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SAMPLING AND QUALITY ASSURANCE PROJECT PLAN
REVISION 1

FOR

Libby, Montana

Environmental Monitoring for Asbestos

Baseline Monitoring for Source Area and Residential Exposure
to
Tremolite-Actinolite
Asbestos Fibers

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DOCUMENT REVISION LOG

Revision	Date	Major Changes
0	12/6/99	--
1	1/4/00	a. Revised text to clarify study design and DQOs b. Added SOP for surface water to allow collection and evaluation of surface water as a transport medium c. Added alternative SOP for asbestos analysis in soil that may have higher sensitivity than other methods. d. Added figures to help illustrate key steps from sample collection to analysis e. Added final SOPs as appendices to the revision.

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A. PROJECT TASK ORGANIZATION

A3 PROJECT MANAGEMENT

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A4 PROBLEM DEFINITION and BACKGROUND

Problem: This sampling plan has been developed in response to requests from the State of Montana, Lincoln County Health Board (meeting minutes, 11/23/99), and City officials of Libby, MT, to address questions and concerns raised by citizens of Libby regarding possible ongoing exposures to asbestos fibers as a result of historical mining, processing and exportation of asbestos-containing vermiculite. Over 60 years of mining, milling, packaging and shipping of vermiculite at the mine and associated properties resulted in the environmental release of asbestos fibers during mining operations (McDonald et al., 1986; Amandus et al.,

1987; Amandus and Wheeler; 1987; Amandus et al., 1978). Since closure of the mine in 1990, it is expected that production-related emissions have been greatly reduced or eliminated. However, there are presently insufficient data to conclude that current exposures to residents in Libby and the surrounding area and occasional recreational visitors to the former mining areas are negligible. ***The purpose of this sampling effort is to acquire information suitable for supporting an exposure and risk assessment for current environmental conditions in Libby.***

Background: Asbestos is a generic term for a group of six naturally-occurring, fibrous silicate minerals that have been widely used in commercial products. Asbestos minerals fall into two groups or classes: serpentine asbestos and amphibole asbestos. Serpentine asbestos, which includes the mineral chrysotile, a magnesium silicate mineral, possesses relatively long and flexible crystalline fibers that are capable of being woven. Amphibole asbestos, which includes the minerals amosite, crocidolite, tremolite, anthophyllite, and actinolite, form crystalline fibers that are substantially more brittle than serpentine asbestos.

Asbestos is of potential health concern because chronic inhalation exposure to excessive levels of asbestos fibers suspended in air can result in lung disease such as asbestosis, mesothelioma, and lung cancer. Figure 1 presents a preliminary Site Conceptual Model which identifies exposure pathways by which asbestos fibers from mining-related sources might become entrained in air in Libby, leading to inhalation exposures of residents or workers. The site conceptual model will be refined as site data are acquired and an improved understanding of actual transport and exposure pathways is achieved.

Approach: This sampling plan describes the efforts planned by EPA to monitor and characterize asbestos-containing materials in and about the vicinity of Libby. The plan will be composed of two phases:

Phase 1: This is a rapid pilot-scale investigation that has two main objectives:

a) Obtain information on airborne asbestos levels in Libby in order to judge whether a time-critical intervention is needed to protect public health.

b) Obtain data on asbestos levels in potential source materials, and identify the most appropriate analytical methods to screen and quantify asbestos in source materials.

Phase 2: This will consist of a systematic evaluation of asbestos levels in air in Libby and in appropriate background locations, along with a systematic investigation to identify the actual or potential source(s) and release mechanism(s) of asbestos in Libby and the surrounding area. The implementation, pace and scope of Phase 2 and the methods used to collect and analyze samples in Phase 2 will be determined in large part by the results of the Phase 1 pilot study.

Interpretation. Analyses of asbestos fibers in air and other site media will determine the potential (or lack of potential) for human inhalation exposure under **present** conditions. The environmental fate and transport of asbestos fibers may be such that present measurement conditions (e.g. weather) and/or measurement techniques interfere with the ability to identify and/or quantify asbestos fibers in relevant exposure media (soil, dust, air, or water). Thus, while conclusions drawn from the implementation of this study are applicable to the present conditions at the site, they do not necessarily reflect conditions which may develop in the future.

A5 PROJECT TASK DESCRIPTION

To the extent possible, sampling will be conducted such that data will be meaningful for human exposure and risk assessment. Because the chief exposure pathway is air, emphasis will be placed on collection of air samples. In addition, to help identify potential sources and transport pathways for asbestos, samples of various bulk materials (mine waste, soil, dust, water, sediment) will also be collected in residential and non-residential areas.

Phase 1

Basic tasks needed to complete Phase 1 are listed below:

1. Collect samples of air, soil, dust, water, and insulation from selected locations in and around town, including a number of residential and/or commercial locations, as well as suspected source areas such as historical mining/processing/loading facilities.

2. Perform asbestos analyses on all air samples and a selected set of the dust, soil, insulation and water samples (those judged to be most likely to have either "high" or "low" concentrations) in order to obtain preliminary information on asbestos levels in air and other media, and to identify the optimum conditions for collection and analysis of bulk media.

At this time, the proposed sampling for Phase 1 consists of collection of environmental media from approximately 30 residences and 3 potential source areas. Residential sample locations will be selected from residences volunteering for multimedia sampling. In addition to the collection of samples within the residential area, samples may also be collected in commercial warehouses, agricultural buildings, or businesses in Libby, as needed to support the objectives of the On Scene Coordinator. Potential source area samples will be collected along the mine road (Rainy Creek Road) and at the Former Vermiculite Loading facility near the intersection of Rainy Creek Road and Highway 37.

Media samples will be collected according to Standard Operating Procedures provided by CDM, Inc. or as provided in the attachments to this Sampling and Quality Assurance Plan.

Phase 2

The purpose of Phase 2 is to design and implement a systematic program of sample collection and analysis to fully characterize levels of health risk from long-term inhalation exposure to asbestos in air, and to identify any actual or potential sources and release mechanisms of asbestos. Specific tasks needed to implement Phase 2 will be selected after completion of Phase 1.

A6 QUALITY OBJECTIVES and CRITERIA for MEASUREMENT DATA

Two types of objectives are identified in this quality assurance project plan (QAPP): general objectives and data quality objectives (DQOs). General objectives are statements of practical goals that, if realized, will substantially contribute to achieving the purpose of the study. Development of DQOs is a process that is intended to ensure that task objectives are clearly defined and that data collected are appropriate and of sufficient quality to satisfy the objectives.

Phase 1 General Objective 1

Determine whether current airborne levels of asbestos in Libby are high enough to warrant a time-critical intervention.

Phase 1 General Objective 2

Obtain preliminary data on asbestos concentrations in potential source materials for air (e.g., dust, soil, mine waste), and determine the optimum conditions for sampling and quantifying asbestos levels in source materials.

Phase 2 General Objective

The general objectives for Phase 2 is to collect reliable and systematic data on asbestos levels in air and other media in Libby to allow a reliable evaluation of current human exposure and health risk from asbestos as well as an identification of sources of unacceptable levels of asbestos in air.

Data Quality Objective Process

The DQO process can be an iterative process which is designed to focus on the decisions that must be made and to help ensure that the site activities that acquire data are logical, scientifically defensible, and cost effective. The DQO process is intended to:

- Ensure that task objectives are clearly defined
- Determine anticipated uses of the data

- Determine what environmental data are necessary to meet these objectives
- Ensure that the data collected are of adequate quantity and quality for the intended use

The three stages of the DQO process are identified below and a discussion of how they have been applied in the characterization study described herein. The three stages are undertaken in an interactive and iterative manner, whereby all the DQO elements are continually reviewed and re-evaluated until there is reasonable assurance that suitable data for decision making will be attained.

- Stage I - Identify Decision Types: Stage I defines the types of decisions that will be made by identifying data uses, evaluating available data, developing a conceptual model, and specifying objectives for the project. The conceptual model facilitates identification of decisions that may be made, the end use of the data collected, and the potential deficiencies in the existing information.

- Stage II - Identify Data Uses/Needs: Stage II stipulates criteria for determining data adequacy. This stage involves specifying the quantity and quality of data necessary to meet the Stage I objectives. EPA's Data Usability for Risk Assessment Guidance (DURA) outlines general and specific recommendations for data adequacy. This includes identification of data uses and data types, and identification of data quality and quantity needs.

- Stage III - Design Data Collection Program: Stage III specifies the methods by which data of acceptable quality and quantity will be obtained to make decisions. This information is provided in the SOP.

Through utilization of the DQO process, as defined in EPA guidance (EPA540-R-93-071 and -078, Sep 1993), this QAPP will use several terms that are specifically defined to avoid confusion that might result from any misunderstanding of their use. For each of the tasks identified within this QAPP, a "Task Objective" is specifically defined. The Task Objective is a concise statement of the problem to be addressed by activities under this task. For each Task Objective, a decision (or series of decisions) is identified which addresses the problem contained in the Task Objective.

For each decision, the data necessary to make the decision are identified and described. For all analytical data, quality assurance objectives are specified that describe the minimum quality of data necessary to support the specified decision or test the hypotheses. These quality assurance objectives are specified as objectives for precision, accuracy, representativeness, comparability, and completeness. In addition, data review and validation procedures are specified in the QAPP that evaluate how well the analytical data meet these quality assurance objectives and whether or not the data are of sufficient quality for the intended usage.

The following sections apply the DQO process to the Libby Project, Stage I and Stage II. Stage III is discussed later (see Section B), but sampling and analysis methods presented in this section are considered tentative and final decisions on optimum sampling and analytical methods will be delayed until the findings of Phase 1 are available.

DQO Stage I - Identifying Decision Types

Stage I of the DQO process identifies a primary question and secondary questions that need to be resolved at the completion of the sampling and analyses program.

- PRIMARY QUESTION (Phase 1): Are current airborne levels of asbestos sufficiently high to warrant a time-critical intervention?
- SECONDARY QUESTION (Phase 1): What are the most likely sources of asbestos in air, and what are the best methods for quantifying asbestos levels in potential source materials?

DQO Stage II - Identifying Data Uses/Needs

Stage II of the DQO process also determines what type and quality of data are needed to answer the questions developed in Stage I. EPA has developed a seven-step method for developing the DQOs. This seven-step method is applied below in order to define the data requirements needed to achieve the primary and secondary objectives of the Phase 1 evaluation (and summarized in Table 1).

Primary Objective: Evaluate The Need For Time-critical Action

1. State the Problem

The problem to be addressed by this study is that citizens of Libby appear to have an increased incidence of asbestos-related disease, but there are no data to determine if this disease is attributable solely to historic exposures, or whether current exposures are of continuing health concern.

2. Identify the Decision

The first decision to be made is whether or not time-critical intervention is needed to protect public health. If current exposures are not high enough to warrant time-critical intervention, the next decision is whether or not non-time-critical remedial action is needed.

3. Identify Inputs to the Decision

Decisions on the need for time-critical intervention or non-time-critical remediation will be based on estimated risk of lung disease in current residents and workers in Libby. Two types of lung disease are of concern: asbestosis (a non-cancer effect) and lung cancer and mesothelioma (cancer effects). Limited data suggest that chronic exposures to chrysotile fiber levels of 5-20 f/mL can cause asbestotic changes (ATSDR 1999), but data are not sufficient to derive a reliable chronic MRL or RfC for asbestosis. However, methods have been established for estimating the excess risk of lung cancer and/or mesothelioma, and it is considered likely that exposure levels that protect against unacceptable risk of lung cancer/mesothelioma (in the range of 0.1 to 0.0001 f/mL; see below) will also protect against unacceptable risk of asbestosis.

The basic equation used to estimate cancer risk is:

Risk = Concentration (f/mL) * Unit Risk (risk per f/mL)

Thus, the data needs are an **estimate of airborne asbestos concentration** and an **estimate of cancer risk per unit concentration.**

Measurement of Asbestos Concentration in Air

There are a number of techniques for measuring asbestos fibers in air, all of which are based on visual identification of structures as asbestos fibers. Most historical human health data and many regulatory limits for asbestos exposure in air are based upon asbestos fiber concentrations measured using phase contrast microscopy (PCM) (see Table 2). In this method, **fiber material** is defined as having a length >5 microns and an aspect ratio (length to diameter ratio) of three or more. Results are generally reported as fibers per milliliter of air (f/mL).

More recently, a number of other methods have been developed for quantitative or qualitative measurement of asbestos fibers in air, including transmission electron microscopy (TEM), and x-ray diffraction (XRD). These methods are generally more sensitive than PCM, and also allow visualization and quantification of asbestos fibers that are thinner than those visible under PCM. This is important because it is likely that the toxicity of long thin fibers is greater than that of shorter thicker fibers (Berman et al., 1995). Based on this, **asbestos fibers in air will be quantified by TEM.** Detailed rules for identifying asbestos fibers of biological concern by TEM are provided in ISO method 10312. This method is an international standard procedure that is recommended for quantifying asbestos fibers that are believed to be the chief source of human health concern (Berman and Crump 1999).

Unit Risk for Asbestos in Air

It is mandatory that the unit risk value used to calculate cancer risk be based on the same type of asbestos measurement technique as used to quantify asbestos concentration in air. That is, it is not correct to estimate risk by multiplying a concentration based on TEM fibers per mL by a unit risk based on PCM fibers per mL. Thus, risk-based values shown in Table 2 cannot be used to interpret measurements based on TEM. EPA has developed a model for predicting risk from mesothelioma and lung cancer from TEM-based measurements of asbestos in air (USEPA 1986), and this method has been revised and improved by Berman and Crump (1999) to incorporate the influence of fiber length. The risk factors for the modified mesothelioma and lung cancer model are summarized in Table 3. Note that the risk factor depends not only on the number of TEM fibers greater than 5 um in length, but also on the fraction of all fibers that are longer than 10 um.

The toxicity factors shown in Table 3 are based on the best data currently available, but it is important to recognize that these toxicity factors are uncertain. This is because the values are derived from studies in which important details of exposure (level, duration, fiber size distribution, etc.) are not always known. In particular, the importance of fiber size (length, thickness) and fiber type (tremolite, chrysotile, etc.) on toxicity is difficult to quantify and remains a source of discussion.

4. Define the Study Boundaries

Spatial Bounds

The spatial bounds to be investigated in this project include the community of Libby, and areas associated with former mining activities near the town. Appropriate background areas may be selected for comparative evaluation.

Temporal Bounds

Asbestos fibers enter air mainly as a result of resuspension due to mechanical disturbance or wind erosion. Because mechanical and wind forces may vary substantially over time, asbestos levels in air are also expected to vary substantially over time. Thus, estimates of long term average concentrations are inherently preferable to measurements based on grab samples. Therefore, multiple samples of air will be collected over time at locations of interest. It is likely the highest levels will tend to occur in summer, when source areas tend to be dry and wind and mechanical forces result in significant dust resuspension.

5. Develop a Decision Rule

EPA must identify an actual or potential threat to human health or the environment in order to initiate a time-critical intervention at a site. Based on current EPA guidelines, a lifetime excess cancer risk of 1E-04 is considered to be at the upper end of the acceptable risk

range for chronic (lifetime) exposure. Based on this, this Phase 1 study will use an excess cancer risk of about 1E-03 as the appropriate boundary for decision-making. That is, if asbestos levels in air correspond to an estimated cancer risk of about 1E-03 or higher, time critical actions to identify sources and find appropriate and effective interventions will be considered. If estimated cancer risks from asbestos in Phase 1 air samples do not exceed a level of about 1E-03, then further studies may be pursued to determine if risk levels might exceed 1E-03 at other times or in other places, or if risks might exceed an acceptable chronic risk level (e.g., 1E-04).

6. Specify Limits on Decision Errors

The null hypothesis that will be tested in Phase 1 is that indoor air levels in Libby are sufficiently high to warrant time-critical intervention. Two types of decision error are possible when making this decision:

Type I Error: Rejecting the null hypothesis when it really is true. That is, the site is declared to be below a risk level of 1E-03 when it is really above this level.

Type II Error: Accepting the null hypothesis when it actually is false. That is, the site is declared to be of time-critical concern when it actually is not.

The limits on these two types of errors are risk management judgements. In order to minimize the chances of a Type 1 error (a "false negative"), the decision will be based on the highest concentration of asbestos fibers detected in any currently-occupied residential or occupational building evaluated in the Phase 1 investigation. If one or more samples exceeds the 1E-03 risk level, time critical action may be needed. However, additional samples may be collected to confirm the original measurement and to refine the risk estimate. Because of the time variability in asbestos levels in air, final decisions may be delayed until additional data have been collected, including data in the summer when airborne resuspension and transport of asbestos fibers in outdoor air is considered to be more likely than in winter.

7. Optimize the Design for Obtaining Results

Additional indoor and/or outdoor air samples may be collected and incorporated into either Phase 1 and/or Phase 2 as data become available on actual airborne exposure and risk levels.

Secondary Objective: Preliminary Investigation of Source Materials

Table 4 provides a summary of the seven-step DQO process for achieving the secondary objective. The following text describes each of the DQO steps in detail.

1. State the Problem

The problem to be addressed by this portion of the study is that most methods currently available for measuring asbestos in solid materials (e.g., soil, dust, bulk insulation, mine waste, etc.) are relatively insensitive, and it is not known whether impacts of historic or ongoing asbestos releases on these media can be detected by these techniques.

2. Identify the Decision

The decision to be made is whether analysis of potential source materials and/or transport media in and about the mine (e.g., mine waste, surface water) and in and about the community of Libby (e.g., yard soil, house dust, garden soil) can be reliably quantified using available techniques. If so, then source areas judged to be of potential concern may be removed at the discretion of the OSC. Alternatively, additional sampling and analysis of potential source material may be pursed as needed to identify impacted areas and to focus on sources of unacceptable asbestos levels in air.

3. Identify Inputs to the Decision

Asbestos Measurements in Environmental Media

Inputs to the decision will be the results of asbestos analyses of each medium using the best available technique(s), as follows:

Medium	Proposed Method	
	Sample Preparation	**Sample Analysis**
Yard soil Garden soil Road soil Mine waste Bulk insulation	Collect bulk sample, place on slide	PLM of bulk material
	Collect bulk sample, dry	Visible reflective infrared spectroscopy
	Separate respirable dust fraction using Superfund method, collect dust on filter, collapse filter, prepare TEM grids	TEM of respirable dust
Indoor Dust	Microvacuum into cassette, suspend dust in water/alcohol, collect on filter, dry ash, prepare TEM grids	TEM
Surface Water	Collect bulk sample, filter, collapse filter, prepare TEM grids	TEM

These methods have been selected because they are judged to be the most likely to yield results that will allow qualitative or quantitative evaluation of asbestos levels in

environmental media. Note that several alternative methods are identified for soil and related bulk materials. At present, it is not known which of these will be the most appropriate. It is envisioned that all samples will be screened using visible infrared spectroscopy, since this method is very fast and inexpensive. If successful, the results of this method can be used to rank-order samples into "high", "medium" and "low" concentration ranges. For quantitative assessment, it is envisioned that all samples will be analyzed by PLM, since this method is fast and relatively less expensive than the Superfund TEM method. This evaluation will begin with samples that are known or suspected to be high in asbestos concentration, based either on the infrared results and/or field observations such as the presence of visible levels of vermiculite, proximity to known sources or waste materials, etc. The analyses will continue through the samples to those that are known or suspected to contain "low" levels. When asbestos fiber concentrations are consistently below the detection limit, further analyses by PLM may be discontinued. After the results of the infrared and the PLM analyses are available, a set of samples will be selected for analysis by the Superfund method. This method is expected to be the most sensitive, because it includes a preliminary separation of respirable asbestos fibers from the bulk material, and because quantification is by TEM rather than PLM. However, the method is not yet in wide use, and is associated with a relatively high cost and slow turnaround time. It is for this reason that only about 15-21 samples will be evaluated by this approach. This set will be composed of approximately 5-7 in each of three categories: "high", "medium", and "low". Comparison of results across these three methods will allow an evaluation of which method(s) is (are) most appropriate for on-going evaluation of soils and related materials at the site.

For the other media (dust, surface water), all samples collected will be analyzed by the analytical methods indicated above. A comparison of results across samples will be used to determine whether the method is likely to be reliable and useful for further evaluation of site samples.

Community Interview

EPA will administer a community interview to numerous Libby residents including residents of each household sampled. These interviews will help gauge community members' level awareness about asbestos, their health concerns about asbestos, their knowledge about activities that may results in asbestos exposure, as well as possible sources of asbestos-bearing material. This information may help explain observed asbestos levels in samples from the home. A copy of the interview questionnaire is provided in Section E (Appendices).

4. Define the Study Boundaries

Spatial Bounds

The spatial bounds to be investigated in this project include the community of Libby, and areas associated with former mining activities near the town.

Temporal Bounds

Asbestos levels in source or transport material are expected to be relatively stable. Thus, the time when source area samples are collected is not judged to be critical.

5. Develop a Decision Rule

If no observable difference in asbestos concentration can be detected between the two classes of samples ("high" vs "low"), it will be concluded that a) either the medium is not impacted, or b) the measurement technique is not sufficiently sensitive. If a difference can be detected, it will be concluded that there is an impact to that medium, along with an actual or potential release to the environment, and that the current method can be used to further investigate and quantify that release.

6. Specify Limits on Decision Errors

Because the decision to be made is mainly with regard to method adequacy, no quantitative rules are needed to define decision errors.

7. Optimize the Design for Obtaining Results

Additional source area samples may be collected and incorporated into either Phase 1 and/or Phase 2 as data become available on the ability of current methods to detect and quantify asbestos fibers in each medium.

PARCC Requirements

Within this QAPP, quantitative and qualitative limits are defined for precision, accuracy, representativeness, comparability and analytical completeness. Reporting limits for asbestos fibers are set by the analytical laboratory based on environmental matrix, historical data, and comparison to EPA limits for CLP and other methods. Quantitative limits are also defined by microscopy (light microscopy or TEM) for method detection limits, and for method reporting limits or method quantitation limits. The QA procedures outlined in this section are intended to ensure data quality and to administer corrective actions with the goal of producing data that satisfy the following requirements. General guidelines, policies, and procedures to achieve these objectives are presented below. Where additional, detailed, procedures are required to attain QA objectives and to describe specific methods, these are provided in the SOPs (see attached). The following PARCC requirements apply to more standard chemical analytical analyses, and partially to asbestos analyses (e.g., identifying physico-chemical make-up of specific fibers)

Precision: Precision is defined as the agreement between a set of replicate measurements without assumption or knowledge of the true value. It is a measure of agreement among

individual measurements of the same property under prescribed similar conditions. Agreement is expressed as either the relative percent difference (RPD) for duplicate measurements or the range and standard deviation for larger numbers of replicates. The RPD will be reported on required 5% laboratory duplicates.

Accuracy: Accuracy is a measure of the closeness of individual measurements to the "true" value. Accuracy usually is expressed as a percentage of that value. For a variety of analytical procedures, standard reference materials traceable to or available from National Institute of Standards and Technology (NIST, formerly National Bureau of Standards) or other sources can be used to determine accuracy of measurements. Accuracy will be measured as the percent recovery (%R) of an analyte in a reference standard or spiked samples (>3 at each selected concentration range) that span the limit of linearity for the method.

Ideally, precision and accuracy estimates should represent the entire measurement process, including sampling, analysis, calibration, and other components. From a practical perspective, these estimates usually represent only a portion of the measurement process that occurs in the analytical lab.

Representativeness: Representativeness is the degree to which data accurately and precisely represent characteristics of a population, parameter variations at a sampling point, or an environmental condition. For this QAPP, data and samples representative of chemical and biological exposures in the study and reference areas are to be collected from randomly chosen residences.

Comparability: Data are comparable if site considerations, collection techniques, and measurement procedures, methods, and reporting are equivalent for the samples within a sample set. A qualitative assessment of data comparability will be made of applicable data sets. These criteria allow comparison of data from different sources. Comparable data will be obtained by specifying standard units for physical measurements and standard procedures for sample collection, processing, and analysis. Please see the attached SOPs for sampling and analysis procedures.

Completeness: Data are considered complete when a prescribed percentage of the total intended measurements and samples are obtained. Analytical completeness is defined as the percentage of valid analytical results requested, and >90% of analyzed samples should have results reported. For this sampling program, a minimum of 80 percent of the planned collection of individual samples for quantification and a minimum of 30 percent of related parameters (e.g., physical measurements, fiber type, etc.) must be obtained to achieve a satisfactory level of data completeness.

Method Detection Limits (applicable to chemical analyses only): Method detection limits (MDLs) are minimum values that can be reliably measured to identify the analyte as being present in the matrix, versus method quantitation limits are the minimum values that can

be quantitated with reasonable scientific confidence. The method will also have a maximum linear value in most situations, and analyses should occur within this limit of linearity range. See applicable operating procedures for details.

Table 1. DQOs for Primary Objective: Evaluate the Need for Time-Critical Action

DQO Step	Description
1. Define the problem	The citizens of Libby appear to have an increased incidence of asbestos-related disease, but there are no data to determine if this disease is attributable solely to historic exposures, or whether current exposures are of continuing health concern.
2. Identify the decision	Is time-critical action needed to protect public health? If yes, identify appropriate action and intervene as necessary If no, determine whether or not non-time-critical remediation is necessary
3. Identify inputs to decision	Level of concern for human health (lifetime excess cancer risk of 1E-03) Estimate of airborne asbestos concentration, and cancer risk per unit concentration.
4. Define study boundaries	*Spatial bounds*: Community of Libby, including former mining, milling and processing areas and areas potentially impacted as defined by meteorological conditions. If necessary, appropriate background areas are also included (precise locations to be defined). *Temporal bounds*: multiple air samples will be collected in areas associated with former mining activities near the town seasonally throughout the year
5. Define decision rule	If asbestos levels in indoor air $\geq$ 1E-03 risk level, consider the need for time-critical intervention. If asbestos levels in indoor air < 1E-03 risk level, time-critical intervention may not be necessary. However, additional studies may be needed to determine if non-time-critical remediation is necessary, or if levels might exceed 1E-03 risk levels under different conditions (e.g., seasonal variation)
6. Specify limits on decision errors	Risk management decisions will be based on the highest airborne asbestos concentration found in any residential or occupational building.
7. Optimize the design	Incorporate new information as data become available on actual airborne exposure and risk levels.

Table 2: Summary of Available PCM-Based Exposure Levels for Asbestos

Agency	Description	Nominal Value	Reference
ACGIH	TLV-TWA	0.1 f/cc	ACGIH, 1998
NIOSH	REL 100 minute TWA in a 400L sample (all forms)	0.1 f/cc	NIOSH 1999
OSHA	PEL (TWA) all forms	0.1 f/cc	OSHA 1998 29 CFR 1919.1001
OSHA	PEL (ceiling) 30 minute average - all forms	1.0 f/cc	OSHA 1998 29 CFR 1926.1101
EPA (IRIS)	Inhalation unit risk - all forms	0.23 per (f/mL)	IRIS 1999
EPA (OW)	MCL (f>10 um in length) all forms	7 MFL[a]	EPA 1998

[a] MFL = million fibers per liter

TABLE 3. Unit Risk for Inhalation of Asbestos

Population	Percentage of Fibers Greater than 10 um in Length										
	0.50%	1%	2%	4%	6%	10%	15%	20%	30%	40%	50%
Male Nonsmoker											
Lung Cancer	1.0E-02	1.6E-02	3.0E-02	5.4E-02	8.0E-02	1.3E-01	1.9E-01	2.6E-01	3.8E-01	5.0E-01	6.4E-01
Mesotheliomas	1.1E-01	1.9E-01	3.2E-01	6.2E-01	9.0E-01	1.5E+00	2.2E+00	2.9E+00	4.3E+00	5.8E+00	7.2E+00
Total	1.2E-01	2.0E-01	3.5E-01	6.7E-01	9.8E-01	1.6E+00	2.4E+00	3.2E+00	4.7E+00	6.3E+00	7.8E+00
Female Nonsmoker											
Lung Cancer	7.6E-03	1.2E-02	2.2E-02	4.0E-02	6.0E-02	9.6E-02	1.4E-01	1.9E-01	2.8E-01	3.8E-01	4.8E-01
Mesotheliomas	1.3E-01	2.0E-01	3.6E-01	6.8E-01	1.0E+00	1.7E+00	2.5E+00	3.3E+00	4.9E+00	6.5E+00	8.1E+00
Total	1.4E-01	2.1E-01	3.8E-01	7.2E-01	1.1E+00	1.8E+00	2.6E+00	3.5E+00	5.1E+00	6.8E+00	8.5E+00
Mean Total for Nonsmokers	2.6E-01	4.1E-01	7.3E-01	1.4E+00	2.0E+00	3.4E+00	5.0E+00	6.6E+00	9.8E+00	1.3E+01	1.6E+01
Male Smoker											
Lung Cancer	9.4E-02	1.5E-01	2.6E-01	5.0E-01	7.4E-01	1.2E+00	1.8E+00	2.4E+00	3.5E+00	4.7E+00	5.9E+00
Mesotheliomas	7.6E-02	1.2E-01	2.2E-01	4.2E-01	6.0E-01	9.8E-01	1.5E+00	1.9E+00	2.9E+00	3.8E+00	4.8E+00
Total	1.7E-01	2.8E-01	4.8E-01	9.2E-01	1.3E+00	2.2E+00	3.2E+00	4.3E+00	6.4E+00	8.5E+00	1.1E+01
Female Smoker											
Lung Cancer	6.4E-02	1.0E-01	1.8E-01	3.4E-01	5.0E-01	8.2E-01	1.2E+00	1.6E+00	2.4E+00	3.2E+00	4.0E+00
Mesotheliomas	1.1E-01	1.9E-01	3.2E-01	6.2E-01	9.0E-01	1.5E+00	2.2E+00	2.9E+00	4.3E+00	5.8E+00	7.2E+00
Total	1.8E-01	2.9E-01	5.0E-01	9.6E-01	1.4E+00	2.3E+00	3.4E+00	4.5E+00	6.7E+00	9.0E+00	1.1E+01
Mean Total for Smokers	1.7E-01	2.8E-01	4.9E-01	9.4E-01	1.4E+00	2.2E+00	3.3E+00	4.4E+00	6.6E+00	8.8E+00	1.1E+01

Source: Berman and Crump (1999)

Table 4. DQOs for Secondary Objective: Preliminary Investigation of Source Materials

DQO Step	Description
1. Define the problem	Optimum sampling and analysis techniques for potential source media (soil, dust, mine waste, etc) are not known and available techniques may or may not be able to detect and quantify impacts of asbestos releases
2. Identify the decision	Can asbestos concentrations be reliably quantified using available methods? If yes, source areas may be characterized and removed at the discretion of the OSC If no, additional methods development may be required
3. Identify inputs to decision	Asbestos measurements from selected samples that are expected to have "high" and "low" asbestos levels. Compare results to determine if available methods are sufficiently sensitive to detect and measure differences in potential source materials and/or transport media. Survey information on historic and current factors that may help explain current patterns of asbestos contamination
4. Define study boundaries	*Spatial bounds*: Community of Libby and areas associated with former mining activities near the town *Temporal bounds*: because asbestos levels in source or transport material are relatively stable, the time when these samples are collected is not critical
5. Develop decision rule	If no difference is observed between the two classes of samples, the following conclusions will be drawn: a) the medium is not impacted, or b) the measurement technique is not sufficiently sensitive If a difference between the two classes of samples **can** be detected, the following conclusions will be drawn: a) the medium is impacted b) the method can be used to further investigate and quantify that impact
6. Specify limits on decision errors	NA
7. Optimize the design	Additional source area samples may be collected and incorporated as data becomes available on the ability of current methods to detect and quantify asbestos fibers in each medium

B. MEASUREMENT AND DATA ACQUISITION

B1 SAMPLING PROCESS DESIGN

Study Area: For this investigation, the study area is Libby, MT. Initial sampling will be based upon identification of community volunteers. Initial sampling will consist of two basic parts : 1) sampling in residential areas, including the collection of samples at approximately 30 volunteer residences in Libby, along with samples from appropriate background residential areas not influenced by vermiculite mining, and 2) sampling at suspected asbestos source areas and/or transport pathways. All field samples will be accompanied by appropriate QA/QC (5-20%) samples. If possible, future sampling will be based upon stratified random sampling design to select homes in Libby and appropriate reference areas. Additional samples will be targeted at reference areas. Within each area, homes will be selected semi-randomly and on a volunteer basis and access requested from owners. Sampling will be conducted to improve an understanding of actual or potential exposure pathways as indicated in the Conceptual Site Model (Figure 1).

Sample size and characteristics: For this study, approximately 30 residences will be targeted for sampling. In addition to sampling in these residences, samples may be collected as requested by City or State Officials in public buildings, schools, and/or hospitals. Samples collected at residential locations will usually include indoor air, indoor dust and outdoor soil. Samples collected at suspected source and/or transport media may include mine waste, outdoor soil, surface water, sediment and outdoor air, as judged appropriate by the OSC.

B2 SAMPLING METHODS REQUIREMENTS

Air

Air samples will be collected by drawing air through a cellulose acetate filter (0.45 um pore size) at a specified flow rate for a specified period of time. The details of the method are provided in SOP 2015. The optimal conditions for sample collection depend on the level of health risk that is of concern, the level of asbestos in the air, and the level of interfering particles in air besides asbestos. Presented below is a set of alternative flow conditions that illustrate the range of sampling and analysis conditions that may be needed to achieve differing levels of sensitivity.

Alternative Air Sampling and Analysis Methods

Target Risk	Assumed Percent Fibers > 10 um in Length	Concentration of Concern (f/mL) (a)	Suggested Sampling and Analysis Parameters (b)			
			Grid Openings	V (L)	Flow (L/min)	Time (hrs)
1.0E-02	1%	3.5E-02	4	900	1	15
	10%	4.5E-03	8	3800	3	21
1.0E-03	1%	3.5E-03	10	4000	10	7
	10%	4.5E-04	40	8000	10	13
1.0E-04	1%	3.5E-04	40	8500	10	14
	10%	4.5E-05	60	50000	16	52

(a) Based on average unit risk to smokers (see Table 3). As noted in the text, quantitative toxicity values for asbestos-induced cancer are uncertain, and so the concentration values listed should not be viewed as discrete boundaries between "acceptable" and "unacceptable".

(b) Grid openings and air volume required to quantify asbestos at approximately 1/3 the target risk level.

The collection conditions in the shaded row correspond to the conditions that were selected for initial samples collected in Phase 1. Depending on the ratio of long fibers to total fibers, and on the level of interfering non-asbestos dust, these samples may have a risk detection limit of about 3E-04, sufficiently low to assess the potential need for time critical action.

Soil

Bulk samples (mine waste, yard soil, garden soil, road material, insulation, etc.) will be collected, stored under chain-of-custody, and shipped according to methods outlined in Standard Operating Procedures supplied by CDM, Inc., including SOP 4-1 (Field Logbook Content and Contro), SOP 1-3 (Surface Soil Sampling), SOP 1-2 (Sample Custody), SOP 2-5 (Packaging and Shipping of Environmental Samples), SOP 4-2 (Photographic Documentation of Field Activities), and the Surface/Subsurface Soil Sampling Log (Attached).

Dust

Dust sampling will be conducted using method D 5755 - 95 (structure, number count) provided by the American Society for Testing and Materials (ASTM) (Attached).

Surface Water

Surface water is unlikely to be a source of airborne asbestos fibers, but can serve as a transport pathway. Samples of surface water will be collected according to SOP 2013.

Field QA/QC Samples

The QA/QC samples will consist of field blanks (air and dust only), duplicate samples, background samples collected in areas expected to be unaffected by vermiculite mining. Every reasonable effort will be made to adhere strictly to specified SOPs. Where deviation from SOPs is unavoidable, documentation of the deviation and its potential impact on the outcome of the data collection effort will be clearly indicated in field notes and subsequent reports. Detailed field notes will record information pertinent to each sample collection. These field notes will be indexed and made available for review following sample collection.

B3 SAMPLING, HANDLING AND CUSTODY REQUIREMENT

Documentation of sample collection, handling, and shipment will include completion of chain-of-custody forms in the field, use of field maps and field forms, and entry of data into a field logbook. Each sample will be properly labeled with the a unique sample identifier. A chain-of-custody form shall accompany every shipment of samples to the analytical laboratory. The purpose of the chain-of-custody form is to establish the documentation necessary to trace possession from the time of collection to final disposal. Figures 2 to 5 summarize the sampling, handling, and analyzes for each media type.

The chain-of-custody will be designated by CDM SOP 1-2. Minimally the field form will have the following information:

- Project number
- Sampler's signature
- Date and time of sample collection
- Sample identification number
- Analytical parameters

The shipping forms or transmittal memo from EPA will describe:

- Number of containers
- Sample preservative (N/A)
- Date and time of sample shipments

The labs will enter the following information upon receipt:

- Name of person receiving the sample
- Date of sample receipt
- Sample condition

All corrections to the chain-of-custody record will be initialed and dated by the person making the corrections. Each chain-of-custody form will include signatures of the appropriate

individuals indicated on the form. The originals will accompany the samples to the laboratory, and copies documenting each custody change will be recorded and kept on file.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results by EPA. One copy of the chain-of-custody will be kept by field personnel.

The microscopist will include the following information on the Field Form:

- Date
- Microscopist's name
- Sample identification
- Mineral Type
- Structure Counts

All required paper work, including sample container labels, chain-of-custody forms, custody seals and shipping forms will be fully completed in ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping from sample storage to laboratory will be by overnight delivery.

Upon receipt, the samples will be given to the laboratory sample custodian. The coolers will be opened and the contents inspected. Chain-of custody forms will be reviewed for completeness, and samples will be logged and assigned a unique laboratory sample number. Any discrepancies or abnormalities in samples will be noted. The EPA On Scene Coordinator will maintain original log books and receive all data packages and reports.

B4 ANALYTICAL METHODS REQUIREMENTS

The most appropriate analytical methods for each environmental medium may depend on the type and level of asbestos contamination and on the detection levels needed to assess hazard and/or nature and extent of contamination. For these reasons, the final choice of method for each medium cannot be specified at this time. A number of methods that are considered reliable and that may be utilized in this investigation are provided as SOPs in Section E (Appendices).

In most of these methods, two alternative approaches are available: "direct" and "indirect" analysis. In direct analysis, the sample is prepared with minimal handling, generally by collecting the sample on a filter or by placing the test material directly under the microscope for examination. However, this approach may sometimes be inadequate because a) the fibers are accompanied by an excessive level of non-asbestos material, or b) the concentration of asbestos fibers is either too low or too high for reliable quantification. In these cases, an indirect approach will be considered. In this approach, the sample is generally diluted, concentrated, and/or treated to remove interferences, such that the asbestos fibers can be

more reliably quantified. However, because indirect preparation steps may alter fiber morphology and/or may alter fiber recovery, indirect sampling will be avoided whenever possible.

B5 QUALITY CONTROL REQUIREMENT

The project team organization ensures attainment of QA objectives by:

- Assigning responsibility for performing work according to specifications
- Providing oversight of quality-related activities for verification of conformance with specifications
- Defining the relationships between management and personnel performing quality-related work Corrective Action

The Project Manager will prepare a summary of quality-related activities and problems. This summary will be forwarded to EPA for inclusion in the project file. If deficiencies in the program are identified, the Project Manager will identify recommendations for corrective action.

Communications. Lines of communication between project personnel and project management staff will be appropriate to enable timely response to events that have the potential to affect data quality. Project personnel will be provided with a project contact list that includes telephone numbers for both routine communications and emergency notifications.

Communications will also entail ensuring that information on sample collection, transportation, analysis, and storage; data acquisition, analysis, and reporting; personnel assignments and activities; and other information pertinent to the project are distributed to potentially affected personnel in a timely manner. Changes in procedures, equipment, personnel, or other program elements as a result of an accident or emergency that have the potential to affect data quality or achievement of overall program objectives will be communicated to the Project Manager in writing in a timely manner. Copies of all written communications and written summaries of all substantive telephone conversations will be placed in a permanent project file maintained by the EPA On Scene Coordinator.

Quality Control Methods. Quality control methods will include both a field and laboratory component. Field personnel will prepare two types of quality control samples: replicates and blanks.

Replicates - For air samples, replicates are defined as separate samples that are collected using separate air pumps and filters. These air samples are collected side-by-side at a location and are sampled for the same amount of time. Air pumps are set at the same air

flow rates so that adequate and like air volumes are passed through each filter. Replicate samples will not be collected for any media other than air.

Blanks - Field personal will prepare blank samples for air and dust by labeling un-used filter cassettes and submitting them for analysis.

The laboratory and its staff will have the responsibility for processing all samples submitted according to the specific protocols for sample custody, analysis, reporting, and associated laboratory QA/QC. Laboratory personal will prepare blanks TEM grids from the same lot of filters used to prepare water and soil samples.

Quality assurance programs for analytical chemistry typically include blanks, blind standards, and spikes. Unfortunately, this type of performance evaluation program is not available for asbestos analysis. The laboratory quality assurance program will consist of blanks and replicate analysis. Blank samples will be used to control for possible contamination in the filter medium. A subset of TEM grids (10-20%) will be sent either back to the primary laboratory for re-analysis (blind) or to additional laboratories for replicate analysis. The TEM grids for replicate analysis will be shipped under chain of custody to appropriate laboratories for analysis.

B6 INSTRUMENT CALIBRATION and FREQUENCY

Pumps used for air sampling will be calibrated just prior to sampling and again at the termination of sampling to determine air flow rates across the sampling.

SOPs will identify requirements needed to be met by the laboratories to meet adequate instrument calibration frequency, and QA/QC for raw data and reports.

C. ASSESSMENT OVERSIGHT

C1 ASSESSMENTS and RESPONSE ACTIONS

The EPA On Scene Coordinator and/or Scientific Support Coordinator will be on-site to oversee and inspect sampling activities. Enough sample will be taken and archived to allow for problems (such as loss or spoilage) from transportation or analytical labs.

D. DATA VALIDATION and USABILITY

D1 DATA REVIEW, VALIDATION and VERIFICATION REQUIREMENTS

Data validation will consist of a) establishing an absolute range, acceptance limits (screening criteria), and appropriate statistics for each data parameter, b) describing methods for determining the disposition of suspect data, and c) documenting final disposition of invalid or qualified data, including outliers.

Test Statistic: Quantitative professional judgement will be used to determine fiber counts in sampled media. Toxigenic hazard will be assessed based upon professional identification of asbestos fibers. Toxigenic potential of specific fibers will be determined by thorough review of the peer reviewed literature as well as be professional judgement of qualified toxicologists, physicians, and or industrial hygienists.

If feasible, based upon sampling success, a **one-tailed t-test** will be used to compare the two groups (test area and background or reference area) for appropriate classes of asbestos fibers and/or fiber sizes identified (a two-tailed t-test is not used since any change in fiber concentrations is expected to be one direction above background levels as per EPA Risk Assessment Guidance). If there is statistical probability of (e.g. $\alpha \leq 0.05$) for Libby residences being higher than reference areas, then reject the null hypothesis and conclude that significant difference exists between the two groups for a particular fiber type. Therefore, potential exposure of humans to this species would not be able to be screened out. Conversely, if (e.g.) $\alpha \geq 0.05$ for all appropriate fiber sizes and fiber types in a specific media that is reasonably well defined and comparable with similar media in the test area (i.e., no "hot-spots"), then the null hypothesis is accepted and exposure via this route is able to be screened out with no further evaluation being justifiable.

Out-of-range data (fibers outside of the size range normally considered to be toxigenic) will be excluded from the validated data set unless the appropriate data value and relevant toxicological significance can be positively established and documented. Other suspect data or samples will be examined in detail, including any irregularities in its collection and handling. In the absence of any clear indication that they are invalid (such as equipment failure or operator error), data outliers will remain in the validated data set but will be flagged as outliers per specified criteria (e.g., >3 x SD from the mean). Data points determined to be invalid will be permanently flagged in a clear and consistent manner in the original raw data set and removed from subsequent data summaries and files.

QA for data validation will ensure that the screening criteria are comprehensive, unambiguous, reasonable, and internally consistent; and that data validation activities are properly documented. Data discrepancy reports should be prepared describing any data problems observed and any data correction activities undertaken.

Data analysis will consist of analyzing valid data sets from residential results to provide information in formats appropriate to the objectives of the exposure screening study. All data records should be cataloged and stored in their original form. Calibration adjustments and adjustments to reduce data to standard conditions for comparability will be clearly documented, and raw data clearly distinguished from "corrected" data (i.e., data to which calibration and standardization adjustments have been applied).

Raw data and adjustments should be entered into a computer database and/or spreadsheet for correction, statistical analysis, manipulation, formatting, and summarizing to reduce the potential for human error. EPA requests that all data be placed into the designated data entry sheets.

D2 VALIDATION and VERIFICATION METHODS

Data reporting consists of communicating summarized data in a final form. Quality assurance for reporting consists of measures intended to avoid or detect human error and to correct identified errors. Such methods include specification of standard reporting formats and contents of measures to reduce data transcription errors. Data will undergo peer review by qualified reviewers capable of evaluating reasonableness of the data for the scientific design.

Reports: A report of all the summary study design characteristics, sample collections and analyses, data quality and results shall be presented by the analytical laboratories. Simple statistical tests of group treatment differences should be performed and presented as discussed above and will be conducted by EPA. All raw data and summary results of both data and summary statistics (means, standard deviations, ranges, etc.) should be tabulated by the laboratories. Results should be interpreted to quantitatively estimate the relative frequency of occurrence of specific asbestos fibers above reference levels. Study reports should be available within 60 days of receipt of acceptable laboratory results and reports.

Data will be reviewed by the On Scene Coordinator, EPA Science Support Coordinator and State officials, and by a peer review team to assess data quality in accordance with DURA (1992) for this CERCLA Emergency Response site.

Quality assurance records and project files will be maintained in accordance with standard project procedures. All QA records, logbooks, sample data forms, raw data summaries, and the like will be maintained until written directions for their disposal are provided.

D3 RECONCILIATION with DQOs

The project team will review any results which fall outside the DQOs and decide (per DURA 1992 and RAGS 1992) the extent of useability of results for risk assessment.

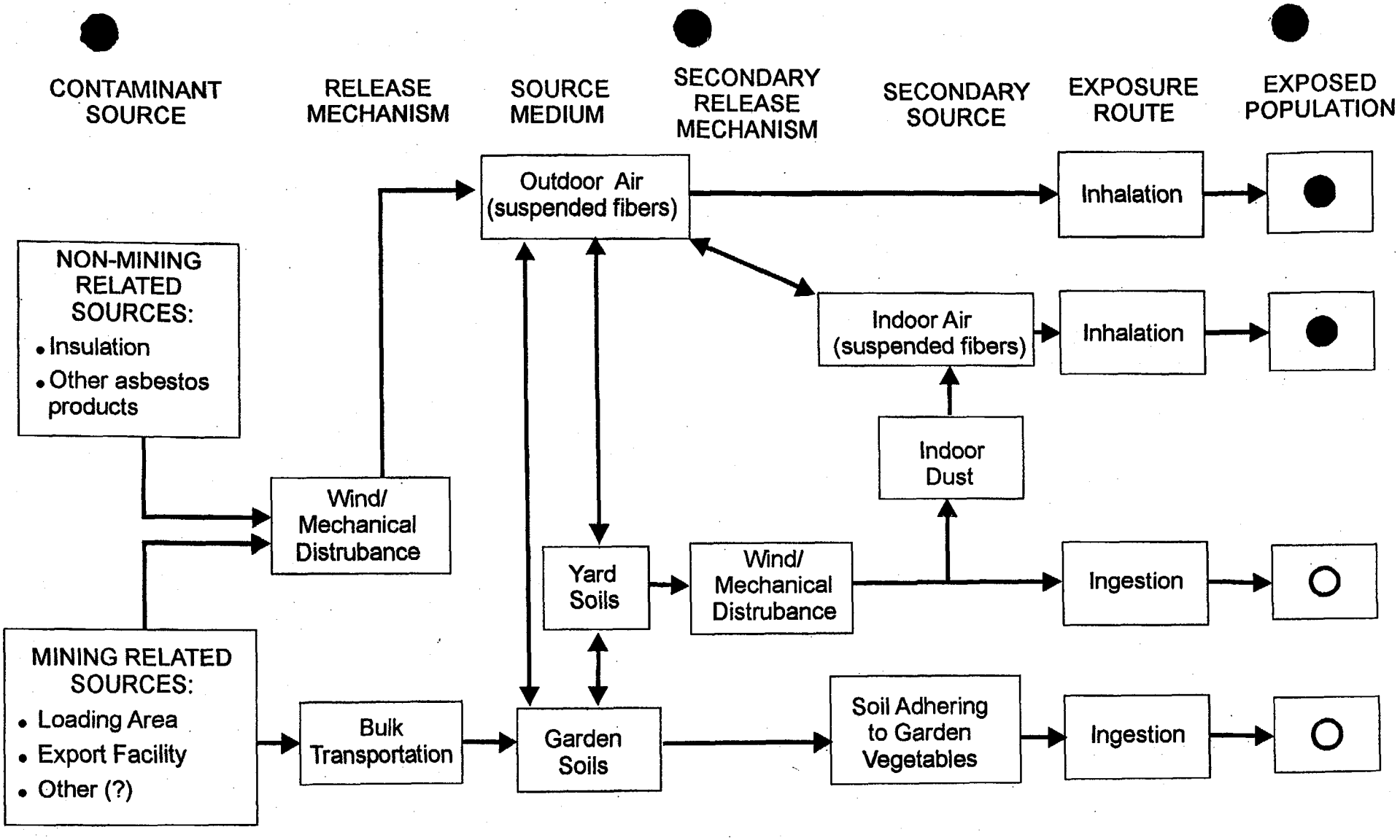

Figure 1. Draft Conceptual Site Model - Potential Human Exposure Pathways to Asbestos at the Libby, Montana Site

○ = Pathway is complete, but minor; qualitative evaluation

● = Pathway is complete and could be significant; quantitative evaluation

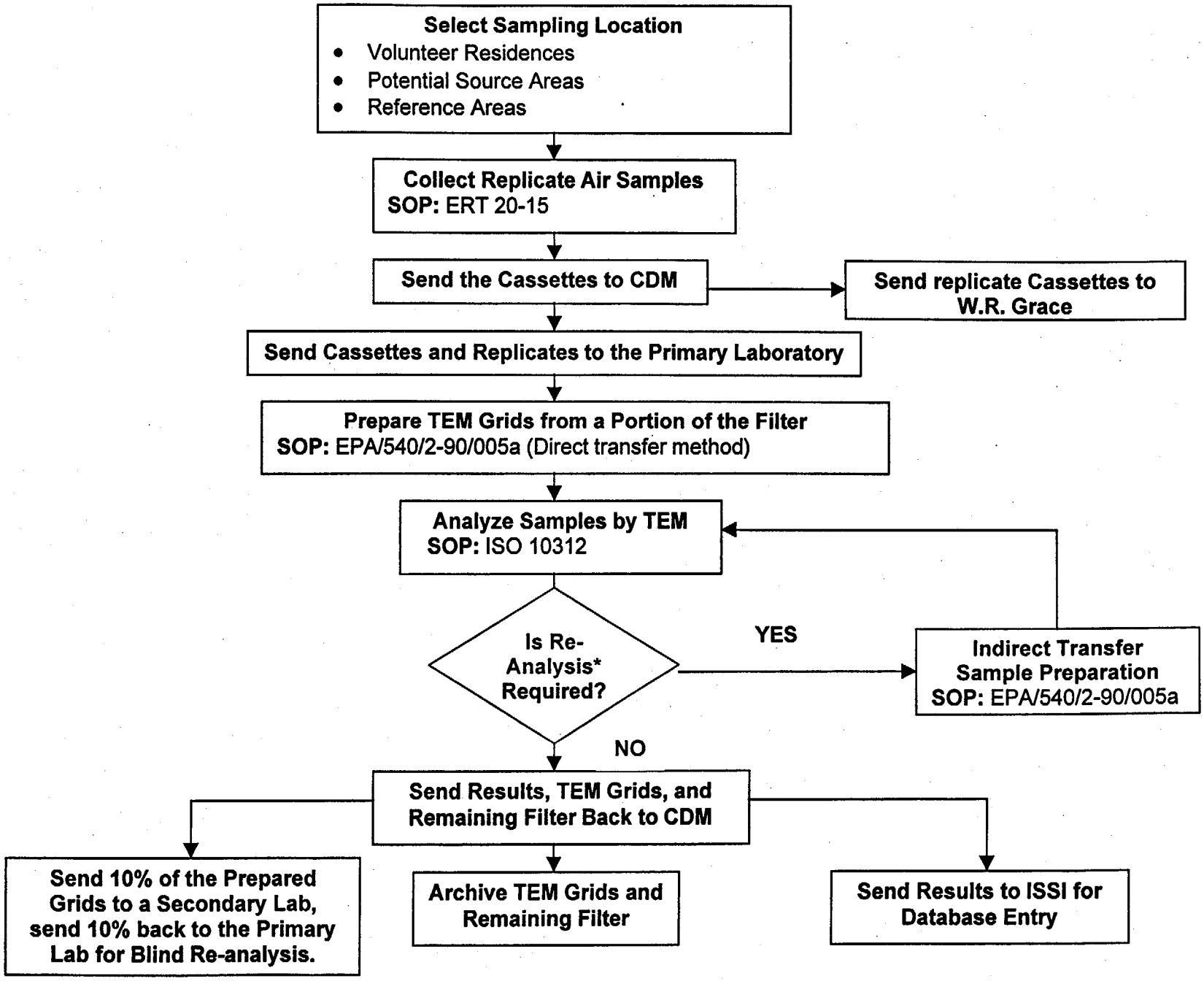

Figure 2. Air Sample Flow-Diagram

*For details on when re-analysis is required refer to the text.

