CES Response to Veolia Incinerator Comments

Summary

Most of the issues raised in the comments were addressed in the Alternative Monitoring Petitions submitted by Eli Lilly and Company in 2005, particularly "Method 301 Evaluation of Candidate Conditional Methods" and more specifically Section 7.0 "Range of Method Application" which is presented in the Appendix of these comments. This appendix demonstrates the robustness of the Xact methodology and instrumentation over a broad range of source conditions. The remaining comments relate to the commercial viability of the Xact. CES conditions of sale are no different than those of other manufacturers of pollution control and monitoring instrumentation. These results plus those in the Appendix clearly show that the Xact has demonstrated its ability to operate accurately and reliably for extended periods of time under a wide range of stack conditions that includes those likely to be found in the Veolia stack, and that such monitors are commercially available from Cooper Environmental Services LLC.

1. Applicability to Veolia Incinerator

a. **Comment**:

#48 Comment: OTM 16 and 20 are site-specific methods and are not applicable to Veolia's operations. OTM 16 and 20 were written specifically for Eli Lilly and Co.'s (Lilly) source and instrument. While they would provide a starting place for another facility to develop their own site-specific method, one cannot assume that either could be used at another facility without significant modification. For example, the sample transport system in OTM 16 provides for a protocol for including the sample transport system into the instrument validation using either dynamic spiking of known gases or comparison to another method (instrumental or reference, M29, filters/tubes and Ohio Luminex, etc.). This was written specifically for the Lilly system using a Sigrist sampling system. It is not at all clear that an appropriate sample transport system could be designed and developed for Veolia's stack gas conditions. Lilly spent considerable time and resources in developing both the laboratory and stack data underlying these methods. It is also not clear whether this instrument would tie into Veolia's daily operations, alarms, and QA/QC procedures. It is likely that these two OTMs would need extensive work before they would be usable for the conditions in Veolia's stack.

In addition, there is a good chance that the current QAG will not be able to produce quantitative mass of metals in the ranges required to calibrate/test the Xact. The QAG operates as a dynamic spiking into the probe/sample loop of the Xact system. Operation of this unit is dependent on the type of probe used, the flow in the sample loop, and gas conditions. Lilly spent a year developing this device to present the mass of metals specific to its sample loop (flow/moisture, etc.).

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In addition, there is a good chance that the current QAG will not be able to produce quantitative mass of metals in the ranges required to calibrate/test the Xact. The QAG operates as a dynamic spiking into the probe/sample loop of the Xact system. Operation of this unit is dependent on the type of probe used, the flow in the sample loop, and gas conditions. Lilly spent a year developing this device to present the mass of metals specific to its sample loop (flow/moisture, etc.).

b. Response:

i. The above comment basically states that the demonstrated applicability of the Xact multi-metals CEMS (Xact) is based on site-specific testing that is not applicable to Veolia's incinerator without significant modifications; and that the QAG has not demonstrated an adequate range.

ii. Our general response to the site specific concern is summarized in the Appendix (Section 7.0), which discusses the broad range of sources and conditions to which the Xact has been successfully applied. It is important to emphasize that while these procedures were developed at Lilly's stack, there is nothing in them that is specific to Lilly's stack. Instead, they provide a general procedure that can be applied in many locations. In fact the Xact and its procedures have been successfully applied to coal combustion sources equipped with a baghouse and a wet scrubber under an EPA SBIR project with great success (EPA Contract Number EPD07026).

iii. Transport systems for the Xact have been developed for many different sampling conditions including at Lilly (downstream of a wet scrubber and a natural gas fired source), coal fired power plants - downstream of multiple control types including baghouses in combination with wet scrubbers, ESPs alone, and downstream of ESP's and wet scrubbers and with bituminuous, sub-bituminous and lignite fuels at munitions incinerators - Diesel fired sources downstream of a baghouse. Appropriate transport systems were installed at all of these locations.

iv. QAG: It is difficult to understand where the commenter came to the conclusion that "…there is a good chance that the current QAG will not be able to produce quantitative mass of metals in the ranges required …". Contrary to this statement, the QAG has been used to generate metals aerosols whose concentrations are traceable to NIST standards in the range of ng/m3 (used for the ambient/fence line Xact monitors) to mg/m3 used for stack monitors. This clearly covers the range required for any multi-metals CEMS based on the emission limits required as well as maximum concentrations expected. The QAG is broadly applicable to ambient as well as stack monitors and its application is independent of such conditions as the type of probe, probe flow or gas conditions. The QAG operating conditions can readily be adjusted to these variables in the field to deliver the concentration to meet the required permit range. The QAG can be mobilized and delivered for field work within days of receiving a purchase order for services.

2. Statement of Basis – A: Commercial Availability

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2. Statement of Basis – A: Commercial Availability

a. **Comment:**

EPA's statement that "Multi-metals CEMS are commercially available and have been demonstrated to be reliable for measuring mercury and other metal emissions from hazardous waste combustors" (Statement of Basis at 22) is incorrect or misleading. When the Statement of Basis was written, the Xact was offered by Pall Corporation. This is no longer correct. Pall no longer manufactures and sells the Xact unit. The marketing rights to the Xact have reverted to Cooper Environmental Services. It should also be noted that this instrument is not an instrument that is carried in inventory. If a facility wanted to purchase an Xact unit, Cooper Environmental Services has indicated that it would take them at least six months to build it. This is the time to build the instrument itself and does not include time to develop the site-specific sampling train or the time to develop the software interface between the instrument and the facility's control systems. The software development process (custom software for that site) would take several months after the instrument was installed. Only one Xact unit has been installed and operated for an extended period of time at only one hazardous waste combustor, the combustor at Evoniks Tippecanoe Laboratories. Due to frequent maintenance issues and equipment failure, that Xact unit is no longer operating. (CRWI 6-7)

b. Response:

i. The fact that the marketing rights to the Xact have reverted back to Cooper Environmental Services LLC (CES) has little to do with its commercial availability. CES is the company that developed and first commercialized the Xact series of monitors and is having no problem returning to this position. In fact, CES took its first new order for an Xact at the end of March of this year and will be shipping that Xact by the end of this month (May, 2013). The fact that CES may have given a longer shipping time shortly after CES was informed of the return of the Xact rights to CES would have been because of the uncertainties associated with this time period. Now that contracts are in place with supply vendors, the uncertainties have been greatly reduced and the delivery times reduced.

ii. The fact that Xacts are not carried in inventor is not unusual in contemporary business, particularly for high end environmental instruments for which the condition, metals, requirements, etc. may be somewhat different for each source. Three to six months is not uncommon. As noted above, the longer lead times were associated with the initial uncertainties shortly after CES was informed of the Pall decision.

iii. Once a purchase order is received, many of the fabrication tasks can be conducted in parallel/simultaneously. As such, components such as probe and transport lines are included in CES' time estimates and it isn't necessary to wait until the unit is delivered. Similarly, although the DAHS interface maybe site specific, the DAHS software development process could begin immediately as the Xact utilizes standard

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communication protocols (Modbus) and has fixed and known register locations for data.

iv. The Xact operated reliably for 6 plus years at Lilly Tippecanoe laboratories incinerator as well as a year under Evonik. It is our understanding that Evonik elected not to complete a required repair on the Xact because there was not a regulatory requirement to operate the instrument. Below are summaries of the Xact's XRF audit and RATA results during its operation at Lilly. (CRWI 6-7)

