QUALITY ASSURANCE PROJECT PLAN (QAPP)

TULSA EVANS/FINTUBE

Parcels # 1, #2, and #3
150/186 North Lansing Avenue
Tulsa, Oklahoma
Site-Specific Brownfields Cleanup Grant
City of Tulsa
ENERCON Project No. CTYTUL0050

Prepared For:
The City of Tulsa, Oklahoma
175 West 2nd Street, 15th Floor
Tulsa, Oklahoma 74103

Date:
Draft 3
January 2016

Prepared By:
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LIST OF ACRONYMS

ABCA ..... Analysis of Brownfield Cleanup Alternatives
ACBM ..... Asbestos Containing Material
ASTM ..... American Society for Testing and Materials
ESA ..... Environmental Site Assessment
GIS ..... Global Information System
LBP ..... Lead Based Paint
ODEQ ..... Oklahoma Department of Environmental Quality
ODOL ..... Oklahoma Department of Labor
PCM ..... Phase Contrast Microscopy
PLM ..... Polarized Light Microscopy
PDF ..... Portable Document Format
QA ..... Quality Assurance
QAPP ..... Quality Assurance Project Plan
QA/QC ..... Quality Assurance/Quality Control
RFP ..... Request for Proposal
TDA ..... Tulsa Development Authority
TCLP ..... Toxicity Characteristic Leaching Procedure
TEM ..... Transmission Electron Microscopy
TIA ..... Tulsa Industrial Authority
TPWD ..... Tulsa Public Works Department
USEPA ..... United States Environmental Protection Agency
XRF ..... X-Ray Fluorescence

TITLE AND APPROVAL SHEET

Project Title: **City of Tulsa – Evans/FinTube Project QAPP**

City of Tulsa
175 East 2nd Street, Suite 260
Tulsa, Oklahoma 74103

Implementing Organization: City of Tulsa

QAPP Effective Date: 2/17/2016

Approving Officials:

For this project the City of Tulsa's Economic Development Director will function as the Project Manager and have primary responsibility for implementation of the Clean-up Grant. The City of Tulsa's Brownfields Program Manager will function as the Assistant Project Manager and have secondary responsibility for the implementation of the program and QA. The City of Tulsa's Project Officer for Evans/Fintube on the staff of the Engineering Department will function as the QA Manager and perform primary QA activities.

The USEPA Project Officer will ensure that the policies, goals, and objectives of the Clean-up Grant are achieved. This QAPP must be reviewed and approved by the Project Manager, Assistant Project Manager, QA Manager, USEPA Project Officer, and the USEPA Brownfields Project Officer prior to implementation and commencement of project activities.

Project Manager, Clay Bird:

Signature — *Date* 2-10-16

Assistant Project Manager, Adrienne Russ:

Signature — *Date* 2-10-16

QA Manager, Doug Wilson:

Signature — *Date* 2-10-16

USEPA Region 6 Project Officer, Paul Johnson:

Signature — *Date* 2/17/16

DISTRIBUTION LIST

Each person listed on the approval sheet and each person listed in the Project Organization List below will receive a copy of this Quality Assurance Project Plan (QAPP). Individuals taking part in the project may request additional copies of the QAPP from the personnel listed in the Project Organization List.

Project Organization List

NAME	TITLE	ENTITY	PROJECT ROLE
Clay Bird	Economic Development Director	City of Tulsa	Project Manager
Adrienne Russ	Brownfields Program Manager	City of Tulsa	Assistant Project Manager
Doug Wilson	Evans/Fintube Project Officer	Engineering Services Department	QA Manager
Paul Johnson	USEPA Brownfields Project Officer	USEPA	USEPA Region 6 Brownfields Project Officer
Michelle Barnett	Senior Project Manager	Enercon Services, Inc.	Consultant Project Manager
TBD	Contractor	TBD	Contractor Project Manager

QUALITY ASSURANCE PROJECT PLAN (QAPP)

Professional Environmental and Engineering Services for
Evans/FinTube
Site-Specific Brownfields Cleanup Grant
City of Tulsa, Oklahoma

1.0 INTRODUCTION

The USEPA has developed the QAPP policy as a source for project managers and planners to document the type and quality of data required for environmental decisions and to describe the methodology for the collection and assessment of data. The Quality System mandated by the USEPA includes the development, review, approval, and implementation of a QAPP. All USEPA funded projects that involve the acquisition of environmental data generated from direct measurement activities collected from other sources or compiled from computerized data bases/information systems must be conducted in accordance with an approved QAPP. These requirements apply to all environmental programs funded by the USEPA that acquire, generate, or compile environmental data through contracts, work assignments, delivery orders, task orders, cooperative agreements, interagency agreements, State-EPA agreements, State, local, or Tribal Financial Assistance/Grants, Research Grants, and in response to statutory or regulatory requirements or consent orders.

No portion of the project work covered by this requirement is to be implemented without prior approval of the QAPP except for emergency circumstances requiring immediate action to protect human health and the environment or operations conducted under police powers. Subject to these emergency exceptions, it is the responsibility of the organization performing the work to assure that no environmental data is gathered or evaluated prior to approval of the QAPP by the appropriate project personnel. The organization performing the work must ensure that the QAPP is implemented as approved and that all personnel involved in the project have direct access to the most current approved version. Any revisions to this QAPP must be performed by the originator with appropriate documentation and approval by the same authorities that performed the original review.

The QAPP may be prepared by a USEPA organization, a contractor, an assistance agreement holder, or other Federal agency under an interagency agreement, however, final approval is reserved for the USEPA Brownfields Project Officer (USEPA PO). This QAPP has been developed by Enercon Services, Inc. (ENERCON) to serve as a method for the documentation of planning, implementation, assessment, and QA/QC procedures implemented in accordance with Part B requirements of ANSI/ASQC E4-1944 for the Professional Environmental and Engineering Services for Evans/FinTube project at hand. To the best of our knowledge and interpretation this QAPP has been prepared in accordance with EPA QA/R-5 (EPA Requirements for Quality Assurance Project Plans – Final March 2001).

1.1 QAPP Plan Amendments

All amendments and/or revisions to this QAPP must be reviewed and approved prior to implementation by the Project Manager, Assistant Project Manager, QA Manager and Consultant's Project Manager, with final approval from the USEPA PO. A record of changes and subsequent reviews shall be maintained in Appendix A.

2.0 PROJECT MANAGEMENT

Project management includes the elements and personnel in place to ensure that the project has defined goals plus participants understand the goals and approach to be used. Furthermore, project management ensures that the planning procedures are properly documented. The title and approval sheet and distribution list are included on pages i and ii previously.

2.1 Project/Task Organization

The individuals and organizations participating in this project along with their specific roles and responsibilities are broken down into primary groups known as Principal Data Users, Decision Makers, QA Manager, Environmental Consultant, and Other Subcontractors as described below. An Organizational Chart is included as Figure 3 of Appendix C in this QAPP.

Principal Data Users

Project Manager

Mr. Clay Bird, City of Tulsa: For this project the City of Tulsa's Economic Director will function as the Project Manager and have primary responsibility for implementation and oversight of the Clean-up Grant. He is responsible for overall contracting and management of the grant; project performance; development of the QAPP; and shares in the responsibility of review/approval of the QAPP on behalf of the City. Mr. Bird will also be the primary individual responsible for Contractor oversight, including review, evaluation and decision-making regarding the Contractor's recommendations. The Project Manager has the authority to stop work at the project.

Assistant Project Manager

Ms. Adrienne Russ, City of Tulsa: For this project City of Tulsa's Brownfields Program Manager will function as the Assistant Project Manager and have secondary responsibility for implementation and oversight of the Clean-up Grant and QA/QC. She is responsible for overall contracting and management of the Grant; project performance; development of the QAPP; and shares in the responsibility of review/approval of the QAPP on behalf of the City. Additionally, she will be responsible for staffing and maintaining the City of Tulsa quality assurance program throughout completion of project activities and coordinating day to day management of this project to assure compliance with the Brownfields Program objectives.

QA Manager

Mr. Doug Wilson, City of Tulsa: The City of Tulsa's Evans/Fintube Project Officer will assume the responsibility of QA Manager for this project. The QA Manager is an environmental professional on the staff of the City Engineering Services Department, and has primary responsibility for QA/QC oversight of environmental contracting during implementation of the Clean-up Grant. The QA Manager is responsible to ensure the work done on this project using secondary data is completed in accordance with the QAPP. Primary responsibilities of the QA Manager include the following:

- Provides technical assistance to ensure environmental compliance.
- Shares in responsibility of review/approval of the QAPP.
- Responsible for maintaining the official, approved QAPP and ensuring that all involved parties have the most recent version of the QAPP and receive all amendments.

- Responsible for QA/QC oversight of environmental contracting during implementation of the Clean-up Grant.
- Serves as the official QA/QC contact for all intramural and extramural QA activities for the City of Tulsa's Brownfields Program.
- Reports directly, as a partner, to the City of Tulsa regarding all QA matters.
- Reviews and concurs with QAPP, and submits to the QAPP to the USEPA Project Officer prior to the planned initiation of secondary environmental data review activities.
- Works with the City of Tulsa's Brownfields-related personnel to take appropriate corrective action when, where, and however needed, during the proposed project activities.
- Assures that all secondary environmental data review activities are accomplished in strict compliance with QAPP requirements.
- The City of Tulsa's QA Manager will have the authority to stop work at the project.

Federal and State of Oklahoma Representatives

Paul Johnson, USEPA Region 6 Project Officer: The USEPA PO ensures that USEPA policies, goals, and objectives are achieved. The USEPA PO's responsibilities include:

- Assists the City of Tulsa Brownfields Program QA/QC staff.
- Provides overall resources to accomplish the implementation of the USEPA Brownfields Program.
- Routinely evaluates the Brownfields Program's effectiveness
- Reviews and provides final approval of the QAPP, including all subsequent revisions.

When necessary, the Assistant Project Manager and/or QA Manager will coordinate with all appropriate State Agencies in a manner that ensures that compliance with all applicable State regulatory requirements are achieved. These agencies may include the Oklahoma Department of Environmental Quality (ODEQ), Oklahoma Department of Labor (ODOL), and/or others, as appropriate.

ODEQ staff that may be assigned to this project include:

Aron Samwell, ODEQ Brownfields Program Manager

Consultants

The Environmental Consultant selected for this project is Enercon Services, Inc. (ENERCON). Herein after, this role is referred to simply as Consultant and is organized as follows:

Consultant Project Manager

Ms. Michelle Barnett, PE, Senior Project Manager: Ms. Barnett will function as the Consultant Project Manager and have primary responsibility for the overall management of all project activities including:

- Provides consultant's final QA/QC of secondary data.
- Interacts with City of Tulsa, and agency staff with regard to the project, provides progress reports and participates in routine work progress meetings.
- Oversees overall project work and scheduling.
- Assures compliance with the QAPP for use of secondary data.
- Assures QA/QC and proper project documentation is completed and maintained.
- Submits project closeout documents to the appropriate parties.

- The Consultant Project Manager will have authority to stop work at the project.

Mr. Kenny Ground, CIH, Consultant QA/QC Manager: Mr. Ground will function as the Consultant QA/QC Manager and have primary responsibility for the oversight of the following project activities:

- Provides consultant's final QA/QC of secondary data.
- Assures compliance with the QAPP for use of secondary data.
- Assures QA/QC and proper project documentation is completed

Figure 3 (Organizational Chart) is included in Appendix C and depicts the overall summary of key personnel associated with this project. The organization chart provides a functional overview of the team that will be used to complete the scope of work, along with lines of authority.

2.1.1 Key Decision Makers

The key decision makers for this project include the following representatives:

- Clay Bird, City of Tulsa's Economic Development Director, is the key decision maker on behalf of the City of Tulsa in regards to site development.
- Paul Johnson, USEPA Region 6 Project Officer, USEPA, is the key decision maker on behalf of the USEPA for approval of the QAPP.
- Aron Samwell, ODEQ Brownfields Program Manager, is the key decision maker on behalf of the State of Oklahoma.
- OKDOL is the key decision maker on asbestos and worker protection issues.

2.1.2 Project Meetings

The following meetings are planned for the project:

Pre-Bid Meeting:A Pre-Bid Meeting shall be held in order to give prospective abatement/remediation contractors the opportunity to review the existing site conditions; confirm estimated quantities; discuss bid requirements; and to clarify any uncertainties concerning the scope and schedule of work. The Pre-Bid meeting shall be considered mandatory for all prospective bidders.

Pre-Work Meeting: A Pre-Work Meeting shall be held to review and resolve any uncertainties of the QAPP and all other plans and specifications prior to commencement of the work. The Pre-Work Meeting is to be held via teleconference, at the project site, or other suitable location prior to commencement of onsite activities. Individuals on the project distribution list and all other appropriate individuals are to be notified of the Pre-Work Meeting.

Weekly Progress Meetings: A meeting will be held weekly to review progress against the planned work schedule and to discuss existing or anticipated problems. Meetings will be held via teleconference, at the site, or other suitable locations. The meeting will also be utilized to provide updates on data acquisition and review, address safety issues, ensure maintenance of quality standards, discuss pending changes and substitutions, and discuss any other items that could affect timely completion of the work. The meeting will be conducted by the Consultant Project Manager or their designee. Minimal attendance at these meetings will include the Consultant Project Manager, or their designee, and one representative from the Contractor. Representatives of the City of Tulsa, ODEQ and others will attend at their discretion. These meetings may be held informally or waived by the Consultant Project Manager at their discretion. A

written record will be maintained as a summary within the monthly Progress Reports. Individuals from the Consultant Team on the project distribution list and all other appropriate individuals are to be notified 72 hours prior to the progress meetings.

Called Meeting: A special meeting will be called by the Consultant Project Manager whenever a significant problem or deficiency is noted or is anticipated. The purpose of the called meeting will be to define and resolve a significant problem or recurring work deficiency that has not been resolved. This will be done by defining the problem/deficiency, reviewing potential solutions, and selecting a course of action to resolve the problem/deficiency. Required attendance at the meeting will depend upon the nature of the problem or deficiency. The Consultant Project Manager, as appropriate, will conduct the meeting, record the proceedings, and maintain a copy in central electronic files.

Project Close-out Meeting: To facilitate the collection of final reports, invoices, and grant required documentation, a Project Close-Out Meeting shall be held. The meeting will be coordinated and facilitated by the Project Manager or QA Manager. Minimal attendance at this meeting will include the Consultant Project Manager, or their designee, and one representative from the Contractor.

2.2 Problem Definition and Background

The Tulsa Evans/Fintube is located at 150/186 North Lansing in Tulsa, Oklahoma. The Evans Building Complex was formerly a steel manufacturing facility that contained a foundry on the northern end. The Fintube Building Complex was formerly used as a metal manufacturing facility and a producer of heat exchangers that consisted of a concrete reservoir, a forge, and welding and fabrication shops. The site has also been a scrap metal recycling facility and a storage yard for a wrecker service and highway construction equipment and materials.

A Phase II Environmental Site Assessment (ESA) conducted in June 2010 by ALL Consulting identified the potential presence of asbestos-containing building materials (ACBM) and lead-based paint (LBP). The City of Tulsa is seeking to remediate these environmental hazards in an effort to increase the safety of the site and prepare for redevelopment efforts.

Currently, the Evans/FinTube site remains vacant, and is suffering from natural decay leading to hazardous conditions in the interior of the building and the soil and groundwater. These problems are prohibiting renovation efforts and ultimate reuse of the building. ACBM are becoming deteriorated leading to the possible disbursement of asbestos fibers into the air, lead paint is chipping and peeling off causing the possible dispersion of lead in the form of dust and paint chips.

The City of Tulsa applied for and received Clean-up Grant Funds for clean-up of these hazards. Funds from this grant will be utilized to develop an abatement project design for ACBM and LBP, contractor specifications, public outreach efforts, and ultimately abatement activities to remove ACBM and LBP hazards in preparation of site renovations.

2.3 Project/Task Description

The project seeks to maximize available grant funding to abate identified ACBM and lead paint within a limited time schedule. The specific project tasks include:

TASK 1 Grant Oversight

This task is the responsibility of the Assistant Project Manager. No additional data needs to be gathered in order to complete this task in accordance with the Workplan.

TASK 2 Document Review and Abatement/Remediation Design

The task will begin with the gathering and evaluation of secondary environmental data. During the gathering and evaluation of these data sources, the Consultant will be responsible to gather and evaluate the data provided by the City of Tulsa. This data will include previous inspection reports, laboratory data, and available regulatory records for the project. No new data will be developed by the Consultant during this task. The secondary data gathered during this task will be evaluated by the Consultant Project Manager and the QA Manager in accordance with this QAPP. The purpose of this evaluation will be to determine the validity and usefulness of the data in the performance of future tasks and to identify data gaps.

This task will also involve the development of an Asbestos Abatement Project Design, herein after referred to as the Project Specifications. These documents will outline the materials to be addressed by the selected Contractor, the prevailing regulations which govern such work, safety requirements, and final clearance criteria. The documents will be prepared under the direction of the Consultant's Project Manager by appropriately trained and licensed staff. An Oklahoma licensed Asbestos Project Designer will provide the final authoring of the abatement project design.

The QA Manager will perform final review of the abatement and remediation design documents prior to distribution during the bid process or submittal to regulatory agencies.

TASK 3 Advertisement for Bid

The project will be advertised for bid to qualified asbestos and lead paint abatement and remediation contractors and through the City of Tulsa's standard procurement process for public projects using a standard Request for Proposal (RFP). No secondary data or new data will be generated or required by this process.

The City of Tulsa's Project Manager, Assistant Project Manager, and QA Manager will review all bid documents prior to publication and verify that proper procurement procedures are followed. In addition to the standard procurement rules of the City of Tulsa, the QA Manager will verify that the grant specific procurement rules of the EPA are addressed.

TASK 4 Pre-Bid Meeting

At least 10 business days prior to the bid expiration date, a mandatory Pre-Bid Meeting will be held for all prospective bidders. Copies of inspection reports, project specifications, available project drawings, and required bid documents will be made available to prospective bidders. No additional data will be gathered or generated during this process. The Consultant Project Manager will facilitate the Pre-Bid Meeting. Prospective bidders will be required to complete an attendance roster to verify attendance. The formal portion of the Pre-Bid Meeting will be recorded via audio tape. Following the formal portion of the meeting, prospective bidders will be permitted a minimum of 2 hours to tour the project site.

Prospective bidders will be permitted to measure and quantify materials during the project site tour. However, this data will remain property of the bidder for purposes of developing their cost estimates and will not be formally reviewed by the Consultant for quality or accuracy.

Questions and answers generated through the Pre-Bid Meeting will be summarized by the Consultant Project Manager and published to all prospective bidders in a series of bid addendums. A schedule and deadline for questions will be announced at the Pre-Bid Meeting.

TASK 5 Bid Selection

Sealed bids will be received until the deadline published in the RFP and all associated bid addendums. The Consultant Project Manager will assist the City of Tulsa in the evaluation of required submittals as defined in the RFP. The QA Manager will utilize a standardized checklist to evaluate each bid package and determine if the bid is a "responsive bid". "Non-responsive" bids will receive no further evaluation.

Bids determined to be "responsive" will be evaluated in accordance with the City of Tulsa's standard procurement process. The apparent successful bidder will be announced and appropriate notification will be made to all bidders.

TASK 6 Abatement/Remediation Activities

The Contractor will be responsible for conducting abatement and remediation activities in accordance with the approved Project Specifications. These specifications will detail both environmental and safety sampling and analysis requirements. Data will be generated by both the Contractor and the Consultant during this task and may include:

- Bulk asbestos sampling and analysis.
- Work area, containment, and perimeter asbestos air sampling and analysis.
- Worker breathing zone asbestos air sampling and analysis.
- Bulk Lead paint analysis by laboratory results or by field instrumentation.

The Consultant Project Manager or his assigned staff is responsible for verifying that data management and verification occurs in accordance with the appropriate sections of this QAPP.

TASK 7 Final Inspections

A final inspection of the project will be scheduled by the Consultant Project Manager and attended by a representative of the Contractor and the QA Manager. The inspection will serve as the final verification of project performance and adherence with specifications. The inspection is anticipated to be visual in nature and no additional data generation is expected.

2.4 Quality Objectives and Criteria

Detailed Performance Measures

This project is primarily interested in the following list of likely performance measures:

1. Successful abatement of known ACBM quantities as identified in existing inspection reports in accordance with federal and state regulations governing asbestos abatement as evidenced by agency approval of a Final Abatement Report.

2. Successful abatement of the lead based paint and associated contaminated media in accordance with federal and state regulations governing lead paint removal.

3. Protection of site workers through adherence to a Site Specific Health and Safety Plan (HASP) and evidenced by data collected through breathing zone samples.

4. Protection of the public through adherence with Project Specifications and evidenced by the data collected through work area, perimeter, and clearance samples.

5. Protection of the environment through adherence with Project Specifications and evidenced by appropriately executed waste manifests.

