Battelle
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Environmental Technology Verification Program
Advanced Monitoring Systems Center

Quality Assurance Project Plan for Verification of the Xact 625 Ambient Particulate Metals Monitor

QUALITY ASSURANCE PROJECT PLAN

for

Verification of the Xact 625 Ambient Particulate Metals Monitor

Version 1.0

August 19, 2011

Prepared by

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SECTION A

PROJECT MANAGEMENT

A1 VENDOR APPROVAL PAGE

ETV Advanced Monitoring Systems Center

Quality Assurance Project Plan for Verification of
the Xact 625 Ambient Particulate Metals Monitor

Version 1.0

August 19, 2011

APPROVAL:

Name: _____

Company: Pall Corporation_____

Date: _____

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A3 LIST OF ACRONYMS AND ABBREVIATIONS

ADQ	audit of data quality
AMS	Advanced Monitoring Systems
CES	Cooper Environmental Services LLC
COC	chain of custody
DC	data completeness
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
ERG	Eastern Research Group
ETV	Environmental Technology Verification
ft	foot or feet
hr	hour(s)
ICP/MS	inductively coupled plasma/mass spectrometry
L	liter(s)
LFB	laboratory fortified blank
LRB	laboratory record book
m	meter(s)
$\mu g/m^3$	micrograms per cubic meter
min	minute(s)
ng	nanogram(s)
NIST	National Institute of Standards and Technology
NRMRL	National Risk Management Research Laboratory
OAQPS	Office of Air Quality Planning and Standards
OEPA	Ohio Environmental Protection Agency
PE	performance evaluation
PM	particulate matter
PM_{10}	particulate matter with aerodynamic diameter less than 10 micrometers
PO	project officer
QA	quality assurance
QAO	quality assurance officer
QAPP	quality assurance project plan
QC	quality control
QCS	quality control sample
QMP	Quality Management Plan
SAT	school air toxics
SOP	standard operating procedure
RMO	Records Management Office
TSA	technical systems audit
TSP	total suspended particulate
VTC	verification test coordinator
XRF	X-ray fluorescence

A4 DISTRIBUTION LIST

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A5 VERIFICATION TEST ORGANIZATION

The verification test described in this document will be conducted under the Environmental Technology Verification (ETV) Program. Verification activities will be overseen by Battelle, which is managing the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with the U.S. Environmental Protection Agency (EPA) National Risk Management Research Laboratory (NRMRL). Verification testing will be carried out in collaboration with EPA Region 5, EPA's Office of Air Quality Planning and Standards (OAQPS), and the Ohio Environmental Protection Agency (OEPA). The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil.

This verification test will be conducted at two OEPA monitoring sites in eastern Ohio. The testing will involve continuous operation of a commercial monitor for determining toxic metals in atmospheric particulate matter, at sites where reference samples will be collected on filters for subsequent laboratory analysis for metals. The commercial monitor will be purchased by EPA, and will be housed for testing in a shelter purchased by EPA and built by the vendor of the monitor. The continuous monitor results and the filter-based reference results will be compared to evaluate the monitor's capability for metals monitoring. The vendor of the metals monitor being tested will install, and if necessary repair or maintain, their monitor during the verification test, and will train OEPA, EPA Region 5, and OAQPS staff in operation of the monitor.

Quality assurance (QA) oversight of this EPA Category III verification test will be provided by the Battelle AMS Center Quality Manager and the EPA AMS Center Quality Manager. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Roles and responsibilities are defined further below.

A5.1 Battelle

Dr. Thomas Kelly is the AMS Center Verification Test Coordinator (VTC) for this test. In this role, Dr. Kelly will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, he will:

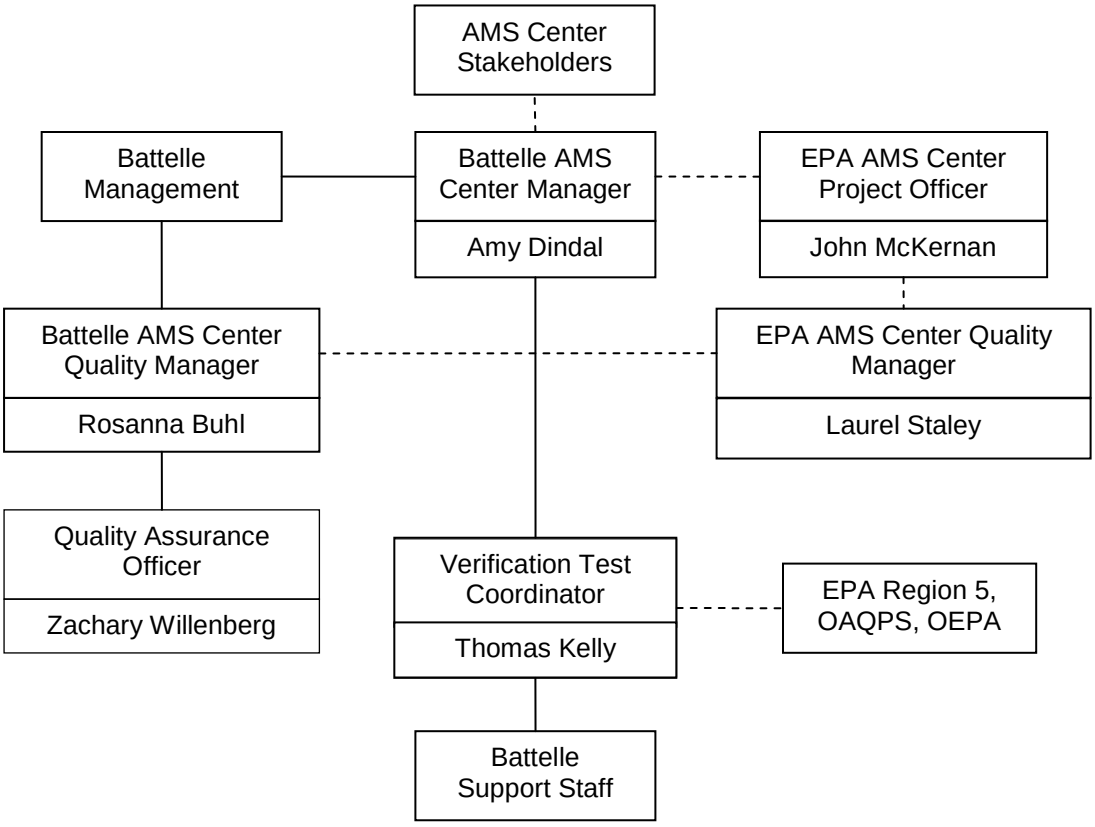

Figure 1. Organizational Chart

- Hold a kick-off meeting approximately one week prior to the start of the verification test to review the critical logistical, technical, and administrative aspects of the verification test,
- Coordinate with EPA Region 5, OAQPS, and OEPA in performing the verification test in accordance with this QA project plan (QAPP),
- Ensure that all quality procedures specified in the QAPP and in the AMS Center Quality Management Plan[1] (QMP) are followed,
- Maintain real-time communication with the Battelle AMS Center Manager and Quality Manager, and EPA AMS Center Project Officer and Quality Manager on any potential or actual deviations from the QAPP,
- Prepare the draft and final QAPP, verification report, and verification statement,
- Revise the draft QAPP, verification report, and verification statement in response to reviewers' comments,

- Support OEPA as needed in establishing the necessary infrastructure at the test site to carry out the test,
- Coordinate a performance evaluation (PE) audit of the reference sampling and analysis methods,
- Provide test data, including data from the first day of testing, to the Battelle AMS Center Manager and EPA AMS Center Project Officer and Quality Manager,
- Conduct a technical review of all test data,
- Perform analysis of the collected data to carry out the statistical evaluations defined in this QAPP,
- Call on Battelle staff as needed for support in test coordination, data analysis, and reporting,
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary,
- Serve as the primary point of contact for vendor representatives and collaborators,
- Coordinate distribution of the final QAPP, verification report, and verification statement, and
- Establish a budget for Battelle activities in the verification test and manage staff to ensure the budget is not exceeded.

Ms. Amy Dindal is Battelle's Manager for the AMS Center. Ms. Dindal will:

- Review the draft and final QAPP,
- Review the draft and final verification report and verification statement,
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test,
- Ensure that confidentiality of sensitive vendor information is maintained,
- Review QAPP deviations and any issues raised in assessment reports, audits, or from test staff observations, and institute corrective action as necessary,
- Maintain communication with EPA's AMS Center Project Officer and Quality Manager, and

- Facilitate a stop work order if Battelle or EPA QA staff discovers adverse findings that will compromise data quality or test results.

Ms. Rosanna Buhl is Battelle's Quality Manager for the AMS Center. Ms. Buhl will:

- Review the draft and final QAPP,
- Assign a Quality Assurance Officer (QAO) for this verification test,
- Delegate to other Battelle quality staff any QAO responsibilities assigned below as needed to meet project schedules,
- Review any audit checklists prepared by the QAO for completeness and detail,
- Review draft and final audit reports prior to release to the VTC and/or EPA for clarity and appropriate assessment of findings,
- Review audit responses for appropriateness,
- Review and approve the QAPP and any deviations,
- Review draft and final verification report(s) and verification statement(s),
- Maintain real-time communication with the QAO on QA activities, audit results, and concerns,
- Work with the QAO, VTC, and Battelle's AMS Center Manager to resolve data quality concerns and disputes, and
- Recommend a stop work order if audits indicate that data quality or safety is being compromised.