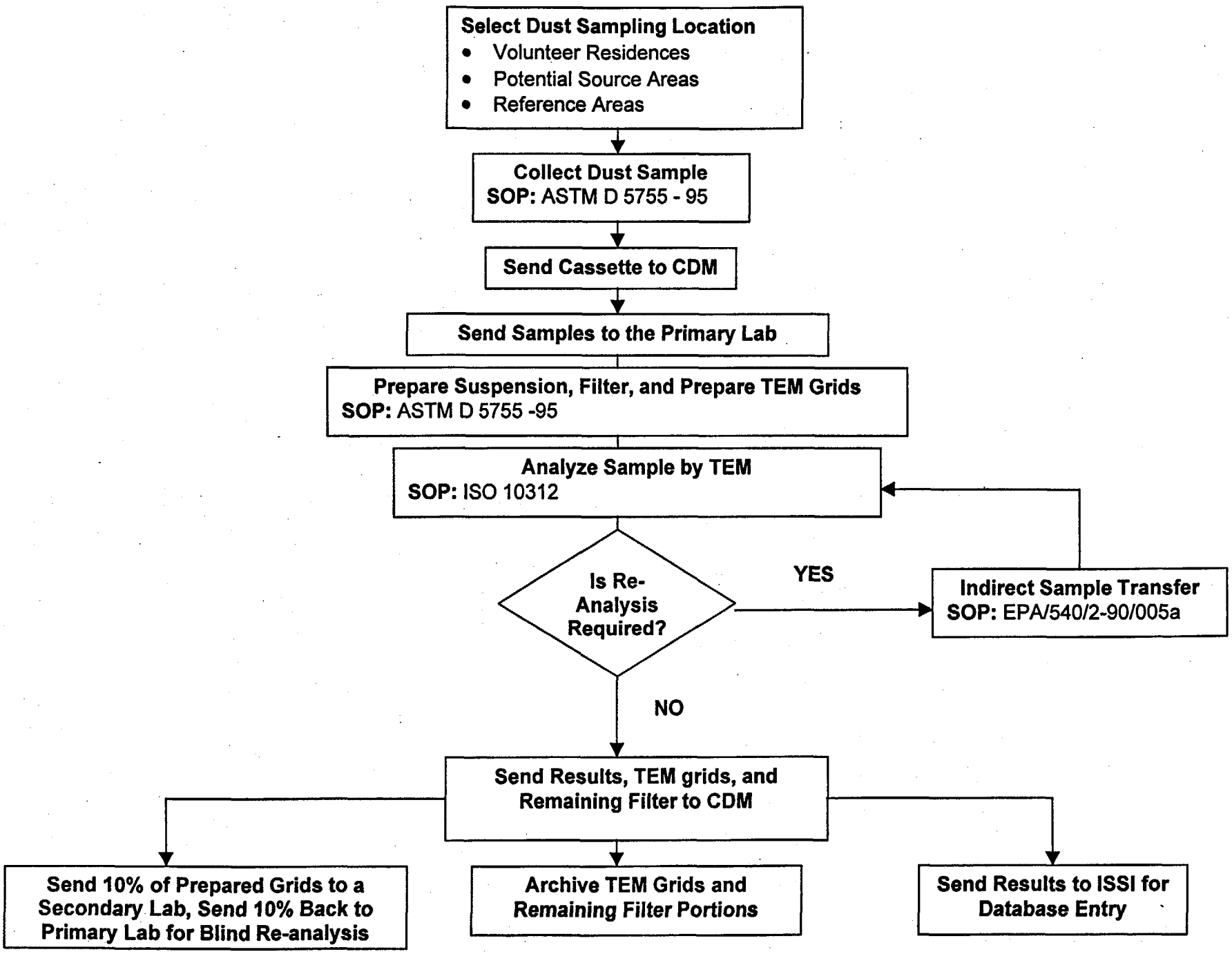

Figure 3. Dust Sample Flow Diagram

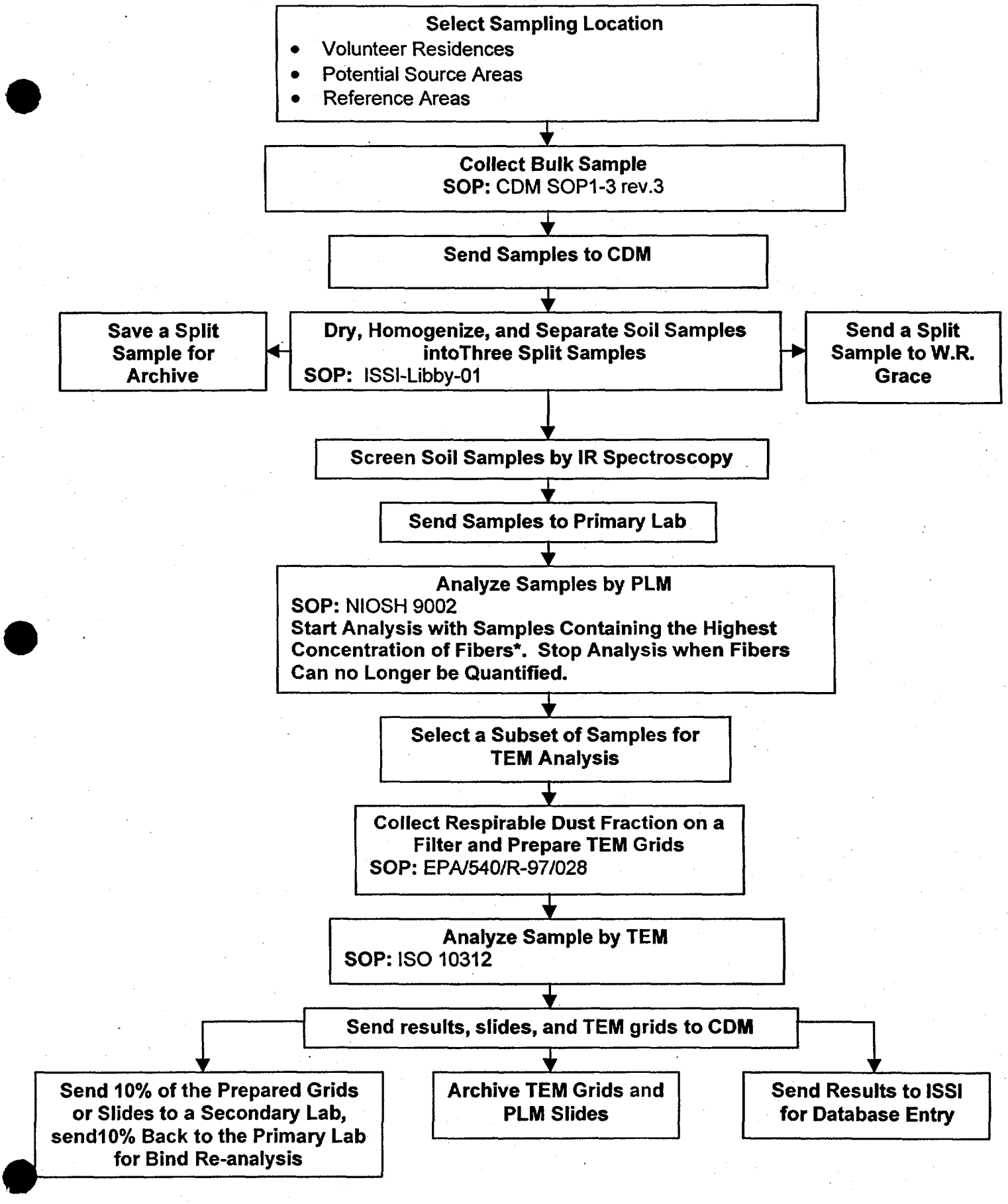

Figure 4. Sample Flow Diagram for Soil and Other Bulk Materials

*Refer to the text for a description of how the samples with the highest concentration of fibers will be selected.

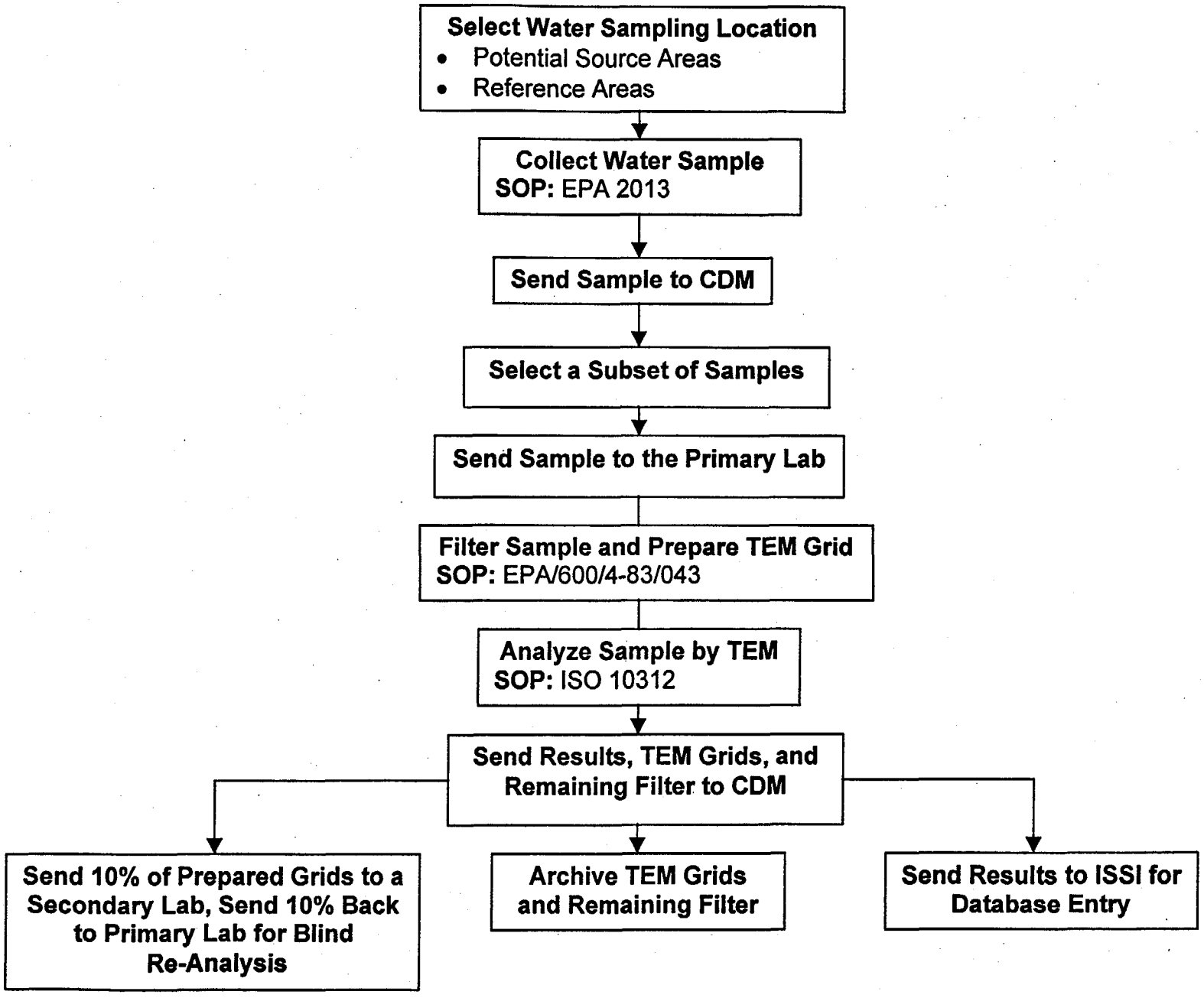

Figure 5. Water Sample Flow-Diagram

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E. APPENDICES

Appendix	Title	
1	Community Interview for Residents of Libby, Montana	
2	SOP 4-1	Field Logbook Content and Control
3	SOP 1-3	Surface Soil Sampling
4	SOP 1-2	Sample Custody
5	SOP 2-5	Packaging and Shipping of Environmental Samples
6	SOP 4-2	Photographic Documentation of Field Activities
7	SOP 4-5	Field Equipment Decontamination at Non-Radioactive Sites
8	Surface/Subsurface Soil Sampling Log	
9	ASTM D 5755-95	Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations
10	NIOSH Method 9002	Asbestos (bulk) by PLM
11	ISO 10312	Ambient Air - Determination of Asbestos Fibers: Direct -Transfer Transmission Electron Microscopy Method
12	EPA SOP 2015	Asbestos Sampling
13	ISSI- Libby-01	Soil Sample Preparation
14	EPA 540/2-90/005a	Superfund Method for the Determination of Asbestos in Ambient Air
15	EPA 540-R-97-028	Superfund Method for the Determination of Releasable Asbestos in Soil and Bulk Materials
16	EPA SOP 2013	Surface Water Sampling
17	EPA 600/4-84-043	Analytical Method for Determination of Asbestos Fibers in Water
18	ASTM D 3195 -90	Standard Practice for Rotameter Calibration
19	ISSI-Libby-02	Reflectance Spectroscopy Screening for Asbestos in Soil

Appendix 1

Community Interview for Residents of Libby, Montana

EPA Community Interview for Residents of Libby, Montana

Date:_____ Time:_____
Interviewers: _____
_____
_____

PART A

1. Address:

2. Phone:

3A. Please list the names and DOB of all immediate family members (enter information into table below): (indicate with an * all members that participated in the interview)

3B1. Are your children living 1) at home? Y / N / refused to answer (RA) (enter in table below)

3B2. If no, is he/she living in Libby? Y / N / RA (enter response and current address in table below)

NAME	DOB (Age, if DOB is unknown)	Children are living At home	In Libby	If a child is not at home, Please list current address:

4A. How long has your family has been living in Libby, MT: _____(years)

4B. How long has your family has been living at your current address: _____(years)

PART B

5. If you have health concerns about asbestos, where do you go for information?

6. Does your local physician provide you with information? Y / N / RA

7A. Do you have any specific concerns? Y / N / Not sure or don't know (NA)

7B. If yes, please describe:

8A. Would you like to be involved in any investigation/risk assessment that EPA carries out in Libby? Yes / No / NA / RA

8B. If yes, how?

PART C

9A. Did anyone in the family work at the Zonolite mine or the vermiculite processing operations?
Yes / No / RA

If Yes, please answer the following questions, if No or RA, skip to Question 12.

9B. Name(s) of workers	9C. Job Title(s) or short description of work duties:	9D1. Dates of employment (yr. to yr.)	9D2. If dates unknown, provide duration of employment and approximate start and/or stop dates	9E. Location(s) of work 1. Mine 2. RR loading area 3. Export site 4. Other (describe) 5. Unknown (mark all that apply)

10. Typically after a day of work, did family members, working in the vermiculite mining or processing operations: (Circle one) a) change clothes at work, b) wear their work clothes home, or c) other? (please describe)

11A. How did family members most frequently get to and from the vermiculite worksite? (Circle one) 1. Personal vehicle, 2. Ride from co-worker, 3. Bus, 4. Other (please describe):

11B. If a personal vehicle was used for work transportation was it frequently found to have accumulations of dust/dirt from the worksite in it? Yes / No / NA / RA

12A. Was the vermiculite from the mine used in or around your home ? Yes / No/ NA / RA
(if No, NA, or RA, skip to Question 18.)

12B. Was the vermiculite that was used in/around or your home purchased from a store? Yes / No / NA / RA
If No, 1. Where did you get it from?

2. Was it free? Y / N / NA / RA

13A. Was the vermiculite used for insulation? Yes / No / NA / RA (if no skip to 13B):
A1. If yes, describe locations (e.g., attic, walls)

A2. Is dust from the vermiculite insulation often noticeable in any of the living areas of the house ? Yes / No / NA / RA

13B. Was the vermiculite used for 1. Gardens, 2. Planting, 3. Greenhouse (circle all that apply)

13C. Were there other ways that vermiculite was used? Yes / No / NA / RA

13D. If yes, please describe:

14A. Aside from working at the mine, has anyone in the family been employed in a job with potential exposure to asbestos such as welding, automotive repair, plumbing, ductwork, roofing, carpentry, insulation installation?
Y / N / NA / RA

14B and 14C. If yes, please tell us:

B. Who	C. What kind of work: 1. welding, 2. automotive repair, 3. plumbing, 4. ductwork, 4. roofing, 5. carpentry, 6. Insulation installation, 7. Other (please describe)

15A. What year was your house built:_____
or if unknown, what is the approximate age of the residence_____ (years)(enter NA if unknown)

15B. Are you aware of any asbestos-containing-products other than vermiculite in your home such as floor tiles, pipe insulation, siding? Yes / No / NA / RA

15C. If yes, what are they: (circle all that apply) 1. Floor tiles, 2. Pipe Insulation, 3. Siding, 4. Insulation, 5. Other (please describe):

PART D

16A. Besides work, did you or family participate in any activities that bring you frequently into contact with the Zonolite mine, processing facilities, or the vermiculite? Y / N / NA / RA

16B. If yes, please describe these activities:

16C. Where did these activities occur:

17. Is there anything else you would like to say about the Zonolite mine?

18A. When the mine was operating, or more recently, do you believe that dust from the vermiculite mining or processing operations was accumulating on your property or in your home? Yes / No / NA

18b. If Yes, describe why you believed this was occurring:

19. Can you think of any way that vermiculite might have gotten into your home now or when you were growing up (i.e., on clothing) If so, please describe these activities:

20. Do you know of areas around town where vermiculite from the mine has been placed?

21A. As a child, where did you most frequently play?

21B. What was the area like? (please describe)

21C. Where do your children most frequently play?

22A. Are there areas of Libby that you believe present a risk from asbestos to residents? Y / N / NA / RA

22B. If yes, where:

23A. Is there anything you'd like more information about? Y / N / NA

23B. If yes, what:

24. What do you think is the best way to communicate with people in Libby?

1. Newspaper
2. Newsletter
3. Radio
4. Civic Organizations
5. Meetings
6. Other (please describe):

25. Where do you think would be a good place for meetings?

26. Who do you think are the formal and informal leaders in your community?

27A. Can you think of other people that we should talk with? Y / N / NA / RA

27B. Is yes, who?

28. Is there anything you would like to add?

Thank you!

Appendix 2

(SOP 4-1) Field Logbook Content and Control

FIELD LOGBOOK CONTENT AND CONTROL

Prepared: Del Baird

Technical Review: Jackie Mosher

QA Review: David O. Johnson

Approved: 2/23/99
Signature/Date

Issued: Rosemary Austin 2/24/99
Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to set CDM Federal criteria for content entry and form of field logbooks.

2.0 BACKGROUND

2.1 Definitions

Biota - The flora and fauna of a region.

Decontamination - To remove contaminants from field sampling equipment that might bias analytical results.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

2.2 Discussion

Information recorded in field logbooks include observations, data, calculations, time, weather, description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 RESPONSIBILITIES

Field Team Leader (FTL) - The FTL is responsible for ensuring the nature and form of data entries are conducted in accordance with this procedure.

Site Personnel - All CDM Federal employees who make entries in field logbooks during on-site activities are required to read this procedure prior to engaging in this activity. The FTL will assign field logbooks to

site personnel who will be responsible for their care and maintenance.

4.0 REQUIRED EQUIPMENT

- Site-specific plans
- Field notebook
- Indelible black or blue ink pen
- Ruler or similar scale (in some circumstances)

5.0 PROCEDURES

5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with other pertinent CDM Federal and site SOPs. These should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered prior to initial use of the logbook. Prior to use in the field, each logbook will be marked with a specific document control number issued by the document control administrator, if required by the QIP. Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field Logbook Document Control Number
- Activity (if the logbook is to be activity-specific)
- Name of CDM Federal contact and phone number(s)
- Start date

The first few (approximately five) pages of the logbook will be reserved for a table of contents. Mark the first page with the heading and enter the following:

TABLE OF CONTENTS

Date/Description	Page
(Start Date)/Reserved for TOC	1-5

The remaining pages of the Table of Contents will be designated as such with "TOC" written on the top center of each page.

5.2 Operation

The following is a list of requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information need not be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.

- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.

- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.

- Do not remove any pages from the book.

- Record as much information as possible.

Specific requirements for field logbook entries include:

- Initial and date each page
- Sign and date the final page of entries for each day
- Initial and date all changes
- Multiple authors must sign out the logbook by inserting the following:

  Above notes authored by:
  - (Sign name)
  - (Print name)
  - (Date)

- A new author must sign and print his/her name before additional entries are made
- Draw a diagonal line through the remainder of the final page at the end of the day
- Record the following information on a daily basis:

  - Date and time
  - Name of individual making entry

- Names of field team and other persons on-site
- Description of activity being conducted including station (i.e., well, boring, sampling location number) if appropriate
- Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
- Level of personal protection to be used
- Serial numbers of instruments
- Required calibration information
- Serial/tracking numbers on documentation (e.g., carrier airbills)

Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays
- Upgrade or downgrade of personal protection equipment

5.3 Post-Operation

To guard against loss of data due to damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated, and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

6.0 RESTRICTIONS/LIMITATIONS

Field logbooks constitute the official record of on-site technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM Federal personnel and their subcontractors. They are documents that may be used in court to indicate and defend dates, personnel, procedures, and techniques employed during site activities. Entries made in these notebooks should be factual, clear, precise, and as non-subjective as possible. Field logbooks, and entries within, are not to be utilized for personal use.

7.0 REFERENCES

Sandia National Laboratories, *Procedure for Preparing, Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures*, QA-02-03, Albuquerque Environmental Program Department 3220, Albuquerque, New Mexico, 1991.

Sandia National Laboratories, Division 7723, *Field Operation Procedure for Field Logbook Content and Control*, Environmental Restoration Department, Albuquerque, New Mexico, 1992.

Appendix 3

(SOP 1-3) Surface Soil Sampling

SURFACE SOIL SAMPLING

Prepared: Del R. Baird

Technical Review: Ken Black

QA Review: David O. Johnson

Approved: 2/24/99
Signature/Date

Issued: 2/24/99
Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to define the techniques and the requirements for collecting surface soil samples.

2.0 BACKGROUND

2.1 Definitions

Surface Soil - The soil that exists down from the surface approximately one foot (30 centimeters). Depending on application, the soil interval to be sampled will vary.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time.

Composite - Two or more subsamples taken from a specific media and site at a specific point in time. The subsamples are collected and mixed, then a single average sample is taken from the mixture.

Spoon/Scoop - A small stainless steel or Teflon utensil approximately 6 inches in length with a stem-like handle.

Trowel - A small stainless steel or Teflon shovel approximately 6 to 8 inches in length with a slight (approximately 140°) curve across. The trowel has a stem-like handle (for hand operation). Samples are collected with a spooning action.

2.2 Discussion

Surface soil samples are collected to determine the type(s) and level(s) of contamination and are often important to risk assessment. These samples may be collected as part of an investigative plan, site-

specific sampling plan, and/or as a screen for "hot spots," which may require more extensive sampling. Sediment(s) and sludge(s) that have been exposed by evaporation, stream rerouting, or any other means are collected by the same methods as those for surface soil(s). Typically, the top 1 to 2 centimeters (cm) of material, including vegetation, are carefully removed before collection of the sample.

Surface soil and exposed sediment or sludge are collected using stainless steel and/or Teflon-lined trowels or scoops.

2.3 Associated Procedures

- CDM Federal SOP 1-2, Sample Custody
- CDM Federal SOP 2-5, Packaging and Shipping of Environmental Samples
- CDM Federal SOP 4-1, Field Logbook Content and Control
- CDM Federal SOP 4-5, Field Equipment Decontamination at Nonradioactive Sites

3.0 RESPONSIBILITIES

Site Manager - The Site Manager is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to specific media sampling.

Field Team Leader - The Field Team Leader is responsible for ensuring that field personnel collect surface soil samples in accordance with this and other relevant procedures.

4.0 REQUIRED EQUIPMENT

- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Latex or appropriate gloves
- Plastic zip-top bags
- Personal protective clothing and equipment
- Stainless steel and/or Teflon-lined spatulas and pans, trays, or bowls
- Stainless steel and/or Teflon-lined trowels or spoons (or equipment as specified in the site-specific plans)

- Plastic sheeting

Additional equipment are discussed in Section 5.2.2, VOC Field Sampling/Preservation Methods.

5.0 PROCEDURES

5.1 Preparation

The following steps must be followed when preparing for sample collection:

1. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.

2. The collection points shall be stated, located on a site map, and referenced in the field logbook.

3. Processes for verifying depth of sampling must be specified in the site-specific plans.

4. Place clean plastic sheeting on a flat, level surface near the sampling area, if possible, and place equipment to be used on the plastic; place the insulated cooler(s) on separate plastic sheeting. Cover all equipment and supplies with clean plastic sheeting when not in use.

5. A clean, decontaminated trowel, scoop, or spoon will be used for each sample collected. Other equipment may be used (e.g., shovels) if constructed of stainless steel.

5.2 Collection

The following general steps must be followed when collecting surface soil samples:

1. Surface soil samples are normally collected from the least-contaminated to the most-contaminated areas. Stay outside a specific sampling location until all samples are collected at that location.

2. Document the sampling events, recording the information in the designated field logbook. Document any and all deviations from SOPs in the field logbook and include rationale for changes. See CDM Federal SOP 4-1.

3. Carefully remove stones, vegetation, snow, etc. from the sampling location surface.

4. First collect sample portions or aliquots for volatile analyses as well as any other samples that would be degraded by aeration. Follow with collection of samples for other analyses.

6. Decontaminate sampling equipment between locations. See CDM Federal SOP 4-5.

5.2.1 Method for Collecting Samples for Volatile Organic Compound (VOC) Analysis

The requirements for collecting grab samples of surface soil for VOCs or other samples degraded by aeration are as follows:

1. VOC samples shall be collected with the least disturbance possible.

2. VOC samples shall be collected as grab samples; however, the method of collection will vary from site to site, based on data quality objectives and the degree of known or suspected contamination.

3. Label the sample containers with the appropriate information. Secure the label, covering it with a piece of clear tape.

4. Use a clean stainless steel or Teflon-lined trowel or spoon (or tube) to collect sufficient material in one grab, over the required sampling interval, to fill the sample containers.

5. With the aid of a clean stainless steel spatula, quickly fill the sample containers directly from the sampling device, removing stones, twigs, grass, etc., from the sample. Fill the containers as full and compact as possible to minimize headspace.

6. Immediately secure the Teflon-lined caps on the sample container.

7. Wipe the containers clean with a clean Kimwipe or paper towel.

8. Place the containers in individual zip-top plastic bag(s) and seal the bag(s).

9. Pack all samples as required. Include properly completed documentation, and affix signed and dated custody seals to the cooler lid.

5.2.2 Field Sampling/Preservation Methods

The following four sections contain SW 846 methods for sampling and field preservation. These methods include EN CORE™ Sampler Method for low level detection limits, EN CORE™ Sampler Method for high level/detection limits/screening, Acid preservation, and Methanol preservation. These methods may be used if required by the EPA Region, client, or governing sample plan. These methods are very detailed and contain equipment requirements at the beginning of each section.

Note: Some variations from these methods may be required depending on the contracted analytical laboratory, such as sample volume.

5.2.2.1 EN CORE™SAMPLER COLLECTION FOR LOW LEVEL ANALYSES (≥1 UG/KG)

EN CORE™Sampling Equipment Requirements

The following equipment is required for low level analysis:

- Two 5g samplers.
- One 25g sampler or one 5g sampler for screening and or high level analysis. (The sampler
- size used will be dependent on who is doing the sampling and who is doing the laboratory
- analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

EN CORE™ Sampling Steps for Low Level Analysis

1. Remove sampler and cap from package and attach T-handle to sampler body.

2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.

3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.

4. Push cap on with a twisting motion to attach cap.

5. Fill out label and attach to sampler.

6. Repeat procedure for the other two samplers.

7. Collect dry weight sample.

8. Store samplers at 4 degrees Celsius.

Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

5.2.2.2 ACID PRESERVATION SAMPLING FOR LOW LEVEL ANALYSES (≥1 UG/KG)

Acid Preservation Sampling Equipment Requirements

The following equipment and supplies are required for field Acid preservation:

- One 40mL VOA vial with acid preservation (for field testing of soil pH). Two pre-weighed 40mL VOA vials with acid preservative and stir bar (for lab analysis)
- Two pre-weighed 40mL VOA vials with water and stir bar (in case samples effervesces).
- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a
- pre-weighed vial that contains methanol (for screening sample and/or high level analysis).
- One dry weight cup.
- One 2oz jar with acid preservative (in case additional acid is needed due to high
- soil pH).
- One scoop capable to deliver about one gram of solid sodium bisulfate.
- pH paper.
- Weighing balance that weighs to 0.01 g (with an accuracy of ±0.1g).
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper toweling.
- Sodium bisulfate acid ($NaHSO_4$) acid.
- A cutoff plastic syringe or other coring device to deliver 5 g or 25g of soil.

Testing Effervescing Capacity of Soils

Soils must be tested with acid to determine the amount of effervescing that will occur when preserved with acid. Effervescing will drive off VOCs as well as create a very high pressure in a sealed vial which could explode. The following steps will provide information on the effervescing capacity of the soil.

1. Place~5g of soil into a vial that contains acid preservative and no stir bar.

2. Do not cap this vial as it may EXPLODE upon interaction with the soil.

3. Observe the sample for gas evolution (due to carbonates in the soil).

4. If vigorous or sustained gas evolution occurs; then acid preservation is not acceptable to preserve the sample.

- In this case the samples need to be collected in the VOA vials with only water and a stir bar. The vials with acid preservative CANNOT be used.

5. If a small amount or no gas evolution occurs: then acid preservation is acceptable to preserve the sample. Keep this testing vial for use in the buffering testing detailed below.

- In this case the samples need to be collected in the VOA vials with the acid preservative and a stir bar.

Testing Buffering Capacity of Soils

The soils must be tested to determine the quantity of acid that is required to reach a less than 2 pH reading. The following steps will assist in determining this quantity.

1. If acid preservation is acceptable for sampling soils then the sample vial that was used in the effervescing testing can be used here for the buffering testing.

2. Cap the vial that contains 5g of soil, acid preservative and no stir bar from step 1 in the effervescing testing.

3. Shake the vial gently to attempt to make a homogenous solution.

4. When done, open the vial and check the pH of the acid solution with the pH paper.

- If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA trials with the acid preservative and stir bar. Since the pH was below 2, it is not necessary to add additional acid to the vials.

- If the pH paper reads above 2, then additional acid needs to be added to the sample vial.

5. Use the jar with the solid sodium bisulfate acid and add another one gram of acid to the sample.

6. Cap the vial and shake thoroughly again.

7. When done, open the vial and check the pH of the acid solution with a new piece of paper.

- If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA trials with the acid preservative and stir bar and one extra gram of acid.

- Make a note of the extra gram of acid needed so the same amount of extra acid can be added to the vials the lab will analyze.

- If the pH paper reads above 2, then add another gram of acid and repeat this procedure one more time.

Now that the soil chemistry has been determined the actual sampling can occur. The procedure stated below assumes the correct vials are used based on the guidance discussed.

Sample Preservation Steps

1. Wear gloves during all handling of pre-weighed vials.

2. Quickly collect a 5g sample using a cut off plastic syringe or other coring device designed to deliver 5g of soil from a freshly exposed surface of soil.

3. Carefully wipe exterior of sample collection device with clean paper toweling.

4. Quickly transfer to the appropriate VOA trial, extruding with caution so that the solution does not splash out of the vial.

5. Add more acid if necessary (this is based on the buffering testing discussed in the previous section).

6. Use the paper toweling and quickly remove any soil off the vial threads.

7. Cap vial and weigh the jar to the nearest 0.01 g.

8. Record exact weight on sample label.

9. Repeat sampling procedure for the duplicate VOA vial.

10. Weigh the vial with methanol preservative in it to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation below.

11. Take the empty jar or the jar that contains the methanol preservative.

12. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size is dependent on who is doing the sampling and who is doing the laboratory analysis.

13. Carefully wipe the exterior of the collection device with clean paper toweling.

14. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar received is dependent on who is doing the laboratory analysis.

15. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided-using only one vial of methanol preservative per sample jar.

16. Use the paper toweling and remove any soil off of the vial threads and cap the jar.

17. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.

18. Collect dry weight sample-fill container.

19. Store samples at 4 degrees Celsius.

20. Ship sample containers with plenty of ice and per Department of Transportation (DOT) regulations (CORROSIVE. FLAMMABLE LIQUID. POISON) to the laboratory.

5.2.2.3 EN CORE SAMPLER COLLECTION FOR HIGH LEVEL ANALYSES (≥200 UG/KG)

<u>EN CORE™ Sampling Equipment Requirements</u>

The following equipment is required for high level analysis:

- One 25g sampler or one 5g sampler (The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

EN CORE™Sampling Steps for High Level Analysis

1. Remove sample and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out label and attached to sampler.
6. Collect dry weight sample.
7. Store samplers at 4 degrees Celsius.
8. Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

5.2.2.4 METHANOL PRESERVATION SAMPLING FOR HIGH LEVEL ANALYSES (≥200 UG/KG)

Methanol Preservation Sampling Equipment Requirements

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (laboratory grade).
- One dry weight cup.
- Weighing balance that accurately weighs to 0.01g (with accuracy of ±0.1g).
- Set of balance weights used in daily balance calibration.
- Latex gloves.
- Paper towel.

- Cutoff plastic syringe or other coring device to deliver 5g or 25g of soil.

Sampling Preservation Steps

1. Wear gloves during all handling of pre-weighed vials.

2. Weigh the vial with methanol preservative in it to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation/collection below.

3. Take the empty jar or the jar that contains the methanol preservative.

4. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size used is dependent on who is doing the sampling and who is doing the laboratory analysis.

5. Carefully wipe the exterior of the collection device with clean paper toweling.

6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on who is doing the laboratory analysis.

7. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided-using only one vial of methanol preservative per sample jar.

8. Using the paper toweling-remove any soil off of the vial threads and cap the jar.

9. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.

10. Collect dry weight sample-fill container.

11. Store samples at 4 degrees Celsius.

12. Ship sample containers with plenty of ice and per DOT regulations (CORROSIVE. FLAMMABLE LIQUID. POISON) to the laboratory.

5.2.3 Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis

The requirements for collecting samples of surface soil for nonvolatile organic or inorganic analyses are as follows:

1. Label each sample container with the appropriate information. Secure the label by covering it with a piece of clear tape.

2. Use a decontaminated stainless steel or Teflon-lined trowel or spoon to obtain sufficient sample from the required interval and subsampling points, if necessary, to fill the specified sample containers.

3. Empty the contents of each fill of the sampling device directly into a clean stainless steel or Teflon-lined tray or bowl.

4. Homogenize the sample by mixing with a spoon, spatula, or trowel.

5. Use the spoon, spatula, or trowel to distribute the uniform mixture into the labeled sample containers. Fill organic sample containers first, then inorganics.

6. Secure the appropriate cap on each container immediately after filling it.

7. Wipe the sample containers clean with a clean Kimwipe or paper towel.

8. Place sample containers in individual zip-top plastic bags and seal the bags.

9. Pack all samples as required. Include properly completed documentation, and affix custody seals to the cooler lid.

10. Decontaminate sampling equipment according to CDM Federal SOP 4-5.

6.0 RESTRICTIONS/LIMITATIONS

When grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and, hence, analyze loss. The representativeness of this sample, however, is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

7.0 REFERENCES

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements For Field Methods,* DOE/HWP-69/R1, July 1990 or current revision.

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures For Site Characterizations*, DOE/HWP-100, July 1990 or current revision.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods,* EPA/540/P-87/001, December 1987 or current revision.

U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846),* Third Edition, November 1986, (as amended by Update III, June 1997). Method 5035: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.

Appendix 4

(SOP 1-2) Sample Custody

SAMPLE CUSTODY

SOP 1-2
Revision: 1
Date: June 30, 1994

Prepared: David O. Johnson 7-14-94 Signature/Date Technical Review: [illegible] 7/21/94 Signature/Date

QA Review: Marguerite E. Jones 7/22/94 Signature/Date Approved: [illegible] 8/12/94 Signature/Date

Issued: Rose Mary Ellebrick 8/19/94 Signature/Date

1.0 OBJECTIVE

Due to the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in CDM Federal Programs Corporation (CDM Federal) files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

2.0 BACKGROUND

2.1 Definitions

Sample - A material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

Sample Custody - A sample is under custody if:

1. It is in your possession.
2. It is in your view, after being in your possession.
3. It was in your possession and you locked it up.
4. It is in a designated secure area.

Chain-of-Custody Record - Form used to document the transfer of custody of samples from one individual to another.

Custody Seal - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

Sample Label - Adhesive label placed on sample containers to designate a sample identification number and other sampling information.

Sample Tag - Tag attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes).

3.0 RESPONSIBILITIES

Sampler - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Field Team Leader - The Field Team Leader is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The Field Team Leader is also responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on custody records.

4.0 REQUIRED SUPPLIES

- Chain-of-Custody Records (applicable CDM Federal forms)
- Custody seals
- Sample labels or tags
- Clear Tape

5.0 PROCEDURES

5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a Chain-of-Custody Record. This procedure will be followed for all samples collected or split samples accepted.

Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations prior to the actual fieldwork. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

3. Sample labels or tags shall be completed for each sample, using waterproof ink.

4. The Field Team Leader determines whether proper custody procedures were followed during the fieldwork and decides if additional samples are required.

Transfer of Custody and Shipment

1. Samples are accompanied by a Chain-of-Custody Record (see Figure 1 for example of Chain-of-Custody Record). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in the appropriate laboratory.

   - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the form.

   - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.

   - If samples are left unattended or a person refuses to sign, this must be documented and explained on the Chain-of-Custody Record.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment.

3. All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and the copies will be retained by the Field

Team Leader and if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the Field Team Leader as part of the permanent documentation. (Refer to Figure 1)

Procedure for Completing CDM Federal Chain-of-Custody Record (Refer to Figure 1.)

1. Record project number.

2. Record Field Team Leader for the project.

3. Record the name and address of the laboratory to which samples are being shipped.

4. Record the record number and total number of records being shipped for the day.

5. Enter the project name/location or code number.

6. Record overnight courier's airbill number.

7. Note sample type (matrix) and reference number. Include reference number on the Chain-of-Custody Record, box #9.

8. Record sample identification number.

9. Enter the reference number from box #7.

10. Enter date of sample collection.

11. Enter time of sample collection in military time.

12. Enter an X in appropriate box for sample designation - composite or grab.

13. Samplers must enter their initials next to the samples they collected.

14. List parameters for analysis and the number of containers submitted for each analysis.

15. Enter MS/MSD (matrix spike/matrix spike duplicate) if sample is for laboratory quality control, or other remarks (e.g. sample depth).

Figure 1
EXAMPLE CDM Federal Chain-of-Custody Record

CDM FEDERAL PROGRAMS CORPORATION

98 North Washington Street, Suite 200
Boston, MA 02114
617-742-2658

CHAIN OF CUSTODY RECORD

PROJECT NO. 01	FIELD TEAM LEADER 02	LABORATORY AND ADDRESS 03	RECORD ___ OF 04
PROJECT NAME/LOCATION 05			AIRBILL NO. 06

SAMPLE TYPES 07
1. SURFACE WATER
2. GROUNDWATER
3. LEACHATE
4. RINSATE
5. SOIL/SEDIMENT
6. OIL
7. WASTE
8. OTHER _____

SAMPLE NO	SAMPLE TYPE	YR DATE	TIME SAMPLED	COMP	GRAB	SAMPLING DETAILS*	PARAMETERS DESIRED / ANALYSES	REMARKS	PRESERVATIVES ADDED
08	09	010	011	-01	2-	013	014	015	016

* SAMPLER SIGNATURES: 017

RELINQUISHED BY: 018	DATE/TIME	RECEIVED BY 019	RELINQUISHED BY:	DATE/TIME	RECEIVED BY:
RELINQUISHED BY	DATE/TIME	RECEIVED BY	RELINQUISHED BY	DATE/TIME	RECEIVED BY

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory, yellow copy returned by laboratory. Pink copy returned by sampler. (1992)

NOTE: If requested by the client, different Chain-of-Custody records may be used. Copies of the template for this record may be obtained from the Fairfax Graphics Department.

16. Record the type of the preservative added by reference number and sample pH. Use the remarks column if no space is dedicated to preservative.

17. All samplers must sign in the space provided.

18. The originator checks information entered in items 1 through 17 and then signs the top left "Relinquished by" box, prints his/her name, and enters the current date and time (military).

    - Upon completion of the custody record form, the top two copies (usually white and yellow) shall be sent with the samples to the laboratory; the bottom copy (usually pink) is retained for the project files. Additional copies will be retained for the project file or distributed as required to the appropriate sample coordinators.

19. The laboratory sample custodian receiving the samples checks the sample label information against the custody record form. He or she also checks sample condition and notes anything unusual under "Remarks" on the custody record form. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the pink copy. The white copy is returned to CDM Federal.

5.2 Sample Labels and Tags

Sample labels or tags will be utilized for all samples collected or accepted for CDM Federal projects.

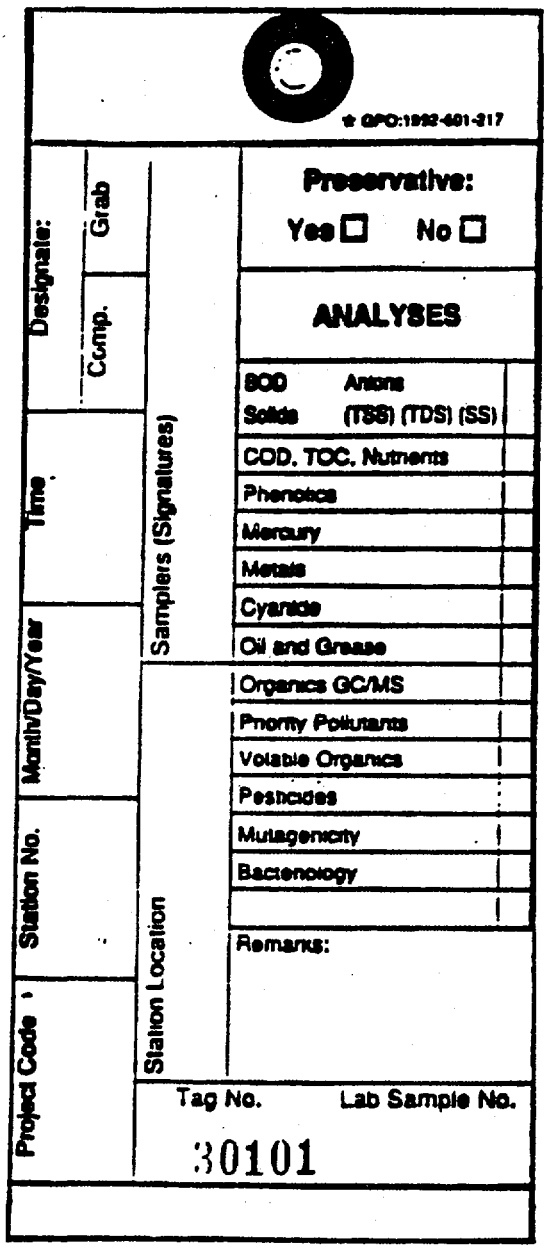
1. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.

2. Sample tags will be securely attached to the sample bottle. On 80 oz. amber bottles, the tag string may be looped through the ring style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted and relooped around the neck until the slack in the string is removed.

3. One label or tag will be completed for each sample container collected. Each label or tag will be completed as follows (see Figure 2 for example of sample tag); labels are completed with the equivalent information:

    - Record the Project Code (i.e., project or task number).
    - Enter the Station Number if applicable.
    - Record the date to indicate the month, day, and year of sample collection.
    - Enter the time (military) of sample collection.

Figure 2
EXAMPLE Sample Tag



NOTE: Equivalent sample labels or tags may be used.

- Place a check to indicate composite or grab sample.
- Record the sample location.
- Samplers must sign in the space provided.
- Place a check next to "yes" or "no" to indicate if a preservative was added.
- Under "analyses," place a check next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number."
- Under "remarks," add additional, relevant information.

5.3 Custody Seals

Custody seals must be placed on the shipping containers prior to shipment. The seal should be signed and dated by a field team member.

Custody seals may also be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction.

5.4 Sample Shipping

CDM Federal's Standard Operating Procedure 2-5: Packaging and Shipping of Environmental Samples establishes a uniform method for packaging and shipping low-level environmental samples.

6.0 RESTRICTIONS/LIMITATIONS

For EPA Contract Laboratory Program (CLP) sampling events, combined chain-of-custody/traffic report forms or other EPA-specific records may be utilized. Refer to regional guidelines for completing these forms.

7.0 REFERENCES

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.

U.S. Environmental Protection Agency, *Samplers Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December 1990.

Appendix 5

(SOP 2-5) Packaging and Shipping of Environmental Samples

PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

SOP: 2-5
Revision: 2
Date: November 15, 1995
Page: 1 of 4

Prepared: David O. Johnson 10/5/95 Signature/Date
Technical Review: [illegible] 10/17/95 Signature/Date
QA Review: Teresa A. Nelis 10/20/95 Signature/Date
Approved: [illegible] 11/8/95 Signature/Date
Issued: Rose Mary Ellersick 11/14/95 Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to establish packaging and shipping requirements and guidelines for environmental sample shipping.

2.0 BACKGROUND

2.1 Definitions

Environmental Sample - An Environmental Sample is any sample that has less than reportable quantities for any hazardous constituents according to Department of Transportation (DOT) regulations promulgated in 49 CFR - Part 172.

Chain of Custody Seal - Narrow strip of adhesive paper used to demonstrate that no tampering has occurred.

2.2 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis.

2.3 Associated Procedure

- CDM Federal SOP 1-2, Sample Custody

3.0 RESPONSIBILITIES

Field Team Leader - The Field Team Leader is responsible for ensuring that packaging and sampling procedures are conducted in accordance with this SOP. The Field Team Leader is also responsible for ensuring that laboratory analysis of samples is properly coordinated by CDM Federal.

4.0 REQUIRED EQUIPMENT

- Coolers with return address of CDM Federal office
- Heavy-duty plastic garbage bags
- Plastic zip-top bags, small and large
- Clear Tape
- Fiber tape
- Duct tape
- Vermiculite (or equivalent)*
- Bubble wrap (optional)
- Ice
- Chain-of-Custody seals
- Completed Chain-of-Custody record or CLP custody records if applicable
- Completed Bill of Lading
- "This End Up" and directional arrow labels

* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

5.0 PROCEDURES

The following steps must be followed when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of "Environmental Sample" and are not a hazardous material as defined by DOT. Professional judgement and/or consultation with the appropriate Health and Safety Coordinator or the Health and Safety Manager should be observed.

2. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape. Line the cooler with a large heavy-duty plastic garbage bag.

3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite or equivalent. **Note:** Trip blanks must be included in coolers containing VOA samples.

5. Place two to four inches of vermiculite (or equivalent) into a cooler that has been lined with a trash bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of more packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Due to the strength properties of a glass container, there is much less chance for breakage when the container is packed vertically rather than horizontally.

6. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at less than or equal to 4° Centigrade. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.

7. Place the completed Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler's lid, and then close the cooler.

8. Fiber tape shall be wrapped around each end of the cooler two times, and completed Chain-of-Custody seals affixed to the top opposite sides of the cooler, half on the fiber tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape; place clear tape over custody seals.

9. The shipping container lid must be marked "THIS END UP" and arrow labels which indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper (CDM Federal) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of the container used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the Bill of Lading shall be completed and attached to the lid of the shipping container.

