Marian Murphy
Project Manager

September 19, 2007

Mr. Charlie Fitzsimmons, On-Scene Coordinator
U.S. Environmental Protection Agency, Region 3
1160 Arch Street
Philadelphia, PA

Subject: **Revised Final Air Monitoring and Sampling and Analysis Plan for the Elkton Farms Firehole Site – DTN 0391**
EPA Contract No. EP-S3-05-02
TDD No. E23-014-07-07-001

Dear Mr. Fitzsimmons:

The Tetra Tech EM Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) is submitting the final air monitoring and air sampling and analysis plan (AM/SAP), which details START's approach to collecting industrial hygiene samples for asbestos and monitoring at the Elkton Farms Firehole site located in Elkton, Maryland.

Please call Marian Murphy (610-364-2129) or Rick Ecord, Certified Industrial Hygienist (404-225-5527) if you have any questions about the AM/SAP.

Sincerely,

Marian Murphy

Marian Murphy

Project Manager

cc: START TDD Files

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Revised Final Air Monitoring and Sampling and Analysis Plan
FOR THE
ELKTON FARMS FIREHOLE SITE
ELKTON, CECIL COUNTY, MARYLAND

Prepared for

U.S. Environmental Protection Agency
1650 Arch Street
Philadelphia, PA 19103

Prepared by

Tetra Tech EM Inc.
7 Creek Parkway
Boothwyn, PA 19061

EPA Contract No. EP-S3-05-02

Technical Direction Document No. E23-014-07-07-001
Document Tracking No. 0391

September 19, 2007

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Approved by

Andy Mazzeo
Philadelphia Operations Manager

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1.0 INTRODUCTION

Under Eastern Area Superfund Technical Assessment and Response Team (START) Contract No. EP-S3-05-02, Technical Direction Document (TDD) No. E23-014-07-07-001, U.S. Environmental Protection Agency (EPA) Region 3 tasked Tetra Tech EM Inc. (Tetra Tech) to conduct air monitoring and air sampling at the Elkton Farms Firehole (Elkton Farm) in Elkton, Cecil County, Maryland. The purpose of the sampling is to determine the quantity of total dust generated by site activities and concentration of asbestos fibers in ambient air in support of the site Phase II Work Plan. Implementation of this air monitoring and sampling and analysis plan (AMSAP) will provide continuous, real-time air monitoring data and validated analytical data that can be used to determine if proper site safety (worker protection) and engineering controls are being used to control fugitive dust emissions during intrusive activities at the site and to protect the safety of the surrounding community. Data will assist onsite personnel in determination of proper levels of protection to be worn during excavation activities.

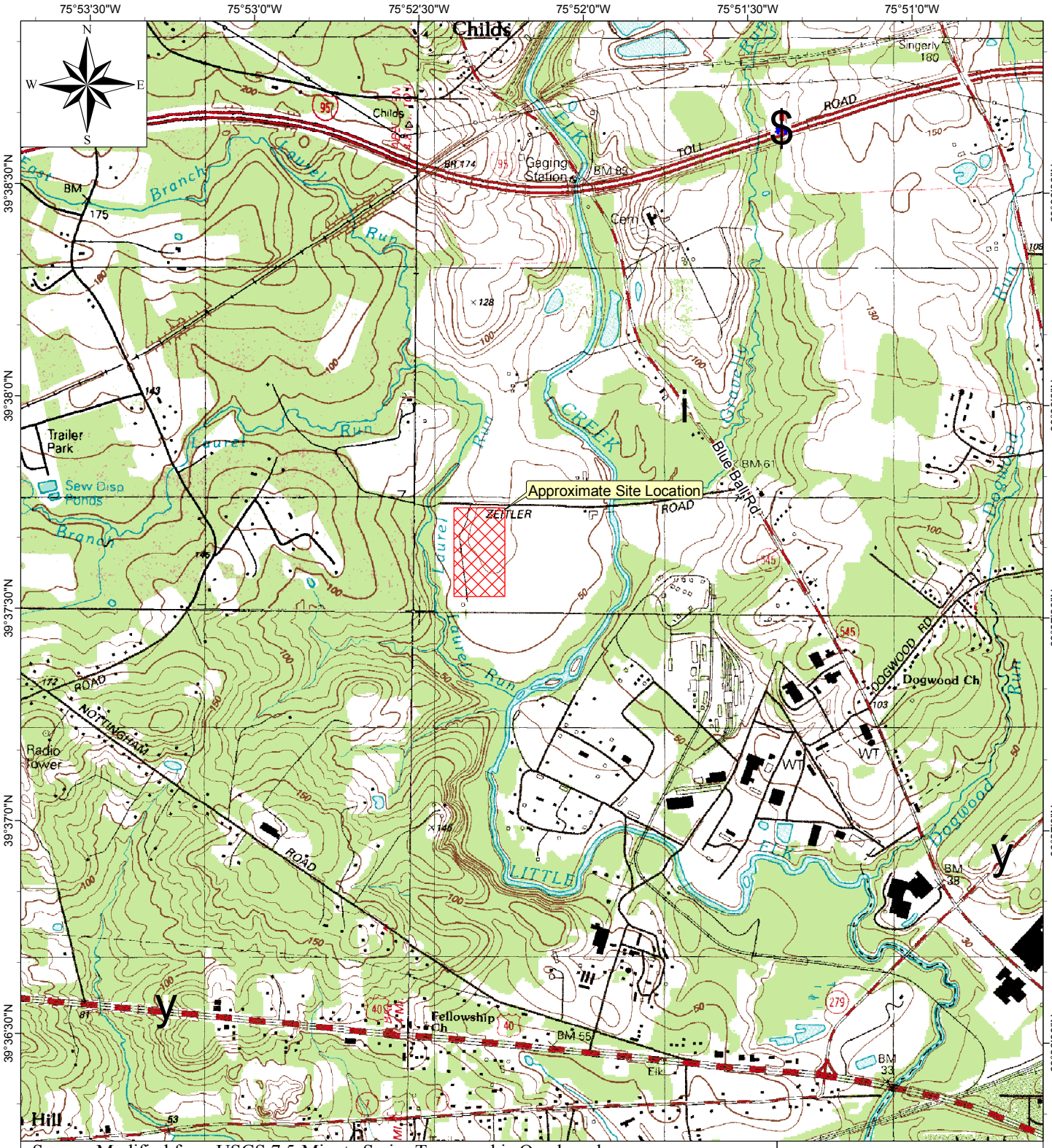
This AMSAP discusses the site background information in Section 2.0, project objectives and data use in Section 3.0, proposed air monitoring and sampling activities in Section 4.0, quality assurance (QA) and quality control (QC) procedures in Section 5.0, project deliverables in Section 6.0, and the project schedule in Section 7.0. All references cited in this plan are listed after Section 7.0.

2.0 SITE BACKGROUND

This section discusses the site location and description, and summarizes previous site investigation activities.