Mr. Zachary Willenberg is Battelle's QAO for this test. Mr. Willenberg will:

- Attend the verification test kick-off meeting and lead the discussion of the QA elements of the kick-off meeting checklist,
- Prior to the start of verification testing, verify the presence of applicable training records, including any vendor-provided training on the metals monitor being tested,
- Conduct a technical systems audit (TSA) of the field testing and the reference analytical laboratory near the beginning of the verification test,
- Review results of the PE audit of reference sampling and analysis methods,
- Conduct audits of data quality (ADQs) to verify data quality,

- Prepare and distribute an audit report for each audit,
- Verify that audit responses for each audit finding and observation are appropriate and that corrective action has been implemented effectively,
- Communicate to the VTC and/or technical staff the need for immediate corrective action if an audit identifies QAPP deviations or practices that threaten data quality,
- Provide a summary of the QA/QC activities and results for the verification report,
- Review the draft and final verification report and verification statement,
- Maintain real-time communication with the Battelle Quality Manager on QA activities, audit results, and concerns, including potential schedule and budget problems, and
- Communicate data quality concerns to the VTC and/or Battelle's AMS Center Quality Manager; recommend the need for a stop work order if audits indicate that data quality or safety is being compromised.

A5.2 Vendor

The metals monitor to be tested was developed and will be manufactured by Cooper Environmental Services LLC (CES), but the rights to the metals monitor technology are owned by Pall Corporation. Pall Corporation and CES will jointly fulfill the following responsibilities of the metals monitor vendor, except as noted when one or the other organization has responsibility for a particular item:

- Review and provide comments on the draft QAPP,
- Pall Corporation will approve the final QAPP prior to test initiation,
- Provide a representative to participate in the kick-off meeting for the verification test,
- CES will provide all supplies/reagents/consumables needed to operate the metals monitor for the duration of the verification test upon purchase of the monitor by EPA,
- Pall Corporation will supply a representative to assist with installing the metals monitor at the field site, and to be available for consultation in maintenance or repair of the monitor,
- Provide training to EPA Region 5, OAQPS, and OEPA personnel in operating and maintaining the metals monitor, and written consent for OEPA staff to operate the monitor for purposes of verification testing,

- CES will provide written instructions for routine operation of the metals monitor, such as a user's manual, Standard Operating Procedure (SOP), and/or a daily checklist of diagnostic and/or maintenance activities, and
- Pall Corporation will review and provide comments on the draft verification report and verification statement.

A5.3 U.S. EPA ETV Program

EPA's responsibilities in the ETV program are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan"[2] (ETV QMP). The roles of specific EPA testing staff in this verification will be as follows:

Dr. John McKernan is EPA's Project Officer for the AMS Center. In this verification Mr. Julius Enriquez will assist in the role of Project Officer while Dr. McKernan is on another assignment. Mr. Enriquez will review the draft QAPP. Dr. McKernan will:

- Approve the final QAPP,
- Review and approve deviations to the approved final QAPP,
- Review the first day of data from the verification test and provide immediate comments if concerns are identified,
- Review the draft verification report and statement,
- Oversee the EPA review process for the QAPP, verification report, and statement, and
- Coordinate the submission of the QAPP, verification report, and statement for final EPA approval.

EPA's AMS Center Quality Manager or designated QA representative will:

- Review the draft QAPP,
- Review deviations to the approved final QAPP,
- Review the first day of data from the verification test and provide immediate comments if concerns are identified,
- Perform at his/her option one external TSA and/or audit of data quality during the verification test,

- Notify the EPA AMS Center Project Officer of the need for a stop work order if an external audit indicates that data quality or safety is being compromised,
- Prepare and distribute an assessment report summarizing results of the external audit, and
- Review the draft verification report and statement.

A5.4 U.S. EPA Region 5 and OAQPS

EPA Region 5 and OAQPS will coordinate with Battelle and OEPA in performance of the verification test. Ms. Motria Caudill will be the primary EPA Region 5 point of contact, and Mr. Michael Jones will be the primary OAQPS point of contact. EPA OAQPS will:

- Purchase an Xact 625 metals monitor for testing,
- Purchase a Partisol Plus 2025 Sequential Ambient Particulate Sampler for use as the reference sampler in the field test of the Xact 625.

Additional responsibilities of EPA Region 5 and OAQPS will be as follows:

- Coordinate with the vendor for training of EPA Region 5, OAQPS, and OEPA staff in operation of the metals monitor,
- Provide an SOP for operation and sample recovery with the reference method sampling system at the field site,
- Arrange for laboratory analysis of collected reference method samples by a certified analytical laboratory [Eastern Research Group (ERG)],
- Review the draft QAPP,
- Provide a representative to participate in the kick-off meeting for the verification test, and
- Review the draft verification report and statement.

A5.5 Ohio EPA

Ohio EPA will provide field sites and supporting infrastructure to carry out the test, and OEPA staff will operate the metals monitor and conduct reference method sampling at the site. OEPA staff will be in frequent communication with the Battelle VTC and EPA Region 5 and OAQPS staff, and with the technology vendor as needed. All such communication will be documented, and the Battelle VTC will keep EPA's Project Officer for the AMS Center informed of these communications. Mr. Randy Hock, in OEPA's Columbus, Ohio headquarters, will be the primary OEPA point of contact. The responsibilities of OEPA staff will be to:

- Coordinate with EPA Region 5, OAQPS, and the vendor to install the metals monitor to be tested at the test sites,
- Provide a representative to participate in the kick-off meeting for the verification test,
- Prepare the test sites to ensure suitable space and electrical power to perform the necessary testing activities,
- Receive training in operation of the metals monitor from the vendor,
- Operate the metals monitor throughout the field test,
- Coordinate the operation of the test sites for the purposes of ETV testing,
- Communicate needs regarding access, security, and safety to any staff working or visiting at the test sites,
- Perform ambient particulate sampling using an appropriate filter-based collection method and send samples to the analytical laboratory conducting metals analysis,
- Record qualitative observations about the maintenance and operation of the metals monitor during testing,
- Ensure that the metals monitor data and any field records are reviewed within one week of generation, and transmitted to the Battelle VTC on at least a weekly basis,
- Provide input in responding to any issues raised in assessment reports and audits related to site operations,
- Provide input on test procedures, technology operation and maintenance, and field conditions for the draft verification report, and
- Review the draft verification report and statement as needed.

A5.6. Verification Test Peer Reviewers

This QAPP and the verification report and verification statement based on testing described in this document will be reviewed by experts in the fields related to particulate metals and/or analytical instrumentation. The following experts have agreed to provide a peer review of this QAPP and the resulting verification report and statement.

- Dennis Mikel, EPA/Office of Air Quality Planning and Standards [EPA QA coordinator for the School Air Toxics (SAT) program]
- Rudy Eden, South Coast Air Quality Management District

The responsibilities of verification test peer reviewers include:

- Participate in technical panel discussions (when available) to provide input to the test design,
- Review and provide input to the QAPP, and
- Review and provide input to the verification report and verification statement.

A6 BACKGROUND

A6.1 Technology Need

The ETV Program's AMS Center conducts third-party performance testing of commercially-available technologies that detect or monitor natural species or contaminants in air, water, and soil. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. Among the technology categories recommended for testing are continuous monitors for toxic metals present in atmospheric particulate matter (PM). In particular, the use of metals monitors for determining toxic metals in PM having aerodynamic diameters smaller than 10 micrometers (known as PM_{10}) was identified as an area of interest for technology verification.

Many industrial communities are burdened by emissions of toxic metals such as lead (Pb), manganese (Mn), and chromium (Cr). For example, EPA's SAT Monitoring Initiative[3] has disclosed that Mn, a neurotoxic metal, is one of the pollutants of greatest potential risk near schools in industrialized locations. Existing monitoring methods for metals in airborne PM have significant limitations, in that samples are collected over a 24-hour integrated period and must be

submitted for laboratory analysis, from which results may not be available for several weeks. Commercially-developed metals monitors are now available that can determine 10 or more toxic metals simultaneously in ambient PM_{10} with time resolution of one hour or less. The relatively fast response afforded by such monitors may be useful in identifying emission sources and allocating the contributions of such sources to local levels of toxic metals. Specifically, the advantages of hourly metals data (relative to 24-hour average data) are:

- Improved ability to follow variations in metals concentrations associated with time of day or meteorological conditions, and thus to attribute metals risk to a particular source,
- Ability to compile a dataset large enough to conduct source receptor modeling, and
- Cost reduction and time savings due to the generation of multi-metals data on-site without waiting for laboratory analysis.

Thus deployment of metals monitoring may lead to better characterization of environmental metals exposures and more effective source identification, allowing decision-makers to quickly target industries for enforcement action or voluntary emissions reduction.