6.0 RESTRICTIONS/LIMITATIONS

The holding times for the samples packed for shipment must not be exceeded. It is recommended that samples be packed in time to be shipped nightly for overnight delivery. Use caution when shipping samples for weekend delivery; make arrangements with laboratory before sending samples.

7.0 REFERENCES

U.S. Environmental Protection Agency, *Sampler's Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December 1990.

U.S. Environmental Protection Agency, Region IV, *Standard Operating Procedures and Quality Assurance Manual*, February 1991.

Appendix 6

(SOP 4-2) Photographic Documentation of Field Activities

PHOTOGRAPHIC DOCUMENTATION OF FIELD ACTIVITIES

Prepared: Del R. Baird

Technical Review: Jeanne Litwin

QA Review: David O. Johnson

Approved: 2/23/99
Signature/Date

Issued: 2/24/99
Signature/Date

1.0 OBJECTIVE

The purpose of this standard operating procedure (SOP) is to provide standard guidelines and methods for photographic documentation, which include still photography and video tape recordings, of field activities and site features (geologic formations, core sections, lithologic samples, water samples, general site layout, etc.). This document shall provide guidelines designed for use by a professional or amateur photographer. This SOP is intended for circumstances when formal photographic documentation is required. Based on project requirements, it may not be applicable for all photographic activities.

2.0 BACKGROUND

2.1 Definitions

Photographer - Camera operator (professional or amateur) of either still photography or video tape recording whose primary function with regard to this SOP is to produce documentary or data-oriented visual media.

Identifier Component - Visual components used within a photograph such as visual slates, reference markers, pointers.

Standard Reference Marker - A reference marker that is used to indicate a feature size in the photograph and is a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Slates - Blank, white, index cards used to write information pertaining to the subject/procedure being photographed. Letters and numbers on the slate will be bold and written with black, indelible marking pens.

Arrows and Pointers - A marker used to indicate and/or draw attention to a special feature within the photograph.

Contrasting Backgrounds - Backdrop used to lay soil samples, borings, or other objects for clearer viewing and to delineate features.

Data Recording Camera Back - A camera attachment or built-in feature that will record, at the very least, frame numbers and dates directly on the film.

2.2 Discussion

Photographs and video tape recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, equipment used and possible lithologic interpretation. This SOP is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard reference markers, and pointers. These items shall become an integral part of the "visual media" that, for the purpose of this document, shall encompass both still photographs including digital photographs and video tape recordings (or video footage). The use of a photographic logbook and standardized entry procedures are outlined as well. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the visual media and ensures the representative nature of the photographic documentation.

3.0 RESPONSIBILITIES

Field Team Leader (FTL) - The FTL is responsible for ensuring that the nature and form of data entries are conducted in accordance with this procedure.

Photographer - The photographer shall seek direction from the FTL and discuss the visual documentation requirements and schedule, regularly. The FTL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting. The photographer is responsible for maintaining a logbook per Sections 5.1, 5.2.4, and 5.3.1 of this SOP.

4.0 REQUIRED EQUIPMENT

- 35mm camera or disposable single use camera (35mm or panoramic use)
- Digital Camera
- Video Camera

- Logbook
- Indelible black ink pen
- Standard reference markers
- Slates
- Arrows or pointers
- Contrasting backgrounds
- Medium speed, or multi purpose fine-grain, color, 35-mm, negative film or slide film (project dependent)
- Data recording camera back (if available)
- Storage disk for digital camera

5.0 PROCEDURES

5.1 Documentation

A commercially available, bound logbook will be used to log and document photographic activities. Review the CDM Federal SOP 4-1 Field Logbook Content and Control and prepare all supplies needed for logbook entries.

Note: A separate photographic logbook is not required. A portion of the field logbook or site safety logbook may be designated as the photographic log section.

5.1.1 Field - Health and Safety Considerations

There are no hazards that an individual will be exposed to specific to photographic documentation. However, site-specific hazards may arise depending on location or operation. Personal protective equipment used in this operation will be site specific and dictated through requirements set by the Site Safety Officer, site Health and Safety Plan, and/or prescribed by the CDM Federal Corporate Health and Safety Program. The photographer should contact the Site Safety Officer for health and safety orientation prior to commencing field activities. The Site Health and Safety Plan must be read prior to entry to the site, and all individuals must sign the appropriate acknowledgement that this has been done.

The photographer should be aware of any potential physical hazardous while photographing the subject (i.e., low overhead hazard, edge of excavation).

5.2 OPERATION

5.2.1 General Photographic Activities in the Field

The following sections (5.2.1 - 5.3.2) provide general guidelines that should be followed to visually document field activities and site features using still cameras and video equipment. The photographer shall seek direction from the FTL and discuss the visual documentation requirements and schedule, regularly. The FTL is responsible for directing the photographer to specific situations, site features, or operations which the photographer will be responsible for documenting. Listed below are general suggestions that the photographer should consider when performing activities under this SOP:

- The photographer should be prepared to make a variety of shots, from close-up to wide angle. Many shots will be repetitive in nature or format especially close-up site feature photographs. Consideration should therefore be given to designing a system or technique that will provide a reliable repetition of performance.

- All still photographs should be made using a medium speed, or multi purpose fine-grain, color negative film in the 35-mm format unless otherwise directed by the FTL.

- It is suggested that Kodak brand "Ektapress Gold Deluxe" film or equivalent be used as the standard film for the still photography requirements of the field activities. This film is stable at room temperature after exposure and will better survive the time lag between exposure and processing. It is suggested that film speed ASA 100 should be used for outdoor photographs in bright sunlight, ASA 200 film should be used in cloudy conditions, and ASA 400 film should be used indoors or for very low-light outdoor photographs.

- No preference of video tape brand is specified, and is left to the discretion of the photographer.

- The lighting for sample and feature photography should be oriented toward a flat condition with little or no shadow. If the ambient lighting conditions are inadequate, the photographer should be prepared to augment the light (perhaps with reflectors or electronic flash) to maintain the desired visual effect.

- Digital photography quality is determined by the camera. A camera that obtains a higher resolution has a higher number of pixels, and will store a fewer number of photographs per each disk.

5.2.2 General Guidelines for Still Photography

Each new roll of film, digital storage disk and video tape shall contain upon the first usable frame (for film) or at the beginning of the tape (for video), a slate with the following information:

- A consecutively assigned control number (a consecutive, unique number that is assigned by the photographer to each roll of film or video tape, such as in sample numbers)
- The site location
- FTL's name
- Photographer's name
- Date

Caption Information

All still photographs will have a full caption permanently attached to the back containing the following information:

- Film roll control number and photograph document control number
- A brief site or scene description (What, Where, Who, Why)
- Date
- Time
- Direction
- Photographed by
- Witnessed by (if applicable)

A standard reference marker should be used in all documentary visual media. While the standard reference marker will predominantly be used in close-up feature documentation, inclusion in all scenes should be considered.

Close-up and Feature Photography

All close-up photographs will include a standard reference marker of appropriate size as an indication of the feature size and should contain a slate marked with the site name and any identifying label, such as a well number or core depth, that clearly communicates to the viewer the specific feature being photographed.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their *in situ* locations. This enables a more accurate record of their

initial condition and color. Include a standard reference color strip (color chart such as Munsell Soil Color Chart or that available from Eastman Kodak Co.) within the scene. This is to be included for the benefit of the viewer of the photographic document and serves as a reference aide to the viewer for lithologic observations and interpretations.

The photographer will permanently attach a caption containing pertinent information to the photograph after it has been produced and prior to submitting it to the project files.

Site Photography

Site photography, in general, will consist predominantly of medium and wide angle shots. A standard reference marker should be placed adjacent to the feature, or when this is not possible, within the same focal plane.

While it is encouraged that a standard reference marker and caption/slate be included in the scene, it is understood that situations will arise that preclude their inclusion within the scene. This will be especially true of wide angle shots. In such a case, the film/tape control number shall be entered in the photographic logbook along with the frame number and all other information pertinent to the scene.

The photographer will permanently attach a caption containing this information to the photograph after it has been produced and prior to submitting it to the project files.

Panoramic

In situations where a wide angle lens does not provide sufficient subject detail, a single-use disposal panoramic camera is recommended. If this type of camera is not available, a panoramic series of two or three photos would be appropriate. Panoramics can provide greater detail while covering a wide subject, such as an overall shot of a site.

To shoot a panoramic series using a standard 35mm camera, the following procedure is recommended.

- Utilizing a stable surface or tripod to support the camera is recommended.
- 20 - 30% from overlap while maintaining a uniform horizon is suggested.
- 2 to 3 photos per series is recommended.

5.2.3 General Photographic Documentation Using Video Cameras

As a reminder, it is not within the scope of this document to set guidelines for presentation or "show" video tape recording. The following guidelines are set for documentary video tape recordings only and should be implemented at the discretion of the FTL:

All documentary video tape recordings of field activities shall include an audio slate for all scenes. At the beginning of each video session an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account should include any additional information clarifying the subject matter being recorded.

A standard reference marker should be used when taking close-up shots of site features with a video camera. The scene should also include a caption/slate. It shall be placed adjacent and parallel to the feature being photographed.

It is strongly encouraged that a standard reference marker and caption/slate be included in all scenes. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

Original video tape recordings will not be edited. This will maintain the integrity of the information contained on the video tape. If editing is desired, a working copy of the original video tape recording can be made. The original video tape will be archived in accordance with Section 5.3.2.

5.2.4 Photographic Documentation

Photographic activities must be documented in a photographic logbook or in a section of the field logbook or site safety logbook. Each logbook will be assigned a document control number. The photographer will be responsible for making proper entries.

In addition to following the technical standards for logbook entry as referenced in Section 5.1.2, the following information should be maintained in the appropriate logbook:

- An entry shall be made for each new roll/tape control number assigned. Include information that is recorded on the film/tape control slate (the site location, FTL's name, photographer name, and date).

- A description of the general setup, including approximate distance between the camera and the subject, will be recorded in the appropriate logbook. Include pertinent information that is

necessary to provide a complete and full caption for each scene (refer to Sections 5.2.2 and 5.2.3).

- The laboratory used for photographic processing will be recorded (if known) in the appropriate logbook. Record the time and date that film is submitted to the photographic laboratory, as well as the time and date that the negatives and prints are received from the photographic laboratory.

- Document all situations encountered that cause an interruption in the progress of photographic activities under this SOP.

- Record as much other information as possible to assist in the identification of the photographic document.

5.3 Post Operation

All film will be sent for development and printing to a predetermined photographic laboratory (to be determined by the photographer). The photographer will be responsible for arranging transport of the film from the field to the photographic laboratory. The photographer shall also be responsible for delivery of the negatives and photographs or video tape to the project management.

5.3.1 Documentation

At the end of each day's photographic session, the photographer(s) will ensure that the appropriate logbook has been completely filled out and maintained as outlined in CDM Federal SOP 4-1.

5.3.2 Archive Procedures

1. All rolls of film, video tape recordings, and photographs should be considered controlled documents. Therefore, all photographs and the associated set of negatives and original unedited documentary video tape recordings will be submitted to the project files and handled accordingly. The FTL will ensure their proper distribution.

2. Based on specific project requirements, the photographer will assign each roll of film or photograph a control number. Film control numbers may be photographed on the first usable frame of the film and permanently attached to the negative sleeve/holders. Control numbers for photographs may contain the control number of the film and the frame number from which the photograph was made and be written on the back of the photograph. All control numbers will be written with black indelible ink.

3. Original video tape footage shall be assigned a document control number. Video tapes must have a document control number written on the tape case as well as the video tape cassette. All document control numbers will be written with black indelible ink. Original video footage shall not be edited but may be copied.

4. Completed pages of the appropriate logbook will be copied weekly and submitted to the project files.

6.0 RESTRICTIONS/LIMITATIONS

This document is designed to provide a set of guidelines for the field amateur or professional photographer to ensure that an effective and standardized program of visual documentation is maintained.

It is not within the scope of this document to provide instruction in photographic procedures, nor is it within the scope of this document to set guidelines for presentation or "show" photography.

The procedures outlined herein are general by nature. The FTL is responsible for directly specific operational activity or procedure. Questions concerning specific procedures or requirements should be directed to the FTL.

7.0 REFERENCES

Sandia National Laboratories, *Procedure for Preparing Sampling and Analysis Plans, Site-Specific Sampling Plans, and Field Operating Procedures*, QA-02-03, Environmental Programs Department 3220, Albuquerque, New Mexico, 1991.

Sandia National Laboratories, Division 7723, *Field Operating Procedure for Field Logbook Content and Control*, PRO 92-04, Environmental Restoration Department, Albuquerque, New Mexico, 1992.

U.S. Department of Energy, Yucca Mountain Project, *Branch Technical Procedure: Field Logging, Handling, and Documenting Borehole Samples*, Las Vegas, Nevada, 1989.

Appendix 7

(SOP 4-5) Field Equipment Decontamination at Non-Radioactive Sites

FIELD EQUIPMENT DECONTAMINATION AT NONRADIOACTIVE SITES

Prepared: Susan Flakus

Technical Review: Dave Schroeder

QA Review: David O. Johnson

Approved: 3/13/98
Signature/Date

Issued: 3/13/98
Signature/Date

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to describe the procedures required for decontamination of field equipment.

2.0 BACKGROUND

2.1 Definitions

Clean - Free of visible contamination and when decontamination has been completed in accordance with this SOP.

Cross-Contamination - The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or noncontaminated samples or areas.

Decontamination - The process of rinsing or otherwise cleaning the surfaces of equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure of personnel.

2.2 Discussion

Decontamination of field equipment is necessary to ensure the quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants off-site.

3.0 RESPONSIBILITIES

Field Team Leader - The Field Team Leader (FTL) ensures that field personnel are trained in the performance of this procedure and that decontamination is conducted in accordance with this procedure. The FTL may also be required to collect and document rinsate samples to provide quantitative verification that these procedures have been correctly implemented.

4.0 REQUIRED EQUIPMENT

- Stiff-bristle scrub brushes
- Plastic buckets and troughs
- Laboratory-grade detergent (low phosphate)
- Nalgene or Teflon Sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Plastic sheeting
- Disposable wipes or rags
- Potable water and/or deionized water and/or American Society for Testing and Materials (ASTM) Type II or better, as defined by ASTM Standard Specification for Reagent Water, Standard D 1193-77 (re-approved 1983)*
- Gloves, safety glasses, and other protective clothing as specified in the site-specific health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source (e.g. 10% and/or 1% nitric acid (HNO_3), acetone, methanol, isopropanol, hexane)
- Tools for equipment assembly and disassembly (as required)
- 55-gallon drums or tanks (as required)
- Pallets for drums or tanks holding decontamination water (as required)

* Potable, deionized, and ASTM Type II water may be required to be tested for contaminants before use. Check field plan for requirements.

5.0 PROCEDURES

All reusable equipment (non-dedicated) used to collect, handle, or measure samples will be decontaminated before coming into contact with any sample. Decontamination of equipment will occur either at the central decontamination station or at portable decontamination stations set up at the sampling location, drill sites, or monitoring well locations. The centrally located decontamination station will include an appropriately sized bermed area on which equipment decontamination will occur and shall be equipped with a collection system and storage vessels. In certain circumstances, berming is not required when small quantities of water are being generated and for some short duration field activities (i.e., pre-remedial sampling).

The decontamination area will be constructed so that contaminated water is either collected directly into appropriate containers (5-gallon buckets or steel wash tubs) or within the berms of the decontamination area which then drains into a collection system. Water from the collection system will be pumped into 55-gallon drums or portable tanks for storage. Typically, decontamination water will be staged until sampling results or waste characterization results are obtained and evaluated and the proper disposition

of the waste is determined. The exact procedure for decontamination waste disposal should be discussed in the field plan. Also, decontamination fluids, such as solvents may need to be segregated from other investigation derived wastes.

All items that will come into contact with potentially contaminated media will be decontaminated before use and between sampling and/or drilling locations. If decontaminated items are not immediately used, they will be covered either with plastic or aluminum foil depending on the size of the item. All decontamination procedures for the equipment being used are as follows:

General Guidelines

- Potable and deionized water should be free of all contaminants of concern. Following the field plan, analytical data from the water source may be required. If required, either existing analytical data from the water source supplier (i.e., municipality, bottled water company, deionized water producer) may be obtained or chemical testing may be performed on the selected source.
- Soap used in the soap and water rinse step will be a low phosphate detergent.
- Sampling equipment that has come into contact with oil and grease will be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or client requirements will be stated in the field plan.
- Decontaminated equipment will be allowed to air dry before being used.
- Documentation for all cleaning will be recorded in the appropriate logbook.
- All solvents will be pesticide grade or better and traceable to a source. The corresponding lot numbers will be recorded in the appropriate logbook.
- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment will be used as specified in the site-specific health and safety plan.

5.1 Heavy Equipment Decontamination

Heavy equipment includes drilling rigs and backhoes. Follow these steps when decontaminating this equipment:

1. Establish a decontamination area with berms that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be utilized; otherwise, use one or more layers of heavy plastic sheeting to cover the

ground surface and berms. All decontamination pads should be upwind of the area to investigated.

2. With the rig in place, spray areas (rear of rig or backhoe) exposed to contaminated soils using a hot water high-pressure sprayer. Be sure to spray down all surfaces, including the undercarriage.

3. Use brushes, and low phosphate detergent and potable water to remove dirt whenever necessary.

4. Remove equipment from the decontamination pad and allow it to air dry before returning it to the work site.

5. Record equipment type, date, time, and method of decontamination in the appropriate logbook.

6. After decontamination activities are completed, collect all contaminated waste water, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the field plan. Liquids and solids must be drummed separately.

5.2 Downhole Equipment Decontamination

Downhole equipment decontamination includes hollow-stem augers, drill pipes, casings, screens, etc. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area should be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.

2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination pads should be upwind of any areas under investigation.

3. Place the object to be cleaned on aluminum foil or plastic-covered wooden sawhorses or other supports.

4. Using low phosphate detergent and potable water in the hot water high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.

5. If using soapy water, rinse the equipment using clean, potable water. If using hot water, the rinse step is not necessary if the hot water does not contain a detergent. If the hot water contains a detergent, this final clean water rinse is required.

6. Using the manual-pump sprayer, rinse the equipment thoroughly with deionized water (ASTM Type II or better).

7. Remove the equipment from the decontamination area and place in a clean area upwind to air dry.

8. Record equipment type, date, time, and method of decontamination in the appropriate logbook.

9. After decontamination activities are completed, collect all contaminated waste waters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

5.3 Sampling Equipment Decontamination

Sampling equipment includes split spoons, spatulas, and bowls used for sample homogenization that directly contact sample media. Follow these steps when decontaminating this equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line should progress from "dirty" to "clean" and have an area located upwind for drying decontaminated equipment. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or the surfaces on which decontaminated equipment is to be placed for drying.

2. Before washing, disassemble any items that might trap contaminants internally. Do not reassemble these items until decontamination and air drying are complete. Wash items thoroughly in a bucket of low phosphate detergent and potable water. Use a stiff-bristle brush to dislodge any gross contamination (soil or debris).

3. Rinse the item in potable water. Rinse water should be replaced as needed, generally when cloudy.

4. Using a hand sprayer, wash bottles, or manual-pump sprayer, rinse the item with deionized water (ASTM Type II or better).

5. If required by the site-specific field plans, rinse the item with 10% nitric acid (for stainless steel, glass, plastic, and Teflon), or 1% nitric acid (for items made of low-carbon steel) followed by a deionized water (ASTM Type II or better) rinse.

   NOTE: Care should be taken not to get nitric acid on skin or clothing. This step should not be used unless required by sampling needs as dictated in the field plan.

   CAUTION: Do not allow nitric acid to contact methanol or hexane. Contain nitric acid waste separate from organic solvents.

6. If sampling for organic analytes, rinse the item with methanol or approved organic solvent.

7. Rinse the item with deionized water (ASTM Type II or better).

8. If required by the field plan, when sampling for polar organic compounds such as pesticides, polychlorinated biphenyls (PCBs), and fuels, rinse the item with hexane or approved alternatives, followed by a second methanol rinse.

9. Allow the item to air dry completely.

10. After drying, wrap the clean item in plastic wrap or in aluminum foil, shiny side out.

11. Record equipment type, date, time, and method of decontamination in the appropriate logbook.

12. After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable gloves, boots, and clothing. Place contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. (Refer to site-specific plans for labeling and waste management requirements).

5.4 Pump Decontamination

Follow these steps when decontaminating pumps:

1. Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other porous surfaces. Set up three 55-gallon drums and one or more containers of ASTM Type II water (or as specified in the field plan) with one drum containing dilute (nonfoaming) soapy water, the second drum containing potable water, and the third drum receiving waste water.

2. The pump should be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first drum. Place the discharge outlet in the waste water drum above the level of the waste water. Pump soapy water through the pump assembly until it discharges to the waste drum.

3. Move the pump assembly to the potable water drum while leaving discharge outlet in the waste drum. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.

4. Move the pump intake to the ASTM Type II water can. Pump the ASTM Type II water through the pump assembly. Usually, three pump-and-line-assembly volumes will be required.

5. Decontaminate the discharge outlet by hand following the steps outlined in Section 5.3.

6. Remove the decontaminated pump assembly to the "clean" area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices should be covered with aluminum foil to prevent the entry of airborne contaminants and particles.

7. Record the equipment type, serial number, date, time, and method of decontamination in the appropriate logbook.

5.5 Instrument Probe Decontamination

Instrument probes used for field instruments such as pH meters, conductivity meters etc. will be decontaminated between samples and after use with ASTM type II, or better, water.

5.6 Waste Disposal

Refer to site-specific plans for waste disposal requirements. The following are guidelines for disposing of wastes:

1. All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste.

2. Small quantities of decontamination solutions may be allowed to evaporate to dryness.

3. If large quantities of used decontamination solutions will be generated, it may be best to separate each type of waste in a separate container. This may permit the disposal of wash

water and rinse water onsite or in a sanitary sewage treatment plant rather than as a hazardous waste. If an industrial waste water treatment plant is available onsite, the disposal of acid solutions and solvent-water solutions may be permitted.

4. Unless required, plastic sheeting and disposable protective clothing may be treated as a solid, nonhazardous waste.

6.0 RESTRICTIONS/LIMITATIONS

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics respectively. These steps should not be used unless required because of acid burn and ignitability hazards.

If the field equipment is not allowed to air dry properly before use, volatile organic residue which interferes with the analysis may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted; in the summer, volatilization is rapid and in the winter, volatilization is slow. Check with your EPA region, state and client for approved decontamination solvents.

7.0 REFERENCES

Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures For Site Characterization*, DOE/HWP-100, July 1990.

Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements For Field Methods*, DOE/HWP-69/RI.

American Society for Testing and Materials, *Standard Practice for Decontamination of Field Equipment at Nonradioactive Waste Sites*, ASTM D5088-90, June 29, 1990.

U.S. Environmental Protection Agency, Region II, "*CERCLA Quality Assurance Manual*, Revision 1, 1989.

U.S. Environmental Protection Agency, Region IV, *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual*, 1986.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001.1, 1987.

Appendix 8

Surface/Subsurface Soil Sampling Log

Surface/Subsurface Soil Sampling Log

Project Name:	Sampling Location:
Project Number:	Sampler(s):
Site Location:	Date:

Sample Information

Sample Time:	USCS Abbreviation:
Grab Composite	Color:
Depth Interval:	Staining:
Type of Soil Cover:	Odor:
Soil Type:	

Sample No./QC Sample No.	Analysis	Type of Container	Quantity	Preservative

Comments:

Composite Sketch:

Appendix 9

(ASTM D 5755-95) Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations

Designation: D 5755 – 95

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the concentration of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the concentration of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1739 Test Method for the Collection and Measurement of Dustfall (Settleable Particulate Matter)[3]

D 3195 Practice for Rotameter Calibration[3]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D-22[3]

D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (**1**),[4] (**2**) and (**3**).

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

DISCUSSION—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (**3**).

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into

[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[5] The non-asbestiform variations of the minerals indicated in 5.1.3 have different Chemical Abstract Service (CAS) numbers.

smaller components without losing its fibrous properties or appearance.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of ≤1 mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).

3.2.6 *fiber*—a structure having a minimum length of 0.5 µm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous solution of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the concentration of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.

5.2 This test method uses the definition, settleable particulate material, found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and a minimum of three samples analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall 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.[6]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA).*

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood.*

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers.*

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF).*

7.25 *Glacial Acetic Acid.*

7.26 *1-methyl-2-pyrrolidone.*

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper.*

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter.*

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8 µm or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon*[7] *Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods.*

7.41 *Lens Tissue.*

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings.*

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica.*

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information (5).

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Tygon is a registered trademark of the DuPont Co.

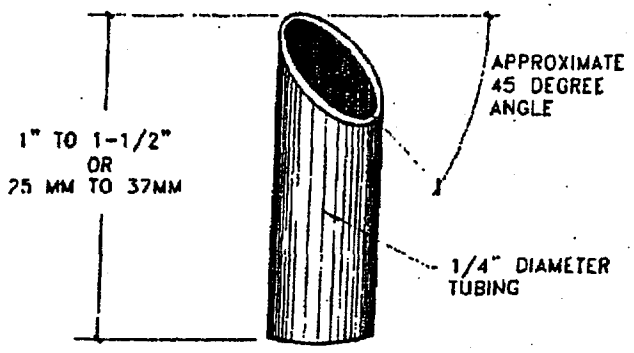

FIG. 1 Example of the Tubing Nozzle

sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 11).

10.4 All sample preparation steps 10.4.1 through 10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust suspension by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of solution in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate suspension such as diluted India ink (suspension of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 µm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 µm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring

that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the solution in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8 through 10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of solution has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original solution or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. *TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters*

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method:*

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method:*

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer:*

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

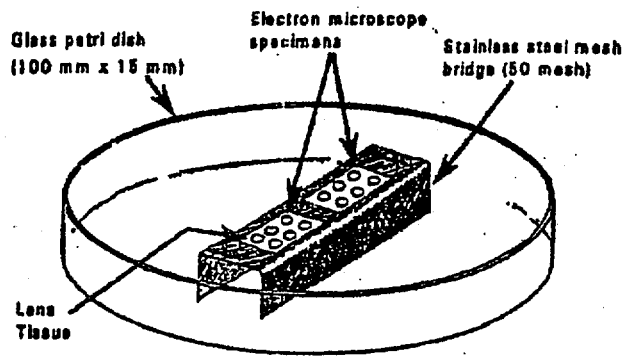

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2: Caution—Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 µm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability:*

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the X and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability:*

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing*

Materials in Schools document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules:*

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity*—An analytical sensitivity of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter. Occasionally, due to high particle loadings or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply.

16.9 *Limit of Detection*—The limit of detection for this method is defined as, at a minimum, the counting of four asbestos structures during the TEM analysis. If less than four asbestos structures are counted during the analysis then the analytical result which will be reported will be less than the limit of detection and a "less than" sign (<) will appear before the number. All data shall be provided in the laboratory report.

16.10 *Stopping Rules:*

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an analytical sensitivity of less than 1000 asbestos structures per square centimetre.

16.10.2 If an analytical sensitivity of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Concentration in structures/cm^2.

18.1.2 The analytical sensitivity.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ \text{mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \qquad (1)$$

where:

$\#STR$ = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a

regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM:*

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(*a*) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(*b*) Frequency of calibration will depend on the service history of the particular microscope.

(*c*) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(*d*) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(*e*) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(*a*) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(*b*) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA:*

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \quad (2)$$

where:

4.185 = Joules/cal,

R = energy deposition, watts/mL,

θ_1 = temperature rise with the ultrasonic bath not operating, °C,

θ_2 = temperature rise with the ultrasonic bath operating, °C,

t = time in seconds, 60 s (20.5.3 and 20.5.5),

σ = specific heat of the liquid in the glass sample container, 1.0 cal/g, and

ρ = density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client:		Accelerating Voltage:	
Sample ID:		Indicated Mag:	KX
Job Number:		Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:		Filter Type:	
Grid Accepted, 600X:	Yes No	Filter Size:	
Percent Loading:	%	Filter Pore Size (μm):	
Grid Box #1:		Grid Opening:	1) μm x μm
			2) μm x μm

Analyst: _____

Reviewer: _____ Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm²:	(EFA)	
Number of Grid Openings Counted:	(GO)	
Avderage Grid Opening Area in mm²:	(GOA)	
Volume of sample Filtered in ml:	(V)	
Surface area Sampled in cm²:	(SPL)	
Number of Asbestos Structures Counted:*	(#STR)	

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM²:

$$\frac{EFA \times 100 \times \#STR}{GO \times GOA \times V \times SPL} = \text{(Asbestos Structures per cm}^2\text{)}$$

Results for Total Asbestos Structures: _____ (Structures per cm²)

Results for Structures ≥ microns: _____ (Structures per cm²)

FIG. X1.1 Dust Sample Analysis Work Sheet

Job Number: _____

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: Keys to Abbreviations Used in Figure:

Type:

C = Chrysotile
AM = Amosite
CR = Crocidolite
AC = Actinolite
TR = Tremolite
AN = Anthophyllite
N = Non Asbestos

Structure:

F = Fiber
B = Bundle
C = Cluster
M = Matrix

Others:

NSD = No Structures Detected
Morph = Morphology
SAED = Selected Area Electron Diffraction
EDS = Energy Dispersive X-Ray Spectroscopy
ER = Inter-Row Spacing
NP = No Pattern

FIG. X1.2 TEM Count Sheet

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Appendix 10

(NIOSH Method 9002) Asbestos (bulk) by PLM

ASBESTOS (bulk) by PLM

9002

various MW: various CAS: 1332-21-4 RTECS: CI6475000

METHOD: 9002, Issue 2 **EVALUATION:** PARTIAL **Issue 1:** 15 May 1989 **Issue 2:** 15 August 1994

EPA Standard (Bulk): 1% **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].

SAMPLING

BULK SAMPLE:	1 to 10 grams
SHIPMENT:	seal securely to prevent escape of asbestos
SAMPLE STABILITY:	stable
BLANKS:	none required

ACCURACY

RANGE STUDIED:	<1% to 100% asbestos
BIAS:	not determined
PRECISION:	not determined
ACCURACY:	not determined

MEASUREMENT

TECHNIQUE:	MICROSCOPY, STEREO AND POLARIZED LIGHT, WITH DISPERSION STAINING
ANALYTE:	actinolite asbestos, amosite, anthophyllite asbestos, chrysotile, crocidolite, tremolite asbestos
EQUIPMENT:	microscope, polarized light; 100-400X dispersion staining objective, stereo microscope: 10-45X
RANGE:	1% to 100% asbestos
ESTIMATED LOD:	<1% asbestos [1]
PRECISION:	not determined

APPLICABILITY: this method is useful for the qualitative identification of asbestos and the semi-quantitative determination of asbestos content of bulk samples. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience. The method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope.

INTERFERENCES: Other fibers with optical properties similar to the asbestos minerals may give positive interferences. Optical properties of asbestos may be obscured by coating on the fibers. Fibers finer than the resolving power of the microscope (ca. 0.3 μm) will not be detected. Heat and acid treatment may alter the index of refraction of asbestos and change its color.

OTHER METHODS: This method (originally designated as method 7403) is designed for use with NIOSH Methods 7400 (phase contrast microscopy) and 7402 (electron microscopy/EDS). The method is similar to the EPA bulk asbestos method [1].

REAGENTS:

1. Refractive index (RI) liquids for Dispersion Staining: high-dispersion (HD) series, 1.550, 1.605, 1.620.
2. Refractive index liquids: 1.670, 1.680, and 1.700.
3. Asbestos reference samples such as SRM #1866, available from the National Institute of Standards and Technology.*
4. Distilled Water (optional).
5. Concentrated HCl: ACS reagent grade.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sample containers: screw-top plastic vials of 10- to 50-mL capacity.
2. Microscope, polarized light, with polarizer, analyzer, port for retardation plate, 360° graduated rotating stage, substage condenser with iris, lamp, lamp iris, and:
   a. Objective lenses: 10X, 20X, and 40X or near equivalent.
   b. Ocular lense: 10X minimum.
   c. Eyepiece reticle: crosshair.
   d. Dispersion staining objective lens or equivalent.
   e. Compensator plate: ca. 550 nm ± 20 nm, retardation: "first order red" compensator.
3. Microscope slides: 75 mm x 25 mm.
4. Cover slips.
5. Ventilated hood or negative-pressure glove box.
6. Mortar and pestle: agate or porcelain.
7. Stereomicroscope, ca. 10 to 45X.
8. Light source: incandescent or fluorescent.
9. Tweezers, dissecting needles, spatulas, probes, and scalpels.
10. Glassine paper or clean glass plate.
11. Low-speed hand drill with coarse burr bit (optional).

SPECIAL PRECAUTIONS: Asbestos, a human carcinogen, should be handled only in an exhaust hood (equipped with a HEPA filter) [2]. Precautions should be taken when collecting unknown samples, which may be asbestos, to preclude exposure to the person collecting the sample and minimize the disruption to the parent material [3]. Disposal of asbestos-containing materials should follow EPA Guidelines [4].

SAMPLING:

1. Place 1 to 10 g of the material to be analyzed in a sample container.
   NOTE: For large samples (i.e., whole ceiling tiles) that are fairly homogenous, a representative small portion should be submitted for analysis. Sample size should be adjusted to ensure that it is representative of the parent material.
2. Make sure that sample containers are taped so they will not open in transit.
3. Ship the samples in a rigid container with sufficient packing material to prevent damage or sample loss.

SAMPLE PREPARATION:

4. Visually examine samples in the container and with a low-magnification stereomicroscope in a hood. (If necessary, a sample may be carefully removed from the container and placed on glassine transfer paper or clean glass plate for examination). Break off a portion of the sample and examine the edges for emergent fibers. Note the homogeneity of the sample. Some hard tiles can be broken, and the edges examined for emergent fibers. If fibers are found, make an estimate of the amount and type of fibers present, confirm fiber type (step 14) and quantify (step 15).

5. In a hood, open sample container and with tweezers remove small, representative portions of the sample.
   a. If there are obvious separable layers, sample and analyze each layer separately.
   b. If the sample appears to be slightly inhomogeneous, mix it in the sample container with tweezers or a spatula before taking the portion of analysis. Alternatively, take small representative portions of each type of material and place on a glass slide.
   c. On hard tiles that may have thin, inseparable layers, use a scalpel to cut through all the layers for a representative sample. Then cut it into smaller pieces after placing RI liquid on it before trying to reduce the thickness. Alternatively, use a low-speed hand drill equipped with a burr bit to remove material from hard tiles. Avoid excessive heating of the sample which may alter the optical properties of the material.
      NOTE: This type of sample often requires ashing or other specialized preparation, and may require transmission electron microscopy for detection of the short asbestos fibers which are characteristic of floor tiles.
   d. If the sample has large, hard particles, grind it in a mortar. Do not grind so fine that fiber characteristics are destroyed.
   e. If necessary, treat a portion of the sample in a hood with an appropriate solvent to remove binders, tars, and other interfering materials which may be present in the sample. Make corrections for the non-asbestos material removed by this process.
      NOTE: Other methods of sample preparation such as acid washing and sodium metaphosphate treatment and ashing may be necessary, especially to detect low concentrations of asbestos. If needed, use as described in Reference [1].
6. After placing a few drops of RI liquid on the slide, put a small portion of sample in the liquid. Tease apart with a needle or smash small clumps with the flat end of a spatula or probe, producing a uniform thickness or particles so that better estimates of projected area percentages can be made. Mix the fibers and particles on the slide so that they are as homogeneous as possible.
   NOTE: An even dispersion of sample should cover the entire area under the cover slip. some practice will be necessary to judge the right amount of material to place on the slide. Too little sample may not give sufficient information and too much sample cannot be easily analyzed.

CALIBRATION AND QUALITY CONTROL:

7. Check for contamination each day of operation. Wipe microscope slides and cover slips with lens paper before using. Check refractive index liquids. Record results in a separate logbook.
8. Verify the refractive indices of the refractive index liquids used once per week of operation. Record these checks in a separate logbook.
9. Follow the manufacturer's instructions for illumination, condenser alignment and other microscope adjustments. Perform these adjustments prior to each sample set.
10. Determine percent of each identified asbestos species by comparison to standard projections (Figure 1) [1]. If no fibers are detected in a homogeneous sample, examine at least two additional preparations before concluding that no asbestos is present.
11. If it appears that the preparation technique might not be able to produce a homogeneous or representative sample on the slide, prepare a duplicate slide and average the results. Occasionally, when the duplicate results vary greatly, it will be necessary to prepare additional replicate slides and average all the replicate results. Prepare duplicate slides of at least 10% of the samples analyzed. Average the results for reporting.
12. Analyze about 5% blind samples of known asbestos content.
13. Laboratories performing this analytical method should participate in the National Voluntary Laboratory Accreditation Program [5] or a similar interlaboratory quality control program. Each analyst should have complete formal training in polarized light microscopy and its application to crystalline materials. In lieu of formal training, laboratory training in asbestos bulk analysis under the direction of a trained asbestos bulk analyst may be substituted. Owing to the subjective nature of the method, frequent practice is essential in order to remain proficient in estimating projected area percentages.

QUALITATIVE ASSESSMENT:

14. Scan the slide to identify any asbestos minerals using the optical properties of morphology, refractive indices, color, pleochroism, birefringence, extinction characteristics, sign of elongation, and dispersion staining characteristics.

    NOTE: Identification of asbestos using polarized light microscopy is unlike most other analytical methods. The quality of the results is dependent on the skill and judgment of the analyst. This method does not lend itself easily to a step-wise approach. Various procedures devised by different analysts may yield equivalent results. The following step-wise procedure repeatedly utilizes the sample preparation procedure previously outlined.

    a. Prepare a slide using 1.550 HD RI liquid. Adjust the polarizing filter such that the polars are partially crossed, with ca. 15° offset. Scan the preparation, examining the morphology for the presence of fibers. If no fibers are found, scan the additional preparations. If no fibers are found in any of the preparations, report that the sample does not contain asbestos, and stop the analysis at this point.

    b. If fibers are found, adjust the polarizing filter such that the polars are fully crossed. If all of the fibers are isotropic (disappear at all angles of rotation) then those fibers are not asbestos. Fibrous glass and mineral wool, which are common components of suspect samples, are isotropic. If only isotropic fibers are found in the additional preparations, report no asbestos fibers detected, and stop the analysis.

    c. If anisotropic fibers are found, rotate the stage to determine the angle of extinction. Except for tremolite-actinolite asbestos which has oblique extinction at 10-20°, the other forms of asbestos exhibit parallel extinction (Table 1). Tremolite may show both parallel and oblique extinction.

    d. Insert the first order red compensator plate in the microscope and determine the sign of elongation. All forms of asbestos have a positive sign of elongation except for crocidolite. If the sign of elongation observed is negative, go to step "g."

       NOTE: To determine the direction of the sign of elongation on a particular microscope configuration, examine a known chrysotile sample and note the direction (NE-SW or NW-SE) of the blue coloration. Chrysotile has a positive sign of elongation.

    e. Remove the first-order red compensator and uncross the polarizer. Examine under plane polarized light for blue and gold-brown Becke colors at the fiber-oil interface (i.e., index of refraction match). Becke colors are not always evident. Examine fiber morphology for twisted, wavy bundles of fibers which are characteristic of chrysotile. Twisted, ribbon-like morphology with cellular internal features may indicate cellulose fibers. It may be necessary to cross the polars partially in order to see the fibers if the index of refraction is an exact match at 1.550. If the fibers appear to have higher index of refraction, go to step "h," otherwise continue.

    f. Identification of chrysotile. Insert the dispersion staining objective. Observation of dispersion staining colors of blue and blue-magenta confirms chrysotile. Cellulose, which is a common interfering fiber at the 1.550 index of refraction, will not exhibit these dispersion staining colors. If chrysotile is found, go to step 15 for quantitative estimation.

    g. Identification of crocidolite. Prepare a slide in 1.700 RI liquid. Examine under plane-polarized light (uncrossed polars); check for morphology of crocidolite. Fibers will be straight, with rigid appearance, and may appear blue or purple-blue. Crocidolite is pleochroic, i.e., it will appear to change its color (blue or gray) as it is rotated through plane polarized light. Insert the dispersion staining objective. The central stop dispersion staining color are red magenta and blue magenta, however, these colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If observations above indicate crocidolite, go to step 15 for quantitative estimation.

    h. Identification of amosite. Prepare a slide in 1.680 RI liquid. Observed the fiber morphology for amosite characteristics: straight fibers and fiber bundles with broom-like or splayed ends. If the morphology matches amosite, examine the fibers using the dispersion staining objective. Blue and pale blue colors indicate the cummingtonite form of amosite, and gold and blue colors indicate the grunerite form of amosite. If amosite is confirmed by this test,

go to step 15 for quantitative estimation, otherwise continue.

i. Identification of anthophyllite-tremolite-actinolite. Prepare a slide in 1.605 HD RI liquid. Examine morphology for comparison to anthophyllite-tremolite-actinolite asbestos. The refractive indices for these forms of asbestos vary naturally within the species. Anthophyllite can be distinguished from actinolite and tremolite by its nearly parallel extinction. Actinolite has a light to dark green color under plane-polarized light and exhibits some pleochroism. For all three, fibers will be straight, single fibers possibly with some larger composite fibers. Cleavage fragments may also be present. Examine using the central stop dispersion staining objective. Anthophyllite will exhibit central stop colors of blue and gold/gold-magenta; tremolite will exhibit pale blue and yellow; and actinolite will exhibit magenta and golden-yellow colors.

NOTE: In this refractive index range, wollastonite is a common interfering mineral with similar morphology including the presence of cleavage fragments. It has both positive and negative sign of elongation, parallel extinction, and central stop dispersion staining colors of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is needed, go to step "j". If any of the above forms of asbestos were confirmed above, go to step 15 for quantitative estimation. If none of the tests above confirmed asbestos fibers, examine the additional preparations and if the same result occurs, report the absence of asbestos in this sample.

j. Wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with cover slip in place, on a warm hot plate until dry. By capillary action, place 1.620 RI liquid under the cover clip and examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show their original dispersion colors.

NOTE: There are alternative analysis procedures to the step-wise approach outlined above which will yield equivalent results. Some of these alternatives are:

i. Perform the initial scan for the presence of asbestos using crossed polars as well as the first-order red compensator. This allows for simultaneous viewing of birefringent and amorphous materials as well as determine their sign of elongation. Some fibers which are covered with mortar may best be observed using this configuration.

ii. Some analysts prefer to mount their first preparation in a RI liquid different than any asbestos materials and conduct their initial examination under plane-polarized light.

iii. If alternative RI liquids are used from those specified, dispersion staining colors observed will also change. Refer to an appropriate reference for the specific colors associated with asbestos in the RI liquids actually used.

QUANTITATIVE ASSESSMENT:

15. Estimate the content of the asbestos type present in the sample using the 1.550 RI preparation. Express the estimate as an area percent of all material present, taking into account the loading and distribution of all sample material on the slide. Use Figure 1 as an aid in arriving at your estimate. If additional unidentified fibers are present in the sample, continue with the qualitative measurement (step 14).

NOTE: Point-counting techniques to determine percentages of the asbestos minerals are not generally recommended. The point-counting method only produces accurate quantitative data when the material on the slide is homogeneous and has a uniform thickness, which is difficult to obtain [6]. The point-counting technique is, recommended by the EPA to determine the amount of asbestos in bulk [1]; however, in the more recent Asbestos Hazard Emergency Response Act (AHERA) regulations, asbestos quantification may be performed by a point-counting or equivalent estimation method [7].

16. Make a quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. If asbestos fibers are identified, report the material as "asbestos-containing". Asbestos content should be reported as a range of percent content. The range reported should be indicative of the analyst's precision in estimating asbestos content. For greater quantities use Figure 1 in arriving at your estimate.

EVALUATION OF METHOD:

The method is compiled from standard techniques used in mineralogy [8-13], and from standard laboratory procedures for bulk asbestos analysis which have been utilized for several years. These techniques have been successfully applied to the analysis of EPA Bulk Sample Analysis Quality Assurance Program samples since 1982 [1,5]. However, no formal evaluation of this method, as written, has been performed.

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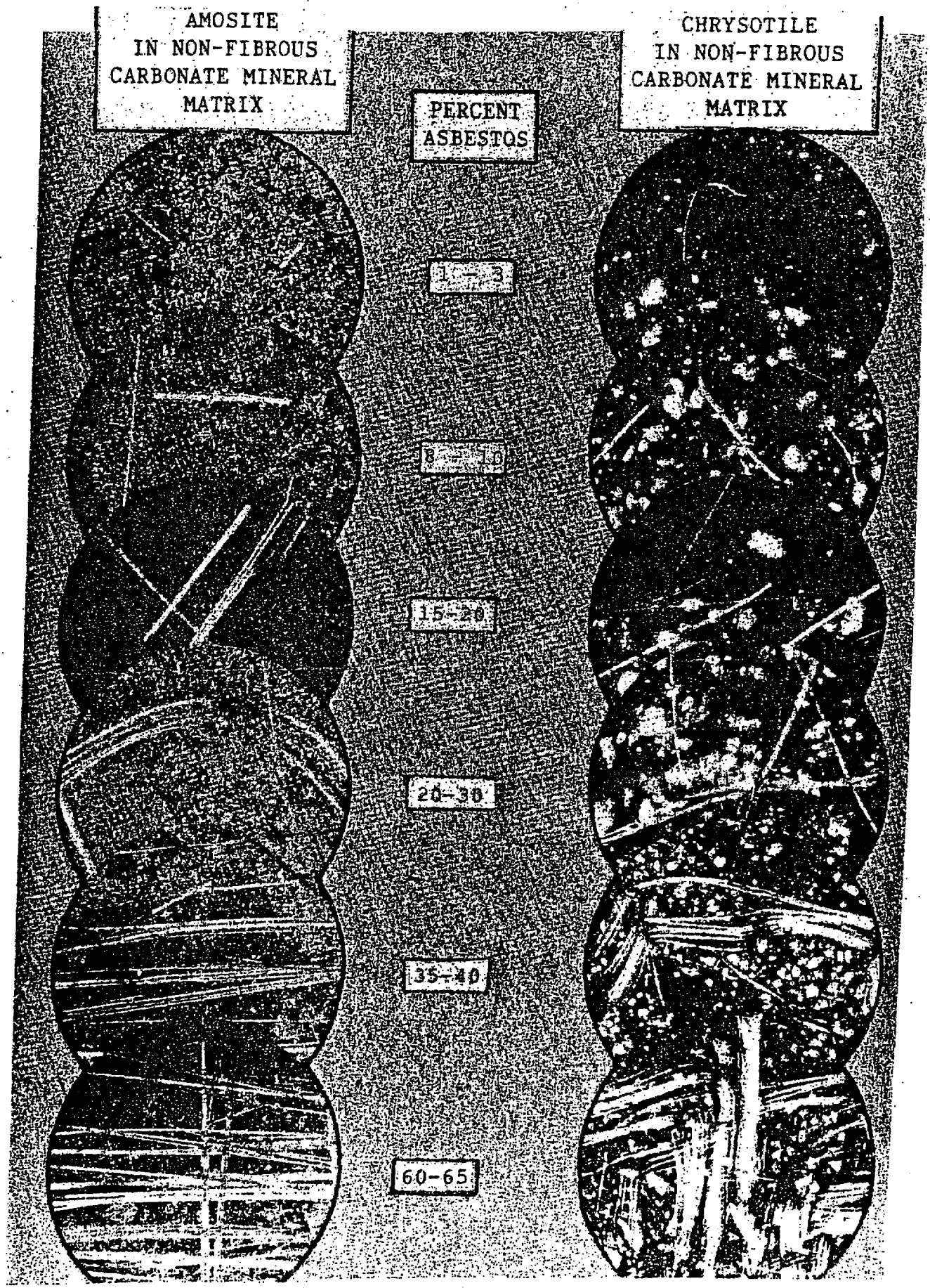
AMOSITE
IN NON-FIBROUS
CARBONATE MINERAL
MATRIX
PERCENT
ASBESTOS
CHRYSOTILE
IN NON-FIBROUS
CARBONATE MINERAL
MATRIX
1 - 3
8 - 10
15-20
20-30
35-40
60-65

Table 1. Optical Properties of Asbestos Fibers

		Refractive Index (Approximate Values)		
Mineral	Morphology and Color	⊥ to Elongation	∥ to Elongation	Birefringence
Chrysotile	Wavy fibers with kinks. Splayed ends on larger bundles. Colorless to light brown upon being heated. Nonpleochroic. Aspect ratio typically >10:1.	1.54	1.55	0.002 - 0.014
Cummingtonite-Grunerite (Amosite)	Straight fibers and fiber bundles. Bundle ends appear broom-like or splayed. Colorless to brown upon heating. May be weakly pleochroic. Aspect ratio typically >10:1.	1.67	1.70	0.02 - 0.03
Crocidolite (Riebeckite)	Straight fibers and fiber bundles. Longer fibers show curvature. Splayed ends on bundles. Characteristic blue color. Pleochroic. Aspect ratio typically >10:1.	1.71	1.70	0.014 - 0.016 Interference colors may be masked by blue color.
Anthophyllite	Straight fibers and fiber bundles. Cleavage fragments may be present. Colorless to light brown. Nonpleochroic to weakly pleochroic. Aspect ratio generally <10:1.	1.61	1.63	0.019 - 0.024
Tremolite-Actinolite	Straight and curved fibers. Cleavage fragments common. Large fiber bundles show splayed ends. Tremolite is colorless. Actinolite is green and weakly to moderately pleochroic. Aspect ratio generally <10:1.	1.60 - 1.62 (tremolite) 1.62 - 1.67 (actinolite)	1.62 - 1.64 (tremolite) 1.64 - 1.68 (actinolite)	0.02 - 0.03

Table 1. Optical Properties of Asbestos Fibers (Continued)

			Central Stop Dispersion Staining Colors		
Mineral	Extinction	Sign of Elongation	RI Liquid	⊥ to Vibration	∥ to Vibration
Chrysotile	Parallel to fiber length	+ (length slow)	1.550 HD	Blue	Blue-magenta
Cummingtonite-Grunerite (Amosite)	Parallel to fiber length	+ (length slow)	1.670 Fibers subjected to high temperatures will not dispersion-stain.	Red magenta to blue	Yellow
Cummingtonite Grunerite			1.680 1.680	pale blue blue	blue gold
Crocidolite (Riebeckite)	Parallel to fiber length	- (length fast)	1.700 1.680	Red magenta yellow	Blue-magenta pale yellow
Anthophyllite	Parallel to fiber length	+ (length slow)	1.605 HD 1.620 HD	Blue Blue-green	Gold to gold-magenta Golden-yellow
Tremolite-Actinolite	Oblique - 10 to 20° for fragments. Some composite fibers show ∥ extinction.	+ (length slow)	1.605 HD	Pale blue (tremolite) Yellow (actinolite)	Yellow (tremolite) Pale yellow (actinolite)

HD = high-dispersion RI liquid series.