2.1 SITE LOCATION

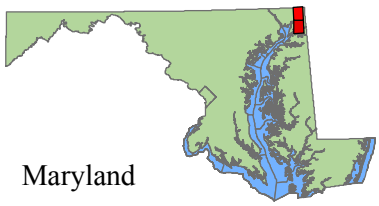
The Elkton Farm site is located on the south side of Zeitler Road, Elkton, Cecil County, Maryland, as shown on Figure 1 (U.S. Geological Survey 1992). The geographic coordinates of the approximate center of the site are 39.62813° north latitude and75.8477° west longitude. It is



Source: Modified from USGS 7.5-Minute Series Topographic Quadrangles,
Elkton, Maryland - Delaware, 1992
Newark West, Maryland - Delaware - Pennsylvania, 1992

0 0.25 0.5 Miles

Quadrangle Location =



Maryland

Elkton Farms Firehole Site
Elkton, Cecil County, Maryland

Figure 1
Site Location Map

TDD No. E23-014-07-07-001
EPA Contract No. EP-S3-05-02

Map created on September 8, 2006
by D. Call, Tetra Tech EMI

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situated between Maryland Route 40, Pulaski Highway, and Route 545, Blue Ball Road. (EPA 2006).

2.2 SITE DESCRIPTION

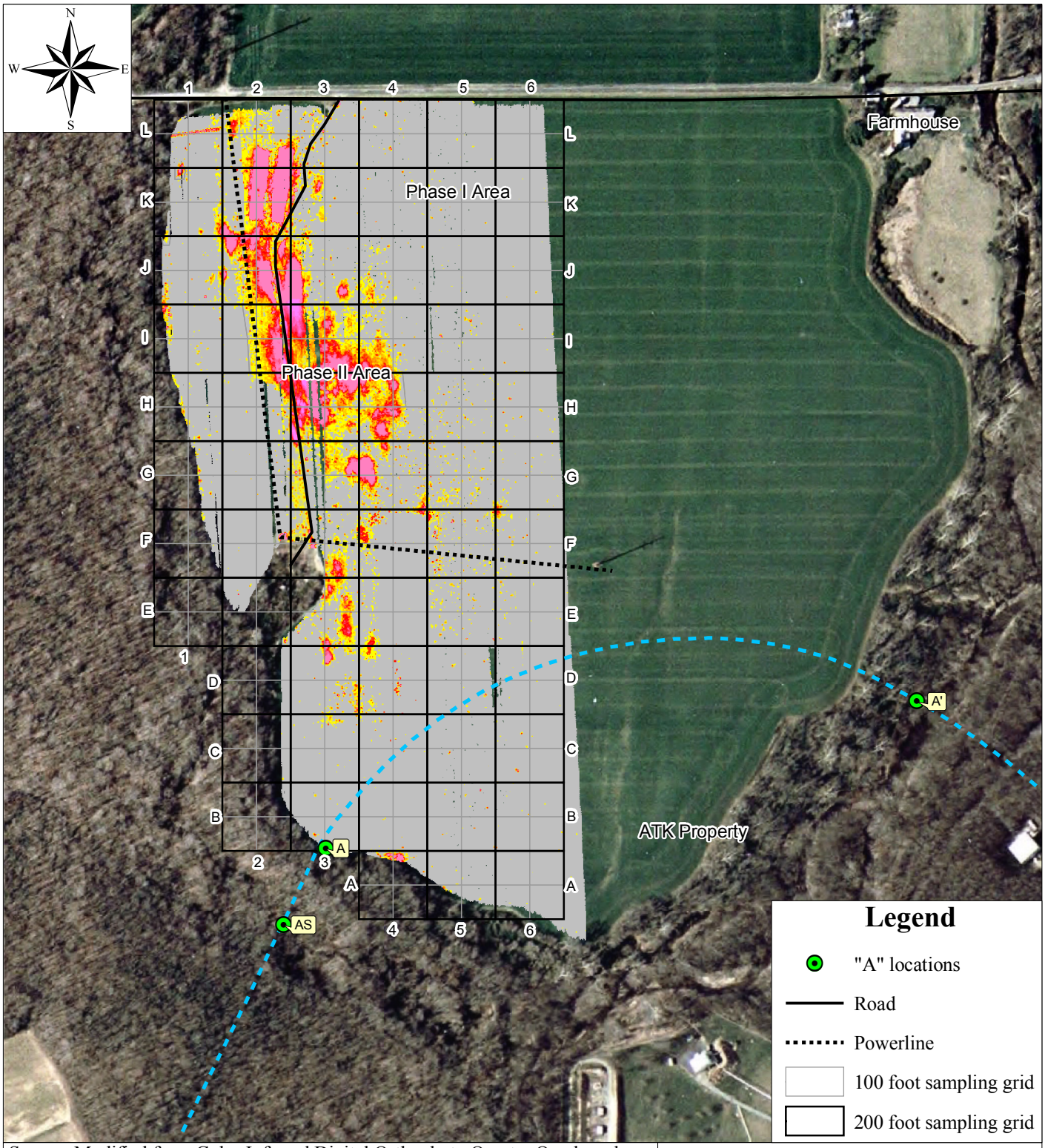
The Elkton Farms site is located at 183 Zeitler Road approximately 2 miles northwest of the city of Elkton, Cecil County, Maryland. The site occupies approximately 55 acres of an approximately 400-acre farm property presently owned by the Herron Ltd. Partnership. The site is bounded on the west by Laurel Run, to the north by Zeitler Road, and to the East by Little Elk Creek. A gravel access road bisects the western quadrant of the site. Land use surrounding the site is primarily agricultural/residential with an area of medium to heavy industry property to the southeast across Little Elk Creek (EPA 2006).

2.3 SITE HISTORY

Refer to EPA Action Memorandum for the site for the history (EPA 2006).

2.4 PREVIOUS SITE INVESTIGATIONS

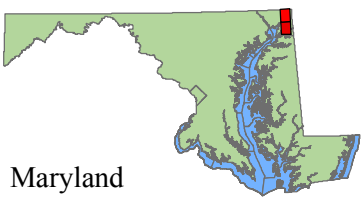
On June 12, 2006, Tetra Tech collected six asbestos bulk samples (009-BA01-A01 to 009-BA06-A01) from various materials located in the soil piles excavated from the east and west pits in the high anomaly area located in Phase 2. These samples were collected due to the identification of presumed-asbestos containing materials (PACM). Each bulk asbestos sample collected was of a different type of PACM. Asbestos bulk samples were double bagged, labeled, and secured in a cooler, and delivered to the laboratory for asbestos analysis (Tetra Tech 2006a).



Source: Modified from Color Infrared Digital Orthophoto Quarter Quadrangles, Newark Southwest and Elkton Northwest, Maryland Quadrangles, Maryland Department of Natural Resources, 1988 - 1995

0 250 500 Feet

Quadrangle Location =



Maryland

Elkton Farms Firehole Site
Elkton, Cecil County, Maryland

Figure 2
Site Layout Map

TDD No. E23-014-07-07-001
EPA Contract No. EP-S3-05-02

Map created on June 13, 2007
by D. Call, Tetra Tech EMI

TETRA TECH

Additionally, two composite soil samples (009-SA01-A01 and 009-SA02-A01) were collected for asbestos analysis. Each sample was a composite of each soil pile at the site. The samples were collected to identify if asbestos may be present at levels of concern in the soil, not to definitively determine whether the soils contain less than one percent asbestos at the site. Soil samples were double bagged, labeled, and secured in a cooler. Dedicated sampling equipment including plastic scoops and disposable aluminum pans were used to collect the soil samples for asbestos analysis. The bulk asbestos and soil asbestos samples were collected to determine if asbestos was present in PACMs and if asbestos was present in soils excavated from the Phase 2 pits. A total of five out of six bulk asbestos samples contained chrysotile asbestos. The bulk material contained 35 to 55 percent asbestos in the materials sampled. Two out of the five bulk samples containing asbestos may be friable types of asbestos. The bulk samples were analyzed using phase contrast microscopy analysis (Tetra Tech 2006a).