6. Informing the public of project activities by publishing at least two informational notices regarding the project.

Quality Objectives

To the extent that these performance measures rely on the generation of data, this QAPP seeks to verify that the quality objectives are appropriate for the regulatory and non-regulatory decisions to be made based upon that data. The data quality objectives will take into account both the best practices for similar projects and the resources available for this project. If necessary, the Project Manager will rely upon EPA's *Generic Guide to Statistical Aspects of Developing and Environmental Results Program* (2003) for advice in making decisions related to optimizing the following aspects of data quality for this project:

Precision

Precision is the measurement of agreement or reproducibility among replicate samples of the same media under prescribed similar conditions. It is normally expressed as the relative percent difference (RPD) between two values.

Collecting field duplicate samples customarily assesses sample collection precision. Field duplicate samples are used to evaluate errors associated with sample heterogeneity, sampling methodology and analytical procedures. The analytical results from these samples are important because they provide data to evaluate overall measurement precision.

Accuracy

Accuracy is a measure of the closeness of an individual measurement; or the average of a number of measurements, to the true value. Bias is the systematic or persistent distortion of a measurement process that causes error in one direction. Accuracy is normally expressed as a percent recovery.

To assess sample accuracy, field QC samples such as field blanks, are typically incorporated into the sampling scheme. The data acquired from the analysis of blanks are useful in their ability to evaluate errors, which can arise from cross-contamination. The occurrence of cross contamination can result from the improper handling of samples by field and/or lab personnel, improper decontamination procedures, improper shipment and storage, and on-site atmospheric contaminants. Therefore, to facilitate sample collection accuracy, it is essential to maintain frequent and thorough review of field procedures so that deficiencies can be quickly documented and corrected.

Representativeness

Representativeness is an expression of the degree to which a sample accurately and precisely represents a characteristic of a population, parameter variations at a sampling point or an environmental condition. Representativeness is a qualitative parameter, which relies upon the proper design of a sampling program

and proper laboratory protocol. Making certain that sampling locations are selected properly and a sufficient number of samples are collected that best satisfies this criterion. Therefore, collecting field duplicates will assess sample representativeness. Traditionally, field duplicates are by definition, equally representative of a given point in space and time.

Comparability

Comparability is defined as an expression of the confidence with which one data set can be compared to another. In most instances, the proficiency of field sampling efforts will be the determining factor that affects the overall comparability of environmental measurement data. To optimize the comparability of environmental measurement data, sample collection activities should always be performed using standardized procedures whenever possible. When performing a site investigation, adhering to the quality control criteria will facilitate these efforts.

Completeness

Completeness is defined as the measurement of the amount of data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. Data completeness is often expressed as the percentage of valid data obtained from a given measurement system. To consider data valid, it is customary to assess if a set of data satisfies all of the specified acceptance/performance criteria (accuracy measures, precision measures, etc.) to render a determination. This necessitates that the data acquired for all confirmatory analysis critical to a site investigation sampling program be validated (100%).

2.5 Special Training and Certification

Individuals conducting work on this project directly related to asbestos abatement including the Contractor, the Consultant, and all associated field staff and subcontractors, are required to have specialized training in accordance with CFR 40, Part 61, Subpart M – National Emission Standards for Hazardous Air Pollutants (NESHAP) pursuant to their individual asbestos functions. This training shall be evidenced by the possession of a valid license issued by the Oklahoma Department of Labor for the appropriate functional area.

Laboratories utilized for asbestos and lead paint related analysis must be accredited by the American Industrial Hygiene Association (AIHA) and/or participate in the National Voluntary Laboratory Accreditation Program (NVLAP).

2.6 Documents and Records

The Project Manager will be responsible for distributing copies of the approved QAPP. The QA Manager is responsible for maintaining the official, approved QAPP. Furthermore, the QA Manager will be responsible for ensuring that appropriate project personnel have the most current approved version of the QAPP and distributing all amendments. A record of all changes and approval dates shall be maintained in Appendix A.

Copies of documentation and records will be maintained in the central electronic repository and will be available for review upon request by client representatives, regulatory agency representatives, or any other authorized individuals. A copy of the signed approved QAPP will be kept on file by the QA Manager, at a minimum. All files and records will be maintained according to the City of Tulsa and

USEPA records retention policies. Pre-existing data shall be reviewed as to suitability for secondary use in adherence with the EPA New England's *Quality Assurance Project Plan Guidance for Environmental Project Using Only Existing (Secondary) Data*. USEPA's *Guidance for Data Usability in Site Assessment* document may also be used as a QA tool for the methodology used in secondary data sources. All records generated under the Brownfields Program, with exception of information that may be considered "confidential business information," are subject to the Open Records Act and are available for review to the public upon request.

Documents likely to be produced during this project include:

- Quality Assurance Project Plan (QAPP)
- Analysis of Brownfield Clean-up Alternatives (ABCA)
- Community Relations Plan
- Review of Pre-existing Data
- Project Specifications (Asbestos Abatement Project Design and Lead Based Paint Abatement Design)
- Remediation Contractor Bid Documents
- Quarterly Reports
- Contractor Progress Reports
- Asbestos Abatement and Lead Based Paint Abatement Documentation and Data
- Project Closure Documentation

2.7 Project Schedule

It is anticipated that the project will begin immediately upon approval of the QAPP by all responsible parties. The total estimated project duration is 190 calendar days. If necessary, time extension notices will be submitted by the City of Tulsa representatives in accordance with the Brownfields grant requirements to meet applicable deadlines. A complete Project Schedule is included in the appendix.

3.0 DATA SELECTION AND MANAGEMENT

The data related to this project includes existing facility inspection reports and field-generated data during abatement/remediation activities.

3.1 Sources of Existing Data

Existing data sources include Phase I Environmental Sites Assessments, Phase II Environmental Site Assessments, and Asbestos Inspection reports for the Evans/FinTube facility which are in the possession of the City of Tulsa.

3.2 Intended Use and Limitations on the Use of Existing Data

Data from previous inspection reports will be utilized to identify the source, location, and quantity of contaminated media to be addressed during the project.

Initial QA/QC will be initiated by the Consultant QA Manager and will include verification of source credibility by experienced/qualified team members performing within their disciplines and practicing professional judgment. The next layer of QA/QC will be provided during reviews by the Consultant

Project Manager or their designee. The final layer of QA/QC for the data and information acquired will be accomplished by the QA Manager and Assistant Project Manager during their reviews of Draft and Final Reports.

Data that has been prepared under an approved QAPP or other sampling document will be assumed as suitable for use in this project without further review. All secondary data sets to be used in the project must have been generated using comparable sampling and analytical methods.

Data that have incomplete or non-standard methodologies for sampling or analysis may be accepted for use, but with stated limitations. These sources will be reviewed for conformance with standard EPA protocols for sampling and analysis. USEPA's "Guidance for Data Usability in Site Assessment" document may be used as a QA tool for these data sources. In addition, data that is deemed suitable based upon methodology or a previous QAPP may yet include a qualifying statement, if the secondary source includes such.

Data that has been developed without documentation of methodologies for sampling or analysis may be rejected. However, if included, for example if it were the only data available for a particular material, the secondary data will include the disclaimer "The quality of this data is unknown." or "Data Quality Unknown" for tabular formats.

3.3 Field-Generated Data

The project will involve the field generation of data by both the Consultant and the Contractor. The major quality objective will be to collect representative data that truly reflect the site conditions before, during and after project activities. Data generated may be focused on environmental controls or protection of worker and public health.

3.4 Sampling Methods

Primary guidance for sampling methodology relative to asbestos will be 40 *CFR Chapter 1, Subchapter R - AHERA, 40 CFR, Part 61, Subpart M – NESHAP and Title 40 § 450-456 Oklahoma Asbestos Control Act, Abatement of Friable Asbestos Materials Rules 380:50-1-1 through 38:50-29-1.*

Exposure assessment, air monitoring, and analysis of airborne concentration of asbestos fibers shall be performed in accordance with 29 CFR 1926.1101.

Analysis of personnel air samples (breathing zone) shall be conducted using the methods prescribed in 29 CFR 1926.1101 to include NIOSH 2003-154 Method 7400 (PCM).

Environmental and final clearance air monitoring shall be performed in accordance with NIOSH 2003-154 Method 7400 (PCM) with optional confirmation by TEM.

Project Specifications will provide additional details regarding specific sampling efforts. In cases of conflict between Project Specifications and regulatory requirements, the more stringent requirement shall be used.

If unique sampling efforts are required for this project, a standard sampling procedure shall be prepared and included as an amendment to this report.

Primary guidance for sampling methodology relative to lead will be 29CFR, Part 1926, subpart 62 and standard industrial hygiene practices.

Field screen analysis of paint for lead weight percentage shall be performed using X-Ray Fluorescence (XRF) using manufacturer's protocols and the EPA/HUD Performance Characteristic Sheet (PCS).

Quantitative Analysis of paint for lead weight percentage shall be performed using EPA 7420 for waste disposal characterization.

Quantitative Analysis of air and/or dust for lead shall be performed using either NIOSH 7000B or 9100 methods.

RCRA rules for toxicity characteristic leaching procedure (TCLP) for characterizing wastes would also need to be implemented.

Sampling Protocols for NIOSH Method 7400 PCM Environmental Air Sampling and EPA AHERA Method TEM Air Sampling are included in Appendix E.

3.5 Calibration of Data Collection Instruments

Data collection instruments utilized for this project may include:

a) High-volume sampling pumps that can be calibrated and operated at a constant airflow up to 16 liters per minute.
b) Low-volume, battery powered, body-attachable, portable personal pumps that can be calibrated to a constant airflow up to approximately 3.5 liters per minute, and a self-contained rechargeable power pack capable of sustaining the calibrated flow rate for a minimum of 10 hours. The pumps shall also be equipped with an automatic flow control unit which shall maintain a constant flow, even as filter resistance increases due to accumulation of fiber and debris on the filter surface.
c) Single use standard 25 mm diameter cassette, open face, 0.8 micron pore size, mixed cellulose ester membrane filters and cassettes with 50 mm electrically conductive extension cowl, and shrink bands for personal air sampling.
d) Single use standard 25 mm diameter cassette, open face, 0.45 micron pore size, mixed cellulose ester membrane filters and cassettes with 50 mm electrically conductive cowl, and shrink bands when conducting environmental area sampling using NIOSH 2003-154 Methods 7400 and 7402, and the TEM method specified at 40 CFR 763 if required.
e) A flow calibrator (rotameter) capable of calibration to within plus or minus 2 percent of reading over a temperature range of minus 4 to plus 140 degrees F and traceable to a NIST primary standard within 6 months.
f) A polarized light microscope (PLM) calibrated daily to include a check of the polarizer and analyzer to ensure that they are at 90 degrees to each other, and centering of the condenser, the stage and objectives.

All data collection instruments will be calibrated in accordance with the manufacturer's specifications prior to use to help assure the collection of high-quality and representative data. Equipment calibrated by the manufacturer or vendor prior to shipment will be documented with a calibration certification shipped with the unit. Equipment which is field calibrated will be self-certified by the instrument user on a standard calibration checklist. The calibration checklist, whether generated by Consultant or Contractor, will be reviewed prior to generation of final reports. The Consultant Project Manager is responsible for final verification of calibration requirements and corrective action of all deficiencies.

3.6 Sample Handling and Custody

Samples generated during this project will be packed in laboratory provided sample containers or other containers approved by the Consultant's QA manager. Sampling identification will follow a logical pattern which is unique to each sample and which allows efficient identification of the sample's date of collection, location, and sample type. Samples will be logged on a laboratory provided chain-of-custody by the sample collector and signed and dated prior to transfer to the laboratory. Upon transfer to the analytical laboratory, the chain-of-custody will be signed and dated by the receiving lab and a copy of the completed chain will be distributed as follows: (1) copy to be retained by the laboratory and kept with the samples; (1) copy to be retained by the Contractor or Consultant's Project Manager; (1) copy to be retained by the Consultant's QA Manager.

A completed copy of each chain-of-custody will be included with all publications of laboratory analytical results.

The following guidelines should be utilized in the packing and shipping of samples:

1. When a sample is shipped to the laboratory, it must be packaged in a proper shipping container to avoid leakage or breakage. The sampler completes the chain-of-custody form for all samples in the cooler, places the form in a water-tight plastic bag, and places it inside the cooler with the samples.

2. If the samples are mailed, they must be sent by registered mail. The package may also be placed with a common carrier, such as a bus company, airline, or freight company. In any case, all receipts must be retained and should be attached to the sample tag or other permanent record after the samples arrive at their destination.

3. When transfer of custody takes place, it is the responsibility of the sampler to ensure the integrity of the samples. This includes making sure that all samples are accounted for, properly marked, sealed, and documented.

4. The field sampler is responsible for personally transporting or arranging for the shipment of samples to the laboratory. Transfer of custody of samples between field personnel and laboratory personnel authorized to receive the sample (often referred to as the laboratory sample custodian) will be accomplished by the use of the chain-of-custody form.

5. The field portion of the chain-of-custody form must be completed by the person collecting the sample and should include most of the pertinent information noted in the logbook. (The information on the form intended to be completed by the laboratory personnel includes the date and time of sample receipt and the signature of the person receiving the sample.)

6. Upon delivery of the samples to the laboratory, the laboratory sample control supervisor or designee will verify the number of sample and their identification, and inspect the seal on the cooler to ensure that it has not been tampered with. If it has not been disturbed, the cooler is opened and the chain-of-custody form is removed and signed. Field custody problems identified by the laboratory personnel will be immediately relayed by the laboratory director to the program quality assurance representative for corrective action and/or resampling.

3.7 Analytical Methods

Samples collected during this project will be analyzed by an accredited laboratory meeting the requirements of Section 2.5 of this QAPP. The anticipated laboratory analytical methods for each media type are as follows:

Media Type	Analytical Method	Container Type	Preservation	Holding Time
Bulk Asbestos	Polarized Light Microscopy (PLM) – NIOSH 9002	Whirl-Pak or plastic sandwich bag	None	>30 days
Asbestos Fibers in Air	Phase Contrast Microscopy (PCM) – NIOSH 7400	PCM canister with MCEM filter (0.8 micron pore size)	None	>30 days
Asbestos Fibers in Air	Transmission Electron Microscopy (TEM) – NIOSH 7402	PCM canister with MCEM filter (0.8 micron pore size)	None	>30 days
Bulk Lead Paint	Atomic Absorption Spectrophotometer, Flame – NIOSH 7000B	Cellulose ester membrane filter (with 0.8 micron pore size)	None	>30 days

Test methods may be modified by the Consultant Project Manager and the QA Manager as necessary based upon project needs by amendment of this QAPP.

3.8 Data Management

The acquired data and information will be catalogued and organized by property in a central repository to be accessed throughout the project. The catalogue and organization of information into the central repository and initial reviews of information gathered in order to identify large data gaps will be performed by the experienced/qualified team members performing within their disciplines and practicing professional judgment. Reviews of the secondary data will be summarized within a one-page form. The summary form for each data source will include at least the following information:

- Data summary including data source, author, site name, address, former use, and historical issues if known.
- Status of secondary data acceptance, conditional acceptance, or rejection and rational.
- The reviewers name, accreditations, and firm as well as the date of review.

The secondary data and the summary form will be maintained by the Consultant QA Manager or their designee within the central electronic repository. Every effort will be made to maintain electronic files and minimize hard copy file management.

The Consultant QA Manager will review all of the summary forms and any other data review records for completeness and identify any discrepancies or missing information. Upon his or her approval, noted on the summary form, clerical personnel will load the secondary data and summary forms into dedicated electronic files. Problems with the summary form review findings may be resolved through

communication with the reviewer, cross-referencing the review with other documents from the same site, review of regulatory standards, or documented tightened or loosening of the acceptance criteria.

At the completion of the project, documents in the repository will be reviewed by the Consultant Project Manager to ensure that referenced secondary data sources are present along with their summary forms. The repository file for each data source will include at least the following documents:

- Copies of the secondary data source, including appendices, and the summary form.
- Relevant correspondence regarding QA of the secondary data.
- Other documents relevant to the data source QA not listed here.

Once the project has been completed, and as part of project close-out the Consultant Project Manager will reviewed the repository files and CDs or DVDs of the documents will be assembled. One (1) copy will be maintained in the Consultant's file, one (1) copy will be for the City of Tulsa's files, one (1) copy for ODEQ files, and one (1) copy for EPA files. Project documentation will be assembled as work progresses and the documents become available. Final review of all reports and environmental data prepared by the Consultant will be performed by the Project Manager, QA Manager, USEPA Brownfields Project Officer, Consultant Project Manager, and Consultant QA Manager, at a minimum.

4.0 ASSESSMENT AND OVERSIGHT

Assessment and oversight procedures are needed for assessing the effectiveness of project implementation and the associated QA/QC activities. The purpose of assessment is to ensure proper implementation of the QAPP.

4.1 Assessments and Response Actions

Self-assessment and performance evaluations will be conducted weekly to evaluate the effectiveness of project implementation and determine whether QAPP procedures are being properly enforced. The self-assessments and performance evaluations will be conducted by the Consultant Project Manager, whom will have the authority to stop work in the event that non-conforming conditions are identified that cannot be remedied or resolved with immediate actions in a manner that protects the validity of the information being gathered.

The Consultant Project Manager will review the documentation required to be maintained, including but not limited to the central data repository and summary forms. A record of any significant deviations from normal procedures will be documented to ensure that corrective actions are taken to correct any noted deficiencies. Minor deviation items will be corrected on the spot. Significant deviations or recurring deviations will be recorded and addressed at the Weekly Progress Meeting. All records of deviations will be signed by the Consultant Project Manager and placed in the central repository.

4.2 Reports to Management

A meeting will be held weekly to review progress against the planned work schedule and to discuss existing or anticipated problems. Meetings will be held via teleconference, at the site, or other suitable locations. The meeting will also be utilized to provide updates on data acquisition and review, address safety issues, ensure maintenance of quality standards, discuss pending changes and substitutions, and

discuss any other items that could affect timely completion of the work. The meeting will be conducted by the Consultant Project Manager and/or Consultant Deputy Project Manager or their designee. Minimal attendance at these meetings will include the Consultant Project Manager and a representative of the Contractor. Representatives of the Owner and others will attend at their discretion. These meetings may be held informally or waived by the Consultant Project Manager at their discretion. A written record will be maintained in summary form within the monthly Progress Reports. Individuals on the project distribution list and all other appropriate individuals are to be notified 72 hours prior to the progress meetings.

5.0 DATA REVIEW – VERIFICATION, VALIDATION AND EVALUATION

This QAPP shall govern the operation of the project at all times. Each responsible party listed in the distribution list shall adhere to the procedural requirements of the QAPP and ensure that subordinate personnel do likewise.

This QAPP shall be reviewed at least quarterly to ensure that the project will achieve all intended purposes. All the persons listed in the distribution list shall participate in the review of the QAPP. The Project Manager and the QA Manager are responsible for determining that data are of adequate quality to support this project. The project will be modified as directed by the Project Manager. The Project Manager shall be responsible for the implementation of changes to the project and shall document the effective date of all changes made.

It is expected that from time to time ongoing and perhaps unexpected changed will need to be made to the project. The Project Manager shall authorize all changes or deviations in the operation of the project. Any significant changes will be noted in the next monthly or quarterly report, and shall be considered an amendment to the QAPP. All verification and validation methods will be noted in the analysis provided in the final report.

5.1 Data Review, Verification, and Validation

Initial data review, verification, and validation of all data and information acquired will be accomplished by experienced/qualified team members performing within their disciplines and practicing professional judgment. The next layer of data review, verification, and validation will be performed by the Consultant Project Manager during their reviews. The final layer of data review, verification, and validation will be accomplished by the Assistant Project Manager and QA Manager during their reviews of Draft and Final Reports. Additionally, when available the QAPPs associated with the data and/or information gathered from existing reports will be taken into consideration. During either phase of these reviews the data can be accepted, rejected, or qualified by any of these individuals.

5.2 Verification and Validation Methods

Asbestos

The following tables provide laboratory measurement performance criteria for asbestos analyzed by Polarized Light Microscopy (PLM) Method, EPA Method 600/R-93/116; Transmission Electron Microscopy (TEM) via EPA/600/R-93/116 Section 2.5; by NIOSH Method 7400 – Asbestos and Other Fibers by PCM, and NIOSH Method 7402 – Asbestos by TEM. The quantification limit for all bulk samples shall be ≤ 1%. The quantification limit for air samples taken for personal protection shall be ≤

0.1 f/cc. The quantification limit for air samples taken for re-occupancy clearance shall be ≤ 0.01 f/cc PCM and < 70 s/mm^2 (TEM).