Xact Quarterly Audit results Results

Audit Year	Quarter	Audit Date	Flow Error	XRF Audit Error				
				Cr	As	Cd	Hg	Pb
2007	1	3/1/2007	3.04%	1.46%	2.18%	2.84%	1.75%	2.34%
	2	5/25/2007	3.13%	3.32%	0.13%	4.23%	2.60%	2.96%
	3	8/29/2007	0.68%	-3.74%	2.18%	-4.13%	2.36%	2.76%
	4	12/26/2007	1.42%	6.50%	0.36%	7.36%	4.18%	0.40%
2008	1	2/15/2008	NA	0.22%	0.11%	1.20%	-0.99%	0.87%
	2	3/17/2008	1.07%	1.93%	0.66%	1.82%	2.16%	2.34%
	3	5/20/2008	0.53%	2.96%	2.61%	3.30%	2.53%	0.68%
	4	10/20/2008	1.37%	1.77%	0.72%	1.48%	0.56%	1.06%
2009	1	1/7/2009	2.10%	0.60%	2.94%	1.52%	3.45%	0.46%
	2	5/6/2009	3.67%	1.49%	4.09%	1.07%	1.78%	0.30%
	3	7/9/2009	0.84%	2.00%	5.43%	2.78%	0.72%	0.22%
	4	NA	NA	NA	NA	NA	NA	NA
2010	1	1/12/2010	NA	1.60%	5.13%	0.45%	0.71%	2.11%
	2	4/25/2010	NA	1.61%	5.92%	1.12%	4.00%	0.96%
	3	7/13/2010	1.68%	-3.47%	-0.92%	-2.82%	-2.42%	-2.11%
	4	12/10/2010	NA	2.00%	0.82%	2.70%	1.83%	2.91%
AVE			**1.77%**	**1.35%**	**2.16%**	**1.66%**	**1.68%**	**1.22%**

NA - Data Not Available

Xact RATA Results

Year	Slope					
	Cr	As	Cd	Hg	Pb	Average
2006	0.83	0.90	0.85	0.82	0.85	0.85
2006 (Quarterly)	0.91	0.77	0.95	0.92	0.93	0.89
2007	0.84	0.82	0.88	0.84	0.81	0.84
2008	0.96	0.71	0.98	0.99	0.97	0.92
2009	0.96	0.99	0.99	1.10	1.00	1.01
2010	0.97	1.06	1.11	1.02	1.04	1.04
Average	**0.91**	**0.87**	**0.96**	**0.95**	**0.93**	**0.93**

In addition, similar CES multi-metal CEMS have been installed and operated on US Army hazardous waste incinerators.

3. Lack of Data Demonstrating Applicability at Veolia Moisture Conditions

a. **Comment:**

1 - The Statement of Basis states in subsection 4.0(D)(c): "Eli Lilly's stack gases at the Tippecanoe facility averaged approximately 8 percent moisture content and 140 degree F while the multi-metals CEMS was being operated. However, Pall Corporation has assured EPA that trial tests on its CEMS demonstrate that

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the unit can operate reliably at moisture contents above 40 percent. The only document in the record produced by EPA to support this statement is one email exchanged between Pall and EPA. EPA failed to consider the fact that Pall, which manufactures the multi-metals CEMS, has a financial interest to overstate the CEMS capabilities. No data or objective evidence exist in the record to demonstrate the Xact multi-metals CEMS can operate at moisture contents at or above 40 percent.

b. Response:

i. As noted in the Appendix (Section 7.0 of the Lilly AMP), the Xact has operated at a wide range of temperature and moisture conditions.

ii. It has operated successfully on saturated stacks at 125 to 140 degrees C (SBIR study and a coal fired power plant).

iii. Clearly, it should have no problem operating at the conditions in the Veolia stack.

4. Statement of Basis – B

a. **Comment:** 2- The Xact Multi-Metals CEMS is identified in the Statement of Basis as a system that uses reel-to-reel filler tape sampling technology followed by X-ray fluorescence analysis of metals in the deposit. The Statement of Basis represents that this is the only multi-metals CEMS technology currently commercially available. Pall Corporation is identified in the Statement of Basis as the marketer for the Xact multi-metals CEMS. The Statement of Basis discusses the use of a Xact CEMS at the former Eli Lilly incinerator. Eli Lilly's incinerator and Veolia's Unit 3 are not comparable. Most incinerators including the Eli Lilly incinerator employ wet scrubbers as their pollution control equipment or a combination of wet scrubbers and baghouses. The off gases from incinerators using wet scrubbers have similar moisture and temperature ranges. The suppliers of die Xact multi-metals CEMS claim that the Xact analyzes the off gas emitted from an incinerator to determine the amount of metals in the emissions stream. Unlike these wet scrubber systems, Veolia operates a dry pollution control system on Units 2, 3 and 4 of its Sauget facility. Dry systems are unique and to my knowledge the only other exclusively dry system in the United States is the Clean Harbors incinerator in Kimball, Nebraska. Veolia's dry pollution control systems operate at much higher moisture and temperature ranges than wet scrubber systems. The Xact multi-metals CEMS has never been demonstrated to successfully operate in the high moisture and high temperature environment presented by Unit 3.

b. Response:

i. As noted in the Appendix (Section 7.0 of the Lilly AMP), the Xact has operated at a wide range of temperature and moisture conditions.

ii. More specifically, the Xact has demonstrated operation at temperatures up to about 500 degrees F (demilitarization incinerator) and in flue gases

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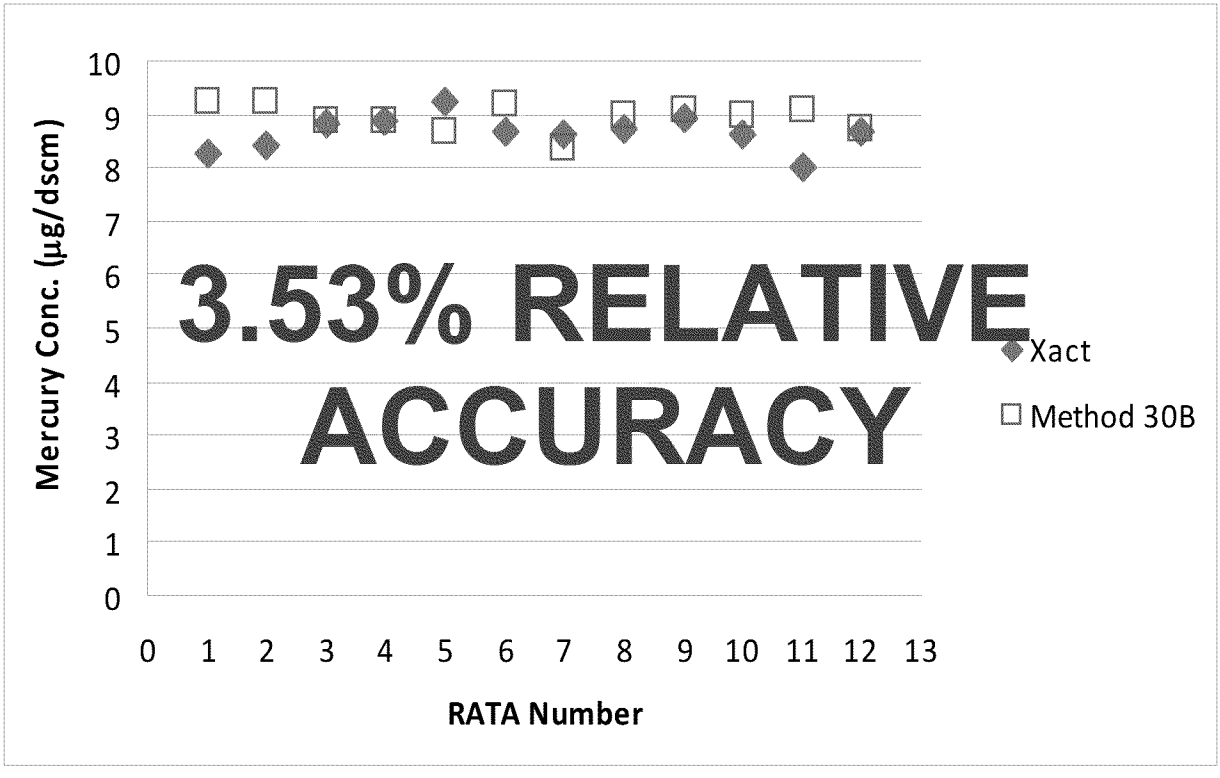
saturated at 140 degrees F.