A.6.2 Technology Description

Commercial instruments for monitoring of metals in atmospheric PM_{10} are based on the principle of X-ray fluorescence (XRF), in which X-rays from a source cause removal of an electron from an inner electronic shell of a metal atom in a sample. The vacancy in the inner shell is then filled by an electron from an outer shell, with resulting emission of an X-ray whose wavelength is longer than that of the original excitation and characteristic of the metal in question. The wavelength (or, equivalently, the energy) and intensity of the emitted X-rays are determined to identify and quantify the metal in the sample. Multiple metals in the sample are excited and detected simultaneously. For monitoring of metals in atmospheric PM, an automated moveable filter tape system is used, in which a sample air flow is drawn through a small spot on the tape, collecting and concentrating PM onto that spot. After a pre-set sampling interval, the tape advances, placing the collected sample spot in the X-ray excitation and analysis section of the instrument, and initiating sampling onto a previously unexposed spot on the tape. The sequence of sampling and analysis can continue automatically, limited only by the supply of filter tape.

The duration of sample collection at each spot can be varied to maintain detection performance in the face of varying atmospheric PM levels.

The specific commercial metals monitor evaluated under this QAPP is the Xact 625, previously manufactured by Cooper Environmental Services LLC (Portland, OR). Commercial rights to the Xact technology are owned by Pall Corporation (Port Washington, NY). The following description of the Xact 625 technology is based on information provided by the vendor (this information was not verified in this test). The Xact 625 employs energy-dispersive X-ray fluorescence (EDXRF), in which the detector and electronics resolve emitted X-rays based on their energy. The EDXRF approach allows use of a relatively simple optical path and a relatively low power X-ray source, and thus reduces the instrument cost. The EDXRF approach also provides acquisition of the entire X-ray spectrum very rapidly, so that many elements in the periodic table can be detected within a few seconds. The Xact 625 samples ambient air at a constant flow rate of 16.7 L/min (i.e., 1 m^3/hr). The Xact 625's sample inlet is designed to provide uniform sample deposition, and the instrument analyzes approximately 90% of the sample spot area, to minimize effects of sample inhomogeneity. In addition to the general features of ambient metals monitors outlined above, the Xact 625 employs automatic internal stability checks (described in Section B5) to track the monitor's performance

A7 VERIFICATION TEST DESCRIPTION AND SCHEDULE

The purpose of this verification test is to generate performance data on metals monitoring for airborne PM, with a particular focus on monitoring of toxic metals in atmospheric PM_{10} in industrialized areas. The data generated from this verification test are intended to provide organizations and users interested in metals monitoring with information on the potential use of these monitors for that application and on their performance under such conditions.

A7.1 Verification Test Description

The test described in this QAPP will be conducted by operating a metals monitor at each of two field sites for up to eight weeks, and comparing the monitor results for several metals to the results from conventional reference measurements made by laboratory analysis of 24-hour filter samples collected at the same site. The monitor readings for each metal will be averaged over

the appropriate 24-hour periods for comparison to the reference results. The metals monitor will be evaluated on the following performance parameters.

- Comparability
- Correlation
- Bias
- Data completeness
- Operational factors

Comparability, correlation, and bias will be assessed by comparison of monitor and reference results, using the statistical approaches described in Section B1.2. Data completeness will be determined from the number of hourly periods the metals monitor provides readings of PM_{10} metals concentrations (allowing for daily periods of approximately 30 minutes in which the Xact 625 performs internal QA checks). Data completeness will be determined both relative to the entire test period and relative to each 24-hour reference sampling period. Operational performance factors such as maintenance requirements, ease of use, and effectiveness of data acquisition will be determined from observations by the field testing staff. This test is not intended to simulate long-term performance of the metals monitor at a monitoring site, but rather to assess the feasibility of its use for ambient metals monitoring in near-source locations. In long-term use, metals monitoring procedures might be tailored to address the specific conditions, metals concentrations, and sources for a given site. This test also will not evaluate the precision of the individual metals monitor in repetitive analysis of the same sample, as ambient atmospheric sampling does not provide known constant samples to assess precision. That performance factor will be evaluated in a separate project.[4] Similarly, with only a single unit of the metals monitor involved in the test, the unit-to-unit duplication of multiple monitors of the same type cannot be determined.

Subsequent to the verification test, Battelle will draft a verification report for the metals monitor. The report will be reviewed by the technology vendor, by EPA Region 5 and OAQPS staff, and by peer reviewers, and will be revised and submitted to EPA NRMRL for final approval. In performing the verification test, Battelle will follow the technical and QA procedures specified in

this QAPP and will comply with the data quality requirements in the AMS Center QMP and the ETV QMP.[1]

A7.2 Proposed Testing Schedule

Table 1 shows the expected schedule of testing and data analysis/reporting activities to be conducted in this verification. The metals monitor field testing is planned to take place from August to December 2011, with equal time spent at two test sites within that period. Table 1 also notes the approximate timing of the field and laboratory TSAs and of the ADQs.

A7.3 Test Sites

The sites for testing will be at OEPA air quality monitoring sites in East Liverpool and Marietta, Ohio, on Ohio's eastern and southeastern border, respectively. Both sites are located near the Ohio River, in industrialized areas along the river that are characterized by restricted air flow due to local terrain. Elevated levels of Mn and other metals have been found in ambient total suspended particulate (TSP) at these sites, and at nearby elementary schools.

The metals monitor to be tested will be installed in an air-conditioned trailer provided by EPA, parked in a secure location at each OEPA site. The trailer in which the metals monitor will be installed is approximately 6.9 m (22.5 ft) overall, including the hitch. The air conditioned enclosure for the metals monitor is approximately 3 m (L) x 2 m (W) x 2.6 m (H) [10 ft (L) x 6.7 ft (W) x 8.5 ft (H)]. The PM_{10} inlet of the Xact 625 metals monitor will be placed on the roof of this enclosure as close as possible to, and no more than 10 meters from, the inlet of the PM_{10} reference sampler, and will be connected to the metals monitor by a straight vertical pipe connection to minimize any loss of ambient particles during passage of sample air to the monitor. The Xact 625 metals monitor, reference sampler, and other equipment will be powered by fixed electrical power lines installed at the sites for this test.

Table 1. Planned Verification Test Schedule

Approximate Dates	Location	Verification Activities	
		Testing	**Data Analysis and Reporting**
August 9-10, 2011	Columbus, OH	Training in operation of the monitor	
August 9-11, 2011	East Liverpool, OH	Installation of monitor at test site Kickoff meeting with all participants	
August 12 – October 7, 2011	East Liverpool, OH	Continuous monitoring of target metals Reference method sampling	Conduct TSA of field testing and reference laboratory Compile data from metals monitor Review and summarize observations from field operators Compile metals data from reference method Begin statistical comparisons of reference and monitor results Conduct audit of data quality Develop ETV draft report template
October 8-12, 2011	Marietta, OH	Installation of monitor at test site Refresher training in operation of the monitor	
October 13 – December 8, 2011	Marietta, OH	Continuous monitoring of target metals Reference method sampling	Compile data from metals monitor Review and summarize observations from field operators Compile metals data from reference method Continue statistical comparisons of reference and monitor results Continue draft ETV report
December 9-10, 2011	Marietta, OH	Conclusion of verification test Remove monitor from test site	
December 2011-January, 2012	Columbus, OH		Complete statistical comparisons of reference and monitor results Complete draft ETV report Conduct final audit of data quality
February, 2012	Columbus, OH		Submit draft ETV report for peer review, and revise
March, 2012	Columbus, OH		Submit draft ETV report for EPA QA review, and revise
April, 2012	Columbus, OH		Submit final ETV report for EPA approval

A8 QUALITY OBJECTIVES FOR MEASUREMENT DATA

The objective of this verification test is to evaluate the performance of the Xact 625 metals monitor under conditions relevant to ambient particulate metals monitoring. This evaluation will assess the capabilities of the monitor for determining metals concentrations in the ambient air. The verification test will also rely upon operator observations to assess other performance characteristics of the monitor being tested including data completeness, ease of use, and maintenance requirements.

Data quality indicators (DQIs) ensure that this verification test provides suitable data for a robust evaluation of performance. DQIs have been established for reference sampler flow rate accuracy and the accuracy of the reference method measurements. The DQIs were established to ensure that data used to support the quantitative performance evaluations of the Xact 625 metals monitor are of sufficient quality.

Quantitative performance of the metals monitor will be assessed by comparison to well-established reference sampling and analytical methods. To assure that those methods provide data of sufficient quality for a robust evaluation of metals monitor performance, accuracy data quality indicators (DQI's) have been established for the reference sampler flow rate and for the analytical method. Specifically, the measurement quality objective (MQO) for the reference sampler flow rate is that it be within ±5% of the target flow rate for the sampler. The MQO for the analytical accuracy is that reported metals concentrations be within ±10% of the true value. The degree of attainment of both of these DQO's will be assessed by means of PE audits conducted during the testing. The procedures and schedule for the PE audits are described in Section C1.1.

The Battelle QAO will perform a TSA of laboratory and field-based testing activities to augment the normal operational and QA/QC requirements of the metals monitor and the reference sampling and analytical methods. A TSA of the field testing activities will be performed within the first two weeks of the field testing, and will be followed by a TSA of the analytical laboratory within the first two weeks that reference samples from the field are being analyzed. The EPA Quality Manager also may conduct an independent TSA, at her discretion.