Data Quality Indicator	Measurement Performance Criteria	QC Sample and/or Activity Used to Measure Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Bulk Samples			
Precision-Laboratory (TEM)	≥80% true positive ≤20% false negative ≤10% false positive	Verified Analysis	A
Precision – Laboratory (PLM/TEM)	TEM: <5 structures ± 1 structure 5-20 structures ± 2 structures >20 structures ± 3 structures PLM: RPD≤100	Inter-analyst QC	A
Accuracy/Bias (PLM/TEM)	Vendor-specific Limits	Standard Reference Manuals	A
Accuracy/Bias – Contamination (PLM/TEM)	Asbestos < QL	Method Blanks	A
Data Completeness (PLM/TEM)	Field 90%; Laboratory 95%	Data Completeness Check	S&A
Precision-Laboratory (PLM)	RPD≤100	Intra-analyst QC	A
Precision – Laboratory (PLM/TEM)	TEM: <5 structures ± 1 structure 5-20 structures ± 2 structures >20 structures ± 3 structures PLM: RPD≤100	Laboratory Duplicate	A
Air Samples			
Precision-Laboratory (PCM/TEM)	TEM: <5 structures ± 1 structure 5-20 structures ± 2 structures >20 structures ± 3 structures PCM: The following must be false: $[(E_1)^{1/2} – (E_2)^{1/2}] >$ $2.8 \times ((E_1)^{1/2} + (E_2)^{1/2}) \times CV/2$	Verified Analysis	A
Accuracy/Bias (PCM)	Vendor-specific limits	Daily Reference Sample	A
Accuracy/Bias – Contamination	Asbestos < QL	Media Certification Check	S&A
Data Completeness (PLM/TEM)	Field 90%; Laboratory 95%	Data Completeness Check	S&A

The following table details the Laboratory analytical quality assurance samples for asbestos analyses:

Laboratory QC	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible	Data Quality Indicator
Bulk Samples					
Method Blank (PLM/TEM)	One per day	Asbestos < QL	Re-clean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination
Verified Analysis (TEM)	1%	≥80% true positive ≤20% false negative ≤10% false positive	Reanalyze and qualify data	Analyst	Precision
Inter-analyst QC (PLM/TEM)	4% TEM 7% PLM	Vendor-specific limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias
Standard Reference Materials (PLM/TEM)	TEM: Annually PLM: 1%	Vendor-specific limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias
Intra-analyst QC (PLM)	2%	RPD≤100	Reanalyze and qualify data	Analyst and Data Validator	Precision
Laboratory duplicate (PLM/TEM)	One per 10 samples	TEM: <5 structures ± 1 structure 5-20 structures ± 2 structures >20 structures ± 3 structures PLM: RPD≤100	Reanalyze and qualify data	Analyst and Data Validator	Precision
Air Samples					
Method Blank (PCM/TEM	Two per day	Asbestos < QL	Re-clean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination
Replicated Analysis (PCM/TEM)	One per 10 samples	TEM: <5 structures ± 1 structure 5-20 structures ± 2 structures >20 structures ± 3 structures PCM: The following must be false: [(E1)1/2 –(E2)1/2] > 2.8 x ((E1)1/2 + (E2)1/2) x CV/2	Reanalyze and qualify data	Analyst and Data Validator	Precision
Daily Reference Sample	One per day	Vendor-specific limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias

Lead Based Paint

Screening samples for lead-based paint may be conducted by XRF (X-Ray Fluorescence) technology. Procedures for operation of the XRF machine will be those provided by the manufacturer. Selection of sample locations and material types will be performed in general accordance with the U.S. Department of Housing and Urban Development's *Guidelines for the Evaluation and Control of Lead-Based Paint Hazardous in Housing, Chapter 7: Lead-Based Paint Inspection*, 1997 Revision modified as appropriate for a commercial "non-target housing" "non-child occupied" facility.

Lead paint screening using the XRF will not be considered definitive data. The purpose of the sampling is to identify lead-containing paint so that the contractor performing abatement activities can appropriately protect workers that may disturb this material in accordance with OSHA construction standards. Lead paint screening will be conducted by State of Oklahoma certified lead inspectors. The method used for field inspection will be XRF utilizing an on-site Niton XRF Analyzer with a detection limit of 0.1 mg/cm^2. Use of the Niton XRF will be in accordance with the manufacturer's protocols and the EPA/HUD Performance Characteristic Sheet (PCS) for the Niton. Samples may also be collected for analysis by an accredited laboratory and in accordance with Method 7000B as an alternative to, or as supporting evidence of the results attained with the Niton XRF sampling. Representative measurements of the painted building components will be conducted throughout the subject buildings to determine the general presence of any detectable amounts of lead. In addition to having the appropriate State of Oklahoma certified inspector credentials, the sampler will have specific Niton training and manufacturer certification. RCRA rules for toxicity characteristic leaching procedure (TCLP) for characterizing wastes would also need to be implemented if disposing of the waste.

5.3 Reconciliation with User Requirements

The results obtained from the project will be reconciled with the requirements defined by the data user and/or decision makers. The data will be analyzed to determine possible anomalies and/or departures from any assumptions made during the planning phase. The data will be analyzed by experienced/qualified team members performing within their disciplines and practicing professional judgment initially followed by reviews performed by the Consultant Project Manager, Assistant Project Manager, and QA Manager. The decision makers will be made aware of any limitations associated with the data and/or information collected.

The work to be done on this project is fairly straightforward in that standard work procedures are to be used. No deviations from the QAPP are anticipated at this time. Should unforeseen conditions arise that warrant a deviation from the QAPP, the Consultant Project Manager and/or Consultant Deputy Project Manager are to notify the QA Manager and a determination will be made regarding notification of the appropriate regulatory agencies and decision makers.

Corrective action will be taken whenever data are determined unacceptable by comparison to pre-established quality control limits. Corrective actions will be the responsibility of the Consultant Project Manager or, in the case of laboratory error, the laboratories QA/QC staff.

Corrective action will, in general, consist of the following:

- Review of raw data and calculations
- Review of procedures to determine that appropriate sample collection and analytical methods were followed.
- Review of instrumentation operation, calibration, and maintenance.
- Other actions as deemed necessary by ODOL, ODEQ, and the USEPA.

As a result of the above, corrective action may be identified and will be pursued as necessary. This action may include:

- Reanalysis of sample or samples.
- Recalibration and restandardization of equipment.
- Resampling if possible.
- Additional staff training.
- Perform additional decontamination of equipment.
- Other action as deemed necessary by ODOL, ODEQ, and the USEPA.

A Deviation Record must be completed and approved by the Consultant Project Manager and QA Manager prior to any changes in work scope being performed. A copy of the Deviation Record must be placed in ENERCON's Project Records File. The Final Report will include a description of any deviations, assumptions, or limitations along with a summary of any associated reconciliation that occurred during the course of the project.

6.0 WORK COMPLETION

After completion of all work as outlined in this QAPP, the Consultant Project Manager shall deliver to the Owner's Representative via the QA Manager, one electronic copy of the Final Report containing the documents listed below. A Certificate of Work Completion and authorization for final payment will not be issued by the consultant representative until the documents and data are reviewed and approved by the client representatives, and the documents are submitted in satisfactory form. Final work product should include, at a minimum, the following;

- List of any deviations, assumption, or limitations along with a summary of any associated reconciliation.
- Certification that all work specified in the QAPP has been completed.
- Final report complete with a description of activities completed, overview of QA/QC procedures, findings, and recommendations.

APPENDIX A
(QAPP Amendments)

ENERCON
Excellence—Every project. Every day.

QAPP AMENDMENT RECORD

Date:
Project Name: Former Evans-Fintube Property – City of Tulsa, Oklahoma
Reason For Amendment:

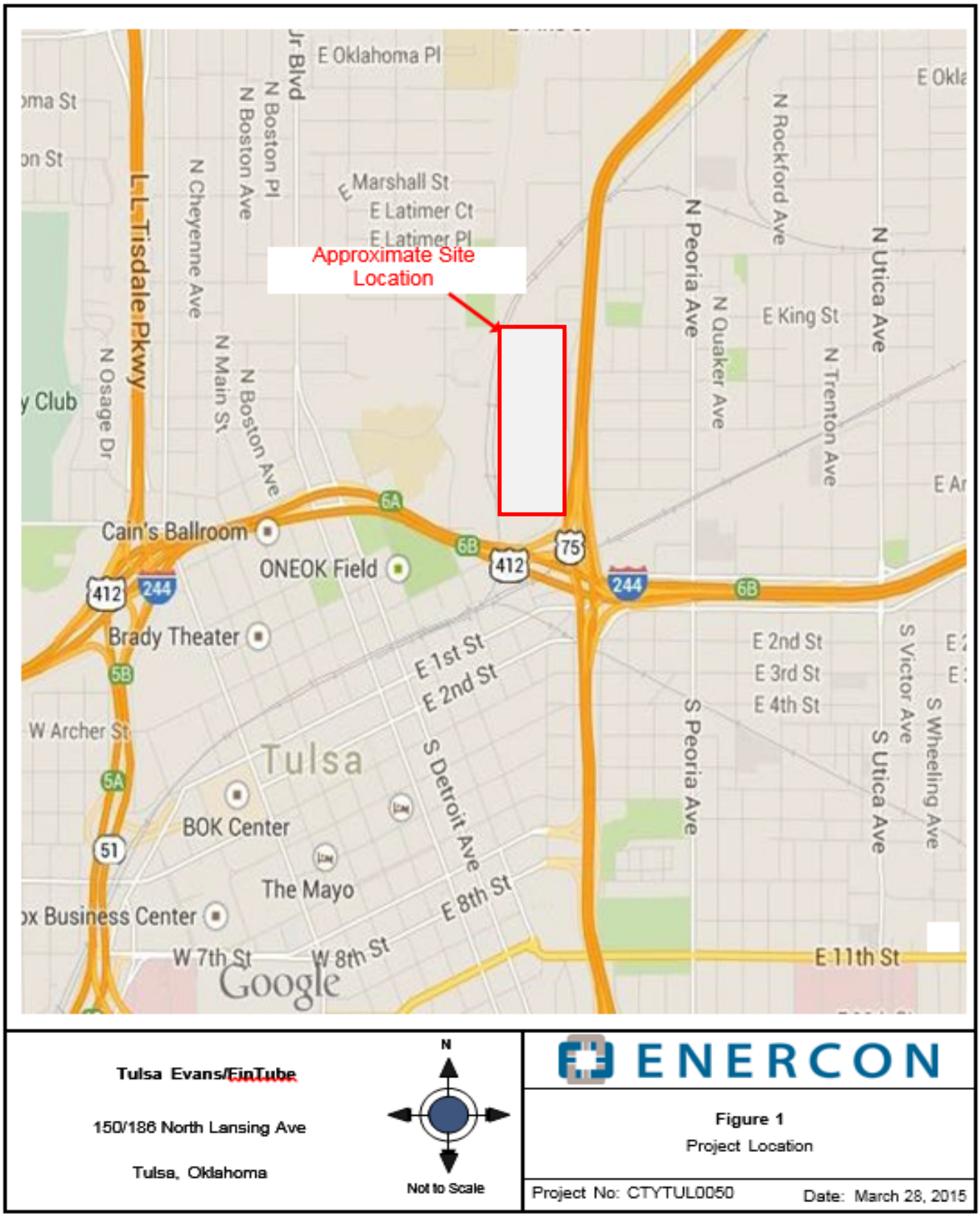
PROJECT PLAN AFFECTED	PAGE NUMBERS AFFECTED

AMENDMENT DESCRIPTION

APPROVAL (print name)	ROLE TITLE	SIGNATURE	DATE
	Project Manager		
	QA Manager		
	USEPA Brownfields Project Officer		
	Consultant Project Manager		
	Consultant Deputy Project Manager		

All amendments must be in compliance with the approved project QAPP.

APPENDIX B
(Site Figures)



Tulsa Evans/FinTube

150/186 North Lansing Ave

Tulsa, Oklahoma

N

Not to Scale

ENERCON

Figure 1

Project Location

Project No: CTYTUL0050 Date: March 28, 2015

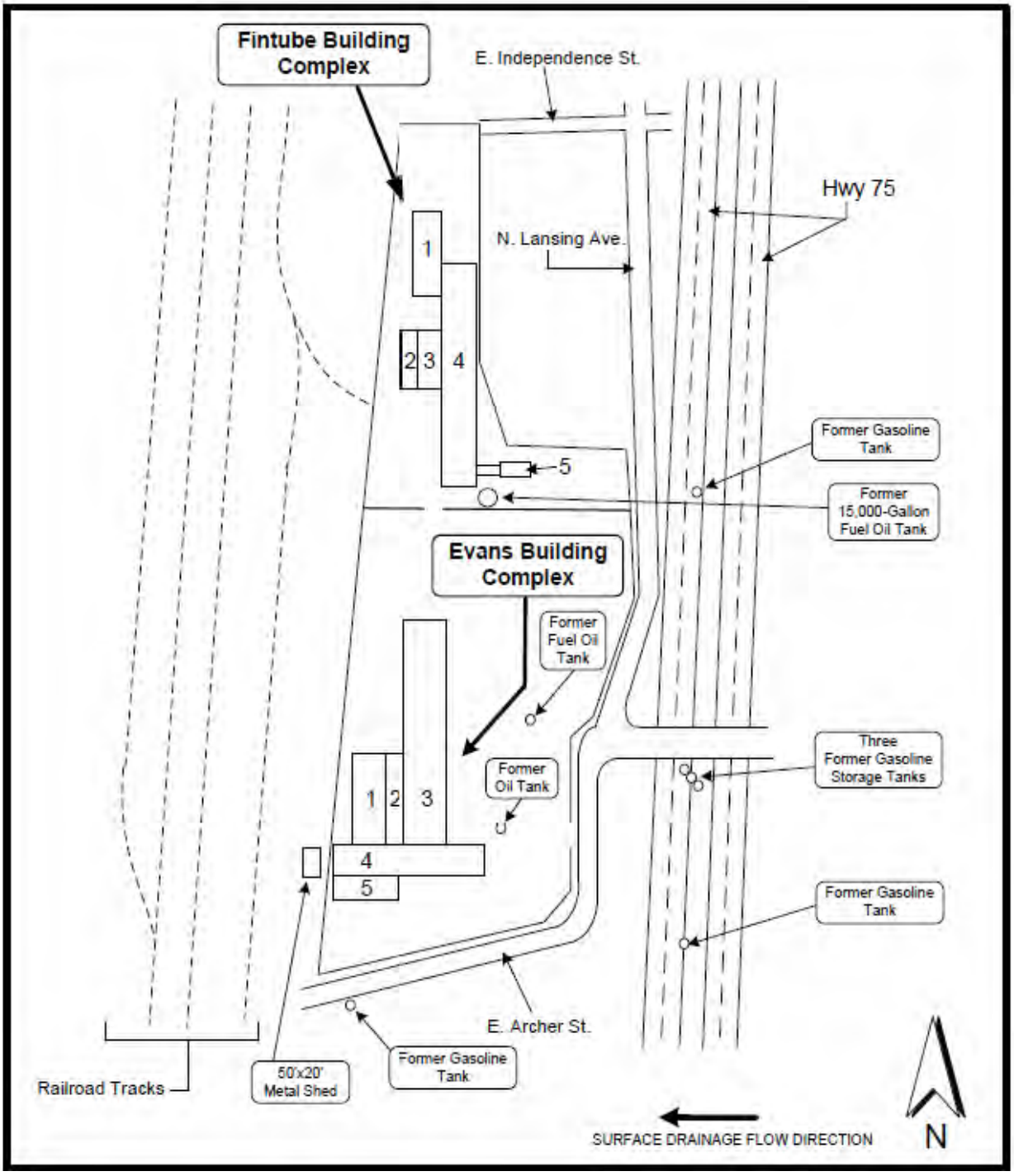


Figure 2: Site Map

Former Evans Fintube Property

APPENDIX C
(Organizational Chart)

Organizational Chart

Professional Environmental and Engineering Services for Tulsa Evans/FinTube

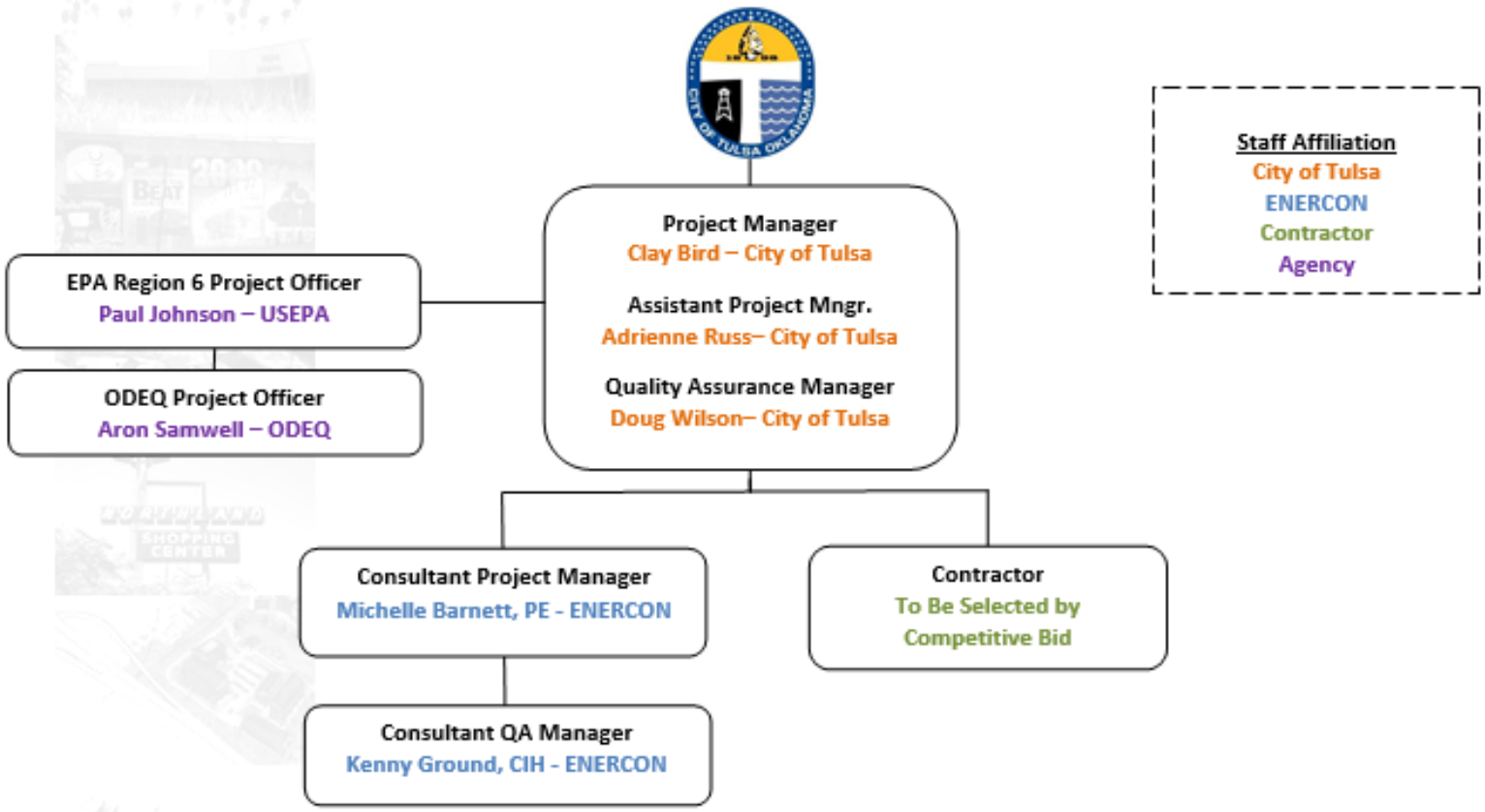


Figure 3

APPENDIX D
(Project Schedule)

PL-1201 Professional Environmental and Engineering Services for Evans/FinTube

PROJECT SCHEDULE

Task	July, 2015		August, 2015		September, 2015		October, 2015		November, 2015		December, 2015		January, 2016		February, 2016		March, 2016		April, 2016		May, 2016		June, 2016	
	1-15	16-31	1-15	16-31	1-15	16-30	1-15	16-31	1-15	16-30	1-15	16-31	1-15	16-31	1-15	16-29	1-15	16-31	1-15	16-30	1-15	16-31	1-15	16-30
1 - GRANT OVERSIGHT																								
2 - DOCUMENT REVIEW AND ABATEMENT/REMEDIATION DESIGN																								
3 - ADVERTISEMENT FOR BID																								
4 - PRE-BID MEETING																								
5 - BID SELECTION																								
6 - ABATEMENT/REMEDIATION ACTIVITIES																								
7- FINAL INSPECTIONS																								
GRANT CLOSEOUT DOCUMENTATION																								

Assumptions

Approved QAPP received by January 15, 2016

15 Days for review and approval of Abatement/Remediation Design

30 Days for final procurement of selected contractor

APPENDIX E
(Standard Protocols)

METHOD 7000B

FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 Metals in solution may be readily determined by flame (direct aspiration) atomic absorption spectrophotometry. The method is simple, rapid, and applicable to a large number of environmental samples including, but not limited to, ground water, aqueous samples, extracts, industrial wastes, soils, sludges, sediments, and similar wastes. With the exception of the analyses for dissolved constituents, all samples require digestion prior to analysis (see Chapter Three). Analysis for dissolved elements does not require digestion if the sample has been filtered and then acidified.

NOTE: Organo-metallic species may not be detected if the sample is not digested.