Appendix 11

(ISO 10312) Ambient Air - Determination of Asbestos Fibers: Direct - Transfer Transmission Electron Microscopy Method

INTERNATIONAL STANDARD

ISO 10312

First edition
1995-05-01

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

Air ambiant — Détermination des fibres d'amiante — Méthode de microscopie électronique à transmission directe

Reference number
ISO 10312:1995(E)

Variance of the Published Methodology

For the purposes of this study, ISO 10312 will be modified as follows:
Line 9 of paragraph 3 in Section 1.4 will be changed to "The direct analytical technique method cannot be used if the general particulate loading of the sample collection filter exceeds approximately 25 ug/cm^2 of filter surface, which corresponds to approximately 25% coverage of the collection filter by particulate."

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10312 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Annexes A, B, C, D, E and F form an integral part of this International Standard. Annexes G, H and J are for information only.

Introduction

This International Standard is applicable to the determination of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for detailed evaluation of any atmosphere in which asbestos structures are likely to be present. Because the best available medical evidence indicates that the numerical fibre concentration and the fibre sizes are the relevant parameters for evaluation of the inhalation hazards, a fibre counting technique is the only logical approach. Most fibres in ambient atmospheres are not asbestos, and therefore there is a requirement for fibres to be identified. Many airborne asbestos fibres in ambient atmospheres have diameters below the resolution limit of the optical microscope. This International Standard is based on transmission electron microscopy, which has adequate resolution to allow detection of small fibres and is currently the only technique capable of unequivocal identification of the majority of individual fibres of asbestos. Asbestos is often found, not as single fibres, but as very complex, aggregated structures which may or may not be also aggregated with other particles. The fibres found suspended in an ambient atmosphere can often be identified unequivocally, if a sufficient measurement effort is expended. However, if each fibre were to be identified in this way, the analysis would become prohibitively expensive. Because of instrumental deficiencies or because of the nature of the particulate, some fibres cannot be positively identified as asbestos, even though the measurements all indicate that they could be asbestos. Subjective factors therefore contribute to this measurement, and consequently a very precise definition of the procedure for identification and enumeration of asbestos fibres is required. The method specified in this International Standard is designed to provide the best description possible of the nature, numerical concentration, and sizes of asbestos-containing particles found in an air sample. This International Standard is necessarily complex, because the instrumental techniques used are complex, and also because a very detailed and logical procedure must be specified to reduce the subjective aspects of the measurement. The method of data recording specified in this International Standard is designed to allow re-evaluation of the structure counting data as new medical evidence becomes available. All of the feasible specimen preparation techniques result in some modification of the airborne particulate. Even the collection of particles from a three-dimensional airborne dispersion onto a two-dimensional filter surface can be considered a modification of the particulate, and some of the particles in most samples are modified by the specimen preparation procedures. However, the procedures specified in this International Standard are designed to minimize the disturbance of the collected particulate material, and the effect of those disturbances which do occur can be evaluated.

This International Standard describes the method of analysis for a single air filter. However, one of the largest potential errors in characterizing asbestos in ambient atmospheres is associated with the variability between filter samples. For this reason, it is necessary to design a replicate sampling scheme in order to determine this International Standard's accuracy and precision.

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

1 Scope

1.1 Substance determined

This International Standard specifies a reference method using transmission electron microscopy for the determination of the concentration of asbestos structures in ambient atmospheres and includes measurement of the lengths, widths and aspect ratios of the asbestos structures. The method allows determination of the type(s) of asbestos fibres present. The method cannot discriminate between individual fibres of the asbestos and non-asbestos analogues of the same amphibole mineral.

1.2 Type of sample

The method is defined for polycarbonate capillary-pore filters or cellulose ester (either mixed esters of cellulose or cellulose nitrate) filters through which a known volume of air has been drawn. The method is suitable for determination of asbestos in both exterior and building atmospheres.

1.3 Measuring range

The range of concentration which can be determined is 50 structures/mm^2 to 7 000 structures/mm^2 on the filter. The air concentrations represented by these values are a function of the volume of air sampled. There is no lower limit to the dimensions of asbestos fibres which can be detected. In practice, microscopists vary in their ability to detect very small asbestos fibres. Therefore, a minimum length of 0,5 µm has been defined as the shortest fibre to be incorporated in the reported results.

1.4 Limit of detection

The limit of detection theoretically can be lowered indefinitely by filtration of progressively larger volumes of air and by extending the examination of the specimens in the electron microscope. In practice, the lowest achievable limit of detection for a particular area of TEM specimen examined is controlled by the total suspended particulate concentration.

For total suspended particulate concentrations of approximately 10 µg/m^3, corresponding to clean, rural atmospheres, and assuming filtration of 4 000 litres of air, an analytical sensitivity of 0,5 structure/l can be obtained, equivalent to a limit of detection of 1,8 structure/l, if an area of 0,195 mm^2 of the TEM specimens is examined. If higher total suspended particulate concentrations are present, the volume of air filtered must be reduced in order to maintain an acceptable particulate loading on the filter, leading to a proportionate increase in the analytical sensitivity.

Where this is the case, lower limits of detection can be achieved by increasing the area of the TEM specimens that is examined. In order to achieve lower limits of detection for fibres and bundles longer than 5 µm, and for PCM equivalent fibres, lower magnifications are specified which permit more rapid examination of larger areas of the TEM specimens when the examination is limited to these dimensions of fibre. The direct analytical method cannot be used if the general particulate loading of the sample collection filter exceeds approximately 10 µg/cm^2 of filter surface, which corresponds to approximately 10 % coverage of the collection filter by particulate. If the total suspended particulate is largely organic material, the limit of detection can be lowered significantly by using an indirect preparation method.

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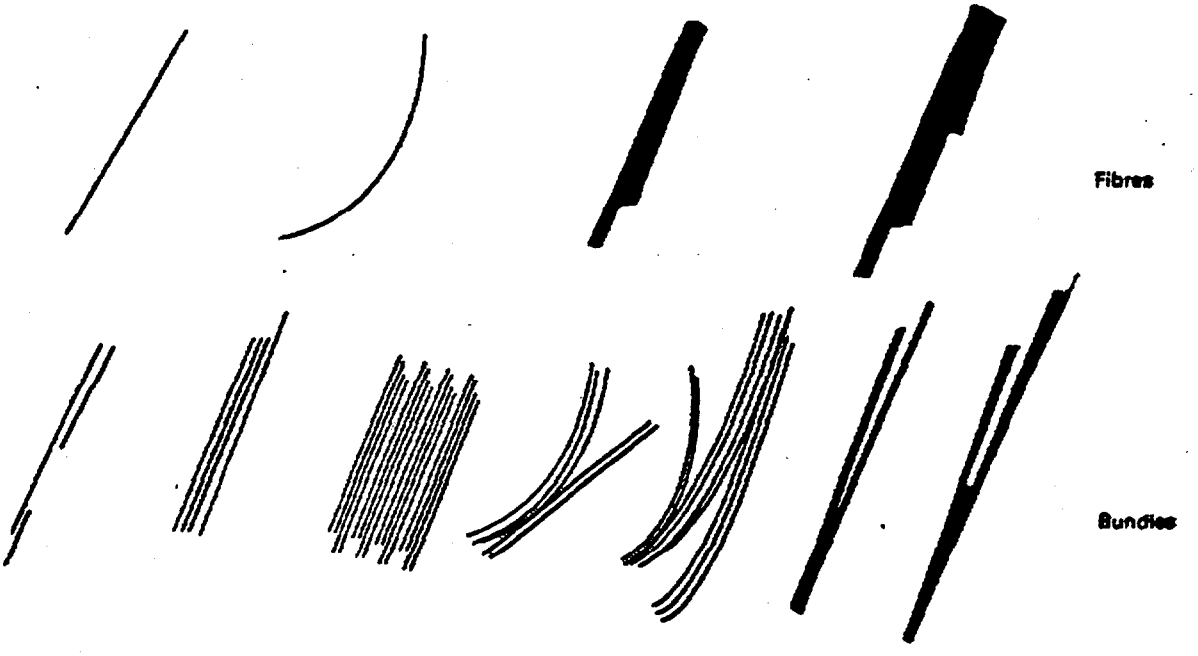
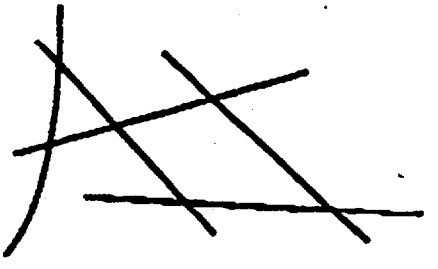
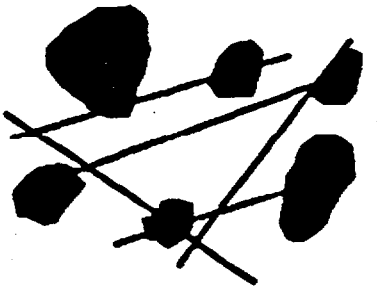


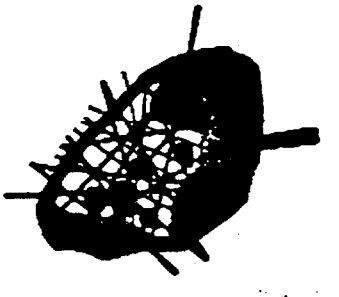


a) Disperse cluster (type D)

b) Compact cluster (type C)



c) Disperse matrix (type D)

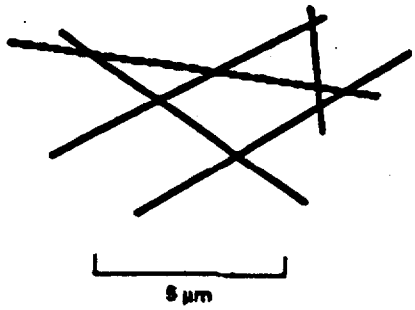


d) Compact matrix (type C)

Figure C.1 — Fundamental morphological structure types

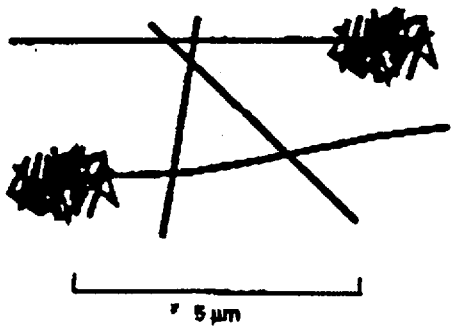
Count as 1 compact cluster containing more than 9 fibres (all fibres shorter than 5 µm)

Record as CC+0



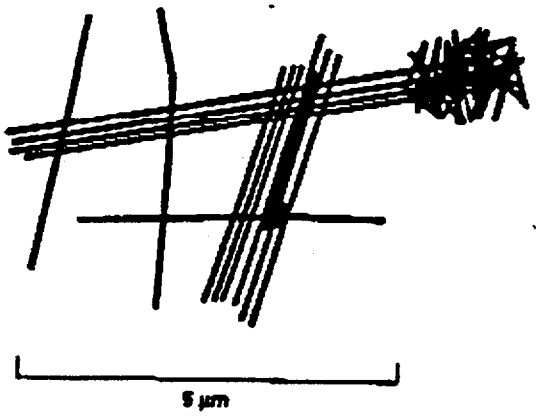
Count as 1 disperse cluster consisting of 5 fibres, 4 of which are longer than 5 µm

Record as CD54, followed by 5 fibres, each recorded as CF



Count as 1 disperse cluster consisting of 4 fibres, 2 of which are longer than 5 µm, and 2 cluster residuals, each containing more than 9 fibres

Record as CD+2, followed by 4 fibres, each recorded as CF, and 2 cluster residual, each recorded as CR+0



Count as 1 disperse cluster consisting of 3 fibres, 2 bundles, 1 of which is longer than 5 µm, and 1 cluster residual containing more than 9 fibres

Record as CD+1, followed by 3 fibres, each recorded as CF, 2 bundles, each recorded as CB, and 1 cluster residual recorded as CR+0

Figure C.2 — Examples of recording of complex asbestos clusters

C.2.4 Matrix

One or more fibres, or fibre bundles, may be attached to, or partially concealed by, a single particle or group of overlapping nonfibrous particles. This structure shall be defined as a matrix. The TEM image does not discriminate between particles which are attached to fibres, and those which have by chance overlapped in the TEM image. It is not known, therefore, whether such a structure is actually a complex particle, or whether it has arisen by a simple overlapping of particles and fibres on the filter.

Since a matrix structure may involve more than one fibre, it is important to define in detail how matrices shall be counted. Matrices exhibit different characteristics, and two types can be defined.

C.2.4.1 disperse matrix (type D): Structure consisting of a particle or linked group of particles, with overlapping or attached fibres or bundles in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured.

C.2.4.2 compact matrix (type C): Structure consisting of a particle or linked group of particles, in which fibres or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, matrices can occur in which the characteristics of both types of matrix occur in the same structure. Where this occurs, the structure should be assigned as a disperse matrix, and then a logical procedure should be followed by recording structure components according to the counting criteria. Examples of the procedure which shall be followed are shown in figure C.3.

C.2.5 Asbestos structure larger than 5 µm

Any fibre, bundle, cluster or matrix for which the largest dimension exceeds 5 µm. Asbestos structures larger than 5 µm do not necessarily contain asbestos fibres or bundles longer than 5 µm.

C.2.6 Asbestos fibre or bundle longer than 5 µm

An asbestos fibre of any width, or bundle of such fibres, which has a length exceeding 5 µm.

C.2.7 PCM equivalent structure

Any fibre, bundle, cluster or matrix with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. PCM equivalent structures do not necessarily contain fibres or bundles longer than 5 µm, or PCM equivalent fibres.

C.2.8 PCM equivalent fibre

Any particle with parallel or stepped sides, with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. For chrysotile, PCM equivalent fibres will always be bundles.

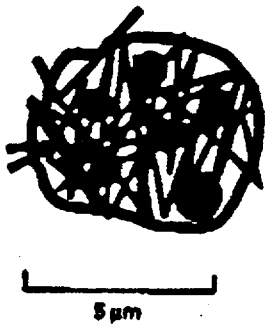
C.3 Other structure counting criteria

C.3.1 Structures which intersect grid bars

A structure which intersects a grid bar shall only be counted on two sides of the grid opening, as illustrated in figure C.4. Record the dimensions of the structure such that the obscured portions of components are taken to be equivalent to the unobscured portions, as shown by the broken lines in figure C.4. For example, the length of a fibre intersecting a grid bar is taken to be twice the unobscured length. Structures intersecting either of the other two sides shall not be included in the count.

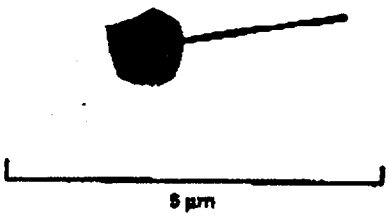
C.3.2 Fibres which extend outside the field of view

During scanning of a grid opening, count fibres which extend outside the field of view systematically, so as to avoid double-counting. In general, a rule should be established so that fibres extending outside the field of view in only two quadrants are counted. The procedure is illustrated by figure C.5. Measure the length of each of these fibre by moving the specimen to locate the other end of the fibre, and then return to the original field of view before continuing to scan the specimen. Fibres without terminations within the field of view shall not be counted.



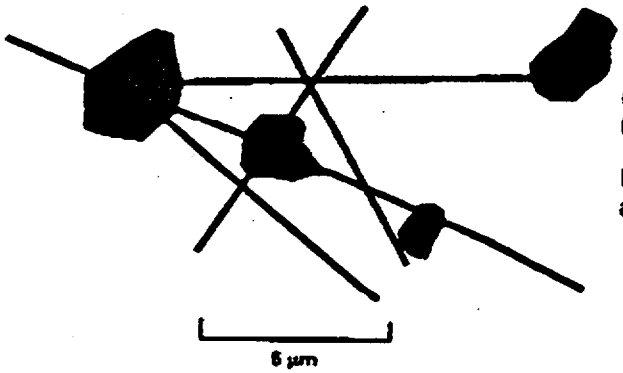
Count as 1 compact matrix, with all fibres shorter than 5 µm

Record as MC+0



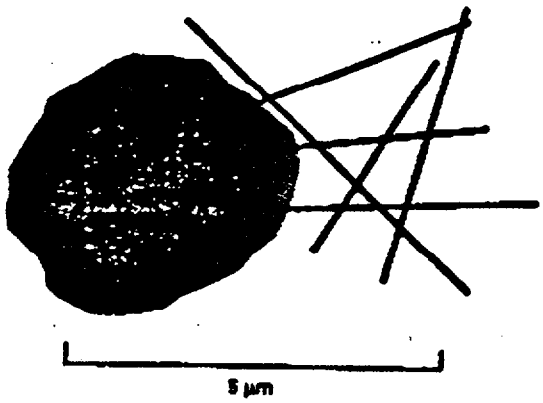
Count as 1 disperse matrix consisting of 1 fibre shorter than 5 µm

Record as MD10, followed by 1 fibre recorded as MF



Count as 1 disperse matrix consisting of 5 fibres, all longer than 5 µm

Record as MD55, followed by 5 fibres, each recorded as MF



Count as 1 disperse matrix, consisting if 3 fibres, 1 of which is longer than 5 µm, and 1 matrix residual containing 3 fibres

Record as MD61, followed by 3 fibres, each recorded as MF, and 1 matrix residual recorded as MR30

Figure C.3 — Examples of recording of complex asbestos matrices

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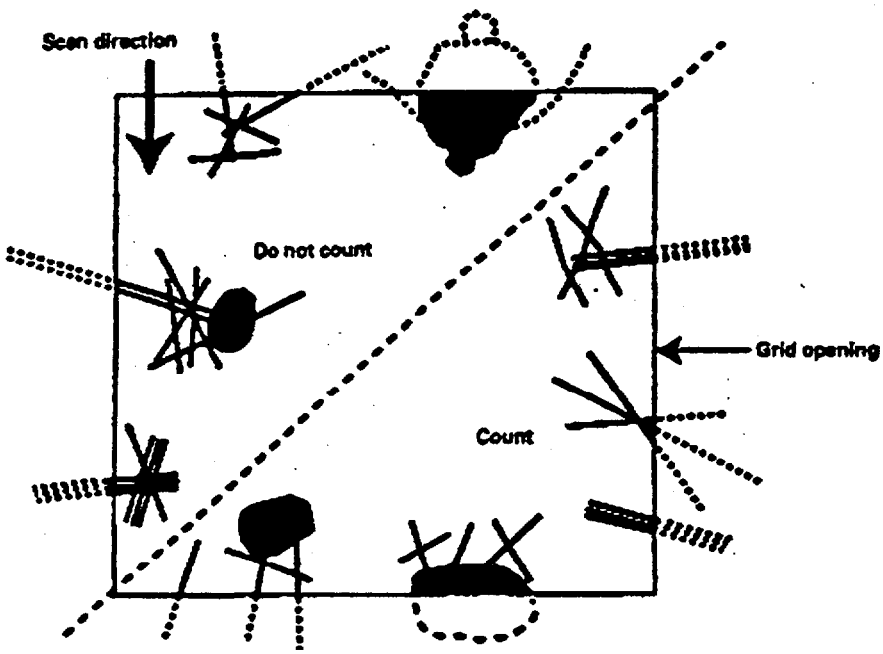


Figure C.4 — Example of counting of structures which intersect grid bars

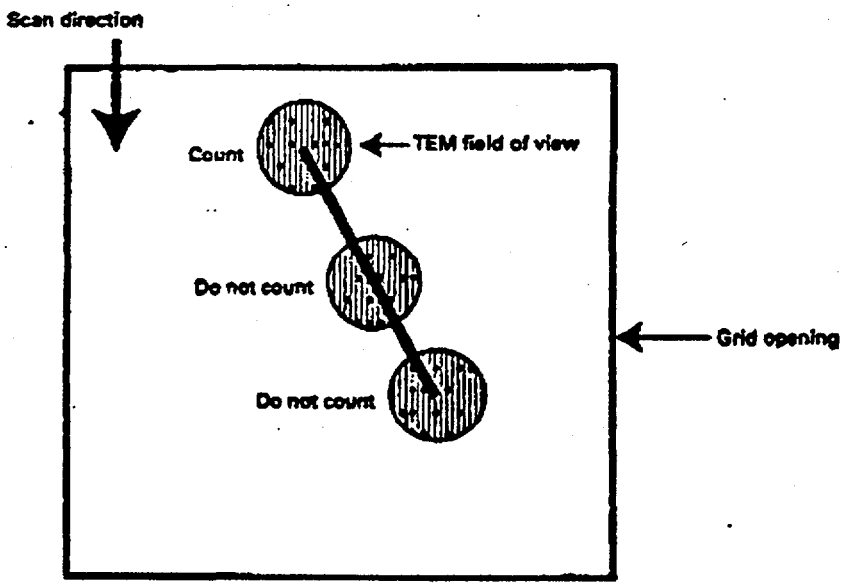


Figure C.5 — Example of counting of fibres which extend outside the field of view

C.4 Procedure for data recording

C.4.1 General

The morphological codes specified are designed to facilitate computer data processing, and to allow recording of a complete representation of the important features of each asbestos structure. The procedure requires that the microscopist classify each primary fibrous structure into one of the four fundamental categories: fibres, bundles, clusters and matrices.

C.4.2 Fibres

On the structure counting form, a fibre as defined in C.2.1 shall be recorded by the designation "F". If the fibre is a separately-counted part of a cluster or matrix, the fibre shall be recorded by the designation "CF", or "MF", depending on whether it is a component of a cluster or matrix.

C.4.3 Bundles

On the structure counting form, a bundle as defined in C.2.2 shall be recorded by the designation "B". If the bundle is a separately-counted part of a cluster or matrix, the bundle shall be recorded by the designation "CB", or "MB", depending on whether it is a component of a cluster or matrix.

C.4.4 Disperse clusters (type D)

On the structure counting form, an isolated cluster of type D as defined in C.2.3 shall be recorded by the designation "CD", followed by a two-digit number. The first digit represents the analyst's estimate of the total number of fibres and bundles comprising the structure. The digit shall be from 1 to 9, or designated as "+" if there are estimated to be more than 9 component fibres or bundles. The second digit shall represent, in the same manner, the total number of fibres and bundles longer than 5 µm contained in the structure. The overall dimensions of the cluster, in two perpendicular directions representing the maximum dimensions, shall be recorded. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "CF" (cluster fibre) and "CB" (cluster bundle). If, after accounting for prominent component fibres and bundles, a group of clustered fibres remains, this shall be recorded by the designation "CR" (cluster residual). If the remaining clustered fibres are present as more than one localized group, it may be necessary to record more than one cluster residual. Do not record more than 5 cluster residuals for any cluster. A cluster residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall cluster. Optionally, if the number of component fibres and bundles in either the original cluster or the cluster residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.5 Compact clusters (type C)

On the structure counting form, an isolated cluster of type C as defined in C.2.3 shall be recorded by the designation "CC", followed by a two-digit number. The two-digit number describing the numbers of component fibres and bundles shall be assigned in the same manner as for clusters of type D. The overall dimensions of the cluster in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact clusters cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.6 Disperse matrices (type D)

On the structure counting form, an isolated matrix of type D as defined in C.2.4 shall be recorded by the designation "MD", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "MF" (matrix fibre) and "MB" (matrix bundle). If after accounting for prominent component fibres and bundles, matrix material containing asbestos fibres remains, this shall be recorded by the designation "MR" (matrix residual). If the remaining matrix fibres are present as more than one localized group, it may be necessary to record more than one matrix residual. Do not record more than 5 matrix residuals for any matrix. A matrix residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall matrix. Optionally, if the number of component fibres or bundles in either the original matrix or the matrix residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.7 Compact matrices (type C)

On the structure counting form, an isolated matrix of type C as defined in C.2.4 shall be recorded by the

designation "MC", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact matrices cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.8 Procedure for recording of partially obscured fibres and bundles

The proportion of the length of a fibre or bundle that is obscured by other particulates shall be used as the basis for determining whether a fibre or bundle is to be recorded as a separate component or is to be considered as a part of a matrix of type C or part of a matrix residual. If the obscured length could not possibly be more than one-third of the total length, the fibre or bundle shall be considered a prominent feature to be separately recorded. The assigned length for each such partially obscured fibre or bundle shall be equal to the visible length plus the maximum possible contribution from the obscured portion. Fibres or bundles which appear to cross the matrix, and for which both ends can be located approximately, shall be included in the maximum of 5 and recorded according to the counting criteria as separate fibres or bundles. If the obscured length could be more than one third of the total length, the fibre or bundle shall be considered as a part of a compact matrix of type C or part of a matrix residual.

C.5 Special considerations for counting of PCM equivalent structures

Use 3/1 as the minimum aspect ratio for counting of PCM equivalent structures. This aspect ratio definition is required in order to achieve comparability of the results for this size range of structure with historical optical measurements, but use of this aspect ratio definition does not significantly affect the ability to interpret the whole fibre size distribution in terms of a minimum 5/1 aspect ratio. Some applications may require that a count be made of PCM equivalent structures only. The coding system permits discrimination between PCM equivalent structures that contain fibres and bundles longer than 5 µm and those that do not.

NOTE 16 In general, clusters and matrices will yield fewer components as the minimum dimensions specified for countable fibres are increased. Thus, it may be found that a particular structure yields a higher number of component fibres and bundles in a count for all fibre sizes than it does at a reduced magnification when only fibres and bundles longer than 5 µm are being counted. However, the requirement that component fibres and bundles be recorded in decreasing length order ensures that the data are consistent for a particular structure, regardless of the size category of fibres being counted and the magnification in use.

Annex D
(normative)

Fibre identification procedure

D.1 General

The criteria used for identification of asbestos fibres may be selected depending on the intended use of the measurements. In some circumstances, there can be a requirement that fibres shall be unequivocally identified as a specific mineral species. In other circumstances, there can be sufficient knowledge about the sample, so that rigorous identification of each fibre need not be carried out. The time required to perform the analysis, and therefore the cost of analysis, can vary widely depending on the identification criteria considered which are to be sufficiently definitive. The combination of criteria considered definitive for identification of fibres in a particular analysis shall be specified before the analysis is made, and this combination of criteria shall be referred to as the "level" of analysis. Various factors related to instrumental limitations and the character of the sample may prevent satisfaction of all of the specified fibre identification criteria for a particular fibre. Therefore, a record shall be made of the identification criteria which were satisfied for each suspected asbestos fibre included in the analysis. For example, if both ED and EDXA were specified to be attempted for definitive identification of each fibre, fibres with chrysotile morphology which, for some reason, do not give an ED pattern but which do yield an EDXA spectrum corresponding to chrysotile, are categorized in a way which conveys the level of confidence to be placed in the identification.

D.2 ED and EDXA techniques

D.2.1 General

Initially, fibres are classified into two categories on the basis of morphology: those fibres with tubular morphology, and those fibres without tubular morphology. Further analysis of each fibre is conducted using ED and EDXA methods. The following procedures should be used when fibres are examined by ED and EDXA.

The crystal structures of some mineral fibres, such as chrysotile, are easily damaged by the high current densities required for EDXA examination. Therefore, investigation of these sensitive fibres by ED should be completed before attempts are made to obtain EDXA spectra from the fibres. When more stable fibres, such as the amphiboles, are examined, EDXA and ED may be used in either order.

D.2.2 ED techniques

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel with the incident electron beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a "zone-axis ED pattern". In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but is is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone-axis. The angle between the two zone-axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when

the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or to be recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focussed in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it will be necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, an internal calibration standard shall be used. A thin coating of gold, or another suitable calibration material, shall be applied to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

If a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in the references by J.A. Gard [11] P.B. Hirsch *et al* [14] and H.R. Wenck [42] included in annex J. Not all zone-axis patterns which can be obtained are definitive. Only those which have closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d-spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the first gold diffraction ring (111), and that patterns with smaller distances between reflections are usually the most definitive.

Five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern shall be selected for measurement, as shown in figure D.1. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very overexposed, it does not provide a well-defined origin for these measurements. The required distances shall therefore be obtained by measuring between pairs of spots symmetrically disposed about the centre spot, preferably separated by several repeat distances. The distances shall be measured with a precision of better than 0,3 mm, and the angles to a precision of better than 2,5°. The diameter of the first or second ring of the calibration pattern (111 and 200) shall also be measured with a precision of better than 0,3 mm.

Using gold as the calibration material, the radius-based camera constant is given by

$$\lambda L = 0{,}117\,74D \text{ mm·nm (first ring)}$$

$$\lambda L = 0{,}101\,97D \text{ mm·nm (second ring)}$$

D.2.3 EDXA measurements

Interpretation of the EDXA spectrum may be either qualitative or quantitative. For qualitative interpretation of a spectrum, the X-ray peaks originating from the elements in the fibre are recorded. For quantitative interpretation, the net peak areas, after background subtraction, are obtained for the X-ray peaks originating from the elements in the fibre. This method provides quantitative interpretation for those minerals which contain silicon.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4225:1994, *Air quality — General aspects — Vocabulary.*

ISO 4226:1993, *Air quality — General aspects — Units of measurement.*

ISO Standard Handbook No. 2:1993, *Quantities and units.*

ISO Standard Handbook No. 3:1989, *Statistical Methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply (see also ISO 4225).

3.1 acicular: The shape of an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like.

3.2 amphibole: A group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, with the nominal formula:

$$A_0 \text{ or } {}_1B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where

A = K, Na

B = Fe^{2+}, Mn, Mg, Ca, Na

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+}

T = Si, Al, Cr, Fe^{3+}, Ti

In some varieties of amphibole, these elements can be partially substituted by Li, Pb or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

3.3 amphibole asbestos: Amphibole in an asbestiform habit.

3.4 analytical sensitivity: The calculated airborne asbestos structure concentration in asbestos structures/litre, equivalent to counting of one asbestos structure in the analysis. The method in this International Standard does not specify an analytical sensitivity.

3.5 asbestiform: A specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility.

3.6 asbestos: A term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, strong fibres when crushed or processed. The Chemical Abstracts Service Registry Numbers of the most common asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grünerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

3.7 asbestos structure: A term applied to any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles.

3.8 aspect ratio: The ratio of length to width of a particle.

3.9 blank: A structure count made on TEM specimens prepared from an unused filter, to determine the background measurement.

3.10 camera length: The equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action.

3.11 chrysotile: A fibrous mineral of the serpentine group which has the nominal composition

$$Mg_3Si_2O_5(OH)_4$$

Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

3.12 cleavage: The breaking of a mineral along one of its crystallographic directions.

3.13 cleavage fragment: A fragment of a crystal that is bounded by cleavage faces.

3.14 cluster: A structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping.

3.15 d-spacing: The distance between identical adjacent and parallel planes of atoms in a crystal.

3.16 electron diffraction: A technique in electron microscopy by which the crystal structure of a specimen is examined.

3.17 electron scattering power: The extent to which a thin layer of substance scatters electrons from their original directions.

3.18 energy dispersive X-ray analysis: Measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyser system.

3.19 eucentric: The condition when the area of interest of an object is placed on a tilting axis at the intersection of the electron beam with that axis and is in the plane of focus.

3.20 field blank: A filter cassette which has been taken to the sampling site, opened, and then closed. Such a filter is used to determine the background structure count for the measurement.

3.21 fibril: A single fibre of asbestos, which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.22 fibre: An elongated particle which has parallel or stepped sides. For the purposes of this International Standard, a fibre is defined to have an aspect ratio equal to or greater than 5:1 and a minimum length of 0,5 µm.

3.23 fibre bundle: A structure composed of parallel, smaller diameter fibres attached along their lengths. A fibre bundle may exhibit diverging fibres at one or both ends.

3.24 fibrous structure: A fibre, or connected grouping of fibres, with or without other particles.

3.25 habit: The characteristic crystal growth form, (or combination of these forms), of a mineral, including characteristic irregularities.

3.26 limit of detection: The calculated airborne asbestos structure concentration in structures per litre, equivalent to counting 2,99 asbestos structures in the analysis.

3.27 matrix: A structure in which one or more fibres, or fibre bundles, touch, are attached to, or partially concealed by, a single particle or connected group of nonfibrous particles.

3.28 Miller index: A set of either three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes.

3.29 PCM equivalent fibre: A fibre of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.30 PCM equivalent structure: A fibrous structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0.2 µm and 3,0 µm.

3.31 primary structure: A fibrous structure that is a separate entity in the TEM image.

3.32 replication: A procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

3.33 selected area electron diffraction: A technique in electron microscopy in which the crystal structure of a small area of a sample is examined.

3.34 serpentine: A group of common rock-forming minerals having the nominal formula

$$Mg_3Si_2O_5(OH)_4$$

3.35 structure: A single fibre, fibre bundle, cluster or matrix.

3.36 twinning: The occurrence of crystals of the same species joined together at a particular mutual orientation, such that the relative orientations are related by a definite law.

3.37 unopened fibre: An asbestos fibre bundle of large diameter which has not been separated into its constituent fibrils or fibres.

3.38 zone-axis: The line or crystallographic direction through the centre of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone.

4 Principle

A sample of airborne particulate is collected by drawing a measured volume of air through either a

capillary-pore polycarbonate membrane filter of maximum pore size 0,4 µm or a cellulose ester (either mixed esters of cellulose or cellulose nitrate) membrane filter of maximum pore size 0,45 µm by means of a battery-powered or mains-powered pump. TEM specimens are prepared from polycarbonate filters by applying a thin film of carbon to the filter surface by vacuum evaporation. Small areas are cut from the carbon-coated filter, supported on TEM specimen grids, and the filter medium is dissolved away by a solvent extraction procedure. This procedure leaves a thin film of carbon which bridges the openings in the TEM specimen grid, and which supports each particle from the original filter in its original position. Cellulose ester filters are chemically treated to collapse the pore structure of the filter, and the surface of the collapsed filter is then etched in an oxygen plasma to ensure that all particles are exposed. A thin film of carbon is evaporated onto the filter surface and small areas are cut from the filter. These sections are supported on TEM specimen grids and the filter medium is dissolved away by a solvent extraction procedure.

The TEM specimen grids from either preparation method are examined at both low and high magnifications to check that they are suitable for analysis before carrying out a quantitative structure count on randomly-selected grid openings. In the TEM analysis, electron diffraction (ED) is used to examine the crystal structure of a fibre, and its elemental composition is determined by energy dispersive X-ray analysis (EDXA). For a number of reasons, it is not possible to identify each fibre unequivocally, and fibres are classified according to the techniques which have been used to identify them. A simple code is used to record, for each fibre, the manner in which it was classified. The fibre classification procedure is based on successive inspection of the morphology, the electron diffraction pattern for a selected area, and the qualitative and quantitative energy dispersive X-ray analyses. Confirmation of the identification of chrysotile is done only by quantitative ED, and confirmation of amphibole is done only by quantitative EDXA and quantitative zone axis ED.

In addition to isolated fibres, ambient air samples often contain more complex aggregates of fibres, with or without other particles. Some particles are composites of asbestos fibres with other materials. Individual fibres and structures that are more complex are referred to as "asbestos structures". A coding system is used to record the type of fibrous structure, and to provide the optimum description of each of these complex structures. The two codes remove the requirement to interpret the structure counting data from the microscopist, and allow this evaluation to be made later without the requirement for re-examination of the TEM specimens. Several levels of analysis are specified, the higher levels providing a more rigorous approach to the identification of fibres. The procedure permits a minimum required fibre identification criterion to be defined on the basis of previous knowledge, or lack of it, about the particular sample. Attempts are then made to achieve this minimum criterion for each fibre, and the degree of success is recorded for each fibre. The lengths and widths of all classified structures and fibres are recorded. The number of asbestos structures found on a known area of the microscope sample, together with the equivalent volume of air filtered through this area, is used to calculate the airborne concentration in asbestos structures/litre of air.

5 Symbols of units and abbreviations

5.1 Symbols of units (see also ISO 4226 and ISO No. 2)

eV = electron volt

kV = kilovolt

l/min = litres per minute

µg = microgram (10^{-6} gram)

µm = micrometre (10^{-6} metre)

nm = nanometre (10^{-9} metre)

W = watt

5.2 Abbreviations

DMF	Dimethylformamide
DE	Electron diffraction
EDXA	Energy dispersive X-ray analysis
FWHM	Full width, half maximum
HEPA	High efficiency particle absolute
MEC	Mixed esters of cellulose
PC	Polycarbonate
PCM	Phase contrast optical microscopy
SAED	Selected area electron diffraction
SEM	Scanning electron microscope
STEM	Scanning transmission electron microscope
TEM	Transmission electron microscope

UICC Union Internationale Contre le Cancer

6 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water (6.1).

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

6.1 water, fibre-free.

A supply of freshly distilled, fibre-free water, or another source of fibre-free, pyrogen-free water shall be used.

6.2 Chloroform, analytical grade, distilled in glass, preserved with 1 % (*V/V*) ethanol.

6.3 1-Methyl-2-pyrrolidone.

6.4 Dimethylformamide.

6.5 Glacial acetic acid.

6.6 Acetone.

7 Apparatus

7.1 Air sampling — Equipment and consumable supplies

7.1.1 Filter cassette

Field monitors, comprising 25 mm to 50 mm diameter three-piece cassettes, with cowls which project less than 2 cm in front of the filter surface shall be used for sample collection. The cassette shall be loaded with either a capillary pore polycarbonate filter of maximum pore size 0,4 µm or an MEC or cellulose nitrate filter of maximum pore size 0,45 µm. Either type of filter shall be backed by a 5 µm pore size MEC or cellulose nitrate filter, and supported by a cellulose back-up pad. When the filters are in position, an elastic cellulose band or adhesive tape shall be applied to prevent air leakage. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly, so that significant air leakage around the filter cannot occur.

Representative filters from the filter lot shall be analysed as specified in 9.7 for the presence of asbestos structures before any are used for air sample collection.

7.1.2 Sampling pump

The sampling pump shall be capable of a flow-rate sufficient to achieve the desired analytical sensitivity. The face velocity through the filter shall be between 4,0 cm/s and 25,0 cm/s. The sampling pump used shall provide a non-fluctuating airflow through the filter, and shall maintain the initial volume flow-rate to within ± 10 % throughout the sampling period. A constant flow or critical orifice controlled pump meets these requirements. Flexible tubing shall be used to connect the filter cassette to the sampling pump. A means for calibration of the flow-rate of each pump is also required.

7.1.3 Stand

A stand shall be used to hold the filter cassette at the desired height for sampling, and shall be isolated from the vibrations of the pump (7.1.2).

7.1.4 Variable area flowmeter

A calibrated variable are a flowmeter with a range of approximately 1 l/min to 10 l/min is required for calibration of the air sampling system.

The variable area flowmeter shall be cleaned before use to avoid transfer of asbestos contamination from the flowmeter to the sample being collected.

7.2 Specimen preparation laboratory

Asbestos, particularly chrysotile, is present in varying quantities in many laboratory reagents. Many building materials also contain significant amounts of asbestos or other mineral fibres which may interfere with the analysis if they are inadvertently introduced during preparation of specimens. It is most important to ensure that, during preparation, contamination of TEM specimens by any extraneous asbestos fibres is minimized. All specimen preparation steps shall therefore be performed in an environment where contamination of the sample is minimized. The primary requirement of the sample preparation laboratory is that a blank determination shall yield a result which will meet the requirements specified in 9.7. A minimum facility considered suitable for preparation of TEM specimens is a laminar flow hood with positive pressure. However, it has been established that work practices in specimen preparation appear to be more important than the tape of clean handling facilities in use. Preparation of samples shall be carried out only after acceptable blank values have been demonstrated.

NOTE 1 It is recommended that activities involving manipulation of bulk asbestos samples not be performed in the

same area as TEM specimen preparation, because of the possibilities of contaminating the TEM specimens.

7.3 Equipment for analysis

7.3.1 Transmission electron microscope

A TEM operating at an accelerating potential of 80 kV to 120 kV, with a resolution better than 1,0 nm, and a magnification range of approximately × 300 to × 100 000 shall be used. The ability to obtain a direct screen magnification of about × 100 000 is necessary for inspection of fibre morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. It is also required that the viewing screen of the microscope be calibrated such that the lengths and widths of fibre images down to 1 mm width can be measured in increments of 1 mm, regardless of image orientation. This requirement is often fulfilled through the use of a fluorescent screen with calibrated gradations in the form of circles, as shown in figure 1.

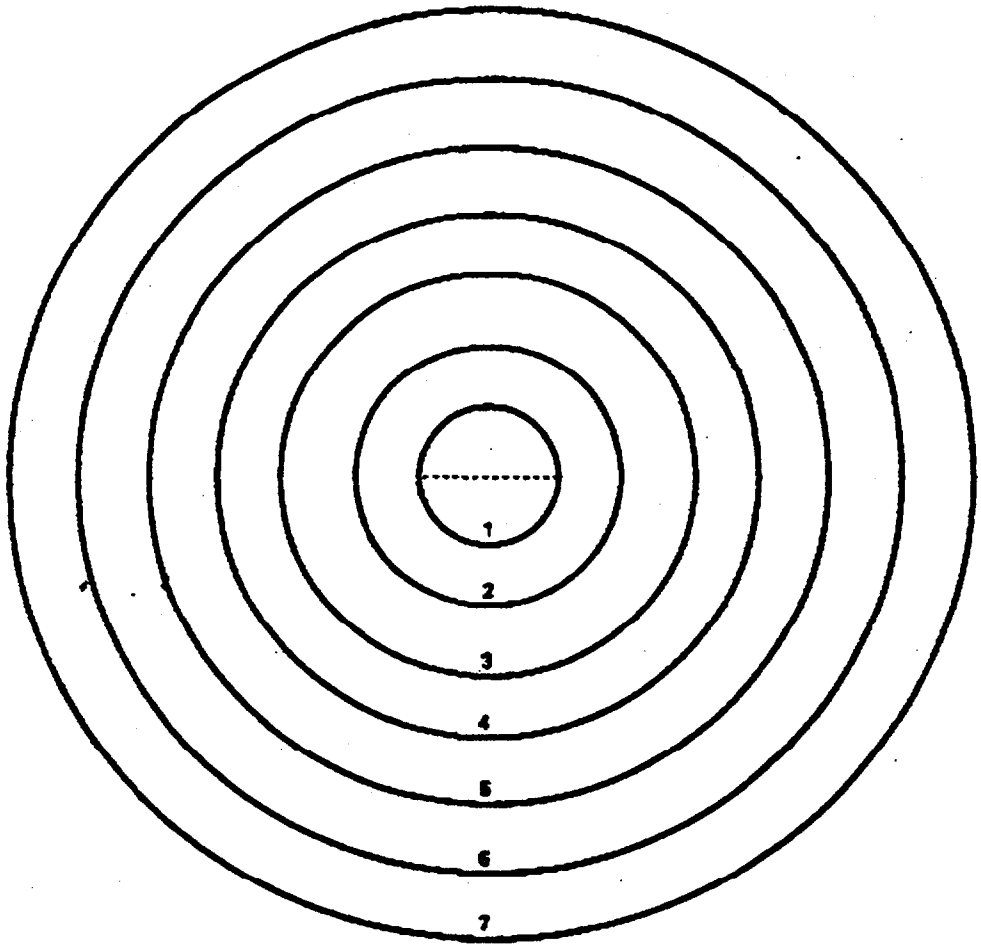


Figure 1 — Example of calibration markings on TEM viewing screen

For Bragg angles less than 0,01 rad, the TEM shall be capable of performing ED from an area of 0,6 μm^2 or less, selected from an in-focus image at a screen magnification of × 20 000. This performance requirement defines the minimum separation between particles at which independent ED patterns can be obtained from each particle. If SAED is used, the performance of a particular instrument may normally be calculated using the following equation

$$A = 0{,}785\,4 \times \left(\frac{D}{M} + 2\,000 C_s \theta^3\right)^2$$

where

- A is the effective SAED area, in square micrometres;
- D is the diameter, in micrometres, of the SAED aperture;
- M is the magnification of the objective lens;
- C_s is the spherical aberration coefficient, in millimetres, of the objective lens;
- θ is the maximum required Bragg angle, in radians.

It is not possible to reduce the effective SAED area indefinitely by the use of progressively smaller SAED apertures, because there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

If zone-axis ED analyses are to be performed, the TEM shall incorporate a goniometer stage which permits the TEM specimen to be either

a) rotated through 360°, combined with tilting through at least + 30° to − 30° about an axis in the plane of the specimen;

b) tilted through at least + 30° to − 30° about two perpendicular axes in the plane of the specimen.

The analysis is greatly facilitated if the goniometer permits eucentric tilting, although this is not essential. If EDXA and zone-axis ED are required on the same fibre, the goniometer shall be of a type which permits tilting of the specimen and acquisition of EDXA spectra without changing the specimen holder.

The TEM shall have an illumination and condenser lens system capable of forming an electron probe of diameter less than 250 nm.

NOTE 2 Use of an anti-contamination trap around the specimen is recommended if the required instrumental performance is to be obtained.

7.3.2 Energy dispersive X-ray analyser

The TEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 180 eV (FWHM) on the MnKα. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid state X-ray detectors are least sensitive in the low energy region, and so measurement of sodium in crocidolite shall be the performance criterion. The combination of electron microscope and X-ray analyser shall yield, under routine analytical conditions, a background-subtracted NaKα integrated peak count rate of more than 1 count per second (cps) from a fibre of UICC crocidolite, 50 nm in diameter or smaller, when irradiated by an electron probe of 250 nm diameter or smaller at an accelerating potential of 80 kV. The peak/background ratio for this performance test shall exceed 1,0.

The EDXA unit shall provide the means for subtraction of the background, identification of elemental peaks, and calculation of background-subtracted peak areas.

7.3.3 Computer

Many repetitive numerical calculations are necessary, and these may be performed conveniently by relatively simple computer programmes. For analyses of zone-axis ED pattern measurements, a computer with adequate memory is required to accommodate the more complex programmes involved.

7.3.4 Plasma asher

For preparation of TEM specimens from MEC filters, a plasma asher, with a radio frequency power rating of 50 W or higher, shall be used to etch the surface of collapsed MEC filters. The asher shall be supplied with a controlled oxygen flow, and shall be modified, if necessary, to provide a valve to control the speed of air admission so that rapid air admission does not disturb particulates from the surface of the filter after the etching step.

NOTE 3 It is recommended that filters be fitted to the oxygen supply and the air admission line.

7.3.5 Vacuum coating unit

A vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon on the membrane filters. A sample

holder is required which will allow a glass microscope slide to be continuously rotated during the coating procedure.

NOTE 4 A mechanism which also allows the rotating slide to be tilted through an angle of approximately 45° during the coating procedure is recommended. A liquid nitrogen cold trap above the diffusion pump may be used to minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum coating unit may also be used for deposition of the thin film of gold, or other calibration material, when it is required on TEM specimens as an internal calibration of ED patterns.

7.3.6 Sputter coater

A sputter coater with a gold target may be used for deposition of gold onto TEM specimens as an integral calibration of ED patterns. Other calibration materials are acceptable. Experience has shown that a sputter coater allows better control of the thickness of the calibration material.

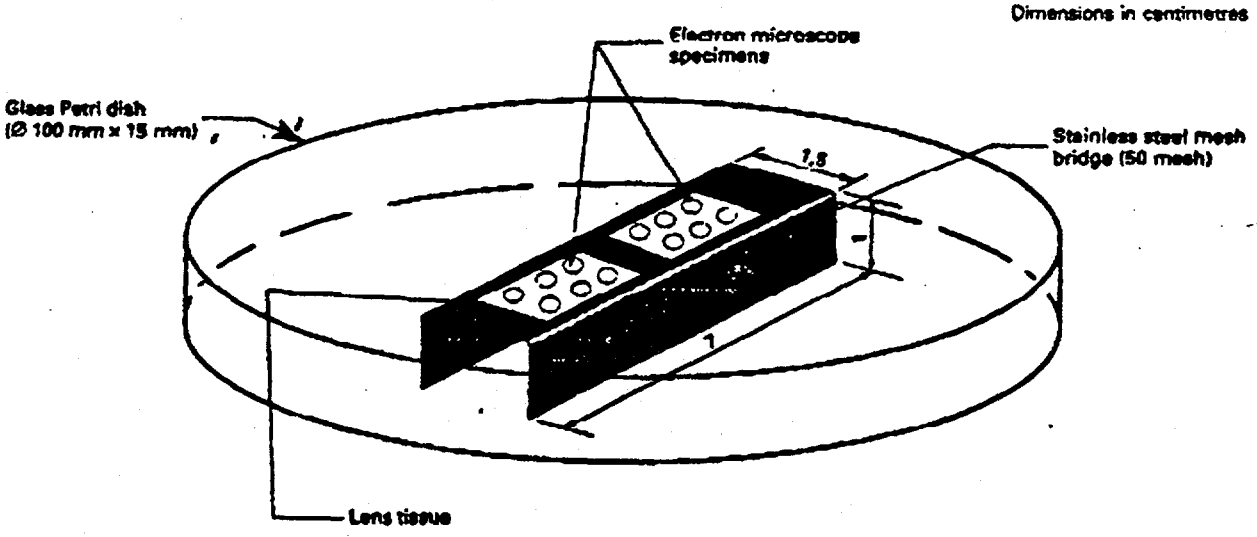
7.3.7 Solvent washer (Jaffe washer)

The purpose of the Jaffe washer is to allow dissolution of the filter polymer while leaving an intact evaporated carbon film supporting the fibres and other particles from the filter surface. One design of a washer which has been found satisfactory for various solvents and filter media is shown in figure 2. In general, either chloroform or 1-methyl-2-pyrrolidone has been used for dissolving polycarbonate filters and dimethylformamide or acetone has been used for dissolving MEC or cellulose nitrate filters. The higher evaporation rates of chloroform and acetone require that a reservoir of 10 ml to 50 ml of solvent be used, which may need replenishment during the procedure. Dimethylformamide and 1-methyl-2-pyrrolidone have lower vapour pressures and much smaller volumes of solvent may be used. It is recommended that all washers be used in a fume hood, and when specimens are not being inserted or removed, the Petri dish lid shall be in place during the solvent dissolution. The washer shall be cleaned before it is used for each batch of specimens.

