 20 mA / 4 ... 20 mA
Display of measuring values	numerically and graphically
Warm-up period	approx. 1 h
Admissible ambient temperature (Operation)	5 - 40 °C
Consumption gases	N_2 and instrument air
Operational position	according to design
Auxiliary energy: mains connection Power consumption	 380 V max. 4610 VA at 10 m sampling tube
Weight	340 kg
Material of the parts in contact with the sample gas	high-grade stainless steel, glass, PTFE
Electrically heated sample tube Length: Temperature: Power consumption (Operation):	 10 m 185 °C 1500 VA max.

4 TEST PROGRAM

The pilot aptitude test was performed according to the "guidelines concerning the aptitude test, installation, calibration and maintenance of measuring devices for continuous emission measurements" by the Federal Minister for the Environment, Environmental Protection and Reactor Safety [1] and according to the test catalogue [2].

4.1 Laboratory test

The following laboratory test program was established:

- Examination of all instrument functions,
- Determination of the instrument characteristics by means of test gases,
- Determination of cross sensitivities of the measuring system towards escort substances in the waste gas.
- Test of the zero and reference point stability within the admissible ambient temperature range.
- Determination of the influence of supply voltage fluctuations to the measuring signal,
- Determination of the influence of relative air humidity, content of spray-water in the air, vibrations and operational position,
- Influence of pressure changes and changes in the flow rate.

Two identical measuring devices with following instrument numbers were tested:

Device 1 : TÜV 01
Device 2 : TÜV 02.

4.2 Field Test

The long-duration test was conducted during approx. three months (from December 2, 1995 through March 1, 1996) with two identical systems of the type MERCEM with following instrument numbers:

Device 1 : TÜV 01
Device 2 : TÜV 02.

The following measuring range had been selected for the long-duration test:

0 - 100 µg/m^3 ≙ 4 - 20 mA.

Based on these measuring ranges, the following device characteristics were tested during the specified test period:

- Dead and response time
- Calibrating properties
- Detection limit
- Reproducibility
- Constancy of device characteristics (zero point, sensitivity drift)
- Maintenance intervals
- Availability
- Function test and calibration.

The field test was performed at a communal refuse incineration plant.

The plant consists of four parallel incinceration lines with grate furnace, additional incineration chamber and waste-heat boiler, as well as electric filter, spray adsorber and fabric filter.

To separate organical trace components and mercury to meet the requirements of the 17. BImSchV (German regulation on the emission ...) hearth furnace coke (HOK) is added before the fabric filters.
The mercury concentration in the waste gas could be influenced by switching the HOK-addition on, respectively off.

The waste gas of the four furnaces is conducted outside via a stack. The measuring devices were installed in the purified waste gas of the line 1 at a vertical waste gas channel. The measuring point is in accordance with the requirements of the VDI 2066 guideline. At the beginning of the field test the measuring devices had been installed at the waste gas channel of line 2 because of revision works. For connection to the line 1 channel, only the sampling probes with the sampling tubes had to be installed at the other line. The measuring devices did not have to be moved for this purpose as the sample gas/measuring outlets are in the same room. Both calibration measurements were conducted at line 1. The reference measurement outlet is located close to the sampling point.

The peripheral waste gas conditions were as follows:

Flow rate:	9 - 13	m/s
Content of O_2:	10 - 14	Vol.-%
Waste gas moisture (n,f):	approx. 20	Vol.-%
Flue gas temperature:	approx. 140	°C

4.3 Test Gases

With thermal processes the heavy metal mercury is mainly released in gaseous form and can be existant in the waste gases in various compounds. Apart from its elemental form (Hg(0) it can also be emitted as $HgCl_2$ Hg (II). Furthermore organic mercury compounds, mercury sulfide or mercury oxide can be emitted. To a small extent, it can also be adsorbed to dust. Table 4.1 provides a general overview over some of the possible compounds of mercury in waste gases.

Table 4.1: Mercury compounds in waste gases

Formula	Solubility in water	Boiling point °C
Hg	-	356
$HgCl_2$	+	303
Hg_2Cl_2	-	385 (Sublimation) (Disproportioning at 400 °C)
HgS	-	586 (Sublimation)
HgO	-	Decay into the elements at 400 °C
$Hg(CH_3)_2$	-	96

For measurements at e.g. refuse incineration plants only Hg(0) and $HgCl_2$ are of interest. For determination of the total mercury emissions it is usually sufficient to measure these two species.

4.3.1 Production of Test Gases

During the laboratory tests the test gases $HgCl_2$ and Hg(0) were produced by means of thermically controlled test gas generators *) as shown in fig. 4.1 and 4.2. At every setting point the mercury content was determined by means of the reference procedure (VDI 3868) parallel to the sampling of the test gas to the analyzer system. The complete setup of the test gas production is shown in fig. 4.3.

In addition to the temperature control the concentration adjustment was also performed by diluting the test gas in a N_2 main volume stream by means of electronic mass flow meters.

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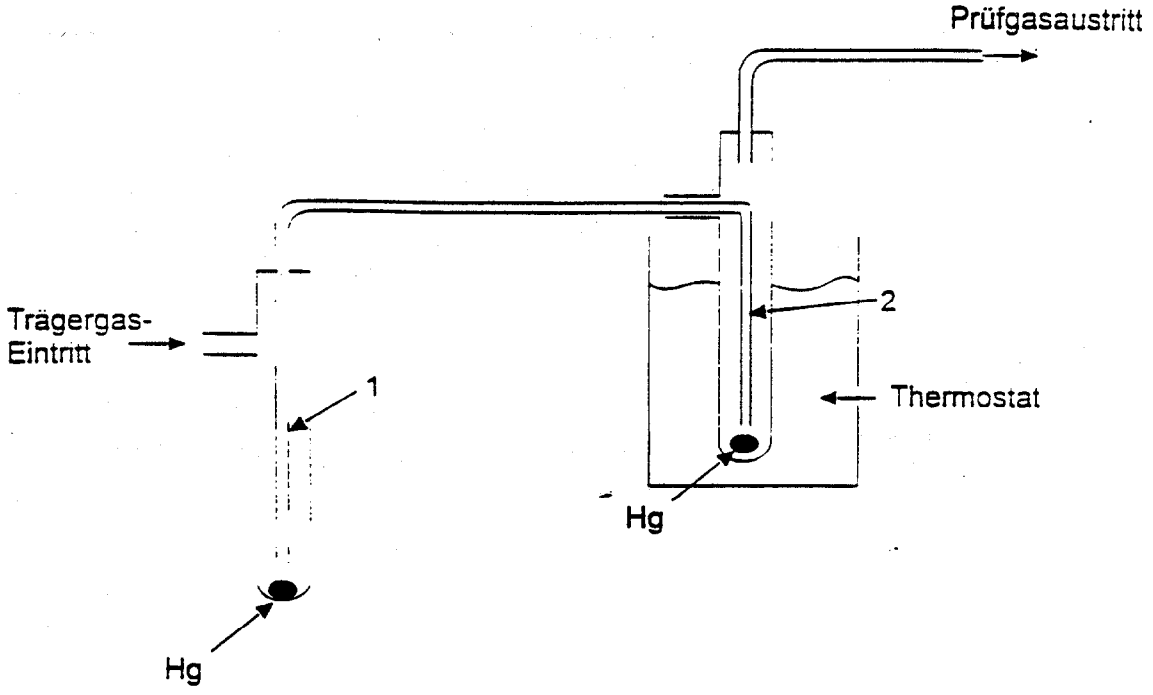
*) W. Jockel und P. Wilbring: Stand und Perspektiven der automatischen Emissionsüberwachung für besondere Stoffe, VDI-Berichte Nr. 1059, 1993



1 Test gas cell 1 for enrichment of the carrier gas stream with mercury at ambient temperature

2 Test gas cell 2 for stable adjustment of the Hg concentration ($T_{cell\ 1} > T_{cell\ 2}$)

Fig. 4.1: Hg(0) - Test gas cell

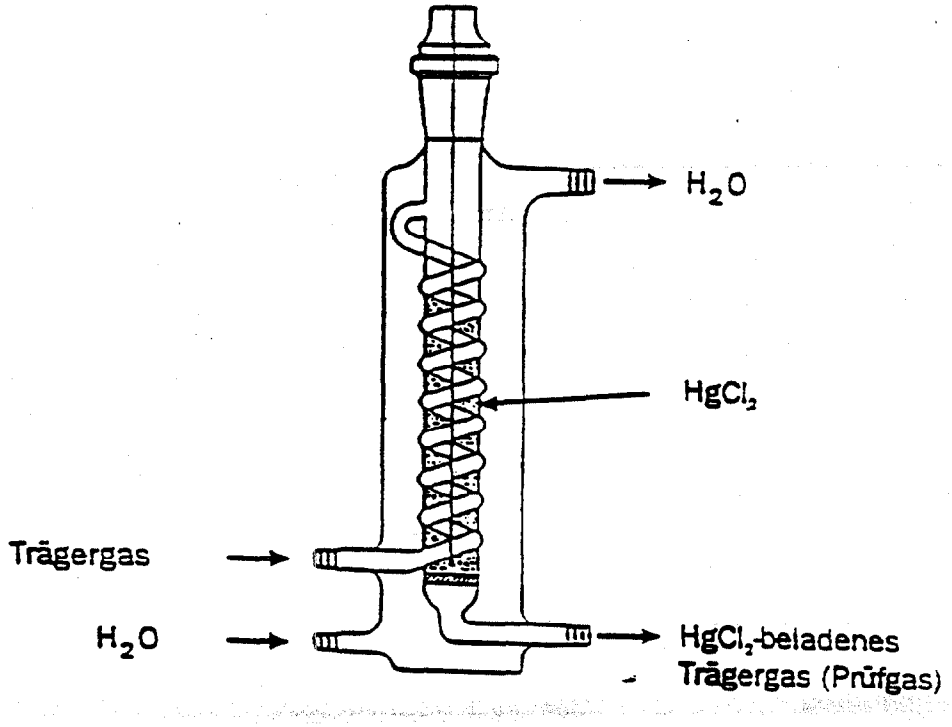
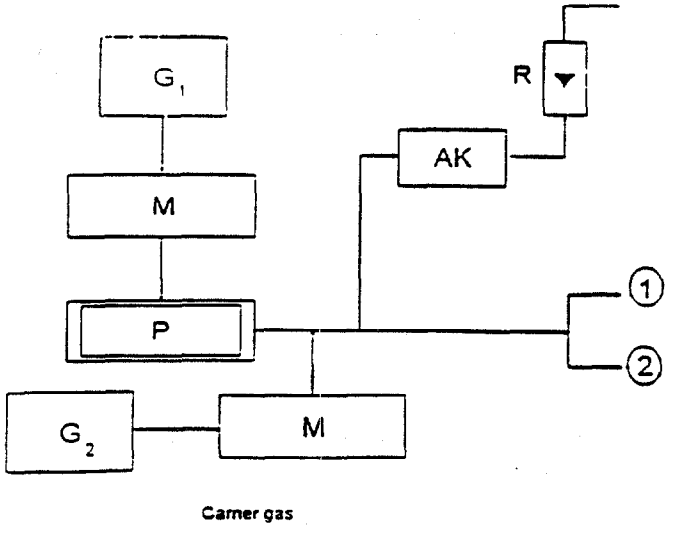


Fig. 4.2: $HgCl_2$ - Test gas cell



G_1	Trägergasstrom
G_2	Verdünnungsgasstrom
M	Elektronischer Massendurchflußregler
P	Prüfgaszelle
AK	Quecksilber Adsorptionskartusche
Hg-M	Hg MAK-Monitor zur Überwachung der Konstanz der erze Prüfgaskonzentration (bei Hg (II) - Erzeugung mit vorgesc Reduktionseinheit)
R	Volumenstrommeßgerat
1, 2	Prüfgasausgange (fur zu prufendes Gerat und fur naßcne Vergleichsanalyse

Fig. 4.3: Total view of the setup of the Hg test gas production

4.3.2 Test Gases from Compression Cylinders

During the pilot aptitude test also mercury test gases from cylinders were used. Hg(0) test gases in special high-grade steel pressurized cylinders were obtained from the Messer Griesheim company. A feature of these was the good long-term stability of the mercury concentration, however, the actual concentration in the pressurized cylinder was considerably different from the value indicated (up to 100%). Consequently, each individual cylinder had to be analyzed prior to using it in the test. Due to the high gas flow of the MERCEM measuring system cylinder gas could only be used when not operating the big gas diaphragm vaccum pump (1000 l/h).

4.4 Reference Procedures

Object to be measured : Mercury in gaseous form

Measuring procedure : VDI 3868 guideline, page 2, draft of Oct. 1995

Sampling Devices:

- Sampling probe : heated by waste gas
  Material : glass

- Sampling tube : heated
  Material : glass
  (in the laboratory without probe and sampling tube)

- Gas volume meter : Gas meter (type: dry)

- Absorption devices : 100 ml washing bottles with D2-frit, cleaned with 20% nitirc acid.

- Sorption medium : Solution consisting of 20 g $KMnO_4$, 2 ml HCl (c = 1 mol/l) in 1 l 10% H_2SO_4

  35 ml each of the solution were filled into the two washing bottles in cascade connection.

  After finishing the sampling procedure the excess oxidation medium was reduced in the washing bottles by adding 10% aqueous $(NH_3OH)Cl$ solution.

- Transport and storage : refrigerated in PP-cups with PE lids
- Service life of the samples : max. 5 days
- Distance between sampling point of the probe and separating element : 1,2 m

Analytical Determination

- An aliquote proportion of the adsorption solution was conducted into the atomic absorption cold vapor analyzer where it was mixed with the reaction solution (10 g $SnCl_2 \cdot 2\ H_2O$ in diluted hydrochloric acid)
- Analytical instrument
  Manufacturer / Type : Fa. Seefelder / Hg 254 A
  TÜV-Part-No. : 805
- The standard calibration procedure was applied for calibration.
- Standards : Standard solution of Messrs. Merck
  c(Hg) = 1000 mg/l

Characteristic Values of the Procedure / Quality Control

- Detection limit
  - absolute : 0,1 µg
  - relative : 2 µg/m^3 (sample gas volume 0,06 m^3)
- Measures for quality control:
  - Duplicate determinations were carried out.
  - Blind values and standards were also measured and taken into consideration.
  - Reference measurements with mercury test gases.

Measurement accuracy (U):

Mean mg/m^3	U (95 %) mg/m^3	Sample gas volume l
0,02	0,007	60
0,05	0,01	60
0,08	0,02	60
0,20	0,04	60

Own duplicate determinations from 1991 to 1995.

5 LABORATORY TESTS

5.1 Verification of the Instrument Characteristics (Calibration Function)

In the laboratory the correlation between the measured value and the specified quantity of the measuring object was determined. For this purpose, the devices were sampled with various test gas concentrations (as shown in fig. 5.1).