The following elements have been determined by this method:

ELEMENT		CASRN[a]
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Lithium	(Li)	7439-93-2
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Osmium	(Os)	7440-04-2
Potassium	(K)	7440-09-7
Silver	(Ag)	7440-22-4

Sodium	(Na)	7440-23-5
Strontium	(Sr)	7440-24-6
Thallium	(Tl)	7440-28-0
Tin	(Sn)	7440-31-5
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

[a] Chemical Abstract Service Registry Number

1.2 Lower limits of quantitation and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. The data shown in Table 1 provide some indication of the lower limits of quantitation obtainable by the direct aspiration technique. For clean aqueous samples, the quantitation limits shown in the table by direct aspiration may be extended downward with scale expansion and upward by using a less sensitive wavelength or by rotating the burner head. Quantitation limits by direct aspiration may also be extended through concentration of the sample and/or through solvent extraction techniques.

1.3 Users of this method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using this method for analysis.

1.4 Where direct-aspiration atomic absorption techniques do not provide adequate sensitivity, refer to specialized procedures such as graphite furnace atomic absorption (Method 7010) or the gaseous-hydride methods.

1.5 Other elements and matrices may be analyzed by this method as long as the method performance is demonstrated for these additional elements of interest, in the additional matrices of interest, at the concentration levels of interest in the same manner as the listed elements and matrices (see Sec. 9.0).

1.6 Prior to employing this method, analysts are advised to consult each type of procedure (e.g., sample preparation methods) that may be employed in the overall analysis for additional information on quality control procedures, development of QA acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.7 Use of this method is restricted to use by, or under supervision of, properly experienced and trained personnel, including analysts who are knowledgeable in the chemical and physical interferences described in this method. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Although methods have been reported for the analysis of solids by atomic absorption spectrophotometry, the technique generally is limited to metals in solution or dissolved through some form of sample processing (see Chapter Three). Preliminary treatment of waste water, ground water, extracts, and industrial waste is always necessary because of the complexity and variability of sample matrix. Solids, slurries, and suspended material must be subjected to a solubilization process before analysis. This process may vary because of the metals to be determined and the nature of the sample being analyzed. Solubilization and digestion procedures are presented in Chapter Three.

2.2 In direct-aspiration atomic absorption spectrophotometry, a sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp or an electrodeless discharge lamp is directed through the flame into a monochromator, and onto a detector that measures the amount of absorbed light. Absorption depends upon the presence of free unexcited ground-state atoms in the flame. Because the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectrophotometry.

3.0 DEFINITIONS

Refer to Chapter One, Chapter Three, and the manufacturer's instructions for a definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Three for general guidance on the cleaning of glassware.

4.2 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular combination in the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or when the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The addition of lanthanum will overcome phosphate interference in magnesium, calcium, and barium determinations. Similarly, silica interference in the determination of manganese can be eliminated by the addition of calcium. A nitrous oxide/acetylene gas mixture may be used to help prevent interferences from refractory compounds.

4.3 Chemical interferences may also be eliminated by separating the metal from the interfering material. Although complexing agents are employed primarily to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.

4.4 The presence of high dissolved solids in the sample may result in an interference from non-atomic absorbance such as light scattering. In the absence of background correction, this can result in false positives and/or falsely elevated values. If background correction is not

available, a non-absorbing wavelength should be checked. Signal contribution from uncorrected background can not be diagnosed through the analysis of spike recovery, nor is it compensated for by the application of the method of standard additions (MSA). If background correction is not available and the non-absorbing wavelength test indicates the presence of background interference, the sample digestates must be extracted (liquid-liquid or solid phase) prior to analysis, or another analytical method must be selected.

4.5 Ionization interferences occur when the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positively charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess (1,000 mg/L) of an easily ionized element such as K, Na, Li or Cs. Each sample and standard should contain 2 mL KCl/100 mL of solution. Use 95 g of potassium chloride in 1 L of reagent water for the KCl solution.

4.6 Spectral interference can occur when an absorbing wavelength of an element present in the sample, but not being determined, falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal. Interference can also occur when resonant energy from another element in a multielement lamp, or from a metal impurity in the lamp cathode, falls within the bandpass of the slit setting when that other metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.

4.7 The analyst should be aware that viscosity differences and/or high dissolved or suspended solids may alter the aspiration rate.

4.8 All metals are not equally stable in the digestate, especially if it only contains nitric acid and not a combination of acids including hydrochloric acid. The addition of HCl helps stabilize Sn, Sb, Mo, Ba, and Ag in the digestate. The digestate should be analyzed as soon as possible, with preference given to these analytes. Refer to Chapter Three for suggested decomposition methods.

4.9 Specific interference problems related to individual analytes

4.9.1 Aluminum -- Aluminum may be as much as 15% ionized in a nitrous-oxide/acetylene flame. Use of an ionization suppressor (1,000 µg/mL of K as KCl) as described in Sec. 4.5 will eliminate this interference.

4.9.2 Antimony -- In the presence of lead (1,000 mg/L), a spectral interference may occur at the 217.6-nm resonance line. In this case, the 231.1-nm resonance line should be used. Excess concentrations of copper and nickel (and potentially other elements), as well as acids, can interfere with antimony analyses. If the sample contains these matrix types, either matrices of the standards should be matched to those of the sample or the sample should be analyzed using a nitrous oxide/acetylene flame.

4.9.3 Barium -- Barium undergoes significant ionization in the nitrous oxide/acetylene flame, resulting in a significant decrease in sensitivity. All samples and standards must contain 2 mL of the KCl ionization suppressant per 100 mL of solution (refer to Sec. 4.5). In addition, high hollow cathode current settings and a narrow spectral band pass must be used because both barium and calcium emit strongly at barium's analytical wavelength.

4.9.4 Beryllium -- Concentrations of Al greater than 500 ppm may suppress beryllium absorbance. The addition of 0.1% fluoride has been found effective in

eliminating this interference. High concentrations of magnesium and silicon cause similar problems and require the use of the method of standard additions.

4.9.5 Calcium -- All elements forming stable oxyanions will complex calcium and interfere unless lanthanum is added. Addition of lanthanum to prepared samples rarely presents a problem because virtually all environmental samples contain sufficient calcium to require dilution to be within the linear range of the method.

4.9.6 Chromium -- An ionization interference may occur if the samples have a significantly higher alkali metal content than the standards. If this interference is encountered, an ionization suppressant (KCl) should be added to both samples and standards (refer to Sec. 4.5).

4.9.7 Magnesium -- All elements forming stable oxyanions (P, B, Si, Cr, S, V, Ti, Al, etc.) will complex magnesium and interfere unless lanthanum is added. Addition of lanthanum to prepared samples rarely presents a problem because virtually all environmental samples contain sufficient magnesium to require dilution.

4.9.8 Molybdenum -- Interferences in an air/acetylene flame from Ca, Sr, SO_4, and Fe are severe. These interferences are greatly reduced in the nitrous oxide flame and by the addition of 1,000 mg/L of aluminum to samples and standards (refer to Sec. 7.7).

4.9.9 Nickel -- High concentrations of iron, cobalt, or chromium may interfere, requiring either matrix matching or use of a nitrous-oxide/acetylene flame. A non-response line of Ni at 232.14 nm causes non-linear calibration curves at moderate to high nickel concentrations, requiring sample dilution or use of the 352.4 nm line.

4.9.10 Osmium -- Due to the volatility of osmium, standards must be made on a daily basis, and the applicability of sample preparation techniques must be verified for the sample matrices of interest.

4.9.11 Potassium -- In air/acetylene or other high temperature flames (>2800 EC), potassium can experience partial ionization, which indirectly affects absorption sensitivity. The presence of other alkali salts in the sample can reduce ionization and thereby enhance analytical results. The ionization-suppressive effect of sodium is small if the ratio of Na to K is under 10. Any enhancement due to sodium can be stabilized by adding excess sodium (1,000 µg/mL) to both sample and standard solutions. If more stringent control of ionization is needed, the addition of cesium should be considered.

4.9.12 Silver -- Since silver nitrate solutions are light sensitive and have the tendency to plate silver out on the container walls, they should be stored in dark-colored bottles. In addition, it is recommended that the stock standard concentrations be kept below 2 ppm and the chloride content increased to prevent precipitation. If precipitation is occurring, a 5%:2% HCl:HNO_3 stock solution may prevent precipitation. Daily standard preparation may also be needed to prevent precipitation of silver.

4.9.13 Strontium -- Chemical interference caused by silicon, aluminum, and phosphate are controlled by adding lanthanum chloride. Potassium chloride is added to suppress the ionization of strontium. All samples and standards should contain 1 mL of lanthanum chloride/potassium chloride solution per 10 mL of solution (refer to Sec. 7.8).

4.9.14 Vanadium -- High concentrations of aluminum or titanium, or the presence of Bi, Cr, Fe, acetic acid, phosphoric acid, surfactants, detergents, or alkali

metals, may interfere. The interference can be controlled by adding 1,000 mg/L of aluminum to samples and standards (refer to Sec. 7.7).

4.9.15 Zinc -- High levels of silicon, copper, or phosphate may interfere. Addition of strontium (1,500 mg/L) removes the copper and phosphate interference.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a hood whenever possible and, if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection when working with these reagents.

5.3 Hydrofluoric acid is a very toxic acid and penetrates the skin and tissues deeply if not treated immediately. Injury occurs in two stages; first, by hydration that induces tissue necrosis and then by penetration of fluoride ions deep into the tissue and by reaction with calcium. Boric acid and other complexing reagents and appropriate treatment agents should be administered immediately. Consult appropriate safety literature and have the appropriate treatment materials readily available prior to working with this acid. See Method 3052 for specific suggestions for handling hydrofluoric acid from a safety and an instrument standpoint.

5.4 Many metal salts are extremely toxic if inhaled or swallowed. Extreme care must be taken to ensure that samples and standards are handled properly and that all exhaust gases are properly vented. Wash hands thoroughly after handling.

5.5 Protective eyeware and/or flame shields should be used when conducting analyses by acetylene-nitrous oxide flame due to the emission of UV light.

5.6 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. For this reason, the acidification and digestion of samples should be performed in an approved fume hood.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 Atomic absorption spectrophotometer -- Single- or dual-channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for a computer or graphical interface.

6.2 Burner -- The burner recommended by the particular instrument manufacturer should be used. For certain elements the nitrous oxide burner is needed. Under no circumstance should an acetylene-air burner head be used with an acetylene-nitrous oxide flame.

6.3 Hollow cathode lamps -- Single-element lamps are preferred, but multielement lamps may be used. Electrodeless discharge lamps may also be used when available. Other types of lamps meeting the performance criteria of this method may be used.

6.4 Graphical display and recorder -- A recorder is recommended for flame work so that there will be a permanent record and that any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, peak signal, etc., can be easily recognized.

6.5 Pipets -- Class A or microliter, with disposable tips. Sizes can range from 5 to 100 uL as needed. Pipet tips should be checked as a possible source of contamination when contamination is suspected or when a new source or batch of pipet tips is received by the laboratory. The accuracy of variable pipets must be verified daily. Class A pipets can be used for the measurement of volumes equal to or larger than 1 mL.

6.6 Pressure-reducing valves -- The supplies of fuel and oxidant should be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

6.7 Glassware -- All glassware, polypropylene, or fluorocarbon (PFA or TFM) containers, including sample bottles, flasks and pipets, should be washed in the following sequence -- 1:1 hydrochloric acid, tap water, 1:1 nitric acid, tap water, detergent, tap water, and reagent water. (Chromic acid should not be used as a cleaning agent for glassware if chromium is to be included in the analytical scheme.) If it can be documented through an active analytical quality control program using spiked samples and method blanks that certain steps in the cleaning procedure are not needed for routine samples, those steps may be eliminated from the procedure. Alternative cleaning procedures must also be documented.

6.8 Volumetric flasks of suitable precision and accuracy.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade- or trace metals-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All reagents should be analyzed to demonstrate that the reagents do not contain target analytes at or above the lowest limit of quantitation.

7.2 Reagent water -- All references to water in the method refer to reagent water, unless otherwise specified. Reagent water must be free of interferences.

7.3 Nitric acid, HNO_3 -- Use a spectrograde acid certified for AA use. Prepare a 1:1 dilution with water by adding the concentrated acid to an equal volume of water. If the method blank does not contain target analytes at or above the lowest limit of quantitation, then the acid may be used.

7.4 Hydrochloric acid (1:1), HCl -- Use a spectrograde acid certified for AA use. Prepare a 1:1 dilution with water by adding the concentrated acid to an equal volume of water. If the method blank does not contain target analytes at or above the lowest limit of quantitation, then the acid may be used.

7.5 Fuel and oxidant -- High purity acetylene is generally acceptable. Air may be supplied from a compressed air line, a laboratory compressor, or a cylinder of compressed air and should be clean and dry. Nitrous oxide is also required for certain determinations. A centrifuge filter on the compressed air lines is also recommended to remove particulates.

7.6 Stock standard metal solutions -- Stock standard solutions are prepared from analytical reagent grade high purity metals, oxides, or nonhygroscopic salts using reagent water and redistilled nitric or hydrochloric acids. Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements. The stock solutions are prepared at concentrations of 1,000 mg of the metal per liter. Commercially available standard solutions may also be used. When using pure metals (especially wire) for standards preparation, cleaning procedures, as detailed in Chapter Three, should be used to ensure that the solutions are not compromised. Stability of standards will be verified through the use of the QC protocols as specified in this method. Comparison of the daily ICVs and CCVs with the calibration curve enables the standards to be prepared as needed.

7.6.1 Aluminum -- Dissolve 1.000 g of aluminum metal in dilute HCl with gentle warming and dilute to 1 L with reagent water.

7.6.2 Antimony -- Carefully weigh 2.743 g of antimony potassium tartrate, $K(SbO)C_4H_4O_6$C1/2H_2O, and dissolve in reagent water. Dilute to 1 L with reagent water.

7.6.3 Barium -- Dissolve 1.779 g of barium chloride, $BaCl_2$C2H_2O, analytical grade and dilute to 1 L with reagent water.

7.6.4 Beryllium -- Dissolve 11.659 g of beryllium sulfate, $BeSO_4$, in reagent water containing 2 mL of nitric acid (conc.) and dilute to 1 L with reagent water.

7.6.5 Cadmium -- Dissolve 1.000 g of cadmium metal in 20 mL of 1:1 HNO_3 and dilute to 1 L with reagent water.

7.6.6 Calcium -- Suspend 2.500 g of calcium carbonate, $CaCO_3$, dried for 1 hr at 180 EC in reagent water and dissolve by adding a minimum of dilute HCl. Dilute to 1 L with reagent water.

7.6.7 Chromium -- Dissolve 1.923 g of chromium trioxide, CrO_3, in reagent water, acidify (to pH # 2) with redistilled HNO_3 (conc.), and dilute to 1 L with reagent water.

7.6.8 Cobalt -- Dissolve 1.000 g of cobalt metal in 20 mL of 1:1 HNO_3 and dilute to 1 L with reagent water. Chloride or nitrate salts of cobalt(II) may be used. Although numerous hydrated forms exist, they are not recommended unless the exact composition of the compound is known.

7.6.9 Copper -- Dissolve 1.000 g of electrolytic copper in 5 mL of redistilled HNO_3 (conc.) and dilute to 1 L with reagent water.

7.6.10 Iron -- Dissolve 1.000 g of iron wire in 10 mL redistilled HNO_3 (conc.) and reagent water and dilute to 1 L with reagent water. Note that iron passivates in conc. HNO_3, and therefore some water should be present.

7.6.11 Lead -- Dissolve 1.599 g of lead nitrate, $Pb(NO_3)_2$, in reagent water, acidify with 10 mL of redistilled HNO_3 (conc.), and dilute to 1 L with reagent water.

7.6.12 Lithium -- Dissolve 5.324 g of lithium carbonate, Li_2CO_3, in a minimum volume of 1:1 HCl and dilute to 1 L with reagent water.

7.6.13 Magnesium -- Dissolve 1.000 g of magnesium metal in 20 mL 1:1 HNO_3 and dilute to 1 L with reagent water.

7.6.14 Manganese -- Dissolve 1.000 g of manganese metal in 10 mL of redistilled HNO_3 (conc.) and dilute to 1 L with reagent water.

7.6.15 Molybdenum -- Dissolve 1.840 g of ammonium molybdate, $(NH_4)_6Mo_7O_{24}$C$4H_2O$, and dilute to 1 L with reagent water.

7.6.16 Nickel -- Dissolve 1.000 g of nickel metal or 4.953 g of nickel nitrate, $Ni(NO_3)_2$C$6H_2O$, in 10 mL of HNO_3 (conc.) and dilute to 1 L with reagent water.

7.6.17 Osmium -- Procure a certified aqueous standard from a supplier and verify by comparison with a second standard. If necessary, standards can be made from osmium compounds. However, due to the toxicity of these compounds, this approach is not advised.

7.6.18 Potassium -- Dissolve 1.907 g of potassium chloride, KCl, dried at 110 EC, in reagent water and dilute to 1 L with reagent water.

7.6.19 Silver -- Dissolve 1.575 g of anhydrous silver nitrate, $AgNO_3$, in reagent water. Add 10 mL of HNO_3 (conc.) and dilute to 1 L with reagent water. Store in a dark-colored glass bottle in a refrigerator.

7.6.20 Sodium -- Dissolve 2.542 g of sodium chloride, NaCl, in reagent water, acidify with 10 mL of redistilled HNO_3 (conc.), and dilute to 1 L with reagent water.

7.6.21 Strontium -- Dissolve 2.415 g of strontium nitrate, $Sr(NO_3)_2$, in 10 mL of conc. HCl and 700 mL of reagent water. Dilute to 1 L with reagent water.

7.6.22 Thallium -- Dissolve 1.303 g of thallium nitrate, $TlNO_3$, in reagent water, acidify (to pH # 2) with 10 mL of conc. HNO_3, and dilute to 1 L with reagent water.

7.6.23 Tin -- Dissolve 1.000 g of tin metal in 100 mL conc. HCl and dilute to 1 L with reagent water.

7.6.24 Vanadium -- Dissolve 1.785 g of vanadium pentoxide, V_2O_5 , in 10 mL of conc. HNO_3 and dilute to 1 L with reagent water.

7.6.25 Zinc -- Dissolve 1.000 g of zinc metal in 10 mL of conc. HNO_3 and dilute to 1 L with reagent water.

7.7 Aluminum nitrate solution -- Dissolve 139 g of aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, in 150 mL reagent water and heat to effect solution. Allow to cool and make to 200 mL. Add 2 mL of this solution to a 100 mL volume of standards and samples.

7.8 Lanthanum chloride/potassium chloride solution -- Dissolve 11.73 g of lanthanum oxide, La_2O_3, in a minimum amount (approximately 50 mL) of conc. HCl. Add 1.91 g of potassium chloride, KCl. Allow solution to cool to room temperature and dilute to 100 mL with reagent water.

WARNING: REACTION IS VIOLENT!

Add acid slowly and in small portions to control the reaction rate upon mixing.

7.9 Blanks

Two types of blanks are required for the analysis of samples prepared by any method other than Method 3040. The calibration blank is used in establishing the analytical curve and the method blank is used to identify possible contamination resulting from either the reagents (acids) or the equipment used during sample processing including filtration.

7.9.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations.

7.9.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis (refer to Sec. 9.5).

7.10 The initial calibration verification (ICV) standard is prepared by the analyst (or a purchased second source reference material) by combining compatible elements from a standard source different from that of the calibration standard, and at concentration near the midpoint of the calibration curve (see Sec. 10.2.1 for use). This standard may also be purchased.

7.11 The continuing calibration verification (CCV) standard should be prepared in the same acid matrix using the same standards used for calibration, at a concentration near the mid-point of the calibration curve (see Sec. 10.2.2 for use).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

See the introductory material in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan

(QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to a 3000 series method (Method 3005, 3010, 3015, 3031, 3040, 3050, 3051, or 3052) for appropriate QC procedures to ensure the proper operation of the various sample preparation techniques.

9.3 Instrument detection limits (IDLs) are a useful tool to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified according to the guidance in Sec. 10.2.3.

IDLs in µg/L can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least every three months or at a project-specific designated frequency and kept with the instrument log book.

9.4 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency with each sample preparation (a 3000 series method) and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean matrix. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made.

9.5 For each batch of samples processed, at least one method blank must be carried throughout the entire sample preparation and analytical process, as described in Chapter One. A method blank is prepared by using a volume or weight of reagent water at the volume or weight specified in the preparation method, and then carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, prefiltering, digestion, dilution, filtering, and analysis. If the method blank does not contain target analytes at a level that interferes with the project-specific DQOs, then the method blank would be considered acceptable.

In the absence of project-specific DQOs, if the blank is less than 10% of the lower limit of quantitation check sample concentration, less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater, then the method blank is considered acceptable. If the method blank cannot be considered acceptable, the method blank should be re-run once, and if still unacceptable, then all samples after the last acceptable method blank should be reprepared and reanalyzed along with the other appropriate batch QC samples. These blanks will be useful in determining if samples are being contaminated. If the method blank exceeds the criteria, but the samples are all either below the reporting level or below the applicable action level or other DQOs, then the

sample data may be used despite the contamination of the method blank. Refer to Chapter One for the proper protocol when analyzing blanks.