iii. Below in the following figure, are the results of a comparison of mercury results reported by EPA reference method 30B and the CES Xact. This data was collected at a coal fired utility boiler operating with dry controls at about 350 degrees F.

iv. These results plus those in the Appendix clearly show that the Xact has demonstrated its ability to operate accurately and reliably for extended periods of time under a wide range of stack conditions that includes those likely to be found in the Veolia stack.



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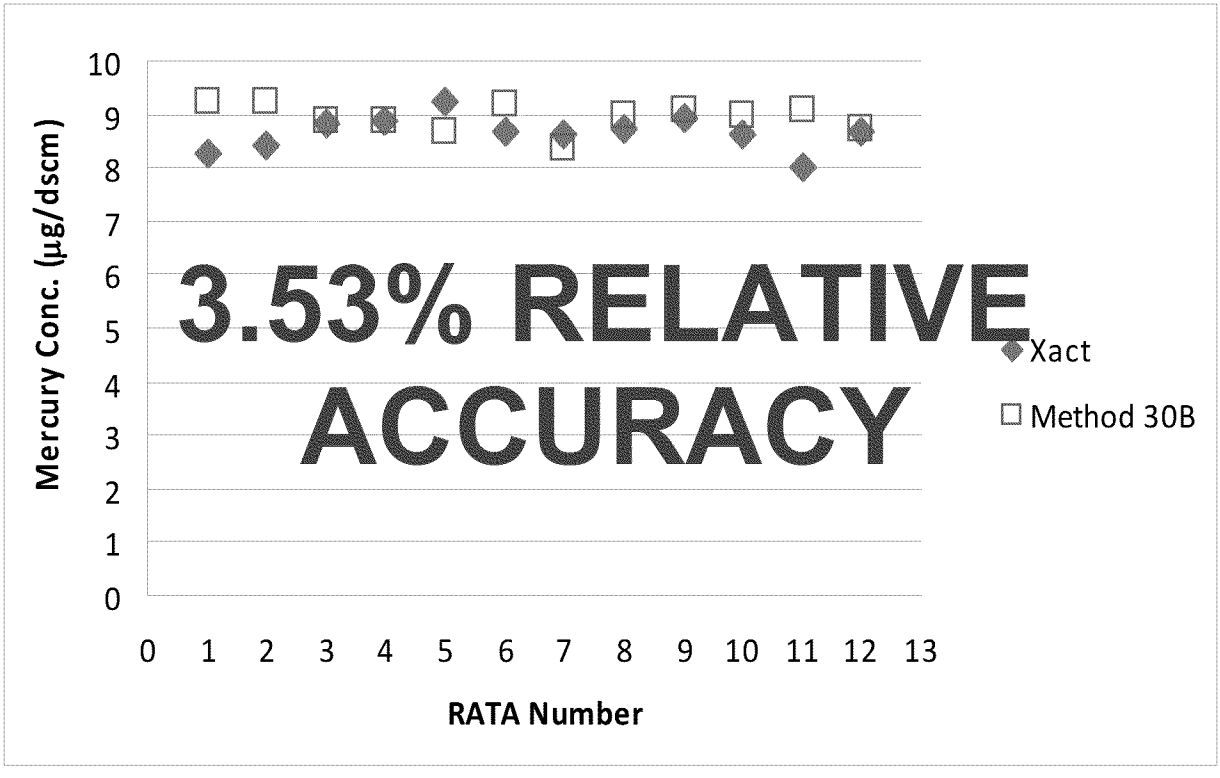

Appendix

7.0 Range of Method Application

7.1 Overview

The results from the preceding section clearly demonstrate the ability of the XFM and Xact-IAP to accurately and precisely quantify metals in stack emissions over a broad range of concentrations, and that these methods are applicable to the quantitative measurement of the five validated metals in hazardous waste incinerators. The question then arises as to

What other source categories and elements is this technology applicable?

The answer to this question lies in how key features of the sampling and analysis procedures depend on and respond to changes in emission characteristics associated with different source categories. The EPA suggests in Method 301 that this might be addressed in part by conducting a series of ruggedness tests, which are laboratory tests of a method's sensitivity to key method variables[1,29,30]. This type of evaluation of the XFM and Xact-IAP was conducted during Phase I laboratory testing. In addition, a substantial body of previous laboratory and field test data on this reactive-filter based technology is also available that supports the general applicability of these methods to most sources and elements.

The above tests and data are summarized in this section along with key aspects of the XFM and Xact-IAP methods, and relative characteristics of emissions. The objective of this review is to demonstrate that these methods should be considered generally applicable to a wide range of emissions and elements including those from regulated sources. This review is presented in the following subsections:

- Method Applicability (7.2)
- Key Sampling and Analysis Features (7.3)
- Key Emission Characteristics (7.4)
- Filter Trapping Efficiency for Vapor Phase Metals (7.5)
- Applicable Concentration Range (7.6)

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What other source categories and elements is this technology applicable?

The answer to this question lies in how key features of the sampling and analysis procedures depend on and respond to changes in emission characteristics associated with different source categories. The EPA suggests in Method 301 that this might be addressed in part by conducting a series of ruggedness tests, which are laboratory tests of a method's sensitivity to key method variables[1,29,30]. This type of evaluation of the XFM and Xact-IAP was conducted during Phase I laboratory testing. In addition, a substantial body of previous laboratory and field test data on this reactive-filter based technology is also available that supports the general applicability of these methods to most sources and elements.

The above tests and data are summarized in this section along with key aspects of the XFM and Xact-IAP methods, and relative characteristics of emissions. The objective of this review is to demonstrate that these methods should be considered generally applicable to a wide range of emissions and elements including those from regulated sources. This review is presented in the following subsections:

- Method Applicability (7.2)
- Key Sampling and Analysis Features (7.3)
- Key Emission Characteristics (7.4)
- Filter Trapping Efficiency for Vapor Phase Metals (7.5)
- Applicable Concentration Range (7.6)

- Deposit Stability (7.7)
- Moisture (7.8)
- Holding Times (7.9)
- Other Related Tests (7.10)
- Applicable Elements (7.11)
- Sensitivity to Reactive Stack Gases (7.12)

The details of this review are presented in Appendix L and supporting reports are provided in Appendix M. The conclusion of this review is that there is a substantial body of information about the chemistry and physics of the methods and emissions as well as specific test data covering some of the more challenging of conditions that strongly supports the extension of these methods to other regulated source categories and elements.