A9 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective locations. The Battelle QAO may verify the presence of appropriate training records prior to the start of testing. Battelle technical staff supporting this verification test will have a minimum of a bachelor's degree in science/engineering. Training of EPA Region 5, OAQPS, and OEPA staff by the vendor will be documented, and the vendor will indicate by signature that those staff are suitably trained to operate the Xact 625 monitor for testing. The metals monitor vendor will only operate the technology for training of testing staff and as needed for maintenance or repair activities during the verification test.

The verification test described in this QAPP will be performed at sites operated by the OEPA. All participants in this verification test (i.e., Battelle, EPA, and vendor staff) will adhere to the health and safety requirements of the OEPA sites. OEPA testing staff will give a site-specific safety briefing to all staff visiting either of the test sites. This briefing will include a description of:

- Any emergency operating procedures (i.e., in case of fire, tornado),
- Identification, location, and operation of any safety equipment (e.g., fire alarms, fire extinguishers, eye washes, exits), and
- Any personal protective equipment requirements and site-specific procedures.

A10 DOCUMENTATION AND RECORDS

The documents and records for this verification test include this QAPP, chain-of-custody forms, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and the final verification report. All of these documents and records will be maintained at the field site during the test, and will be reviewed on at least a weekly basis by OEPA to ensure that records have been filled in and are up to date. Copies of all reference sample chain-of-custody forms will be retained on site. These documents and records will be transferred to permanent storage at Battelle's Records Management Office (RMO) at the conclusion of the verification test. Documents and records generated at the test site will be

stored in a secure location until they can be copied and/or transferred to the VTC. Electronic documents and records will also be uploaded to a SharePoint site designated for this test and will be available to EPA upon request. All Battelle LRBs will be stored indefinitely by Battelle's RMO. EPA will be notified before disposal of any files. Section B10 further details the data recording practices and responsibilities.

All data generated during the conduct of this project will be recorded directly, promptly, and legibly in ink. All data entries will be dated on the date of entry and signed or initialed by the person entering the data. Any changes in entries will be made so as not to obscure the original entry, will be dated and signed or initialed at the time of the change and shall indicate the reason for the change. Data recording and chain-of-custody forms for the reference sampling method will be used in testing at both sites; an example of those forms is shown as Appendix A of this QAPP. The draft forms will be provided to the Battelle QAO for review prior to use so that appropriate changes, if any, can be made.

SECTION B

DATA GENERATION AND ACQUISITION

B1 EXPERIMENTAL DESIGN

The verification test described in this QAPP will be conducted consistent with the requirements of the ETV AMS Center QMP,[1] the ETV Program QMP,[2] and the draft SAT Program QAPP.[4] This QAPP specifically addresses verification of continuous metals monitors for ambient air monitoring applications by evaluating the following performance parameters.

- Comparability
- Correlation
- Bias
- Data completeness
- Operational factors

The verification test will be conducted over a period of approximately four months. The installation and training period at the first test site is scheduled to begin in early August, 2011. The Xact 625 monitor undergoing verification will report ambient concentrations of approximately 20 metals on an hourly basis. The monitored metals concentrations will be averaged over 24 hour time periods for comparison to ambient concentrations determined from time-integrated filter sampling and subsequent laboratory analysis. Section B1.1 describes the test procedures, and Section B1.2 describes the statistical calculations that will be used in those evaluations.

A verification report will be prepared for the metals monitor upon completion of testing, presenting the test procedures and test data, as well as the results of the statistical evaluation of those data. The verification report will briefly describe the ETV program, the AMS Center, and the procedures used in verification testing. The results of the verification test regarding metals monitor performance will be stated quantitatively. The draft verification report will be subjected to review by the metals monitor vendor, EPA, and other peer reviewers. The resulting review

comments will be addressed in a subsequent revision of the report, and the peer review comments and responses will be tabulated to document the peer review and QA review process for EPA. The reporting and review process will be conducted according to the requirements of the ETV/AMS Center QMP.[1]

B1.1 Test Procedures

The Xact 625 metals monitor undergoing verification will be installed at an appropriate secure location at each of the two OEPA test sites in turn, according to the schedule shown in section A7.1, and between 2 and 10 meters from the inlet of the reference PM_{10} sampler. The Xact 625 metals monitor will operate continuously and on an hourly basis will report the measured ambient concentration of each of the analytes listed in Table 2.

Table 2. Target Analytes for Xact 625 Metals Monitor

Metal	Symbol	Metal	Symbol
Antimony	Sb	Mercury	Hg
Arsenic	As	Molybdenum	Mo
Barium	Ba	Nickel	Ni
Bromine	Br	Potassium	K
Cadmium	Cd	Rubidium	Rb
Calcium	Ca	Selenium	Se
Chromium	Cr	Strontium	Sr
Cobalt	Co	Thallium	Tl
Copper	Cu	Thorium	Th
Iron	Fe	Titanium	Ti
Lead	Pb	Zinc	Zn
Manganese	Mn		

Table 3 presents a summary of the quantitative tests to be performed, showing the performance parameters, the objective of each parameter, the basis for evaluation of each parameter, and the number of data points or comparisons to be included in the evaluation. Table 3 assumes that a 24-hour reference sample will be obtained on every day over a 16-week period (112 total reference samples for each target metal), and that the metals monitor will report data on an hourly basis (2,688 total measurements of each target metal over the test period). The hourly monitoring data will be converted to daily averages for comparison with the reference data. The

numbers of data points indicated in Table 3 apply to each of the more than 20 metals to be determined in the test.

Table 3. Summary of Tests and Testing Frequency

Performance Parameter	Objective	Evaluation Based On	Number of Data Points
Comparability	Determine relationship of metals monitor results to reference results	Slope and intercept of linear regression of averaged metals monitor results against reference results	112[a]
Correlation	Determine degree of correlation of metals monitor results with reference results	Coefficient of determination of linear regression of averaged metals monitor results against reference results	112[a]
Bias	Determine quantitative agreement of metals monitor results and reference results	Mean, standard deviation, and range of percent difference values of metals monitor results relative to corresponding reference results	112[a]
Data Completeness	Determine data return of metals monitor relative to maximum possible	Number of hours of valid data relative to all hours in the test	2,688[a]
	Determine data return of metals monitor relative to reference sampling periods	Number of 24-hour reference periods in which metals data are obtained for over half the hours	112[a]

a: For each target metal, if detected in all samples

Throughout the verification test, the metals monitor undergoing testing will be operated by OEPA staff trained by the vendor. However, the intent of the testing is for the metals monitor to operate in a manner simulating use by site operators to monitor ambient air with little user intervention. As a result, it is expected that once the verification test has begun, no adjustment or recalibration will be performed. Repair or maintenance procedures may be carried out at any time, but testing will not be interrupted, and data completeness will be reduced if such activities prevent collection of metals monitor data required for verification. All repair and maintenance efforts will be documented including the type of repair or maintenance, the length of time needed to make the repair or conduct maintenance, and the cost of any consumables or replacement

parts. OEPA staff will download metals monitor data from the monitor's software for transfer to permanent electronic records.

B1.2 Statistical Methods

The statistical methods and calculations to be used for evaluation of quantitative performance are described in the following subsections. All measurement results from both the Xact 625 monitor (i.e., hourly data) and the reference method (i.e., 24-hour integrated data) will be reported as ambient metals concentrations [e.g., in nanograms per cubic meter of air (ng/m^3)] and graphically displayed in the test report. Care will be taken to assure that the volume units used in reported metals concentrations from the two methods are identical (i.e., standard conditions, or actual volume). Non-detect results from the reference method for any metal will be flagged as such, and excluded from any data comparisons. Although data comparisons are nominally based on a 24 hour sampling period, the Xact 625 data will actually consist of 23 measurements of 1-hour duration and one measurement of 0.5- hour duration on each test day (due to the performance of 0.5-hour internal QA checks by the Xact 625 each day). It is planned that the 0.5-hour measurement will be made from 12:30 to 1:00 am, following the performance of the internal QA checks between midnight and 12:30 am. To assure accurate comparisons, the reference sampling will also be conducted over the same 23.5 hour duration within each 24-hour period.

The ambient concentration results from the reference method and the Xact 625 metals monitor are calculated based on the known sample flow rate and sample duration of each respective method. Consequently, no further normalization of the concentration results relative to those factors will be necessary. However, the Xact 625 readings on each test day consist of 23 1-hour measurements and one 0.5-hour measurement, with all sampling done at the same flow rate (i.e., 16.7 L/min). Consequently, integration of the Xact 625 results over a daily 23.5 hour time period requires simple arithmetic averaging of the 24 total concentration results, but with the 0.5-hour result given half the weight of the 23 1-hour results, i.e.:

$$23.5 \text{ Hour Average} = (0.5c_1 + c_2 + \ldots c_{24})/23.5 \qquad (1)$$

Where c_1, c_2, …c_{24} are the metals concentrations reported each hour by the Xact 625. The resulting daily concentration results from the Xact 625 metals monitor will be compared to the corresponding 23.5-hour integrated sample results from the reference method.