Fig. 5.1: Test set-up for registration of the instrument display

The devices were sampled both with Hg(0) as well as with $HgCl_2$ test gas. The values resulting from these tests are shown in appendix 10.4.

The specified mercury test gas concentration (x-axis) in $\mu g/m^3$ were correlated to the corresponding measuring signals of the analyzer (y-axis) in mA. The resulting characteristic values are shown in table 5.1. The instrument characteristics $y = b * x + c$ were determined by linear regression calculation.

Table 5.1: Values of instrument characteristics

Component	Measuring Range	Number of Measured Values	b	c	r^2	V_b	V_c
Device 01	0 - 100	16	0,168	3,572	0,9952	0,003	0,371
Device 02	0 - 100	18	0,158	4,120	0,9962	0,002	0,307

b = Inclination of the instrument characteristics (mA / $\mu g/m^3$)
c = Axis segment of the instrument characteristics (mA)
r = Correlation coefficient
V_b = Confidence interval of the inclination b (mA / $\mu g/m^3$)
V_c = Confidence interval of the axis segment c (mA).

5.2 Determination of Cross-Sensitivities

The interferences with usually occuring escort substances in the waste gases were determined using test gases of specific concentrations.

The following test gases were sampled to the analyzer:

Component		Concentration	
Carbon dioxide	CO_2	15,0	Vol.-% in N_2
Carbon monoxide	CO	300	mg/m³ in N_2
Sulfur dioxide	SO_2	1000	mg/m³ in N_2
Nitrogen monoxide	NO	300	mg/m³ in N_2
Nitrogen dioxide	NO_2	29	mg/m³ in SL
Ammonia	NH_3	20	mg/m³ in N_2
Di-Nitrogen monoxide	N_2O	20	mg/m³ in N_2
Hydrochloric acid	HCl	50	mg/m³ in N_2
Moisture	H_2O	30	Vol.-% in N_2
Methane	CH_4	50	mg/m³ in N_2
Benzole	C_6H_6	2	mg/m³ in N_2

The interference tests for the nitrogen oxides (NO, NO_2) and for HCl were carried out with humid test gases. Sampling dry NO-, NO_2- and HCl test gases can lead to negative deviations of the measuring signal at the reference point.
The results are shown in table 5.2.

Table 5.2: Influence of escort substances to the measuring result at the zero and reference point

	Device 1		Device 2	
Escort substance	Deviation in % from fullscale			
	NP	RP	NP	RP
CO_2	< 1,0	< 1,0	< 1,0	< 1,0
CO	< 1,0	< 1,0	< 1,0	< 1,0
SO_2	< 1,0	< 1,0	< 1,0	< 1,0
NO	< 1,0	< 1,0	< 1,0	- 1,1
NO_2	< 1,0	< 1,0	< 1,0	< 1,0
NH_3	< 1,0	- 1,0	< 1,0	< 1,0
N_2O	< 1,0	< 1,0	< 1,0	< 1,0
HCl	< 1,0	- 1,0	< 1,0	- 1,0
CH_4	< 1,0	- 1,4	< 1,0	- 1,3
H_2O	< 1,0	< 1,0	< 1,0	< 1,0
C_6H_6	< 1,0	< 1,0	< 1,0	< 1,0
Sum of positive deviation	< 1,0	< 1,0	< 1,0	< 1,0
Sum of negative deviation	< 1,0	- 3,4	< 1,0	- 3,4

In total the sum of all individually determined changes of the measured values when sampling interfering components was less than 4%, referred to fullscale.

The requirements (sum of positive and negative deviations ± 4 % of fullscale each) are met.

5.3 Influence of the Ambient Temperature

Within the admissible temperature range of +5 °C to +40 °C (ambient temperature) the measuring devices were tested with two test gas concentrations within the measuring range including zero gas (N_2). The ambient temperatures were varied in steps of 10 resp. 5 K within an air-conditioned chamber. The relative humidity of the ambient air was kept constant to 60 % (relative). Adjustment resp. calibration of the measuring devices was carried out with nitrogen and test gas at an initial temperature of 20 °C. The steady-state period for each temperature step was 3 hours. The results shown in tables 5.3 and 5.4 were determined in two temperature test series. At each temperature setting zero gas (N_2) and test gas were sampled.

Table 5.3: Influence of the ambient temperature on the measuring result at the zero point
Measuring range: 0 to 100 µg/m³
Deviation in: % fullscale

	Temperature	°C	5	10	20	30	40
Device 1	Deviation	%	0,2	0,2	-	0,1	0,5
	Zero point		0,2	0,2	0,0	0,1	0,5
Device 2	Deviation	%	0,2	0,2	-	0,4	0,8
	Zero point		0,1	0,1	0,0	0,4	0,8

Table 5.4: **Influence of the ambient temperature on the measuring result at the reference point** (approx. 70 % of fullscale)
Measuring range: 0 to 100 µg/m³
Deviation in: % of the set value

	Temperature	°C	5	10	20	30	40
Device 1	Deviation	%	1,5 *	0,9	-	2,3	- 1,5
	Reference point		79,7	79,1	78,5	80,3	79,1
Device 2	Deviation	%	1,1 *	1,2	-	- 1,2	1,0
	Reference point		74,2	74,3	73,4	72,5	73,2

*) Deviation in reference to 15 K temperature step.

According to the BMU guidelines dated 01.03.1990 the minimal requirements for measuring devices for gaseous emissions allow the following changes in the measuring signals when the ambient temperature is varied by 10°:

Zero point	± 2 % of fullscale
Sensitivity	± 3 % of the required value.

The maximum deviations at the zero point are actually 0.8 % of fullscale and 2.3 % of the set value at the reference point. The minimal requirement in the range of +5 °C to +40 °C are met.

5.4 Influence of mains voltage variations

To determine the influence of mains voltage variations on the measuring results of the instrument, the power supply was varied by means of a transformer in steps of 20 V each within the voltage range of 190 V to 250 V. The influence of voltage variations was examined while sampling zero gas (N_2) as well as when sampling test gas in the measuring range of 0 to 100 µg/m^3. The test series were carried out twice.

Table 5.8 shows the maximum changes stated within a voltage range of 190 V to 250 V.

The voltage tests were only performed for the following components of the measuring system as the measuring system is operated with three-phase current:

- Computer
- Photometer
- Photometer heating.

Table 5.8: Influence of mains voltage variations on the measuring results

	Zero point		Reference point			
Mains voltage	max. deviation in % of the measuring range		Required value		Deviation in % of the required value	
[V]	Device 1	Device 2	Device 1	Device 2	Device 1	Device 2
190	< 0,5	< 0,5	77,0	70,8	< 0,5	- 0,7
210	< 0,5	< 0,5	77,0	70,8	< 0,5	- 0,8
230	-	-	77,0	70,8	-	-
250	< 0,5	< 0,5	77,0	70,8	< 0,5	- 0,7

According to the minimal requirements the following changes of the measuring signals are admissible:

Zero Point	± 2 % of fullscale
Sensitivity	± 2 % of the required value

The deviations at the zero point and at the reference point were less than 1 % for both devices. The minimal requirements are met.

5.5 Influence of relative air humidity, content of spray-water in the air, vibrations and operational position

The influence of the air humidity was not especially examined. It can be assumed, however, that the instrument - due to its design - is insensitive to air humidity as long as it does not fall below the dew point. The analyzer has to be protected against spray water by mounting it in a cabinet.

During the long-duration tests the devices were subjected to the vibrations occuring at the measuring place. Influences on the instrument function could not be stated. As a precaution the place of installation should as free as possible of vibrations.

The manufacturer does not mention any special details about the operational position. It is defined by the design of the device.

5.6 Influence of barometrical and flow variations

By sampling test gas with and without operating the big sample gas pump it was possible to vary the flow rate and pressure in the measuring system. These variations were carried out several times and in parallel the content of Hg was examined in a wet chemical procedure and compared with the instrument display.

For MERCEM the manufacturer specifies a flow rate of approx 1000 L/h in the operating mode. The flow rate was varied between 400 L/h and 1200 L/h and the measuring signals were recorded. The measuring signal was not found to be dependent from the flow rate.

The volumina at the photometer and at the gold trap required to calculate the content of Hg are determined continuously by means of an electronic mass flow meter. Any variations are thus integrated in the measuring signal immediately.

6 LONG-DURATION TEST UNDER FIELD CONDITIONS

The minimal requirements include a long-duration test of minimum 3 months time at a test location in the field, if possible. The measuring device to be tested should be operated under conditions that are very similar to field conditions, thus allowing to apply the test results to other comparable operating conditions.

The long-duration test was performed at a refuse incineration plant. Following are the characteristics of the operating conditions:

Type of plant	:	Communal Refuse Incineration
Waste gas purification	:	quasi dry sorption, dust adsorber system with fabric filter
Installation position of the measuring device	:	purified gas
Channel dimensions	:	rectangular channel, 1,35 m · 2,75 m
Waste gas temperature	:	ca. 140 °C
Installation location	:	Room for measuring instruments (room temperature).

Two MERCEM measuring systems of the same type were used during the complete period of the long-duration test. Maintenance was performed once a week at the instruments by TÜV staff. During the complete period of the long-duration test all measuring signals were continuously recorded by a data logger and subsequently condensed to average values of half an hour resp. two hours.

6.1 Dead time and response time

According to the VDI 2449 guideline, page 2, the dead time is defined as the interval between the rapid change of the state value and the rise of the measured value to 10 % of the expected height of the step response.
The rise time is defined as the interval after a rapid change of the expected measured value to 10 % and 90%-value of the expected height of the step response.
The response time is defined as the sum of the dead time and the rise time and should not exceed 200 sec.

Dead and response times of the laboratory measuring device were determined in the laboratory with a heated tube of max. 5 m and at the waste gas channel with approx. 10 m of heated tube.

According to the instrument description the MERCEM measuring system operates quasi-continuously with a cycle time of 3 minutes. During the laboratory and field tests it could be stated the the measuring system usually reaches 90 % of its measuring value within one cycle, certainly, however, after two cycles. This results in a maximum T_{90}-time of 360 sec.

6.2 Analysis function of the measuring device

At the start and at the end of the long-duration operation of the measuring device the analysis function was verified by means of reference measurements according to section 4.4. of this report. The sampling time was approx. 30 minutes. For the measured values refer to appendix 10.2.

By means of regression calculation a correlation between the measured values of the two devices and the measuring object in the matrix of the waste gas was determined. These calculations were based on a statistical reliability of P = 95 %.

Table 6.1 shows the results of the regression calculation. In fig. 6.1 to 6.4 examples of regression lines with their tolerance ranges and confidence intervals for one instrument each are presented graphically.

Table 6.1: Results of the regression calculation between the MERCEM instruments and the reference method.

	Device 1		Device 2	
	Start of the long-duration test	End of the long-duration test	Start of the long-duration test	End of the long-duration test
Measuring range	0 - 100 µg/m³			
Number of spot checks	17	17	17	17
Arithmetic mean of the measured values of the measuring device in mA	8,65	7,80	9,03	7,72
Arithmetic mean of the measured values of the reference procedure in µg/m³	26,2	21,9	26,2	21,9
Inclination of the regression line in µg/m³ / mA	5,207	5,337	5,179	5,442
Ordinate segment of the regression line in µg/m³	- 18,84	- 19,72	- 20,53	- 20,09
Standard deviation of the regression line in µg/m³	2,26	1,57	2,16	2,11

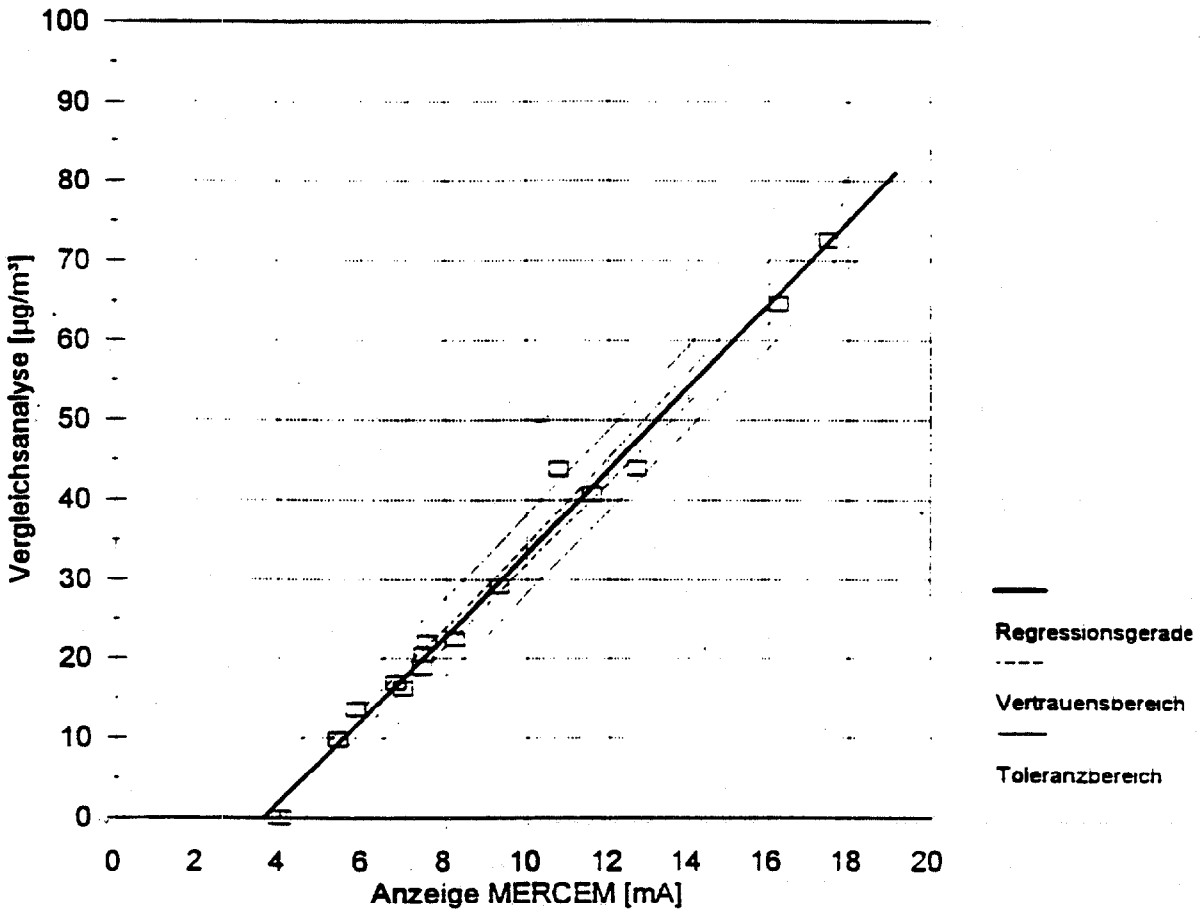


Fig. 6.1: Calibration curve (analysis function) for device 1 at the start of the long-duration test, measuring range 0 - 100 µg/m^3