9.6 Laboratory control sample (LCS)

For each batch of samples processed, at least one LCS must be carried throughout the entire sample preparation and analytical process as described in Chapter One. The laboratory control samples should be spiked with each analyte of interest at the project-specific action level or, when lacking project-specific action levels, at approximately mid-point of the linear dynamic range. Acceptance criteria should either be defined in the project-specifc planning documents or set at a laboratory derived limit developed through the use of historical analyses. In the absence of project-specific or historical data generated criteria, this limit should be set at ± 20% of the spiked value. Acceptance limits derived from historical data should be no wider that ± 20%. If the laboratory control sample is not acceptable, then the laboratory control sample should be re-run once and, if still unacceptable, all samples after the last acceptable laboratory control sample should be reprepared and reanalyzed.

Concurrent analyses of reference materials (SRMs) containing known amounts of analytes in the media of interest are recommended and may be used as an LCS. For solid SRMs, 80 - 120% accuracy may not be achievable and the manufacturer's established acceptance criterion should be used for soil SRMs.

9.7 Matrix spike, unspiked duplicate, or matrix spike duplicate (MS/Dup or MS/MSD)

Documenting the effect of the matrix, for a given preparation batch consisting of similar sample characteristics, should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch or as noted in the project-specific planning documents. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

For each batch of samples processed, at least one MS/Dup or MS/MSD sample set should be carried throughout the entire sample preparation and analytical process as described in Chapter One. MS/MSDs are intralaboratory split samples spiked with identical concentrations of each analyte of interest. The spiking occurs prior to sample preparation and analysis. An MS/Dup or MS/MSD is used to document the bias and precision of a method in a given sample matrix.

Refer to Chapter One for definitions of bias and precision, and for the proper data reduction protocols. MS/MSD samples should be spiked at the same level, and with the same spiking material, as the corresponding laboratory control sample that is at the project-specific action level or, when lacking project-specific action levels, at approximately mid-point of the linear dynamic range. Acceptance criteria should either be defined in the project-specifc planning documents or set at a laboratory-derived limit developed through the use of historical analyses per matrix type analyzed. In the absence of project-specific or historical data generated criteria, these limits should be set at ± 25% of the spiked value for accuracy and 20 relative percent difference (RPD) for precision. Acceptance limits derived from historical data should be no wider that ± 25% for accuracy and 20% for precision. Refer to Chapter One for additional guidance. If the bias and precision indicators are outside the laboratory control limits, if the percent recovery is less than 75% or greater than 125%, or if the relative percent

difference is greater than 20%, then the interference test discussed in Sec. 9.8 should be conducted.

9.7.1 The relative percent difference between spiked matrix duplicate or unspiked duplicate determinations is to be calculated as follows:

$$RPD = \frac{*D_1 - D_2*}{\left(\frac{*D_1 + D_2*}{2}\right)} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (spiked or unspiked duplicate).

9.7.2 The spiked sample or spiked duplicate sample recovery should be within ± 25% of the actual value, or within the documented historical acceptance limits for each matrix.

9.8 If less than acceptable accuracy and precision data are generated, the following additional quality control tests are recommended prior to reporting concentration data for the elements in this method. At a minimum these tests, outlined in Secs. 9.8.1 and 9.8.2, should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests will then serve to ensure that neither positive nor negative interferences are affecting the measurement of any of the elements or distorting the accuracy of the reported values. If matrix effects are confirmed, the laboratory should consult with the data user when feasible for possible corrective actions which may include the use of alternative or modified test procedures or possibly the method of standard additions so that the analysis is not impacted by the same interference.

9.8.1 Post digestion spike addition

The same sample from which the MS/MSD aliquots were prepared (asuming the MS/MSD recoveries are unacceptable) should also be spiked with a post digestion spike. Otherwise another sample from the same preparation should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. If this spike fails, then the dilution test (Sec. 9.8.2) should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

9.8.2 Dilution test

If the analyte concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, then a chemical or physical interference effect should be suspected. For both a failed post digestion spike or an unacceptable dilution test agreement result, the method of standard additions should be used as the primary means to quantitate all samples in the associated preparation batch.

9.9 Where the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard additions (MSA) is recommended (see Sec. 9.10 below). Other options including the use of different matrix modifiers, different furnace conditions, different preparatory methods or different analytical methods may also be attempted to properly characterize a sample. Sec. 9.8 provides tests to determine the potential of an interference and evaluates the need for using the MSA.

9.10 Method of standard additions -- The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique attempts to compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions may be appropriate for analysis of extracts, on analyses submitted as part of a delisting petition, whenever a new sample matrix is being analyzed and on every batch that fails the recovery test.

9.10.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of reagent water. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated:

$$C_x = \frac{S_B V_S C_S}{(S_A - S_B) V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

9.10.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the indigenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1. A linear regression program may be used to obtain the intercept concentration.

9.10.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

1. The apparent concentrations from the calibration curve must be linear (0.995 or greater) over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve.

2. The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3. The determination must be free of spectral interference and corrected for nonspecific background interference.

9.11 Ultra-trace analysis requires the use of clean chemistry preparation and analysis techniques. Several suggestions for minimizing analytical blank contamination are provided in Chapter Three.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration standards -- For those instruments which do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. Usually, this means the preparation of a blank and standards which produce an absorbance of 0.0 to 0.7. Calibration standards can prepared by diluting the stock metal solutions in the same acids and acid concentrtions as the samples.

10.1.1 Calibration standards can be prepared fresh each time a batch of samples is analyzed. If the ICV solution is prepared daily and the ICV is analyzed within the acceptance criteria, calibration standards do not need to be prepared daily and may be prepared and stored for as long as the calibration standard viability can be verified through the use of the ICV. If the ICV is outside of the acceptance criteria, the calibration standards must be prepared fresh and the instrument recalibrated. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range of the linear part of the curve.

10.1.2 The calibration standards should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing.

10.1.3 Beginning with the calibration blank and working toward the highest standard, aspirate the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure an average reading for each solution. Calibration curves are always required.

10.2 A calibration curve must be prepared each day with a minimum of a calibration blank and three standards. The curve must be linear and have a correlation coefficient of at least 0.995.

10.2.1 After initial calibration, the calibration curve must be verified by use of an initial calibration blank (ICB) and an initial calibration verification (ICV) standard. The ICV standard must be made from an independent (second source) material at or near mid-range. The acceptance criteria for the ICV standard must be ±10% of its true value and the ICB must not contain target analytes at or above the lowest limit of quantitation for the curve to be considered valid. If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are

analyzed. The analysis data for the ICV must be kept on file with the sample analysis data.

10.2.2 The calibration curve must also be verified at the end of each analysis batch and/or after every 10 samples by use of a continuing calibration blank (CCB) and a continuing calibration verification (CCV) standard. The CCV standard should be made from the same material as the initial calibration standards at or near midrange. The acceptance criteria for the CCV standard must be ±10% of its true value and the CCB must not contain target analytes at or above the lowest limit of quantitation for the curve to be considered valid. If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable CCV/CCB must be reanalyzed. The analysis data for the CCV/CCB must be kept on file with the sample analysis data.

10.2.3 The lower limits of quantitation should be established for all analytes for each type of matrix analyzed and for each preparation method used and for each instrument. These limits are considered the lowest reliable laboratory reporting concentrations and should be established from the lower limit of quantitation check sample and then confirmed using either the lowest calibration point or from a low-level calibration check standard.

10.2.3.1 Lower limit of quantitation check sample

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ± 30% of their true value. This check should be used to both establish and confirm the lowest quantitation limit.

10.2.3.2 The lower limits of quantitation determination using reagent water represents a best case situation and does not represent possible matrix effects of real-world samples. For the application of lower limits of quantitation on a project-specific basis with established data quality objectives, low-level matrix-specific spike studies may provide data users with a more reliable indication of the actual method sensitivity and minimum detection capabilities.

10.3 It is recommended that each standard should be analyzed (injected) twice and an average value determined. Replicate standard values should be within ±10% RPD.

10.4 If conducting trace analysis, it is recommended that the lowest calibration standard be set at the laboratory's lower limit of quantitation. The laboratory can use a reporting limit that is below the lower limit of quantitation but all values reported below the low standard should be reported as estimated values.

11.0 PROCEDURE

11.1 Preliminary treatment of aqueous and solid wastes is always necessary because of the complexity and variability of sample matrices. Solids, slurries, and suspended material must be subjected to a solubilization process before analysis. This process may vary because of the

metals to be determined and the nature of the sample being analyzed. Solubilization and digestion procedures are presented in Chapter Three. Samples which are to be analyzed for dissolved constituents need not be digested if they have been filtered and then acidified. Also see the note in Sec. 1.1.

11.2 All atomic absorption analyses must be performed using a suitable form of background correction. Refer to Chapter Three for a information regarding background correction.

11.3 Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for a particular instrument.

11.3.1 In general, after choosing the proper lamp for the analysis, allow the lamp to warm up for a minimum of 15 minutes.

11.3.2 During this period, align the instrument, position the monochromator at the correct wavelength, select the proper monochromator slit width, and adjust the current according to the manufacturer's recommendation.

11.3.3 Light the flame and regulate the flow of fuel and oxidant. Adjust the burner and nebulizer flow rate for maximum percent absorption and stability. Balance the photometer.

11.3.4 Run a series of standards of the element under analysis. Construct a calibration curve by plotting the concentrations of the standards against absorbances. Set the curve corrector of a direct reading instrument to read out the proper concentration.

11.3.5 Aspirate the samples and determine the concentrations either directly or from the calibration curve. Standards must be run each time a sample or series of samples is run.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 For determination of metal concentration, read the concentration from the calibration curve or directly from the read-out system of the instrument.

12.1.1 If dilution of the sample was required:

$$\mu g/L \text{ metal in sample} = \frac{A\ (C\%B)}{C}$$

where:

A = µg/L of metal in diluted aliquot from calibration curve.
B = Starting sample volume, mL.
C = Final volume of sample, mL.

12.1.2 For solid samples, report all concentrations in consistent units based on weight. Ensure that, if the dry weight was used for the analysis, percent solids are reported to the client.

$$\text{mg metal)kg sample} = \frac{A \times V}{W}$$

where:

A = mg/L of metal in processed sample from calibration curve.
V = Final volume of the processed sample, L.
W = Weight of sample, Kg.

12.1.3 Different integration times must not be used for samples and standards. Instead, the sample should be diluted and the same integration time should be used for both samples and standards. If dilution of the sample was required:

$$\mu\text{/L of metal sample} = \frac{Z\ (C\ \%\ B)}{C}$$

where:

Z = µg/L of metal read from calibration curve or read-out system.
B = Starting sample volume, mL.
C = Final volume of sample, mL.

12.2 Results need to be reported in units commensurate with their intended use and all dilutions need to be taken into account when computing final results.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 For relevant performance data, see the methods of Ref. 1.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention

techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes; U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory. ORD Publication Offices of Center for Environmental Research Information: Cincinnati, OH, 1983; EPA-600/4-79-020.

2. W. G. Rohrbough, et al., Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.

3. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables and figure referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE ATOMIC ABSORPTION LOWER LIMITS OF QUANTITATION AND SENSITIVITY FOR ANALYTES IN REAGENT WATER

	Direct Aspiration	
Metal	Lower Limit of Quantitation (mg/L)	Sensitivity (mg/L)
Aluminum	0.1	1
Antimony	0.2	0.5
Barium	0.1	0.4
Beryllium	0.005	0.025
Cadmium	0.005	0.025
Calcium	0.01	0.08
Chromium	0.05	0.25
Cobalt	0.05	0.2
Copper	0.02	0.1
Iron	0.03	0.12
Lead	0.1	0.5
Lithium	0.002	0.04
Magnesium	0.001	0.007
Manganese	0.01	0.05
Molybdenum	0.1	0.4
Nickel	0.04	0.15
Osmium	0.03	1
Potassium	0.01	0.04
Silver	0.01	0.06
Sodium	0.002	0.015
Strontium	0.03	0.15
Thallium	0.1	0.5
Tin	0.8	4
Vanadium	0.2	0.8
Zinc	0.005	0.02

These data are provided for guidance purposes only.

TABLE 2

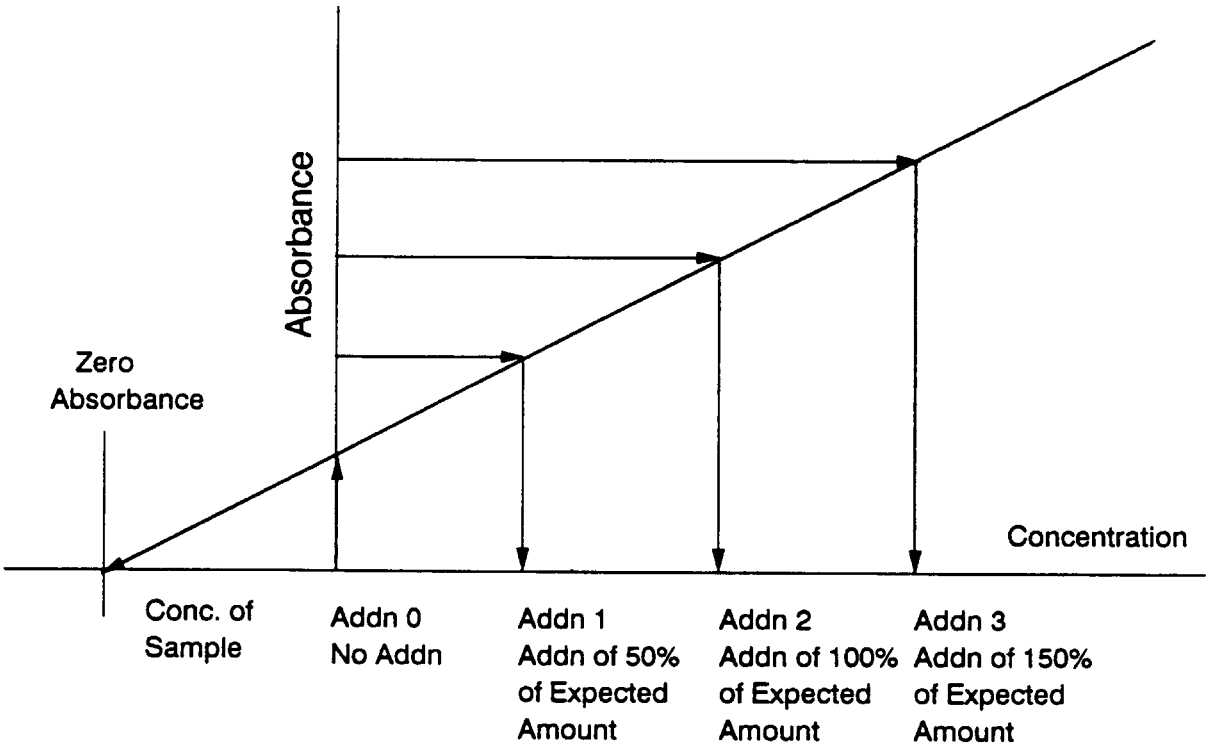
INSTRUMENT PARAMETERS (Ref. 1)

ELEMENT	WAVELENGTH (nm)	FUEL	OXIDANT	TYPE OF FLAME
Al	324.7	acetylene	nitrous oxide	fuel rich
Sb	217.6, 231.1	acetylene	air	fuel lean
Ba	553.6	acetylene	nitrous oxide	fuel rich
Be	234.9	acetylene	nitrous oxide	fuel rich
Cd	228.8	acetylene	air	fuel lean
Ca	422.7	acetylene	nitrous oxide	stoichiometric
Cr	357.9	acetylene	nitrous oxide	fuel rich
Co	240.7	acetylene	air	fuel lean
Cu	324.7	acetylene	air	fuel lean
Fe	248.3, 248.8, 271.8, 302.1, 252.7	acetylene	air	fuel lean
Pb	283.3, 217.0	acetylene	air	fuel lean
Li	670.8	acetylene	air	fuel lean
Mg	285.2	acetylene	air	fuel lean
Mn	279.5, 403.1	acetylene	air	fuel lean to stoichiometric
Mo	313.3	acetylene	nitrous oxide	fuel rich
Ni	232.0, 352.4	acetylene	air	fuel lean
Os	290.0	acetylene	nitrous oxide	fuel rich
K	766.5	acetylene	air	fuel lean
Ag	328.1	acetylene	air	fuel lean
Na	589.6	acetylene	air	fuel lean
Sr	460.7	acetylene	air	fuel lean
Tl	276.8	acetylene	air	fuel lean
Sn	286.3	acetylene	nitrous oxide	fuel rich
V	318.4	acetylene	nitrous oxide	fuel rich
Zn	213.9	acetylene	air	fuel lean

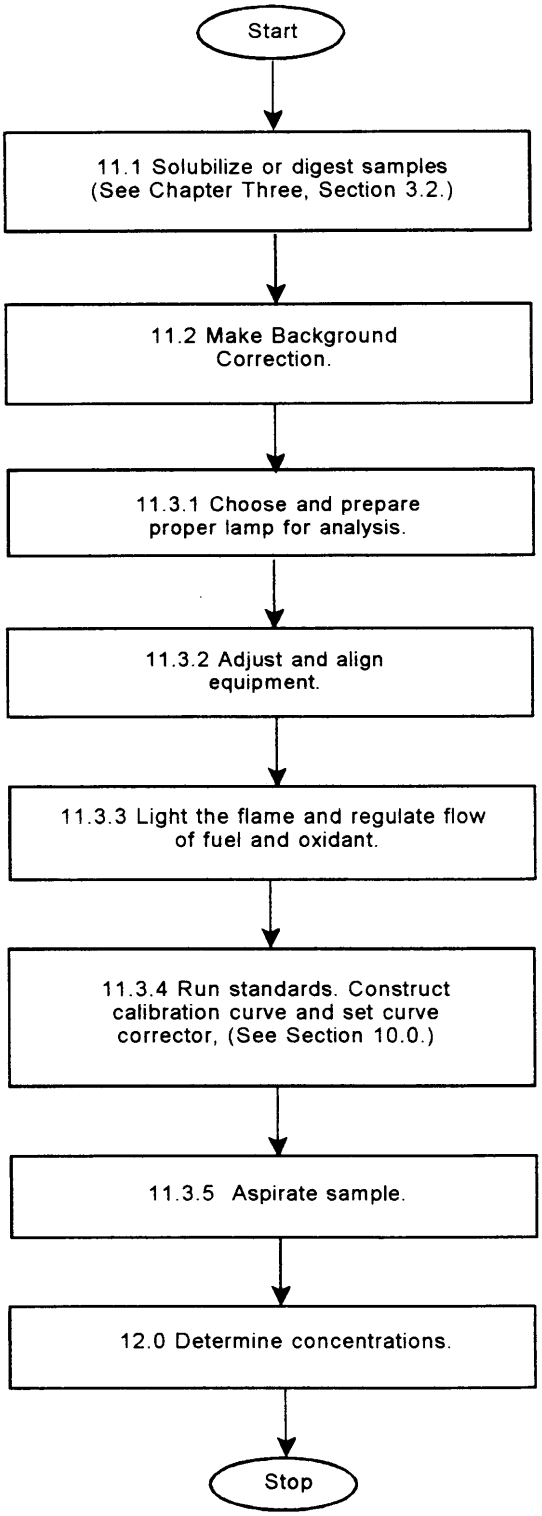
Note: If more than one wavelength is listed, the primary line is underlined.

FIGURE 1

STANDARD ADDITION PLOT



FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY



ASBESTOS and OTHER FIBERS by PCM 7400

FORMULA: Various MW: Various CAS: see Synonyms RTECS: Various

METHOD: 7400, Issue 2 **EVALUATION: FULL** **Issue 1: Rev. 3 on 15 May 1989**
Issue 2: 15 August 1994

OSHA: 0.1 asbestos fiber (> 5 µm long)/cc; 1 f/cc, 30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 µm long), 400 L; carcinogen
ACGIH: 0.2 f/cc crocidolite; 0.5 f/cc amosite; 2 f/cc chrysotile and other asbestos; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4]; refractory ceramic fibers [142844-00-6]; fibrous glass

SAMPLING

SAMPLER: FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm; conductive cowl on cassette)

FLOW RATE*: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)

*Adjust to give 100 to 1300 fiber/mm^2

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION ($\hat{S}_{rT}$): 0.115 to 0.13 [1]

ACCURACY: see EVALUATION OF METHOD

MEASUREMENT

TECHNIQUE: LIGHT MICROSCOPY, PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone - collapse/triacetin - immersion method [2]

COUNTING RULES: described in previous version of this method as "A" rules [1,3]

EQUIPMENT:
1. positive phase-contrast microscope
2. Walton-Beckett graticule (100-µm field of view) Type G-22
3. phase-shift test slide (HSE/NPL)

CALIBRATION: HSE/NPL test slide

RANGE: 100 to 1300 fibers/mm^2 filter area

ESTIMATED LOD: 7 fibers/mm^2 filter area

PRECISION ($\bar{S}_r$): 0.10 to 0.12 [1]; see EVALUATION OF METHOD

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fibers < ca. 0.25 µm diameter will not be detected by this method [4]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

INTERFERENCES: If the method is used to detect a specific type of fiber, any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This revision replaces Method 7400, Revision #3 (dated 5/15/89).