The soil samples collected from the Phase 2 pit materials were analyzed using transmission electron microscopy (TEM) to provide a lower detection limit. Asbestos was not detected above the detection limit of 0.01 percent (Tetra Tech 2006a).

3.0 PROJECT OBJECTIVES AND DATA USE

The objectives of the air monitoring and air sampling activities are to determine the concentrations of dust and dust laden with asbestos in on- and off-site air during intrusive activities starting at the southern end of Phase II and working north. Implementation of this AMSAP will provide continuous, real-time air monitoring data and validated analytical data that can be used to determine if proper worker protection levels and site safety and engineering controls are being used to control fugitive dust emissions during excavation activities at the site and to protect the safety of the residents living around the site.

This AMSAP consists of the appropriate Occupational Safety and Health Administration (OSHA) and National Institute for Occupational Safety and Health (NIOSH) methods to be used to collect these samples, the rationale supporting the sampling schemes, and the sampling

schemes for both employee and community exposure determination. Sample collection locations including personnel samplers will be more accurately determined in the field at the time samples are collected, thus are not provided as a figure in this document. Tetra Tech field personnel will record in the official field logbooks any deviations from the sampling approach and the analytical approach for the fixed laboratory discussed in this AMSAP.

4.0 PROPOSED AIR MONITORING AND SAMPLING ACTIVITIES

This section describes the scope of work, summarizes Tetra Tech personnel responsible for performing the tasks discussed in this AMSAP, and discusses the proposed air monitoring and air sampling activities.

4.1 SCOPE OF WORK

The Tetra Tech START will conduct the following air monitoring and sampling activities at the Elkton Farm site during intrusive activities to ensure the health and safety of surrounding community:

- Conduct daily air monitoring to determine fugitive dust emissions leaving the exclusion zone and wash area using an MIE Data RAM
- Conduct daily personnel air monitoring of personnel in the exclusion zone and wash area using a MIE Personnel Data RAM
- Record data logged from the air monitoring equipment at the end of each day
- Provide downloaded and tabulated air monitoring data daily to the EPA on-scene coordinator (OSC)
- Perform air sampling and monitoring to confirm that engineering controls in place at the site are protective of the surrounding community.
- Perform personnel air sampling for asbestos impact on site workers to determine if proper levels of protection are being utilized with quick turnaround results.

4.2 KEY PROJECT PERSONNEL

The Tetra Tech START project manager for the TDD is Ms. Marian Murphy. As the project manager, Ms. Murphy is responsible and accountable for all aspects of the project scope of work, including achieving the technical, financial, and scheduling objectives for the project. Ms. Murphy will communicate directly with the EPA OSC for this project, Mr. Charlie Fitzsimmons. Table 1 lists the other Tetra Tech START personnel proposed for this the project and their roles and responsibilities. The technical or field personnel used to support the project may vary depending on project-specific needs, site conditions, and staff availability.

TABLE 1

KEY PROJECT PERSONNEL

Role	Name	Responsibility
Project Manager	Marian Murphy	The project manager is responsible for implementing all activities identified in the TDD; responsible for developing and implementing the site health and safety plan; has authority to commit resources necessary to complete the work; prepares all deliverables required by the TDD; communicates directly with the EPA OSC, the project team, and any other personnel needed to complete the project .
Field Support Personnel	Joe Taraba/ Steve Morpus	Field support personnel performs necessary sampling or monitoring activities as well as other tasks defined in the TDD or assigned by the EPA OSC or the Tetra Tech project manager; communicates directly with the Tetra Tech project manager.
Health and Safety Officer	Pete Hoover	The health and safety officer oversees and supports development of the site health and safety plan; communicates directly with the Tetra Tech project manager to ensure that all corporate health and safety protocols applicable to the site are being followed.
Chemist	Marian Murphy	The project chemist coordinates with the Tetra Tech project manager regarding the analytical requirements for the project; solicits and procures necessary laboratory services; reviews sample data and validates data, if necessary; communicates directly with the Tetra Tech project manager, field support personnel, EPA OSC, and START program manager as necessary.

TABLE 1 (Continued)

KEY PROJECT PERSONNEL

Role	Name	Responsibility
Graphics and Mapping Specialist	Dan Call	Graphic and mapping specialists generates maps and other figures for project deliverables or presentations; assists the Tetra Tech project manager or other personnel when global positioning system activities are required.
Point of Contact	Marian Murphy	The point of contact assists the Tetra Tech project manager as necessary to implement the project; commits or helps obtain all necessary company resources to meet the objectives of the TDD; provides document quality control reviews; and addresses and helps resolve project management issues with the Tetra Tech project managers.

Notes:

EPA	=	U.S. Environmental Protection Agency
OSC	=	On-Scene Coordinator
START	=	Superfund Technical Assessment and Response Team
TBD	=	To be determined
TDD	=	Technical Direction Document
Tetra Tech	=	Tetra Tech EM Inc.

4.3 PROPOSED AIR MONITORING ACTIVITIES

The Tetra Tech START will conduct air monitoring at the Elkton Farm site daily during intrusive activities. Tetra Tech START will measure dust particulate concentrations using a Thermo Anderson Data RAM (Data RAM) in four separate areas surrounding the activities proposed for that day. One location will be upwind of the activities, one downwind between the excavation area and surrounding community, one in the wash area, these monitors will measure PM10 dust. One DataRam will be placed inside the exclusion zone near intrusive activities to measure total dust. In addition to the Data RAMs, Tetra Tech will place Personal Data Rams (PDRs) on personnel doing intrusive activities in the work area for that day, at minimum one PDR will be placed in the cab of any equipment in the exclusion zone and one in the wash area in the breathing zone. A weather station will be located on site during intrusive activities to

determine wind speed and direction. These activities will be conducted in accordance with Tetra Tech's Standard Operating Procedure (SOP) No. 073 "Air Quality Monitoring" (Tetra Tech 1999b).

Tetra Tech START will use the data logging function of the Data RAMs and PDRs throughout the work day. Tetra Tech START will record all activities in the site logbook in accordance with Tetra Tech's SOP No. 024, "Recording of Notes in Field Logbook" (Tetra Tech 1999a). The Occupational Health and Safety Administration (OSHA) permissible exposure limit (PEL) on an 8-hour time weighted average (TWA) is 15 milligrams per cubic meter (mg/m^3) total dust and 5 mg/m^3 for respirable dust (NIOSH 2005).