7.3.8 Condensation washer

For more rapid dissolution of the filter polymer, or if difficulties are experienced in dissolving the filter polymer, use a condensation washer, consisting of a flask, condenser and cold finger assembly, with a heating mantle and means for controlling the temperature. A suitable assembly is shown in figure 3, using either acetone or chloroform as the solvent, depending on the type of filter.

Dimensions in centimetres



NOTE — Solvent is added until the meniscus contacts the underside of the stainless steel mesh bridge.

Figure 2 — Example of design of solvent washer (Jaffe washer)

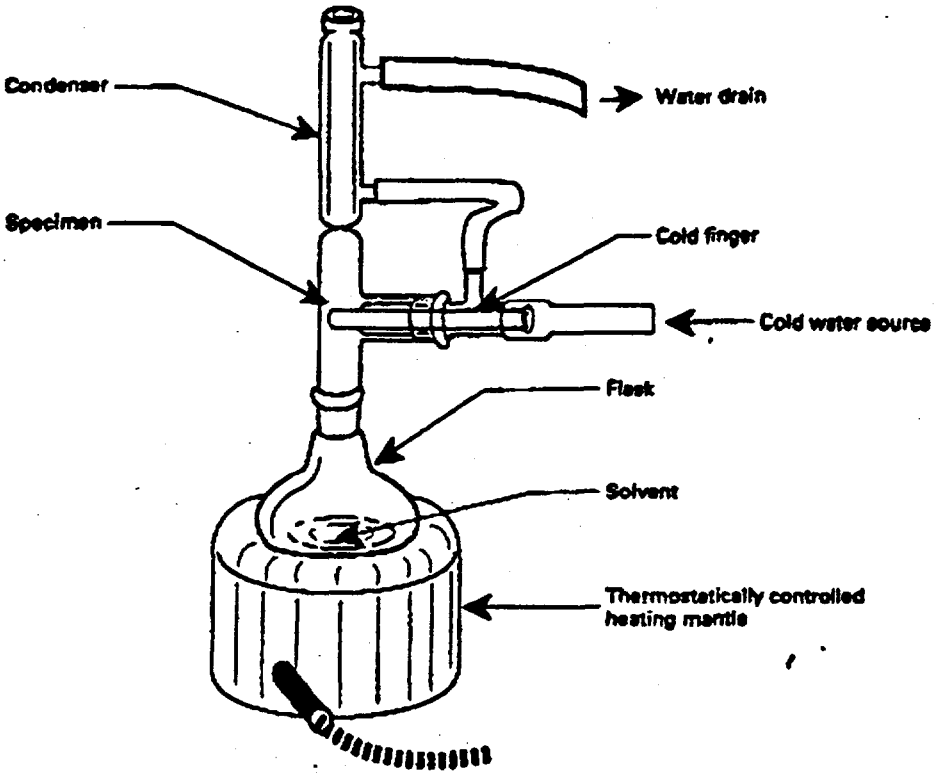


Figure 3 — Example of design of condensation washer

7.3.9 Slide warmer or oven

Use either a slide warmer or an oven for heating slides during the preparation of TEM specimens from MEC or cellulose nitrate filters. It is required to maintain a temperature of 65 °C to 70 °C.

7.3.10 Ultrasonic bath

An ultrasonic bath is necessary for cleaning the apparatus used for TEM specimen preparation.

7.3.11 Carbon grating replica

A carbon grating replica with about 2 000 parallel lines per millimetre shall be used to calibrate the magnification of the TEM.

7.3.12 Calibration specimen grids for EDXA

TEM specimen grids prepared from dispersions of calibration minerals are required for calibration of the EDXA system. Some suitable calibration minerals are riebeckite, chrysotile, halloysite, phlogopite, wollastonite and bustamite. The mineral used for calibration of the EDXA system for sodium shall be prepared using a gold TEM grid.

7.3.13 Carbon rod sharpener

The use of necked carbon rods, or equivalent, allows the carbon to be evaporated onto the filters with a minimum of heating.

7.3.14 Disposable tip micropipettes

A disposable tip micropipette, capable of transferring a volume of approximately 30 µl, is necessary for the preparation of TEM specimen grids from MEC filters.

7.4 Consumable supplies

7.4.1 Copper electron microscope grids

Copper TEM grids with 200 mesh are recommended. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.2 Gold electron microscope grids

Gold TEM grids with 200 mesh are recommended to mount TEM specimens when sodium measurements are required in the fibre identification procedure. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.3 Carbon rod electrodes

Spectrochemically pure carbon rods, shall be used in the vacuum evaporator (7.3.5) during carbon coating of filters.

7.4.4 Routine electron microscopy tools and supplies

Fine-point tweezers, scalpel holders and blades, microscope slides, double-coated adhesive tape, lens tissue, gold wire, tungsten filaments and other routine supplies are required.

7.4.5 Reference asbestos samples

Asbestos samples, shall be for preparation of reference TEM specimens of the primary asbestos minerals. The UICC set of minerals is suitable for this purpose.

8 Air sample collection

The desired analytical sensitivity is a parameter that shall be established for the analysis prior to sample collection. It is defined as the structure concentration corresponding to the detection of one structure in the analysis. For direct transfer methods of TEM specimen preparation, the analytical sensitivity is a function of the volume of air sampled, the active area of the collection filter, and the area of the TEM specimen over which structures are counted. If total airborne dust levels are high, it may be necessary to terminate sampling before the required volume has been sampled. If this happens, the analytical sensitivity required can be achieved only by counting structures on more grid openings, or by selective concentration of asbestos structures using an indirect TEM specimen preparation technique. Select the sampling rate and the period of sampling to yield the required analytical sensitivity, as detailed in table 1. Before air samples are collected, unused filters shall be analysed as described in 9.7 to determine the mean asbestos structure count for blank filters.

Air samples shall be collected using filter cassettes (7.1.1). During sampling, the cassette shall be supported on a stand (7.1.3) which is isolated from the vibrations of the pump (7.1.2). The cassette shall be held facing vertically downwards at a height of approximately 1,5 m to 2,0 m above ground/floor level, and shall be connected to the pump with a flexible tube.

Measure the sampling flow-rate at the front end of the cassette, both at the beginning and end of the sampling period, using a calibrated variable area flowmeter (7.1.4) temporarily attached to the inlet of the cassette. The mean value of these two measurements shall be used to calculate the total air volume sampled.

Basic strategies for monitoring environmental sources of airborne asbestos are described in annex G. After sampling, a cap shall be placed over the open end of the cassette, and the cassette packed with the filter face-upwards for return to the laboratory. Field blank filters shall also be included, as specified in 9.7, and submitted to the remaining analytical procedures along with the samples.

NOTES

5 In table 1 a collection filter area of 385 mm^2 is assumed, and the TEM grid openings are assumed to be 85 μm^2 square. The limit of detection is defined as the upper 95 % confidence limit of the Poisson distribution for a count of 0 structures. In the absence of background, this is equal to 2.99 times the analytical sensitivity. Backgrounds that are different from 0 observed during analysis of blank filters will degrade the limit of detection.

6 The analytical sensitivity S, expressed in number of structures per litre, is calculated using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

A_f is the active area, in square millimetres, of sample collection filter;

A_g is the mean area, in square millimetres, of grid openings examined;

k is the number of grid openings examined;

V is the volume of air sampled, in litres.

Table 1 — Examples of the minimum number of grid openings required to achieve a particular analytical sensitivity and limit of detection

Analytical sensitivity structures/l	Limit of detection structures/l	Volume of air sampled (litres) 500	1 000	2 000	3 000	4 000	5 000
0,1	0,30	1 066	533	267	178	134	107
0,2	0,60	533	267	134	89	67	54
0,3	0,90	356	178	89	60	45	36
0,4	1,2	267	134	67	45	34	27
0,5	1,5	214	107	54	36	27	22
0,7	2,1	153	77	39	26	20	16
1,0	3,0	107	54	27	18	14	11
2,0	6,0	54	27	14	9	7	6
3,0	9,0	36	18	9	6	5	4
4,0	12	27	14	7	5	4	4
5,0	15	22	11	6	4	4	4
7,0	21	16	8	4	4	4	4
10	30	11	6	4	4	4	4

9 Procedure for analysis

9.1 General

The techniques used to prepare TEM specimens are different for polycarbonate and cellulose ester filters. The preparation method to be used shall be either 9.3 or 9.4, depending on the type of membrane filter used for air sampling. Cleaning of the sample cassettes before they are opened, preparation of the carbon evaporator, criteria for acceptable specimen grids, and the requirement for blank determinations are identical for the two preparation techniques. TEM examination, structure counting, fibre identification and reporting of results are independent of the type of filter or preparation technique used.

The ability to meet the blank sample criteria is dependent on the cleanliness of equipment and supplies. Consider all supplies such as microscope slides and glassware as potential sources of asbestos contamination. It is necessary to wash all glassware before it is used. Wash any tools or glassware which come into contact with the air sampling filters or TEM specimen preparations both before use and between handling of individual samples. Where possible, disposable supplies should be used.

9.2 Cleaning of sample cassettes

Asbestos fibres can adher to the exterior surfaces of air sampling cassettes, and these fibres can be inadvertently transferred to the sample during handling. To prevent this possibility of contamination, and after ensuring that the cassette is tightly sealed, wipe the exterior surfaces of each sampling cassette before it is placed in the clean facility or laminar flow hood.

9.3 Direct preparation of TEM specimens from polycarbonate filters

9.3.1 Selection of filter area for carbon coating

Use a cleaned microscope slide to support representative portions of polycarbonate filter during the carbon evaporation. Double-coated adhesive tape is used to attach the filter portions to the glass slide. Take care not to stretch the polycarbonate filters during handling. Using freshly cleaned tweezers, remove the polycarbonate filter from the sampling cassette, and place it on to a second cleaned glass microscope slide which is used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut the filter by rocking the blade from the point, pressing it into contact with the filter. Repeat the process as necessary. Several such portions may be mounted on the same microscope slide. The scalpel blade and tweezers shall be washed and dried between the handling of each filter. Identify the filter portions by writing on the glass slide.

9.3.2 Carbon coating of filter portions

Place the glass slide holding the filter portions on the rotation-tilting device, approximately 10 cm to 12 cm

from the evaporation source, and evacuate the evaporator chamber (7.3.5) to a vacuum better than 0,013 Pa. The evaporation of carbon shall be performed in very short bursts, separated by a few seconds to allow the electrodes to cool. If evaporation of carbon is too rapid, the strips of polycarbonate filter will begin to curl, and cross-linking of the surface will occur. This cross-linking procedures a layer of polymer which is relatively insoluble in organic solvents, and it will not be possible to prepare satisfactory TEM specimens. The thickness of carbon required is dependent on the size of particles on the filter, and approximately 30 nm to 50 nm has been found to be satisfactory. If the carbon film is too thin, large particles will break out of the film during the later stages of preparation, and there will be few complete and undamaged grid openings on the specimen. Too thick a carbon film will lead to a TEM image which is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film thickness should be the minimum possible, while retaining most of the grid openings of the TEM specimen intact.

9.3.3 Preparation of the Jaffe washer

Place several pieces of lens tissue, as shown in figure 2, on the stainless steel bridge (7.1.3) and fill the washer (see 7.3.7) with chloroform (6.2) or 1-methyl-2-pyrrolidone (6.3) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.3.4 Placing of specimens in the Jaffe washer

Using a curved scalpel blade, cut three 3 mm square pieces of carbon-coated polycarbonate filter form the carbon-coated filter portion. Select three squares to represent the centre and the periphery of the active surface of the filter. Place each square of filter, carbon side up, on a TEM specimen grid, and place the grid and filter on the saturated lens tissue in the Jaffe washer. Place the three specimen grids from one sample on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. Cover the Jaffe washer with the lid, and allow the washer to stand for at least 8 h.

NOTE 7 It has been found that some polycarbonate filters will not completely dissolve in the Jaffe washer, even after exposure to chloroform for as long as 3 d. This problem is more severe if the surface of the filter was overheated during the carbon evaporation. It has been found that the problem of residual undissolved filter polymer can be overcome in several ways:

a) condensation washing of the grids, using chloroform as the solvent, after the initial Jaffe washer treatment, can often remove much of the residual filter medium in a period of approximately 30 min. To carry out this procedure, transfer the piece of lens tissue supporting the specimen grids to the cold finger of the condensation washer (7.3.8), which has achieved stable operating conditions. Operate the washer for approximately 30 min after inserting the grids;

b) used in a Jaffe washer, 1-methyl-2-pyrrolidone has been found to be a more effective solvent than chloroform for polycarbonate filters. This solvent is more effective if the lens paper is not used and grids are placed directly on the stainless steel mesh of the Jaffe washer. A dissolution period of 2 h to 6 h has been found to be satisfactory. After dissolution is complete, remove the stainless steel mesh from the Jaffe washer and allow the grids to dry. 1-methyl-2-pyrrolidone evaporates very slowly. If it is required to dry the grids more rapidly, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash;

c) a mixture of 20 % 1,2-diaminoethane [ethylenediamine] and 80 % 1-methyl-2-pyrrolidone, used in a Jaffe washer, completely dissolves polycarbonate filters in 15 min, even if the surface of the filter has been overheated. To use this solvent, place the grids directly on the stainless steel mesh of the Jaffe washer, do not use the lens paper. After a period of 15 min, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash.

9.3.5 Rapid preparation of TEM specimens from PC filters

TEM specimens can be prepared rapidly from PC filters, if desired, by washing for approximately 1 h in a Jaffe washer, followed by washing for 30 min in a condensation washer using chloroform as the solvent. The alternative filter dissolution procedures described in note 7 may also be used.

9.4 Direct preparation of TEM specimens from cellulose ester filters

9.4.1 Selection of area of filter for preparation

Using clean tweezers, remove the filter from the filter cassette, and place it on a cleaned microscope slide. Using a clean, curved scalpel blade, cut out a portion of the filter.

9.4.2 Preparation of solution for collapsing cellulose ester filters

Mix 35 ml of dimethylformamide (6.4), and 15 ml of glacial acetic acid (6.5) with 50 ml of water (6.1). Store this mixture in a clean bottle. The mixture is stable and suitable for use for up to 3 months after preparation.

9.4.3 Filter collapsing procedure

Using a micropipette with a disposable tip (7.3.14), place 15 µl/cm^2 to 25 µl/cm^2 of the solution prepared in 9.4.2 on a cleaned microscope slide, and using the end of the pipette tip, spread the liquid over the area to be occupied by the filter portion. Place the filter portion, active surface upwards, on top of the solution, lowering the edge of the filter at an angle of about 20° so that air bubbles are not created. Remove any solution not absorbed by the filter by allowing a paper tissue to contact the liquid at the edge of the filter. More than one filter portion may be placed on one slide. Place the slide either on a thermostatically controlled slide warmer (7.3.9) at a temperature of 65 °C to 70 °C, or in an oven (7.3.9) at this temperature, for 10 min. The filter collapses slowly to about 15 % of its original thickness The procedure leaves a thin, transparent polymer film, with particles and fibres embedded in the upper surface.

9.4.4 Plasma etching of the filter surface

The optimum conditions and time for plasma etching (see 7.3.4) have been determined experimentally from the recovery of fine chrysotile fibrils on 0,8 µm pore size MEC filters. The conditions required in a particular plasma asher shall be established using the procedure specified in annex A. Place the microscope slide holding the collapsed filter portions in the plasma asher, and etch for the time and under the conditions determined. Take care to ensure that the correct conditions are respected. After etching, admit air slowly to the chamber and remove the microscope slide.

Adjust the air admission valve of the plasma asher such that the time taken for the chamber to reach atmospheric pressure exceeds 2 min. Rapid air admission may disturb particulates on the surface of the etched filter.

9.4.5 Carbon coating

Coat the microscope slide holding the collapsed filter portions with carbon as specified in 9.3.2.

9.4.6 Preparation of the Jaffe washer

Place several pieces of lens tissue on the stainless steel bridge, and fill the washer with dimethylformamide (6.4) or acetone (6.5) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.4.7 Placing of specimens in the Jaffe washer

Place the specimens in the Jaffe washer as specified in 9.3.4. Specimens are normally cleared after approximately 4 h.

9.4.8 Rapid preparation of TEM specimens from cellulose ester filters

An alternative washing procedure may be used to prepare TEM specimens from cellulose ester filters more rapidly than can be achieved by the Jaffe washing procedure. After the specimens have been washed in a Jaffe washer for approximately 1 h, transfer the piece of lens tissue supporting the specimens to the cold finger of a condensation washer (7.3.8) operating with acetone as the solvent because dimethylformamide shall not be used in a condensation washer. Operate the condensation washer for approximately 30 min. This treatment removes all the remaining filter polymer.

9.5 Criteria for acceptable TEM specimen grids

Valid data cannot be obtained unless the TEM specimens meet specified quality criteria. Examine the TEM specimen grid in the electron microscope at a sufficiently low magnification (x 300 to x 1 000) for complete grid openings to be inspected. Reject the grid if

a) the TEM specimen has not been cleared of filter medium by the filter dissolution step. If the TEM specimen exhibits areas of undissolved filter medium, and if at least two of the three specimen grids are not cleared, either additional washing with solvent shall be carried out, or new specimens shall be prepared from the filter;

b) the sample is overloaded with particulate. If the specimen grid exhibits more than approximately 10 % obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. This filter cannot be alanysed satisfactorily using the direct preparation methods because the grid is too heavily loaded with debris to allow separate examination of individual particles by ED and EDXA, and obscuration of fibres by

other particulates may lead to underestimation of the asbestos structure count;

c) the particulate deposits on the specimen are not uniformly distributed from one grid opening to the next. If the particulate deposits on the specimen are obviously not uniform from one grid opening to the next, the specimen shall be designated as non-uniform. This condition is a function either of the air sampling conditions or of the fundamental nature of the airborne particulate. Satisfactory analysis of this filter may not be possible unless a large number of grid openings is examined;

d) the TEM grid is too heavily loaded with fibrous structures to make an accurate count. Accurate counts cannot be made if the grid has more than approximately 7 000 structures/mm^2; or

e) more than approximately 25 % of the grid openings have broken carbon film over the whole grid opening. Since the breakage of carbon film is usually more frequent in areas of heavy deposit, counting of the intact openings can lead to an underestimate of the asbestos structure count.

NOTE 8 If the specimens are rejected because unacceptable numbers of grid openings exhibit broken carbon replica, an additional carbon coating may be applied to the carbon coated filter, and new specimen grids prepared. The larger particles can often be supported by using a thicker carbon film. If this action does not produce acceptable specimen grids, this filter cannot be analysed using the direct preparation methods.

If one or more of the conditions described in b), c), d) or e) exists, it may not be possible to analyse the sample by this method.

9.6 Procedure for structure counting by TEM

9.6.1 General

The examination consists of a count of asbestos structures which are present on a specified number of grid openings. Fibres are classified into groups on the basis of morphological observations, ED patterns and EDXA spectra. The total number of structures to be counted depends on the statistical precision desired. In the absence of asbestos structures, the area of the TEM specimen grids which must be examined depends on the analytical sensitivity required. The precision of the structure count depends not only on the total number of structures counted, but also on their uniformity from one grid opening to the next. Additional structure counting will be necessary if greater precision is required.

In order that the estimate of the structure density on the sampling filter shall not be based on the small area represented by one specimen grid, grid openings shall be examined on two of the three specimen grids prepared. Then combine the results in the calculation of the structure density. Structure counts shall be made at a magnification of approximately × 20 000, and shall be terminated at the end of the examination of the grid opening on which the 100th asbestos structure is observed, except that the count shall be continued until a minimum of 4 grid openings have been examined. Otherwise, the structure count shall continue to that number of grid openings at which the specified analytical sensitivity has been achieved.

NOTE 9 The normal range for the number of grid openings which should be examined is from 4 to 20. If insufficient air has been sampled through the filter, the calculation in 9.6.4 can indicate that an impractically large number of grid openings should be examined. When this situation occurs, a larger value of analytical sensitivity may have to be accepted.

9.6.2 Measurement of mean grid opening area

The mean grid opening area shall be measured for the type of TEM specimen grids in use. The standard deviation of the mean of 10 openings selected from 10 grids should be less than 5 %. As an optional procedure, or if the 5 % standard deviation criterion cannot be demonstrated, the dimensions of each grid opening examined in the TEM shall be measured at a calibrated magnification.

9.6.3 TEM alignment and calibration procedures

Before structure counting is performed, align the TEM according to instrumental specifications. Calibrate the TEM and EDXA system according to the procedures specified in annex B.

9.6.4 Determination of stopping point

Before structure counting is begun, calculate the area of specimen to be examined in order to achieve the selected analytical sensitivity. Calculate the maximum number of grid openings to be examined using the following equation:

$$k = \frac{A_f}{A_g V S}$$

where

k is the number of grid openings to be examined, rounded upwards to the next highest integer;

A_f is the area, in square millimetres, of sample filter;

A_g is the area, in square millimetres, of TEM specimen grid opening;

V is the volume of air sampled, in litres;

S is the required analytical sensitivity, expressed in number of structures per litre.

9.6.5 General procedure for structure counting and size analysis

Use at least two specimen grids prepared from the filter in the structure count. Select at random several grid openings from each grid, and combine the data in the calculation of the results.

Use a form similar to that shown in figure 4 to record the data. Insert the first specimen grid into the TEM.

NOTE 10 In order to facilitate quality assurance measurements which require re-examination of the same grid opening by different microscopists, the grid should be inserted into the specimen holder in a standard orientation with the grid bars parallel and perpendicular to the axis of the specimen holder. This will provide scan directions parallel to the edges of the grid opening. It should be ensured that all microscopists begin scanning at the same starting point on the grid opening, and that they use similar scan patterns. This procedure permits rapid relocation of fibrous structures for further examination if necessary.

Select a typical grid opening and set the screen magnification to the calibrated value (approximately × 20 000). Adjust the sample height until the features in the centre of the TEM viewing screen are at the eucentric point. Set the goniometer tilt angle to zero. In column 1 of the data recording form, record the number or letter used to identify the grid. In column 2, record the identification of the particular grid opening. Position the specimen so that the grid opening is positioned with one corner visible on the screen. Move the image by adjustment of only one translation control, carefully examining the sample for fibres, until the opposite side of the grid opening is encountered. Move the image by a predetermined distance less than one screen diameter, using the other translation control, and scan the image in the reverse direction. Continue the procedure in this manner until the entire grid opening has been inspected in a pattern similar to that shown in figure 5. When a fibrous structure is detected, assign a sequential number to the primary structure in column 3, perform the identification procedures required as detailed in annex E, and enter the appropriate compositional classification on the structure counting form in column 5. Assign a morphological classification to the structure according to the procedures specified in annex D, and record this in column 6. Measure on the TEM viewing screen the length and width of the image of the primary structure, in millimetres, and record these measurements in columns 7 and 8. For a disperse cluster or matrix, assign a compositional classification and a morphological classification to each structure component, measure the length and width, and enter the data in columns 4 to 8. Use column 4 of the data recording form to tabulate the sequential number of total structures taking into account structure components. If non-asbestos fibres are observed, note their presence and type, if known. After a fibrous structure has been examined and measured, relocate the original field of view accurately before continuing scanning of the specimen. Failure to do this may cause structures to be overlooked or counted twice. Continue the examination until the completion of the grid opening on which the 100th asbestos structure has been recorded, or until the number of grid openings required to achieve the specified analytical sensitivity, calculated according to 9.6.4, have been examined whichever occurs first. The data shall be drawn approximately equally from a minimum of two grids. Regardless of the value calculated according to 9.6.4, fibrous structures on a minimum of four openings shall be counted.

9.6.6 Measurement of concentration for asbestos fibres and bundles longer than 5 µm

Consider improving the statistical validity for measurement of asbestos fibres and bundles longer than 5 µm by additional examination at a lower magnification, taking account only of the longer fibres and bundles. Perform this extended examination for fibres and bundles longer than 5 µm in accordance with the procedures specified in annex E. Use a magnification of approximately × 10 000 for counting all asbestos fibres and bundles longer than 5 µm, or approximately × 5 000 if only fibres and bundles within the diameter range 0,2 µm to 3,0 µm are to be counted. Continue the count until completion of the grid opening on which 100 fibres and bundles have been recorded, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity. Only those structures which are identified as, or are suspected to be, either chrysotile or one of the amphibole minerals will be reported in either the original or the extended TEM examination. Other materials, such as gypsum, cellulose fibres, and filter artifacts such as undissolved filter strands, will not be included in the fibre count. This restriction is intended to ensure that the best statistical validity is obtained for the materials of interest.

TEM asbestos structure count (page of)

Report number: .....
Sample number: .....
File name: .....
Sample description: .....
.....
.....
Preparation date: ..... By: .....
Analysis date: ..... By: .....
Computer entry date: ..... By: .....

Air volume: ..... litres
Sample filter area: ..... mm^2
Magnification: .....
Grid opening dimension: ..... µm
Level of analysis (C): .....
(A): .....

Grid	Grid opening	Number of structures		Class	Type of structure	Length	Width	Comments
		primary	total			mm	mm	

Figure 4 — Example of structure counting form

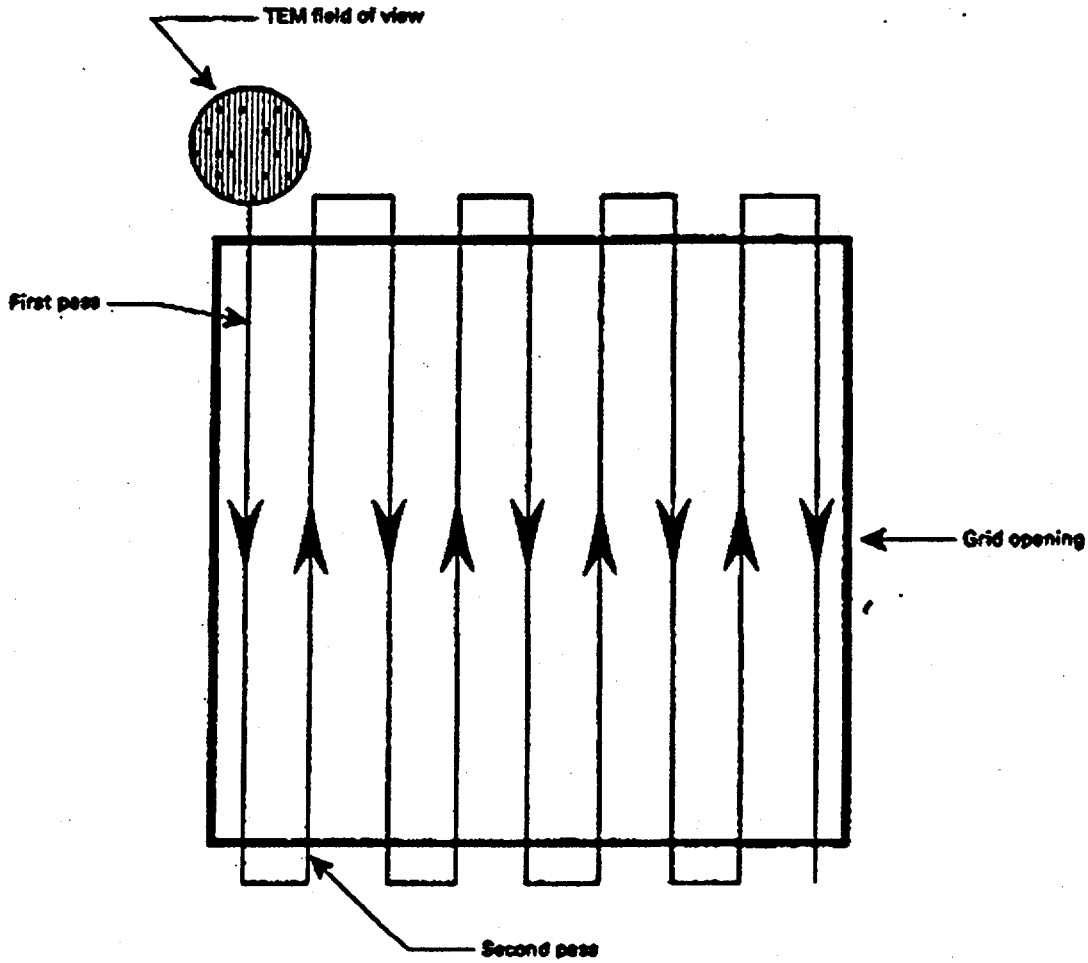


Figure 5 — Example of scanning procedure for TEM specimen examination

9.7 Blank and quality control determinations

Before air samples are collected, a minimum of two unused filters from each filter lot of 100 filters shall be analysed to determine the mean asbestos structure count. If the mean count for all types of asbestos structures is found to be more than 10 structures/mm^2, or if the mean fibre count for asbestos fibres and bundles longer than 5 µm is more than 0,1 fibre/mm^2, reject the filter lot.

To ensure that contamination by extraneous asbestos fibres during specimen preparation is insignificant compared with the results reported on samples, establish a continuous programme of blank measurements. At least one field blank shall be processed along with each batch of samples. In addition, at least one unused filter shall be included with every group of samples prepared on one microscope slide.

Initially, and also at intervals afterwards, ensure that samples of known asbestos concentrations can be analysed satisfactorily. Since there is a subjective component in the structure counting procedure, it is necessary that recounts of some specimens be made by different microscopists, in order to minimize the subjective effects. Such recounts provide a means of maintaining comparability between counts made by different microscopists. Variability between and within microscopists and between laboratories shall be characterized. These quality assurance measurements shall constitute approximately 10 % of the analyses. Repeat results should not differ at the 5 % significance level.

9.8 Calculation of results

Calculate the results using the procedures detailed in annex F. Prior to the TEM examination of the specimens, the level of analysis was specified. Before the results are calculated, the compositional and morphological classifications to be included in the result shall be specified. The chi-squared uniformity test shall be conducted using the number of primary asbestos structures found on each grid opening, prior to the application of the cluster and matrix counting criteria. The concentration result shall be calculated using the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

10 Performance characteristics

10.1 General

It is important to use this analytical method in conjunction with a continuous quality control programme. The quality control programme should include use of standard samples, blank samples, and both interlaboratory and intralaboratory analyses.

10.2 Interferences and limitations of fibre identification

Unequivocal identification of every chrysotile fibre is not possible, due to both instrumental limitations and the nature of some of the fibres. The requirement for a calibrated ED pattern eliminates the possibility of an incorrect identification of the fibre selected. However, there is a possibility of misidentification of fibres for which both the morphologies and the ED patterns are reported on the basis of visual inspection only. The only significant possibilities of misidentification occur with halloysite, vermiculite scrolls or palygorskite, all of which can be discriminated from chrysotile by the use of EDXA and by observation of the 0,73 nm (002) reflection of chrysotile in the ED pattern.

As in the case of chrysotile fibres, complete identification of every amphibole fibre is not possible due to instrumental limitations and the nature of some of the fibres. Moreover, complete identification of every amphibole fibre is not practical due to the limitations of both time and cost. Particles of a number of other minerals with compositions similar to those of some amphiboles could be erroneously classified as amphibole when the classification criteria do not include zone-axis ED techniques. However, the requirement for quantitative EDXA measurements on all fibres as support for the random orientation ED technique makes misidentification very unlikely, particularly when other similar fibres in the same sample have been identified as amphibole by zone-axis methods. The possibility of misidentification is further reduced with increasing aspect ratio, since it is rare for the minerals with which amphibole may be confused to display an asbestiform habit.

10.3 Precision and accuracy (see ISO Standard Handbook No. 3)

10.3.1 Precision

The analytical precision that can be obtained is dependent upon the number of structures counted, and also on the uniformity of the particulate deposit on the original filter. Assuming that the structures are randomly deposited on the filter, if 100 structures are counted and the loading is at least 3,5 structures/grid opening, computer modelling of the counting procedure shows that a coefficient of variation of about 10 % can be expected. As the number of structures counted decreases, the precision will also decrease approximately as $\sqrt{N}$, where N is the number of structures counted. In practice, particulate deposits obtained by filtration of ambient air samples are rarely ideally distributed, and it is found that the precision is correspondingly reduced. Degradation of precision is a consequence of several factors, such as:

a) non-uniformity of the filtered particulate deposit;

b) distorsion of the fibre distribution by application of the structure counting criteria;

c) variation between microscopists in their interpretation of the fibrous structures;

d) variation between microscopists in their ability to detect and identify fibres.

The 95 % confidence interval about the mean for a single structure concentration measurement using this analytical method should be approximately ± 25 % when 100 structures are counted over 10 grid openings.

10.3.2 Accuracy

There is no independent method available to determine the accuracy.

NOTE 11 It has been demonstrated that, after polycarbonate membrane filters have been coated with carbon, particulate material is transferred to the TEM specimens without measurable losses. However, if the filters are heavily loaded by particulate material, some of this may be lost before they are coated with carbon. Good comparability between the capillary-pore polycarbonate pro-

cedure and the cellulose ester filter procedure has been demonstrated for laboratory-generated aerosols of chrysotile asbestos.

10.3.3 Interlaboratory and intralaboratory analyses

Interlaboratory and intralaboratory analyses are required in order to monitor systematic errors that may develop among microscopists when using this method. These analyses should be designed to test both the overall method and the performance of individual microscopists. Repeating preparation of TEM grids from different sectors of a filter, followed by examination of the grids by a different microscopist, is a test for the reproducibility of the whole method. However, non-uniformity of the particulate deposit on the filter may lead to differences which are not related to the performance of the microscopists. Verified fibre counting (counting of asbestos structures on the same grid opening of a TEM grid by two or more operators, followed by resolution of any discrepancies) may be used both as a training aid and to determine the performance of different microscopists. The use of indexed TEM grids as described in 7.4.1 and 7.4.2 is recommended in order to facilitate relocation of specific grid openings.

10.4 Limit of detection

The limit of detection of the method can be varied by choice of the area of the collection filter, the volume of air sampled and the area of the specimen examined in the TEM. It is also a function of the background of asbestos structures on unused filters. A limit of detection shall be quoted for each sample analysis.

NOTE 12 In practice, the lowest limit of detection is frequently determined by the total suspended particulate concentration, since each particle on the filter must be separated from adjacent ones by a distance large enough for the particle to be identified without interference. Particulate loadings on sampling filters greater than 25 µg/cm^2 usually preclude preparation of TEM specimens by the direct methods. If the analysis is to be performed with an acceptable expenditure of time, the area of the specimen examined in the TEM for structures of all sizes is limited in most cases to between 10 and 20 grid openings. In typical ambient or building atmospheres, it has been found that an analytical sensitivity of 1 structure/l can be achieved. In some circumstances, where the atmosphere is exceptionally clean, this can be reduced to 0,1 structure/l or lower. For fibres and bundles longer than 5 µm, the reduced magnifications specified permit larger areas of the TEM specimens to be examined with an acceptable expenditure of time, resulting in proportionately lower limits of detection. If no structures are found in the analysis, the upper 95 % confidence limit can be quoted as the upper boundary of the concentration, corresponding to 2,99 times the analytical sensitivity if a Poisson distribution of structures on the filter is assumed. This 95 % confidence limit for 0 structures counted is taken as the detection limit. Since there is sometimes contamination of unused samples filters by asbestos structures, this should also be taken into account in the discussion of limits of detection.

11 Test report

The test report shall include at least the following information:

a) reference to this International Standard;

b) identification of the sample;

c) the date and time of sampling, and all necessary sampling data;

d) the date of the analysis;

e) the identity of the analyst;

f) any procedure used that is not specified in this International Standard or regarded as optional;

g) a complete listing of the structure counting data (the following data should be included: grid opening number, structure number, identification category, structure type, length and width of the structure in micrometres, and any comments concerning the structure);

h) a statement of the minimum acceptable identification category and the maximum identification category attempted (refer to tables D.1 and D.2);

i) a statement specifying which identification and structure categories have been used to calculate the concentration values;

j) separate concentration values for chrysotile and amphibole structures, expressed in number of asbestos structures per litre;

k) the 95 % confidence interval limits for the concentration values, expressed in number of asbestos structures per litre;

l) the analytical sensitivity, expressed in number of asbestos structures per litre;

m) the limit of detection, expressed in number of asbestos structures per litre;

n) compositional data for the principal varieties of amphibole, if present;

o) items g) to m) for asbestos fibres and bundles longer than 5 µm;

p) items g) to m) for PCM equivalent asbestos fibres and bundles.

An example of a suitable format for the structure counting data is shown in figures 6 and 7.

Sample analysis information (page 1)

Laboratory name	**Report number**	**Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

Air volume:	2 150,0 litres
Area of collection filter:	385,0 mm^2
Level of analysis (chrysotile):	CD or CMQ
Level of analysis (amphibole):	ADQ
Magnification used for fibre counting:	× 20 500
Aspect ratio for fibre definition:	5/1
Mean dimension of grid openings:	95,4 µm
Initials of analyst:	JMW
Number of grid openings examined:	10
Analytical sensitivity:	1,968 structures/l
Number of primary asbestos structures:	13
Number of asbestos structures counted:	26
Number of asbestos structures > 5 µm :	7
Number of asbestos fibres and bundles > 5 µm :	10
Number of PCM equivalent asbestos structures:	3
Number of PCM equivalent asbestos fibres:	5

Figure 8 — Example of format for reporting sample and preparation data

Sample analysis information (pages 2 and following)

Laboratory name **Report number** **Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

TEM asbestos structure count — Raw data

Grid	Grid opening	Number of structures primary	Number of structures total	Identification[1]	Structure type	Length µm	Width µm	Comments
A	F4-4	1	1	CD	F	1,7	0,045	
		2	2	CMQ	B	2,6	0,09	
		3	3	ADQ	F	4,0	0,15	Crocidolite
	E3-6	4	4	CD	MC+0	3,5	1,3	
	E5-1	5		CD	MD43	7,5	5,0	
			5	CD	MB	7,7	0,30	
			6	CMQ	MF	5,6	0,045	
			7	CD	MB	5,1	0,30	
			8	CD	MF	1,7	0,045	
B	F4-1	6		CD	CD+0	6,5	3,0	
			9	CD	CB	3,5	0,15	
			10	CD	CF	3,5	0,045	
			11	CMQ	CR+0	2,6	1,9	
	G5-1	7		CD	CD31	6,1	3,2	
			12	CD	CB	5,6	0,3	
			13	CMQ	CF	4,0	0,045	
			14	CMQ	CB	3,2	0,090	
	E4-4	8	15	CD	B	1,5	0,23	
		9	16	AD	F	8,7	0,15	
C	G4-4	10		CMQ	CD42	25	5,6	
			17	CMQ	CB	15	0,15	
			18	CMQ	CF	9,4	0,045	
			19	ADQ	CF	3,6	0,30	Tremolite
			20	CM	CF	4,2	0,045	
	E4-4			No fibres				
	E5-6	11		ADQ	CD+3	9,4	2,5	
			21	ADQ	CF	7,1	0,30	Amosite
			22	ADQ	CF	6,2	0,10	Crocidolite
			23	CM	CB	5,1	0,2	
			24	CM	CR+0	3,3	1,8	
	F4-1	12	25	CMQ	MC10	3,7	2,1	
		13	26	CD	CC+0	7,4	0,5	

1) Identification codes listed in tables D.1 and D.2.

Figure 7 — Example of format for reporting structure counting data

Annex A
(normative)

Determination of operating conditions for plasma asher

A.1 General

During the preparation of TEM specimens from an MEC or cellulose nitrate filter, the spongy structure of the filter is collapsed into a thinner film of polymer by the action of a solvent. Some of the particles on the surface of the original filter become completely buried in the polymer, and the specimen preparation procedure incorporates a plasma etching step to oxidize the surface layer of the polymer. Particles buried by the filter collapsing step are then exposed so that they can become subsequently affixed to the evaporated carbon film without altering their position on the original filter. The amount of etching is critical, and individual ashers vary in performance. Therefore, the plasma asher (7.3.4) shall be calibrated to give a known amount of etching of the surface of the collapsed filter. This is carried out by adjusting the radio-frequency power output and the oxygen flow-rate, and measuring the time taken to completely oxidize an uncollapsed cellulose ester filter with 25 mm diameter of the same type and pore size as those used in the analysis.

A.2 Procedure

Place an unused cellulose ester filter, with 25 mm diameter, of the same type as that being used, in the centre of a glass microscope slide. Position the slide approximately in the centre of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 ml/min to 20 ml/min. Adjust the tuning of the system so that the intensity of the plasma in maximized. Measure the time required for complete oxidation of the filter. Determine operating parameters which result in complete oxydation of the filter in a period of approximately 15 min. For etching of collapsed filters, these operating parameters shall be used for a period of 8 min.

NOTE 13 Plasma oxidation at high radio-frequency powers will cause the filter to shrink and curl, followed by sudden violent ignition. At lower powers, the filter will remain in position and will slowly become thinner until it is nearly transparent. It is recommended that a radio-frequency power be used such that violent ignition does not occur. When multiple filters are etched, the rate of etching is reduced, and the system should be calibrated accordingly.

Annex B
(normative)

Calibration procedures

B.1 Calibration of the TEM

B.1.1 Calibration of TEM screen magnification

The electron microscope should be aligned according to the specifications of the manufacturer. Initially, and at regular intervals, calibrate the magnifications used for the analysis using a diffraction grating replica (7.3.11). Adjust the specimen height to the eucentric position before carrying out the calibration. Measure the distance on the fluorescent viewing screen occupied by a convenient number of repeat distances of the grating image, and calculate the magnification. Always repeat the calibration after any instrumental maintenance or change of operating conditions. The magnification of the image on the viewing screen is not the same as that obtained on photographic plates or film. The ratio between these is a constant value for the particular model of TEM.

B.1.2 Calibration of ED camera constant

Calibrate the camera constant of the TEM when used in ED mode. Use a specimen grid supporting a carbon film on which a thin film of gold has been evaporated or sputtered. Form an image of the gold film with the specimen adjusted to the eucentric position and select ED conditions. Adjust the objective lens current to optimize the pattern obtained, and measure the diameters of the innermost two rings either on the fluorescent viewing screen or on a recorded image. Calculate the radius-based camera constant, λL, for both the fluorescent screen and the photographic plate or film, using the following equation:

$$\lambda L = \frac{aD}{2{,}0\sqrt{h^2 + k^2 + l^2}}$$

where

- λ is the wavelength, in nanometres, of the incident electrons;
- L is the camera length, in millimetres;
- a is the unit cell dimension of gold, in nanometres (= 0,407 86 nm);
- D is the diameter, in millimetres, of the (hkl) diffraction ring.

Using gold as the calibration material, the radius-based camera constant is given by

$\lambda L = 0{,}117\ 74D$ mm·nm (smallest ring)

$\lambda L = 0{,}101\ 97D$ mm·nm (second ring)

B.2 Calibration of the EDXA system

Energy calibration of the EDXA system for a low energy and high energy peak shall be performed regularly. Calibration of the intensity scale of the EDXA system permits quantitative composition data, at an accuracy of about 10 % of the elemental concentration, to be obtained from EDXA spectra of reference silicate minerals involving the elements Na, Mg, Al, Si, K, Ca, Mn and Fe, and applicable certified reference materials. If quantitative determinations are required for minerals containing other elements, reference standards other than those referred to below will be required. Well-characterized mineral standards permit calibration of any TEM-EDXA combination which meets the instrumental specifications of 7.3.1 and 7.3.2, so that EDXA data from different instruments can be compared. Reference minerals are required for the calibration; the criteria for selection being that they should be silicate minerals with matrices as close as possible to those of the amphiboles or serpentine, and that small individual fragments of the minerals are homogeneous in composition within a few percent.

Determine the compositions of these standards by electron microprobe analysis or chemicals methods. Crush fragments of the same selected mineral standards and prepare filters by dispersal of the crushed material in water and immediate filtration of the suspensions. Prepare TEM specimens from these filters according to the procedures specified in clause 9. These TEM specimens can then be used to calibrate any TEM-EDXA system so that comparable composi-

tional results can be obtained from different instruments.

NOTES

14 The microprobe analysis of the mineral standards are carried out by conventional techniques which can be found in annex J. The mineral is first embedded in a mount of poly(methyl methacrylate) or epoxy resin. The mount is then ground and polished to achieve a flat, polished surface of the mineral fragment. This surface is then analysed, using suitable reference standards, preferably oxide standards of the individual elements wherever these are available. It is necessary to take into account the water concentration in the minerals, which in the case of chrysotile amounts to 13 % by mass. This water content may vary due to losses in the vacuum system.

15 Aqueous suspensions of mineral standards should be filtered immediately after preparation, since alkali and alkali earth metals may be partially leached from minerals containing these elements.

Express the results of the electron microprobe analyses as atomic or mass percentage ratios relative to silicon. X-ray peak ratios of the same elements relative to silicon, obtained from the EDXA system, can then be used to calculate the relationship between peak area ratio and atomic or mass percentage ratio. The technique was described by Cliff and Lorimer (see annex J, reference [8]).

The X-rays generated in a thin specimen by an incident electron beam have a low probability of interacting with the specimen. Thus, mass absorption and fluorescence effects are negligible. In a silicate mineral specimen containing element i, the following equation can be used to perform quantitative analyses in the TEM:

$$\frac{C_i}{C_{Si}} = k_i \times \frac{A_i}{A_{Si}}$$

where

C_i is the concentration or atomic percentage of element i;

C_{Si} is the concentration or atomic percentage of silicon;

A_i is the elemental integrated peak area for element i;

A_{Si} is the elemental integrated peak area for silicon;

k_i is the k-ratio for element i relative to silicon.

For a particular instrumental configuration and a particular particle size, the value of k_i is constant.

To incorporate correction for the particle size effect on peak area ratios (see annex J, references [35] and [36], extend the Cliff and Lorimer technique by obtaining separate values of the constant k_i for different ranges of fibre diameter. It is recommended that 20 EDXA measurements be made for each range of fibre diameters. Suitable ranges of fibre diameter are:

< 0,25 µm; 0,25 µm to 0,5 µm; 0,5 µm to 1,0 µm; > 1,0 µm.

Insert the TEM grid into the transmission electron microscope, obtain an image at the calibrated higher magnification of about × 20 000, and adjust the specimen height to the eucentric point. If the X-ray detector is a side-entry variety, tilt the specimen towards the X-ray detector. Select an isolated fibre or particle less than 0,5 µm in width, and accumulate an EDXA spectrum using an electron probe of suitable diameter. When a well-defined spectrum has been obtained, perform a background subtraction and calculate the background-corrected peak areas for each element listed, using energy windows centred on the peaks. Calculate the ratio of the peak area for each specified element relative to the peak area for silicon. All background-subtracted peak areas used for calibration shall exceed 400 counts.

Repeat this procedure for 20 particles of each mineral standard. Reject analyses of any obviously foreign particles. Calculate the arithmetic mean concentration to peak area ratio, k_i (k-ratio), for each specified element of each mineral standard and for each of the fibre diameter ranges. Periodic routine checks shall be carried out to ensure that there has been no degradation of the detector performance. These k-ratios are used to calculate the elemental concentrations of unknown fibres, using the Cliff and Lorimer relationship.

Annex C
(normative)

Structure counting criteria

C.1 General

In addition to isolated fibres, other assemblages of particles and fibres frequently occur in air samples. Groupings of asbestos fibres and particles, referred to as "asbestos structures", are defined as fibre bundles, clusters and matrices. The numerical result of a TEM examination depends largely on whether the analyst assigns such an assemblage of fibres as a single entity, or as the estimated number of individual fibres which form the assemblage. It is therefore important that a logical system of counting criteria be defined, so that the interpretation of these complex structures is the same for all analysts, and so that the numerical result is meaningful. Imposition of specific structure-counting criteria generally requires that some interpretation, partially based on uncertain information on health effects, be made of each asbestos structure found. It is not the intention of this International Standard to make any interpretations based on health effects, and it is intended that a clear separation shall be made between recording of structure counting data, and later interpretation of those data. The system of coding specified in this International Standard permits a clear morphological description of the structures to be recorded in a concise manner suitable for later interpretation, if necessary, by a range of different criteria, without the necessity for re-examination of the specimens. In particular, the coding system is designed to permit the dimensions of each complex fibrous structure, and also whether these structures contain fibres longer than 5 µm, to be recorded. This approach permits later evaluations of the data to include considerations of particle respirability and comparisons with historical indices of asbestos exposure. Examples of the various types of morphological structure, and the manner in which these shall be recorded, are shown in figure C.1.

C.2 Structure definitions and treatment

Each fibrous structure that is a separate entity shall be designated as a primary structure. Each primary structure shall be designated as a fibre, bundle, cluster or matrix.

C.2.1 Fibre

Any particle with parallel or stepped sides, of minimum length 0,5 µm, and with an aspect ratio of 5/1 or greater, shall be defined as a fibre. For chrysotile asbestos, the single fibril shall be defined as a fibre. A fibre with stepped sides shall be assigned a width equal to the average of the minimum and maximum widths. This average shall be used as the width in determination of the aspect ratio.

C.2.2 Bundle

A grouping composed of apparently attached parallel fibres shall be defined as a bundle, with a width equal to an estimate of the mean bundle width, and a length equal to the maximum length of the structure. The overall aspect ratio of the bundle may have any value, provided that it contains individual constituent fibres with aspect ratios equal to or greater than 5/1. Bundles may exhibit diverging fibres at one or both ends.

C.2.3 Cluster

An aggregate of two or more randomly oriented fibres, with or without bundles, shall be defined as a cluster. Clusters occur as two varieties.

C.2.3.1 disperse cluster (type D): Disperse and open network, in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured;

C.2.3.2 compact cluster (type C): Complex and tightly bound network, in which one or both ends of each individual fibre or bundle is (are) obscured, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, clusters can occur in which the characteristics of both types of cluster occur in the same structure. Where this occurs, the structure should be defined as a disperse cluster, and then a logical procedure should be followed by recording structure components according to the counting criteria. The procedure for treatment of clusters is illustrated by examples in figure C.2.