Statistical comparisons are not needed for evaluation of the qualitative performance of the metals monitor, i.e., the operational factors. Those factors will include the ease of operation and data collection, the need for consumables and routine maintenance, the amount and nature of any waste generated, and the extent of repair or replacement efforts. These factors will be evaluated based on observations of the OEPA site operators, EPA Region 5 staff, and Battelle staff, and by communication with vendor representatives. Examples of information to be recorded may include the daily status of diagnostic indicators, use or replacement of any consumables, the effort or cost associated with maintenance or repair, vendor effort (e.g., time on site) for repair or maintenance, the duration and causes of any down time or data acquisition failure, operator observations about technology startup, ease of use, clarity of the vendor's training, and user-friendliness of monitor software. Battelle will summarize these observations to aid in describing the metals monitor performance in the test report.

B1.2.1 Comparability

The overall comparability of the continuous monitors to the reference method will be assessed by comparison of the ambient metals concentrations determined simultaneously by the two methods. For each target metal, comparability will be assessed from a linear regression of the data using the reference method results as the independent variable and the continuous monitor results as the dependent variable, as follows.

$$C_i = (m \cdot R_i) + b \qquad (2)$$

Where R_i is the ith reference measurement (for a 23.5-hour period), C_i is the average of the Xact 625 continuous metals monitor measurements over the same 23.5-hour time period, and b and m are the intercept and slope of the linear regression, respectively. Comparability will be reported in terms of the slope and intercept values obtained from the linear regression for each metal determined by the metals monitor and the reference method. In the absence of bias, the

regression slope would be 1.0 and the intercept would be zero. The slope and intercept values for each metal will be reported, along with associated 95% confidence intervals, by means of the LINEST linear regression function in Microsoft Excel®. The test report will indicate whether each reported slope or intercept value is significantly different from 1.0 or from zero, respectively (i.e., whether the 95% confidence interval excludes a slope of 1.0 or an intercept of zero, respectively).

B1.2.2 Correlation

The degree of correlation between the metals monitor results and the reference method results will be determined by the coefficient of determination (r^2) of the linear regression between the 23.5-hour reference method results and the corresponding averages of the metals monitor measurements over the same time periods. A value of r^2 close to 1.0 means that the amount of random error is small, that is, the variability in the continuous measurements is almost entirely explained by the variability in the reference measurements. The r^2 value will be determined and reported separately for each metal measured.

B1.2.3 Bias

The bias of the metals monitor readings will be evaluated in terms of the percent difference of the average metals monitor readings relative to the corresponding 23.5-hour reference method result. That is, for each target metal (i) in each 23.5-hour reference method period (j), bias for that metal in that period ($B_{i,j}$) will be calculated as:

$$B_{i,j} = \frac{(\text{Average Monitor Reading} - \text{Reference Reading}) \times 100}{\text{Reference Reading}} \qquad (3)$$

Before conducting the calculation shown in Equation 3, the 23.5-hour average concentrations from the Xact 625 for each test day will be compared to the quantitation limit of the Xact 625 for each target metal. The quantitation limit is a defined value for each target metal using the Xact 625, and roughly corresponds to 10% uncertainty in an interference-free situation.[(5)] Daily 23.5-hour averages for any metal that are less than the Xact 625 quantitation limit for that metal will be excluded from the calculation of bias using Equation 3. Quantitation limits established by the vendor for the Xact 625 are listed in Appendix A.

As noted in Table 3, up to 112 $B_{i,j}$ values will be obtained for each target metal. The impact of low metals concentrations on the bias results will be investigated by reporting the range, median, mean, and standard deviation of the individual $B_{i,j}$ results for each metal. Also, the individual bias results for each metal will be plotted against the ambient metal concentration to determine whether bias is a function of the ambient concentration of the metal.

B1.2.4 Data Completeness

Data completeness (DC) will be determined in two different ways. The first will be as the percentage of all hours in the field period in which the metals monitor reported ambient metals data, relative to the total number of hours in the field period:

$$DC1 = (\#\ \text{Hours data return/Total hours in period}) \times 100 \qquad (4)$$

Data completeness will also be calculated as the percentage of all 24-hour reference measurement periods during which the monitor reported at least 12 hours of monitoring data (i.e., produced data for at least half of the reference monitoring period):

$$DC2 = (\#\ \text{Periods with} \geq 12\ \text{hours monitor data/\# Reference periods}) \times 100 \qquad (5)$$

Both forms of data completeness will be calculated for each target metal.

B2. SAMPLING METHODS

Ambient PM_{10} will be sampled for reference metals analysis using a Partisol Plus 2025 Sequential Ambient Particulate Sampler (Thermo Scientific, Hopkinton, MA) equipped with a PM_{10} size-selective inlet (http://pj.b5z.net/i/u/1640578/f/Thermo/Partisol_Plus_2025.pdf). This sampler is designated by EPA as Reference Method RFPS-1298-127 for sampling of ambient PM_{10}. The Partisol Plus 2025 exchanges 47 mm diameter Teflon® sample filters automatically after pre-set sampling intervals, and can hold up to 16 filter cassettes, sufficient for more than two weeks of unattended daily 24-hour sampling. Sample air is pulled by an internal pump through the filter currently being sampled, with the sample air flow regulated by a mass

flow controller. Ambient temperature and pressure readings are used along with the mass flow control to maintain a constant volumetric sampling rate. Previously sampled and yet-to-be-sampled filters are sealed off within the Partisol Plus 2025 to prevent contamination. The sampler stores sampling and meteorological data in internal memory, including the average ambient temperature, ambient pressure, and average ambient relative humidity for each filter sample.

The Partisol Plus 2025 will be operated by OEPA personnel at the test site. The Partisol Plus 2025 sampling flow rate will be set at approximately 16.7 L/min (equivalent to approximately 1 m^3/hr). Samples will be collected every day throughout the field period, with a 23.5 hour sampling duration (i.e., one sample per day), so that reference sampling does not take place during the 0.5 hours per day when the Xact 625 performs its internal QA checks. That 0.5 hour period will be scheduled at the same time of day on every test day so that reference and Xact 625 sampling intervals are consistent throughout the test period. Collected filter samples can be removed from the Partisol Plus 2025 sampler without interruption of sampling onto the current filter. Collected samples will be shipped to ERG in batches on a weekly basis, or more frequently, for extraction and analysis. OEPA staff will download sampling data from the Partisol Plus 2025 internal software by means of an RS232 interface, and provide the electronic files of such data to EPA Region 5, OAQPS, Battelle, and ERG.

B3. SAMPLE HANDLING AND CUSTODY

Reference samples for determination of ambient PM_{10} mass and metals concentration will be collected by OEPA staff using the Partisol Plus 2025 sampler, according to EPA Method IO-3.1[6] that calls for sampling of ambient air at a known constant flow rate for a specified time interval through filters that have been weighed before sampling, and that are then weighed again after sampling. The atmospheric PM_{10} mass concentration [in micrograms per cubic meter ($\mu g/m^3$)] is determined from the difference in the filter weights and the volume of air sampled. Method IO-3.1 also defines procedures for the extraction of metals from the collected particulate filter for subsequent analysis.

Pre-weighed, individually identified 47 mm Teflon filters will be prepared by ERG and shipped to OEPA personnel. OEPA will install clean filters in the sampler at the field site, document the identification of each filter, collect ambient PM_{10} samples according to the schedule defined in Section B2, and ship the collected filter samples back to ERG for weighing, extraction, and reference metals analysis. Filters will be returned to ERG in the same individual filter containers in which they were sent to the field. The particulate trace metals to be determined are non-volatile and not reactive, so no storage temperature or holding time requirements apply to the collected samples. Chain-of-custody (COC) forms (see example in Appendix B) will accompany the filter samples on return to ERG, and for each reference sample will indicate the filter identification number, sampling date, start and stop times, flow rate, and requested analytical method. COC forms will track sample release from the sampling location to the analysis laboratory. COC forms will be signed by the person relinquishing samples once that person has verified that the COC form is accurate. Upon arrival at the analysis laboratory, COC forms will be signed by the person receiving the sample once that person has verified that all samples identified on the COC forms are present. All COC forms will be delivered to the VTC and maintained with the test records.

B4. ANALYTICAL METHODS

The reference PM_{10} filter samples will be analyzed by ERG for metals content according to EPA Method IO-3.5[7] where collected PM_{10} is digested using microwave or hot acid according to EPA Method IO-3.1[6] and then analyzed by inductively coupled plasma/mass spectrometry (ICP/MS). All samples will be digested using the same procedure to ensure comparability. This analysis will be performed according to Standard Operating Procedures (SOPs) that are incorporated into ERG's QAPP for sampling and analysis in EPA's national monitoring programs.[8] The QC requirements of Method IO-3.5 will be followed, including the use of blanks, quality control samples (QCSs), laboratory fortified blanks (LFBs), internal standards, and tuning solutions, and an instrument performance demonstration program that includes duplicate samples, standards, blanks, interference check standards, continuing calibration blanks and verification standards, and laboratory control and matrix spikes.[8] The QC requirements and acceptance criteria in Table 8 of Method IO-3.5[7] will serve as the MQO's for the metals analysis.