4.4 PROPOSED AIR SAMPLING ACTIVITIES

Tetra Tech field personnel will record detailed descriptions about how samples are collected on an industrial hygiene air sampling collection form provided as Appendix A to this document. In particular, the field sampling personnel will describe the following:

- Sampling equipment used
- Calibration information
- Pump identification number
- Pump flow rate
- Sample run-time (including start and end times)
- Total volume of air collected

The following two populations of concern will be evaluated by the sample collection schemes described in this document: employees working on the Elkton Farm site; and the community surrounding the Elkton Farm site. Procedures for sample collection for each population are described below. Samples collected are for employee and community protection only, and not for the purpose of compliance with the EPA Asbestos Hazard Emergency Response Act or the National Emissions Standards for Hazardous Air Pollutants program for asbestos.

4.4.1 SITE WORKER AND COMMUNITY SAMPLE COLLECTION

All sampling and monitoring will take place in the Phase II grid areas.

4.4.1.1 Sampling Materials and Equipment – Worker and Community

Personal sample collection pumps will be used to collect required samples. A set of five pumps is immediately available to use for sample collection. NIOSH Method 7400, included as Attachment 1 (NIOSH 1994), describes the sample collection technique to be used at the Elkton Farm site and should be consulted in addition to the following instructions. One blank cassette will be delivered to the laboratory as a field blank with each new batch of sample cassettes used or at least once per work week.

4.4.1.2 Sample Equipment Calibration – Worker and Off-Site

Each sample pump will be calibrated using a DC-Lite low-flow meter. Each pump will be turned on and warmed up for 5 minutes before calibration. Each pump will be calibrated with a representative sample collection cassette in-line. The meter will read three consecutive measurements to determine the flow-rate of the pump. This calibration sequence will occur at the beginning of the work-shift and again at the end. The average of the two will be used as the overall flow rate for the sampling event and recorded on the sample collection documentation form, included as Attachment 1.

4.4.1.3 Asbestos Sampling and Hazard Communications

For the purposes of worker protection, intrusive activities will be considered any activity that has the potential to actively disturb the soil specifically excavation within the Phase II geographic zone. All employees will be required to attend site-specific asbestos hazard communications training before entering the exclusion zone during intrusive activities. This communication will be provided by EPA's prime contractor Guardian Environmental Services (GES) and is included in the site Health and Safety Plan and Phase II Work Plan.

4.4.1.4 Sample Collection Procedure – Worker

A sample will be collected from two employees one in the excavation area and one in the wash area each day based on anticipated asbestos contact for that area. The samples will be collected

in areas where intrusive activities occur or during any activities where asbestos may be disturbed as a result of work being performed in the area. Initially one set of asbestos samples for worker safety will be collected when mechanical means are used to remove soil. After the initial sample collection, samples will be collected when visible asbestos is detected or at the discretion of the OSC. The sampling pumps will be placed on employees expected to be in contact with the highest levels of asbestos. The sampling pumps used for sample collection will be set to draw approximately 2.0 liters per minute and will have a run time as close to 8 hours as possible. To prepare the cassette for sampling, it should be opened, with the entire bottom cap removed, and the open-face of the cassette should face downward when attached to the lapel of the employee whose breathing zone is being sampled.

4.4.1.5 Sample Collection Procedure – Site and off-Site

To determine exposure to other site workers and the surrounding community three samples will be collected each day intrusive activities are performed (as determined by the OSC and PM) where asbestos may be disturbed on the site. Two sample collection devices will be placed in the path of prevailing wind direction, one upwind and one downwind in between excavation area and crews performing other work on-site. Initially one set of asbestos samples for site and off-site exposure measurement will be collected when mechanical means are used to remove soil. After the initial sample collection, samples will be collected when visible asbestos is detected or at the discretion of the OSC. The pumps will be placed on the exclusion zone fencing surrounding the area of asbestos contamination at the site, or will be placed on metal stakes outside of the 200-foot exclusion zone distance established for the MEC. The exclusion zone fencing has been placed in a 50-foot buffer zone around the area of asbestos contamination. The calibration, cassette preparation, and sample collection procedures will be the same as worker collection procedures.

4.4.1.6 Sample Preparation Procedure

At the end of each sampling event, the cover and end-plugs will be placed back on each cassette. Sample cassettes will not be shipped in untreated polystyrene foam due to the possibility of fiber losses from electrostatic charges. A traffic report/chain-of-custody form and sample collection information form will be submitted with each shipment of samples.

In addition to the samples collected during the sampling event, a minimum of two field blanks or 10 percent of the total number of samples collected , (whichever is greater), will be sent to the laboratory for QC purposes.

4.4.2 SAMPLE PROCESSING AND LABORATORY ANALYSIS

Results from laboratory analysis will be given in terms of exposure to both on-site workers and off-site receptors. Asbestos analytical results will be reported within 1 day of sample collection. The units for each will be fibers per cubic centimeter (f/cc). If asbestos fibers are identified by phase contrast microscopy, then Tetra Tech will collect another air sample in the same area and have the sample analyzed by transmission emission spectroscopy to verify the type of asbestos fibers present (see Attachments 1 and 2).

Asbestos air samples will be transported to a fixed laboratory for analysis for the following analytical parameters in Table 2:

TABLE 2

ANALYTICAL PARAMETERS

Matrix	Analyses	Analytical Method	Container Quantity, Type, and Volume	Preservatives	Detection Limit	Maximum Holding Time
Air	Asbestos	NIOSH Method 7400 - PCM	(0.45- to 1.2-:µm cellulose ester membrane, 25-mm; conductive cowl on cassette)	None	0.05 f/cc	Not Applicable
Air	Asbestos	NIOSH Method 7402 - TEM	(0.45- to 1.2-:µm cellulose ester membrane, 25-mm; conductive cowl on cassette)	None	0.05 f/cc	Not Applicable

Notes:

µm	=	Micrometer	NIOSH	=	National Institute for Safety and Health
f/cc	=	Fibers per cubic centimeter	PCM	=	Phase Contrast Microscopy
mm	=	Millimeter	TEM	=	Transmission emission spectroscopy

4.4.5 ASBESTOS ANALYTICAL RESULTS

Asbestos analytical results will be compared to the OSHA Permissible Exposure Limit of 0.1 f/cc for employee exposure monitoring. EPA shall develop or determine an asbestos community exposure guideline for use in comparing the asbestos analytical results. Respirator use at the site shall adhere to the respiratory protection for asbestos fibers table below, as taken from OSHA 1910.1001 (g) (3).

TABLE 1.—RESPIRATORY PROTECTION FOR ASBESTOS FIBERS

Airborne concentration of asbestos or conditions of use	Required respirator
Not in excess of 1 f/cc (10 X PEL)	Half-mask air purifying respirator other than a disposable respirator, equipped with high efficiency filters.

Airborne concentration of asbestos or conditions of use	Required respirator
Not in excess of 5 f/cc (50 X PEL)	Full facepiece air-purifying respirator equipped with high efficiency filters.
Not in excess of 10 f/cc (100 X PEL)	Any powered air-purifying respirator equipped with high efficiency filters or any supplied air respirator operated in continuous flow mode.
Not in excess of 100 f/cc (1,000 X PEL)	Full facepiece supplied air respirator operated in pressure demand mode.
Greater than 100 f/cc (1,000 X PEL) or unknown concentration.	Full facepiece supplied air respirator operated in pressure demand mode, equipped with an auxiliary positive pressure self-contained breathing apparatus.