REAGENTS:

1. Acetone,* reagent grade.
2. Triacetin (glycerol triacetate), reagent grade.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically conductive extension cowl and cellulose ester filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is ≥ 5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.
   NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.
   NOTE 3: Use 0.8-µm pore size filters for personal sampling. The 0.45-µm filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.
   NOTE 4: Other cassettes have been proposed that exhibit improved uniformity of fiber deposit on the filter surface, e.g., bellmouthed sampler (Envirometrics, Charleston, SC). These may be used if shown to give measured concentrations equivalent to sampler indicated above for the application.
2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow-rate requirements (see step 4 for flow rate), with flexible connecting tubing.
3. Wire, multi-stranded, 22-gauge; 1″ hose clamp to attach wire to cassette.
4. Tape, shrink- or adhesive-.
5. Slides, glass, frosted-end, pre-cleaned, 25- × 75-mm.
6. Cover slips, 22- × 22-mm, No. 1½, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

EQUIPMENT (continued):

10. Acetone flash vaporization system for clearing filters on glass slides (see ref. [5] for specifications or see manufacturer's instructions for equivalent devices).
11. Micropipets or syringes, 5-µL and 100- to 500-µL.
12. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10× eyepiece, and 40 to 45× phase objective (total magnification ca. 400×); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100-µm diameter circular field (area = 0.00785 mm^2) at the specimen plane (Type G-22). Available from Optometrics USA, P.O. Box 699, Ayer, MA 01432 [phone (508)-772-1700], and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
    NOTE: The graticule is custom-made for each microscope. (see APPENDIX A for the custom-ordering procedure).
14. HSE/NPL phase contrast test slide, Mark II. Available from Optometrics USA (address above).
15. Telescope, ocular phase-ring centering.
16. Stage micrometer (0.01-mm divisions).

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.
    NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E, of 100 to 1300 fibers/mm^2 (3.85×10^4 to 5×10^5 fibers per 25-mm filter with effective

collection area A_c = 385 mm^2) for optimum accuracy. These variables are related to the action level (one-half the current standard), *L* (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \times E}{Q \times L \times 10^3}.$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [7]. Relatively large diameter fibers (>3 µm) may exhibit significant aspiration loss and inlet deposition. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If ≥50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min [3].

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤ 1.46. This method collapses the filter for easier focusing and produces permanent (1–10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400—revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7–9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9–11].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [2].
   NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
   a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.
      NOTE: Static electricity will usually keep the wedge on the slide.
   b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250 µL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the

acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 s for the filter to clear, remove pipet and slide from their ports.

CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce explosive acetone vapor concentrations.

c. Using the 5-µL micropipet, immediately place 3.0 to 3.5 µL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.

NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.

e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.

NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturer's instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.

    a. Each time a sample is examined, do the following:

        (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.

        (2) Focus on the particulate material to be examined.

        (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

    b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:

        (1) Center the HSE/NPL phase-contrast test slide under the phase objective.

        (2) Bring the blocks of grooved lines into focus in the graticule area.

        NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting, the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.

        (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Document the laboratory's precision for each counter for replicate fiber counts.

    a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer should maintain custody of the reference slides and should supply each counter with a minimum of one reference

slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter precision. Obtain separate values of relative standard deviation (S_r) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.

NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [9].

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm^2) exceeds $2.77XS_r'$ where X = average of the square roots of the two fiber counts (in fiber/mm^2) and $S_r' = S_r / 2$ where S_r is the intracounter relative standard deviation for the appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [13].

NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10- to 20-minute breaks from the microscope every one or two hours to limit fatigue [14]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.
15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program for asbestos and routinely exchange field samples with other laboratories to compare performance of counters.

MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.
17. Adjust the microscope (Step 10).

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm) [4].

18. Counting rules: (same as P&CAM 239 rules [1,10,11]: see examples in APPENDIX B).

a. Count any fiber longer than 5 µm which lies entirely within the graticule area.
   (1) Count only fibers longer than 5 µm. Measure length of curved fibers along the curve.
   (2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.

b. For fibers which cross the boundary of the graticule field:
   (1) Count as ½ fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.

(2) Do not count any fiber which crosses the graticule boundary more than once.
(3) Reject and do not count all other fibers.

c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.

19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 s per field is appropriate for accurate counting.

NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 µm), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].

NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.

NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].

NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/mm^2) is defined as the average count (fibers/field) divided by the field (graticule) area (mm^2/field).

CALCULATIONS AND REPORTING OF RESULTS

20. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the average fiber count per graticule field, F / n_f, minus the mean field blank count per graticule field, B / n_b, by the graticule field area, A_f (approx. 0.00785 mm^2):

$$E = \frac{(F/n_f - B/n_b)}{A_f}, \text{ fibers/mm}^2.$$

NOTE: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased." Other fiber counts outside the 100–1300 fiber/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm^2 for a 25-mm filter):

$$C = \frac{EA_c}{V \times 10^3}.$$

NOTE: Periodically check and adjust the value of A_c, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.
    NOTE: Precision depends on the total number of fibers counted [1,16]. Relative standard deviation is documented in references [1,15–17] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Figure 1).

EVALUATION OF METHOD:

Method Revisions:

This method is a revision of P&CAM 239 [10]. A summary of the revisions is as follows:

1. Sampling:
   The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].
2. Sample preparation technique:
   The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.
3. Measurement:
   a. The Walton-Beckett graticule standardizes the area observed [14,18,19].
   b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].
   c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm^2 filter area (a total of 78.5 fibers counted in 100 fields, each with field area = 0.00785 mm^2.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S_r in the range of 0.10 to 0.17 [21–23].

Interlaboratory Comparability:

An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [9]. The relative standard deviations (S_r) varied with sample type and laboratory. The ranges were:

Rules	Intralaboratory S_r	Interlaboratory S_r	Overall S_r
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)†	0.11 to 0.29	0.20 to 0.35	0.25

*Under AIA rules, only fibers having a diameter less than 3 µm are counted and fibers attached to particles larger than 3 µm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.
†See Appendix C.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S_r in the range 0.17 to 0.25 and an interlaboratory S_r of 0.45 [21]. This agrees well with other recent studies [9,14,16].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S_r. (NOTE: The following discussion does not include bias estimates and should not be taken to indicate that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an S_r that depends on the number, N, of fibers counted:

$$S_r = 1/N^{1/2}.$$

Thus S_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S_r found in a number of studies is greater than these theoretical numbers [17,19–21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [15] found this subjective component of intralaboratory S_r to be approximately 0.2 and estimated the overall S_r by the term:

$$\frac{[N + (0.2 \times N)^2]^{1/2}}{N}.$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 S_r and −1.5 S_r. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [14]. These results gave a subjective interlaboratory component of S_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [21]. This value falls slightly above the range of S_r (0.25 to 0.42 for 1984–85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [17].

A number of factors influence S_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is hoped that the laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the S_r.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [16].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 counting field area). If this same sample were counted by

a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the S_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory S_r is smaller, then it is more correct to use that smaller S_r. However, the estimated S_r of 0.45 is to be used in the absence of such information. Note also that it has been found that S_r can be higher for certain types of samples, such as asbestos cement [9].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., $0.16 + 2.13 \times 0.16 = 0.5$.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and −49% as the upper and lower confidence values of the mean for a 100-fiber count.

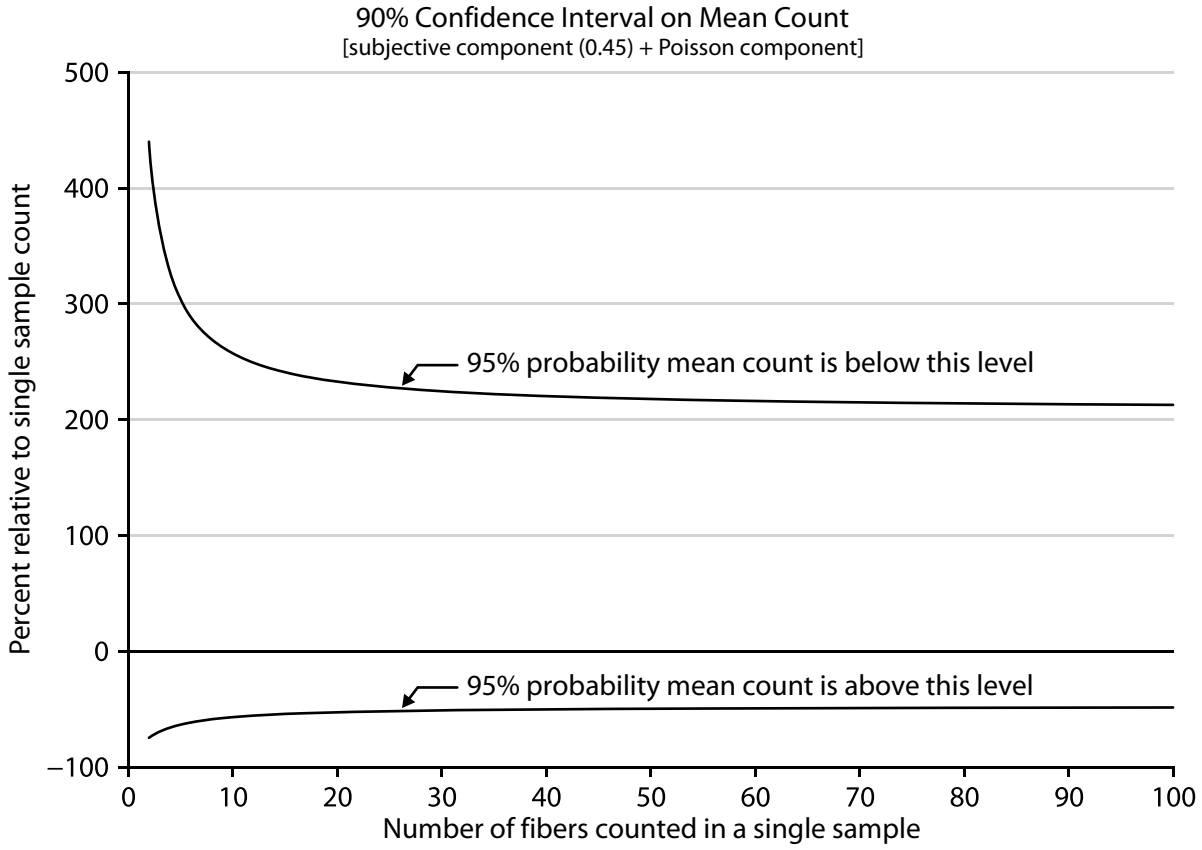


Figure 1. Interlaboratory precision of fiber counts.

The curves in Figure 1 are defined by the following equations:

$$U_{CL} = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25S_r^2)X^2]^{½}}{2(1 - 2.25S_r^2)} \text{ and}$$

$$L_{CL} = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4S_r^2)X^2]^{½}}{2(1 - 4S_r^2)},$$

where S_r = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory S_r when approximately 100 fibers are counted,
X = total fibers counted on sample,
L_{CL} = lower 95% confidence limit, and
U_{CL} = upper 95% confidence limit.

Note that the range between these two limits represents 90% of the total range.

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APPENDIX A. CALIBRATION OF THE WALTON-BECKETT GRATICULE

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (*D*) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45× phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_o} \times D.$$

   Example: If L_o = 112 µm, L_a = 4.5 mm, and D = 100 µm, then d_c = 4.02 mm.
8. Check the field diameter, *D* (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 mm^2 to 0.00817 mm^2).

APPENDIX B. COMPARISON OF COUNTING RULES

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. The rules will be discussed as they apply to the labeled objects in the figure.

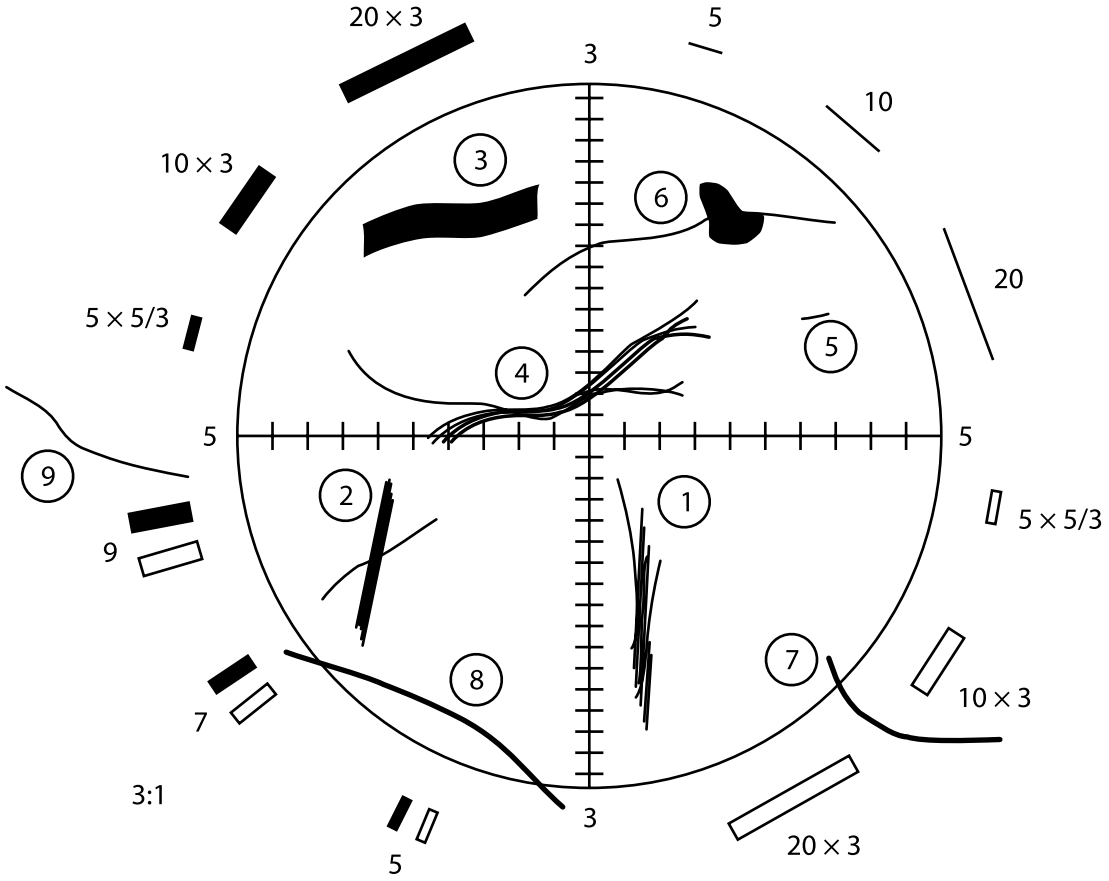

Figure 2. Walton-Beckett graticule with fibers.

These rules are sometimes referred to as the "A" rules:

Object	Count	Discussion
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle, the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fibers	If fibers meeting the length and aspect ratio criteria (length >5 µm and length-to-width ratio > 3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 µm), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is ≤ 5 µm long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	½ fiber	A fiber which crosses into the graticule area one time is counted as ½ fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.

APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules given below (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [24], and the NIOSH fibrous glass criteria document method [25]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" Counting Rules

1. Count only *ends* of fibers. Each fiber must be longer than 5 µm and less than 3 µm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 µm in diameter.

5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION

	Fiber density on filter*		Fiber concentration in air, f/cc	
	Fibers per 100 fields	Fibers/mm^2	400-L air sample	1000-L air sample
	200	255	0.25	0.10
	100	127	0.125	0.05
LOQ	80.0	102	0.10	0.04
	50	64	0.0625	0.025
	25	32	0.03	0.0125
	20	25	0.025	0.010
	10	12.7	0.0125	0.005
	8	10.2	0.010	0.004
LOD	5.5	7	0.00675	0.0027

*Assumes 385 mm^2 effective filter collection area, and field area = 0.00785 mm^2, for relatively "clean" (little particulate aside from fibers) filters.

United States
Environmental Protection
Agency

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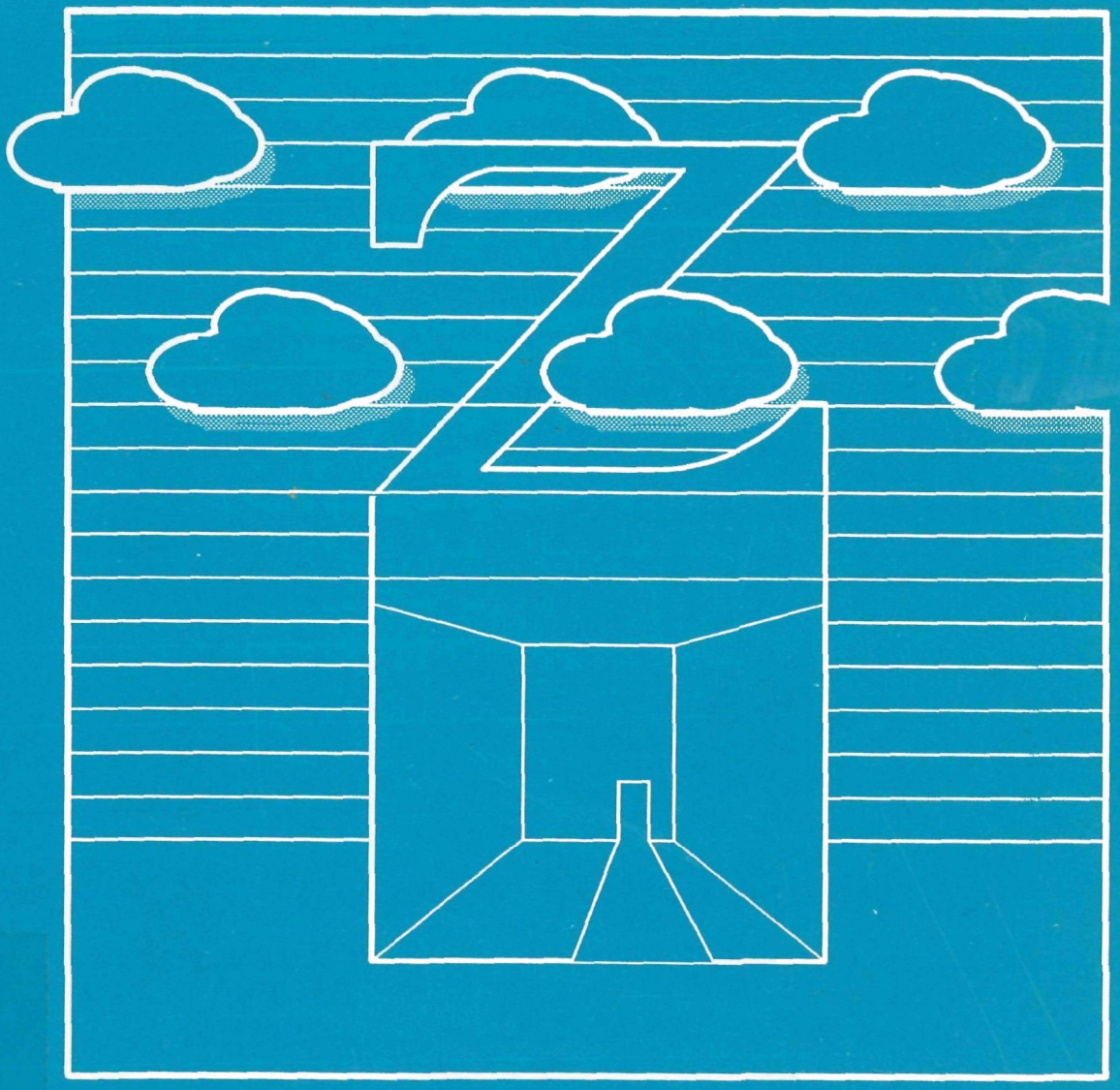
Toxic Substances

EPA

Guidelines for Conducting the AHERA TEM Clearance Test to Determine Completion of an Asbestos Abatement Project



EPA 560/5-89-001
May 1989

FINAL REPORT

GUIDELINES FOR CONDUCTING THE AHERA TEM CLEARANCE TEST TO DETERMINE COMPLETION OF AN ASBESTOS ABATEMENT PROJECT

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SUMMARY

Asbestos abatement carried out in schools is subject to regulations under the Asbestos Hazard Emergency Response Act of 1986 (AHERA). The AHERA rule (40 CFR Part 763) includes procedures for determining when an asbestos abatement site is sufficiently clean for the containment barriers to be removed. After the abatement site has been subject to a thorough visual inspection, air samples are collected. In most cases, the samples must be analyzed by transmission electron microscopy (TEM).

This document provides guidance for conducting the TEM clearance test with emphasis on interpretation of the results. The three components of the test -- the Initial Screening Test, the Blank Contamination Test, and the Z-test -- are described and illustrated with numerical examples.