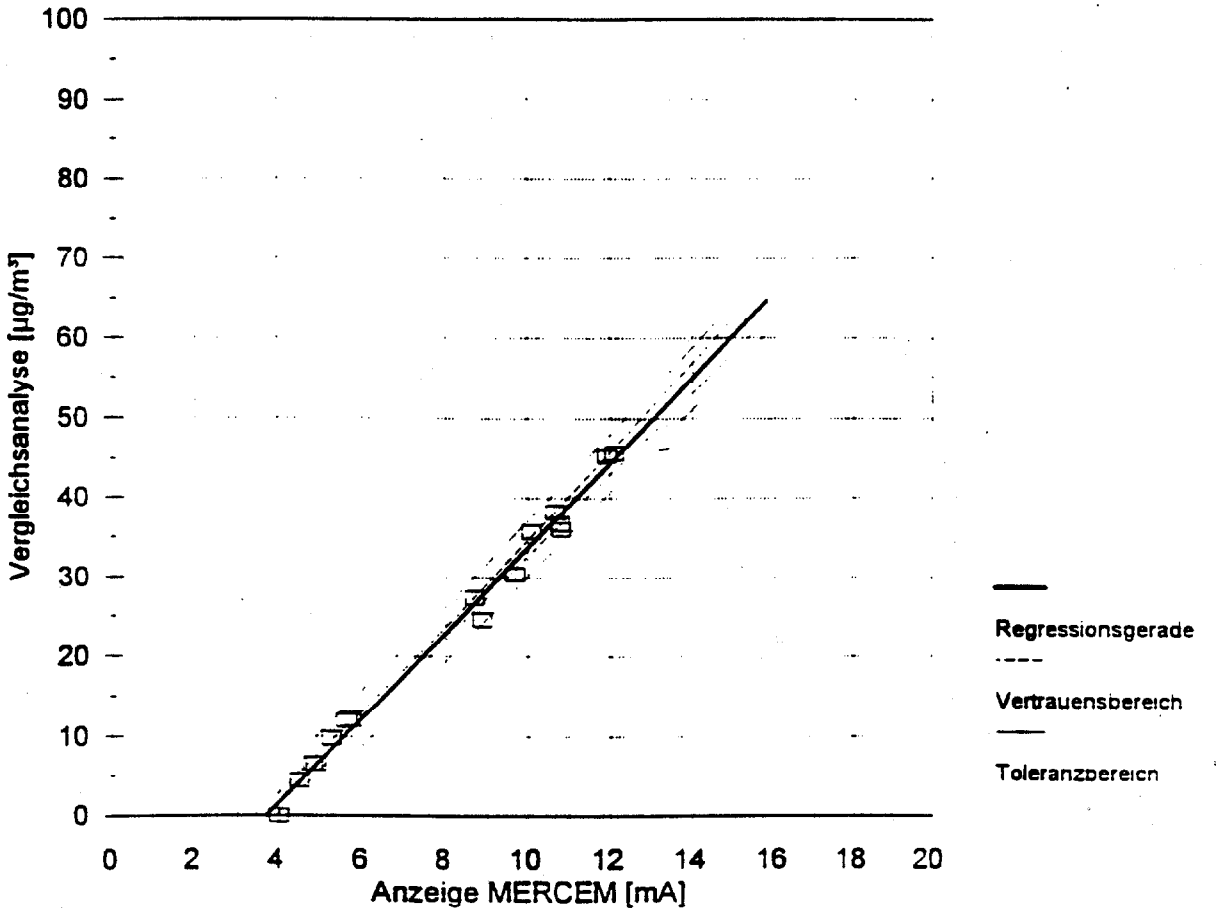


Fig. 6.2: Calibration curve (analysis function) for device 1 at the end of the long-duration test, measuring range 0 - 100 µg/m^3

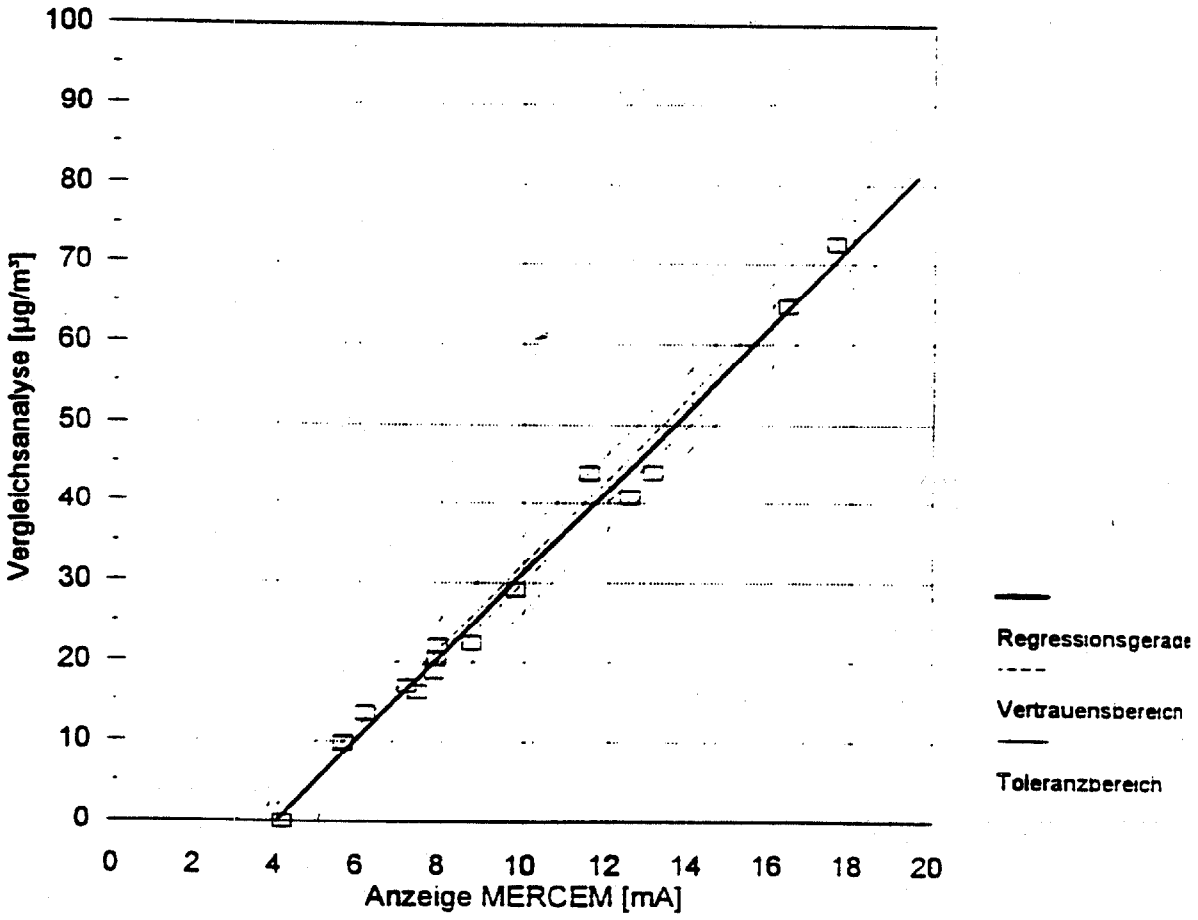


Fig. 6.3: Calibration curve (analysis function) for device 2 at the start of the long-duration test, measuring range 0 - 100 µg/m³

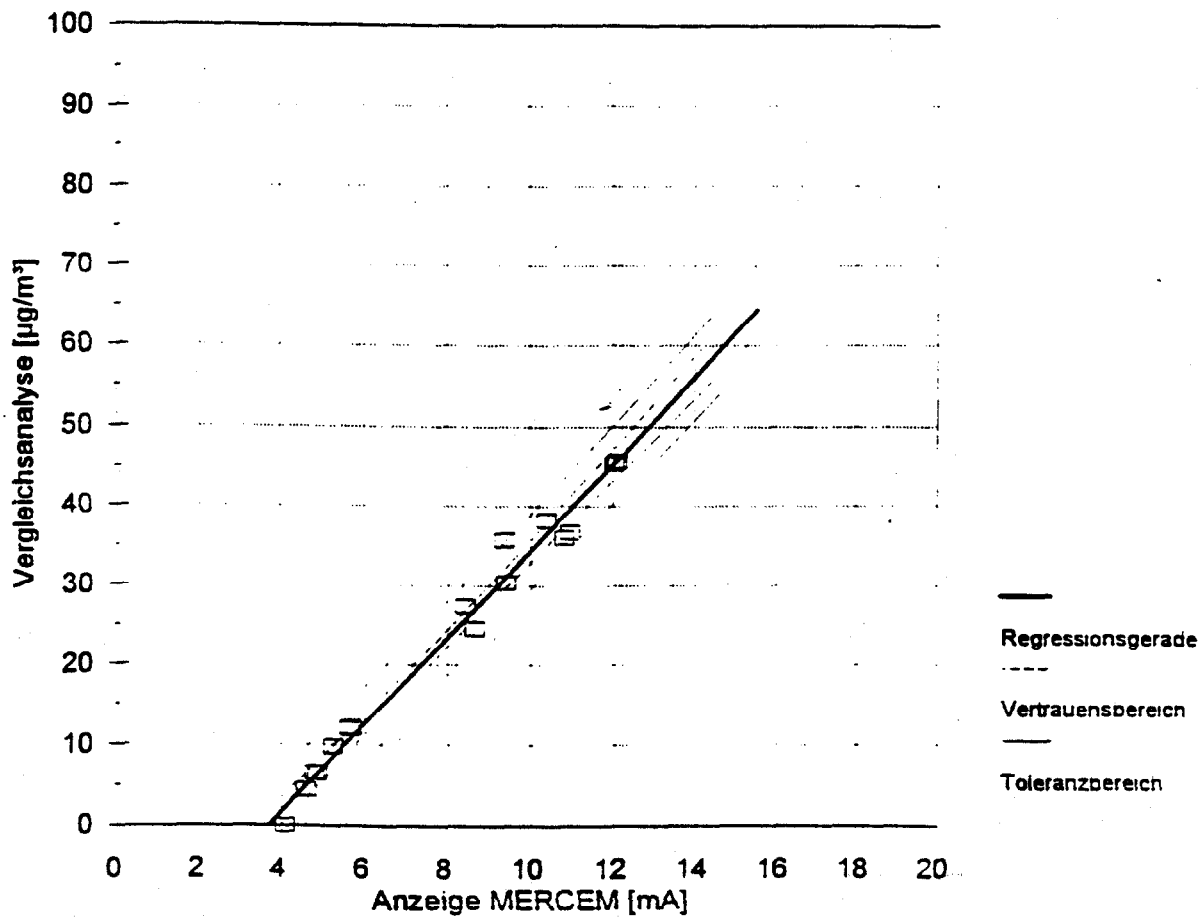


Fig. 6.4: Calibration curve (analysis function) for device 2 at the end of the long-duration test, measuring range 0 - 100 µg/m^3

6.3 Determination of the detection limits

The detection limits of both instruments were determined by sampling zero gas during the field test. The results are shown in table 6.2. These examinations were based on guideline VDI 2449, page 1. The detection limit should not exceed 2 % of fullscale (i.e. 100 µg/m³).

Table 6.2: Detection limits

		Device 1	Device 2
Number of values n		30	30
Average value of the zero values x	mA	4,01	4,09
Standard deviation of the values s	mA	0,04	0,06
Detection limit 3 · s	mA	0,11	0,19
Detection limit 3 · s	µg/m³	1,04	1,81
Detection limit	%fullscale	1,0	1,8

6.4 Reproducibility of the measuring values display

Two measuring devices were used to determine the reproducibility of the measured values, while the concentration of the object to be measured in the waste gas was simultaneously recorded. The measured values were evaluated as 2h-average values in the measuring range of 0 to 100 µg/m³ after the usual spot check selection for 3 classes.

The results are shown in table 6.3; the measured values are contained in appendix 10.3. The duplicate determinations are presented graphically in fig. 6.5. Class III could not be completed, because of the low emission level of the plant. Even when reducing the integration interval the measuring values could not be completed for Class III, as the measured values > 66 µg/m³ often exceeded > 100 µg/m³.

Table 6.3: Reproducibility R from duplicate determinations with both measuring devices, measuring range 0 to 100 µg/m³

Selection criteria		Number of pairs of values	R'	R
Class I	0 ... 33 µg/m³	50	99	33
Class II	33 ... 66 µg/m³	50	30	20
Class III	66 ... 100 µg/m³	29	31	31
Class I, II, III	0 ... 100 µg/m³	129	38	38

R' = referred to class fullscale

R = referred to fullscale

Reproducibility R = MBE / (s*t) with MBE = measuring range fullscale resp. class fullscale

t = Student-Faktor for 95% safety/reliability

s = Standard deviation from duplicate determination

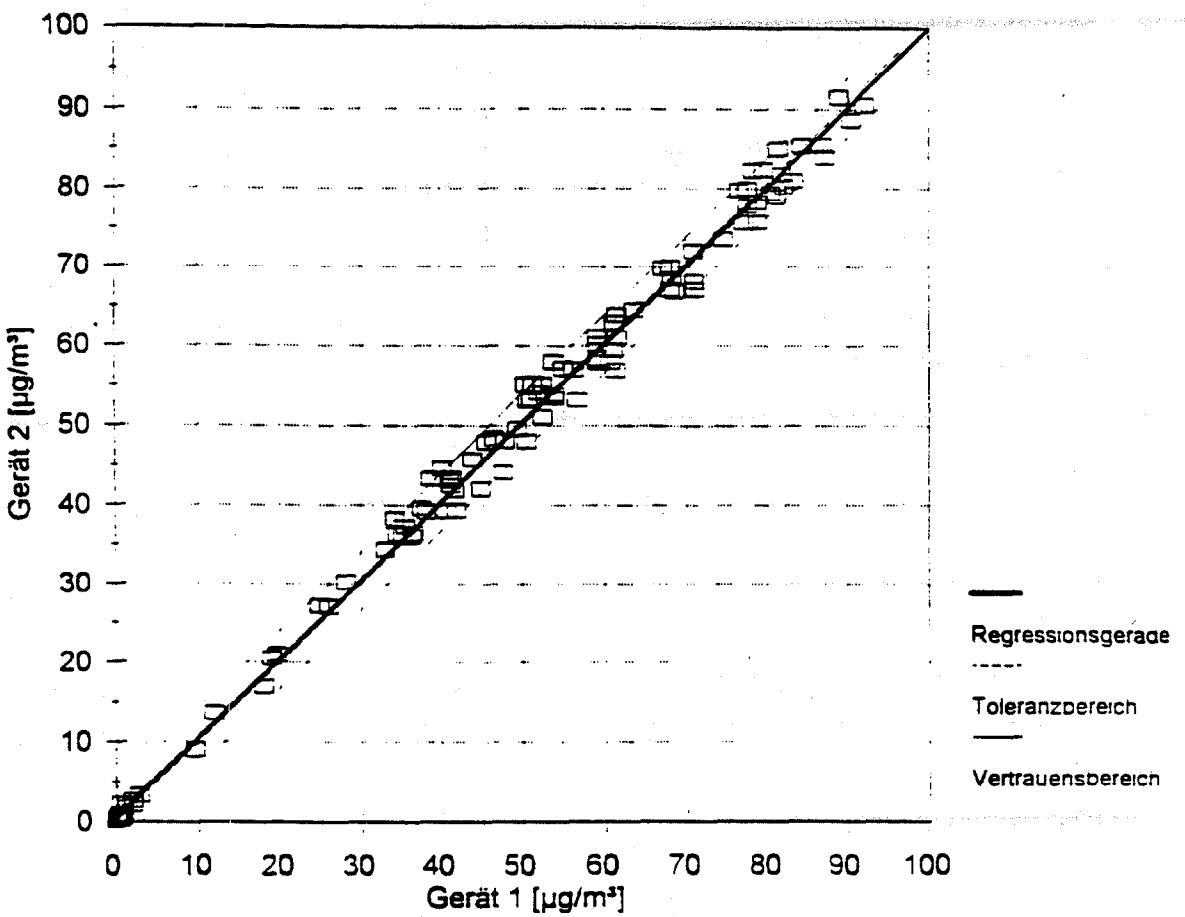


Fig. 6.5: Reproducibility of display of measuring values for the MERCEM instruments in the long-duration test.