NOTE: a. Respirators assigned for high environmental concentrations may be used at lower concentrations, or when required respirator use is independent of concentration.
b. A high efficiency filter means a filter that is at least 99.97 percent efficient against mono-dispersed particles of 0.3 micrometers in diameter or larger.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

This section describes the QA/QC procedures for the sampling event of the Elkton Farm site. Specifically, this section addresses responsibility, sampling equipment decontamination, field controls, laboratory controls, data validation, and data evaluation and management.

5.1 RESPONSIBILITIES

The Tetra Tech project manager, Ms. Murphy, will be responsible for ensuring that sample quality and integrity are maintained in accordance with the "Quality Assurance Project Plan (QAPP) for START" (Tetra Tech 2006b), and that sample labeling and documentation procedures are carried out in accordance with Tetra Tech SOP No. 019, "Packaging and

Shipping Samples" (Tetra Tech 2000).

Regulations for packaging, marking, labeling, and shipping hazardous materials and wastes are promulgated by the U.S. Department of Transportation. Air carriers that transport hazardous materials (in particular, Federal Express) require compliance with the current International Air Transport Association (IATA) regulations, which apply to shipment and transport of hazardous materials by air carrier. Tetra Tech will follow all applicable IATA regulations.

5.2 EQUIPMENT DECONTAMINATION AND WASTE HANDLING

Non-dedicated equipment will be decontaminated in accordance with Tetra Tech SOP No. 002, "General Equipment Decontamination" (Tetra Tech 1999c). Decontamination of non-dedicated sampling equipment will consist of a tap water and Liquinox wash, a triple de-ionized water rinse, and air drying.

5.3 FIELD QUALITY CONTROL

Field QC measures will consist of collecting a field blank and will include sample documentation in the site logbook, as described in the Tetra Tech QAPP for START (Tetra Tech 2006b) and Tetra Tech SOP No. 024, "Recording of Notes in Field Logbook" (Tetra Tech 1999a). Tetra Tech will also prepare well installation logs documenting the temporary well installation parameters for the site. Field blanks will be collected to verify that the samples were properly handled during sample collection, sample shipment, and laboratory analysis.

5.4 LABORATORY QUALITY CONTROL

Samples will be hand delivered to the laboratory assigned by the EPA Region 3 Office of Analytical Services and Quality Assurance (OASQA). Laboratory QC measures will consist of all QC elements identified in the analytical method and will include completion of all forms and deliverables required by the method.

5.5 DATA VALIDATION

EPA Region 3 Central Regional Laboratory's QA staff will perform data validation in accordance with EPA Region 3 modifications to the EPA Contract Laboratory Program national functional guidelines for data review (EPA 1993 and 1994). Data will be validated an IM1 validation level for inorganic compounds.

5.6 DATA EVALUATION AND MANAGEMENT

This section describes how Tetra Tech will (1) evaluate the data generated from the sampling event, (2) determine whether the data are representative of site conditions and complete enough for use in making confident risk management decisions, and (3) ensure that the data are secure and retrievable.

5.6.1 Data Evaluation

Tetra Tech will review the analytical package to determine whether any major deficiencies were encountered during analysis and to ensure that the data are interpreted correctly. The analytical results will be compared against applicable benchmarks and background concentrations to evaluate conditions at the site.

5.6.2 Data Representativeness and Completeness

This AMSAP is designed to obtain data that are representative of environmental conditions at the site. If sampling varies significantly from this plan because of unexpected field conditions or other unforeseeable factors, Tetra Tech will discuss how those variations affect data representativeness in the trip report.

This AMSAP is also designed to obtain enough valid and acceptable data to achieve 90 percent completeness when compared against the amount of data planned. When validated analytical results are received, Tetra Tech will calculate the percent completeness based on an equation in the QAPP for START (Tetra Tech 2006b). If 90 percent completeness is not achieved because

fewer samples than anticipated are collected or because data are rejected during the validation process, the Tetra Tech project manager will discuss the matter with the EPA work assignment manager (WAM) and will include a discussion of the matter in the trip report.

5.6.3 Data Management

Tetra Tech will request that the laboratory submit the analytical data in electronic form as well as in the required hard copy analytical data package. Tetra Tech will compare the electronic data deliverables with the hard copy data package to ensure their consistency. When EPA Region 3 OASQA staff has validated the data set with the appropriate data qualifiers, the electronic data will be released to the Tetra Tech project manager for reporting. Tetra Tech will use the data to prepare a trip report for the project. All electronic data will be stored in a Microsoft (MS) Excel or Access database for future retrieval and reference based on the WAM's requirements. Each hard copy data package will be kept in the project file in the Tetra Tech office in Boothwyn, Pennsylvania, until the data package is officially transferred to EPA.

6.0 DELIVERABLES

Information obtained during and after the continuous air monitoring and sampling activities will be compiled on site for immediate use during intrusive activities and for future reference. All information gathered during these activities will be kept in the files at the site for future use by EPA.

7.0 PROJECT SCHEDULE

Tetra Tech anticipates mobilizing equipment to the site the second week in September 2007. Completion of all site activities is expected to require several weeks.

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APPENDIX A

INDUSTRIAL HYGIENE AIR SAMPLE COLLECTION FORM

(1 Page)

Air Sampling Data Sheet

Date: _____ **Sample ID No.:** _____

Time: _____ **Pump No.:** _____

Site Name: _____

Pump Type

( ) Gillian GilAir5 ( ) Meta Lite

Particulate Collection Devices

( ) 37 mm Polystyrene 3-piece filter cassette

( ) 25 mm Carbon filled black polypropylene w/cowl filter cassette

Particulate Collection Media

( ) Cellulose*
( ) MCE (.8 µm)*
( ) MCE (.45 µm)*
( ) MCE (1.2 µm)*
( ) PVC (.8 µm)*
( ) PVC (.45 µm)*
( ) PVC (5.0 µm)*
( ) PTFE (.8 µm)*
( ) PTFE (.45µm)*
( ) PTFE (.5 µm)*
( ) PTFE (1.0 µm)*
( ) PTFE (2.0 µm)*
( ) PTFE (5.0 µm)*
( ) Glass fiber (1.0 µm)
( ) VAC (.8 µm)
( ) Silver membrane (.45µm)
( ) Silver membrane (.8 µm)

* Requires support pad

Gas/Vapor Collection Devices

( ) Charcoal Sorbent Tube
( ) Chromosorb Sorbent Tube
( ) Porapak-T Sorbent Tube
( ) Cryogenic Trap
( ) Tenax Sorbent Tube
( ) Anasorb Sorbent Tube
( ) Hopcalite Sorbent Tube
( ) Impinger
( ) XAD Sorbent Tube
( ) Silica Gel Sorbent Tube
( ) Tedlar Bag
( ) PUF

Calibration Data

	Run #1	Run #2	Run #3	Average	Pre-Post Average
Pre-Cal	lpm	lpm	lpm	lpm	
Post-Cal	lpm	lpm	lpm	lpm	
Pre-Post					lpm(V)

Sample Start Time: _____ **Sample Stop Time:** _____

Total Time Sampled: _____ minutes (TIME)

Volume Calculations

_____ (TIME) x _____ (V) = _____ liters (VOLUME SAMPLED)

ATTACHMENT 1

NIOSH METHOD 7400
Asbestos and Other Fibers by PCM

(15 Pages)

ASBESTOS and OTHER FIBERS by PCM 7400

Various MW: Various CAS: Various 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 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; 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

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 (date 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 x 75-mm.
6. Cover slips, 22- x 22-mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

EQUIPMENT:

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 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100-µm diameter circular field (area = 0.00785 mm2) 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 ($3.85 \cdot 10^4$ to $5 \cdot 10^5$ 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:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

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 [3,7,8,9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9,10,11].