1. INTRODUCTION

As required under the Asbestos Hazard Emergency Response Act of 1986 (AHERA), EPA has promulgated a rule regarding inspections, abatement, and management of asbestos-containing material in schools (40 CFR Part 763). The rule includes procedures for determining when an asbestos abatement site is sufficiently clean for the containment barriers to be removed. After the abatement site has been subject to a thorough visual inspection, air samples are collected. In most cases, the samples must be analyzed by transmission electron microscopy (TEM).

This document provides guidance for conducting the TEM clearance test with emphasis on interpretation of the results. The guidance is intended for abatement project monitors, industrial hygienists, asbestos consultants and others who are responsible for interpreting air sampling data. Details on sampling and analytical protocols are not included. Readers should refer to the AHERA rule for details on these topics and to determine the circumstances under which alternative clearance procedures may be used. Readers are also urged to consult the latest version of the AHERA rule for revisions that may have been made since the publication of this document.

2. BACKGROUND

The AHERA TEM clearance test is based on a comparison of airborne asbestos levels inside the work site with those outside the work site. This approach was adopted since an abatement contractor could not be expected to achieve airborne asbestos concentrations inside the work site lower than those of the incoming air. "Outside" does not necessarily mean outdoors. When air intake to the work site is from other parts of the building rather than from outdoors, indoor samples may be collected as the basis for comparison. However, outdoor samples are recommended in most circumstances because they are unlikely to be affected by poor work practices that might contaminate areas outside the work site.

Airborne asbestos measurements are subject to variation due to variability of the distribution of asbestos in the air and variability introduced by the sampling and analytical procedures. The clearance test must account for both sources of variation in order to achieve a high probability of correct clearance decisions. Therefore the test requires a minimum of five samples collected inside the work site and five samples collected outside the work site, and uses a statistical test, the Z-test, to determine if inside levels are statistically higher than outside levels. The Z-test differs slightly from the t-test recommended in previous EPA guidance documents (USEPA 1985a, 1985b) by fixing the amount of variability associated with the measurements rather

than estimating it from the data. Consequently, the Z-test is simpler to calculate.

The Z-test is preceded by two preliminary tests, an initial screening test and a blank contamination test. The initial screening test is intended to reduce the cost of analysis when the concentration of asbestos structures on sample filters collected inside the work site is comparable to the concentration typically observed on blank filters (filters through which no air has been drawn). Asbestos structures on blank filters may be the result of contamination during filter manufacture, for example. If the concentration on filters used to sample inside the work site is comparable to typical blank contamination levels, the work site passes the clearance test without requiring analysis of the outside samples. The initial screening test is based on the concentration per area of filter, not concentration per volume of air, because it considers asbestos structures from sources other than the sampled air. Filter concentrations less than or equal to 70 s/mm^2 are considered indistinguishable from blank contamination levels. A measurement within this range suggests that few, if any, asbestos structures have been contributed by the sampled air.

If the average filter concentration for the inside samples is greater than 70 s/mm^2, a minimum of three blanks are checked to insure that the particular filter lot used was not contaminated beyond typical levels. Like the initial screening test, the blank contamination test is based on structures per area of filter because it involves asbestos structures from sources other than the sampled air.

Together, the three tests (the initial screening test, the blank contamination test, and the Z-test) make up the AHERA TEM clearance test.

3. SAMPLING

Sampling must be performed by a qualified individual who is completely independent of the abatement contractor. Although the AHERA rule does not impose specific requirements, it is suggested that the person should be professionally licensed and/or have received relevant training.

Since circumstances vary among abatement sites, professional judgment is needed to ensure that the samples accurately represent airborne asbestos levels inside and outside the work site. This section provides guidance on the number and location of samples. Refer to Section III B of the AHERA rule and USEPA (1985b) for further details on the sampling protocol.

3.1 Number of Samples

The clearance test requires a minimum of five samples inside the work site, five samples outside the work site, and three blanks. Additional samples will improve the performance of the clearance test (i.e., increase the probability of making a correct clearance decision). Additional samples are recommended for large or complex work sites that consist of several rooms or distinct areas. Provided the minimum requirements are met, it is not necessary to have an equal number of samples inside and outside the work site.

For example, if the work site consists of eight adjacent rooms, the local educational authority (LEA) may choose to collect a sample in each of the eight rooms and five samples outside the work site. The additional three samples inside the work site improve the performance of the clearance test and also provide extra assurance that each room has been adequately cleaned. If the work site fails the clearance test, the additional samples may help isolate the problem to one or more rooms and indicate where cleaning efforts should be concentrated. Note, however, that the entire work site must be resampled, not just the room or area in which high concentrations were measured during the original test.

The desirability of exceeding the minimum requirements, especially for large or complex abatement projects, should be considered when the abatement project is being planned. Specifications for sampling and interpretation of results should be clearly stated in the contract between the LEA and the abatement contractor.

The clearance test must be based on all samples analyzed. It is not permissible to analyze more than the minimum number of samples then choose the "best" results for inclusion in the calculations. It is acceptable, however, to analyze a subset of the samples collected, provided the subset of samples is selected at random without knowledge of the concentration of asbestos structures on the filters.

For example, a school district may set up 6 samplers in the work site to insure against the possibility of a lost or damaged sample. At the laboratory, 5 samples are selected at random from the 6 provided. If a sample is unsuitable for analysis, or is accidentally destroyed, the remaining sample is analyzed and the minimum of 5 samples is still achieved. If all samples are suitable for analysis and the school district decides to analyze all 6, then the results of the 6 analyses must be included in the clearance test calculations.

The AHERA rule does not explicitly address the case of samples that are so overloaded with material (asbestos or otherwise) that

they cannot be analyzed by the laboratory. In most cases, an overloaded sample is indicative of a dirty work site that needs recleaning.

3.2 Location of Samples

Inside the Work Site

Samplers should be located to provide a representative sample of air within the work site. If the work site is a single room, locate the samplers throughout the area. If the work site consists of several rooms, place a sampler in each room, or in a representative subset of rooms. Random numbers may be used to select a representative subset. Each sampler should be placed so that it is subject to normal air circulation. Avoid room corners, obstructed locations, and sites near windows, doors, or vents. Ensure that the sampler is not sampling exhaust fumes from the pump. Oil droplets on the filter can adversely affect the analysis.

Outside the Work Site

Samples should be representative of air entering the work site. Place samplers so that they do not sample any air that may escape from the work site. Recommended distances are at least 50 feet from the entrance to the work site and at least 25 feet from the containment barriers. If any potential sources of fiber release (e.g., tears in the containment barrier, spillage of asbestos waste) are identified while the abatement work is in progress, these locations should be avoided when selecting the outside sampling locations.

Taking makeup air from outside the building, or passing makeup air through a HEPA filtration system before it enters the work site eliminates the need to use indoor samples taken outside the work site as the basis for comparison. The contractor no longer has to contend with other sources of airborne asbestos and should be able to clean the work site to levels comparable with outdoors. Sampling outdoors is recommended because it reduces the likelihood of the contractor improperly passing the clearance test due to poor work practices that increase airborne asbestos levels outside the work site.

Outdoor samplers should be placed at least 3 feet apart, preferably at ground level rather than on a roof. Protect the samplers from adverse weather and avoid obstructions that may influence wind patterns. If a roof-top site is necessary, avoid locations near vents or other structures.

3.3 Sample Volume

The AHERA rule specifies a minimum air volume of 560 liters for 25 mm diameter filters and 1,250 liters for 37 mm diameter filters. Recommended ranges are 1,200 liters to 1,800 liters and 2,800 liters to 4,000 liters respectively. Note that the initial screening test cannot be used when the volume of air is below the recommended range. Lower volumes require TEM analysis of a larger area of filter in order to achieve the required analytical sensitivity. This is discussed further in the next section.

4. LABORATORY ANALYSIS

Air samples are analyzed by TEM according to the protocol specified in the rule. The rule allows flexibility in the choice of volume of air sampled and the amount of filter examined, provided the analytical sensitivity (the concentration represented by a single fiber) is no greater than 0.005 structures per cubic centimeter of air (s/cc). To maintain the analytical sensitivity at 0.005 or less, a small volume of air must be compensated for by examining a larger area of the filter. Conversely, examination of a small area of the filter must be compensated for by collecting a larger volume of air.

A laboratory accreditation program for TEM analysis is being established by the National Institute of Standards and Technology (NIST, formerly the National Bureau of Standards). Only accredited laboratories will be permitted to analyze samples for compliance with AHERA. Until the accreditation program is operational, LEAs must use laboratories which follow the protocol stated in Appendix A of the rule.

5. INTERPRETATION OF RESULTS

Figure 1 shows each step in the clearance test. The initial screening test is performed first. If the inside samples pass the initial screening test, the work site passes and no further analysis is required. If the inside samples do not pass the initial screening test, the blanks are examined. If the blanks fail the blank contamination test, the source of contamination must be identified and corrected, and new samples collected. If the blanks pass the blank contamination test, the Z-test is performed to determine whether the work site passes or fails the clearance test.

The following sections describe how to perform the initial screening test, the blank contamination test, and the Z-test. The numerical examples are hypothetical and represent only a subset of the possible outcomes and decisions.

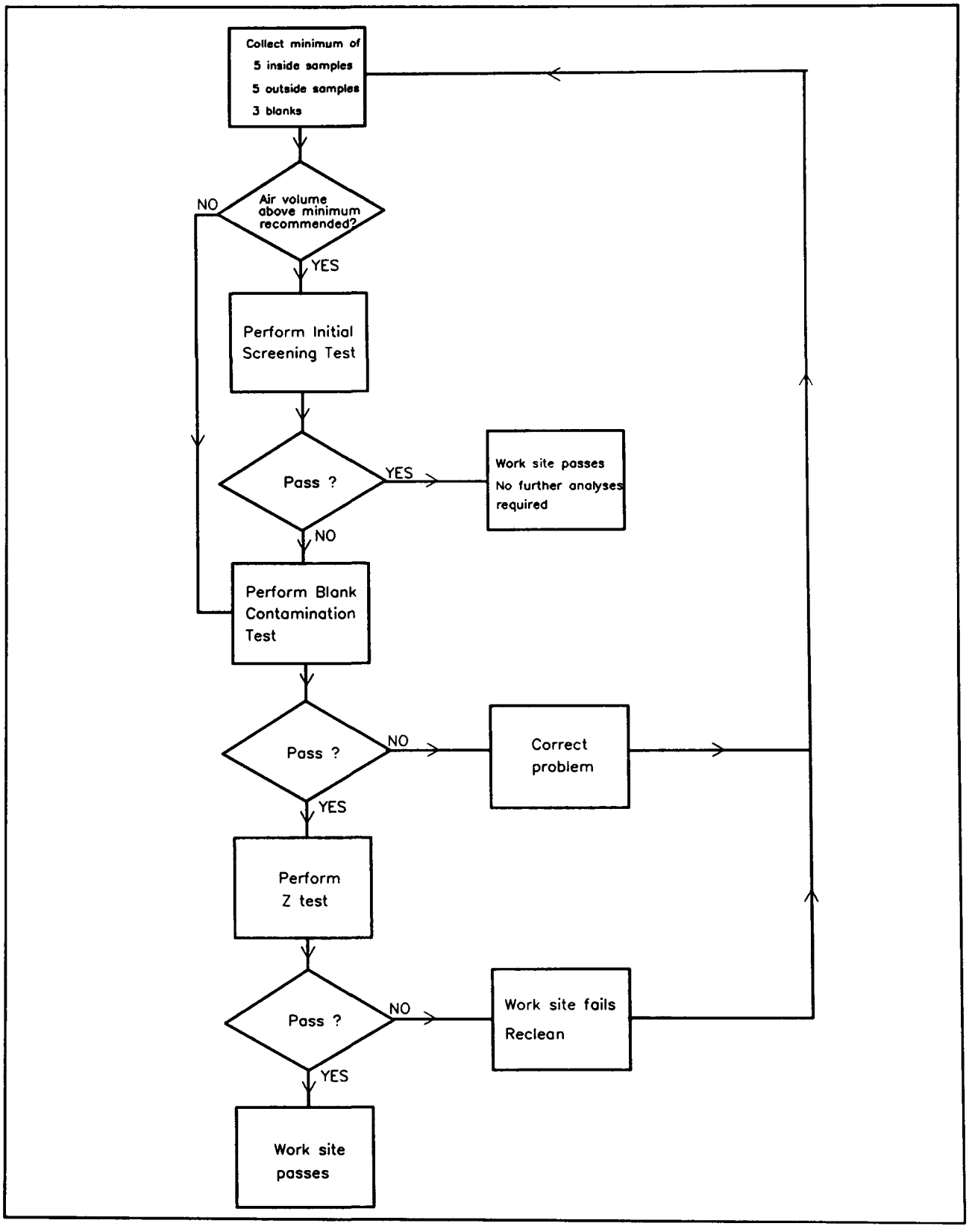


Figure 1. The AHERA TEM clearance test.

5.1 Initial Screening Test

The laboratory should report the asbestos structure concentration per square millimeter of filter (s/mm^2) for each inside sample. Calculate the arithmetic mean (average) of all the inside samples. If the arithmetic mean is less than or equal to 70 s/mm^2, the inside samples pass the initial screening test and the work site passes the clearance test.

In the three examples below it is assumed that the minimum volume requirements for the initial screening test have been met. When this is not the case, the analysis proceeds directly to the blank contamination test.

Example 1. In this example the LEA decided to collect and analyze 8 inside samples. The results are shown in Figure 2. The arithmetic mean is less than 70 s/mm^2 and therefore the samples pass the initial screening test. Note that zeros are included in the calculation of the arithmetic mean. The work site passes the clearance test and no further analyses are required.

Example 2. The results of analysis of 5 inside samples are shown in Figure 3. The arithmetic mean is greater than 70 s/mm^2 and therefore the samples do not pass the initial screening test. The LEA gives the abatement contractor the option of recleaning immediately or carrying on with the blank contamination test and Z-test. The abatement contractor is convinced that the work site has been thoroughly cleaned and opts for continuing with the test.

Example 3. Five inside samples give the concentrations shown in Figure 4. The arithmetic mean is less than 70 s/mm^2 and therefore the samples pass the initial screening test. The result of the fourth analysis, however, appears to be unusually high compared with the other values. The LEA decides as a precautionary measure to ask the abatement contractor to reclean the room where the fourth sample was collected. Although this eventuality was not covered in the original contract and no action is required under the AHERA rule, the LEA decides that the additional expense is justified. (Note that there is a great deal of uncertainty associated with a single estimated concentration. That is why the clearance test requires multiple samples. Therefore caution should be used in interpreting individual values.)

INITIAL SCREENING TEST

Work Sheet

Inside Samples

inside samples, n_I = 8

Sample 1	35.80	s/mm^2
Sample 2	0.00	s/mm^2
Sample 3	17.90	s/mm^2
Sample 4	0.00	s/mm^2
Sample 5	17.90	s/mm^2
Sample 6	0.00	s/mm^2
Sample 7	17.60	s/mm^2
Sample 8	35.90	s/mm^2
.	.	
.	.	
.	.	
Sample n_I	_____	s/mm^2
Total	125.10	

Mean = Total/n_I = 125.10 / 8
= 15.64

Result

Mean ≤ 70	Mean > 70
PASS X	FAIL

Figure 2. The Initial Screening Test -- Example 1. In this example the work site passes and no further analyses are required.

INITIAL SCREENING TEST

Work Sheet

Inside Samples

inside samples, n_I, = 5

Sample 1 71.60 s/mm^2

Sample 2 107.40 s/mm^2

Sample 3 89.50 s/mm^2

Sample 4 53.70 s/mm^2

Sample 5 89.50 s/mm^2

Sample 6 _____ s/mm^2

Sample 7 _____ s/mm^2

Sample 8 _____ s/mm^2

Sample n_I _____ s/mm^2

Total 411.70

Mean = Total/n_I = 411.70 / 5

= 82.34

Result

Mean ≤ 70	Mean > 70
PASS	FAIL X

Figure 3. The Initial Screening Test -- Example 2. In this example the work site fails. The abatement contractor, convinced that the site is clean, opts to continue with the blank contamination and Z-tests.

INITIAL SCREENING TEST

Work Sheet

Inside Samples

inside samples, n_I = 5

Sample 1 35.80 s/mm^2

Sample 2 53.70 s/mm^2

Sample 3 0.00 s/mm^2

Sample 4 232.70 s/mm^2

Sample 5 17.90 s/mm^2

Sample 6 _____ s/mm^2

Sample 7 _____ s/mm^2

Sample 8 _____ s/mm^2

. .

. .

. .

Sample n_I _____ s/mm^2

Total 340.10

Mean = Total/n_I = 340.10 / 5

= 68.02

Result

Mean ≤ 70	Mean > 70
PASS X	FAIL

Figure 4. The Initial Screening Test -- Example 3. In this example the work site passes and no further analyses are required under the AHERA rule. However, as a precautionary measure, the LEA decides to reclean the room where sample 4 was collected.

5.2 Blank Contamination Test

The results of the analyses of the three blanks are reported as structures per square millimeter of filter. Calculate the arithmetic mean of the three values. If the arithmetic mean is less than or equal to 70 s/mm^2, the blanks pass the blank contamination test and one may proceed to the Z-test. If the arithmetic mean is greater than 70 s/mm^2, it is likely that the blanks have been contaminated by asbestos structures from a source other than the sampled air. The validity of the inside and outside samples is questionable. They must be discarded and new samples collected.

Before collecting new samples, attempt to identify and eliminate the source of contamination. If asbestos structures are present on the sealed blank, the filters may have been contaminated during manufacture. Select new filters from lots that have been prescreened. (Laboratories are required to screen filters from the lots they use for their internal quality control.) If the majority of asbestos structures are on the field blanks, contamination may have occurred during the field sampling or laboratory analysis. Check field procedures and ask the laboratory for the results of blank analyses that they are required to perform as a part of their laboratory quality control program. During the resampling ensure that the sampling, handling, and analysis protocols are strictly followed to avoid a repeat of the contamination problem.

Example 4. Analysis of three blanks gives the results shown in Figure 5. The arithmetic mean of the three concentrations is less than 70 s/mm^2 and the blanks pass the contamination test. The laboratory proceeds with the analysis of the outside samples.

Example 5. The three blanks in Figure 6 do not pass the blank contamination test. The cause of the failure appears to be the field blank collected inside the work site. The sampling technician recalls that the sampling cassettes were accidentally stored in the work site while the abatement was in progress. A new supply of cassettes is obtained and the entire sampling and analysis procedure is repeated.

BLANK CONTAMINATION TEST

Work Sheet

Blanks

blanks, n_B, = 3

Blank 1 0.00 s/mm^2

Blank 2 17.60 s/mm^2

Blank 3 0.00 s/mm^2

Blank 4 _____ s/mm^2

Blank 5 _____ s/mm^2

Blank 6 _____ s/mm^2

Blank 7 _____ s/mm^2

Blank 8 _____ s/mm^2

. .

. .

. .

Blank n_B _____ s/mm^2

Total 17.60

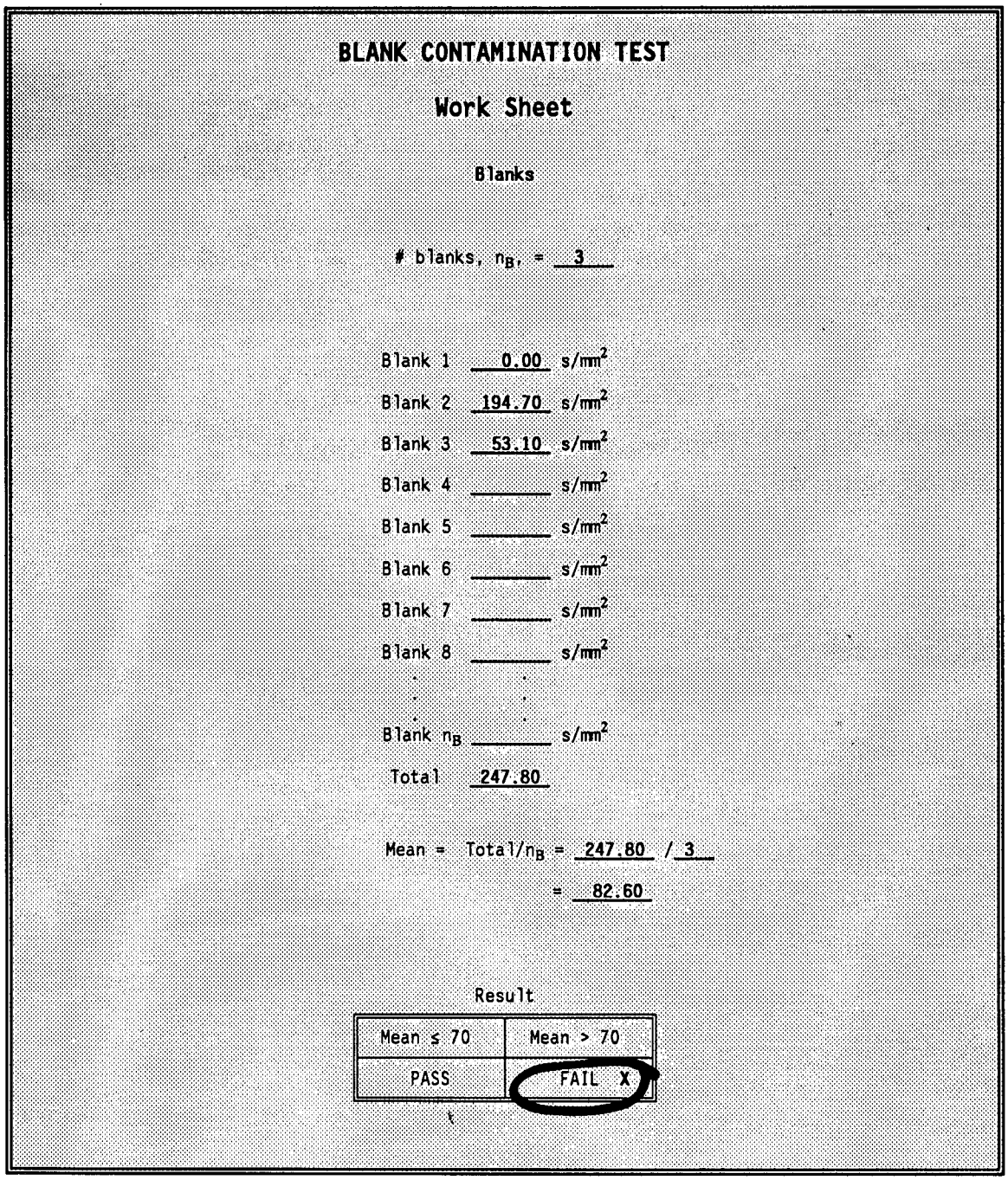
Mean = Total/n_B = 17.60 / 3

= 5.87

Result

Mean ≤ 70	Mean > 70
PASS X	FAIL

Figure 5. The Blank Contamination Test -- Example 4. In this example the blanks pass the test and the laboratory proceeds to the analysis of the outside samples.