The Method IO-3.5 target analyte list for this test is shown in Table 4, along with estimated detection limits (in ng/m^3) provided by ERG for Method IO-3.5 based on the sampling conditions described in Section B2 and extraction of the entire 47 mm filter. Not all of the analytes targeted by the metals monitor (Table 2) are on the analyte list for Method IO-3.5; bromine, calcium, iron, potassium, rubidium, strontium, and titanium are on the Xact 625 list (Table 2) but not on the Method IO-3.5 list (Table 4).

Table 4. Target Analytes and Expected Detection Limits for Reference Method IO-3.5

Metal	Expected[a] Detection Limit (ng/m^3)	Metal	Expected[a] Detection Limit (ng/m^3)
Antimony	0.010	Manganese	0.051
Arsenic	0.017	Mercury	0.13
Barium	0.083	Molybdenum	0.017
Cadmium	0.26	Nickel	0.57
Chromium	5.6	Selenium	0.14
Cobalt	0.019	Thallium	0.0080
Copper	0.66	Thorium	0.020
Lead	0.082	Vanadium	0.010
		Zinc	5.5

a: The values listed were obtained from ERG personnel as their best estimates of method performance

Reference and Xact 625 metals monitor data will be compared for all metals that are common to the analyte lists of the two measurement methods, and present at detectable levels in ambient air. Data for additional metals determined by either method will also be reported to EPA, though not used in the metals monitor evaluation. ERG will complete the metals analysis of reference filter samples and provide draft analytical results to EPA Region 5, Battelle, and OEPA within approximately seven days after receiving each batch of samples. ERG will provide final analytical results on each batch of samples in electronic format to EPA Region 5, Battelle, and OEPA immediately after completion of internal QA review of the results. The ERG analytical laboratory will be subject to a TSA during analysis of the first week's field samples, as stated in Section A8.

As an additional analytical effort, the first 25% of reference filter samples (i.e., approximately 28 samples) collected in the field will be subjected to laboratory metals analysis by XRF prior to

analysis by ICP-MS. The laboratory XRF analysis will be conducted by EPA's National Exposure Research Laboratory according to an established EPA SOP.[9] Collected reference method samples will be shipped with accompanying chain of custody (CoC) documentation from the field to the EPA laboratory for XRF analysis. That analysis is non-destructive, so upon completion of the XRF analysis the samples will be forwarded with accompanying CoC documentation to ERG for ICP-MS analysis. The ICP-MS results will be the reference data used to assess the performance of the Xact 625, and the laboratory XRF analysis is not a component of the ETV verification effort. However, the laboratory XRF data may provide additional insights for EPA and the Xact 625 vendor into the performance of the Xact 625, by virtue of employing the same analytical principle as the Xact 625 measurement.

As a check on sample integrity in the XRF analysis process, field blank samples will be submitted along with the reference samples for XRF analysis, at a rate of 20% (i.e., five field blanks accompanying the 25 reference samples). Blank results exceeding the 95% confidence interval of the results from field blanks not subjected to XRF analysis will be taken as evidence of contamination for an individual metal. In addition, the statistical comparison of ICP-MS data to Xact 625 data defined in Section B1.2 will be carried out separately for the reference samples subjected to EPA laboratory XRF analysis and for those not subjected to that analysis. The results from the two sets of data for comparability, correlation, and bias will be evaluated and reported separately. Only if the 95% confidence intervals of the linear regression slope, linear regression intercept, and average bias overlap will the two sets of reference data be judged equivalent and combined into one complete data set for reporting and comparison of ICP-MS and Xact 625 results. If those confidence intervals do not overlap, the two data sets will be reported and compared to the corresponding Xact 625 results separately. In that case the 75% of reference samples not subjected to XRF analysis will be the primary reference data used to verify the Xact 625. This approach will indicate whether the additional sample handling and XRF analysis process affected the reference samples.

The comparison of ICP-MS and laboratory XRF analysis results from the 25% of reference samples subjected to both analyses is not part of the ETV verification process. None of the XRF

data will be included in the verification report. Consequently the procedures for comparing those data are not presented in this QAPP.

B5. QUALITY CONTROL

Quality control efforts for operation of the Xact 625 metals monitor undergoing testing will include documenting that OEPA and EPA staff have been properly trained to operate the monitor, and performing both automated and manual QC checks at vendor-specified intervals. Adequacy of training will be assured by requiring the metals monitor vendor to certify that specific EPA and OEPA staff have been trained on operation of the metals monitor and are approved to operate the monitor for testing.

Automated QC checks performed by the Xact 625 during continuous operation will consist of the following.

- Internal energy alignment check, performed by XRF analysis of a copper rod; conducted over a 15-minute period starting at midnight each day
- Upscale rod check, performed by XRF analysis of a metal rod containing chromium, lead, copper, and cadmium; conducted once per day over a 15-minute period
- Flow check, conducted at the same time as the upscale rod check, determines the Xact 625 sample air flow by insertion of a second mass flow meter into the flow path
- Palladium rod stability check, conducted by XRF analysis of a palladium rod in every ambient sample analysis (i.e., hourly)

These automated checks serve to monitor trends in the Xact 625's status. The criterion for stability of the palladium rod check is 10 to 15%, that for the flow check is 10%, and that for the upscale rod check is more stringent, at 5% for each of the four metals. Variations in check results outside these criteria result in flags in the monitor's data. The data from the metals monitor will be reviewed by CES personnel and OEPA field staff, and CES personnel will assess whether adjustment or maintenance of the Xact 625 monitor is needed based on the observed data flags. If such actions are needed CES will perform them or instruct OEPA staff how to perform them.

Manually initiated QC checks will be conducted upon installation of the Xact 625 at each field site. In long-term operation these checks would then be repeated quarterly. The manually initiated checks will consist of the following.

- Quarterly flow check, in which a NIST-traceable flow measurement standard is used to verify the Xact 625's sample flow measurement. Agreement of the NIST and Xact 625 flow measurements must be within 10%, or the Xact 625's flow sensor will be recalibrated at 15.0, 16.7, and 18.4 L/min.
- Quarterly leak check, in which the Xact 625's sample inlet is plugged, the sample pump is allowed to create a vacuum in the system, and then the pump is turned off. A rise in the internal system pressure of greater than 150 mm Hg/min is indication of a leak and calls for identification and correction of the leak.

Quality control efforts for the reference sampling will include operation of the Partisol Plus 2025 according to the manufacturer's instructions, periodic calibration checks of the sampling flow rate, collection of field blank filter samples for analysis, and completion of chain-of-custody forms for all samples collected. These efforts are familiar to the OEPA field staff, as they are carried out for all ambient monitoring efforts conducted by OEPA staff. The target metals will be determined on all field blank filters, and all blank results for each target metal will be reported as part of the test data. The blank results will not be subtracted from the sample results.

Quality control efforts for the laboratory analysis of metals by IO-3.5[6] will include continuation of the chain-of-custody of received field samples, and adherence to the QC requirements of the method, as noted in Section B4. Those requirements, including required frequencies and appropriate corrective actions in case of inadequate results, are stated in Section 10 (Calibration and Standardization) and Section 11 (Quality Control) of Method IO-3.5.[7] Examples of corrective actions that may be called for based on calibration, instrument performance, and QC checks include instrument adjustments, repetition of sample preparation, repetition of procedures, dilution of samples, and flagging of data.[7]

B6 INSTRUMENT/ EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The metals monitor undergoing testing will be subjected to a daily inspection and diagnostic check by OEPA staff to assure proper operation. Instrument internal diagnostics and the completeness of data output (relative to the expected daily set of analyses) will be the primary items noted in this daily check. Any issues or questions will trigger communication with the vendor of the metals monitor to assess whether maintenance and/or repair is needed.

The Partisol Plus 2025 sampler will be inspected by OEPA staff prior to the field test and every time collected filter samples are recovered from the sampler throughout the test. This inspection will use a brief checklist of key Partisol Plus 2025 operating indicators, such as flow readings, displays, and data acquisition status. The consistency of the automatic changeover of filters, and the sampling flow rate displayed and recorded by the sampler's software, will be the primary features addressed in the inspection. Any issues or questions will trigger communication with the vendor of the Partisol Plus 2025 to assess whether maintenance and/or repair is needed.

Laboratory analytical equipment used for the reference metals analysis will be tested, inspected, and maintained according to manufacturer's instructions and ERG SOPs. The results of maintenance will be documented according to the laboratory procedures.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

The Xact 625 metals monitor undergoing testing will be calibrated by the vendor prior to delivery to the first field site. Once the Xact 625 is installed at the field site, a calibration check will be performed using NIST-traceable thin film standards of four metals: chromium, selenium, lead, and cadmium. Each thin film standard will be analyzed by the Xact 625 at each of three excitation energy conditions. The analysis results for each metal must agree with the original calibration within 10%, or a re-calibration will be done. If needed, such a re-calibration will involve sending the thin film analysis spectra to CES for calculation of a new calibration curve. Also upon installation at the field site, a QA blank check will be performed, where a blank portion of the Xact 625's sampling tape is analyzed by the monitor. The blank tape is expected to show no detectable levels of the target analytes. Failure of the blank check will require installation of a new tape and conducting the check again.