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 sec 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 manufacturers 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 2.77 (X)S_r', where X = average of the square roots of the two fiber counts

    (in fiber/mm^2) and $S_r' = \frac{S_r}{2}$, 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 1/2 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 seconds 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):

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

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):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}.$$

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 (Fig. 1).

EVALUATION OF METHOD:

A. 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 = .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 [21,22,23].

B. 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:

	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 indicated 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:

$$S_r = 1/(N)^{1/2} \qquad (1)$$

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 [17,19,20,21].

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:

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

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 x 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.

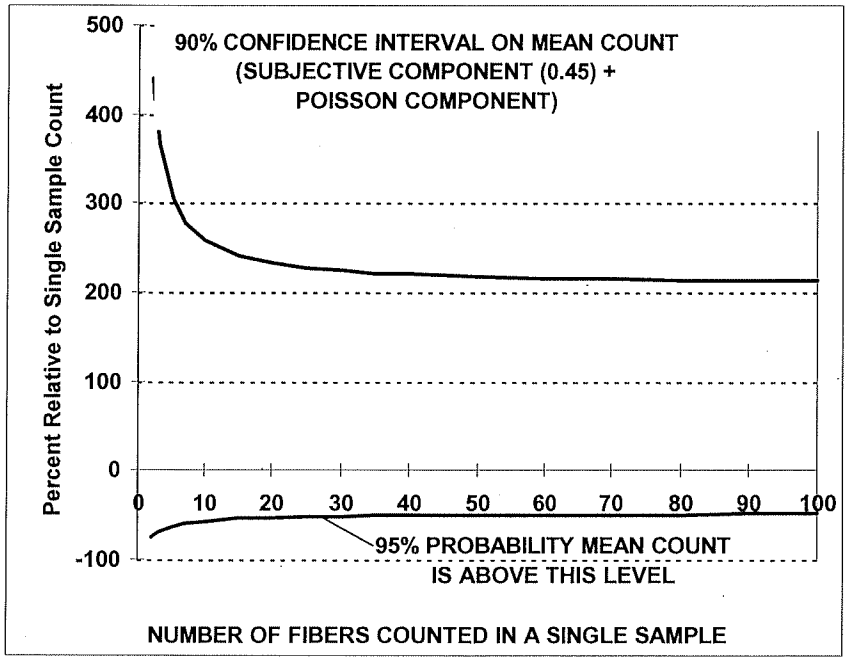

Figure 1. Interlaboratory Precision of Fiber Counts

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

$$UCL = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25 S_r^2) X^2]^{1/2}}{2(1 - 2.25 S_r^2)} \quad (3)$$

$$LCL = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4 S_r^2) X^2]^{1/2}}{2(1 - 4 S_r^2)} \quad (4)$$

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
LCL = lower 95% confidence limit.
UCL = upper 95% confidence limit.
Note that the range between these two limits represents 90% of the total range.

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METHOD WRITTEN BY:

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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 45X 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:

$$d_c = \frac{L_a}{L_o} \times D. \qquad (5)$$

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.

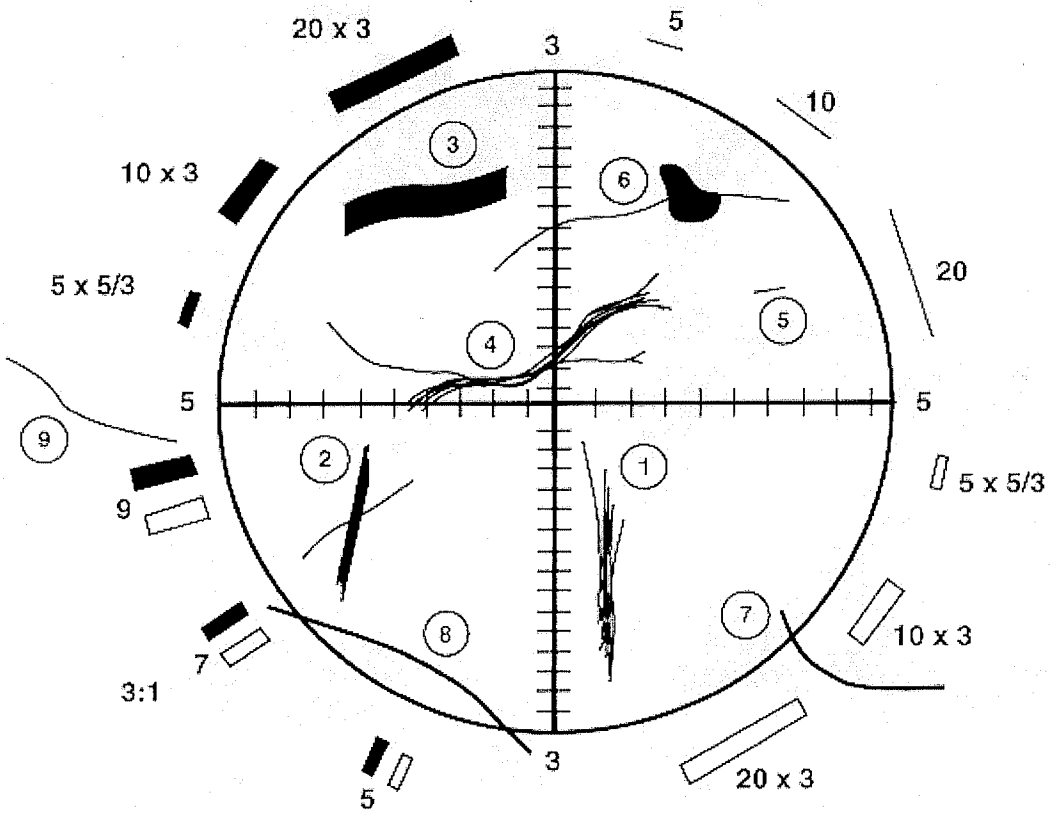

Figure 2. Walton-Beckett graticule with fibers.

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

FIBER COUNT

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 fiber	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	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once. count
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	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.

ATTACHMENT 2

NIOSH METHOD 7402
Asbestos and Other Fibers by TEM

(7 Pages)

ASBESTOS by TEM 7402

FORMULA: Various MW: Various CAS: Various RTECS: Various

METHOD: 7402 **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

OSHA : 0.1 asbestos fibers (>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 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anistropic

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].