BLANK CONTAMINATION TEST

Work Sheet

Blanks

blanks, n_B, = 3

Blank 1 0.00 s/mm^2

Blank 2 194.70 s/mm^2

Blank 3 53.10 s/mm^2

Blank 4 _____ s/mm^2

Blank 5 _____ s/mm^2

Blank 6 _____ s/mm^2

Blank 7 _____ s/mm^2

Blank 8 _____ s/mm^2

Blank n_B _____ s/mm^2

Total 247.80

Mean = Total/n_B = 247.80 / 3

= 82.60

Result

Mean ≤ 70	Mean > 70
PASS	FAIL X

Figure 6. The Blank Contamination Test -- Example 5. In this example the blanks fail the test. The problem must be corrected and a new set of samples (inside, blank, and outside) collected.

5.3 Z-test

After passing the blank contamination test, the outside samples are analyzed and compared with the inside samples using the Z-test. Note that the Z-test is a comparison of airborne asbestos levels inside and outside the work site and therefore is based on the concentrations per cubic centimeter of air.

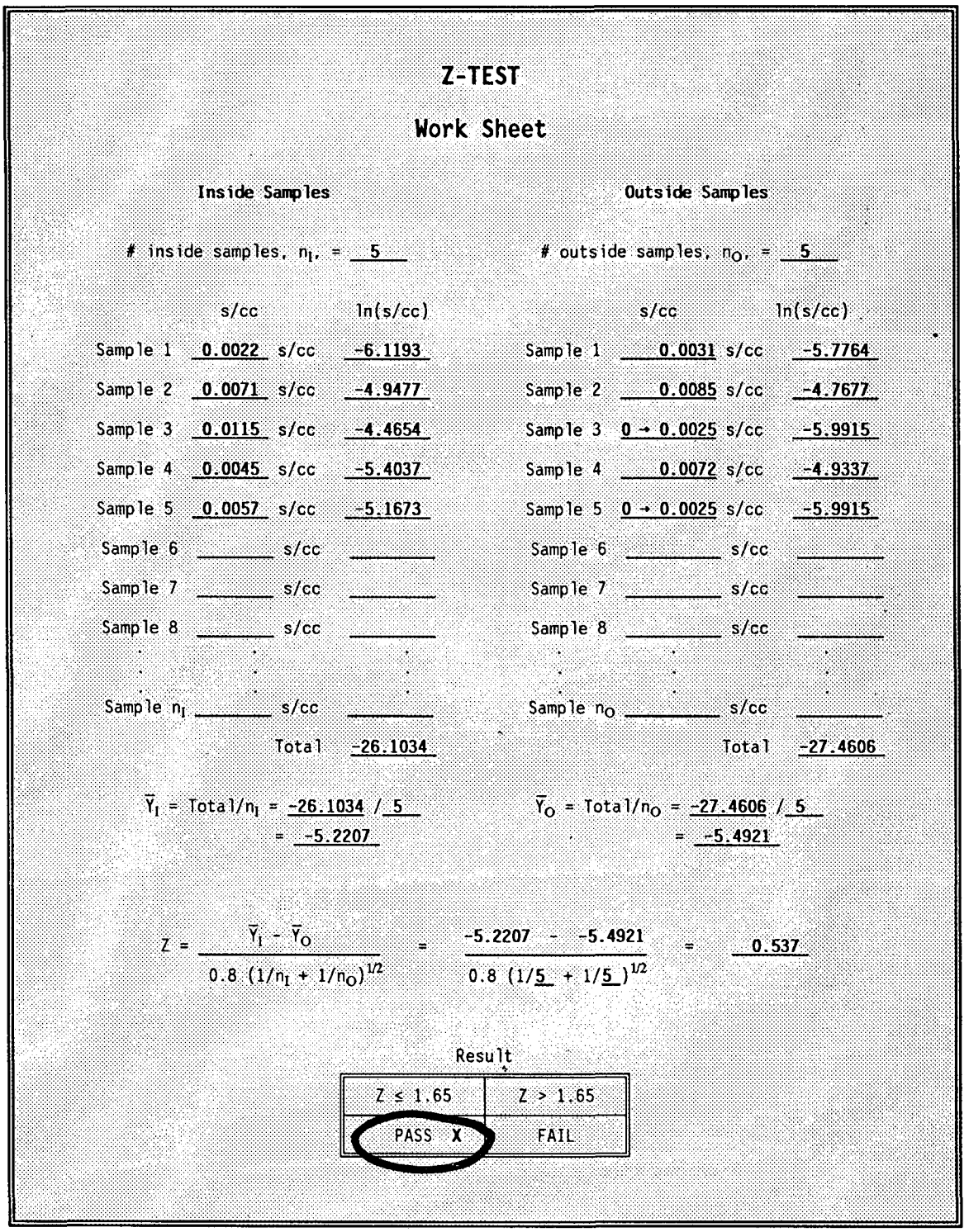
The Z-test uses natural logarithms. Since it is not possible to take the logarithm of zero, samples on which no asbestos structures were detected are given a small positive value. In the AHERA rule this small value is referred to as the "detection limit," although this term is used differently in other contexts. The statement is meant to apply only to the Z-test, not to the initial screening test. In its current form, the wording of the rule is ambiguous. (A clarification is anticipated in an upcoming revision.) In line with the expected revision, it is recommended that 0.0025 s/cc be substituted for any zero concentration prior to taking logarithms. Calculate the arithmetic mean of the logarithms for the inside samples ($\overline{Y}_I$) and the arithmetic mean of the logarithms for outside samples ($\overline{Y}_0$). Then calculate Z according to the formula

$$Z = \frac{\overline{Y}_I - \overline{Y}_0}{0.8\ (1/n_I + 1/n_0)^{1/2}}$$

where n_I is the number of inside samples and n_0 is the number of outside samples. If Z is less than or equal to 1.65, the inside and outside concentrations are not statistically different from each other and the work site passes the clearance test. If Z is greater than 1.65 the work site fails the clearance test. The work site must be recleaned and the sampling and analysis procedures repeated.

Example 6. Figure 7 shows the structure concentrations per cubic centimeter of air for five inside samples and five outside samples. No structures were observed on two of the outside samples. Their concentrations were replaced by 0.0025 before taking logarithms. Z is less than 1.65 and therefore the work site passes the clearance test.

Example 7. In this example the LEA decided to collect and analyze 8 inside samples (one per room) and 5 outside samples. The results are shown in Figure 8. Z is greater than 1.65 and the work site failed the clearance test. The entire work site was recleaned with special emphasis given to the two rooms in which the fifth and seventh samples were collected.



Z-TEST

Work Sheet

Inside Samples

inside samples, n_I, = 5

	s/cc		ln(s/cc)
Sample 1	0.0022	s/cc	-6.1193
Sample 2	0.0071	s/cc	-4.9477
Sample 3	0.0115	s/cc	-4.4654
Sample 4	0.0045	s/cc	-5.4037
Sample 5	0.0057	s/cc	-5.1673
Sample 6		s/cc	
Sample 7		s/cc	
Sample 8		s/cc	
Sample n_I		s/cc	
		Total	-26.1034

$\bar{Y}_I$ = Total/n_I = -26.1034 / 5 = -5.2207

Outside Samples

outside samples, n_O, = 5

	s/cc		ln(s/cc)
Sample 1	0.0031	s/cc	-5.7764
Sample 2	0.0085	s/cc	-4.7677
Sample 3	0 → 0.0025	s/cc	-5.9915
Sample 4	0.0072	s/cc	-4.9337
Sample 5	0 → 0.0025	s/cc	-5.9915
Sample 6		s/cc	
Sample 7		s/cc	
Sample 8		s/cc	
Sample n_O		s/cc	
		Total	-27.4606

$\bar{Y}_O$ = Total/n_O = -27.4606 / 5 = -5.4921

$$Z = \frac{\bar{Y}_I - \bar{Y}_O}{0.8\,(1/n_I + 1/n_O)^{1/2}} = \frac{-5.2207 - -5.4921}{0.8\,(1/5 + 1/5)^{1/2}} = 0.537$$

Result

Z ≤ 1.65	Z > 1.65
PASS X	FAIL

Figure 7. The Z-test -- Example 6. In this example the work site passes the clearance test.

Z-TEST

Work Sheet

Inside Samples			Outside Samples		
# inside samples, n_I, = 8			# outside samples, n_O, = 5		
	s/cc	ln(s/cc)		s/cc	ln(s/cc)
Sample 1	0.0093 s/cc	-4.6777	Sample 1	0.0065 s/cc	-5.0360
Sample 2	0.0071 s/cc	-4.9477	Sample 2	0.0070 s/cc	-4.9618
Sample 3	0.0115 s/cc	-4.4654	Sample 3	0.0053 s/cc	-5.2400
Sample 4	0.0045 s/cc	-5.4037	Sample 4	0.0035 s/cc	-5.6550
Sample 5	0.0418 s/cc	-3.1749	Sample 5	0.0043 s/cc	-5.4491
Sample 6	0.0082 s/cc	-4.8036	Sample 6	_____ s/cc	_____
Sample 7	0.0245 s/cc	-3.7091	Sample 7	_____ s/cc	_____
Sample 8	0.0085 s/cc	-4.7677	Sample 8	_____ s/cc	_____
Sample n_I	_____ s/cc	_____	Sample n_O	_____ s/cc	_____
	Total	-35.9498		Total	-26.3419

$\bar{Y}_I$ = Total/n_I = -35.9498 / 8 = -4.4937

$\bar{Y}_O$ = Total/n_O = -26.3419 / 5 = -5.2684

$$Z = \frac{\bar{Y}_I - \bar{Y}_O}{0.8\,(1/n_I + 1/n_O)^{1/2}} = \frac{-4.4937 - -5.2684}{0.8\,(1/8 + 1/5)^{1/2}} = 1.699$$

Result

Z ≤ 1.65	Z > 1.65
PASS	FAIL X

Figure 8. The Z-test -- Example 7. In this example the work site fails the clearance test. The site must be recleaned and a new set of samples collected.

Example 8. The results shown in Figure 9 give a value of Z that is less than 1.65 and therefore the work site passes the test. However, airborne asbestos levels both inside and outside the work site were somewhat higher than those measured after abatement projects in neighboring schools. The LEA decided that it would be prudent to investigate the matter further and asked its asbestos consultant to investigate and report on other potential sources of asbestos both inside and outside the building. Meanwhile, a special asbestos operations and maintenance program remained in effect throughout the school.

Example 9. The Z-value in this example is greater than 1.65 (Figure 10). Therefore the work site does not pass the clearance test. Examination of the laboratory report revealed that the asbestos structures identified on the inside samples were amosite, while the removal had involved only chrysotile asbestos. After a thorough inspection, previously undetected amosite insulation was discovered on pipes above the suspended ceiling. Minor damage to the pipe insulation was repaired and the work site, including above the ceiling, was recleaned before collecting a new set of air samples.

The preceding examples, as well as demonstrating how to perform the Z-test, indicate that the LEA can and should take into account additional information to decide when an abatement project is complete. Where possible, the contract with the abatement contractor should reflect this to avoid later disagreements. The laboratory report will indicate the type of asbestos (for example, chrysotile or amphibole) and the types of asbestos structures observed (individual fibers or complex structures such as bundles, clusters, or matrices consisting of multiple fibers). The LEA might consider recleaning, if, for example, the work site just passes the Z-test but the inside samples are predominantly amphibole, whereas the outside are exclusively chrysotile. A predominance of complex structures inside the work site compared to single fibers outside the work site, may also suggest that conditions inside the work site are not yet comparable with those outside the work site.

Z-TEST

Work Sheet

Inside Samples			Outside Samples		
# inside samples, n_I, = 8			# outside samples, n_O, = 8		
	s/cc	ln(s/cc)		s/cc	ln(s/cc)
Sample 1	0.0093 s/cc	-4.6777	Sample 1	0.0065 s/cc	-5.0360
Sample 2	0.0154 s/cc	-4.1734	Sample 2	0.0154 s/cc	-4.1734
Sample 3	0.0115 s/cc	-4.4654	Sample 3	0.0341 s/cc	-3.3785
Sample 4	0.0245 s/cc	-3.7091	Sample 4	0.0287 s/cc	-3.5509
Sample 5	0.0418 s/cc	-3.1749	Sample 5	0.0043 s/cc	-5.4491
Sample 6	0.0098 s/cc	-4.6254	Sample 6	0.0136 s/cc	-4.2977
Sample 7	0.0162 s/cc	-4.1227	Sample 7	0.0057 s/cc	-5.1673
Sample 8	0.0326 s/cc	-3.4234	Sample 8	0.0223 s/cc	-3.8032
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
Sample n_I _____	s/cc	_____	Sample n_O _____	s/cc	_____
	Total	-32.3720		Total	-34.8559

$\bar{Y}_I$ = Total/n_I = -32.3720 / 8 = -4.0465

$\bar{Y}_O$ = Total/n_O = -34.8559 / 8 = -4.3570

$$Z = \frac{\bar{Y}_I - \bar{Y}_O}{0.8\,(1/n_I + 1/n_O)^{1/2}} = \frac{-4.0465 - -4.3570}{0.8\,(1/8 + 1/8)^{1/2}} = 0.776$$

Result

Z ≤ 1.65	Z > 1.65
PASS X	FAIL

Figure 9. The Z-test -- Example 8. In this example the work site passes the clearance test.

Z-TEST

Work Sheet

Inside Samples			Outside Samples		
# inside samples, n_I, = 5			# outside samples, n_O, = 5		
	s/cc	ln(s/cc)		s/cc	ln(s/cc)
Sample 1	0.0071 s/cc	-4.9477	Sample 1	0.0065 s/cc	-5.0360
Sample 2	0.0204 s/cc	-3.8922	Sample 2	0.0037 s/cc	-5.5994
Sample 3	0.0079 s/cc	-4.8409	Sample 3	0.0050 s/cc	-5.2983
Sample 4	0.0150 s/cc	-4.1997	Sample 4	0.0028 s/cc	-5.8781
Sample 5	0.0095 s/cc	-4.6565	Sample 5	0.0043 s/cc	-5.4491
Sample 6	_____ s/cc	_____	Sample 6	_____ s/cc	_____
Sample 7	_____ s/cc	_____	Sample 7	_____ s/cc	_____
Sample 8	_____ s/cc	_____	Sample 8	_____ s/cc	_____
Sample n_I	_____ s/cc	_____	Sample n_O	_____ s/cc	_____
	Total	-22.5370		Total	-27.2609

$\bar{Y}_I$ = Total/n_I = -22.5370 / 5 = -4.5074

$\bar{Y}_O$ = Total/n_O = -27.2609 / 5 = -5.4522

$$Z = \frac{\bar{Y}_I - \bar{Y}_O}{0.8\,(1/n_I + 1/n_O)^{1/2}} = \frac{-4.5074 - -5.4522}{0.8\,(1/5 + 1/5)^{1/2}} = 1.867$$

Result

$Z \leq 1.65$	$Z > 1.65$
PASS	FAIL X

Figure 10. The Z-test -- Example 9. In this example the work site fails the clearance test. The site must be recleaned and new samples collected.

REFERENCES

USEPA. 1985a. U.S. Environmental Protection Agency. Guidance for controlling asbestos-containing materials in buildings. Washington, DC: Office of Toxic Substances, USEPA. EPA 560/5-85-019.

USEPA. 1985b. U.S. Environmental Protection Agency. Measuring airborne asbestos following an abatement action. Research Triangle Park, NC: Environmental Monitoring Systems Laboratory and Office of Toxic Substances, USEPA. EPA 600/4-85-049.

APPENDIX A

WORK SHEETS

Note: Identifying information such as location, sample ID, date, and signature of the evaluator should be added if the work sheets are to be used as permanent documentation.

INITIAL SCREENING TEST

WORK SHEET

Inside Samples

inside samples, n_I, = _____

Sample 1 _____ s/mm^2

Sample 2 _____ s/mm^2

Sample 3 _____ s/mm^2

Sample 4 _____ s/mm^2

Sample 5 _____ s/mm^2

Sample 6 _____ s/mm^2

Sample 7 _____ s/mm^2

Sample 8 _____ s/mm^2

. .
. .
. .

Sample n_I _____ s/mm^2

Total _____

Mean = Total/n_I = _____ /____

= _____

Circle Result

Mean ≤ 70	Mean > 70
PASS	FAIL

BLANK CONTAMINATION TEST

WORK SHEET

Blanks

blanks, n_B, = _____

Blank 1 _____ s/mm^2

Blank 2 _____ s/mm^2

Blank 3 _____ s/mm^2

Blank 4 _____ s/mm^2

Blank 5 _____ s/mm^2

Blank 6 _____ s/mm^2

Blank 7 _____ s/mm^2

Blank 8 _____ s/mm^2

. .
. .
. .

Blank n_B _____ s/mm^2

Total _____

Mean = Total/n_B = _____ /____

= _____

Circle Result

Mean ≤ 70	Mean > 70
PASS	FAIL

Z-TEST

Work Sheet

Inside Samples

inside samples, n_I, = _____

	s/cc	ln(s/cc)
Sample 1	_____ s/cc	_____
Sample 2	_____ s/cc	_____
Sample 3	_____ s/cc	_____
Sample 4	_____ s/cc	_____
Sample 5	_____ s/cc	_____
Sample 6	_____ s/cc	_____
Sample 7	_____ s/cc	_____
Sample 8	_____ s/cc	_____
.	.	.
.	.	.
.	.	.
Sample n_I	_____ s/cc	_____
	Total	_____

$\bar{Y}_I$ = Total/n_I = _____ /____

= _____

Outside Samples

outside samples, n_O, = _____

	s/cc	ln(s/cc)
Sample 1	_____ s/cc	_____
Sample 2	_____ s/cc	_____
Sample 3	_____ s/cc	_____
Sample 4	_____ s/cc	_____
Sample 5	_____ s/cc	_____
Sample 6	_____ s/cc	_____
Sample 7	_____ s/cc	_____
Sample 8	_____ s/cc	_____
.	.	.
.	.	.
.	.	.
Sample n_O	_____ s/cc	_____
	Total	_____

$\bar{Y}_O$ = Total/n_O = _____ /____

= _____

$$Z = \frac{\bar{Y}_I - \bar{Y}_O}{0.8\,(1/n_I + 1/n_O)^{1/2}} = \frac{\quad - \quad}{0.8\,(1/\_\_ + 1/\_\_)^{1/2}} = \_\_\_\_\_$$

Circle Result

$Z \leq 1.65$	$Z > 1.65$
PASS	FAIL

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16. Abstract (Limit: 200 words)

Guidance is provided for conducting the transmission electron microscopy clearance test required under the Asbestos Hazard Emergency Response Act (40 CFR Part 763) to determine when an asbestos abatement site is sufficiently clean for the containment barriers to be removed. Emphasis is on interpretation of the results. The three components of the clearance test -- the initial screening test, the blank contamination test, and the Z-test -- are described and illustrated with numerical examples.

17. Document Analysis a. Descriptors

AHERA, asbestos, asbestos air monitoring, asbestos abatement, clearance, TEM, transmission electron microscopy.

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