6.5 Stability of Zero and Reference Point

During the weekly checks the zero and reference points of the instruments were verified regularly and corrected, if necessary, with the following results:

Drift of zero point within maintenance interval Device 1: < 1,0 % of fullscale
Gerät 2: < 1,0 % of fullscale

Drift of reference point within maintenance interval Device 1: < 2 % of required value
Device 2: < 4 % of required value

The stability of the reference point was verified by sampling test gas and by wet chemical reference analyses.

Within the maintenance interval of one month the maximum admissible changes of ± 2 % in the zero point and ± 4 % in the reference point were not exceeded.

6.6 Availability

According to the minimal requirements the availability of the measuring devices must be 90 %, at the aptitude test 95 % should be reached. The term "availability" describes the period of time in which usable measurement values are available for the evaluation of the emission properties of a plant.

Table 6.4 shows the availability values determined in the course of the long-duration test.

Table 6.4: Availability

		Device 1	Device 2
Total operating time	h	2154	2154
Instrument failure	h	9	12
Maintenance, Adjusment	h	18	18
Availability	%	98,7	98,6

6.7 Maintenance Interval

A maintenance interval of 1 month resulted from the long-duration test at the refuse incineration plant. The following maintenance work has to be carried out (expenditure of time 1-2 h):

- Visual check of the complete measuring system.
- Verification of zero point by sampling zero gas to the probe, clean sampling system, if necessary.
- Verification of the reference point of the measuring device by sampling test gas to the probe.
- Verifying the contents of the reaction solution and condensate container (once a week).
- Leakage test of the measuring system.

In addition to this, maintenance work as described in the manual has to be done. The maintenance interval may become smaller for plants with higher dust (< 5mg/m^3) and mercury contents (> 100 mg/m^3) and, if so, must be determined anew.

6.8 Function Test and Calibration

Function test and calibration of the MERCEM measuring system have to be performed according to VDI 3950 guideline, page 1 /July 1994). Special attention must be paid to the following points:

- The plant operator's test standards must be checked (calibrated Hg measuring instrument or wet chemical procedure).

- During the visual check of the instrument special attention must be paid to contaminations and deposits in the gas path of the 30 L/h range of the measuring instrument.

- The instrument zero point must be checked by dismantling the sampling probe and sampling zero air via the probe.

- Test gas sampling should likewise be carried out via the probe.

- Interference tests should be made using humid test gases.

- Wet chemical reference analyses must be performed at the function test.

7 COMPARISON TEST RESULTS AND MINIMAL REQUIREMENTS

Minimal requirements	Test result	Evaluation
1.1.1 The aptitude test has to be carried out with consideration to the definitions of the guideline VDI 2449, page 1 of February 1995, the DIN ISO 6879 standards of January 1984 and the DIN 43745 standard of February 1975.	The aptitude test was performed in compliance with the regulations and standards mentioned.	complies with the require-ments
1.1.2 Compliance with minimum requirements during the aptitude test should be ensured in the course of a long-duration test of at least three months duration. If possible, the long-duration test should be performed at one single site and during one single, continuous period. Shorter test periods or tests carried out at different test locations can only be considered to be part of the long-duration test in exceptional cases.	The test was performed at one single site. From Dec. 2, 1995 until Jan. 2, 1996 the sampling probe was, however, moved and mounted to another flue gas purification line of the same type and with identical waste gas conditions.	complies with the require-ments
1.1.3 During the aptitude test, the connection between the device indication/display and the value of the measured object in the waste gas (determined by means of a reference procedure, e.g. as a mass concentration, volume concentration or volume flow) had to be determined by means of regression analysis (Analysis function).	Calibration was performed with 30 reference measurements. A statistically secured correlation between the device indication/display and the mercury concentration in the waste gas was stated.	complies with the require-ments
1.1.4 The device settings should be protected against unauthorized or unintended mis-alignment during operation.	Due to the MERCEM design unauthorized or unintentional misalignment is not possible. In addition to this instrument operation is secured by a number code and can be locked.	complies with the require-ments
1.1.5 The position of the zero point should range between approx. 10 % or 20 %, the position of the reference point at approx. 70 % fullscale.	MERCEM is equipped with a 4-20 mA measurement value output. The position of the reference points is dependent on the selected test gas concentration.	complies with the require-ments

Minimal requirements	Test result	Evaluation
1.1.6 The display elements should be such that the displayed range can be adapted to the corresponding measuring job. As a rule, the displayed range should amount to 2.5 up to 3 times the valid emission threshold value.	The measuring range is adjustable to the required measuring job.	complies with the require-ments
1.1.7 Measuring devices should be equipped with a measuring signal output to which an additional display or recording device can be connected.	MERCEM is equipped with an analog output with optional 0-20 mA or 4-20 mA.	complies with the require-ments
1.1.8 Measuring devices should be able to transmit messages (status signals) on the current operating status to a subsequent evaluation system (readiness for operation, maintenance, disturbances).	MERCEM has the required status signals.	complies with the require-ments
1.1.9 the availability of measuring device should be at least 90 % in continuous operation, but should reach 95 % in the course of the aptitude test. (The term "availability" describes the period of time in which usable measured values are available for evaluation of the emission properties of a plant).	The availability was determined for both MERCEM instruments: It amounted to 98,7 resp. 98,6 %.	complies with the require-ments
1.1.10 The maintenance interval of the measuring devices has to be determined and indicated.	The maintenance interval is 1 month.	complies with the require-ments

Minimal requirements	Test result	Evaluation
1.1.11 The reproducibility $R = \frac{\bar{x}}{U}$ ($\bar{X}$ = final value of the measuring range, U = instability range according to VDI 2449, Page 1) has to be determined by means of repeat determinations. To this end, measurements must be performed at the same test location with two measuring devices of the same type.	Reproducibility was determined by duplicate determination with two MERCEM instruments of the same type at the same test location.	complies with the requirements
1.1.12 The aptitude test refers to the complete measuring device, including the sampling line, sample preparation, and data acquisition or data output. The manufacturer's operating instructions must be included in the aptitude test, as well.	During the long-duration test the complete system of the MERCEM monitoring system was examined including the sampling line and sampling flange.	complies with the requirements
1.1.13 Minimum performance requirements should be complied with, according to the nominal conditions of use, standard DIN 43745 of February 1975, measuring group II, listed below:		
a) Mains voltage	a) The influence of mains voltage fluctuations on the measuring signal was < 1,0 % at the zero and reference point.	complies with the requirements
b) Relative air humidity	b) and c):	complies with the requirements
c) Content of spray-water in the air	MERCEM is protected against spray water. An influence of the relative air humidity on the instrument function could not be stated.	complies with the requirements
d) Vibrations	d) In the course of the long-duration test no negative influences of vibrations on the instrument function could be stated.	complies with the requirements
e) Operational position	e) The measuring system must be installed in an upright positon.	complies with the requirements

Minimal requirements	Test result	Evaluation
1.1.14 If measuring devices with automatic operation test and adjusting functions are involved, the devices provided for this purpose must be included in the aptitude test. If the adjusting range is exceeded by ± 6 % of the indication range during the automatic correction, a status signal has to be returned.		does not apply
1.1.15 Multi-component measuring devices must comply with the requirements stipulated for each individual component and must be tested accordingly.		does not apply
1.4 Gaseous emissions		
1.4.1 General		
1.4.1.1 The detection limit of the measuring device should not exceed 2 % of the max. sensitivity range.	The detection limit of MERCEM was at 1,0 resp. 1,8 %.	complies with the require-ments
1.4.1.2 The admissible ambient temperature range is between +5°C and +35°C. It should reach -10°C and +55°C, according to DIN standard 43745 of February 1975, measuring device group II.	MERCEM can be used within a temperature range of +5 to +40 °C.	complies with the require-ments
1.4.1.3 Temperature-dependence of the zero point indication should not exceed ± 2 % of the indication range in the event of a change in the ambient temperature by 10K. Influences of the zero point due to temperature changes of the measured objects should be compensated for by means of suitable measures.	The admissible deviations of the zero point were not exceeded.	complies with the require-ments

Minimal requirements	Test result	Evaluation
1.4.1.4 Changes in the reference point indication, which are caused by temperature-dependent sensitivity, should not exceed ± 3 % of the nominal value after a change in the ambient temperature by 10 K within the admissible temperature range. Influences on the reference point, which are caused by changes in the temperature of the measured material should be compensated for by means of suitable measures.	The admissible deviations of the reference point were not exceeded.	complies with the require-ments
1.4.1.5 Interferences caused by cross-sensitivities to escort substances contained in the measured material and at the mass concentrations usually found in waste gases, should not exceed a total of ± 4 % of the indication range. If this requirement cannot be met, the influence of the interference component on the measuring signal should be taken into consideration by means of appropriate measures.	No relevant interference effects did occur.	complies with the require-ments
1.4.1.6 The response time (90%-time) of the measuring device, including the sampling system, should not exceed 200 seconds.	MERCEM supplies a measured value every 180 s. In case of a rapid change, the 90%-value is reached at the latest after the second measuring cycle (360 s).	einge-schränkt erfüllt
1.4.1.7 During the maintenance interval, the temporal change in the zero point indication should not exceed ± 2 % of the nominal value..	The temporal change in the zero point indication was detertmined in the laboratory and in the long-duration test. It amounted to max. 1 % of the indication range.	complies with the require-ments
1.4.1.8 Temporal changes in the reference point indication, which are caused by a change in the sensitivity, should not exceed ± 4 % of the nominal value during the maintenance interval.	During the field test the reference point was examined by means of manual reference measurements and with Hg(0) test gases. The temporal change of the sensitivity during the maintenance interval was stated as < ± 4 % of the nominal value.	complies with the require-ments

Minimal requirements	Test result	Evaluation
1.4.1.9 Sampling and sample preparation, with regard to the material and heating, must be implemented in such a way that satisfactory filtering of solid substances is ensured and transformations and memory effects by adsorption and desorption characteristics can be avoided to the greatest possible extent.	Sampling and sample preparation is designed in such a way that memory effects by adsorption and desorption characteristics are practically impossible.	complies with the require-ments
1.4.1.10 Reproducibility should not fall below a value of 30.	During the long-duration test a value of 38 was reached for reproducibility.	complies with the require-ments

8 SUMMARY

Bodenseewerk Perkin Elmer GmbH commissioned the TÜV Rheinland Sicherheit und Umweltschutz GmbH to conduct a pilot aptitude test of the MERCEM measuring device for gaseous mercury and its compounds.

The aptitude test was carried out according to the minimum requirements [1] and the test plan [2].

Based on the positive results that could be achieved, it is recommended that the measuring device be declared and announced as an aptitude-tested instrument as follows:

Total Mercury (gaseous)
MERCEM

Manufacturer	:	**Bodenseewerk Perkin Elmer GmbH, Meersburg**
Aptitude	:	**Plants according to the 17. BImSchV and TA Luft**
Smallest measuring range used in the aptitude test	:	**0 - 100 µg/m^3**
Remark	:	**- The response time (90%-time) of the measuring system amounts to max. 360 s (minimal requirements: 200 s).** **- During the annual function check manual reference measurements are required as long as no specified test gases can be used.**

Abteilung Luftreinhaltung

Dipl.-Ing. C. Röllig

Dr. P. Wilbring

Cologne, April 25, 1996
936-rö-hä

9 LITERATURE

[1] Bundeseinheitliche Praxis bei der Überwachung der Emissionen: Richtlinien über die Eignungsprüfung, den Einbau, die Kalibrierung und die Wartung von Meßeinrichtungen für kontinuierliche Emissionsmessungen.
(Circular letter of the German Federal Environmental Agency dated March 1, 1990, IGI-2-556 134/4-GMBl. No. 226/230)

[2] Länderausschuß für Immissionsschutz Unterausschuß Luft/Überwachung
Prüfkatalog für die Eignungsprüfung von Meßeinrichtungen für kontinuierliche Emissionsmessungen (Ausgabe: September 1994)

10 APPENDICES

10.1 Measured values for determination of the detection limit

10.2 Measured values of the calibration measurements

10.3 Measured values of reproducibility

10.4 Measured values of linearity test

10.5 Operating instructions

Appendix 10.1: Measured Values for Determination of the Detection Limit

Table 1: Detection limit and measuring threshold MERCEM
Measuring range 0 - 100 µg/m³ ≙ 4 - 20 mA

lfd. Nr.	Device 1 Hg µg/m³	Device 1 Hg mA	Device 2 Hg µg/m³	Device 2 Hg mA
1	-0,1	3,98	0,2	4,03
2	-0,2	3,97	0,1	4,02
3	-0,1	3,98	0,3	4,05
4	-0,1	3,98	0,1	4,02
5	0	4,00	0,3	4,05
6	0,1	4,02	0,6	4,10
7	0,2	4,03	1,6	4,26
8	-0,1	3,98	0,7	4,11
9	-0,1	3,98	0,1	4,02
10	-0,1	3,98	0,1	4,02
11	0,3	4,05	0,4	4,06
12	0,2	4,03	0,1	4,02
13	0,1	4,02	0,5	4,08
14	-0,2	3,97	0,4	4,06
15	-0,2	3,97	0,4	4,06
16	-0,1	3,98	0,4	4,06
17	-0,1	3,98	0,7	4,11
18	0,4	4,06	1,2	4,19
19	0,1	4,02	0,6	4,10
20	-0,1	3,98	0,5	4,08
21	0	4,00	0,3	4,05
22	0,1	4,02	1	4,16
23	-0,2	3,97	1,5	4,24
24	-0,2	3,97	1	4,16
25	-0,1	3,98	0,9	4,14
26	-0,2	3,97	0,8	4,13
27	0,3	4,05	1,1	4,18
28	0,4	4,06	0,3	4,05
29	0,2	4,03	0,4	4,06
30	0,8	4,13	0,6	4,10