SAMPLING

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

FLOW RATE: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)
*Adjust for 100 to 1300 fibers/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: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): see EVALUATION OF METHOD

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: MICROSCOPY, TRANSMISSION ELECTRON (TEM)

ANALYTE: asbestos fibers

SAMPLE PREPARATION: modified Jaffe wick

EQUIPMENT: transmission electron microscope; energy dispersive X-ray system (EDX) analyzer

CALIBRATION: qualitative electron diffraction; calibration of TEM magnification and EDX system

RANGE: 100 to 1300 fibers/mm^2 filter area [1]

ESTIMATED LOD: 1 confirmed asbestos fiber above 95% of expected mean blank value

PRECISION ($\hat{S}_r$): 0.28 when 65% of fibers are asbestos; 0.20 when adjusted fiber count is applied to PCM count [2].

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. This method is use d to determine asbestos fibers in the optically visible range and is intended to complement the results obtained by phase con trast microscopy (Method 7400).

INTERFERENCES: Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification. Some non-asbestos amphibole m inerals may give electron diffraction patterns similar to asbestos amphiboles.

OTHER METHODS: This method is designed for use with Method 7400 (phase contrast microscopy).

REAGENTS:

1. Acetone. (See SPECIAL PRECAUTIONS.)

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl, cellulose ester membrane filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean count is >5 fibers/100 fields. These are defined as laboratory blanks.
   NOTE 2: Use an electrically-conductive extension cowl to reduce electrostatic effects on fiber sampling and during sample shipment. Ground the cowl when possible during sampling.
   NOTE 3: 0.8-µm pore size filters are recommended for personal sampling. 0.45-µm filters are recommended for sampling when performing TEM analysis on the samples because the particles deposit closer to the filter surface. However, the higher pressure drop through these filters normally preclude their use with personal sampling pumps.
2. Personal sampling pump, 0.5 to 16 L/min, with flexible connecting tubing.
3. Microscope, transmission electron, operated at ca. 100 kV, with electron diffraction and energy-dispersive X-ray capabilities, and having a fluorescent screen with inscribed or overlaid calibrated scale (Step 15).
   NOTE: The scale is most efficient if it consists of a series of lines inscribed on the screen or partial circles every 2 cm distant from the center.
4. Diffraction grating replica with known number of lines/mm.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Knife, surgical steel, curved-blade.
7. Tweezers.
8. Grids, 200-mesh TEM copper, (optional: carbon-coated).
9. Petri dishes, 15-mm depth. The top and bottom of the petri dish must fit snugly together. To assure a tight fit, grind the top and bottom pieces together with an abrasive such as carborundum to produce a ground-glass contact surface.
10. Foam, clean polyurethane, spongy, 12-mm thick.
11. Filters, Whatman No. 1 qualitative paper or equivalent, or lens paper.
12. Vacuum evaporator.
13. Cork borer, (about 8-mm).
14. Pen, waterproof, marking.
15. Reinforcement, page, gummed.
16. Asbestos standard bulk materials for reference; e.g. SRM #1866, available from the National Institute of Standards and Technology.
17. Carbon rods, sharpened to 1 mm x 8 mm.
18. Microscope, light, phase contrast (PCM), with Walton-Beckett graticule (see method 7400).
19. Grounding wire, 22-gauge, multi-strand.
20. Tape, shrink- or adhesive-.

SPECIAL PRECAUTIONS: Acetone is extremely flammable (flash point = 0 °F). Take precautions not to ignite it. Heating of acetone must be done in a fume hood using a flameless, spark-free heat source. Asbestos is a confirmed human carcinogen. Handle only in a well-ventilated fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. For personal sampling, fasten sampler to worker's lapel near worker's mouth. Remove the top cover from cowl extension ("open-face") and orient sampler face down. Wrap joint between extender and monitor body with tape to help hold the cassette together and provide a marking surface to identify the cassette. Where possible, especially at low %RH, attach sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove top covers from the field blank cassettes and store top covers and cassettes in a clean area (e.g., closed bag or box) during sampling. Replace top covers when sampling is completed.
4. Sample at 0.5 to 16 L/min [3]. Adjust sampling rate, Q (L/min), and time, t (min), to produce fiber density, E, of 100 to 1300 fibers/mm ^{2} [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area (A_c = 385 mm^2)] for optimum accuracy. Do not exceed ca. 0.5 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

   NOTE: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 h (700 to 2800 L) 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 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 [3].
5. At the end of sampling, replace top cover and small end caps.
6. Ship samples upright with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in the shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

7. Remove circular sections from any of three quadrants of each sample and blank filter using a cork borer [4]. The use of three grid preparations reduces the effect of local variations in dust deposit on the filter.
8. Affix the circular filter sections to a clean glass slide with a gummed page reinforcement. Label the slide with a waterproof marking pen.
   NOTE: Up to eight filter sections may be attached to the same slide.
9. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 min for the sample filter(s) to fuse and clear.
   NOTE: The "hot block" clearing technique [5] of Method 7400 or the DMF clearing technique [6] may be used instead of steps 8 and 9.
10. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1-by 5-mm section of a graphite rod onto the cleared filter(s). Remove the slide to a clean, dry, covered petri dish [4].
11. Prepare a second petri dish as a Jaffe wick washer with the wicking substrate prepared from filter or lens paper placed on top of a 12-mm thick disk of clean, spongy polyurethane foam [7].

Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent.

NOTE: The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

12. Place the TEM grid on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish halves and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated.

NOTE: The level of acetone should be just high enough to saturate the filter paper without creating puddles.

13. Remove about a quarter section of the carbon-coated filter from the glass slide using a surgical knife and tweezers. Carefully place the excised filter, carbon side down, on the appropriately-labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to raise the acetone level as high as possible without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it (allowing drops of condensed acetone to form near the edge rather than in the center where they would drip onto the grid preparation).

CALIBRATION AND QUALITY CONTROL:

14. Determine the TEM magnification on the fluorescent screen:
    a. Define a field of view on the fluorescent screen either by markings or physical boundaries.
       NOTE: The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric) [7].
    b. Insert a diffraction grating replica into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that goniometer stage tilt is zero.
    c. Adjust microscope magnification to 10,000X. Measure the distance (mm) between the same relative positions (e.g., between left edges) of two widely-separated lines on the grating replica. Count the number of spaces between the lines.
       NOTE: On most microscopes the magnification is substantially constant only within the central 8- to 10-cm diameter region of the fluorescent screen.
    d. Calculate the true magnification (M) on the fluorescent screen:

$$m = \frac{X \cdot G}{Y}$$

       where: X = total distance (mm) between the two grating lines;
              G = calibration constant of the grating replica (lines/mm);
              Y = number of grating replica spaces counted
    e. After calibration, note the apparent sizes of 0.25 and 5.0 µm on the fluorescent screen. (These dimensions are the boundary limits for counting asbestos fibers by phase contrast microscopy.)
15. Measure 20 grid openings at random on a 200-mesh copper grid by placing a grid on a glass slide and examining it under the PCM. Use the Walton-Beckett graticule to measure the grid opening dimensions. Calculate an average graticule field dimension from the data and use this number to calculate the graticule field area for an average grid opening.
    NOTE: A grid opening is considered as one graticule field.
16. Obtain reference selected area electron diffraction (SAED) or microdiffraction patterns from standard asbestos materials prepared for TEM analysis.
    NOTE: This is a visual reference technique. No quantitative SAED analysis is required [7]. Microdiffraction may produce clearer patterns on very small fibers or fibers partially obscured by other material.
    a. Set the specimen holder at zero tilt.

b. Center a fiber, focus, and center the smallest field-limiting aperture on the fiber. Obtain a diffraction pattern. Photograph each distinctive pattern and keep the photo for comparison to unknowns.