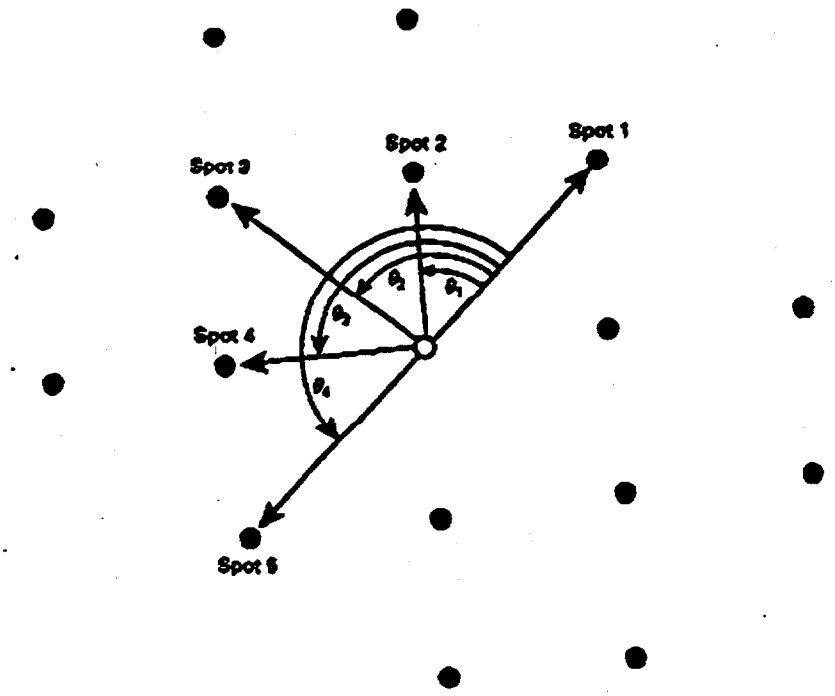


Figure D.1 — Example of measurement of zone-axis SAED patterns

To obtain an EDXA spectrum, move the image of the fibre to the centre of the screen and remove the objective aperture. Select an appropriate electron beam diameter and deflect the beam so that it impinges on the fibre. Depending on the instrumentation, it may be necessary to tilt the specimen towards the X-ray detector and, in some instruments, to use the Scanning Transmission Electron Microscopy (STEM) mode of operation.

The time for acquisition of a suitable spectrum varies with the fibre diameter, and also with instrumental factors. For quantitative interpretation, spectra should have a statistically valid number of counts in each peak. Analyses of small diameter fibres which contain sodium are the most critical, since it is in the low energy range that the X-ray detector is least sensitive. Consequently, it is necessary to acquire a spectrum for a period that is sufficiently long for the sodium to be detected in such fibres. It has been found that satisfactory quantitative an analyses can be obtained if acquisition is continued until the background subtracted silicon $K\alpha$ peak integral exceeds 10 000 counts. The spectrum should then be manipulated to subtract the background and to obtain the net areas of the elemental peaks.

After quantitative EDXA classification of some fibres by computer analysis of the net peak areas, it may be possible to classify further fibres in the same sample on the basis of comparison of spectra at the intrument. Frequently, visual comparisons can be made after somewhat shorter acquisition times.

D.3 Interpretation of fibre analysis data

D.3.1 Chrysotile

The morphological structure of chrysotile is characteristic, and with experience, can be recognized readily. However, a few holder minerals have a similar appearance, and morphological observation by itself is inadequate for most samples. The ED pattern obtained from chrysotile is quite specific for this mineral if the specified characteristics of the pattern correspond to those from reference chrysotile. However, depending on the past history of the fibre, and on a number of other factors, the crystal structure of a particular fibre may be damaged, and it may not yield and ED pattern. In this case, the EDXA spectrum may be the only data available to supplement the morphological observations.

D.3.2 Amphiboles

Since the fibre identification procedure for asbestos fibres other than chrysotile can be involved and time-consuming, computer programmes, such as that developed by B.L. Rhoades (see annex J, reference

[32]), are recommended for interpretation of zone-axis ED patterns. The published literature contains composition and crystallographic data for all of the fibrous minerals likely to be encountered in TEM analysis of air samples, and the compositional and structural data from the unknown fibre should be compared with the published data. Demonstration that the measurements are consistent with the data for a particular test mineral does not uniquely identify the unknown, since the possibility exists that data from other minerals may also be consistent. It is, however, unlikely that a mineral of another structural class could yield data consistent with that from an amphibole fibre identified by quantitative EDXA and two zone-axis ED patterns.

Suspected amphibole fibres should be classified initially on the basis of chemical composition. Either qualitative or quantitative EDXA information may be used as the basis for this classification. From the published data on mineral compositions, a list of minerals which are consistent in composition with that measured for the unknown fibre should be compiled. To proceed further, it is necessary to obtain the first zone-axis ED pattern, according to D.2.2.

It is possible to specify a particular zone-axis pattern for identification of amphibole, since a few patterns are often considered to be characteristic. Unfortunately, for a fibre with random orientation on a TEM grid, no specimen holder and goniometer currently available will permit convenient and rapid location of two preselected zone-axes. The most practical approach has been adopted, which is to accept those low index patterns which are easily obtained, and then to test their consistency with the structures of the minerals already preselected on the basis of the EDXA data. Even the structures of non-amphibole minerals in this preselected list shall be tested against the zone-axis data obtained for the unknown fibre, since non-amphibole minerals in some orientations may yield similar patterns consistent with amphibole structures.

The zone-axis ED interpretation shall include all minerals previously selected from the mineral data file as being chemically compatible with the EDXA data. This procedure will usually shorten the list of minerals for which solutions have been found. A second set of zone-axis data from another pattern obtained on the same fibre can then be processed, either as further confirmation of the identification, or to attempt elimination of an ambiguity. In addition, the angle measured between the orientations of the two zone-axes can be checked for consistency with the structures of the minerals. Caution should be exercised in rationalizing the inter-zone-axis angle, since if the fibre contains $\vec{c}$-axis twinning, the two zone-axis ED patterns may originate from the separate twin crystals. In practice, the full identification procedure will normally be applied to very few fibres, unless precise identification of all fibres is required for a particular reason.

D.4 Fibre classification categories

It is not always possible to proceed to a definitive identification of a fibre; this may be due to instrumental limitations or to the actual nature of the fibre. In many analyses, a definitive identification of each fibre may not actually be necessary if there is other knowledge available about the sample, or if the concentration is below a level of interest. The analytical procedure shall therefore take into account both instrumental limitations and varied analytical requirements. Accordingly, a system for fibre classification is used to permit accurate recording of data. The classifications are shown in tables D.1 and D.2, and are directed towards identification of chrysotile and amphibole respectively. Fibres shall be reported in these categories.

The general principle to be followed in this analytical procedure is first to define the most specific fibre classification which is to be attempted, or the "level" of analysis to be conducted. Then, for each fibre examined, record the classification which is actually achieved. Depending on the intended use of the results, criteria for acceptance of fibres as "identified" can then be established at any time after completion of the analysis.

In an unknown sample, chrysotile will be regarded as confirmed only if a recorded, calibrated ED pattern from one fibre in the CD categories is obtained, or if measurements of the ED pattern are recorded at the instrument. Amphibole will be regarded as confirmed only by obtaining recorded data which indicates exclusively the presence of amphiboles for fibres classified in the AZQ, AZZ or AZZQ categories.

Table D.1 — Classification of fibres with tubular morphology

Category	Description
TM	Tubular Morphology, not sufficiently characteristic for classification as chrysotile
CM	Characteristic Chrysotile Morphology
CD	Chrysotile SAED pattern
CQ	Chrysotile composition by Quantitative EDXA
CMQ	Chrysotile Morphology and composition by Quantitative EDXA
CDQ	Chrysotile SAED pattern and composition by Quantitative EDXA
NAM	Non-Asbestos Mineral

Table D.2 — Classification of fibres without tubular morphology

Category	Description
UF	Unidentified Fibre
AD	Amphibole by random orientation SAED (shows layer pattern of 0,53 nm spacing)
AX	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by Quantitative EDXA
AZ	Amphibole by one Zone-axis SAED pattern
ADQ	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	Amphibole by one Zone-axis SAED pattern and Quantitative EDXA
AZZ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle
AZZQ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle, and Quantitative EDXA
NAM	Non-Asbestos Mineral

D.4.1 Procedure for classification of fibres with tubular morphology suspected to be chrysotile

Occasionally, fibres are encountered which have tubular morphology similar to that of chrysotile, but which cannot be characterized further either by ED or EDXA. They may be non-crystalline, in which case ED techniques are not useful, or they may be in a position on the grid which does not permit an EDXA spectrum to be obtained. Alternatively, the fibre may be of organic origin, but the morphology and composition may not be sufficiently definitive enough to be disregarded. Accordingly, there is a requirement to record each fibre, and to specify how confidently each fibre can be identified. Classification of fibres will meet with various degrees of success. Figure D.2 shows the classification procedure to be used for fibres which display any tubular morphology. The chart is self explanatory, and every fibre is either rejected as a non-asbestos mineral (NAM), or classified in some way which by some later criterion could still contribute to the chrysotile fibre count.

Morphology is the first consideration, and if this is not similar to that usually seen in chrysotile standard samples, designate the initial classification as TM. Regardless of the doubtful morphology, examine the fibre by ED and EDXA methods according to figure D.2. Where the morphology is more definitive, it may be possible to classify the fibre as having chrysotile morphology (CM).

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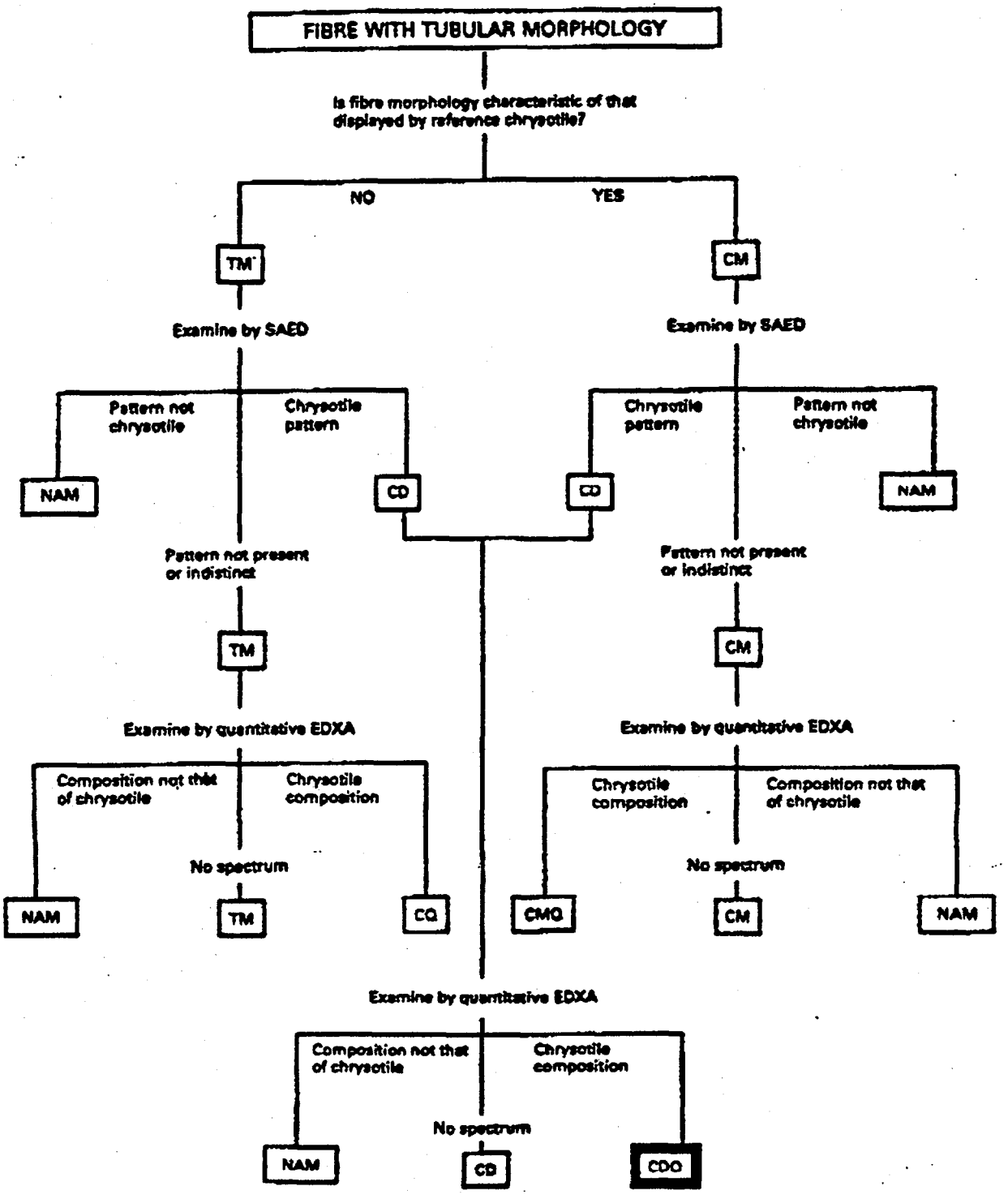

Figure D.2 — Classification chart for fibre with tubular morphology

For classification as CM, the morphological characteristics required are the following:

a) the individual fibrils should have high aspect ratios exceeding 5/1, and be about 30 nm to 40 nm in diameter;

b) the electron scattering power of the fibre at 60 kV to 100 kV accelerating potential should be sufficiently low for the internal structure to be visible;

c) there should be some evidence of an internal structure suggesting a tubular appearance similar to that shown by reference UICC chrysotile, which may degrade in the electron beam.

Examine every fibre having these morphological characteristics by the ED technique, and classify as chrysotile by ED (CD) only those which give diffraction patterns with the precise characteristics shown in figure D.3. The relevant features in this pattern for identification of chrysotile are as follows:

a) the (002) reflections should be examined to determine that they correspond closely to a spacing of 0,73 nm;

b) the layer line repeat distance should correspond to 0,53 nm;

c) there should be "streaking" of the (110) and (130) reflections.

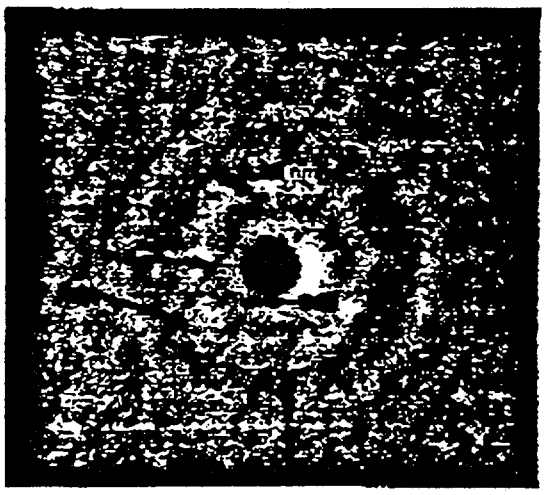

Figure D.3 — Chrysotile SAED pattern

Using the millimetre calibrations on the TEM viewing screen, these observations can readily be made at the instrument. If documentary proof of fibre identification is required, record a TEM micrograph of at least one representative fibre, and record its ED pattern on a separate film or plate. This film or plate shall also carry calibration rings from a known polycrystalline substance such as gold. This calibrated pattern is the only documentary proof that the particular fibre is chrysotile, and not some other tubular or scrolled species such as halloysite, palygorskite, talc or vermiculite. The proportion of fibres which can be successfully identified as chrysotile by ED is variable, and to some extent dependent on both the instrument and the procedures of the operator. The fibres that fail to yield an identifiable ED pattern will remain in the TM or CM categories unless they are examined by EDXA.

In the EDXA analysis of chrysotile there are only two elements which are relevant. For fibre classification, the EDXA analysis shall be quantitative. If the spectrum displays prominent peaks from magnesium and silicon, with their areas in the appropriate ratio, and with only minor peaks from other elements, classify the fibre as chrysotile by quantitative EDXA, in the categories CQ, CMQ, or CDQ, as appropriate.

D.4.2 Procedure for classification of fibres without tubular morphology, suspected to be amphibole

Every particle without tubular morphology and which is not obviously of biological origin, with an aspect ratio of 5/1 or greater, and having parallel or stepped sides, shall be considered as a suspected amphibole fibre. Further examination of the fibre by ED and EDXA techniques will meet with a variable degree of success, depending on the nature of the fibre and on a number of instrumental limitations. It will not be possible to identify every fibre completely, even if time and cost are of no concern. Moreover, confirmation of the presence of amphibole can be achieved only by quantitative interpretation of zone-axis ED patterns, a very time-consuming procedure. Accordingly, for routine samples from unknown sources, this analytical procedure limits the requirement for zone-axis ED work to a minimum of one fibre representative of each compositional class reported. In some samples, it may be necessary to identify more fibres by the zone-axis technique. When analysing samples from well-characterized sources, the cost of identification by zone-axis methods may not be justified.

The 0,53 nm layer spacing of the random orientation ED pattern is not by itself diagnostic for amphibole. However, the presence of $\bar{c}$-axis twinning in many fi-

bres leads to contributions to the layers in the patterns by several individual parallel crystals of different axial orientations. This apparently random positioning of the spots along the layer lines, if also associated with a high fibre aspect ratio, is a characteristic of amphibole asbestos, and thus has some limited diagnostic value. If a pattern of this type is not obtained, the identity of the fibre is still ambiguous, since the absence of a recognizable pattern may be a consequence of an unsuitable orientation relative to the electron beam, or the fibre may be some other mineral species.

Figure D.4 shows the fibre classification chart to be used for suspected amphibole fibres. This chart shows all the classification paths possible in analysis of a suspected amphibole fibre, when examined systematically by ED and EDXA. Two routes are possible, depending on whether an attempt to obtain an EDXA spectrum or a random orientation ED pattern is made first. The normal procedure for analysis of a sample of unknown origin will be to examine the fibre by random orientation ED, qualitative EDXA, quantitative EDXA, and zone-axis ED, in this sequence. The final fibre classification assigned will be defined either by successful analysis at the maximum required level, or by the instrumental limitations. Any instrumental limitations which affect the quality of the results shall be noted. Record the maximum classification achieved for each fibre on the counting sheet in the appropriate column. The various classification categories can then be combined later in any desired way for calculation of the fibre concentration. The complete record of the results obtained when attempting to identify each fibre can also be used to re-assess the data if necessary.

In the unknown sample, zone-axis analysis will be required if the presence of amphibole is to be unequivocally confirmed. For this level of analysis, attempt to raise the classification of every suspected amphibole fibre to the ADQ category by inspection of the random orientation ED pattern and the EDXA spectrum. In addition, examine at least one fibre from each type of suspected amphibole found by zone-axis methods to confirm their identification. In most cases, because information exists about possible sources of asbestos in close proximity to the air sampling location, some degree of ambiguity of identification can be accepted. Lower levels of analysis can therefore be accepted for these situations.

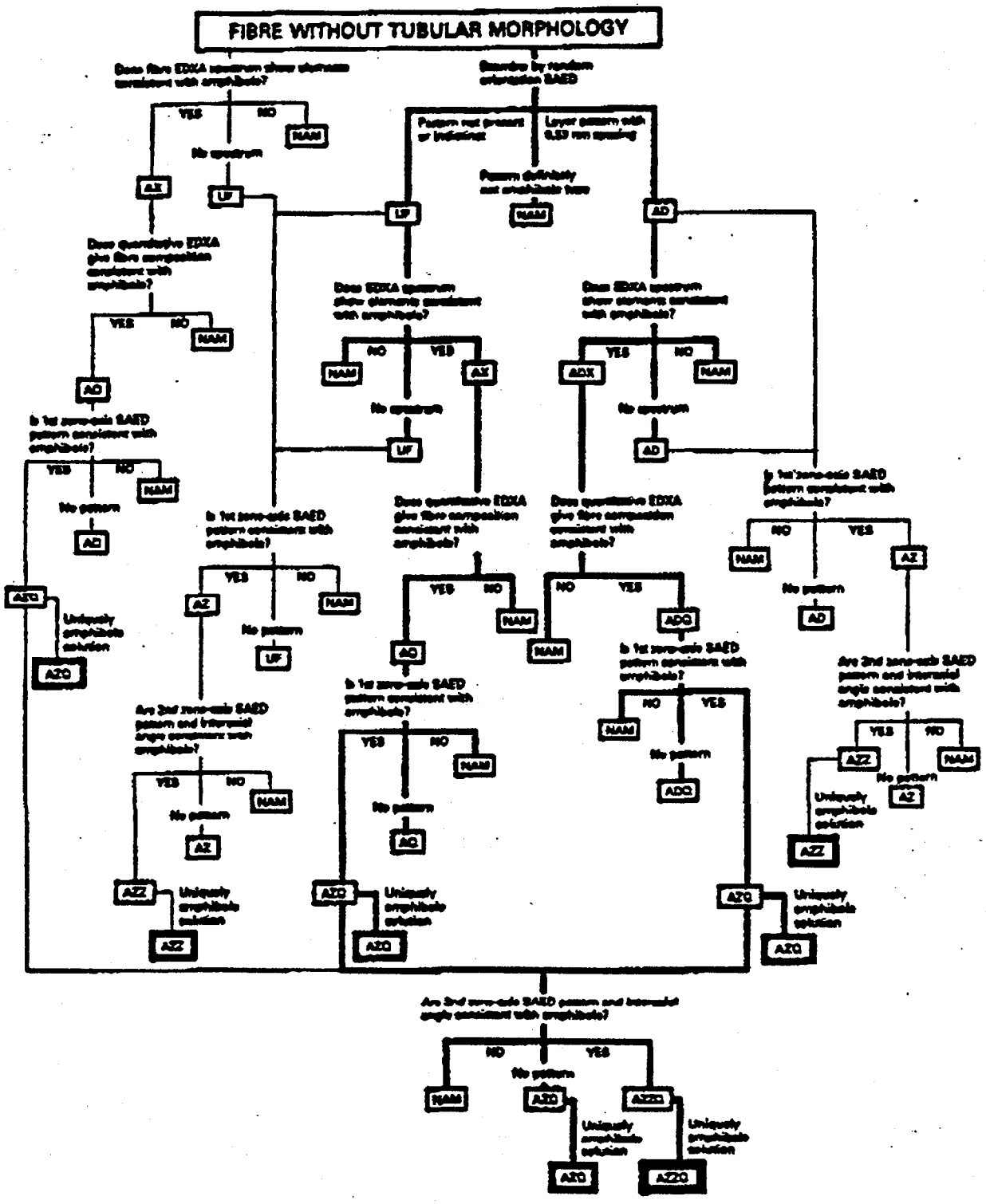

Figure D.4 — Classification chart for fibre without tubular morphology

Annex E
(normative)

Determination of the concentrations of asbestos fibres and bundles longer than 5 µm, and PCM equivalent asbestos fibres

In order to provide increased statistical precision and improved analytical sensitivity for those asbestos fibres and bundles longer than 5 µm, it may be decided to perform additional fibre counting at a lower magnification, taking account only into fibres and bundles within this dimensional range. The result shall be specified as "number of asbestos fibres and bundles longer than 5 µm". For this examination, use a magnification of approximately × 10 000, and continue to assign a morphological code to each structure according to the procedures specified in annex C. Record fibres and bundles only if their lengths exceed 5 µm. Record cluster and matrix components only if their lengths exceed 5 µm.

It may also be decided to provide increased statistical precision and improved analytical sensitivity for fibrous structures longer than 5 µm, with diameters between 0,2 µm and 3,0 µm, which have historically been the basis of risk estimation in the occupational environment (PCM equivalent asbestos fibres). Use a magnification of approximately × 5 000 for this extended fibre count. The result shall be specified as "number of PCM equivalent asbestos fibres". Asbestos structures within this dimensional range do not necessarily incorporate asbestos fibres or bundles longer than 5 µm.

Continue the extended sample examination until 100 asbestos structures have been counted, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity calculated according to table 1. The grid openings examined shall be divided approximately equally between a minimum of two specimen grids.

NOTES

17 The specimen area corresponding to the area of filter examined in the PCM fibre counting methods is 0,785 mm^2, and is equivalent to approximately 100 grid openings of a 200 mesh grid.

18 Some National Standards require that asbestos fibres longer than 2,5 µm, with diameters between 0,2 µm and 3,0 µm be counted. Use a magnification of × 5 000 for counting fibres within these dimensional ranges.

19 The minimum aspect ratio for definition of a fibre in PCM fibre counting methods and in some National Standards is 3/1. Use of a 3/1 aspect ratio is permitted in this International Standard, if this aspect ratio is mentioned in the test report.

The test reports shall include all of the items listed in clause 11.

Annex F
(normative)

Calculation of results

F.1 General

The results should be calculated using the procedures specified below. The results can be conveniently calculated using a computer programme.

F.2 Test for uniformity of distribution of fibrous structures on TEM grids

A chek shall be made using the chi-square test, to determine whether the asbestos structures found on individual grid openings are randomly and uniformly distributed among the grid openings. If the total number found in k grid openings is n, and the areas of the k individual frid openings are designated A_1 to A_k, then the total area of TEM specimen examined is

$$A = \sum_{i=1}^{i=k} A_i$$

The fraction of the total area examined which is represented by the individual grid opening area, p_i, is given by A_i/A. If the structures are randomly and uniformly dispersed over the k grid openings examined, the expected number of structures falling in one grid opening with area A_i is np_i. If the observed number of structures found on that grid opening is n_i, then

$$x^2 = \sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

This value shall be compared with significance points of the chi-square distribution, having $(k-1)$ degrees of freedom. Significance levels lower than 0,1 % may be cause for the sample analysis to be rejected, since this correspond to a very inhomogeneous deposit. If the structure count fails this test, the precision of the result will be uncertain, and if new air samples cannot be collected, additional grid openings may be examined or the sample may be prepared by an indirect method.

F.3 Calculation of the analytical sensitivity

Calculate the required analytical sensitivity S, expressed in number of structures per litre, using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

- A_f is the area, in square millimetres, of sample collection filter;
- A_g is the area, in square millimetres, of TEM specimen grid opening;
- k is the number of grid openings examined;
- V is the volume of air sampled, in litres.

F.4 Calculation of the mean and confidence interval of the structure concentration

In the structure count made according to this International Standard, a number of grid openings have been sampled from a population of grid openings, and it is required to determine the mean grid opening structure count for the population on the basis of this small sample. The interval about the sample mean which, with 95 % confidence, contains the population mean, is also required.

F.4.1 Calculation of the mean structure concentration

Calculate the mean structure concentration C, expressed in number of structures per litre, using the following equation:

$$C = Sn$$

where

S is the analytical sensitivity, expressed in number of structures per litre;

n is the total number of structures found on all grid openings examined.

F.4.2 Calculation of confidence intervals

The distribution of structures on the grid openings should theoretically approximate to a Poisson distribution. Because of fibre aggregation and size-dependent identification effects, the actual structure counting data often does not conform to the Poisson distribution, particularly at high structure counts. An assumption that the structure counting data are distributed according to the Poisson distribution can therefore lead to confidence intervals narrower than are justified by the data. Moreover, if the Poisson distribution is assumed, the variance is related only to the total number of structures counted. Thus, a particular structure count conducted on one grid opening is considered to have the same confidence interval as that for the same number of structures found on many grid openings. However, the area of sample actually counted is very small in relation to the total area of the filter, and for this reason, structures shall be counted on a minimum of four grid openings taken from different areas of the filter in order to ensure that a representative evaluation of the deposit is made.

At high structure counts, where there are adequate numbers of structures per grid opening to allow a sample estimate of the variance to be made, the distribution can be approximated to a Gaussian, with independent values for the mean and variance. Where the sample estimate of variance exceeds that implicit in the Poissonian assumption, use of Gaussian statistics with the variance defined by the actual data is the most conservative approach to calculation of confidence intervals.

At low structure counts, it is not possible to obtain a reliable sample estimate of the variance, and the distribution also becomes asymmetric but not necessarily Poissonian. For 30 structures and below, the distribution becomes asymmetric enough for the fit to a Gaussian to no longer be a reasonable one, and estimates of sample variance are unreliable. Accordingly, for counts below 31 structures, the assumption of a Poisson distribution shall be made for calculation of the confidence intervals.

F.4.3 Example of calculation of Poissonian 95 % confidence intervals

For total structure counts less than 4, the lower 95 % confidence limit corresponds to less than 1 structure. Therefore, it is not meaningful to quote lower confidence interval points for structure counts of less than 4, and the result shall be recorded as "less than" the corresponding one-sided upper 95 % confidence limit of the Poisson distribution, as follows:

0 structure ≙ 2,99 times the analytical sensitivity

1 structure ≙ 4,74 times the analytical sensitivity

2 structures ≙ 6,30 times the analytical sensitivity

3 structures ≙ 7,75 times the analytical sensitivity

For total counts exceeding 4, the 95 % confidence interval shall be calculated using the values shown in table F.1. Table F.1 gives the upper and lower limits of the two-sided Poissonian 95 % confidence interval for structure counts up to 470.

F.4.4 Example of calculation of Gaussian 95 % confidence intervals

Calculate the sample estimate of variance s^2 using the following equation:

$$s^2 = \frac{\sum_{i=1}^{i=k} (n_i - np_i)^2}{(k-1)}$$

where

n_i is the number of structures on the ith grid opening;

n is the total number of structures found in k grid openings;

p_i is the fraction of the total area examined represented by the ith grid opening;

k is the number of grid openings examined.

If the mean value of the structure count is calculated to be n, the upper and lower values of the Gaussian 95 % confidence interval are given respectively by

$$L_u = \frac{n}{k} + \frac{ts}{\sqrt{k}}$$

and

$$L_l = \frac{n}{k} - \frac{ts}{\sqrt{k}}$$

where

L_u is the upper 95 % confidence limit;

L_l is the lower 95 % confidence limit;

n is the total number of structures in all grid openings examined;

t is the value of Student's test (probability 0,975) for $(k - 1)$ degrees of freedom;

s is the standard deviation (square root of sample estimate of variance);

k is the number of grid openings examined.

F.4.5 Summary of procedure for calculation of results

In summary, structure counting data shall be calculated as follows:

No structures detected

The structure concentration shall be reported as less than the concentration equivalent of the one-sided upper 95 % confidence limit of the Poisson distribution. This is equal to 2,99 times the analytical sensitivity.

From 1 to 3 structures

When 1 to 3 structures are counted, the result shall be reported as less than the corresponding one-sided upper 95 % confidence limit for the Poisson distribution. These are

1 structure ≙ 4,74 times the analytical sensitivity

2 structures ≙ 6,30 times the analytical sensitivity

3 structures ≙ 7,75 times the analytical sensitivity

From 4 to 30 structures

The mean structure concentration and the 95 % confidence intervals shall be reported on the basis of the Poissonian assumption, using the values shown in table F.1.

More than 30 structures

When more 30 structures are counted, both the Gaussian 95 % confidence interval and the Poissonian 95 % confidence interval shall be calculated. The larger of these two intervals shall be used to express the precision of the structure concentration. When the Gaussian 95 % confidence interval is selected for data reporting, the Poissonian 95 % confidence interval shall also be mentioned.

F.5 Calculation of structure length, width, and aspect ratio distributions

The distributions all approximate to logarithmic-normal, and therefore the size range intervals for calculation of the distribution shall be spaced logarithmically. The other characteristics required for the choice of size intervals are that they should allow for a sufficient number of size classes, while still retaining a statistically valid number of structures in each class. Interpretation is also facilitated if each size class repeats at 10 intervals, and if 5 µm is a size class boundary. A ratio from one class to the next of 1,468 satisfies all of these requirements and this value shall be used. The distributions, being approximately logarithmic-normal, when presented graphically, shall be plotted using a logarithmic ordinate scale and a Gaussian abscissa.

F.5.1 Calculation of structure length cumulative number distribution

This distribution allows the fraction of the total number of structures either shorter or longer than a given length to be determined. It is calculated using the following equation:

$$C(P)_k = \frac{\sum_{i=1}^{i=k} n_i}{\sum_{i=1}^{i=P} n_i} \times 100$$

where

$C(P)_k$ is the cumulative number percentage of structures which have lengths less than the upper bound of the kth class;

n_i is the number of structures in the ith length class;

P is the total number of length classes.

F.5.2 Calculation of structure width cumulative number distribution

This distribution allows the fraction of the total number of structures either narrower or wider than a given width to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure widths.

F.5.3 Calculation of structure aspect ratio cumulative number distribution

This distribution allows the fraction of the total number of structures which have aspect ratios either smaller or larger than a given aspect ratio to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure aspect ratios.

Table F.1 — Upper and lower limits of the Poissonian 95 % confidence interval of a count

Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit
0	0	3,689[1]	46	33,678	61,358	92	74,164	112,83
1	0,025	5,572	47	34,534	62,501	93	75,061	113,94
2	0,242	7,225	48	35,392	63,642	94	75,959	115,04
3	0,619	8,767	49	36,251	64,781	95	76,858	116,14
4	1,090	10,242	50	37,112	65,919	96	77,757	117,24
5	1,624	11,669	51	37,973	67,056	97	78,657	118,34
6	2,202	13,060	52	38,837	68,192	98	79,557	119,44
7	2,814	14,423	53	39,701	69,326	99	80,458	120,53
8	3,454	15,764	54	40,567	70,459	100	81,360	121,66
9	4,115	17,085	55	41,433	71,591	110	90,400	132,61
10	4,795	18,391	56	42,301	72,721	120	99,490	143,52
11	5,491	19,683	57	43,171	73,851	130	108,61	154,39
12	6,201	20,962	58	44,041	74,979	140	117,77	165,23
13	6,922	22,231	59	44,912	76,106	150	126,96	176,04
14	7,654	23,490	60	45,785	77,232	160	136,17	186,83
15	8,396	24,741	61	46,658	78,357	170	145,41	197,59
16	9,146	25,983	62	47,533	79,482	180	154,66	208,33
17	9,904	27,219	63	48,409	80,605	190	163,94	219,05
18	10,668	28,448	64	49,286	81,727	200	173,24	229,75
19	11,440	29,671	65	50,164	82,848	210	182,56	240,43
20	12,217	30,889	66	51,042	83,969	220	191,89	251,10
21	13,00	32,101	67	51,922	85,088	230	201,24	261,75
22	13,788	33,309	68	52,803	86,207	240	210,60	272,39
23	14,581	34,512	69	53,685	87,324	250	219,97	283,01
24	15,378	35,711	70	54,567	88,441	260	229,36	293,62
25	16,178	36,905	71	55,451	89,557	270	238,75	304,23
26	16,983	38,097	72	56,335	90,673	280	248,16	314,82
27	17,793	39,284	73	57,220	91,787	290	257,58	325,39
28	18,606	40,468	74	58,106	92,901	300	267,01	335,96
29	19,422	41,649	75	58,993	94,014	310	276,45	346,52
30	20,241	42,827	76	59,880	95,126	320	285,90	357,08
31	21,063	44,002	77	60,768	96,237	330	295,36	367,62
32	21,888	45,175	78	61,657	97,348	340	304,82	378,15
33	22,715	46,345	79	62,547	98,458	350	314,29	388,68
34	23,545	47,512	80	63,437	99,567	360	323,77	399,20
35	24,378	48,677	81	64,328	100,68	370	333,26	409,71
36	25,213	49,840	82	65,219	101,79	380	342,75	420,22
37	26,050	51,000	83	66,111	102,90	390	352,25	430,72
38	26,890	52,158	84	67,003	104,00	400	361,76	441,21
39	27,732	53,319	85	67,897	105,11	410	371,27	451,69
40	28,575	54,469	86	68,790	106,21	420	380,79	462,18
41	29,421	55,622	87	69,684	107,32	430	390,32	472,65
42	30,269	56,772	88	70,579	108,42	440	399,85	483,12
43	31,119	57,921	89	71,474	109,53	450	409,38	493,58
44	31,970	59,068	90	72,370	110,63	460	418,92	504,04
45	32,823	60,214	91	73,267	111,73	470	428,47	514,50

1) The one-sided upper 95 % confidence limit for 0 structures is 2,99.

Annex G
(informative)

Strategies for collection of air samples

G.1 General

An important part of the sampling strategy is a statement of the purpose of the sampling programme. A sufficient number of samples should be collected so that the site is well characterized to the precision and accuracy desired, and also ensure that sample filters appropriately loaded for TEM analysis are obtained from all of the sampling locations.

G.2 Air sample collection in the outdoors environment

Weather conditions restrict the ability to collect satisfactory air samples in the outdoors environment, and whenever possible, sampling should be carried out in low-wind, low-humidity conditions. Detailed records of the weather conditions, windspeed and direction during the sampling period should be made. All available information concerning local topography, and the types and positions of sources should be recorded.

Sequential multipoint sampling is necessary to provide adequate characterization of complex sites and sources. It is recommended that multiple samples are taken upwind and downwind of the site, with a minimum of two samples in the downwind position expected to experience the maximum airborne concentration. The locations of the samplers should be carefully recorded.

G.3 Air sample collection inside buildings

Air samples are often collected inside buildings in which asbestos-containing construction materials are present, in order to determine whether these materials contribute to the asbestos fibre concentration in the building atmosphere. The optimum positions for collection of air samples can only be determined after a complete survey of the building to establish air movement patterns. Multiple samples should be collected in the area where asbestos building materials are present, and control samples should be collected in an adjacent area where no airborne asbestos fibres would be expected. The intakes for air conditioning systems are frequently used as the collection locations for control samples. Whenever possible, static samples should be taken over a period exceeding 4 h during normal activity in the building, at face velocities of between 4 cm/s and 25 cm/s.

Annex H
(informative)

Methods for removal of gypsum fibres

It is common to find fibres of calcium sulfate (gypsum) in airborne particulates collected in buildings and urban environments, and particularly in samples collected where demolition or construction work is in progress. The fibres are readily released when plasters and cement products are disturbed. In some circumstances, particles of calcite or dolomite collected on an air filter can react with atmospheric sulfur dioxide, to form long fibres of gypsum. Gypsum fibres can give rise to high fibre counts by both optical and electron microscopy. The gypsum fibres are often 2 µm to 6 µm long, with aspect ratios greater than 10/1. Sometimes, these fibres appear similar to amphibole asbestos fibres, and in some samples they can be morphologically very similar to chrysotile. In the TEM, the larger fibres have high contrast and at high magnification often exhibit a characteristic mottled appearance which changes under electron beam irradiation. Some gypsum fibres, however, are not easily discriminated from asbestos without examination by EDXA. TEM specimens which contain many such gypsum fibres require an extended examination time in the TEM, because it is necessary to examine each of these fibres by EDXA before it can be rejected.

It is possible to remove gypsum fibres selectively by water extraction. A Jaffe washer (7.3.7), or a condensation washer (7.3.8), should be prepared, but using a water (6.1) as the solvent. The TEM specimens, which have been previously prepared and initially examined in the TEM, should be placed in the washer to allow dissolution of the fibres. If a Jaffe washer is used, the treatment time can be reduced by heating the washer to 90 °C to 100 °C for a few minutes. If a condensation washer is used, the gypsum fibres will be dissolved by treatment for approximately 10 min. The effect of this treatment is to remove the gypsum fibres, leaving carbon replicas (7.3.11) which are readily distinguished from asbestos fibres.

NOTE 20 This procedure should be used only when examination of the untreated TEM specimen grids shows the gypsum fibres to be isolated from any asbestos fibres present. Losses of asbestos fibres may occur if matrices of gypsum and asbestos are exposed to this procedure.

Annex J
(informative)

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Appendix 12

(EPA SOP 2015) Asbestos Sampling

ASBESTOS SAMPLING

SOP#: 2015
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method for TEM[2]; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)[3]. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)[4] and its addendum 40 CFR 763 (October 30, 1987)[4] provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 μm[5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and

medical surveillance[5,6].

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.
- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

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.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2 µm mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m^3).

	Concentration	Flow Rate
• Low RAM readings:	<6.0 mg/m^3	11-15. L/min
• Medium RAM readings:	>6.0 mg/m$_3$	7.5 L/min
• High RAM readings:	>10. mg/m^3	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, 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 (3,000 to 10,000 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. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump.Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.

3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.

2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.

4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.

(2) Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.

(3) National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.

(4) U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A

Tables

TABLE 1.

SAMPLE STATIONS FOR OUTDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Upwind/Background[1]	Collect a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

APPENDIX A (Cont'd)

Tables

TABLE 2

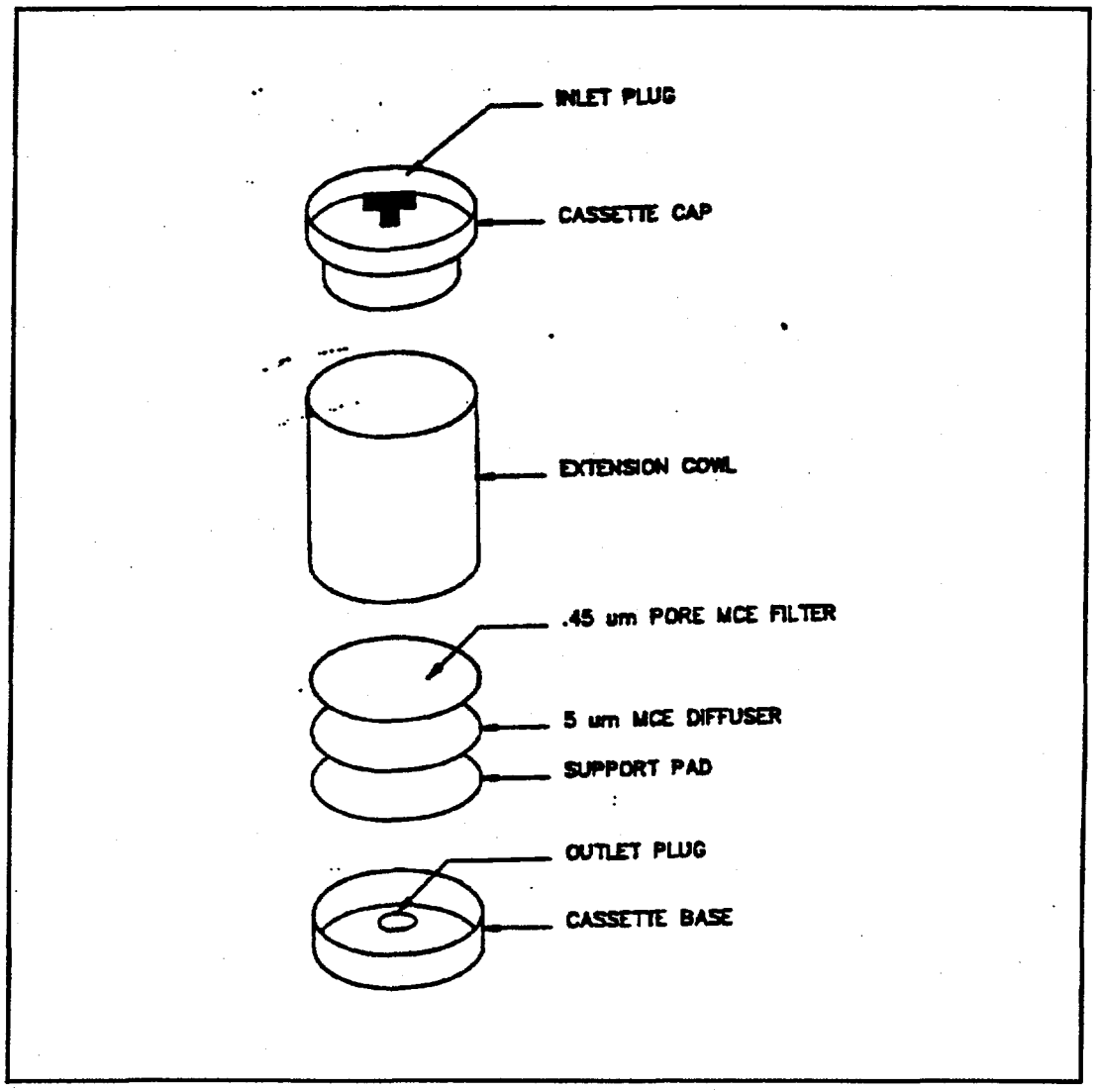
SAMPLE STATIONS FOR INDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

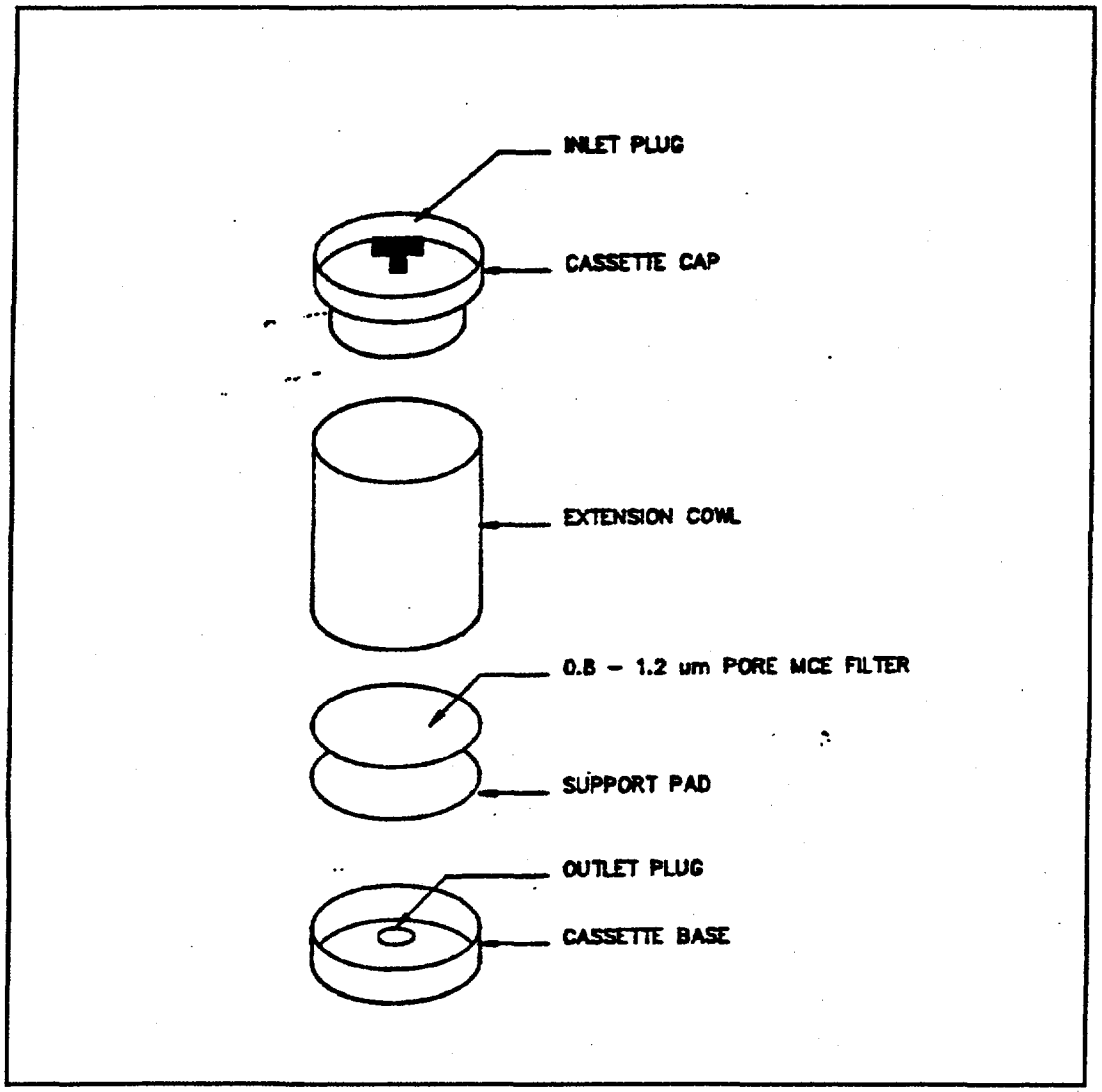
FIGURE 1. Transmission Electron Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

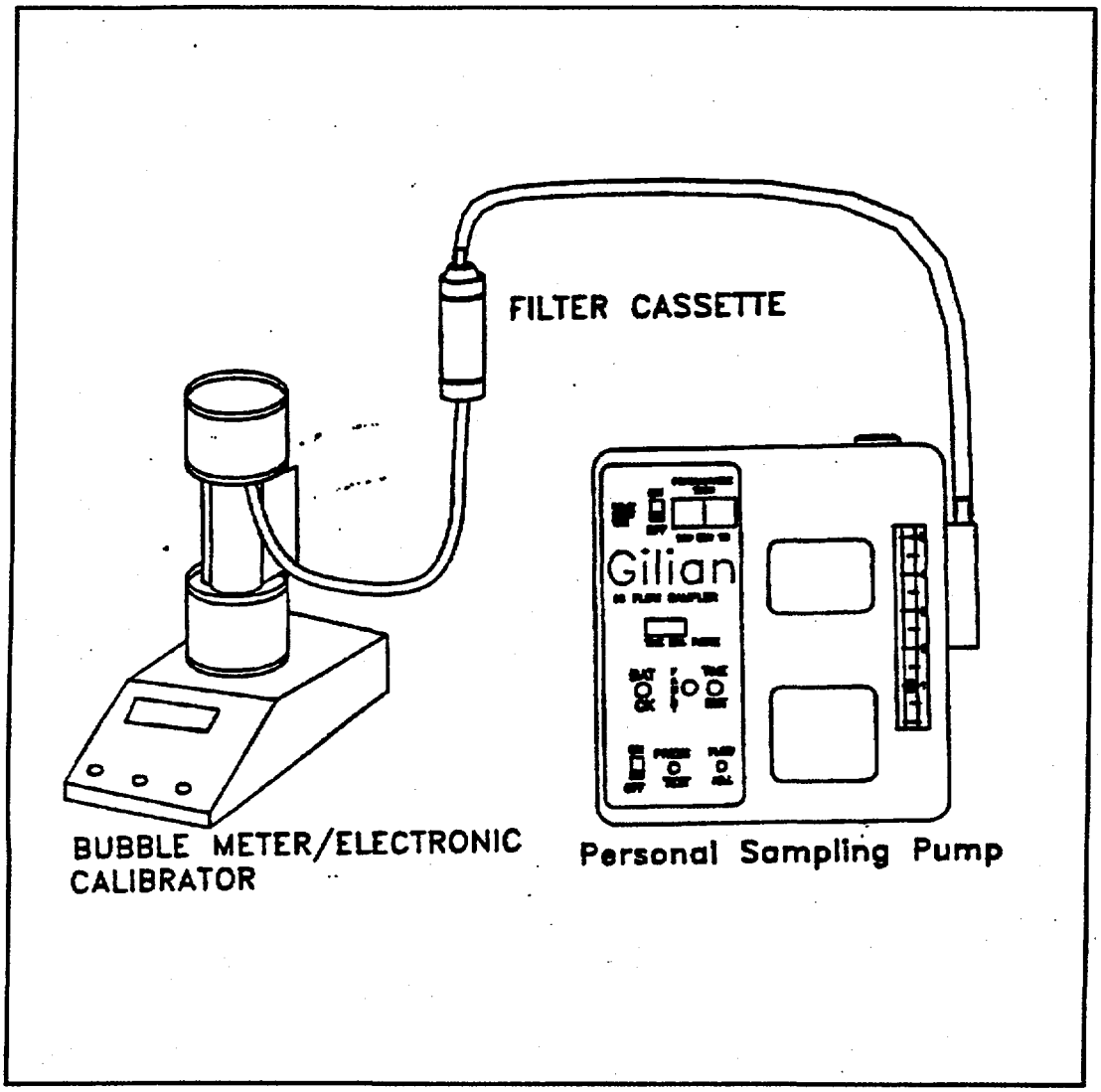
FIGURE 2. Phase Contrast Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