7.2 Criteria for Candidate Method Applicability

It is clear that when a candidate method meets the requirements of Method 301, it is valid for the source tested, and may be applicable to the specific source category. Method 301 recommends that additional test data be submitted to extend the applicability of the candidate method to other source categories. This recommendation, however, does not specify criteria that can be used to evaluate possible extension of a method's applicability when direct test data is not available. For the following discussion, it will be assumed that the candidate methods will be applicable to an untested source category if differences in emission characteristics between sources tested and candidate source emissions are such that they are unlikely to contribute significantly to imprecision and bias in measured metal concentrations.

7.3 Key Sampling and Analysis Features

Both the XFM and Xact-IAP draw a representative sample of stack gas through a filter matrix where metals are concentrated prior to analysis. The XFM draws the stack gas through an upstream PTFE filter that removes PM from a gas stream and then through a downstream reactive filter that traps vapor phase metals. The Xact-IAP, on the other hand, uses a single reactive filter to trap both the PM and vapor phase metals. Both methods measure the mass of each metal deposited on the filter(s) using XRF analysis procedures based on EPA's IO Compendium Method 3.3[14]. The concentration of metal in stack gas (C) is calculated by dividing the mass of metal in the filter(s) deposit (M) by the volume of stack gas (V) that passed through the filter(s). That is;

$$C = M / V \qquad \textbf{Equation 26}$$

Key sampling and analysis features are thus those that contribute to the quantitative accuracy of the metal mass and volume determinations. Consequently, a method will likely be applicable to a particular source emissions category if the differences in stack gas characteristics are such that they do not significantly contribute to inaccuracies in volume and mass measurements.

- Deposit Stability (7.7)
- Moisture (7.8)
- Holding Times (7.9)
- Other Related Tests (7.10)
- Applicable Elements (7.11)
- Sensitivity to Reactive Stack Gases (7.12)

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Key sampling and analysis features are thus those that contribute to the quantitative accuracy of the metal mass and volume determinations. Consequently, a method will likely be applicable to a particular source emissions category if the differences in stack gas characteristics are such that they do not significantly contribute to inaccuracies in volume and mass measurements.

In the case of volume determination, both methods dilute, cool, filter, and dry the stack gas as well as denude the gas of corrosive species such as acids, SO_2, etc. prior to volume determination. This dilution and extensive conditioning of the stack gas prior to volume determination makes it relatively insensitive to differences in stack gas characteristics. Thus, if differences in stack gas characteristics are to significantly impact the accuracy of the measured aerosol concentration, it must be through the metal mass determination.

Accurate determination of metal mass depends on two key factors, which may depend on stack gas characteristics:

- XRF analytical determination of metal mass in the filter deposit
- Filter trapping efficiency

Again, it is important to note that both methods use dilution to maintain and control such sampling conditions as temperature, dew point, and PM concentration in their optimal range. As such, the method's sensitivity to specific stack gas characteristics is significantly reduced even for these two factors. For example, when applying these methods, the applications engineer is directed to adjust non-critical parameters within specified limits such as dilution ratio and sampling time to optimize the method for a particular application. Furthermore, since there is no direct interaction between stack gas and the analysis, the XRF determination of metal mass in the filter deposit is only indirectly dependent on the characteristics of the stack gas through how the stack gas impacts the characteristics of the deposit on the filter. Other than the analyte metals of interest, the only species deposited on the filter are non- analyte PM species, the total mass of which is controlled through the method application by selecting optimal sampling times and dilution ratios. None of the major components of stack gas are trapped on the filters and cannot therefore interfere with the XRF analysis.

For this reason, the only factor that might be sensitive to stack gas characteristics is the filter trapping efficiency for PM and vapor phase metals. Filter trapping efficiencies for PM are well established and generally accepted to be greater than 99%[23,31] are not expected to be dependent on differences in stack gas characteristics. As such, filter trapping efficiency for vapor phase metals is the key factor that might be impacted by stack gas characteristics. The primary focus of the next two sections is on the potential dependence of vapor phase metal trapping efficiency on flue gas conditions and the available data showing the range within which the method has demonstrated acceptable quantitation.

7.4 Key Emission Characteristics

Characterization of emissions depends on such factors as source type (combustion, process, fuel type, etc.), type of emissions (ducted or fugitive) and type of controls (filtration, electrostatic precipitators, scrubbers, absorbers, reactive, etc.). However, from the method applicability evaluation perspective, the characteristics of emissions that are most relevant are those defined in terms of their physical (primarily temperature) and chemical (composition) characteristics. Emissions are generally composed of varying amounts of air (N_2, O_2), combustion products (NO_x, SO_x, CO_2, CO, H_2O, etc.), contributions from controls (Na, Ca, H_2O, NH_3, etc.) and various process species, and can be further divided into categories such as PM, reactive and non-reactive gases, acid vapors, etc.

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As discussed above, the filters used by the XFM and Xact-IAP have a high PM metals trapping efficiency regardless of the stack gas physical and chemical characteristics when sampling within the conditions specified by their SOPs. Some potential exists, however, for the vapor phase collection efficiency to be impacted by the stack gas physical parameters and chemical composition. For both the XFM and Xact-IAP, the sampling temperature, flow rate, and pressures are controlled by the operator within the bounds of the SOP and are independent of the source type. Thus, the key characteristics of emissions that might impact the reactive filter's trapping efficiency are the sampling interferences caused by reactive gases such as NO_x, SO_x, NH_3, Cl_2, HCl, HNO_3, and H_2SO_4. Although this list is not complete, it does represent the more abundant and more reactive species likely to be present at varying concentrations in most stacks of regulatory interest. Oxygen was not listed, since it is expected to be present in most emissions at ambient concentrations (21%) or less, and laboratory tests in Phase I demonstrated high trapping efficiency at the highest likely O_2 concentration. Potentially interfering reactive gases are listed below in Table 23 along with measured stack gas concentrations during supplemental testing discussed below and in Appendix L. Although this list is incomplete, the concentrations and conditions are representative of these sources and cover a broad range of concentrations and temperatures.