Appendix 10.2: Measured Values of the Calibration Measurements

Table 2: Calibration data, MERCEM Device 1

Device 1		Device 1	
First Calibration		Second Calibration	
Measured Value	Reference Method	Measured Value	Reference Value
mA	µg/m³	mA	µg/m³
17,50	72,6	4,50	4,5
16,26	64,7	4,83	6,6
12,72	44,0	5,26	9,8
11,60	40,8	5,62	12,1
10,80	43,9	8,70	27,5
9,31	29,2	10,10	35,8
8,21	22,5	12,11	45,8
7,46	20,5	11,95	45,4
6,78	17,0	10,83	36,2
6,96	16,3	10,80	36,8
5,82	13,6	10,67	38,3
5,38	9,9	9,70	30,5
7,53	22,1	8,90	24,7
7,4	18,9	4,90	6,6
5,4	9,8	5,71	12,2
3,95	0,0	4,02	0,0
4,04	0,0	3,98	0,0

Table 3: Calibration data, MERCEM Device 2

Device 2		Device 2	
First Calibration		Second Calibration	
Measured Value	Reference Method	Measured Value	Reference Method
mA	µg/m³	mA	µg/m³
17,57	72,6	4,59	4,5
16,38	64,7	4,82	6,6
13,07	44,0	5,25	9,8
12,53	40,8	5,63	12,1
11,57	43,9	8,42	27,5
9,82	29,2	9,36	35,8
8,74	22,5	12,10	45,8
7,89	20,5	12,03	45,4
7,15	17,0	10,83	36,2
7,41	16,3	10,96	36,8
6,16	13,6	10,40	38,3
5,62	9,9	9,39	30,5
7,91	22,1	8,64	24,7
7,82	18,9	4,88	6,6
5,61	9,8	5,69	12,2
4,13	0,0	4,1	0,0
4,09	0,0	4,08	0,0

Appendix 10.3: Measured Values of Reproducibility

Table 4: Reproducibility, measured values for mercury

Class 1 (0-33 µg/m³)		Class 2 (33-66 µg/m³)		Class 3 (66-100 µg/m³)	
Device 1 µg/m³	Device 2 µg/m³	Device 1 µg/m³	Device 2 µg/m³	Device 1 µg/m³	Device 2 µg/m³
0,4	0,6	48,1	48,1	81,9	81,9
0,2	0,3	49,5	49,6	77,7	77,9
0,1	0,2	54,2	54,0	68,3	68,6
0,2	0,3	36,6	36,4	78,8	78,3
1,0	1,2	41,7	42,0	68,0	67,2
0,5	0,4	61,5	61,1	84,4	85,4
24,9	27,2	36,4	36,0	74,6	73,6
0,0	0,2	56,4	57,0	80,5	79,5
0,9	0,7	54,1	53,5	70,9	72,1
0,7	0,4	59,2	58,4	68,5	67,0
0,7	1,0	63,6	64,6	92,2	90,6
18,0	17,0	59,2	60,2	82,2	80,4
0,4	1,0	38,2	39,2	68,1	70,0
0,2	0,6	35,2	36,3	77,5	80,0
19,6	21,0	59,2	58,0	71,0	68,2
1,4	1,8	33,1	34,4	67,1	69,9
0,2	0,3	41,3	42,6	81,5	85,1
0,3	0,5	40,7	39,4	71,1	67,2
0,2	0,4	52,6	51,1	78,2	82,3
0,4	0,3	61,3	63,0	77,1	75,8
0,5	0,4	61,1	59,4	76,5	79,8
0,2	0,3	34,5	36,3	79,6	82,5
0,3	0,2	44,0	45,8	81,3	79,0
0,4	0,4	35,5	37,4	90,5	88,8
18,9	20,6	46,6	48,5	87,3	84,0
0,8	0,5	55,2	57,2	87,0	85,6
0,4	0,4	59,2	61,2	89,0	91,6
1,0	0,7	41,1	43,1	83,4	81,1
0,4	0,6	51,2	53,3	79,0	75,8
0,3	0,7	41,4	43,5		
9,6	9,0	37,6	39,7		
2,0	2,1	50,8	53,1		
2,1	2,1	61,6	64,0		
0,3	0,3	41,9	39,4		
0,6	0,2	52,5	55,2		
0,4	0,3	60,8	58,0		
3,0	3,4	51,4	55,3		
0,1	0,3	34,3	38,4		
12,0	13,7	54,0	58,0		
0,2	0,3	61,4	57,0		
0,2	0,3	40,2	44,7		
25,9	27,1	38,8	43,4		
28,1	30,2	50,5	55,2		
2,2	2,6	52,2	54,2		
0,7	0,7	45,7	48,0		
2,9	3,2	50,7	48,1		
0,7	0,8	47,8	44,1		
0,2	0,5	59,6	57,9		
1,0	1,3	56,9	53,2		
0,6	2,2	45,0	42,1		

Appendix 10.4: Measured values of the Linearity Test

Table 5: Recording of the instrument characteristics in the laboratory, MERCEM, measuring range 0,-.100 µg/m³

Device 1		Device 2	
Measured Value	Test gas concentration	Measured value	Test gas concentration
mA	µg/m³	mA	µg/m³
5,38	11,1 [1)]	5,58	10,4 [1)]
6,86	19,6 [1)]	7,17	19,7 [1)]
8,43	31,4 [1)]	8,74	29,1 [1)]
9,97	37,4 [1)]	10,35	40,0 [1)]
11,41	48,7 [1)]	11,89	49,1 [1)]
13,14	56,4 [1)]	13,47	59,3 [1)]
14,62	67,9 [1)]	14,29	67,3 [1)]
16,96	80,4 [1)]	14,38	66,1 [1)]
19,54	96,5 [1)]	14,59	68,8 [1)]
16,16	71,0 [2)]	16,77	79,1 [1)]
18,26	83,9 [2)]	19,30	99,2 [1)]
12,45	54,0 [2)]	5,74	9,6 [2)]
8,59	32,0 [2)]	8,10	24,0 [2)]
5,42	13,0 [2)]	12,14	49,6 [2)]
3,97	0	14,91	66,2 [2)]
4,02	0	18,29	84,8 [2)]
		4,06	0
		4,11	0

1) Hg (0)

2) Hg (II)

Appendix 10.5: Operating Instructions

APPENDIX E

REFERENCE METHOD DATA

Parameter	Units									
Run number		1	2	3	4	5	6	7	8	9
Sampling date	mm/dd/yy	09/16/1998	09/16/1998	09/16/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998
Time sampling started	hh:mm	13:00	16:12	17:56	08:31	09:52	11:18	12:45	14:54	16:54
Time sampling stopped	hh:mm	13:05								
Time sampling resumed	hh:mm	14:20								
Time sampling stopped	hh:mm	14:30								
Time sampli ng resumed	hh:mm	14:50								
Time sampling ended	hh:mm	15:35	17:12	18:56	09:31	10:52	12:18	13:45	15:54	17:54
Absolute stack gas pressure	in. Hg	29.30941	29.30868	29.31015	29.29574	29.29647	29.29794	29.29794	29.29794	29.29794
Average dry gas meter temperature	°R	538.04	542.46	543.79	537.13	542.38	544.63	546.83	543.88	544.04
Average pressure differential across orifice meter	in. Hg	0.1178309	0.0965074	0.1001225	0.0857843	0.080576	0.0698529	0.0696691	0.079473	0.0816176
Average sq. root velocity head of stack gas	in. H2O	0.3003715	0.2892368	0.2935572	0.2734314	0.2636003	0.2465845	0.244949	0.2629115	0.263745
Average stack temperature	°R	639.83	641.92	640.58	638.75	640.17	641	641.58	641.83	641.5
Barometric pressure	in. Hg	29.33	29.33	29.33	29.32	29.32	29.32	29.32	29.32	29.32
Carbon dioxide	vol %	5.37	6.83	7.47	5.57	7.4	6.83	6.93	6.87	6.17
Carbon monoxide	vol %	0	0	0	0	0	0	0	0	0
Nitrogen	vol %	83.83	84.67	84.36	86.3	83.93	83.97	84.3	84.3	82.93
Oxygen	vol %	10.8	8.5	8.17	8.13	8.67	9.2	8.77	8.83	10.9
Cross-sectional area of nozzle	ft2	0.0013283	0.0013391	0.0013326	0.0013283	0.0013326	0.0013429	0.0013391	0.0013326	0.0013456
Cross-sectional area of stack	ft2	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739
Dry gas meter calibration factor, Y		1.01798	1.01798	1.01798	1.01798	1.01798	1.01798	1.01798	1.01798	1.01798

Parameter	Units									
Run number		1	2	3	4	5	6	7	8	9
Sampling date	mm/dd/yy	09/16/1998	09/16/1998	09/16/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998
Orifice meter calibration factor, C		0.75037	0.75037	0.75037	0.75037	0.75037	0.75037	0.75037	0.75037	0.75037
Dry molecular weight of stack gas	lb/lb-mol	29.29	29.43	29.52	29.22	29.53	29.46	29.46	29.45	29.42
Water vapor in the gas stream, proportion by volume		0.5081455	0.5084212	0.5163028	0.5015338	0.5113177	0.5136929	0.4579179	0.4877485	0.486445
Percent of isokinetic sampling		100.0794	100.3912	100.6276	99.19052	100.7201	101.3802	94.04044	99.87516	101.4535
Wet molecular weight of stack gas	lb/lb-mol	23.55363	23.62012	23.57316	23.591	23.6349	23.57347	24.21205	23.86651	23.86644
Pitot tube coefficient		0.84	0.84	0.84	0.84	0.84	0.84	0.84	0.84	0.84
Average stack gas velocity	ft/s	20.76607	20.00084	20.29819	18.877	18.2013	17.05922	16.72871	18.08849	18.14115
Total sampling time	min	60	60	60	60	60	60	60	60	60
Weight of water condensed in impingers & silica gel	g	865.3	840.5	865.9	770.4	769.8	734.3	593.2	721.8	740.9
Volume of gas sample as measured by dry gas meter	dcf	40.1811	39.335	39.3569	36.723	35.637	33.824	34.307	36.822	38.003
Volume of gas sample corrected to std conditions	dscf	39.4909	38.3168	38.2489	36.1022	34.6893	32.7765	33.1101	35.7426	36.8803
Volume of water vapor in gas sample	scf	40.7989	39.62958	40.82719	36.32436	36.29607	34.62225	27.96938	34.03287	34.93344
Lab sample ID		A982670081	A982670082	A982670083	A982670084	A982670085	A982670086	A982670087	A982670088	A982670089
Customer sample ID		T-MERCEM1-1	T-MERCEM2-1	T-MERCEM3-1	T-MERCEM4-1	T-MERCEM5-1	T-MERCEM6-1	T-MERCEM7-1	T-MERCEM8-1	T-MERCEM9-1

Parameter	Units									
Run number		1	2	3	4	5	6	7	8	9
Sampling date	mm/dd/yy	09/16/1998	09/16/1998	09/16/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998
Total mass collected in permanganate	µg	1.48	0.781	4.59	5.01	8.11	6.29	1.05	9.21	2.15
Total mass collected in permanganate for calc	µg	1.48	0.781	4.59	5.01	8.11	6.29	1.05	9.21	2.15
Lab sample ID		A982670090	A982670090	A982670090	A982670090	A982670090	A982670090	A982670090	A982670090	A982670090
Customer sample ID		T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1	T-MERCEM-10-1
Measured train blank value for permanganate	µg	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Blank correction value for permanganate	µg	0	0	0	0	0	0	0	0	0
Lab sample ID		A982670091	A982670092	A982670093	A982670094	A982670095	A982670096	A982670097	A982670098	A982670099
Customer sample ID		T-MERCEM1-1A	T-MERCEM2-1A	T-MERCEM3-1A	T-MERCEM4-1A	T-MERCEM5-1A	T-MERCEM6-1A	T-MERCEM7-1A	T-MERCEM8-1A	T-MERCEM9-1A
Total mass collected in HCl rinse	µg	46.7	44.7	46.5	52.5	46.7	42.2	39.2	33	48.2
Total mass collected in HCl rinse for calc	µg	46.7	44.7	46.5	52.5	46.7	42.2	39.2	33	48.2
Lab sample ID		A982670100	A982670100	A982670100	A982670100	A982670100	A982670100	A982670100	A982670100	A982670100
Customer sample ID		T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A	T-MERCEM-10-1A
Measured train blank value for HCl rinse	µg	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Blank correction value for HCl rinse	µg	0	0	0	0	0	0	0	0	0

Parameter	Units									
Run number		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Sampling date	mm/dd/yy	09/16/1998	09/16/1998	09/16/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998	09/17/1998
Mass used to calculate emission	µg	48.18	45.481	51.09	57.51	54.81	48.49	40.25	42.21	50.35
Gas sample volume	dscf	39.4909	38.3168	38.2489	36.1022	34.6893	32.7765	33.1101	35.7426	36.8803
Oxygen concentration	%dry vol	10.8	8.5	8.17	8.13	8.67	9.2	8.77	8.83	10.9
Concentration @7% O2	µg/dscm	59.13666633	46.9480984	51.47277028	61.19534664	63.35609998	61.98617628	49.14351107	47.97620364	66.829914

Parameter	Units									
Run number		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Sampling date	mm/dd/yy	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/23/1998	10/23/1998	10/23/1998
Time sampling started	hh:mm	10:12	12:20	14:01	15:32	17:53	19:23	08:23	09:49	13:03
Time sampling stopped	hh:mm	10:52							10:25	
Time sampli ng resumed	hh:mm	11:33							11:00	
Time sampling ended	hh:mm	11:53	13:20	15:01	16:32	18:53	20:23	09:23	11:25	14:03
Absolute stack gas pressure	in. Hg	29.61353	29.61647	29.61426	29.61279	29.61353	29.61574	29.63206	29.63427	29.63427
Average dry gas meter temperature	°R	531.67	540.04	541.54	541.54	538.46	537.58	518.33	526.46	536.83
Average pressure differential across orifice meter	in. Hg	0.1102941	0.1232843	0.1121324	0.098652	0.1219363	0.1176471	0.1121324	0.1109069	0.0968137
Average sq. root velocity head of stack gas	in. H2O	0.3012926	0.2992182	0.3012748	0.2719757	0.3047255	0.2998661	0.3065478	0.2917011	0.2750601
Average stack temperature	°R	640.92	638.75	640.67	640.83	639.58	641.33	640.5	640.08	640.33
Barometric pressure	in. Hg	29.64	29.64	29.64	29.64	29.64	29.64	29.66	29.66	29.66
Carbon dioxide	vol %	6.4	6.1	5.37	7.73	6.3	6.5	7.27	7.27	7.17
Carbon monoxide	vol %	0	0	0	0	0	0	0	0	0
Nitrogen	vol %	84.23	84.57	83.5	84.2	84.28	83.8	84.3	84.13	84.13
Oxygen	vol %	9.37	9.33	11.13	8.07	9.42	9.7	8.43	8.6	8.7
Cross-sectional area of nozzle	ft2	0.0013283	0.0013456	0.0013326	0.0013391	0.0013283	0.0013456	0.0013391	0.0013456	0.0013283
Cross-sectional area of stack	ft2	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739	15.75739
Dry gas meter calibration factor, Y		0.99178	0.99178	0.99178	0.99178	0.99178	0.99178	0.99178	0.99178	0.99178