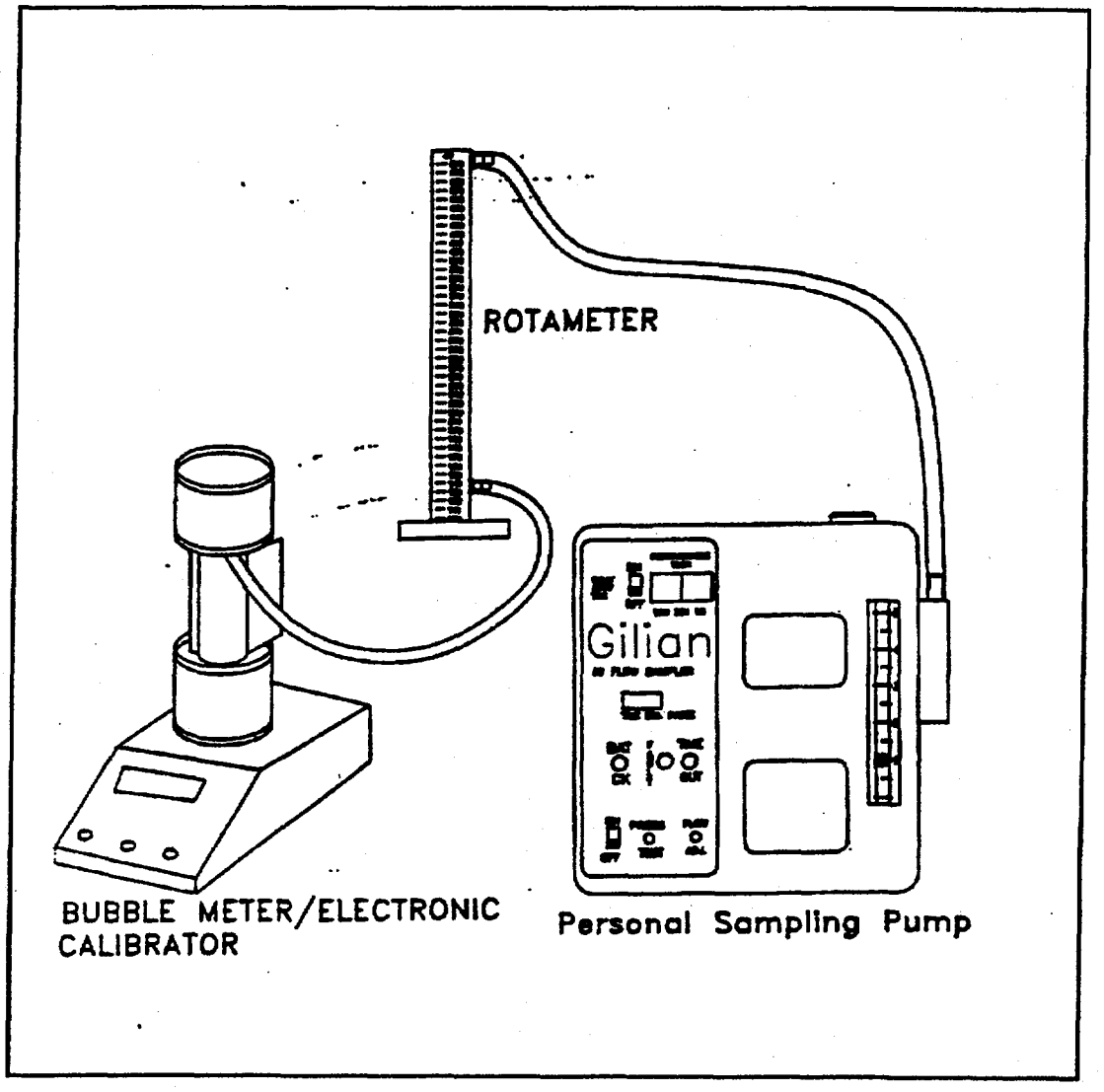
FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter



APPENDIX B (Cont'd)

Figures

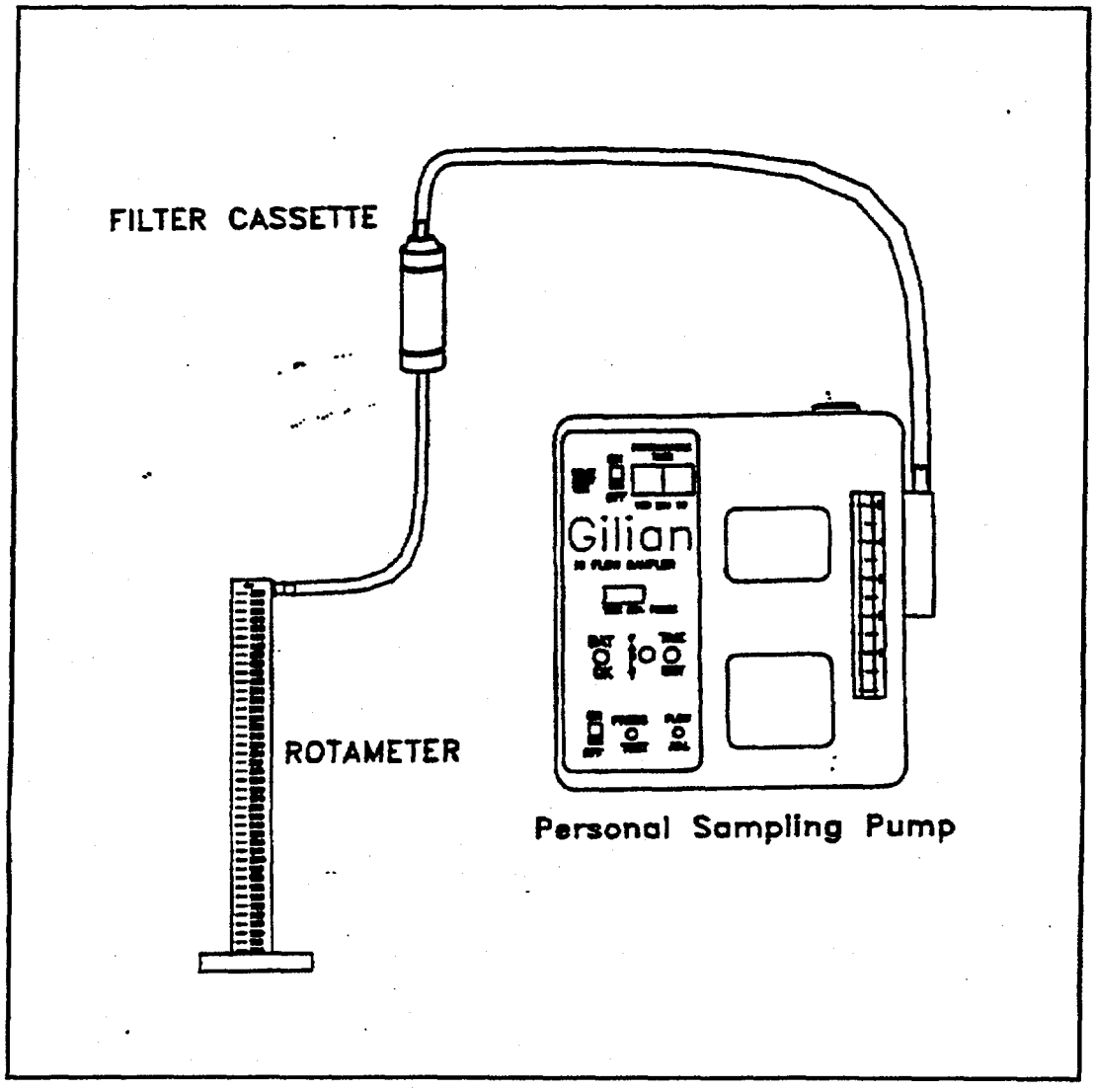
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



APPENDIX B (Cont'd)

Figures

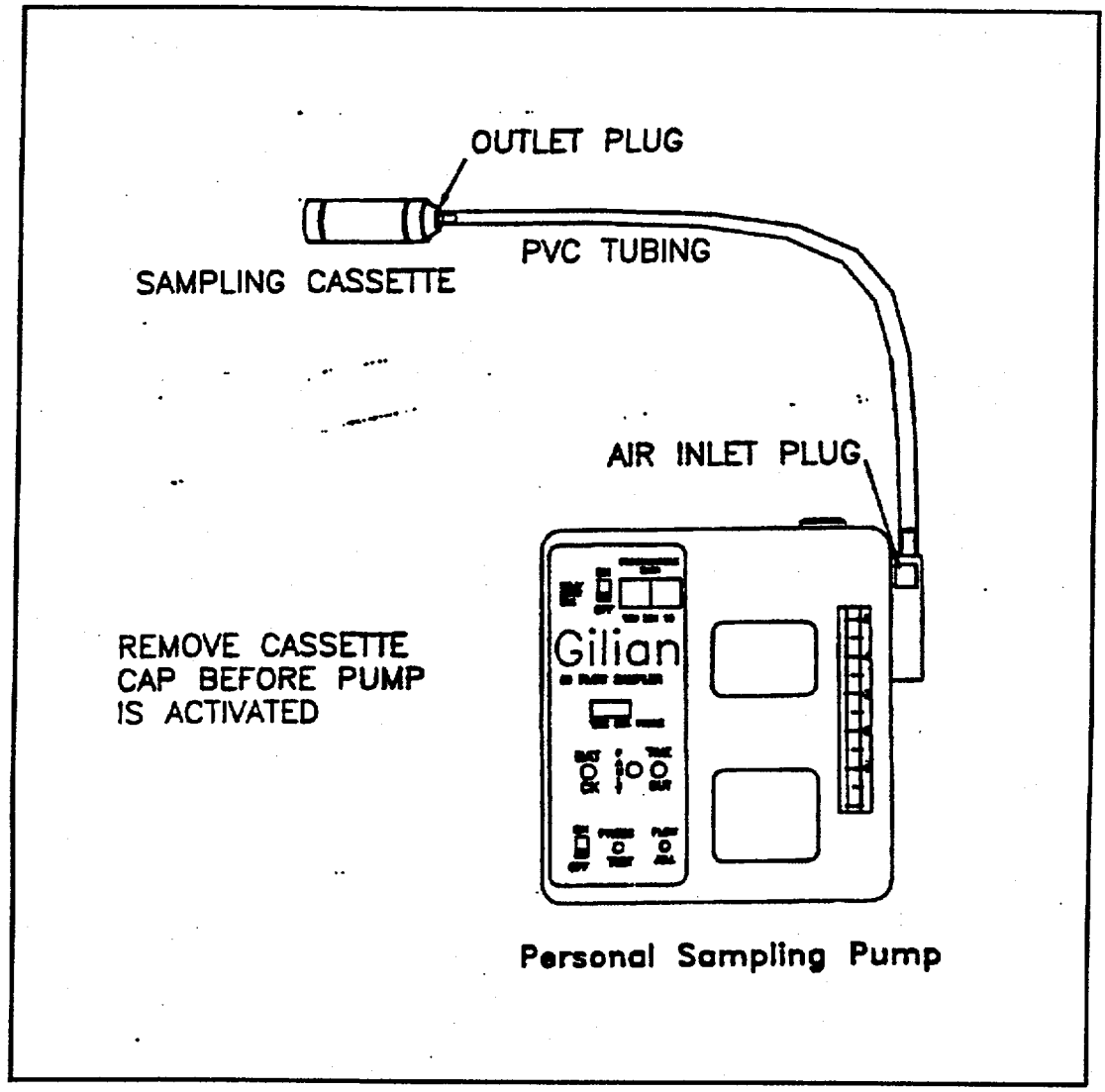
FIGURE 5. Calibrating a Sampling Pump with a Rotameter



APPENDIX B (Cont'd)

Figures

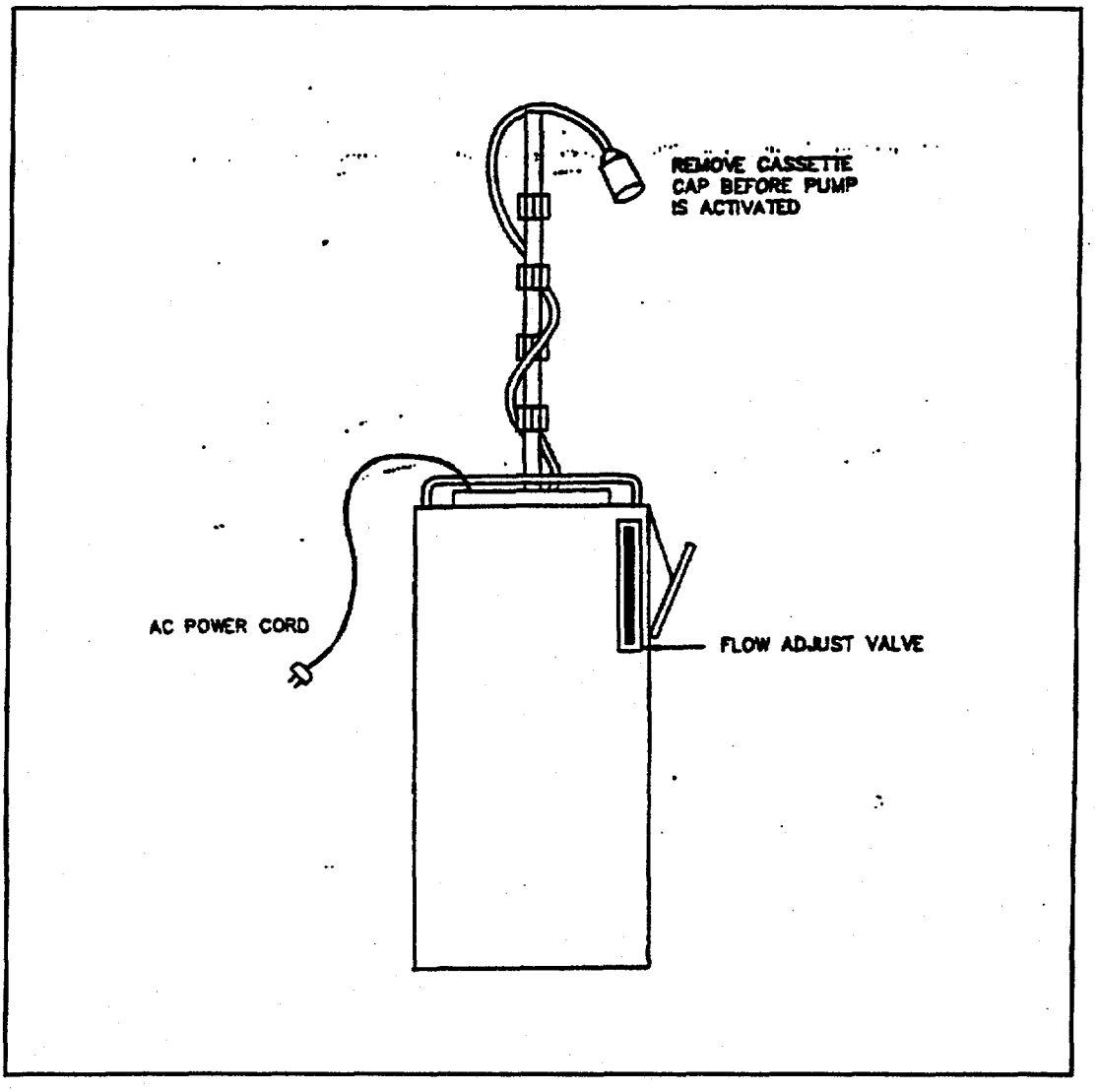
FIGURE 6. Personal Sampling Train for Asbestos



APPENDIX B (Cont'd)

Figures

FIGURE 7. High Flow Sampling Train for Asbestos



Appendix 13

(ISSI-Libby-01) Soil Sample Preparation

Date: January 7, 2000 (Rev. # 1) SOP No. ISSI-LIBBY-01

Title: SOIL SAMPLE PREPARATION

APPROVALS:

Author Adrian Bradley ISSI Consulting Group Inc. Date: December 22, 1999

SYNOPSIS: A standardized method for homogenization of surface soil samples is described. Protocols for sample preparation and handling are provided.

Received by QA Unit:

REVIEWS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8		1/7/00
ISSI Consulting Group, Inc.	W J Beatty	1/7/00

Revision Date	Reason for Revision
1/7/99	Incorporation of sieving to the sample preparation.

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for homogenizing surface soil samples. This procedure will be used by employees of USEPA Region 8 and by contractors/subcontractors supporting USEPA Region 8 projects and tasks. This SOP describes the equipment and operations used for homogenizing surface soil samples in a manner that will produce data that can be used to support risk evaluations. Site-specific deviations from the procedures outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager, or Regional Toxicologist prior to initiation of the sampling activity.

2.0 RESPONSIBILITIES

The Field Project Leader (FPL) may be an USEPA employee or contractor who is responsible for overseeing the surface soil sampling activities. The FPL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to communicate with the Field Personnel regarding specific collection objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the FPL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (Remedial Project Manager, or Regional Toxicologist).

Field personnel performing surface soil sampling are responsible for adhering to the applicable tasks outlined in this procedure while homogenizing surface soil samples.

3.0 EQUIPMENT

- General purpose laboratory oven - must be capable of maintaining a constant temperature of approximately 103-105°C.
- Sample drying trays - capable of holding an even layer of the complete sample volume of each sample. To minimize the decontamination effort, disposable drying trays are recommended.
- Analytical balance - accurate to 0.1 g, range of 0.1 g to 1000 g
- Riffle splitter - with 3/4 to 1 inch chutes to split samples
- Stainless steel or teflon scoop or spoon - for transfering samples
- Collection containers - plastic ziplock bags.

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Field clothing and Personal Protective Equipment - as specified in the Health and Safety Plan.

- Field notebook -used to record progress, any problems or observations.

- Permanent marking pen - used to label sample containers.

- Three-ring binder book - binders will contain Soil Preparation Sheets, Field Split Sample Log sheets, and sample labels.

- Trash Bag - used to dispose of gloves and wipes.

4.0 METHOD SUMMARY

Soil samples will be dried in a standard laboratory oven, then homogenized and split for subsequent analysis.

5.0 BULK SOIL DRYING

Set the oven temperature to 103-105 °C (not to exceed 115 °C). Establish the drying time by weighing a representative sample before drying, at estimated completion, and following an additional 15 minute drying time to confirm stable weight. Verify that the sample is completely dry using the "squeeze test", squeezing a portion of the sample between a freshly gloved thumb and forefinger. Sample dryness is indicated by a lack of cohesiveness in the soil.

Prior to drying each sample, record the weight on the Sample Preparation Logbook Sheet. Spread the sample on the drying tray in an even layer to promote even drying. Check the oven temperature to verify that proper temperature has been reached. Mark each tray with the sample ID number. Place the drying trays containing the samples in the oven. Leave the samples in the oven until completely dry. Verify that each sample is dry by testing cohesiveness using a freshly gloved thumb and forefinger. Record the weight after drying on the Sample Preparation Logbook Sheet. Document the sample drying time for each sample on the Soil Preparation Data Sheet (Attachment 1).

When samples are dry, remove from the oven area and place in the ventilation area. **Before placing samples in the ventilation area, verify that the hood is turned on.** A new pair of gloves must be worn for each sample.

The sample should be coarse sieved and the mesh size recorded. Pour the material which passed through the sieve into a new sample bag, and mark the outside of the bag with the sample ID.

Gently knead contents of the bag to break up any remaining soil clumps. Completely seal the bag, then mix by turning the bag end-over-end slowly, for a minimum of ten times.

6.0 SAMPLE SPLITTING

Following the procedures outlined in Section 5.0, the soil sample should be well-homogenized. With the hood turned on, open the sample bag and use a clean and dry riffle splitter to split each sample.

The following method for splitting a soil sample was adapted from EPA 540-R-97-028 (USEPA, 1997). The sample is split by placing soil onto a splitter tray. Shake the tray to evenly distribute the sample. Place the long lip of the tray against the long lip of the splitter hopper and slowly rotate the tray so that the sample slowly empties into the splitter and slides down the near wall of the hopper to the chutes, collecting the sample in two receiving trays. Tap the sample tray vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving trays. The corners and nooks of the splitter may be cleaned with a coarse nylon brush.

Pour the material from one of the receiving trays into a clean bucket and tap the tray vigorously to assure complete transfer. This portion is designated for archive. The original sample tray (which is now empty), and the emptied receiving tray should be placed under the splitter as the new receiving trays.

Repeat the process of dispersing the remaining sample material (containing half the mass of the original sample) by shaking the sample tray so that it is uniformly distributed. Repeat the procedure described above for splitting the sample. At the end of the second split, carefully transfer the material from each of the receiving trays into a clean, pre-weighed sample bag to weighed and packaged for shipment to the laboratory and to W.R. Grace. Record each split sample ID, and the original sample ID on the Field Split Sample Log sheet (Attachment 1).

7.0 FIELD DOCUMENTATION

Each sample ID must be recorded on the data sheets. Original sample ID numbers are recorded on the Soil Preparation Sheets, and the Field Split Sample Log sheets. When the original sample is split, the original sample ID number, and each new sample, must be recorded.

In addition, a field notebook should be maintained by each individual or team that is preparing samples. For each day that samples are processed, the following information should be collected.

- date
- time
- personnel

- weather conditions
- analytical balance calibration
- drying oven temperature
- descriptions of any deviations to the Project Plan and the reason for the deviation

Field personnel will prepare the proper type and quantity of quality control samples as prescribed in the Project Plan.

8.0 DECONTAMINATION

All non-dedicated equipment used during sample preparation must be decontaminated prior to use. It is recommended that disposable oven trays be used to minimize the decontamination effort. Stainless steel or teflon scoops or spoons, splitters, sieves and drying trays that will be re-used, must be decontaminated with de-ionized (DI) water and disposable wipes or towels. DI water is poured over the equipment, then wiped, then rinsed again with DI water. If soil particles are visible on any of the equipment, repeat this procedure until the equipment is clean. All equipment must be dry before it is re-used.

9.0 GLOSSARY

Project Plan - The written document that spells out the detailed site-specific procedures to be followed by the Project Leader and the Field Personnel.

11.0 REFERENCES

American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size, ASTM Designation: C 702 - 98, 4 p.

USEAP. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. EPA 540-R-97-028.

ATTACHMENT 1

Sample Preparation Logbook Sheet

Sample ID	Prep Batch Number	Sample Drying							Notes			
		Date/Time Drying Begun [a]	Date/Time Drying Completed [a]	Oven Temp (C)	Sample Mass [b] (grams)							
					Before	After 1	After 2	After 3				

[a] - Enter date in the following format: mm/dd/yy; enter time as 24-hour time (e.g., 1340)

[b] - At least 2 mass measurements will be recorded. The sample is completely dry if the mass measurement is stable.

Field Split Sample Logsheet

Date	Sample ID	Original Sample ID	Prepared By	Date Prepared	Notes

Appendix 14

(EPA 540/2-90/005a) Superfund Method for the Determination of Asbestos in Ambient Air

United States
Environmental Protection
Agency

Office of Emergency and
Remedial Response
Washington, DC 20460

EPA/540/2-90/005a
May 1990

Superfund

EPA

Environmental Asbestos Assessment Manual

Superfund Method for the Determination of Asbestos in Ambient Air

Part 1: Method

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7. EQUIPMENT AND APPARATUS

7.1. AIR SAMPLING - EQUIPMENT AND CONSUMABLE SUPPLIES

7.1.1. Filter Cassette

- Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 μm, and supplied from a lot number which has been qualified as low background for asbestos determination. The filter shall be backed by a 5 μm pore size MCE filter, and supported adequately so that distortion of the filter by the differential pressure across it does not occur during sampling. The cassettes shall be purchased with the required filters in position, or shall be assembled in a laminar flow hood or clean area such that the filter blank determinations meet the criteria of Section 10.6. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly so that significant air leakage around the filter cannot occur.

7.1.2. Sampling Pump

The sampling pump shall be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled. The face velocity through the filter shall be between 4.0 cm/s and 65.0 cm/s. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ±10% throughout the sampling period. A constant flow or critical orifice controlled pump meets these requirements. Flexible tubing shall be used to connect the filter cassette to the sampling pump. A means for calibration of the flow-rate of each pump is also required.

7.1.3. Stand

A stand shall be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

7.1.4. Rotameter

A rotameter or other flow measuring device calibrated such that the operator can measure flow rates to ± 5% accuracy at the expected sampling flow rate.

7.2. SPECIMEN PREPARATION LABORATORY

Asbestos, particularly chrysotile, is present in varying quantities in many laboratory reagents. Many building materials also contain significant amounts of asbestos or other mineral fibers that may interfere with the analysis if they are inadvertently introduced during preparation of specimens. It is most important to ensure that during preparation, contamination of TEM specimens by any extraneous asbestos fibers is minimized. All specimen preparation steps shall therefore be performed in an environment where contamination of the sample is minimized. The primary requirement of the sample preparation laboratory is that a blank determination shall yield a result which will meet the specifications in 10.6. A minimum facility considered suitable for preparation of TEM specimens is a laminar flow hood. However, it has been established that work practices in specimen preparation appear to be more important than the type of clean handling facilities in use. Preparation of samples shall be carried out only after acceptable blank values have been demonstrated.

NOTE

Activities involving manipulation of bulk asbestos shall not be performed in the same area as TEM specimen preparation, because of the possibilities of contaminating the TEM specimens.

7.3. LABORATORY EQUIPMENT

7.3.1. Transmission Electron Microscope

A TEM operating at an accelerating potential of 80-120 kV, with a resolution better than 1.0 nm, and a magnification range of approximately 300 to 100,000 shall be used. The ability to obtain a direct screen magnification of about 100,000 is necessary for inspection of fiber morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. It is also required that the viewing screen be calibrated (as shown in Figure 7.1) with concentric circles and a millimeter scale such that the lengths and widths of fiber images down to 1 mm width can be measured in increments of 1 mm.

For Bragg angles less than 0.01 radians, the TEM shall be capable of performing ED from an area of 0.6 μm^2 or less, selected from an in-focus image at a screen magnification of 20,000. This performance requirement defines the minimum separation between particles at which independent ED patterns can be obtained from

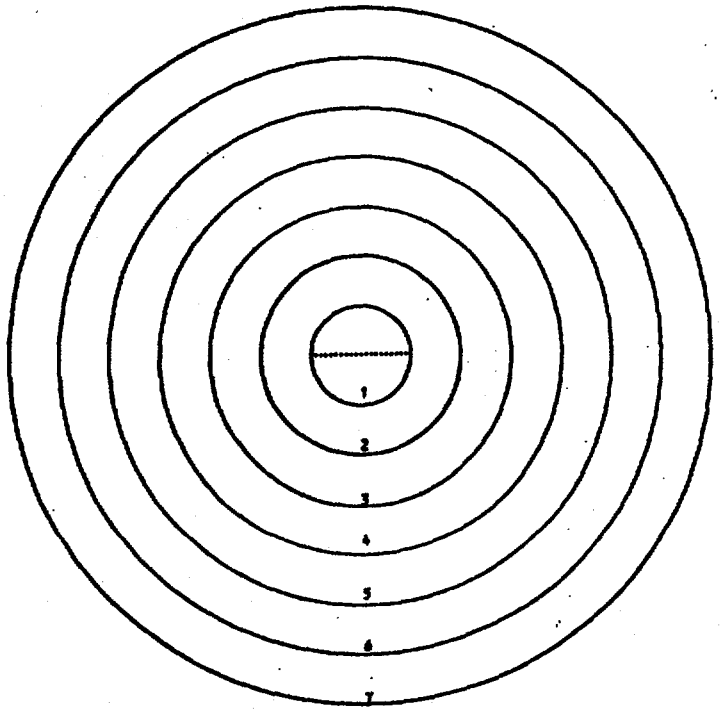

Figure 7.1: Calibration Markings on the TEM Viewing Screen

each particle. If SAED is used, the performance of a particular instrument may normally be calculated using the following relationship:

$$A = 0.7854 \times (D/M + 2000 \times C_s\theta^3)^2$$

Where:
- A = the effective SAED area in μm^2
- D = the diameter of the SAED aperture in μm
- M = the magnification of the objective lens
- C_s = the spherical aberration coefficient of the objective lens in mm
- θ = maximum required Bragg angle in radians

It is not possible to reduce the effective SAED area indefinitely by the use of progressively smaller SAED apertures, because there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

If zone-axis ED analyses are to be performed, the TEM shall incorporate a goniometer stage which permits the TEM specimen to be either:

(a) rotated through 360°, combined with tilting through at least +30° to -30° about an axis in the plane of the specimen; or,

(b) tilted through at least +30° to -30° about two perpendicular axes in the plane of the specimen.

The analysis is greatly facilitated if the goniometer permits eucentric tilting, although this is not essential. If EDXA and zone-axis ED are required on the same fiber, the goniometer shall be of a type which permits tilting of the specimen and acquisition of EDXA spectra without change of specimen holder.

The TEM shall have an illumination and condenser lens system capable of forming an electron probe smaller than 250 nm in diameter.

Use of an anti-contamination trap around the specimen is recommended if the required instrumental performance is to be maintained.

7.3.2. Energy Dispersive X-ray Analyzer

The TEM shall be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 175 eV (FWHM) on the MnK_α peak. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of

geometrical factors, the required performance of the combination of the TEM and X-ray analyzer is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid state X-ray detectors are least sensitive in the low energy region, and so measurement of sodium in crocidolite shall be the performance criterion. The combination of electron microscope and X-ray analyzer shall yield, under routine analytical conditions, a background-subtracted NaK_α integrated peak count rate of more than 1 count per second (cps) from a 50 nm diameter fiber of UICC crocidolite irradiated by a 250 nm diameter electron probe at an accelerating potential of 80 kV. The peak/background ratio for this performance test shall exceed 1.0.

The EDXA unit shall provide the means for subtraction of the background, identification of elemental peaks, and calculation of background-subtracted peak areas.

7.3.3. Computer

Many repetitive numerical calculations are necessary, and these may be performed conveniently by relatively simple computer programs. For analyses of zone-axis ED pattern measurements, a computer with adequate memory is required to accommodate the more complex programs involved.

7.3.4. Plasma Asher

For ashing of filters or particulate deposits washed from filters, a plasma asher, with a radio frequency power rating of 100 W or higher, shall be used. The asher shall be supplied with a controlled oxygen flow, and shall be modified, if necessary, to provide a valve to control the speed of air admission so that rapid air admission does not disturb the ashed particulate. It is recommended that filters be fitted to the oxygen supply and the air admission line.

7.3.5. Filtration Apparatus

After re-dispersal of the ashed particulate in water, the particulate suspension is filtered through a membrane filter of 25 mm diameter. A glass frit support is required in order to obtain a uniform deposit on the filter. The reservoir must be easily cleaned in order to prevent sample cross-contamination. A 25 mm analytical filter holder (Millipore Corporation, Cat. No. XX10 025 00) or equivalent has been found to be suitable.

7.3.6. Filtration Manifold

When a number of samples are to be filtered, several

filtration units can be operated simultaneously from a single vacuum source by using a multiple port filtration manifold (Millipore Corporation, Cat. No. XX26 047 35 or equivalent has been found to be suitable). The manifold should include valves to permit each port to be opened or closed independently.

7.3.7. Vacuum Pump

A pump is required to provide a vacuum of approximately 20 kPa for the filtration of water samples. A water jet pump (Edwards High Vacuum Inc., Grand Island, NY 14072, Cat. No. 01-C046-01-000-female connection or 01-C039-01-000-male connection or equivalent) has been found to provide sufficient vacuum for a 3-port filtration mani-fold and also incorporates a non-return valve to prevent back-streaming.

7.3.8. Vacuum Coating Unit

A vacuum coating unit capable of producing a vacuum better than 0.013 Pa shall be used for vacuum deposition of carbon on the membrane filters. A sample holder is required which will allow a glass microscope slide to be continuously rotated during the coating procedure. A mechanism which allows the rotating slide also to be tilted through an angle of approximately 45° during the coating procedure is recommended. A liquid nitrogen cold trap above the diffusion pump may be used to minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum coating unit may also be used for deposition of the thin film of gold, or other calibration material, when it is required on TEM specimens as an internal calibration of ED patterns.

7.3.9. Sputter Coater

A sputter coater with a gold target may be used for deposition of gold on to TEM specimens as an internal calibration of ED patterns. Experience has shown that a sputter coater allows better control of the thickness of the calibration material.

7.3.10. Solvent Washer (Jaffe Washer)

The purpose of the Jaffe washer is to allow dissolution of the filter polymer while leaving an intact evaporated carbon film supporting the fibers and other particles from the filter surface. One design of a washer that has been found satisfactory for various solvents and filter media is shown in Figure 7.2. Several acceptable variations have been described in the scientific literature. When specimens are not being inserted or removed, the petri dish lid should be in place. The washer should be cleaned before it is used for each batch of specimens.

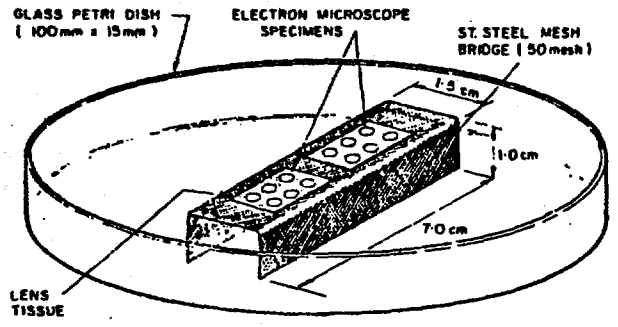

Figure 7.2: Design of a Solvent Washer (Jaffe Washer). Solvent is added until the meniscus contacts the underside of the stainless steel mesh

7.3.11. Condensation Washer

For more rapid dissolution of the filter polymer, a condensation washer, consisting of a flask, condenser and cold finger assembly, with a heating mantle and means for controlling the temperature may be used. A suitable assembly is shown in Figure 7.3, and acetone should be used as the solvent for MCE filters.

7.3.12. Slide Warmer or Oven

Either a slide warmer or an oven shall be used for heating slides during the preparation of TEM specimens. It is required to maintain a temperature of 65-70°C.

7.3.13. Ultrasonic Bath

An ultrasonic bath is required for cleaning of apparatus used for TEM specimen preparation, and for re-dispersal of ashed residues from MCE filters.

7.3.14. Carbon Grating Replica

A carbon grating replica with about 2000 parallel lines per mm shall be used to calibrate the magnification of the TEM.

7.3.15. Calibration Grids for EDXA

For calibration of the efficiency of the EDXA system, reference silicate standard material must be used. The National Institute for Standards and Technology (NIST) Standard Reference Material (SRM) 2063 is a sputtered thin film containing known concentrations of magnesium, silicon, calcium and iron. Other suitable calibration materials include riebeckite, chrysotile, halloysite, phlogopite, wollastonite and bustamite. The mineral used for calibration of the EDXA system for sodium must be prepared using a gold TEM grid.

7.3.16. Carbon Rod Sharpener

The use of necked carbon rods, or equivalent, allows the carbon to be evaporated on to the filters without creating sufficient heat to damage the filters.

7.3.17. Disposable-Tip Micropipettes

A disposable tip micropipette, capable of transferring a volume of approximately 30 μL, is required for the preparation of TEM specimen grids.

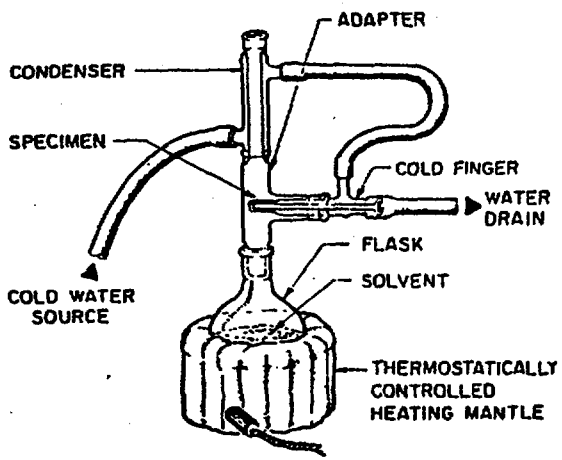

Figure 7.3: Design of a Condensation Washer

7.4. CONSUMABLE LABORATORY SUPPLIES

7.4.1. Glass Beakers

Borosilicate glass beakers of 100 mL capacity are required for ashing of filters and re-dispersal of ashed residues.

7.4.2. Membrane Filters

For filtration of the particulate suspensions, two types of filter are required:

(a) MCE filters of 0.1 μm pore size (Millipore Corporation VCWP 025 00 or equivalent) are used to collect the particles from the liquid suspension;

(b) MCE filters of 5 μm pore size (Millipore Corporation SMWP 025 00 or equivalent). One of these filters is placed between the glass frit of the filtration apparatus and the 0.1 μm pore size filter, in order to ensure a uniform particulate deposit.

7.4.3. Copper Electron Microscope Grids

Two-hundred mesh TEM grids are required. Grids which have grid openings of uniform size such that they meet the requirement of 10.4.2 should be chosen.

7.4.4. Gold Electron Microscope Grids

Two-hundred mesh gold TEM grids are required to mount TEM specimens when sodium measurements are required in the fiber identification procedure. Grids that have grid openings of uniform size such that they meet the requirement of 10.4.2 should be chosen.

7.4.5. Carbon Rod Electrodes

Spectrochemically pure carbon rods are required for use in the vacuum evaporator during carbon coating of filters.

7.4.6. Disposable Tips for Micropipette

Disposable tips for a micropipette are required for measurement of 30 μL volumes of the filter collapsing solution.

7.4.7. Routine Electron Microscopy Tools and Supplies

Fine-point tweezers, scalpel holders and blades, microscope slides, double-coated adhesive tape, lens tissue, gold wire, tungsten filaments, liquid nitrogen, and other routine supplies are required.

7.4.8. Reference Asbestos Samples

Asbestos samples are required for preparation of reference TEM specimens of the primary asbestos minerals. The NIST SRM 1866 contains well-characterized samples of chryostile, crocidolite, and amosite, although compositional data are not available for these standards. The UICC set of minerals may be used as additional reference materials.

8. REAGENTS

8.1. FRESHLY-DISTILLED WATER

A supply of freshly-distilled water must be available. The water must be produced using an all-glass still. Any water used for re-dispersal of ash, or for final rinsing of glassware must have been distilled no longer than 24 hours prior to use.

8.2. DIMETHYL FORMAMIDE, ANALYTICAL GRADE

8.3. GLACIAL ACETIC ACID, ANALYTICAL GRADE

8.4. ACETONE, ANALYTICAL GRADE

8.5. HYDROCHLORIC ACID, ANALYTICAL GRADE

WARNING - Use the reagents in accordance with the appropriate health and safety regulations.

9. AIR SAMPLE COLLECTION

Sampling procedures for this method were developed in consideration of and designed to be compatible with the analytical requirements of this method including, primarily, the need to achieve the desired analytical sensitivity.

Analytical sensitivity is a function of the volume of air sampled, the active area of the collection filter, the dilution or concentration factor introduced during specimen preparation, and the area of the TEM specimen over which asbestos structures are counted. If total airborne dust levels are high, it may be necessary to terminate sampling before the required volume has been sampled. If this happens, the analytical sensitivity required can be achieved only by counting structures over a greater area of the TEM specimen, or by use of a specimen preparation technique that incorporates some selective concentration of asbestos structures relative to other particulate species. For example, depending on the nature of the suspended particulate, a concentration factor up to about 2 may be possible for 25 mm filters, if the entire filter is used in the analysis. Larger concentration factors are possible for larger sample filters. In either case, the number of grid openings to be examined will be proportionately reduced.

The number of grid openings that must be examined in order to achieve a particular analytical sensitivity, without any selective concentration of the asbestos structures, is shown in Table 9.1. An average grid opening size of 0.0081 mm^2 is assumed in this table. However, grid opening sizes vary depending on the manufacturer of the grid. When using this table to derive the number of grid openings to be counted to achieve a defined analytical sensitivity, the number of grid openings to be counted must be adjusted to preserve the total areas represented. For example, if a volume of 5000 liters of air is collected, the table indicates that 96 grid openings must be counted to achieve an analytical sensitivity of 0.1 s/L. This corresponds to an area of 0.78 mm^2 (96 x 0.0081). If the average grid opening size on another grid is 80 μm square (resulting in an average grid opening area of 0.0064 mm^2), than a total of 122 grid openings will have to be counted. to cover the same area and achieve the same analytical sensitivity. Under no circumstances should fewer than 4 grid openings be counted.

The volume in liters required to achieve a specified analytical sensitivity is calculated from the formula:

$$V = A_f/(N \times A_s \times S \times F)$$

where:

A_f = Active area of sample collection filter in mm^2

N = Number of grid openings to be examined. N shall be rounded upwards to the next highest integer

A_s = Mean area of grid openings in mm^2

S = Analytical sensitivity in structures/liter

F = Concentration factor.

Table 9.1 - Examples of the Minimum Number of Grid Openings Required to Achieve a Particular Analytical Sensitivity

Analytical sensitivity	Volume of air sampled (liters)					
s/L	2000	5000	7000	10000	15000	20000
0.01	2377	951	680	476	317	238
0.02	1189	476	340	238	159	119
0.03	793	317	227	159	106	80
0.04	595	238	170	119	80	60
0.05	476	191	136	96	64	48
0.07	340	136	98	68	46	34
0.1	238	96	68	48	32	24
0.2	119	48	34	24	16	12
0.3	80	32	23	16	11	8
0.4	60	24	17	12	8	6
0.5	48	20	14	10	7	5
0.7	34	14	10	7	5	4
1.0	24	10	7	5	4	4
2.0	12	5	4	4	4	4
3.0	8	4	4	4	4	4
4.0	6	4	4	4	4	4
5.0	5	4	4	4	4	4
7.0	4	4	4	4	4	4
10.0	4	4	4	4	4	4

NOTES

In this table, it is assumed that the collection filter area is 385 mm^2, there is no concentration or dilution introduced during specimen preparation, and the TEM grid openings are 90 μm square.

For grid openings with dimensions other than 90μm square, the minimum number of openings to be counted should be adjusted by the ratios of the squares of the grid opening dimensions, such that the area of the TEM specimen examined remains the same.

The concentration factor F arises from the fact that, when using an indirect specimen preparation technique, the fibers on a particular area of the TEM specimen have usually been transferred from a different area of the original sample collection filter. The concentration factor may correspond to a concentration or a dilution of the original fiber density on the collection filter. It is calculated from the ratio between the areas of the original and analytical filters, the volume of water used to re-disperse the ashed particulate, and the volume of the final dispersion used to prepare the analytical filter. For the purposes of calculating the required volume of air to be sampled in order to achieve the specified analytical sensitivity when a direct transfer preparation is to be used, the concentration factor (F) is assumed to be 1.

9.1. REQUIRED SENSITIVITY

The required analytical sensitivities for characterizing environmental samples is established in section 4.3 as 0.5 s/L and 0.02 s/L for asbestos structures of all sizes and asbestos structures longer than 5 μm, respectively. For other applications of this method, different analytical sensitivities should be established prior to sample collection. Air volumes to be collected in this method are selected to provide the desired analytical sensitivity when coupled with the counting of a manageable number of grid openings.

9.2. AIR VOLUME

The sampling rate and the period of sampling shall be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method (See Section 4.2.1). It has been found that the volume cannot normally exceed 5 cubic meters in an urban or agricultural area, and 10 cubic meters in a rural area for samples collected on a 25 mm filter and prepared by a direct transfer technique.

Prior to planning a sampling program, air volumes to be collected should be adjusted based on available information characterizing typical local TSP concentrations (see Section 4.2.1). It has not proven possible to judge the loading of filters in real time during sampling so that filters may easily become overloaded to the point were analysis by the direct method becomes impossible. To minimize the potential that filters will have to be rejected for analysis due to overloading, conservative estimates of the loading rate based on typical TSP concentrations have been developed so that an "optimum" volume of air per unit area of filter may be collected. However, because such estimates tend to be conservative to avoid overloading, a fair percentage of the filters analyzed from such

a sampling effort tend to be underloaded forcing a corresponding increase in analysis costs.

NOTE

One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed.

9.3. FLOW RATE

To remain within known constraints on filter face velocities as reported in Section 2.2, an upper limit to the range of acceptable flow rates for this method is 15 L/min. In practice, available pumps that can be used for environmental sampling at remote locations operate under load at a maximum of approximately 12 L/min. Consequently, the recommended 15 m^3 of air for Phase 1 samples requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The lower volumes required for Phase 2 samples can be collected in shorter periods with a corresponding reduction in stored energy requirements. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options.

9.4. SAMPLING PROCEDURES

Before air samples are collected, unused filters shall be analyzed as described in Section 10.6 to determine the mean background asbestos structure count for the analytical procedure.

Air samples shall be collected using cassettes as described in 7.1.1. The cassettes used for sampling shall be new and unused. Sampling shall be conducted with the cassette open-face. During sampling, the filter cassette shall be supported on a stand so that it is isolated from the vibrations of the pump. The cassette shall be held facing vertically downwards at a height of approximately 1.5-2.0 m above ground/floor level and connected to the pump with a flexible tube. In many cases it will likely be sufficient to collect samples with a short cassette. If conditions dictate the need for additional protection, however, extension cowls may be affixed to the front of the cassette. Such cowls must be

constructed of electrically conducting material to minimize electrostatic effects.

The sampling flow-rate should be measured at the cassette at the beginning and at the end of the sampling period. Sampling equipment shall be selected such that either the sampling flow rate can be maintained to within ± 10%, using a flow controller or critical orifice, or flow rates shall be measured at least every 4 hours. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. After sampling, a cap shall be placed over the open end of the cassette, and the cassette packed in a clean plastic bag for return to the laboratory. Field blank filters shall also be included, as described in Section 10.6, and processed through the remaining analytical procedures along with the samples.

Should intermittent sampling be required, sampling cassettes must be covered between active periods of sampling. To cover the sampler: turn the cassette to face upward, attach a rotameter to measure the flow rate, turn off the sampling pump, and place the cap. To resume sampling: remove the cap, turn on the sampling pump, attach a rotameter to measure the flow rate, and invert the cassette to face downward.

10. PROCEDURE FOR ANALYSIS

10.1. INTRODUCTION

Filters representing Phase 1 samples will be prepared by the indirect method. Filters representing Phase 2 samples will be split. Half of the Phase 2 filters will be prepared by the indirect method and a quarter sector of the filter will be prepared by the direct method.

10.1.1. Indirect TEM Specimen Preparation Method

The indirect method requires collection of particulate from a large volume of air on a 0.45 μm pore size MCE filter. In this procedure, half of the filter is ashed by treatment in a low temperature plasma asher, and the ashed residue is re-dispersed in distilled water. Immediately before filtration, the suspension of particles is acidified using hydrochloric acid to remove any acid-soluble species. The suspension is filtered through a 0.1 μm pore size MCE filter. The filter is collapsed using a mixture of dimethyl formamide, acetic acid and water. A sector of the collapsed filter is carbon coated, and TEM specimen grids are prepared from the carbon coated filter by the Jaffe washer technique, using dimethyl formamide as the solvent.

Use of the 0.1 μm pore size membrane filter eliminates the requirement for the plasma etching step incorporated into both NIOSH Method 7402 and the method of Burdett and Rood. In previous work by Chatfield, Dillon and Stott, no statistically-significant fiber losses could be detected when TEM grids were prepared from 0.1 μm pore size MCE filters without including the etching step.

Indirect preparation of TEM specimens for determination of asbestos is particularly sensitive to problems of contamination by extraneous asbestos. This method requires that very low detection limits be achieved so that control of extraneous asbestos contamination becomes even more important. The ability to meet the blank sample criteria is critically dependent on the cleanliness of equipment and supplies. All supplies such as microscope slides and glassware should be considered as potential sources of asbestos contamination. All solvents used should be filtered through a 0.2 μm pore size filter or they should be distilled in a glass still. It is necessary to wash all glassware before it is used and the final washing of glassware should be performed using freshly-distilled water. Any tools or glassware that come into contact with the air sampling filters or TEM specimen preparations should be washed both before use and between handling of individual samples. Where possible, disposable supplies should be used.

10.1.2. Direct TEM Specimen Preparation Method

The direct TEM specimen preparation method requires that the air volume sampled must not result in a filter that is over-loaded by particulate. Particulate is collected on a 0.45 μm pore size MCE filter. A quarter sector of the MCE filter is collapsed using a mixture of dimethyl formamide, acetic acid and water. The collapsed filter is etched for a short time in a low temperature plasma asher. The collapsed and etched filter is carbon coated, and TEM specimen grids are prepared from the carbon coated filter by the Jaffe washer technique, using dimethyl formamide as the solvent.

10.2. PREPARATION OF TEM SPECIMEN GRIDS BY THE INDIRECT METHOD

10.2.1. Cleaning of Sample Cassettes

Asbestos can adhere to the exterior surfaces of air sampling cassettes and this asbestos can be inadvertently transferred to the sample during handling. To prevent this possibility of contamination, and after ensuring that the cassette is tightly sealed, wipe the exterior surfaces of each sampling cassette with a clean, wet paper towel before it is taken into the clean facility or laminar flow hood.

10.2.2. Ashing of MCE Filter

Open the filter cassette and remove the MCE filter. Place the filter on a clean microscope slide and cut it in half, using a curved scalpel blade. Return one half of the filter to the cassette. Place the other half filter in the bottom of a 100 mL glass beaker and cover the top of the beaker with a piece of aluminum foil. Make approximately 8 1-2 mm perforations in the aluminum foil. Completely ash the half filter in the low temperature plasma asher, using operating conditions as defined in Appendix A.

10.2.3. Re-dispersal of Ashed Residues

After the filter has been completely ashed, remove the beaker from the asher, add 40 mL of distilled water, and place the beaker in the ultrasonic bath for a period of 15 minutes. Using a disposable plastic pipette, add 0.2 mL of concentrated hydrochloric acid and stir the suspension. After addition of the acid, place the beaker in the ultrasonic bath for a period of 5 minutes. Filtration of the suspension must be performed immediately after the ultrasonic treatment. Power in the ultrasonic bath should be maintained below 0.1 watt/mL.

10.2.4. Filtration of the Aqueous Suspension

The separation of suspended particulate by filtration of the aqueous suspension through a membrane filter is a critical step in the analytical procedure. The objective is to produce an analytical filter on which the suspended solids from the sample are distributed uniformly, and at an acceptable loading for the microscopical examination.