Table 23. List of stack conditions for which Xact-IAP and XFM tests have been conducted

Species		Phase II Tippe, IN	CFPP-CES Boardman, OR	CFPP-EPA Midwest	HWC - TEAD Tooele, UT
CO, dry	ppm	14	< 10	NA	10
O_2, dry	%	11	4.5	12-15	14-15
CO_2, dry	%	6	12.5	4-8	5
NO_x, dry	ppm	110	200	70	1200
SO_2, dry	ppm	ND	225	20-35	10
HCl (equiv.), dry	ppm	2	NA	NA	50
H_2O	%	9	10	15-20% NA	5
PM @ 7% O2	mg/dscm	4	27	160	10
Temp	°F	170	300		450

NA: Not Available
ND: Not Detected
HWC-TEAD: US Army sponsored tests at demiliterization HWC
CFPP-CES: CES sponsored collection efficiency test at PGE CFPP
CFPP-EPA: EPA sponsored test of Hg monitors

7.5 Filter Trapping Efficiency for Vapor Phase Metals

The Clean Air Act lists 11 metals as air toxics: Sb, As, Be, Cd, Cr, Co, Pb, Mn, Hg, Ni and Se[10]. Of these metals, Hg is the only one that is predominately in the vapor phase at the operating temperature of the Xact-IAP and XFM (180°F). For this reason, Hg is the most challenging to trap by filtration and most sensitive indicator of vapor phase metal trapping efficiency. Over the past several years, three series of Hg trapping and relative accuracy tests have been conducted in addition to Phase I and II tests described in the first part of this report. Whereas Phase II tests demonstrated high Hg trapping efficiencies under conditions more representative of emissions from modern hazardous waste incinerators, these additional tests focused on emissions from coal-fired boilers. The supplemental tests were more challenging in part because of their lower Hg concentrations (<10 μg/dscm) but also because of the presence of higher concentrations (Table 23) of potentially interfering species such as

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SO_2, dry	ppm	ND	225	20-35	10
HCl (equiv.), dry	ppm	2	NA	NA	50
H_2O	%	9	10	15-20% NA	5
PM @ 7% O2	mg/dscm	4	27	160	10
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7.5.1 CES Sponsored Trapping Efficiency Tests

A series of tests were conducted at CES and a coal-fired power plant in Boardman, OR to evaluate the Hg trapping efficiency of reactive filters under a variety of sampling conditions. For these tests, mercury was injected into either laboratory air or stack effluent and the trapping efficiency evaluated with the XFM using two reactive filters in series. The mercury trapping efficiency was determined by comparing the Hg trapped on the upstream filter with the total mercury collected on both filters. Mercuric chloride laboratory tests in the temperature range between 100 and 212°F exhibited capture efficiencies greater than 99% for concentrations ranging in excess of 800 μg/dscm with sampling times up to one hour. Similar tests were conducted at the coal-fired power plant using elemental Hg spiked into the stack gas. In this case, the elemental Hg trapping efficiency was greater than 99% for concentrations ranging up to 300 μg/dscm.

7.5.2 EPA Sponsored Relative Accuracy Tests

CES tested the XFM filter approach during an EPA sponsored evaluation of mercury measurement methods at a Midwest coal-fired power plant5. These tests, conducted in July 2003, compared simultaneous XFM and Ontario-Hydro Reference Method (OH) measured mercury concentrations between 1 and 6 μg/dscm. The accuracy of the reactive filter based methods at low concentrations was demonstrated using an in-stack version of the XFM. A total of 12 two-hour OH runs were conducted. For each OH run, two simultaneous 50 minute, in-stack XFM samples were collected by CES. For these tests, the XFM and the OH results were in good agreement with a relative accuracy of 11% for the nine valid runs, well within the 20% criteria for conditional methods specified by EPA's proposed Performance Specification 12[32]. Three of the 12 OH test runs were omitted from comparison by the EPA contractors. Two were omitted because of poor OH replication (>40% difference) and one was omitted due to a plant upset. The remaining nine runs were used for comparison, the results of which are plotted in Figure 19. The high correlation of these results and low relative accuracy demonstrates the effectiveness of the reactive filter approach for trapping and quantifying Hg concentrations between 1 and 6 μg/dscm.

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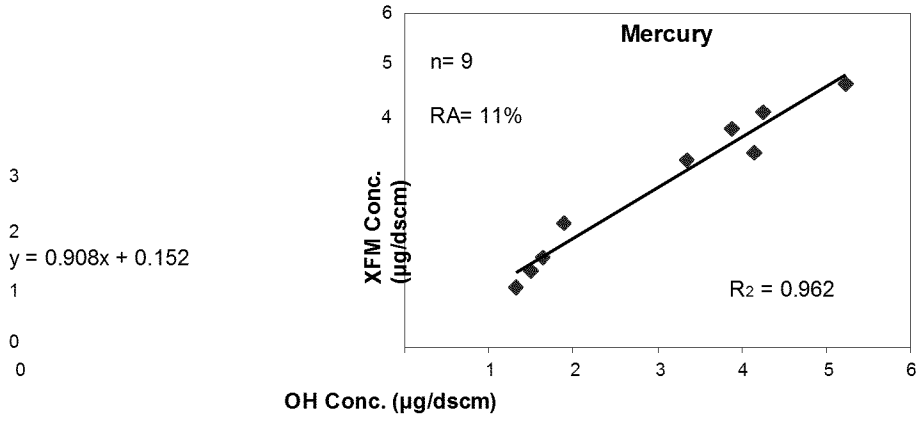


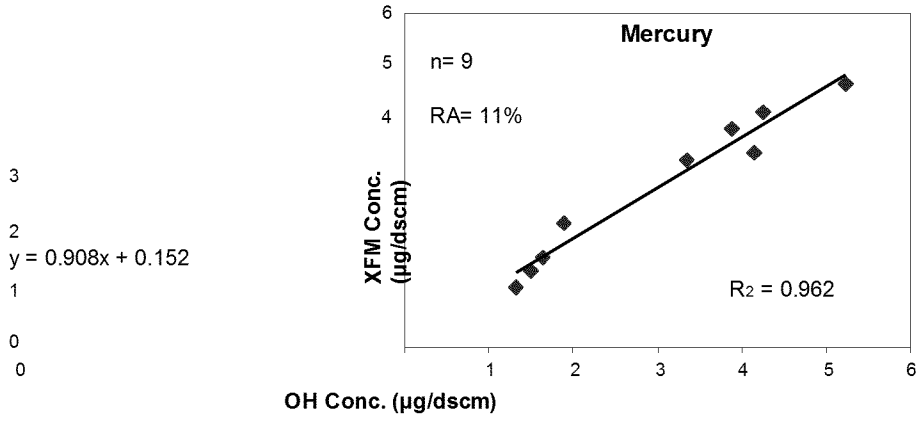
Figure 19. Comparison of Hg concentrations determined by XFM and Ontario Hydro reference method at a coal-fired power plant.

7.5.3 US Army Sponsored Relative Accuracy Tests

The U.S. Army sponsored a series of tests in January 2005 to evaluate a mercury-optimized Xact at a coal-fired power plant[33]. For these tests, mercury and arsenic were spiked into stack gas followed by simultaneous Hg and As measurements using the Xact and Reference Method 29. The results from these tests demonstrated relative accuracies of 17% for these two elements, meeting the proposed 20% criteria proposed for Performance Specification 10.

7.5.4 Summary of Trapping Efficiency Sensitivity to Reactive Stack Gases

Unlike the physical filtration mechanism used to collect particulate metals, vapor phase metals react with the chemically treated filter and bind to the filter substrate. Some stack gas constituents could potentially interfere with this process. However, the more abundant stack gas species such as N_2, O_2, CO_2, CO, and vapor phase H_2O are relatively inert, are not trapped on the reactive filter, and test results indicate that they do not influence the filter trapping efficiency. Oxidants within the gas stream such as Cl_2 have been shown to improve the vapor phase collection efficiency for Hg. Other minor stack gas components such as NO_x, SO_x, and NH_3, have the potential to interfere with vapor phase Hg trapping efficiency. However, this potential to interfere has not been observed in the extensive testing that has been conducted to date. For example, the possibility of NH_3 interference was evaluated during EPA's test of mercury measurement approaches at a Midwest coal-fired power plant with state-of-the-art ammonia injection and selective catalytic reduction NO_x control equipment[34]. Despite the use of ammonia, the XFM reactive filter demonstrated an 11% relative accuracy when compared to the Ontario-Hydro Hg reference method concentrations,
which ranged from 1 to 6 µg/dscm. Similarly, minor reactive components such as NO_x, and SO_x have the potential to impact vapor phase collection efficiency. However, there is no indication that these species affected the Hg trapping efficiency at the concentrations listed in Table 22. Indeed, the 99+% trapping efficiency for elemental Hg at the Boardman site was completed with measured NO_x concentrations of 200 PPM and SO_x concentrations of about 225 PPM. In addition, there is no indication that high acidic concentrations affect the trapping efficiency since nitric acid concentrations during Phase I and II testing was greater



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than 50 PPM. The tests conducted to date and the range of key test parameters are summarized below in Table 24.