In long term operation of the Xact 625, both the calibration check and the QA blank check are performed on a quarterly basis. In this verification these checks will be performed after installation of the monitor at each of the two test sites. The metals monitor vendor will conduct or direct any needed maintenance, repair, and/or recalibration as indicated by the QC checks and calibration checks and restore the monitor to working order for continuation of testing. The nature, extent, and cost of any maintenance, repair, and recalibration efforts will be noted and included in the final report.

Calibration of the Partisol Plus 2025 sampler will be conducted by OEPA staff prior to the start of testing. Recalibration will be performed by OEPA when necessary based on the flow rate checks, i.e., recalibration will be triggered by sampler flow rate readings that differ by more than ±5% from the target flow rate set at the start of field sampling.

Battelle will provide NIST-traceable measurement standards for temperature, barometric pressure, and air flow with which to conduct a PE audit of the Partisol Plus 2025 sampler. All such measurement standards will have been calibrated by Battelle's Instrument Laboratory within no more than 6 months prior to its use in the PE audit.

Calibration of the reference metals analysis equipment will be conducted by ERG, based on the QC procedures stated in Method IO-3.5.[7] The Method's requirements focus on both initial demonstration of analytical performance and continuing checks to confirm that performance is being maintained. The several types of samples used to document method performance include blanks, QCSs, LFBs, internal standards, and tuning solutions, and instrument performance samples that include duplicates, standards, blanks, interference check standards, continuing calibration blanks and verification standards, and laboratory control and matrix spikes.[7] The balances used to determine PM_{10} mass on the Teflon filters will also be calibrated by ERG using NIST-traceable mass standards.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Upon receipt of any sampling supplies or consumables, OEPA and/or Battelle staff will visually inspect and ensure that the materials received are those that were ordered and that there are no visual signs of damage that could compromise the suitability of the materials. If damaged or inappropriate goods are received they will be returned or disposed of and arrangements will be made to receive replacement materials. Forty-seven (47) mm diameter, 2.0 µm pore size (e.g., Zefluor FPTPT247 or equivalent) Teflon filters will be used with the Partisol Plus 2025 sampler. Certificates of analysis or other documentation of analytical purity will be checked for all standards (e.g., PE audit standards) to ensure suitability for this verification test. Unsuitable materials will be returned or disposed of and arrangements for the receipt of replacement materials will be made.

B9 NON-DIRECT MEASUREMENTS

No non-direct measurements will be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by OEPA, EPA Region 5, or Battelle staff during this verification test. All manually-recorded data will be recorded in permanent ink in LRBs or on data forms. The Xact 625 generates electronic data files. These files will be uniquely named, downloaded to a personal computer, and transferred to the VTC on a weekly basis, or more often, by the OEPA staff operating the metals monitor in the field. The metals monitor data will then be uploaded to the project SharePoint site. Data will be uniquely identified in the Xact 625 data files by the date and time of the measurement and the identity of the metal.

Laboratory analytical results for all field samples and blanks will be provided to Battelle and EPA by ERG as a Level III data package in electronic format, and will include supporting QC information documenting the quality of the data obtained. Sample results will be uniquely identified by means of the filter number, the associated date and time of sample collection, and the identity of the target metal. The ERG metals analytical data will be uploaded by Battelle to the project SharePoint site.

Table 5 summarizes the types of data that may be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the monitoring systems being tested will be documented by OEPA or vendor staff in the laboratory record book maintained at the field site. Report formats will include all necessary data to allow traceability from the raw data to final results.

Records received by or generated by any testing staff during the verification test will be reviewed by a Battelle staff member within 14 days of receipt or generation, respectively, before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle technical staff member familiar with the verification test, but not the staff member who originally received or generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. In addition, any calculations or data comparisons performed by Battelle will be spot-checked by Battelle technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this QAPP.

Battelle will provide technology test data and associated reference data (including records; data sheets; notebook records) from the first day of testing within one day of receipt to EPA for simultaneous review. The goal of this data delivery schedule is prompt identification and resolution of any data collection or recording issues. These data will labeled as preliminary and will not have had a QA review before their release.

Table 5. Summary of Data Recording Process

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates, times, and details of test events	ETV LRB, field sampling records, or electronically	Start/end of test event	OEPA, EPA Region 5, or Battelle staff	Used to organize/check test results; manually incorporated in data spreadsheets as necessary
Metals monitor operating information, maintenance, down time, etc.	ETV LRB, field sampling records, or electronically	Start/end of maintenance, down time, change of conditions, etc.	OEPA, EPA Region 5, or Battelle staff	Incorporated in verification report as necessary
Metals monitor readings	Recorded electronically and downloaded to computer daily	Recorded continuously by each metals monitoring system	OEPA staff for transfer to Battelle	Converted to spreadsheet for statistical analysis and comparisons
Reference method sampling information	ETV LRB, field sampling records, chain of custody forms	At each reference method sampling interval	OEPA staff	Converted to spreadsheets for statistical analysis and comparisons
Reference method analytical results	Electronic files of analytical results (Level II data package)	Upon comple-tion and quality review of each batch of samples	ERG analytical staff	Converted to spreadsheets for statistical analysis and comparisons

SECTION C

ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in this verification test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this QAPP is to establish mechanisms necessary to ensure this. Internal quality control measures described in this QAPP, that is peer reviewed by a panel of outside experts, implemented by the technical staff and monitored by the VTC, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the VTC, who will contact the Battelle AMS Center Manager, Battelle AMS Center Quality Manager, EPA AMS Center Project Officer, and EPA AMS Center Quality Manager if any deviations from this QAPP are observed. The VTC will describe any deviations from this QAPP in a teleconference or by email, and once a path forward is determined and agreed upon with EPA, a deviation form will be completed. Any deviations from this QAPP will be noted in the verification report, along with a discussion on the deviation's impact (if any) on data quality.

Technical staffs, including OEPA, EPA Region 5, and Battelle, have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the VTC, who will work with the Battelle Quality Manager to resolve any issues. Action will be taken by the VTC and appropriate testing staff to identify and address the issue, and minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this verification test.

C1.1 Performance Evaluation Audit

Battelle will conduct a PE audit of both the sampling and analysis portions of the reference metals measurements. The PE audit will be carried out early in the field monitoring period, so that any issues raised by the PE results can be addressed early in the period.

Battelle will conduct a PE audit of the Partisol Plus 2025 reference sampling system, by checking the sampler flow rate and measurements of atmospheric pressure and temperature during operation at the field site. This PE audit will be done by Battelle staff in the first week of reference method sampling, using a flow measurement device, thermocouple temperature sensor, and barometric pressure sensor calibrated by Battelle's Instrument Laboratory with NIST-traceable standards. The acceptance criterion for the PE flow audit is that the sampler flow be within ±5% of the target sampler flow rate. Acceptance criteria for temperature and barometric pressure will be ± 2 °C and ± 5 millibars, respectively. Failure to meet the PE audit criterion will result in a repeat of the audit with the same and/or a new audit device; continued failure to meet the PE audit criterion will trigger inspection and/or recalibration of the reference sampler.

Battelle will also conduct a PE audit of the reference metals analysis by submitting one or more sample solutions of selected target metals to ERG for analysis. These PE audit samples will be prepared by Battelle by dilution of aqueous metals standards traceable to the National Institute of Standards and Technology (NIST). The metals concentrations in the PE audit samples will be unknown to ERG, except that the samples will be prepared to be within the normal calibration range of ERG's analytical equipment for Method IO-3.5. The PE audit samples will contain at least eight of the 22 target metals listed in Table 2 and among those will be Pb and Mn. The metals PE audit samples will be submitted to ERG within the first three weeks of the field period. The acceptance criterion for the PE audit is that the concentrations reported by ERG be within ±10% of the prepared concentrations of the PE audit samples. Failure to meet this criterion will result in a reanalysis of the PE audit samples, or preparation and submission of new PE audit samples; continued failure to meet the PE audit criterion will trigger inspection and/or recalibration of the ERG analytical equipment.

C1.2 Technical Systems Audits

The Battelle QAO will perform a TSA during performance of both laboratory and field-testing activities. The purpose of these audits is to ensure that the verification test is being performed in accordance with the AMS Center QMP[1] and this QAPP. In the TSA, the Battelle QAO may compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures. The Battelle QAO will prepare a project-specific checklist

based on the QAPP requirements to guide the TSA, that will include a review of the test location and general testing conditions; observe the testing activities; and review laboratory record books. The Battelle QAO will submit an initial TSA report (with no corrective actions documented) to the EPA Quality Manager and VTC within 10 business days after completion of the audit. A copy of each final TSA report (with corrective actions documented) will then be provided to the EPA AMS Center Project Officer and Quality Manager within 20 business days after receipt of comments on the initial TSA report from the EPA Quality Manager and VTC. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

C1.3 Audit of Data Quality

The Battelle QAO, or designee, will audit at least 10% of the sample results data acquired in the verification test and 100% of the calibration and QC data versus the QAPP requirements. During this audit, the Battelle QAO, or designee, will trace the data from initial acquisition (as received from the vendor's technology), through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit of data quality (ADQ) will be checked. Data must undergo a 100% validation and verification by technical staff (i.e. VTC, or designee) before it will be assessed as part of the data quality audit. All QC data and all calculations performed on the data undergoing the audit will be checked by the Battelle QAO. Results of the ADQ will be documented using the checklist and reported to the VTC and EPA within 10 business days after completion of the audit. A final ADQ that assesses overall data quality, including accuracy and completeness of the technical report, will be prepared as a narrative and distributed to the VTC and EPA within 10 business days of completion of the audit.