Parameter	Units									
Run number		1	2	3	4	5	6	7	8	9
Sampling date	mm/dd/yy	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/23/1998	10/23/1998	10/23/1998
Orifice meter calibration factor, C		0.72586	0.72586	0.72586	0.72586	0.72586	0.72586	0.72586	0.72586	0.72586
Dry molecular weight of stack gas	lb/lb-mol	29.4	29.35	29.3	29.56	29.38	29.43	29.5	29.51	29.5
Water vapor in the gas stream, proportion by volume		0.4990761	0.5023189	0.5095443	0.5065678	0.5048523	0.5000978	0.5013356	0.5056039	0.5112635
Percent of isokinetic sampling		97.23852	101.9524	98.92178	102.054	101.167	102.1939	97.26596	102.6756	104.0508
Wet molecular weight of stack gas	lb/lb-mol	23.70993	23.64828	23.54431	23.70388	23.63716	23.71288	23.73484	23.68911	23.61812
Pitot tube coefficient		0.84	0.84	0.84	0.84	0.84	0.84	0.84	0.84	0.84
Average stack gas velocity	ft/s	20.67158	20.5202	20.73863	18.66156	20.91748	20.57835	21.00769	20.00227	18.8932
Total sampling time	min	60	60	60	60	60	60	60	60	60
Weight of water condensed in impingers & silica gel	g	829.1	898.3	867.1	803.9	884.8	880	854.6	871.1	832
Volume of gas sample as measured by dry gas meter	dcf	40.08	42.776	40.942	38.43	42.3131	42.828	39.885	40.595	38.67
Volume of gas sample corrected to std conditions	dscf	39.2368	41.2446	39.3522	36.921	40.9164	41.4758	40.0797	40.1619	37.5003
Volume of water vapor in gas sample	scf	39.09207	42.35485	40.88377	37.90389	41.71832	41.492	40.29439	41.07237	39.2288
Lab sample ID		A983020156	A983020158	A983020160	A983020162	A983020164	A983020243	A983020245	A983020247	A983020249
Customer sample ID		T2-MERCEM1-1	T2-MERCEM2-1	T2-MERCEM3-1	T2-MERCEM4-1	T2-MERCEM5-1	T2-MERCEM6-1	T2-MERCEM7-1	T2-MERCEM8-1	T2-MERCEM9-1

Parameter	Units									
Run number		1	2	3	4	5	6	7	8	9
Sampling date	mm/dd/yy	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/23/1998	10/23/1998	10/23/1998
Total mass collected in permanganate		6.19	9.57	3.53	2.74	5.51	3.37	1.9	4.42	5.99
Total mass collected in permanganate for calc		6.19	9.57	3.53	2.74	5.51	3.37	1.9	4.42	5.99
Lab sample ID		A983020251	A983020251	A983020251	A983020251	A983020251	A983020251	A983020251	A983020251	A983020251
Customer sample ID		T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1	T2-MERCEM10-1
Measured train blank value for permanganate	µg	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Blank correction value for permanganate	µg	0	0	0	0	0	0	0	0	0
Lab sample ID		A983020157	A983020159	A983020161	A983020163	A983020165	A983020244	A983020246	A983020248	A983020250
Customer sample ID		T2-MERCEM1-1A	T2-MERCEM2-1A	T2-MERCEM3-1A	T2-MERCEM4-1A	T2-MERCEM5-1A	T2-MERCEM6-1A	T2-MERCEM7-1A	T2-MERCEM8-1A	T2-MERCEM9-1A
Total mass collected in HCl rinse	µg	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5
Total mass collected in HCl rinse for calc	µg	0	0	0	0	0	0	0	0	0
Lab sample ID		A983020252	A983020252	A983020252	A983020252	A983020252	A983020252	A983020252	A983020252	A983020252
Customer sample ID		T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A	T2-MERCEM10-1A
Measured train blank value for HCl rinse	µg	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5
Blank correction value for HCl rinse	µg	0	0	0	0	0	0	0	0	0

Parameter	Units									
Run number		1	2	3	4	5	6	7	8	9
Sampling date	mm/dd/yy	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/22/1998	10/23/1998	10/23/1998	10/23/1998
Mass used to calculate emission	µg	6.19	9.57	3.53	2.74	5.51	3.37	1.9	4.42	5.99
Gas sample volume	dscf	39.2368	41.2446	39.3522	36.921	40.9164	41.4758	40.0797	40.1619	37.5003
Oxygen concentration	%dry vol	9.37	9.33	11.13	8.07	9.42	9.7	8.43	8.6	8.7
Concentration @7% O2	µg/dscm	6.706633899	9.830174606	4.493407255	2.837695744	5.74953696	3.55503542	1.864580369	4.388070334	6.420579313

APPENDIX F

RAW FIELD DATA

MERCEM DEMONSTRATION TEST
DOE OAK RIDGE - TSCA INCINERATOR
1998

		DATE	9/12	9/13	9/14	9/14	9/15	9/15	9/15
DAILY INSPECTION LIST		TIME	15:55	8:05	8:30	13:30	10:20	16:45	20:15
		NAME	BAKER	BAKER	BAKER	BAKER	BAKER	BAKER	BAKER
	BURN SHEET	PC	1393	1394	1396	1397	1397	1397	1397
MERCEM	OBSERVE NORMAL TRACE ON MERCURY TREND	LCD	OK	OK	OVERRANGE	OK	OK	OK	OK - 9:45
	ALARMS - LCD Lower Left	LCD	OK	OK	OK	OK	AMB	OK	OK
	TEMPERATURE CONTROLLERS (red or yellow alarm lights)	Upper Panel	OK	OK	OK	OK	OK	OK	OK
	CHECK REAGENT LEVEL - (min 3")	Inside Cabinet	10.5"	10.25"	10"	9"	9"	9.75	8.75
	VERIFY REACTOR LIQUID IS CLEAR	Inside Cabinet	SLIGHTLY	SLIGHTLY	SLIGHTLY	CLEAR	CLEAR	CLEAR	CLEAR
	COOLER STATUS (normal - green flashing light)	Inside Cabinet	OK	OK	OK	OK	OK	OK	OK
	MERCURY CONCENTRATION - ug/m3	LCD - K-1	59.36	16.1	7300				
	SAMPLING VOLUME - ml	LCD - K-2	81.15	81.3			84.28		92.8
	FLOW - l/hr @ MEASURING	LCD - K-3	3.43	3.38	3.53		3.7		3.66
	ENERGY	LCD - K-4	89.0	91.62					79.5
	FLOW @ COOL DOWN L/HR			29.9					33.8
	SCR CONDITION RESERVOIR					3"	3.25"	3.25"	3.4"
UTILITIES	SCADA TERMINAL VALUE CORRELATES WITH MERCEM +/- 0.2	PC at left	OK	OK	NG	OK	OK	OK	OK
	INSTRUMENT AIR SUPPLY READING @ REGULATOR - (SET POINT 4.5 BAR +/- 2.0)	Right of MERCEM	4.7	4.7	4.5	4.6	4.1	4.1	4.6
	NITROGEN SUPPLY READING @ REGULATOR - (SET POINT 1.0 BAR +/- 0.25)	Right of MERCEM	1.0	1.0	1.0	0.8	0.7	0.7	0.7
	PERMEATION PURGE/STANDBY FLOWRATE AT FLOWMETER 20 L/HR	Right of MERCEM	15	15	15	15	15	15	15
	EXHAUST SAMPLE LINE (CLEAN AIR- BLACK HEATED LINE) SET POINT 250 Deg F	Right of MERCEM	250	250	249	250		250	250
	ISOLATE VALVE CLOSED	Right of MERCEM	OK	OK	OK	OK	OK	OK	OK
	TRAILER AMBIENT TEMP - THERMOSTAT - SET POINT 71 Deg F ± 3	Wall at Entrance	70	68	70	69		69	68
CALIBRATION									
	MERCEM ZERO		0.2	0.3/0.6		0.1		0.1	0.1
	PERMEATION TUBE: OPEN ISOLATION VALVE	Right of MERCEM	OK	OK				OK	OK
	CHECK PERMEATION WATER BATH AT 50 DEG C +/- 0.3 DEG C & WATER LEVEL	Right of MERCEM	49.9	50.0	50.0	50.0	50.0	50.0	
	FLOWMETER SETTING - 60 L/HR	Right of MERCEM	60	60		60			
	MERCEM READING - ug/m3	LCD	99.3			99.6			51.7
	ng		51.2			50.4			
	CALIBRATION GAS								
	CYLINDER #		90848	#114		ALL4			51.7
	VERIFY CYLINDER PRESSURE (PSIG)	Left of MERCEM	950	—					900
	FLOW GAS FOR A MINIMUM OF 4 CYCLES (APPROX 12 MIN)								
	WATER BATH FILL LEVEL 1" FROM TOP		OK						ADDED 1 QT

F-3

MERCEM DEMONSTRATION TEST
DOE OAK RIDGE - TSCA INCINERATOR
1998

		DATE	9/16						DATA 1
DAILY INSPECTION LIST		TIME	7:30	8:07	8:17	10:55	13:00	15:00	
		NAME	BAKER			BAKER	BAKER	BAKER	
	BURN SHEET #	PC	1398			1398	1400	1400	
MERCEM	OBSERVE NORMAL TRACE ON MERCURY TREND	LCD	OK			OK	OK	OK	
	ALARMS - LCD Lower Left	LCD	OK			OK	OK	OK	
	TEMPERATURE CONTROLLERS (red or yellow alarm lights)	Upper Panel	OK			OK	OK	OK	
	CHECK REAGENT LEVEL - (min 3")	Inside Cabinet	8.75"			8.75	8.75	8.75	
	VERIFY REACTOR LIQUID IS CLEAR	Inside Cabinet	CLEAR			CLEAR	CLEAR	SLIGHTLY YEL	
	COOLER STATUS (normal - green flashing light)	Inside Cabinet	OK			OK	OK	OK	
	MERCURY CONCENTRATION - ug/m3	LCD - K-1				4.7	4.10	4.0	
	SAMPLING VOLUME - ml	LCD - K-2	84.7			79.4	83.4	78.6	
	FLOW - l/hr @ MEASURING	LCD - K-3	3.38			3.51	3.54	3.50	
	ENERGY	LCD - K-4	81.66			79.8	80.04	80.39	
RED BALL TUBE SCALE	HEATED FLOWMETER - STACK BYPASS (%)	INSIDE CABINET			72	72	77	72	
	FLOW @ COOL DOWN L/HR					29.3	28.3	28.9	
	REAGENT COLLECTION RESERVOIR (in.)		3.25			3.5	3.5"	3.75	
	LEAK TEST - CABINET (Key FG)		< 2 4/MIN						
	RESULT		PASSED						
UTILITIES	SCADA TERMINAL VALUE CORRELATES WITH MERCEM ± 0.2	PC at left	OK			OK	OK	OK	
	INSTRUMENT AIR SUPPLY READING @ REGULATOR - (SET POINT 4.5 BAR +/- 2.0)	Right of MERCEM	4.7			4.8	4.7	4.8	
	NITROGEN SUPPLY READING @ REGULATOR - (SET POINT 0.9 BAR +/- 0.25)	Right of MERCEM	0.7			0.7	0.7	0.7	
	PERMEATION PURGE/STANDBY FLOWRATE AT FLOWMETER 20 L/HR	Right of MERCEM	15			15	15	15	
	EXHAUST SAMPLE LINE (CLEAN AIR- BLACK HEATED LINE) SET POINT 250 Deg F	Right of MERCEM	250			250	250	250	
	ISOLATE VALVE CLOSED	Right of MERCEM	OK			OK	OK	OK	
	TRAILER AMBIENT TEMP - THERMOSTAT - SET POINT 71 Deg F ± 3 DEG F	Wall at Entrance	68						
CALIBRATION									
	MERCEM ZERO PERM TUBE VOL		84.7						
	NG		51.0						
	PERMEATION TUBE: OPEN ISOLATION VALVE	Right of MERCEM	OK						
	CHECK PERMEATION WATER BATH AT 50 DEG C +/- 0.3 DEG C	Right of MERCEM	50.0			50			
	FLOWMETER SETTING - 60 L/HR	Right of MERCEM	60						
	MERCEM READING - ug/m3	LCD	100.07						
	WATER LEVEL IN ISOTEMP BATH (FROM TOP)		1/2"						
	CALIBRATION GAS								
	CYLINDER #		909909						
	VERIFY CYLINDER PRESSURE	Left of MERCEM	900						
	FLOW GAS FOR A MINIMUM OF 4 CYCLES (APPROX 12 MIN) ug/m3		53.2						
	PCC		.0013	.0013				.0001	
	SCC		.0023	.0023				.0017	
SCADA	AQV		0	0				.0007	
	SCRUBS		0.0040	0.000				0	
	TOTAL		.0077	0.0036				.0025	