NOTE: Not all fibers will present diffraction patterns. The objective lens current may need adjustment to give optimum pattern visibility. There are many more amphiboles which give diffraction patterns similar to the analytes named on p. 7402-1. Some, but not all, of these can be eliminated by chemical separations. Also, some non-amphiboles (e.g., pyroxenes, some talc fibers) may interfere.

17. Acquire energy-dispersive X-ray (EDX) spectra on approximately 5 fibers having diameters between 0.25 and 0.5 µm of each asbestos variety obtained from standard reference materials [7].

NOTE: The sample may require tilting to obtain adequate signal. Use same tilt angle for all spectra.

a. Prepare TEM grids of all asbestos varieties.

b. Use acquisition times (at least 100 sec) sufficient to show a silicon peak at least 75% of the monitor screen height at a vertical scale of ≥500 counts per channel.

c. Estimate the elemental peak heights visually as follows:

(1) Normalize all peaks to silicon (assigned an arbitrary value of 10).

(2) Visually interpret all other peaks present and assign values relative to the silicon peak.

(3) Determine an elemental profile for the fiber using the elements Na, Mg, Si, Ca, and Fe. Example: 0-4-10-3-<1 [7].

NOTE: In fibers other than asbestos, determination of Al, K, Ti, S, P, and F may also be required for fiber characterization.

(4) Determine a typical range of profiles for each asbestos variety and record the profiles for comparison to unknowns.

MEASUREMENT:

18. Perform a diffraction pattern inspection on all sample fibers counted under the TEM, using the procedures given in step 17. Assign the diffraction pattern to one of the following structures:

a. chrysotile;

b. amphibole;

c. ambiguous;

d. none.

NOTE: There are some crystalline substances which exhibit diffraction patterns similar to those of asbestos fibers. Many of these, (brucite, halloysite, etc.) can be eliminated from consideration by chemistry. There are, however, several minerals (e.g., pyroxenes, massive amphiboles, and talc fibers) which are chemically similar to asbestos and can be considered interferences. The presence of these substances may warrant the use of more powerful diffraction pattern analysis before positive identification can be made. If interferences are suspected, morphology can play an important role in making positive identification.

19. Obtain EDX spectra in either the TEM or STEM modes from fibers on field samples using the procedure of step 18. Using the diffraction pattern and EDX spectrum, classify the fiber:

a. For a chrysotile structure, obtain EDX spectra on the first five fibers and one out of ten thereafter. Label the range profiles from 0-5-10-0-0 to 0-10-10-0-0 as "chrysotile."

b. For an amphibole structure, obtain EDX spectra on the first 10 fibers and one out of ten thereafter. Label profiles ca. 0-2-10-0-7 as "possible amosite"; profiles ca. 1-1-10-0-6 as "possible crocidolite"; profiles ca. 0-4-10-3-<1 as "possible tremolite"; and profiles ca. 0-3-10-0-1 as "possible anthophyllite."

NOTE: The range of profiles for the amphiboles will vary up to ± 1 unit for each of the elements present according to the relative detector efficiency of the spectrometer.

c. For an ambiguous structure, obtain EDX spectra on all fibers. Label profiles similar to the chrysotile profile as "possible chrysotile." Label profiles similar to the various amphiboles as "possible amphiboles." Label all others as "unknown" or "non-asbestos."

20. Counting and Sizing:
    a. Insert the sample grid into the specimen grid holder and scan the grid at zero tilt at low magnification (ca. 300 to 500X). Ensure that the carbon film is intact and unbroken over ca. 75% of the grid openings.
    b. In order to determine how the grids should be sampled, estimate the number of fibers per grid opening during a low-magnification scan (500 to 1000X). This will allow the analyst to cover most of the area of the grids during the fiber count and analysis. Use the following rules when picking grid openings to count [7,8]:
        (1) Light loading (<5 fibers per grid opening): count total of 40 grid openings.
        (2) Moderate loading (5 to 25 fibers per grid opening): count minimum of 40 grid openings or 100 fibers.
        (3) Heavy loading (>25 fibers per opening): count a minimum of 100 fibers and at least 6 grid openings.

        Note that these grid openings should be selected approximately equally among the three grid preparations and as randomly as possible from each grid.
    c. Count only grid openings that have the carbon film intact. At 500 to 1000X magnification, begin counting at one end of the grid and systematically traverse the grid by rows, reversing direction at row ends. Select the number of fields per traverse based on the loading indicated in the initial scan. Count at least 2 field blanks per sample set to document possible contamination of the samples. Count fibers using the following rules:
        (1) Count all particles with diameter greater than 0.25 µm that meet the definition of a fiber (aspect ratio ≥3:1, longer than 5 µm). Use the guideline of counting all fibers that would have been counted under phase contrast light microscopy (Method 7400). Use higher magnification (10000X) to determine fiber dimensions and countability under the acceptance criteria. Analyze a minimum of 10% of the fibers, and at least 3 asbestos fibers, by EDX and SAED to confirm the presence of asbestos. Fibers of similar morphology under high magnification can be identified as asbestos without SAED. Particles which are of questionable morphology should be analyzed by SAED and EDX to aid in identification.
        (2) Count fibers which are partially obscured by the grid as half fibers.
            NOTE: If a fiber is partially obscured by the grid bar at the edge of the field of view, count it as a half fiber only if more than 2.5 µm of fiber is visible.
        (3) Size each fiber as it is counted and record the diameter and length:
            (a) Move the fiber to the center of the screen. Read the length of the fiber directly from the scale on the screen.
                NOTE 1: Data can be recorded directly off the screen in µm and later converted to µm by computer.
                NOTE 2: For fibers which extend beyond the field of view, the fiber must be moved and superimposed upon the scale until its entire length has been measured.
            (b) When a fiber has been sized, return to the lower magnification and continue the traverse of the grid area to the next fiber.
    d. Record the following fiber counts:
        (1) f_s, f_b = number of asbestos fibers in the grid openings analyzed on the sample filter and corresponding field blank, respectively.
        (2) F_s, F_b = number of fibers, regardless of identification, in the grid openings analyzed on the sample filter and corresponding field blank, respectively.

CALCULATIONS:

21. Calculate and report the fraction of optically visible asbestos fibers on the filter, $(f_s - f_b)/(F_s - F_b)$. Apply this fraction to fiber counts obtained by PCM on the same filter or on other filters for which the TEM sample is representative. The final result is an asbestos fiber count. The type of asbestos present should also be reported.
22. As an integral part of the report, give the model and manufacturer of the TEM as well as the model and manufacturer of the EDX system.

EVALUATION OF METHOD:

The TEM method, using the direct count of asbestos fibers, has been shown to have a precision of 0.275 (s_r) in an evaluation of mixed amosite and wollastonite fibers. The estimate of the asbestos fraction, however, had a precision of 0.11 (s_r). When this fraction was applied to the PCM count, the overall precision of the combined analysis was 0.20 [2].

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