C1.4 QA/QC Reporting

Each assessment and audit will be documented in accordance with Section 3.3.4 of the AMS Center QMP.[1] The results of all audits will be submitted to EPA within 10 business days as noted above. Assessment reports will include the following.

- Identification of any adverse findings or potential problems

- Response to adverse findings or potential problems
- Recommendations for resolving problems. (If the QA audit identifies a technical issue, the VTC or Battelle AMS Center Manager will be consulted to determine the appropriate corrective action
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others

C2 REPORTS TO MANAGEMENT

During the field and laboratory efforts, any QAPP deviations will be reported immediately to EPA. The Battelle Quality Manager and/or VTC, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. A summary of the required assessments and audits, including a listing of responsibilities and reporting timeframes, is included in Table 6. If serious quality problems exist, the Battelle Quality Manager will notify the AMS Center Manager, who is authorized to stop work. Once the assessment report has been prepared, the VTC will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. The QAPP and final report will be reviewed by the EPA AMS Center Quality Manager and the EPA AMS Center Project Officer. Upon final review and approval, both documents will then be posted on the ETV website (www.epa.gov/etv).

Table 6. Summary of Assessment Reports[a]

Assessment	Prepared By	Report Submission Timeframe	Submitted To
Each TSA (Initial)	Battelle	10 business days after TSA is complete	EPA ETV AMS Center
Each TSA (Final)	Battelle	TSA response is due to QAO within 10 business days of receipt from QAO TSA responses will be verified by the QAO and provided within 20 business days after receipt of comments on initial report	EPA ETV AMS Center
ADQ #1	Battelle	10 business days after receipt of first data set	EPA ETV AMS Center
ADQ #2	Battelle	10 business days after completion of the verification report review	EPA ETV AMS Center

[a] Any QA checklists prepared to guide audits will be provided with the audit report

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS

The key data review and data verification requirements for this test are stated in Section B10 of this QAPP. In general, the data review requirements specify that data generated during this test will be reviewed by a Battelle technical staff member within two weeks of generation of the data. The reviewer will be familiar with the technical aspects of the verification test. This process will serve both as the data review and the data verification, and will ensure that the data have been recorded, transmitted and processed properly. Furthermore, this process will ensure that the monitoring systems data were collected under appropriate testing.

The data validation requirements for this test involve an assessment of the quality of the data relative to the data quality objectives for this test referenced in Section A8. Any deficiencies in these data will be flagged and the data will be excluded from any statistical comparisons, unless these deviations are accompanied by descriptions of their potential impacts on the data quality.

D2 VERIFICATION AND VALIDATION METHODS

Data verification is conducted as part of the data review as described in Section B10 of this QAPP. A visual inspection of any handwritten data will be conducted to ensure that all entries were properly recorded or transcribed, and that any erroneous entries were properly noted (i.e., single line through the entry, with an error code, such as “wn” for wrong number, and the initials of the recorder and date of entry). Electronic data from the metals monitor, Partisol Plus 2025 sampler, and analytical equipment used during the test will be inspected to ensure proper recording. Calculations used to transform the data will be reviewed to ensure the accuracy and the appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspection of the equations used for the calculations and verification of selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for

analytical instrumentation) will be reviewed by inspection and, when feasible, verified by handheld calculator, or standard commercial office software.

To ensure that the data generated from this test meet the goals of the test, a number of data validation procedures will be performed. Sections B and C of this QAPP provide a description of the validation safeguards employed for this verification test. Data validation efforts include the completion of QC activities and the performance of a TSA as described in Section C. The data from this test will be evaluated relative to the measurement data quality objectives described in Section A8 of this QAPP.

An ADQ will be conducted by the Battelle QAO to ensure that data review, verification, and validation procedures were completed, and to assure the overall quality of the data.

D3 RECONCILIATION WITH USER REQUIREMENTS

The purpose of this verification test is to evaluate the performance of a continuous monitor for trace metals in ambient PM_{10}. To meet the requirements of the potential user community, including particularly EPA Region 5, EPA OAQPS, and OEPA, input to this QAPP has been provided by external experts. Additional performance data regarding operational characteristics of the metals monitor will be collected by verification test personnel. The data review, verification, and validation procedures described above will assure that data meeting the test requirements are accurately presented in the verification report generated from this test, and will assure that data not meeting these requirements will be appropriately flagged and discussed in the verification report.

This QAPP and the resulting ETV verification report will be subjected to review by the vendor, EPA, and expert peer reviewers. The reviews of this QAPP will help to improve the design of the verification test and the resulting report such that they better meet the needs of potential users of ambient particulate metals monitoring systems.

SECTION E

REFERENCES

1. Battelle, Quality Management Plan for the ETV Advanced Monitoring Systems Center, Version 7.0, U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, November 2008.

2. U.S. EPA, Environmental Technology Verification Program Quality Management Plan, EPA Report No: 600/R-08/009 EPA/600/R-03/021, U.S. Environmental Protection Agency, Cincinnati, Ohio, January 2008.

3. School Air Toxics Monitoring Initiative, information available at http://www.epa.gov/ttn/amtic/airtoxschool.html.

4. Quality Assurance Project Plan for the EPA School Air Toxics Monitoring Program – Phase II Multimetals (Final) – Revision 1.0, U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Research Triangle Park, North Carolina, May 2011.

5. Currie, Lloyd A., Detection and Quantitation in X-Ray Fluorescence Spectrometry. In X-ray Fluorescence Analysis of Environmental Samples, Dzubay, Thomas B., ed.; Ann Arbor Science, Ann Arbor, MI, 1978; pgs. 289-305.

6. Compendium method IO-3.1, Selection, Preparation, and Extraction of Filter Material, in *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air*, EPA/625/R-96/010a, U.S. Environmental Protection Agency Office of Research and Development, June 1999.

7. Compendium Method IO-3.5, Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS), in *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air*, EPA/625/R-96/010a, U.S. Environmental Protection Agency Office of Research and Development, June 1999.

8. Quality Assurance Project Plan for Support for the EPA National Monitoring Programs (NMOC, UATMP, PAMS, HAPs, and NATTS), EPA Contract EP-D-09-048, ERG, Morrisville, North Carolina, 2011.

9. Standard Operating Procedure for Elemental Analysis of Particulate Matter on Membrane Filters by the Kevex XRF Spectrometer, SOP ECAB-048.1E (Alternative Identification SOP WDE-04-18), U.S. EPA National Exposure Research Laboratory, Research Triangle Park, NC, November 4, 2009.

APPENDIX A

QUANTITATION LIMITS OF THE XACT 625

Element	Atomic Number	60 Minute QL (ng/m^3)
K	19	16.73
Ca	20	6.38
Ti	22	2.69
V	23	2.05
Cr	24	2.04
Mn	25	2.00
Fe	26	3.93
Co	27	3.54
Sn	50	18.00
Sb	51	4.69
Ba	56	6.68
Mn	25	2.80
Fe	26	5.37
Co	27	2.24
Ni	28	1.60
Cu	29	1.89
Zn	30	1.64
Ga	31	0.76
Ge	32	0.85
As	33	0.81
Se	34	0.99
Br	35	1.31
Rb	37	2.43
Sr	38	3.16
Y	39	3.82
Mo	42	6.94
Pt	78	1.62
Au	79	1.62
Hg	80	1.33
Tl	81	1.30
Pb	82	1.54
Bi	83	1.68
Ag	47	30.60
Cd	48	40.64
In	49	47.90
Sn	50	52.72

APPENDIX B

EXAMPLE PM_{10} METALS SAMPLE DATA SHEET

ERG Eastern Research Group Inc.

ERG Lab ID #

PM_{10} / TSP METALS DATA SHEET

Lab Pre-Samp.

Site Code: _____ Collection Date: _____

City/State: _____ Duplicate Event (Y/N): _____

AQS Code: _____

Field Setup

Set-Up Date: _____ Operator: _____

Field Recovery

Recovery Date: _____ Sample Duration (i.e. 24 hr): _____

Status: Valid Void (Circle one)

Lab Recovery

Received by: _____ Date: _____

Status: Valid Void (Circle one)

If void, why: _____

PM_{10} / TSP METALS

Sample Date	Start Time	End Time	Total Time	System #	Filter #	Lab ID
	Start MFC	End MFC	Avg Flow (L)			
Sample Date	**Start Time**	**End Time**	**Total Time**	**System #**	**Filter #**	**Lab ID**
	Start MFC	End MFC	Avg Flow (L)			
Sample Date	**Start Time**	**End Time**	**Total Time**	**System #**	**Filter #**	**Lab ID**
	Start MFC	End MFC	Avg Flow (L)			

Comments: _____

White: Sample Traveler Canary: Lab Copy Pink: Field Copy