Table 24. List of test conditions and references used to evaluate method applicability.

Parameter	Test Summary	Test
Concentration Range		
Particulate Metals	3-2200 µg/dscm	1, 2
Elemental Mercury	1-314 µg/dscm	3, 4
Oxidized Mercury	1-880 µg/dscm	3, 5
Holding Time		
XFM: Particulate Filter	12 Months	6, 7
XFM: Vapor Phase Filter	24 Months[a]	8
Xact	12 Months	6, 7, 8, 9
Stack Type & Conditions		
Moisture	Filters acceptable for Wet Stacks	3, 10, 11
	Filters acceptable for Dry Stacks	1, 2, 4, 9
HWC	Filters acceptable for HWC applications	1, 2, 9, 11
CFPP	Filters acceptable for CFPP applications	3, 4, 10
Stack Temperature	450°F	1, 2
Filter Temperature		
XFM Particulate Filter	400°F	12
XFM Vapor Phase Filter	185°F	4, 5
Xact Particulate/Vapor Phase Filter	185°F	
Sampling Time		
XFM	60 min	4, 5
Xact	30 min	10
Sampling Flow Rate		
XFM	4.5 lpm/cm²	4, 12
Xact	2.4 lpm/cm²	12
Field Blank	Det. Limits less than 2 µg/dscm[b]	11

a) Correction factor of 0.5%/month required after six months.
b) One-sigma limits based upon analysis of 3899 filters measurements at Lilly's T149 Incinerator

1 HWC at TEAD production furnace (Section 2.3).
2 HWC at TEAD research furnace (Section 2.3).
3 EPA test at Midwest CFPP (Section 2.3).
4 CES Test at Oregon CFPP (Section 2.3).
5 CES Laboratory Tests (Section 2.3).
6 Reevaluation of Phase I Filters (Section 2.3).
7 EPA $PM_{2.5}$ Speciation Program ; Reevaluation of Phase I Filters (Section 2.3).
8 CES Laboratory Evaluation of XFM Filters (Section 2.3).
9 CES Reanalysis of Xact Filter Tape from TEAD tests (Section 2.3).
10 Xact Test at IAAP CFPP (Section 2.3).
11 Evaluation of Blank Filters at Lilly's T149 Incinerator (Section 2.3).
12 Manufacturers Specifications
13 Phase II Tests at Lilly's T149 Incinerator

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7 EPA $PM_{2.5}$ Speciation Program ; Reevaluation of Phase I Filters (Section 2.3).
8 CES Laboratory Evaluation of XFM Filters (Section 2.3).
9 CES Reanalysis of Xact Filter Tape from TEAD tests (Section 2.3).
10 Xact Test at IAAP CFPP (Section 2.3).
11 Evaluation of Blank Filters at Lilly's T149 Incinerator (Section 2.3).
12 Manufacturers Specifications
13 Phase II Tests at Lilly's T149 Incinerator

7.6 *Applicable Concentration Range*

The applicable concentration range for these reactive filter based methods was extended to over 2000 µg/dscm during Xact and Reference Method 29 relative accuracy tests conducted in 2002 at an Army hazardous waste test incinerator[34]. Twelve Reference Method 29 test runs were conducted while the stack gas Pb concentrations ranged from a low of about 10 µg/dscm to over 2200 µg/dscm. The results from these twelve tests are compared to the corresponding Xact results in Figure 20. These results show a high degree of linearity over this broad range of concentrations and a high relative accuracy of 4% when compared to the Method 29 results. If these results are combined with those in the preceding subsection where the Hg results were in good agreement with the reference method down to almost 1 µg/dscm, it is clear that the method is quantitative over at least three orders of magnitude and at concentrations greater than 2200 µg/dscm.

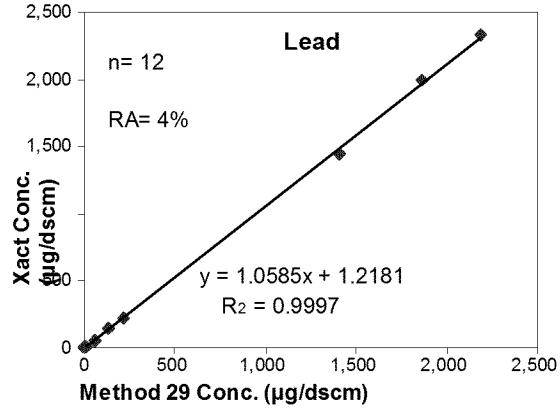

Figure 20 Plot of Xact-IAP concentration versus Reference Method 29 measured concentrations showing excellent agreement over two orders of magnitude and at concentrations in excess of 2,000 µg/dscm.

7.7 *Deposit Stability*

The Xact-IAP uses a reel-to-reel tape system to collect stack gas metal samples, which are then transported to a position for XRF analysis. After analysis, the tape is drawn over a take up spool where the exposed filter deposits come into contact with following tape as it wraps over the previously exposed tape. Some potential exists for transfer of metals from their original deposit to the tape covering the deposit. If this transfer were significant, it would reduce the potential to post-test validate Xact-IAP results with independent analysis of the deposits. As such, post test analyses were conducted on 72 deposits collected during relative accuracy tests conducted in May 2002 at a US Army hazardous waste production incinerator at the Tooele Army Depot (TEAD) in Tooele, UT[34]. During these tests, the Xact monitored emissions in an instrument shed about 40 feet from the incinerator stack while EPA Reference Method 29 samples were collected at the stack. Twelve Method 29 test runs were conducted with concentrations varying from 5 to 300 µg/dscm. There was very good agreement with the reference method as illustrated in Figure 21. The relative accuracy was 4% and the results exhibited a high degree of linearity.

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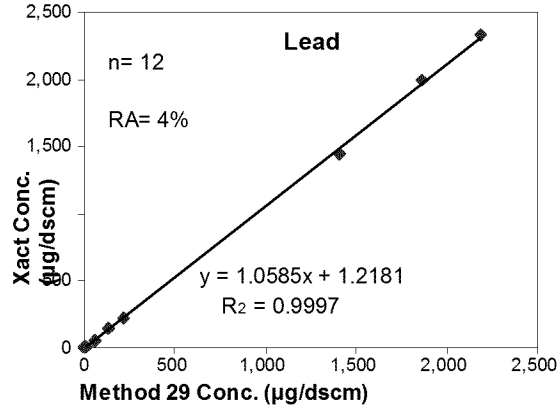

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Following these tests, the exposed filter tape was stored at room temperature at CES. Two months after the field tests were completed, the tape was removed from the spool exposing the previously collected deposits. These deposits were cut from the tape and analyzed with CES' laboratory XRF analyzer. These laboratory results are compared to the original concentrations reported by the Xact in Figure 22. Clearly, there is a high degree of agreement between the original Xact Pb results and the laboratory results. The slope and correlation suggests that there was not a significant transfer of Pb from the deposit to the covering tape at the level of a few percent.