RATA TESTING

MERCEM DEMONSTRATION TEST
DAILY INSPECTION LIST
DOE OAK RIDGE - TSCA INCINERATOR
1998

	9/16/98	DATE		RATA 1	RATA 2	RATA 3		9/17	RATA 4	RATA 6	RATA 8	RATA 9
		TIME		1500	1630	1530	2055	7:30	8:30	11:30	1455	
		LAST NAME		BAKER	BAKER	BAKER	BAKER	BAKER	BAKER	BAKER	BAKER	
TSCA	BURN SHEET #	SCADA PC		1400	1400	1400		1400	1400	1400	1400	
	PCC MERCURY FEED RATE (lb/hr)	SCADA PC		0	0	0		0		0	0	
	SCC MERCURY FEED RATE (lb/hr)	SCADA PC		0017	0019	0017		0020		0018	0020	
	AQU MERCURY FEED RATE (lb/hr)	SCADA PC		0007	0008	0008		0008		0008	0008	
	SOLIDS MERCURY FEED RATE (lb/hr)	SCADA PC		0	0	0		0				
					00							
	TOTAL MERCURY FEED RATE (lb/hr)	SCADA PC		0025	0027	0025	~~0007~~	0028	0028	0027	0028	
MERCEM	OBSERVE NORMAL TRACE ON MERCURY TREND	LCD		OK	OK	OK		AMB	OK	OK	OK	
	ALARMS - LCD Lower Left	LCD		OK	OK	OK		OK	OK	OK	OK	
	TEMPERATURE CONTROLLERS (red or yellow alarm lights)	Upper Panel		OK	OK	OK		OK	OK	OK	OK	
	CHECK REAGENT LEVEL - (min 3")	Inside Cabinet		8.75	8.75	8.5		8.9	8.9	8.9	8.8	
	CHECK REAGENT COLLECTION RESERVOIR LEVEL - (max 9")	Right of MERCEM		3.75	3.75	3.75		3.9	3.9	3.9	4.0	
	VERIFY REACTOR LIQUID IS CLEAR	Inside Cabinet		LT YEL	LT YEL	LT YEL		YEL	YEL	YEL	YEL	
	COOLER STATUS (normal - green flashing light) UN BLACK BOX TO CENTER	Inside Cabinet		OK	OK	OK		OK	OK	OK	OK	
	MERCURY CONCENTRATION - ug/m3	LCD - K-1		45.0	49.2	51.6		1.5 amb	10.3	55.7	45.9	
	SAMPLING VOLUME - ml	LCD - K-2		78.6	74.1	73.4		93.2		77.7	76.3	
	FLOW - (l/hr) while LCD indicates "measuring"	LCD - K-3		3.50	3.51	3.51		3.38		3.48	3.56	
	FLOW - (l/hr) while LCD indicaes "cool down"	LCD - K-3		28.9		26.9				28.5		
	ENERGY (%)	LCD - K-4		80.4	19.2	19.9		80.7		80.2		
	BYPASS PUMP FLOWRATE (%) - AS INDICATED BY RED BALL ON TUBE SCALE	Inside Cabinet		72	72	72		CAL	72	72	72	
	INTERNAL LEAK TEST - PRESS "F6" TO INITIATE - PASSES IF FLOW K-3 UNDER 2.0	LCD									0.6	
	$SnCl_2$ REAGENT - INERT N_2 Y6 - BAR										1.9	
	CONTROL AIR - PROBE VALVE Y7 - BAR										OK	
UTILITIES	SCADA TERMINAL VALUE CORRELATES WITH MERCEM +/- 0.2	PC at left		OK	OK	OK		OK	OK	OK	4.7	
	INSTRUMENT AIR SUPPLY READING @ REGULATOR - (SET POINT 4.5 BAR +/- 2.0)	Right of MERCEM		4.8	4.7	4.7		4.7	4.7	4.7	0.7	
	NITROGEN SUPPLY READING @ REGULATOR - (SET POINT 2 BAR +/- 0.25)	Right of MERCEM		0.7	0.7	0.7		0.7	0.7	0.7	15	
	PERMEATION PURGE/STANDBY FLOWRATE AT FLOWMETER 20 L/HR	Right of MERCEM		15	15	15		15	15	15	250	
	EXHAUST SAMPLE LINE (CLEAN AIR- BLACK HEATED LINE) SET POINT 250 Deg F	Right of MERCEM		250	250	250		250	250	250		
	ISOLATE VALVE CLOSED	Right of MERCEM		OK	OK	OK						
	TRAILER AMBIENT TEMP - THERMOSTAT - SET POINT 71 Deg F (+/- 3)	Wall at Entrance		69	69	70		69	69	69	69	
CALIBRATION												
	MERCEM ZERO						1	1.5				
	PERMEATION TUBE:	Right of MERCEM						50.0				
	CHECK PERMEATION WATER BATH AT 50 DEG C +/- 0.3 DEG C	Right of MERCEM						50.0				
	FLOWMETER SETTING - 60 L/HR	Right of MERCEM						60				
	MERCEM READING - ug/m3	LCD						103.6				
	MERCEM VOL (ML) K3	LCD						85.0				
	CALCULATED MASS (ng)											
	ISOTHERMAL WATER BATH WATER LEVEL (IN. FROM TOP)	Right of MERCEM						16"				
	CALIBRATION GAS	Left of MERCEM						90909				
	CYLINDER #	Left of MERCEM						825				
	VERIFY CYLINDER PRESSURE	Left of MERCEM						52.6				
	MERCEM READING - ug/m3	LCD										
	FLOW GAS FOR A MINIMUM OF 4 CYCLES (APPROX 12 MIN)											
	STACK TEMP							179			179	
	STACK O2							8.4			8.4	

F-5

ANALYZER OPERATING LOG
DOE OAK RIDGE - TSCA INCINERATOR
PERKIN ELMER -MERCEM sn#MER-111 September 15, 1998

DATE	TIME	BURN SHEET	CONDIT	COMMENTS	NOMINAL VALUE	HOURS STACK SAMPLE (APPROX	TOTAL FEED Hg (LB/HR) x 10000	STACK O2 (%)
TUE								
9/18/98	830			ARRIVAL, UNLOAD				
THU								
9/12/98								
	1830			INITIAL SAMPLES DRAWN FROM STACK				
	2035			SYSTEM LEFT SAMPLING AT STACK		5		
FRI								
9/11/98	835	1387		SYSTEM FOUND IN FULL OPERATION		22		
	1700			CALIBRATIONS				
	1950			Hg SPIKE OVER 300 UG/M3				
SAT								
9/12/98	815	1393		SYSTEM FOUND IN FULL OPERATION		21		
	900			DAILY CALIBRATIONS				
	1045			STACK SAMPLING				
	1420			SAMPLING STACK	50			
	1430			CLOCK SYNCHRONIZATION				
	1550			SAMPLING STACK				
				LEFT IN FULL OPERATION				
SUN								
9/13/98	810	1394		SYSTEM FOUND IN FULL OPERATION	30	22.5		
	820			DAILY CALIBRATIONS	ug/m3			
	930			SAMPLING STACK				
	1105			SYSTEM LEFT IN FULL OPERATION				
MON								
9/14/98	830	1296		ANALYZER OVER RANGED AT APPROX 7:45		7.75		
				SYSTEM IN STANDBY				
	1000			JEROME TESTING OF CAL GAS & PERM TUBE				
	2200			DAILY CALIBRATIONS				
	202			SAMPLING AT STACK	45	0.25		
TUE	1315			SAMPLING AT STACK	9	0.25		
9/15/98	1800			CE TEST CONDUCTED				
	1943			COMPLETED				
	2200			SYSTEM SAMPLING AMBIENT AIR				
WED								
9/16/98	730			DAILY CALIBRATIONS				
	818	1398		COMMENCE STACK SAMPLING	7			
	830			JEROME TESTING				
	910			LEAK TEST				
				SYSTEM ZERO				
	920	1398		SAMPLING AT STACK	6			
				WASTE FEED ADJUSTMENTS				
	1300	1400		RATA TEST STARTED				
	1303			WASTE FEED CUT-OFF - KILN ROTATION SWITCH FAILURE				
	1305			TEST STOPPED-AT END OF 5 MIN POINT				
	1420			RESTART RATA TEST #1	40		25	
	1430			RM SAMPLE TRAIN SHUT DOWN - HIGH VAC	42		25	
	1450			RESTART RATA TEST #1				
	1535			END OF RATA TEST #1	44.3		26	
	1612			START OF RATA #2	50		26	
	1712			END OF RATA #2	45		25	
	1756			START OF RATA #3	50		25	
	1856			END OF RATA #3	48		26	
	1930			START OF RATA #4				
	1935			RATA #4 ABORTED - LOST FEEDS				
	2109			SYSTEM IN STANDBY				
9/17/98	720			SYSTEM IN STANDBY - NO ALARMS				
	730			DAILY CALIBRATIONS				
	831			START OF RATA #4	60		28.5	8.4
	931	1400		END OF RATA #4	60		29	8.5
	952			START OF RATA #5				
	1005			FEED UPSETS -3 BRIEF LOSSES OF FEED APPROX 30 SEC				
	1052			END OF RATA #5	65		27	8.3
	1118			START RATA #6			28	
10/19/98	1415			ARRIVE AT PLANT				
				UNLOAD EQUIPMENT				
				SYSTEM INSPECTION				
				DAILY CALS				
10/20/98	815			SETUP FOR INTERFERENCE TEST				
				TOTAL HOURS OF STACK SAMPLING		**•78.75**		

CALIBRATION ERROR TEST
DOE OAK RIDGE - TSCA INCINERATOR
PERKIN ELMER -MERCEM sn#MER-111 September 15, 1998

DATE	TIME	MERCEM ZERO	Cylinder CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3		POINT AVG ug/m3		% DIFF	CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	Jerome ug/m3	CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	Jerome ug/m3
TUE 9/15/98			90848	ZERO-A1		4.07														
				ZERO-A2		4.05														
	1818			ZERO-A3		3.97		4.03												
25				SPAN-A1	900	76.58			77.03	1.35										
				SPAN-A2		77.47														
				SPAN-A3			AWAITING SAMPLE LINE TO REACH SET POINT													
	1902			ZERO-B1		4.1														
				ZERO-B2		5.9		5.23												
				ZERO-B3		5.7														
	1912			SPAN-B1	850	78.9			78.4	-0.41										
				SPAN-B2		78														
				SPAN-B3			AWAITING SAMPLE LINE TO REACH SET POINT													
	1928			ZERO-C1		4.94														
				ZERO-C2		5.08		5.01												
				ZERO-C3																
	1937			SPAN-C1	825	78.7			78.8	-0.93										
				SPAN-C2		78.9														
	1943			SPAN-C3			TEST COMPLETED AT 1943													
							3-POINT AVG	4.6	78.08											

LOCATION- STACK LEVEL 1 PROBE LOCATION

INJECTION POINT - SAMPLE LINE INLET/PROBE OUTLET
AMBIENT CONDITIONS: CLEAR, DRY, 74 DEG F

2nd CALIBRATION ERROR TEST
DOE OAK RIDGE - TSCA INCINERATOR
PERKIN ELMER -MERCEM sn#MER-111 October 20, 1998

	DATE	TIME	MERCEM ZERO	Cylinder CYL #	Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	CYL EXPECTE ug/m3	POINT AVG ug/m3	% DIFF		CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	Jerome ug/m3	CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	Jerome ug/m3
			0.04	amb		0.04	n/a			ABS DIFF UG/M3	% DIFF										
TUE	10/20/98	1636	1	94785	HIGH		250		320												
		1639	1		HIGH			**308**	320												
		1642	1		HIGH			**312**	320	310	8	2.5									
	25	1645	2	90848	MID		550		90												
		1648	2		MID			90.2	90	91	1.0	1.1									
			2		MID			91.9	90												
		1658	3	AMB	ZERO				AMB												
		1701	3		ZERO			1.7	AMB			LINED COOLED ON HIGH VOL UNHEATED AMBIENT									
		1704	3		ZERO			2.6	AMB	2.2	2.20										
		1710	4	90848	MID		550		90												
		1713	4		MID			89.1	90	91.6	1.6	1.8									
		1716	4		MID			94.2	90												
		1719	5	94785	HIGH		250		320												
		1722	5		HIGH			310	320	317	3	0.9									
		1725	5		HIGH			325	320												
		1728	6	AMB	ZERO				AMB												
		1731	6		ZERO			1.7	AMB	1.8	1.8										
		1734	6		ZERO			1.9	AMB												
		1737	7	94785	HIGH		250		320												
		1740	7		HIGH			313	320	313	7	2.2									
		1743	7		HIGH			313	320												
		1746	8		ZERO				AMB												
		1749	8		ZERO			2.5	AMB	2.4	2.4										
		1752	8		ZERO			2.4	AMB												
		1755	9		MID		500		90												
		1758	9		MID			90.2	90	90.5	0.5	0.6									
		1801	9		MID			90.8	90												
		1807			LOW		975	31.7	31	32	1.0	3.2									
		1810			LOW			32.4	31												
		1813			LOW			31.7	31												
		1816			AMB			2.1	AMB												

LOCATION- STACK LEVEL 1 PROBE LOCATION

INJECTION POINT - SAMPLE LINE INLET/PROBE OUTLET
AMBIENT CONDITIONS: CLEAR, DRY, 74 DEG F
BAR PRESS 29.42

PERKIN ELMER -MERCEM DEMONSTRATION TEST
DOE OAK RIDGE - TSCA INCINERATOR
9/8/1998

				CYLINDER# 90849			CYLINDER# 90909			CYLINDER# 90843			CYLINDER# 90913			CYLINDER# 94705			CYLINDER# 94785			
				Non-Certified Value: 78.2 ug/m3			Non-Certified Value: 46.5 ug/m3			Certified Value: 78.2 ug/m3			Certified Value: 48.5 ug/m3			Non-Certified Value: 6 PPB 50.88 UG/M3			Non-Certified Value: 30 PPB 254.4 UG/M3			
DAY OF WEEK	DATE	TIME	MERCEM ZERO (ug/m3)	Cylinder Pressure (PSIG)	MERCEM Reading ug/m3	Jerome ug/m3	Cylinder Pressure (PSIG)	MERCEM Reading ug/m3	Jerome ug/m3	Cylinder Pressure (PSIG)	MERCEM Reading ug/m3	Jerome ug/m3	Cylinder Pressure (PSIG)	MERCEM Reading ug/m3	Jerome ug/m3	Cylinder Pressure (PSIG)	MERCEM Reading ug/m3	Jerome ug/m3	Cylinder Pressure (PSIG)	MERCEM Reading ug/m3	Jerome ug/m3	
FRI	9/11/98	1700	0.16	1000	85.9		1000	57.5		625	85.3		425	53.7								
			0.18		85.4			57.4			86.2			53.4								
														53.5								
SAT	9/12/98	900	0.2	900	84.6		950	54.9		625	84.9		400	52.9								
			0.2		84.7			55.3			85.2			54.5								
SUN	9/13/98	830	0.3	875	86.4		925	55.5		610	87.1		375	56.2								
			0.6		86.8						87.8											
MON	9/14/98	1000		850		44			26			41	350		25							
						41			27			41			25							
						42																
MON	9/14/98	2200	0.1	850	81.8		925	52		600	81.8		325	51.8								
			0.05					52.8			82.8			53.9								
											82.1											
TUE	9/15/98	2000	0.1	850	78.1		925	51.7														
			-0.2		CE Test @Stack			53														
WED	9/16/98	800	0.6	Placed at stack for CE			900	51.5	19	550	81	32	325	52.4	21							
			0.5					53.2	20		81.7	32		51.5	22							
									20			32			23							
									21			32			21							
THU	9/17/98	750	1.5				875	52.6	17	530	82.1	27	325	53.1	16							
		1316						53.4	17			26		53.2	17							
									17			27										
FRI	9/18/98	840		825	82.8	66	875	54.8	49	630	84.0	66										
					84.1	66		55.9	50		85.9	64										
						68			50			66										
						68																
THU	10/8/98	1500	-0.19	725	84.0		860	58.8		520	90.5		325	60.3								
			-0.13		87.2			59.4			91.9			59.7								
MON	10/19/98	1600	-0.43	710	87.9		850	57.8		520	89.0		300	57.4		990	30.1		300	316		
			-0.41		88.9			56.3			89.7			58.5			30.2			328		
TUE	10/20/98	900	0.14	700	89.7		820	59.9		510	92.6		300	60.1		980	30.9		300	316		
			0.11		90.2			60.1			94.4			60.6			31.0			318		
WED	10/21/98	900	-0.2	500	92.4		820	60.5		500	93.1		300	58.9		930	32.1	ppb= 3.8	200	320		ppb= 38.1
			-0.2		92.5			60.8			92.5			61.4			32.6			321		
																	32.0					
							AFTER MANUAL AGGITATION	61.6														
								62.0														
THU	10/22/98	700	-0.20	500	91.8		780	61.1		500	92.3		300	59.2		900	31.3		150	293 PRESS LOW		
			-0.09		91.2			61.2			93.8			59.2			32.2					
FRI	10/23/98	700	0.00	480	90.8		780	61.2		480	90.8		280	60.3		850	31.4		150	SAVED FOR METHOD 101A		
			0.03		91.1			61.4			90.8			61.8			32.1					

PERKIN ELMER -MERCEM DEMONSTRATION TEST
DOE OAK RIDGE - TSCA INCINERATOR
Sep-98

	DATE	TIME	MERCEM ZERO	PERMEATION TUBE CAL					MERCEM (ug/m3)	Jerome ug/m3	
				MERCEM Flow (l/hr	MERCEM Vol (ml)	VICI exp ng/min	MERCEM ng	VICI exp ug/m3			
FRI	9/11/98	1700	0.16	50	139	50.0	61	60.0	73.1		
			0.18	47.5	132	50.0	57	63.1	71.8		
SAT	9/12/98	900	0.2	49.7	138	50.0	55.6	60.4	66.9		
			0.2	49.7	138	50.0	55.5	60.4	66.9		
SUN	9/13/98	830	0.3	30.2	84	50.0	55.6	99.3	110.3		
			0.6	30.2	84	50.0	55.8	99.3	110.8		
MON	9/14/98	1000								39	
										39	
										34	
										56	
MON	9/14/98	2200	0.1	30.5	84.7	50.0	50.8	98.3	99.9	51	
			0.05	30.8	85.5	50.0	50.6	97.4	98.7		
				30.7	85.3	50.0	51.2	97.7	100.1		
TUE	9/15/98	2000	0.1	29.1	80.8	50.0	51.8	103.1	106.8		
			-0.2	28.9	80.4	50.0	51.5	103.8	106.7		
WED	9/16/98	800	0.6	30.5	84.7	50.0	51.1	98.3	100.6	70	
			0.5	30.5	84.7	50.0	50.9	98.3	101.1	41	
										39	
										21	
										22	
										22	
THU	9/17/98	750	1.5	30.3	84.2	50.0	52.7	99.0	104.4	12	
				30.6	85.0	50.0	52.9	98.0	103.7	12	
		1316								13	
FRI	9/18/98	1200		32.0	87.3	50.0	50.6	93.8	96.9	37	
				31.7	87.4	50.0	51.0	94.7	97.4	37	
										36	
										36	
	10/8/98	1500	-0.19		111.2	50.0	57.8	74.9	86.64		Note bath level recently refilled.
			-0.12		110.5	50.0	56.6	75.4	85.3		Bath temp stable at 50 deg C
MON	10/19/98	1700	-0.22		123.8	50.0	60.8	67.3	81.9		ATM PRESS 29.36
			-0.17		125.4	50.0	58.3	66.4	77.5		
					124.0	50.0	56.8	67.2	76.4		
					123.7	50.0	56.3	67.3	75.9		
TUE	10/20/98	1030	0.14	45.5	124.3	50.0	56.8	67.0	76.2		ATM PRESS 29.55
					124.2	50.0	56.9	67.1	76.3		
					124.2	50.0	56.7	67.1	76.1		
WED	10/21/98	1300	-0.13		122.7	50.0	56.9	67.9	77.3		
			-0.08		121.8	50.0	56.5	68.4	77.3		
					122.8	50.0	56.7	67.8	76.9		
THU	10/22/98	745	-0.2	46.5	125.3	50.0	56.2	66.5	74.8		
			-0.1		125.7	50.0	56.3	66.3	74.7		
					125.3	50.0	56.2	66.5	74.8		
FRI	10/23/98	740	0.00		129.5	50.0	59.2	64.3	76.2		
			0.02		128.7	50.0	58.5	64.7	75.8		
						50.0	0.0	#DIV/0!			
						50.0	0.0	#DIV/0!			
						50.0	0.0	#DIV/0!			

INTERFERENCE TEST
DOE OAK RIDGE - TSCA INCINERATOR
PERKIN ELMER -MERCEM sn#MER-111 October 20, 1998

	DATE	TIME	Cylinder Cyl # Gas #1	Cylinder CYL # Gas #2	Conc Gas #2	Cylinder Pressure (PSIG)	Gas #1 Flow (ml/hr)	Gas #2 Flow (ml/hr)	MERCEM ug/m3	DIFF ug/m3	POINT AVG ug/m3	% DIFF
			Plt N2	Plt N2					0.07			
TUE	10/20/98	1208	Plt N2	n/a		n/a	200	0	-0.05			
	Baseline Hg		Plt N2	90848	90	700	100	100	39.8			
									39.9			
									40.2		40.0	
INT & N2	CO2	1245	Plt N2	AH-030932	19.999	1650	100	100	-0.01			
Hg & N2			Plt N2	90848	90	700	100	100	40			-2.25
INT & Hg		1322	CO2	Hg	90	690	100	100	40.7	0.7	40.90	
									41.0	1		
INT & N2	CO			13290-T	1000.8	1750	100	100				
Hg & N2												
INT & Hg			CO	Hg			100	100	41.7	1.7		
									42.2	1.0	41.9	#VALUE!
INT & N2	O2		O2	9266-P	20.898	1750						
Hg & N2		1430	Plt N2	Hg		640	100	100				
INT & Hg		1418					100	100	42.7			
							100	100	42.7			
INT & N2	SO2	1340	SO2	14300	1018	2150	100	100				
Hg & N2							100	100	42.2	2.2		#VALUE!
INT & Hg							100	100	42.4	2.4	42.3	
INT & N2	NO2		NO2	SX-322267	445	2050	100	100				
Hg & N2												
INT & Hg							100	100	40.5	0.5		
							100	100	42.2	2.2		
INT & N2	CL2		CL2	AAL-4513	20.94	1780						
Hg & N2												#VALUE!
INT & Hg							100	100	41.8	1.8	1.6	
							100	100	41.4	1.4		
INT & N2	HCl		HCl	A10665	533	1420						
Hg & N2												
INT & Hg		1410					100	100	41.3	1.3		
							100	100	41.4	1.4		
		1442	Hg with Plt N2 only						41.3			
									41.6			
INT & N2	H2O		H2O	n/a	24.5%	590	100	100			56 ml/hr	
Hg & N2									43.4			
INT & Hg							100	100	43.4			
							100	100	43.0			

CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	Jerome ug/m3

CYL #	Cylinder Value (ug/m3)	Cylinder Pressure (PSIG)	MERCEM ug/m3	Jerome ug/m3

LOCATION - Sample Pump Inlet

INJECTION POINT - SAMPLE pump inlet
AMBIENT CONDITIONS: CLEAR, DRY, 74 DEG F

RELATIVE ACCURACY TESTING RESULTS
DOE OAK RIDGE - TSCA INCINERATOR
PERKIN ELMER -MERCEM sn#MER-111 September 16, 1998

DATE	9/16	9/16	9/16	9/17	9/17	9/17	9/17	9/17	9/17
START	1400	1612	1756	831	952	1118	1245	1454	1654
END	1535	1712	1856	931	1052	1218	1345	1554	1754
RUN #	1	2	3	4	5	6	7	8	9
1	41.8	47.2	59.8	68.3	60.9	SYNCH	SYNCH	SYNCH	SYNCH
2	76.4	48.4	53.5	66.2	65.2	60	64	45	55
3	50.1	46.7	51.8	65.2	66.4	57.7	54.7	43.1	63.2
4	41.9	48.2	51.7	63.8	62.6	55.7	65.3	45.9	59
5	38.9	42.8	48.3	69.6	64	55.6	49.7	44.7	60.9
6	41	45.3	47.4	62.6	62.7	61.9	66	31.6	57
7	41.8	43.1	48.4	63	62.7	58.1	57.6	47.5	59
8	45.9	50	48	67	49.6	53.8	38.1	46.2	59.2
9	46.6	48.2	51.6	61.9	61.8	61.6	66.8	44	62.3
10	47.2	49.3	51.6	74.8	64.7	58	60	47.2	60.4
11	47.3	47.8	44.5	48.2	57.2	61.8	50.9	31.5	59
12	47.5	47.8	64	55.5	62.9	59.2	50.2	47.8	60.8
13	47.5	44.9	53.1	67	61.1	58.9	47.8	44	61
14	44.5	44	50	66.4	61.4	64.9	53.3	32.6	58.8
15	45	46.2	43.7	63.5	63.4	62	48.7	39.9	59.3
16	50.4	48.8	47.8	59.3	57.8	65.4	52	48.9	59.7
17	49	61.7	45.9	57.6	60.3	56.6	50.7	50.4	58
18	47.7	46.6	45.5	57.8	62.1	56.9	42.7	52.9	61.3
19	45.5	47.7	46.9	60.27	59.2	58.8	44.9	53.2	61.3
20	45.2	42.4	47.5	70.8	57.1	57.6	46.9	87.8	55.1
21	43.3					63.1	41	160.5	61.1
22	50.4								
SUM	1034.9	947.1	1001	1268.77	1243.1	1187.6	1051.3	1044.7	1191.4
AVG	47.0	47.4	50.1	63.4	62.2	59.4	52.6	52.2	59.6 TOTAL 493.8
POINTS	22	20	20	20	20	20	20	20	20 AVG 54.9 STD 6.3

Bar Press (in Hg)			29.26		29.23			
Stack O2 (%)			8.4	8.3	8.3	8.6	8.3	8.4
Feed - Hg (lb/hr)			0.0028	0.0027	0.0028	0.0027	0.0026	0.0025
							46.43	
							% DIFF	
Uncorrected for O2								
Uncorrecte for daily cal							-12.5027	

	ug/m3										AVG
MERCEM AVG	47.0	47.4	50.1	63.4	62.2	59.4	52.6	52.2	59.6	TOTAL 493.8	54.9
RM101A	59	47	51	61	63	62	49	48	67	507.0	56.3
DIFF (UG/M3)	12.0	-0.4	1.0	-2.4	0.8	2.6	-3.6	-4.2	7.4	13.2	1.5
%DIFF	20.3	-0.8	1.9	-4.0	1.3	4.2	-7.3	-8.8	11.1	17.9	2.0
abs DIFF(ug/m3)	12	0.4	1	2.4	0.8	2.6	3.6	4.2	7.4	34.4	3.8
Orsat Stk				8.13	8.67	9.2	8.77	8.3	10.3	53.4	5.9
CEM O2				8.4	8.3	8.3	8.6	8.3	8.4	50.3	5.6

RATA

RELATIVE ACCURACY TESTING RESULTS - PHASE II
DOE OAK RIDGE - TSCA INCINERATOR
PERKIN ELMER -MERCEM sn#MER-111 October 22, 1998

	22-Oct 1012 1153	22-Oct 1220 1320	22-Oct 1401 1501	22-Oct 1532 1632	22-Oct 1753 1853	22-Oct 1923 2023	23-Oct 823 923	22-Oct 949 1125	22-Oct 1303 1403
	1	2	3	4	5	6	7	8	9
1012	24.2	19.5		19.7	25.0	17.0	18.6	22.6	16.8
1015	24.3	17.3	16.5	16.6	18.8	15.9	16.6	22.5	20.9
1018	23.1	18.0	17.2	19.6	26	14.5	16.5	22.6	14.1
1021	**32.3**	17.2	16.8	19.6	14.1	10.1	13.0	21.5	16.2
1024	27.7	18.5	20.8	14.8	2.9	10.3	18.8	24.6	21.3
1027	25.3	**27.7**	4.1	18.6	14.5	15.9	18.0	20.4	16.3
1030	24.2	15.2	16.6	21.5	10.7	17.6	22.1	20.3	14.7
1033	**29.8**	13.0	16.4	19.4	10.8	17.2	7.1	14.9	15.3
1036	24.2	15.8	17.8	7.9	21.4	17	15.0	5.6	13.8
1039	20.1	23.8	18.6	19.5	21.5	19.2	17.4	2.3	15.5
1042	19.8	11.8	5	20.4	21.2	19.5	16.1	1.7	16.5
1045	11.6	18.8	15.6	17.2	19.1	18.9	16.8	1.6	16.3
1048	21.6	28.9	15	1.8	22.0	18.1	14.7	23.0	16.9
1051	34.0	18.5	5.5	14.7	22.7	22.1	16.2	18.3	19.9
1054	5.7	15.2	12.3	19.3	9.7	16.9	20.3	19.2	19.8
1057	13.1	18.5	12.2	21.8	11.5	18.4	21.8	23.0	19.6
1100	16.8	23.5	16.3	18.8	16.7	16.7	21.1	27.0	21.9
1103	8.1	19.4	8.6	18.1	17.7	14.4	21.8	25.1	21.8
1106	16.8	19.4	14.4	16.8	17.1	14.2	22.1	27.9	19.6
1109	5.1	21.5	11.3	19.9	17.7	17.3	23.6	27.5	21.3
1112	16.3	3.8	19.9	25.5	19.1	6.1		27.6	20.6
1115	17.8		21.1						
1118	21.5								
SUM	463.4	385.3	302	371.5	360.2	337.3	357.6	399.2	379.1
AVG	20.1	18.3	14.4	17.7	17.2	16.1	17.9	19.0	18.1 TOTAL 158.7
POINTS	23	21	21	21	21	21	20	21	21 AVG 17.6

restarts smooth restarts

Bar Press (in Hg)	29.62		29.6	29.6			29.77		29.65		
Stack	179		179	179			179		178		
O2 (%)	8.19		9	8.7			8.3-9.3				
Feed - Hg (lb/hr)	0.0012		0.0015	0.001			0.0013				
Stk Vel ft/sec							19.02				

ug/m3 AVG

AVG	20.1	18.3	14.4	17.7	17.2	16.1	17.9	19.0	18.1	TOTAL 158.7	17.6

DISTRIBUTION

1. M. Allen, HCET-FIU
2–6. R. Baker, Aldora Technologies
7. J. Bosch, EPA OAQPS
8. T. Brown, DOE-FETC
9. C. G. Davidson
10–14. J. E. Dunn, Jr.
15. N. French, Sky+
16. C. E. Frye
17. S. Gawarecki, ORRLOC
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46. I. Skegg, SICK AG
47. T. Tiesler, TDEC
48. S. Weeks, CMST
49. File—EMEF DMC—RC