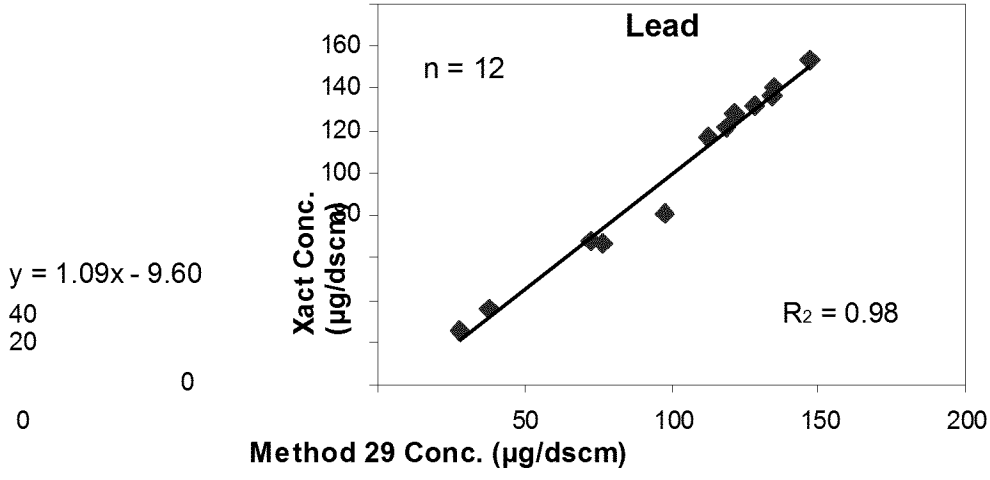

Figure 21. Plot of Pb concentrations determined by Reference Method 29 and the Xact.

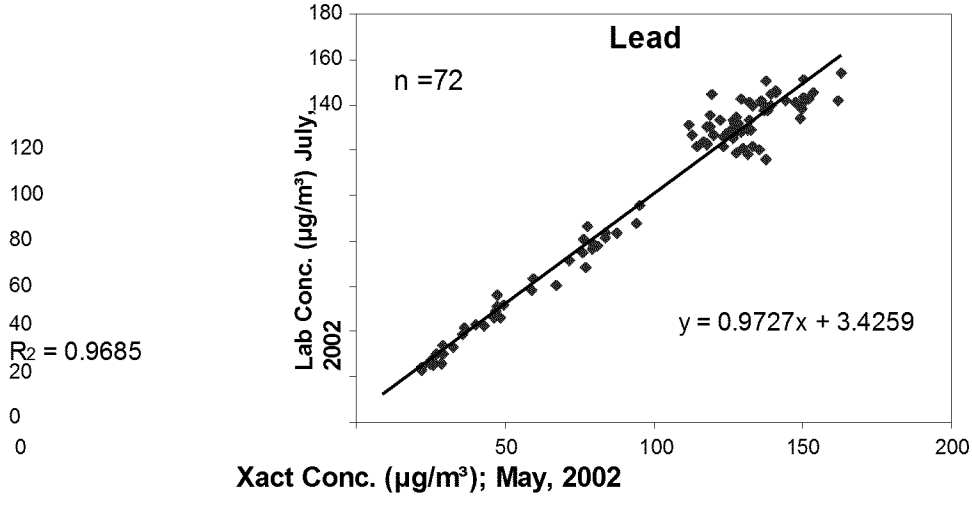

Figure 22: Comparison of results from laboratory XRF analysis in July, 2002 with Xact field test results from May 2002 relative accuracy tests at a hazardous waste incinerator

7.8 Moisture

Over the last three years, the Xact-IAP and XFM have been evaluated on both wet and dry stacks (Table 25) using Method 29, Ontario-Hydro and the QAG reference aerosols. In all

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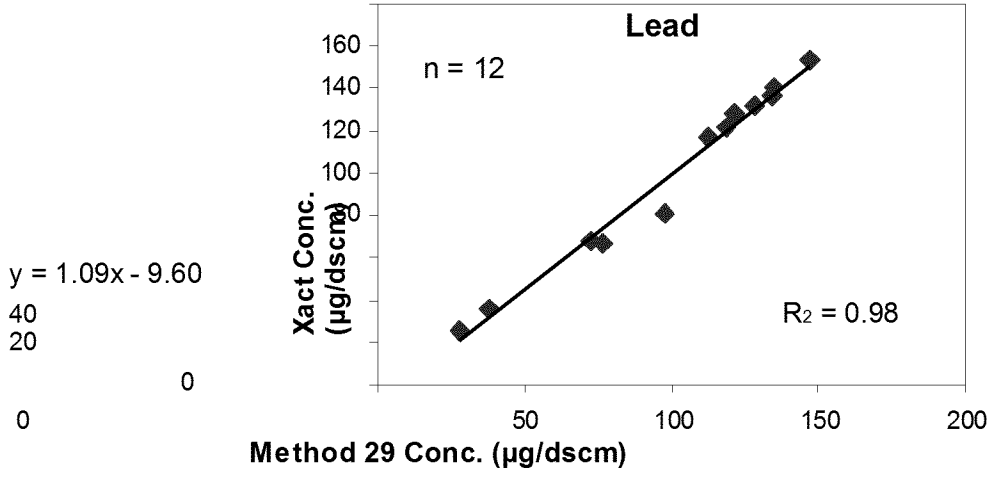

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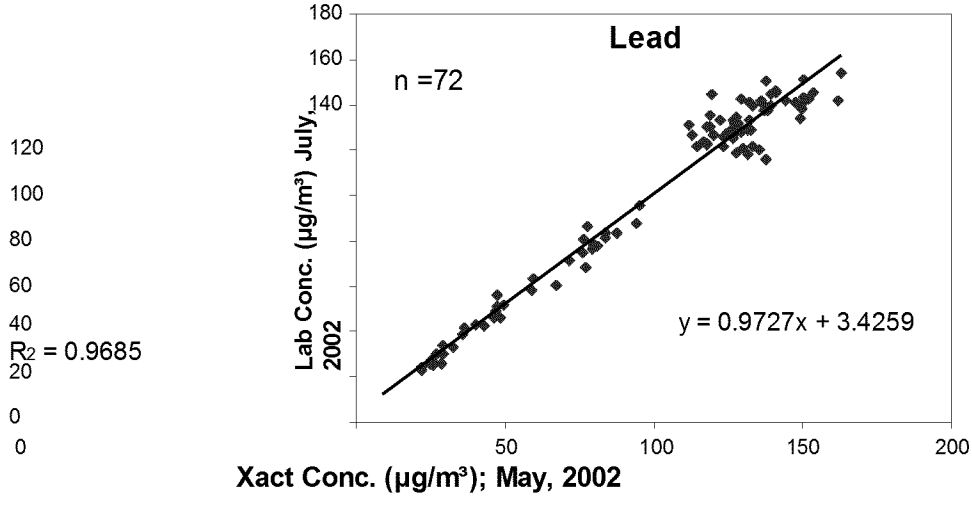

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Table 25: Recent tests of Xact and XFM on wet and dry stacks.

Date	Test Location	Inc. Type	Air Pollution Control Technology	Wet or Dry Stack	Elements Analyzed	Results
March 2005	Eli Lilly Lafayette, IN	HWC	Scrubber	Wet	As, Cr, Cd, Hg, Pb	Xact and XFM within 15% of QAG predicted for As, Cr, Cd, Hg, Pb
Jan. 2005	IAAP Ames, IA	CFPP	Scrubber	Wet	As, Hg	Xact RA of 17% vs. M29 for As, Hg
Sept. 2003	TEAD Tooele, UT	HWC	High Temp. Baghouse	Dry	Pb	Xact RA of 4% vs. M29 for Pb
May 2003	Midwest	CFPP	Ammonia injection, SCR, baghouse	Dry	Hg	XFM RA of 11% vs. OH for Hg
Aug 2002	Boardman, OR	CFPP	Scrubber	Wet	Hg^o	Trapping efficiency of 99% for Hg^o

7.9 Holding Times

Tests were also conducted to evaluate the holding times for the XFM and Xact-IAP filters. These tests included both the XFM PTFE filter and the reactive filter. During the December 2004 Phase I tests, the PTFE filter had collected particulate As, Cd, Cr, Hg, and Pb at concentrations ranging from 20 µg/dscm to 125 µg/dscm. In order to determine PTFE filter holding times, CES reanalyzed filters from seven of these tests (Runs 1-4 and 21-24) in April 2005. During the five months between the initial and second XRF analysis, the filters were stored at room temperature in plastic petrie dishes. Both analyses were conducted using XRF following the IO Compendium Method 3.3[14]. Overall, the average metal concentrations measured in April 2005 were within one percent of the concentrations determined five months earlier. All measurements were within 4% of their original concentration, with no significant bias. The good replication of the metal mass demonstrates the ability of the PTFE filters to retain metal concentrations over a five month period.

Reactive filter tests were conducted to determine retention efficiencies and estimated holding times for vapor phase metals using Hg as an indicator species. These tests are based on filters exposed to both elemental mercury and mercuric chloride vapors during tests conducted in June and August 2002 and filters from coal-fired power plant flue gas tests. All filters were kept at room temperature and analyzed using IO Compendium Method 3.3[14]. These filters were reanalyzed periodically over the past two years and were found to have lost on average about 10% of the original mercury over this period. This finding was

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7.10 Applicable Elements

The XRF analysis determination of metals in the filter deposit is based on EPA IO Compendium Method 3.3, which is generally applicable to elements with atomic numbers ranging from about 13 (Al) through 92 (U). This range of elemental applicability is expected to apply to the XFM PTFE filter but not to the reactive filter. In the case of the reactive filter, the lowest atomic number element quantifiable with XRF analysis is Cr (24) because of significant impurities in the filter matrix.

The elements of primary regulatory interest are those the Clean Air Act Amendments classify as Air Toxics: i.e. Antimony (Sb), Arsenic (As), Beryllium (Be), Cadmium (Cd), Chromium (Cr), Cobalt (Co), Lead (Pb), Manganese (Mn), Mercury (Hg), Nickel (Ni), and Selenium (Se). These eleven elements have been sorted by volatility in the Maximum Achievable Control Technology (MACT) rules[17] as follows:

- Non-enumerated metal compounds (N): Sb, Co, Mn, Ni, Se; equated to particulate matter in the MACT
- Low volatile metal compounds (L): As, Be, Cr
- Semi-volatile metal compounds (S): Pb, Cd
- High volatile metals (V): Hg

The metals tested in Phase I and II represented particulate matter as well as all of the metals in the three volatility categories except the low volatility element Be. The other elements not tested are non-enumerated metals, which the EPA has equated to particulate matter in the MACT rules. Although these eleven elements represent the primary focus of this discussion, it is important to note that these filter based methods are expected to be applicable to most of the elements listed in EPA IO Compendium Methods 3.3[14] and Be by IO Compendium Method 3.4[35].

7.11 Other Related Tests

7.11.1 Sample Collection Times

The theoretical sampling time for the XFM and Xact-IAP is primarily limited by filter loading with PM since XRF correction factors may be required for high particulate deposit densities. However, the filter loading can be controlled in sources with high particulate levels by adjusting the sampling times and dilution ratios up to 4:1. For this reason, it is believed

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that, these filter-based methods can sample for periods up to four times greater than has currently been demonstrated; i.e. four hours for the XFM and two hours for the Xact-IAP.

7.11.2 Xact Blank Concentrations

The Lilly Xact, was installed on the T149 hazardous waste incinerator in May 2004 and operated throughout the summer of 2004. During this time, the Xact completed about 4000 runs while the stack was not burning hazardous waste. These runs were treated as field blanks and were used to statistically determine Xact-IAP detection limits. These statistically determined detection limits were in good agreement with those calculated from the analysis of a few filters.

Table 26: Xact detection limits determined at Lilly's T149 incinerator

Elem	DETECTION LIMITS	
	XFM[1]	Xact[2]
	µg/dscm	µg/dscm
CR	0.08	0.3
AS CD	0.08	0.1
HG PB	0.80	2.4
MN CO	0.14	0.3
NI SE	0.12	0.6
AG SB	0.08	0.2
CU FE	0.08	0.3
ZN BR	0.06	0.3
SR TL	0.08	0.1
	0.60	4.2
	1.60	6.2
	0.08	0.5
	0.12	0.9
	0.06	0.2
	0.20	0.4
	0.18	0.3
	0.14	0.3

1) 95% Confidence, Interference free, 30 minute sample.
2) 95% of 3899 Xact blank measurements at T149 Incinerator

7.12 Applicable Source Categories

As discussed earlier, the filter-based sampling approach has been successfully tested in the laboratory and on two hazardous waste incinerators as well as three coal-fired boilers. Each of these tests showed good relative accuracy and trapping efficiencies. Source emissions can be divided into two categories: fugitive and ducted emissions. Fugitive sources include emissions from processes such as field-burning, slag-pouring, emissions from buildings, demolition activities, forest fires, etc. in addition to passive fugitive emissions from sources such as wind-blown dust and dust suspended by vehicular traffic. These fugitive emissions are typically characterized by substantially lower concentrations of possible reactive species

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than the concentrations in the ducted emissions already successfully tested. As such, the XFM and Xact-IAP should be applicable, in general, to these fugitive emission sources because the characteristics of their emissions are less challenging than the ducted sources already tested.

Ducted sources, on the other hand, can have emission characteristics similar to those already tested, and in some cases may exceed the values for some parameters that may be more challenging than those already tested. As discussed earlier, the filter-based approach has shown consistent success over the past few years while sampling at two hazardous waste incinerators and three coal-fired boilers with a wide range of controls (wet scrubbers, ESPs, high temperature ceramic baghouses, ammonia injection, selective catalytic reduction, lime spray dryer absorber for SO_2 control, and a fabric filter for particulate control). In each case the relative accuracy in comparison to the QAG, Ontario Hydro, and Method 29 was better than the EPA required 20% for conditional method consideration. In general, the filter-based approaches have been accurate regardless of source category, emission control technology used, moisture content and stack chemistry. As such, the Xact-IAP and XFM methods should be applicable to a wide range of source categories including incinerators, boilers, kilns, smelters, plating operations, foundries, mineral processing and other industrial processes using a wide range of pollution control technologies.

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