The volume to be filtered generally depends on either the total suspended solids content or the asbestos structure concentration of the sample, and the intention is to prepare a filter that has a loading not exceeding about 50 asbestos structures per grid opening and with a particulate coverage not exceeding 10% of the filter area. Some judgment is required to achieve the optimum loading and, if the asbestos concentration is very low, it will be found that the suspended solids concentration will limit the volume that can be filtered. The maximum particulate loading on the filter that can be tolerated is about 10 $\mu g/cm^2$.

In practice, the best procedure for optimizing the loading on the final filter is to prepare several filters using different volumes of the suspension. Any fraction of the 40 mL suspension (prepared as per Section 10.2.3) may be selected as a unit for filtration. For example, aliquots containing 1 mL, 4 mL, 8 mL, and 20 mL fractions from the original suspension may be used to prepare a series of increasingly loaded filters. However, if the concentration of suspended solids dictates that a very small volume of the suspension be filtered, small volume aliquots need to be further diluted prior to filtering. Do not attempt to filter a volume of less than 10 mL.

If aliquots smaller than 10 mL from the original suspension are to be filtered, it is difficult to ensure that a uniform deposit of particulate is obtained on the filter unless the aliquot is first diluted. Samples of high solids content, or of high asbestos concentration, may require filtration of volumes less than 10 mL. Such aliquots shall be diluted with freshly-distilled water so that the filtered volumes exceed the minimum of 10 mL. These dilutions shall be made by transferring a known volume of the sample (such as an aliquot from the original suspension) to a disposable plastic beaker and making the volume up to a known volume with freshly-distilled water. The mixture shall be stirred vigorously before sub-samples are taken for filtration.

The following instructions for filtration must be followed precisely:

(a) Assemble the filtration base and turn on the vacuum. The upper surface of the filtration base (both the glass

frit and the ground mating surface) must be dry before the membrane filters are installed. Place a 5 μm pore size MCE filter on the glass frit. If the filter appears to become wet by capillary action on residual water in the glass frit it must be discarded and replaced by another filter. Place a 0.1 μm pore size MCE filter, smooth side facing up, on top of the 5 μm filter. The mating surface of the reservoir component of the filtration apparatus should be dried by shaking off any surplus water and draining on paper towel or tissue. Position the reservoir on the filters and firmly clamp it, taking care not to disturb the filters. The vacuum should not be released until the filtration has been completed.

It is necessary to comment on the use of filtration equipment that is still wet after washing, since improper procedures at this point can seriously compromise the results. If the glass frit is wet when the first filter is applied to it, capillary action will result in some areas of the filter structure being filled by water. When the second, smaller pore size filter is added and the vacuum is applied, the differential pressure across the 5 μm pore size filter will be insufficient to overcome the surface tension of the water in the filled areas. Thus no filtration will take place through the corresponding areas of the 0.1 μm pore size filter, and a grossly non-uniform deposit of particulate will be obtained.

(b) Add the required volume of sample to the filtration funnel. Disposable plastic beakers and pipettes provide a means of measuring the sample volumes without introducing problems of sample cross-contamination. The reservoir may not be sufficiently large to accommodate the total volume of liquid to be filtered. In this case, more of the sample may be added during the filtration, but this should be done carefully and only when the reservoir is more than half full. In this way the addition of liquid will not disturb or affect the uniformity of particulate already deposited on the filter. Do not rinse the sides of the reservoir, and avoid other manipulations which may disturb the particulate deposit on the filter.

(c) Disassemble the filtration apparatus, and transfer the 0.1 μm pore size filter to a labelled, clean plastic petri dish. Place the cover loosely over the top of the dish to limit any deposition of material on the filter, and allow the filter to dry. Discard the 5 μm pore size filter.

10.2.5. Selection of Area of Filter for Preparation

Using clean tweezers, and a clean, curved scalpel blade, cut out a 90 degree sector of the filter while it is still in the plastic petri dish.

10.2.6. Preparation of Solution for Collapsing MCE Filters

Mix 35 mL of dimethyl formamide, 15 mL of glacial acetic acid with 50 mL of freshly-distilled water. Store this mixture in a clean bottle.

10.2.7. Filter Collapsing Procedure

Using a micropipette with a disposable tip, place approximately 30 μL of the collapsing solution on a cleaned microscope slide, and using the end of the pipette tip spread the liquid over the area to be occupied by the filter sector. Place the filter sector, active surface upwards, on top of the solution, lowering the edge of the filter at an angle of about 20 degrees so that air bubbles are not created. Remove any solution not absorbed by the filter by allowing a paper tissue to contact the liquid at the edge of the filter. More than one filter sector may be placed on one slide, and a laboratory blank filter sector shall also be prepared on each slide. Place the slide either on a thermostatically-controlled slide warmer at a temperature of approximately 65-70°C, or in an oven at the same temperature, for about 10 minutes. The filter collapses slowly to about 15% of its original thickness. The procedure leaves a thin, transparent plastic film adhering to the microscope slide, with particles and fibers embedded in the upper surface.

10.2.8. Carbon Coating of Filter Sectors

Place the glass slide holding the filter sectors on the rotation-tilting device, approximately 10-12 cm from the evaporation source, and evacuate the evaporator chamber to a vacuum better than 0.013 Pa. The evaporation of carbon shall be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation of carbon is too rapid, the surface of the filter may be damaged. The thickness of carbon required is dependent on the size of particles on the filter, and approximately 30-50 nm has been found to be satisfactory. If the carbon film is too thin, large particles will break out of the film during the later stages of preparation, and there will be few complete and undamaged grid openings on the specimen. Too thick a carbon film will lead to a TEM image which is lacking in contrast, and the ability to obtain SAED patterns will be compromised. The carbon film thickness should be the minimum possible, while retaining most of the grid openings of the TEM specimen intact.

10.2.9. Preparation of the Jaffe Washer

Place several pieces of lens tissue on the stainless steel bridge, and fill the washer with dimethyl formamide to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue. If it is intended to complete the washing of the specimens in the condensation washer, the pieces of lens tissue should be of such a size that they will fit on to the stainless steel mesh of the condensation washer cold finger. Three TEM specimen grids shall be prepared from each of the coated analytical filters, and for each of the analytical filters, three new specimen grids should be placed on to one of the pieces of lens tissue.

10.2.10. Placing of Specimens Into the Jaffe Washer

Using a curved scalpel blade, cut along the radius of the coated filter sector, approximately 1 mm from the edge. Discard the thin strip of coated filter, which usually exhibits signs of edge effects introduced during the collapsing procedure. Starting from the freshly-cut edge, cut three approximately 3 mm square pieces of the coated filter. Select three squares to represent the center and the outer periphery of the active surface of the filter. Place each square of filter, carbon side up, on one of the TEM specimen grids already on the lens tissue in the Jaffe washer. Cover the Jaffe washer with the lid, and allow the washer to stand. Specimens are normally cleared after approximately 4 hours.

10.2.11. Rapid Preparation of TEM Specimens From MCE Filters

An alternative washing procedure may be used to prepare TEM specimens from MCE filters more rapidly than can be achieved by the Jaffe washing procedure. After the specimens have been washed in a Jaffe washer for approximately 1 hour, transfer the piece of lens tissue supporting the specimens to the cold finger of a condensation washer operating with acetone as the solvent. Operate the condensation washer for approximately 30 minutes. This treatment removes all remaining filter polymer.

10.3. PREPARATION OF TEM SPECIMEN GRIDS BY THE DIRECT METHOD

10.3.1. Cleaning of Sample Cassettes

After ensuring that the cassette is tightly sealed, wipe the exterior surfaces of each sampling cassette with a clean, wet paper towel as described in 10.2.1 before it is taken into the clean facility or laminar flow hood.

10.3.2. Selection of Area of Filter for Preparation

Using clean tweezers, remove the MCE filter from the filter cassette, and cut a sector as described in 10.2.5.

10.3.3. Filter Collapsing Procedure

Collapse the filter sector as described in 10.2.7.

10.3.4. Plasma Etching of the Filter Surface

The conditions required in a particular plasma asher shall first be established using the procedure defined in Appendix A. Place the microscope slide holding the collapsed filter sectors in the plasma asher, and etch for the time and under the conditions determined. Care should be taken to ensure that the correct conditions are used. After etching, admit air slowly to the chamber and remove the microscope slide.

10.3.5. Carbon Coating of Filters

Carbon coat the microscope slide holding the collapsed filter portions as described in 10.2.8.

10.3.6. Preparation of the Jaffe Washer

Prepare the Jaffe washer as described in 10.2.9.

10.3.7. Placing of Specimens into the Jaffe Washer

Place specimens in the Jaffe washer as described in 10.2.10. Specimens are normally cleared after approximately 4 hours.

10.3.8. Rapid Preparation of TEM Specimens from MCE Filters

The alternative rapid washing procedure, described in 10.2.11, may be used to prepare TEM specimens from MCE filters more rapidly than can be achieved by the Jaffe washing procedure.

10.4. CRITERIA FOR ACCEPTABLE TEM SPECIMEN GRIDS

Examine the TEM specimen grid in the TEM at a magnification sufficiently low (300-1000) so that complete grid openings can be inspected. Reject the grid if:

(a) the TEM specimen has not been cleared of filter medium by the filter dissolution step. If the TEM specimen exhibits areas of undissolved filter medium and, if at least two of the three specimen grids are not cleared, either additional solvent washing shall be carried out or new specimens shall be prepared from the filter;

(b) the sample is over-loaded with particulate. If the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen will be designated as over-loaded. This filter cannot be analyzed satisfactorily because the grid is too heavily loaded with debris to allow separate examination of individual particles by ED and EDXA and obscuration of structures by other particulate would lead to under-estimation of the structure count;

(c) the particulate deposits on the specimen are not uniformly distributed from one grid opening to the next. If the particulate deposits on the specimen are obviously not uniform from one grid opening to the next, the specimen will be designated as non-uniform. This condition is caused either by use of an unsatisfactory procedure for the water filtration or it is a consequence of the fundamental nature of the airborne particulate. Such a filter cannot be analyzed satisfactorily;

(d) the TEM grid is too heavily loaded with asbestos structures to make an accurate count. Accurate counts cannot be made if the grid has more than approximately 7000 asbestos structures/mm^2; or,

(e) less than approximately 75% of the grid openings have unbroken carbon film over the whole grid opening. Since the breakage of carbon film is usually more frequent in areas of heavy deposit, counting of the intact openings can lead to an underestimate of the structure count. An additional carbon coating may be applied to the carbon coated filter and new specimen grids prepared. The larger particles can often be supported by using a thicker carbon film. If this action does not produce acceptable specimen grids, this filter cannot be analyzed and a filter prepared with a lighter loading of particulate should be selected.

If any one or more of the conditions described in (b), (c), (d) or (e) exists, the specimen grids cannot be analyzed.

10.5. PROCEDURE FOR STRUCTURE COUNTING BY TEM

10.5.1. Introduction

The TEM examination consists of a count of asbestos structures, which are present on a specified number of grid openings. Asbestos structures are classified into groups on the basis of morphological observations, ED patterns and EDXA spectra. The total number of asbestos structures to be counted depends on the statistical precision desired. In the absence of asbestos structures, the area of the TEM specimen grids which must be examined depends on the analytical sensitivity required. The

precision of the asbestos structure count depends not only on the total number of asbestos structures counted, but also on their uniformity from one grid opening to the next. Additional asbestos structure counting will be necessary if greater precision is required.

In order that the estimate of the asbestos structure density on the sampling filter shall not be based on the asbestos structure deposits found within the small area represented by one specimen grid, grid openings shall be examined on at least two of the three specimen grids prepared. The results shall then be combined in the calculation of the asbestos structure density. Structure counts shall be made at a magnification of approximately 20000, and will be terminated at the number of asbestos structures as defined below, except that the count shall be continued until a minimum of 4 grid openings have been examined. Otherwise, the asbestos structure count shall continue to that number of grid openings at which the specified analytical sensitivity has been achieved.

10.5.2. Measurement of Mean Grid Opening Area

The grid opening area shall be measured for the type of TEM specimen grids in use. This may be performed either on an optical microscope at a calibrated magnification of about 400, or at a calibrated magnification in the TEM. A mean value for the grid opening dimensions may be used if this value can be shown to be sufficiently precise. If a mean value is used, the standard deviation of the mean of 10 openings selected randomly from each of 10 grids shall be less than 5%. Alternately, the dimensions of one grid opening on each of the grids examined shall be measured and the mean of these used in the calculations for the particular analysis.

Values for the number of grid openings to be counted to achieve a desired level of sensitivity (Table 9.1) will be adjusted based on the average grid opening size derived in this section. The procedure for performing the conversion is presented in a note at the bottom of Table 9.1.

10.5.3. TEM Alignment and Calibration Procedures

Before structure counting is performed, align the TEM according to instrumental specifications. Calibrate the TEM and EDXA system according to the procedures described in Appendix B.

10.5.4. Determination of Stopping Point

Before structure counting is commenced, the area of specimen to be examined in order to achieve the required analytical

sensitivity shall be calculated. The maximum number of grid openings to be examined shall be calculated from the formula:

$$N = A_f/(A_g \times V \times S \times F)$$

where:

N = Number of grid openings to be examined. N shall be rounded upwards to the next highest integer

A_f = Area of collection filter in mm^2
A_g = Area of TEM specimen grid opening in mm^2

V = Volume of air sampled in liters

S = Required analytical sensitivity in asbestos structures/liter

F = Concentration factor.

The concentration factor F arises from the fact that the asbestos structures on a particular area of the TEM specimen have usually been transferred from a different area of the original sample collection filter. The concentration factor may correspond to a concentration or a dilution of the original asbestos structure density on the collection filter. It is calculated from the ratio between the areas of the original and analytical filters, the volume of water used to re-disperse the ash, and the volume of the final dispersion used to prepare the analytical filter. F is calculated from the following formula:

$$F = A_a \times V_f/(A_f \times V_r)$$

where:

A_a = Area of filter ashed
A_f = Area of analytical filter
V_r = Volume of water used to re-disperse ashed particulate
V_f = Volume of dispersion filtered through analytical filter

10.5.5. General Procedure for Structure Counting and Size Analysis

Use at least two specimen grids prepared from the filter in the structure count. Select at random several grid openings from each grid, and combine the data in the calculation of the results.

Use a form similar to that shown in Figure 10.1 to record the structure counting data. Insert the first specimen grid into the TEM. Select a typical grid opening and set the screen magnification to the calibrated value (approximately 20,000). Adjust the sample

TEM ASBESTOS COUNT

Page ___ of ___

Preparation Date: ___/___/___ By: ___
Analysis Date: ___/___/___ By: ___
y m d
Report Number: _____
Filename: _____
Sample Description: _____
_____
_____
Sample Number: _____

Sample Filter Area: _____ sq.mm.
Area of Filter Taken: _____ sq.mm.
Analytical Filter Area: _____ sq.mm.
Redisperse Volume: _____ ml
Liquid Volume Filtered: _____ ml
Magnification: _____
Air Volume: _____ liters
Grid Opening Dimension: _____ um

Grid Opening	Structure Number	Class	Structure Type	Length (mm)	Width (mm)	Comments

Figure 10.1: Counting Form

height until the features in the center of the TEM viewing screen are at the eucentric point. Set the goniometer tilt angle to zero.

In column 1 of the structure counting form, record the sequential number of the grid opening. Position the specimen so that the grid opening is positioned with one corner visible on the screen. Move the image by adjustment of only one translation control, carefully examining the sample for asbestos structures, until the opposite side of the grid opening is encountered. Move the image by a pre-determined distance less than one screen diameter, using the other translation control, and scan the image in the reverse direction. Continue the procedure in this manner until the entire grid opening has been inspected in a pattern similar to that shown in Figure 10.2.

When a fibrous structure is detected, assign a sequential number in column 2, perform the identification procedures required as detailed in Appendix D, and enter the appropriate compositional classification on the fiber counting form in column 3. Assign a morphological classification to the structure according to the procedures in Appendix C, and record this in column 4. Measure on the TEM viewing screen the length and width of the image of each of the components of the fibrous structure in mm and record these measurements in columns 5 and 6 of the structure counting form.

If fibrous structures are present that are of obvious biological origin or that are determined to be non-asbestos, record data for a minimum of the first 10 such structures; further recording of data from non-asbestos structures is optional. After a fibrous structure has been examined and measured, re-locate the original field of view accurately before continuing scanning of the specimen. Failure to do this may cause asbestos structures to be overlooked or counted twice. The point at which the examination is to be terminated depends on the specimen, and is defined by either (a), (b) or (c) below:

(a) for Phase 1 samples, which have been prepared by the indirect TEM specimen preparation method <u>only</u>, complete the examination at the end of the grid opening on which the 50th asbestos structure is counted;

(b) for Phase 2 samples, which have been prepared both by the direct and indirect TEM specimen preparation methods, complete the examination at the end of the grid opening on which the 100th asbestos structure is counted; or

(c) for either of the sample types, until the number of grid openings required to achieve the specified analytical sensitivity for asbestos structures of all lengths, calculated according to 10.5.4, have been inspected, whichever occurs

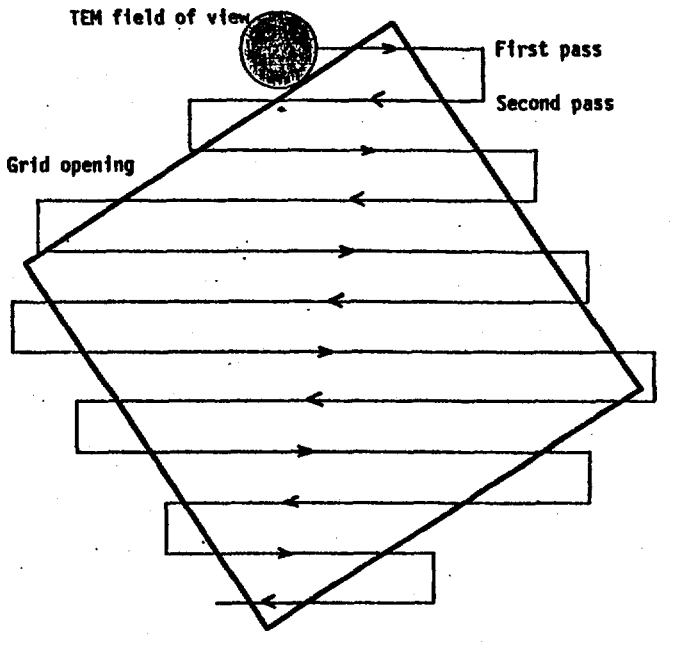

Figure 10.2: Scanning Procedure for TEM Specimen Examination

first. The data shall be drawn approximately equally from the two grids. Regardless of the value calculated according to 10.5.4, asbestos structures shall be counted on a minimum of 4 grid openings. Table 10.1 presents a summary of the counting requirements for Phase 1 and Phase 2 samples.

(d) Only those structures that are identified as, or are suspected to be, either chrysotile or one of the amphibole minerals, will be included in either the original or the extended structure count. Other materials, such as gypsum, cellulose fibers, and filter artifacts such as undissolved filter strands, will not be included in the structure count. This restriction is intended to ensure that the best statistical validity is obtained for the materials of interest.

10.5.6. Measurement of Concentration for Asbestos Structures Longer than 5 μm

To increase the statistical validity of the measurement, this method requires that an extended count be made of those asbestos structures with aspect ratios equal to or greater than 5:1 and longer than 5 μm. This separate structure count takes account only of the asbestos structures longer than 5 μm, and scanning of the TEM specimen shall be performed using a magnification of approximately 10,000.

Measurement of the asbestos structure lengths shall be performed at a magnification of 10,000, but asbestos structure diameters shall be measured at a magnification of a minimum of 20,000. For Phase 1 samples, which have been prepared by indirect transfer only, the count is continued until either 50 asbestos structures have been recorded or an area of specimen has been examined that achieves the specified analytical sensitivity for asbestos structures longer than 5 μm. For Phase 2 samples, which have been prepared both by indirect and direct transfer, the count is continued until either 100 asbestos structures have been counted or an area of specimen has been examined that achieves the specified analytical sensitivity for asbestos structures longer than 5 μm. Table 10.1 presents a summary of the counting requirements for Phase 1 and Phase 2 samples.

Only those structures that are identified as, or are suspected to be, either chrysotile or one of the amphibole minerals, will be reported in either the original or the extended structure count. Other materials, such as gypsum, cellulose fibers, and filter artifacts such as undissolved filter strands, will not be included in the structure count. This restriction is intended to ensure that the best statistical validity is obtained for the materials of interest.

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TABLE 10.1: SPECIFICATIONS FOR PHASE 1 AND 2 SAMPLING AND ANALYSIS

Sampling Definitions		Phase 1 Samples: High Magnification Scan	Low Magnification Scan	Phase 2 Samples: High Magnification Scan	Low Magnification Scan
	Structure sizes to be counted	total	length > 5 μm	total	length > 5 μm
	Number of structures to be counted	50	50	100	100
Ambient:	Volume of air to be collected is 15 m^3				
	Maximum area to be scanned	0.05 mm^2	1.3 mm^2	0.11 mm^2	25 mm^2
	Maximum number of grid openings to be counted	7	160	15	307
	Minimum number of grid openings to be counted	4	4	4	4
Burst:	Volume of air to be collected is 10 m				
	Maximum area to be scanned	0.08 mm^2	2.0 mm^2	0.16 mm^2	3.7 mm^2
	Maximum number of grid openings to be counted	10	240	20	448
	Minimum number of grid openings to be counted	4	4	4	4
Urban and Agricultural:	Volume of air to be collected is 5 m^3				
	Maximum area to be scanned	0.15 mm^2	3.9 mm^2	0.32 mm^2	7.5 mm^2
	Maximum number of grid openings to be counted	20	480	40	900
	Minimum number of grid openings to be counted	4	4	4	4
Activity Specific:	Volume of air to be determined in the field				
	Maximum area to be scanned	TBD	TBD	TBD	TBD
	Maximum number of grid openings to be counted	TBD	TBD	TBD	TBD
	Minimum number of grid openings to be counted	4	4	4	4

TABLE 10.1
(continued)

NOTES:

Phase 1 samples will be prepared by the indirect technique. Filters from Phase 2 samples will be split. Half will be prepared by the indirect technique and half will be prepared by the direct technique. Note that the required sensitivity for the Phase 2 samples is higher than that necessary for the Phase 1 samples. Consequently, it is assumed that 20 structures need to be counted for phase 2 samples at a minimum.

The information presented in this table is derived from the equation presented in the Section 10.5.4 assuming that $A_f = 385\ mm^2$, $A_g = 0.0081\ mm^2$, $F = 1$, and that 10 structures will have to be counted for statistical significance. Based on a review of reported background concentrations and typical concentrations encountered in previous studies, it is also assumed that the critical concentration for total structures is 5.0 structures/liter and the critical concentration for structures longer than 5 micrometers is 0.2 structures/liter.

During any site investigation, it will likely be necessary to characterize both ambient air concentrations and concentrations of asbestos associated with specific human activities. Although the method is designed for ambient conditions, it will be possible to adapt it to activity specific sampling by accounting with some adjustment for expected differences in conditions between the two types of samples. The principle difference likely is that a much higher concentration of non-asbestos particulates will likely be contained in air sampled in association with specific, dust-generating activities. As a consequence, the estimated values for maximum tolerable air volumes presented in section 4.2.1 of the text can not be applied to activity specific sampling and some measure of TSP will have to be provided to derive alternate sampling volumes. Thus, "TBD" means: to be determined.

It must be emphasized that the volume of air recommended for collection in each of the environmental settings listed represents a conservative estimate (based on published data) of the maximum that can be collected and still allow analysis without overloading the filter. If additional information regarding the local average concentration of ambient TSP, air volumes to be collected in any of these settings should be optimized accordingly.

To achieve the desired analytical sensitivity, the numbers of grid openings required for counting (as presented in this Table) may either be distributed over a set of specimen grids prepared from a single sample or a larger set of specimen grids prepared from several samples collected in a manner assuring that the samples are appropriately homogeneous (see Section 4.3 of the Technical Background Document, Part 2 of this report).

10.6. BLANK AND QUALITY CONTROL DETERMINATIONS

Before air samples are collected, a minimum of 2 unused 0.45 μm pore size MCE filters from each lot of 100 filters shall be sectioned and analyzed both by the indirect and the direct transfer procedure to determine the mean background asbestos structure concentration. If the mean concentration for all types of asbestos structures, expressed as the concentration per unit area of the sample collection filter, is found to be more than 10 structures/mm^2, the reasons for the high blank values shall be determined and the situation corrected before air samples are collected.

To ensure that contamination by extraneous asbestos structures during specimen preparation is insignificant compared with the results reported on samples, it is necessary to establish a continuous program of blank measurements. The number of field blanks incorporated into the program shall be at least 10% of the total number of samples, and all of these shall be analyzed. In addition, one unused 0.1 μm pore size filter shall be included with every group of samples prepared on one microscope slide.

It is further recommended that laboratory blanks be collected intermittently at all critical phases of the laboratory program. For example, in addition to the filter blanks and field blanks, suspension blanks should be prepared by subjecting a clean ashing tube to the asher, rinsing the tube and completing the standard indirect preparation on the "clean" rinse. Other blanks may be constructed to test for the potential introduction of contamination at other phases of the process. Such blanks need not be analyzed but may be stored to provide a chain for trouble-shooting should a problem with contamination occur.

Initially, and also included at random points in the sampling program, it is necessary to ensure that samples of known asbestos structure concentrations can be analyzed satisfactorily by this procedure. There are many opportunities in the procedure for a low recovery to be obtained, and the recovery must be measured frequently, particularly if more than one laboratory performs the analysis. Reference filters of known concentration will be incorporated, and shall constitute a minimum of 5% of the total analyses. These reference filters shall not be identified to the analytical laboratory prior to the analysis of any group of samples. The results of these reference analyses shall not differ at the 5% significance level from the mean value obtained by a selected group of experienced laboratories.

Since there is a subjective component in the structure counting procedure, it is necessary that re-counts of some specimens be made by different microscopists, in order to minimize the subjective effects. Such recounts provide a means of maintaining comparability between counts made by different microscopists. Variability between and within microscopists and between laboratories shall be characterized. These quality assurance

measurements shall constitute a minimum of approximately 10% of the analyses. Repeat results should not differ at the 5% significance level.

10.7. CALCULATION OF RESULTS

Calculate the results using the procedures detailed in Appendix E. Prior to the TEM examination of the specimens, the level of analysis was specified. Before the results are calculated, the compositional and morphological classifications to be included in the result shall be specified. The chi-squared uniformity test shall be conducted using the number of isolated asbestos structures (individual entities) found on each grid opening, prior to the application of the cluster and matrix counting criteria. The concentration result shall be calculated using the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

11. PERFORMANCE CHARACTERISTICS

11.1. INTERFERENCES AND LIMITATIONS OF STRUCTURE IDENTIFICATION

Unequivocal identification of every chrysotile structure is not possible, due to both instrumental limitations and the nature of some of the component fibers. The requirement for a calibrated ED pattern eliminates the possibility of an incorrect identification of the structure selected. However, there is a possibility of misidentification of other chrysotile structures for which both a morphology and ED pattern are reported on the basis of visual inspection only. The only significant possibilities of misidentification occur with halloysite, vermiculite scrolls or palygorskite, all of which can be discriminated from chrysotile by the use of EDXA and by observation of the 0.73 nm (002) reflection of chrysotile in the ED pattern.

As in the case of chrysotile structures, complete identification of every amphibole structure is not possible due to instrumental limitations and the nature of some of the component fibers. Moreover, complete identification of every amphibole structure is not practical due to the limitations of both time and cost. Particles of a number of other minerals having compositions similar to those of some amphiboles could be erroneously classified as amphibole when the classification criteria do not include zone-axis ED techniques. However, the requirement for quantitative EDXA measurements on all structures as support for the random orientation ED technique makes misidentification very unlikely, particularly when other similar structures in the same sample have been identified as amphibole by zone-axis methods. The possibility of misidentification is further reduced with increasing aspect ratio, since many of the minerals with which amphibole may be confused do not display its prominent cleavage parallel to the c-axis.

11.2. PRECISION AND ACCURACY

11.2.1. Precision

The analytical precision that can be obtained is dependent upon the number of structures counted, and also on the uniformity of the particulate deposit on the original filter. Assuming that the structures are randomly deposited on the filter, if 100 structures are counted and the loading is at least 3.5 fibers/grid opening, computer modeling of the counting procedure shows that a coefficient of variation of about 10% can be expected. As the number of structures counted decreases, the precision will also decrease approximately as $\sqrt{N}$, where N is the number of structures counted. In practice, particulate deposits obtained by filtration are rarely ideally distributed, and it is found that the precision is correspondingly reduced from that predicted by the Poisson distribution. This degradation is a consequence of:

(a) non-uniformity of the filtered particulate deposit;

(b) distortion of the structure distribution by application of the structure counting criteria;

(c) variation between microscopists in their interpretation of the fibrous structures; and,

(d) variation between microscopists and instruments in their ability to detect and identify structures.

The 95% confidence interval about the mean for a single structure concentration measurement using this analytical method should be about ±25% when about 100 structures are counted.

11.2.2. Accuracy

There is no independent method available to determine accuracy.

11.3. ANALYTICAL SENSITIVITY

Analytical sensitivity is the concentration corresponding to observation of one asbestos structure in an analysis. The analytical sensitivity of the method can be varied by choice of the area of the collection filter, the volume of air sampled, the dilution or concentration factor used in the specimen preparation and the area of the specimen examined in the TEM. The analytical sensitivity shall be quoted for each sample analysis.

NOTE

In practice, the lowest achievable value of analytical sensitivity is frequently determined by the total suspended particulate concentration in the air sample, since each particle on the TEM specimen must be separated from adjacent ones by a sufficient distance that the particle can be identified without interference. Particulate loadings on filters greater than about 25 $\mu g/cm^2$ usually preclude preparation of TEM specimens.

If the analysis is to be performed with an acceptable expenditure of time, the area of the specimen examined in the TEM must also be limited. The analytical sensitivity that can be achieved for any particular sample depends on the air volume, the area of TEM specimen examined, and the dilution or concentration factor used in the analysis, which in turn is controlled by the amount of particulate that is not removed by the selective concentration procedures.

11.4. LIMIT OF DETECTION

Should asbestos be observed during analysis, it is generally important to distinguish whether such asbestos originated in the sampled medium or if it was introduced as contamination during analysis. Asbestos that can be attributed to the sampled medium is generally considered to have been "detected". Thus, a detection limit is defined as the smallest measurement that is unlikely (probability less than a specified value) to be due entirely to contamination from sources other than the air being sampled. Detection limits are generally quantified by considering the magnitude and frequency of occurrence of the analytical background associated with a particular method. However, detection limits for asbestos methods are difficult to quantify because the distribution of analytical background associated with asbestos analysis tends to be poorly behaved.

To avoid the problems typically associated with defining a detection limit for asbestos analysis, an alternate procedure for distinguishing sampled asbestos from contamination due to analytical background has been incorporated into this method[3]. A statistical test is performed to compare structure concentrations observed on sample filters with structure concentrations observed on appropriate blanks to determine whether differences between the concentrations observed on the two sets of filters are statistically significant.

NOTE

Due to the difficulty in characterizing contributions to observed asbestos on individual samples from contamination introduced as analytical background, tests to distinguish "detected" asbestos from such contamination are better applied to groups of samples. Adjustments to account for analytical background are not recommended for individual samples.

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3 A detection limit is more appropriate for analytical methods that employ wet-chemical or spectroscopic techniques rather than structure counts (as in the case of asbestos) because the former are invariably associated with a lower limit to the amount (number of molecules) that can be detected. For asbestos, however, analysis by TEM is sufficiently sensitive to detect the smallest, single asbestos structure. Thus the issues involved in setting detection limits for TEM analyses of asbestos structures are quite different than from those involved with detecting populations of molecules such as in gas chromatograph-mass spectrometry (GC-MS) analyses, for example.

12. REPORTING REQUIREMENTS

Reporting requirements for the laboratory include the individual documentation for each sample (sample analysis report) and the cumulative report for any batch of samples from a particular study. The structure counting form to be used by the analyst was presented in Figure 10.1. Required data reduction by the site data reviewer is presented at the end of this chapter, Section 12.3. It is critical that the following documentation requirements be followed exactly to preserve the ability to interpret results without the need to re-examine samples.

12.1. SAMPLE ANALYSIS REPORT

The sample analysis report for each sample shall include at least the following information:

(a) reference to this analytical method;

(b) identification of the air sample and the batch number of the collection filter used;

(c) the dates and times of the air sample collection period;

(d) the volume of air passed through the sample collection filter, the area of filter used for the sample preparation, the volume of water used for re-dispersal, the batch number of the filters used for filtration of the aqueous suspension, and, for the set of TEM specimen grids examined, the volume of the aliquot used in the aqueous suspension filtered;

(e) a complete listing of the structure counting data. The dimensions of the TEM grid openings shall be specified, and for each asbestos structure (or structure component) the following data shall be included: grid opening number, structure number, identification category (morphological classification), structure type (compositional classification), length and width of the structure in μm, and any comments concerning the structure;

(f) a statement of the minimum acceptable identification category and the maximum identification category attempted;

(g) a statement specifying which identification and structure categories have been used to calculate the concentration values;

(h) separate concentration values for chrysotile and amphibole structures, expressed in asbestos structures/liter[4];

(i) separate concentration values for chrysotile and amphibole structure components, expressed in asbestos structures/liter[4];

(j) the 95% confidence limits for the concentration values, expressed in asbestos structures/liter;

(k) the analytical sensitivity, expressed in asbestos structures/liter;

(l) compositional data for the principal varieties of amphibole, if present;

(m) items d - l for the separate count of asbestos structures longer than 5 μm;

An example of a suitable format for the structure counting data is shown in figure 12.1.

NOTE

As improved definitions of the dimensions of asbestos structures critical to the determination of risk become available, the reporting requirements for this method may be modified accordingly. At the same time, sufficient information is preserved in these reporting requirements to allow later re-evaluation of existing analysis results without the need to re-examine the original sample.

12.2. SAMPLE BATCH REPORT

In addition to the sample analysis report for each sample, a summary page should be provided for each batch of samples representing an entire project. The summary sheet should include the following information:

(a) project title;

(b) date samples received and data results reported;

(c) a summary listing of sample results with chrysotile and amphibole reported separately including:

- the sample number;

---

[4] If asbestos is not detected during a particular analysis, the resulting mean concentration for that sample should be reported as "NF" for "not found".

- the analytical sensitivity for each size range characterized (structures of all sizes and structures longer than 5 mm)
- the total number of structures counted;
- the concentration of asbestos structures of all sizes in structures per liter with appropriate 95% confidence limits;
- the concentration of asbestos structure components in structures per liter with appropriate 95% confidence limits;
- the concentration of asbestos structures longer than 5 μm in structures per liter with appropriate 95% confidence limits;
- the concentration of asbestos structure components longer than 5 μm in structures per liter with 95% confidence limits;

A sample summary sheet is presented in figure 12.2.

12.3. DATA REVIEW REPORT

Once the Sample Batch Report is received from the laboratory, the following reporting procedure will be performed by the project data reviewer. The data reviewer shall provide a summary sheet reflecting specific steps in data reduction. The summary data sheet shall include the following information:

(a) the sample number;

(b) the type of sample (lab blank, field blank, sampling station identifier);

(c) the analytical sensitivity for each size fraction reported (structures of all sizes and structures longer than 5 μm);

(d) the number of total structures counted;

(e) the concentration of asbestos structures of all sizes in structures per liter with 95% confidence limits;

(f) the concentration of asbestos structure components in structures per liter with 95% confidence limits;

(g) the concentration of asbestos structures longer than 5 μm in structures per liter with 95% confidence limits;

(h) the concentration of asbestos structure components longer than 5 μm in structures per liter with 95% confidence limits;

(i) the ratio of free asbestos fibers to total asbestos structures;

SAMPLE ANALYSIS INFORMATION (Page 1)

Laboratory name Report number Date

SAMPLE: 456 Queen Street
San Diego
Downwind sample 1988-09-25
Collected: 8 a.m. to 4 p.m.

ANALYSIS: Name of method
Reference to method

Air volume:	12500.0 liters
Area of collection filter:	385.0 mm^2
Area of filter ashed:	96.3 mm^2
Volume of water used for re-dispersal:	40.0 mL
Volume of suspension filtered:	35.0 mL
Area of analytical filter:	199.0 mm^2
Magnification used for fiber counting:	20500 (high magnification)
	10200 (low magnification)
Mean dimension of grid openings:	90.0 μm

High Magnification Scan

Number of grid openings examined:	38
Analytical Sensitivity:	0.5 s/L
Number of chrysotile structures counted:	63 (including F, M, C and B)
Number of chrysotile structure components counted:	91
Number of amphibole structures counted:	12 (F only)
Number of amphibole structure components counted:	12

Low Magnification Scan

Number of grid openings examined:	357
Analytical sensitivity:	0.02 s/L
Number of long chrysotile structures counted:	3 (B and C)
Number of long chrysotile structure components counted:	14
Number of long amphibole structures counted:	0
Number of long amphibole structure components counted:	0

NOTES: Only structures with components longer than 5μm counted during the low magnification scan.

Figure 12.1A: Format for Reporting of Counting Data, Page 1

ASBESTOS (bulk) by PLM

9002

various MW: various CAS: 1332-21-4 RTECS: CI6475000

METHOD: 9002, Issue 2 **EVALUATION:** PARTIAL **Issue 1:** 15 May 1989 **Issue 2:** 15 August 1994

EPA Standard (Bulk): 1% **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].

SAMPLING

BULK SAMPLE:	1 to 10 grams
SHIPMENT:	seal securely to prevent escape of asbestos
SAMPLE STABILITY:	stable
BLANKS:	none required

ACCURACY

RANGE STUDIED:	<1% to 100% asbestos
BIAS:	not determined
PRECISION:	not determined
ACCURACY:	not determined

MEASUREMENT

TECHNIQUE:	MICROSCOPY, STEREO AND POLARIZED LIGHT, WITH DISPERSION STAINING
ANALYTE:	actinolite asbestos, amosite, anthophyllite asbestos, chrysotile, crocidolite, tremolite asbestos
EQUIPMENT:	microscope, polarized light; 100-400X dispersion staining objective, stereo microscope: 10-45X
RANGE:	1% to 100% asbestos
ESTIMATED LOD:	<1% asbestos [1]
PRECISION:	not determined

APPLICABILITY: this method is useful for the qualitative identification of asbestos and the semi-quantitative determination of asbestos content of bulk samples. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience. The method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope.

INTERFERENCES: Other fibers with optical properties similar to the asbestos minerals may give positive interferences. Optical properties of asbestos may be obscured by coating on the fibers. Fibers finer than the resolving power of the microscope (ca. 0.3 μm) will not be detected. Heat and acid treatment may alter the index of refraction of asbestos and change its color.

OTHER METHODS: This method (originally designated as method 7403) is designed for use with NIOSH Methods 7400 (phase contrast microscopy) and 7402 (electron microscopy/EDS). The method is similar to the EPA bulk asbestos method [1].

REAGENTS:

1. Refractive Index (RI) liquids for Dispersion Staining: high-dispersion (HD) series, 1.550, 1.605, 1.620.
2. Refractive Index liquids: 1.670, 1.680, and 1.700.
3. Asbestos reference samples such as SRM #1866, available from the National Institute of Standards and Technology.*
4. Distilled Water (optional).
5. Concentrated HCl: ACS reagent grade.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sample containers: screw-top plastic vials of 10- to 50-mL capacity.
2. Microscope, polarized light, with polarizer, analyzer, port for retardation plate, 360° graduated rotating stage, substage condenser with iris, lamp, lamp iris, and:
   a. Objective lenses: 10X, 20X, and 40X or near equivalent.
   b. Ocular lense: 10X minimum.
   c. Eyepiece reticle: crosshair.
   d. Dispersion staining objective lens or equivalent.
   e. Compensator plate: ca. 550 nm ± 20 nm, retardation: "first order red" compensator.
3. Microscope slides: 75 mm x 25 mm.
4. Cover slips.
5. Ventilated hood or negative-pressure glove box.
6. Mortar and pestle: agate or porcelain.
7. Stereomicroscope, ca. 10 to 45X.
8. Light source: incandescent or fluorescent.
9. Tweezers, dissecting needles, spatulas, probes, and scalpels.
10. Glassine paper or clean glass plate.
11. Low-speed hand drill with coarse burr bit (optional).

SPECIAL PRECAUTIONS: Asbestos, a human carcinogen, should be handled only in an exhaust hood (equipped with a HEPA filter) [2]. Precautions should be taken when collecting unknown samples, which may be asbestos, to preclude exposure to the person collecting the sample and minimize the disruption to the parent material [3]. Disposal of asbestos-containing materials should follow EPA Guidelines [4].

SAMPLING:

1. Place 1 to 10 g of the material to be analyzed in a sample container.
   NOTE: For large samples (i.e., whole ceiling tiles) that are fairly homogenous, a representative small portion should be submitted for analysis. Sample size should be adjusted to ensure that it is representative of the parent material.
2. Make sure that sample containers are taped so they will not open in transit.
3. Ship the samples in a rigid container with sufficient packing material to prevent damage or sample loss.

SAMPLE PREPARATION:

4. Visually examine samples in the container and with a low-magnification stereomicroscope in a hood. (If necessary, a sample may be carefully removed from the container and placed on glassine transfer paper or clean glass plate for examination). Break off a portion of the sample and examine the edges for emergent fibers. Note the homogeneity of the sample. Some hard tiles can be broken, and the edges examined for emergent fibers. If fibers are found, make an estimate of the amount and type of fibers present, confirm fiber type (step 14) and quantify (step 15).

5. In a hood, open sample container and with tweezers remove small, representative portions of the sample.
   a. If there are obvious separable layers, sample and analyze each layer separately.
   b. If the sample appears to be slightly inhomogeneous, mix it in the sample container with tweezers or a spatula before taking the portion of analysis. Alternatively, take small representative portions of each type of material and place on a glass slide.
   c. On hard tiles that may have thin, inseparable layers, use a scalpel to cut through all the layers for a representative sample. Then cut it into smaller pieces after placing RI liquid on it before trying to reduce the thickness. Alternatively, use a low-speed hand drill equipped with a burr bit to remove material from hard tiles. Avoid excessive heating of the sample which may alter the optical properties of the material.
      NOTE: This type of sample often requires ashing or other specialized preparation, and may require transmission electron microscopy for detection of the short asbestos fibers which are characteristic of floor tiles.
   d. If the sample has large, hard particles, grind it in a mortar. Do not grind so fine that fiber characteristics are destroyed.
   e. If necessary, treat a portion of the sample in a hood with an appropriate solvent to remove binders, tars, and other interfering materials which may be present in the sample. Make corrections for the non-asbestos material removed by this process.
      NOTE: Other methods of sample preparation such as acid washing and sodium metaphosphate treatment and ashing may be necessary, especially to detect low concentrations of asbestos. If needed, use as described in Reference [1].
6. After placing a few drops of RI liquid on the slide, put a small portion of sample in the liquid. Tease apart with a needle or smash small clumps with the flat end of a spatula or probe, producing a uniform thickness or particles so that better estimates of projected area percentages can be made. Mix the fibers and particles on the slide so that they are as homogeneous as possible.
   NOTE: An even dispersion of sample should cover the entire area under the cover slip. some practice will be necessary to judge the right amount of material to place on the slide. Too little sample may not give sufficient information and too much sample cannot be easily analyzed.

CALIBRATION AND QUALITY CONTROL:

7. Check for contamination each day of operation. Wipe microscope slides and cover slips with lens paper before using. Check refractive index liquids. Record results in a separate logbook.
8. Verify the refractive indices of the refractive index liquids used once per week of operation. Record these checks in a separate logbook.
9. Follow the manufacturer's instructions for illumination, condenser alignment and other microscope adjustments. Perform these adjustments prior to each sample set.
10. Determine percent of each identified asbestos species by comparison to standard projections (Figure 1) [1]. If no fibers are detected in a homogeneous sample, examine at least two additional preparations before concluding that no asbestos is present.
11. If it appears that the preparation technique might not be able to produce a homogeneous or representative sample on the slide, prepare a duplicate slide and average the results. Occasionally, when the duplicate results vary greatly, it will be necessary to prepare additional replicate slides and average all the replicate results. Prepare duplicate slides of at least 10% of the samples analyzed. Average the results for reporting.
12. Analyze about 5% blind samples of known asbestos content.
13. Laboratories performing this analytical method should participate in the National Voluntary Laboratory Accreditation Program [5] or a similar interlaboratory quality control program. Each analyst should have complete formal training in polarized light microscopy and its application to crystalline materials. In lieu of formal training, laboratory training in asbestos bulk analysis under the direction of a trained asbestos bulk analyst may be substituted. Owing to the subjective nature of the method, frequent practice is essential in order to remain proficient in estimating projected area percentages.

QUALITATIVE ASSESSMENT:

14. Scan the slide to identify any asbestos minerals using the optical properties of morphology, refractive indices, color, pleochroism, birefringence, extinction characteristics, sign of elongation, and dispersion staining characteristics.

    NOTE: Identification of asbestos using polarized light microscopy is unlike most other analytical methods. The quality of the results is dependent on the skill and judgment of the analyst. This method does not lend itself easily to a step-wise approach. Various procedures devised by different analysts may yield equivalent results. The following step-wise procedure repeatedly utilizes the sample preparation procedure previously outlined.

    a. Prepare a slide using 1.550 HD RI liquid. Adjust the polarizing filter such that the polars are partially crossed, with ca. 15° offset. Scan the preparation, examining the morphology for the presence of fibers. If no fibers are found, scan the additional preparations. If no fibers are found in any of the preparations, report that the sample does not contain asbestos, and stop the analysis at this point.

    b. If fibers are found, adjust the polarizing filter such that the polars are fully crossed. If all of the fibers are isotropic (disappear at all angles of rotation) then those fibers are not asbestos. Fibrous glass and mineral wool, which are common components of suspect samples, are isotropic. If only isotropic fibers are found in the additional preparations, report no asbestos fibers detected, and stop the analysis.

    c. If anisotropic fibers are found, rotate the stage to determine the angle of extinction. Except for tremolite-actinolite asbestos which has oblique extinction at 10-20°, the other forms of asbestos exhibit parallel extinction (Table 1). Tremolite may show both parallel and oblique extinction.

    d. Insert the first order red compensator plate in the microscope and determine the sign of elongation. All forms of asbestos have a positive sign of elongation except for crocidolite. If the sign of elongation observed is negative, go to step "g."

       NOTE: To determine the direction of the sign of elongation on a particular microscope configuration, examine a known chrysotile sample and note the direction (NE-SW or NW-SE) of the blue coloration. Chrysotile has a positive sign of elongation.

    e. Remove the first-order red compensator and uncross the polarizer. Examine under plane polarized light for blue and gold-brown Becke colors at the fiber-oil interface (i.e., index of refraction match). Becke colors are not always evident. Examine fiber morphology for twisted, wavy bundles of fibers which are characteristic of chrysotile. Twisted, ribbon-like morphology with cellular internal features may indicate cellulose fibers. It may be necessary to cross the polars partially in order to see the fibers if the index of refraction is an exact match at 1.550. If the fibers appear to have higher index of refraction, go to step "h," otherwise continue.

    f. Identification of chrysotile. Insert the dispersion staining objective. Observation of dispersion staining colors of blue and blue-magenta confirms chrysotile. Cellulose, which is a common interfering fiber at the 1.550 index of refraction, will not exhibit these dispersion staining colors. If chrysotile is found, go to step 15 for quantitative estimation.

    g. Identification of crocidolite. Prepare a slide in 1.700 RI liquid. Examine under plane-polarized light (uncrossed polars); check for morphology of crocidolite. Fibers will be straight, with rigid appearance, and may appear blue or purple-blue. Crocidolite is pleochroic, i.e., it will appear to change its color (blue or gray) as it is rotated through plane polarized light. Insert the dispersion staining objective. The central stop dispersion staining color are red magenta and blue magenta, however, these colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If observations above indicate crocidolite, go to step 15 for quantitative estimation.

    h. Identification of amosite. Prepare a slide in 1.680 RI liquid. Observed the fiber morphology for amosite characteristics: straight fibers and fiber bundles with broom-like or splayed ends. If the morphology matches amosite, examine the fibers using the dispersion staining objective. Blue and pale blue colors indicate the cummingtonite form of amosite, and gold and blue colors indicate the grunerite form of amosite. If amosite is confirmed by this test,

go to step 15 for quantitative estimation, otherwise continue.

i. Identification of anthophyllite-tremolite-actinolite. Prepare a slide in 1.605 HD RI liquid. Examine morphology for comparison to anthophyllite-tremolite-actinolite asbestos. The refractive indices for these forms of asbestos vary naturally within the species. Anthophyllite can be distinguished from actinolite and tremolite by its nearly parallel extinction. Actinolite has a light to dark green color under plane-polarized light and exhibits some pleochroism. For all three, fibers will be straight, single fibers possibly with some larger composite fibers. Cleavage fragments may also be present. Examine using the central stop dispersion staining objective. Anthophyllite will exhibit central stop colors of blue and gold/gold-magenta; tremolite will exhibit pale blue and yellow; and actinolite will exhibit magenta and golden-yellow colors.

NOTE: In this refractive index range, wollastonite is a common interfering mineral with similar morphology including the presence of cleavage fragments. It has both positive and negative sign of elongation, parallel extinction, and central stop dispersion staining colors of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is needed, go to step "j". If any of the above forms of asbestos were confirmed above, go to step 15 for quantitative estimation. If none of the tests above confirmed asbestos fibers, examine the additional preparations and if the same result occurs, report the absence of asbestos in this sample.

j. Wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with cover slip in place, on a warm hot plate until dry. By capillary action, place 1.620 RI liquid under the cover clip and examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show their original dispersion colors.

NOTE: There are alternative analysis procedures to the step-wise approach outlined above which will yield equivalent results. Some of these alternatives are:

i. Perform the initial scan for the presence of asbestos using crossed polars as well as the first-order red compensator. This allows for simultaneous viewing of birefringent and amorphous materials as well as determine their sign of elongation. Some fibers which are covered with mortar may best be observed using this configuration.

ii. Some analysts prefer to mount their first preparation in a RI liquid different than any asbestos materials and conduct their initial examination under plane-polarized light.

iii. If alternative RI liquids are used from those specified, dispersion staining colors observed will also change. Refer to an appropriate reference for the specific colors associated with asbestos in the RI liquids actually used.

QUANTITATIVE ASSESSMENT:

15. Estimate the content of the asbestos type present in the sample using the 1.550 RI preparation. Express the estimate as an area percent of all material present, taking into account the loading and distribution of all sample material on the slide. Use Figure 1 as an aid in arriving at your estimate. If additional unidentified fibers are present in the sample, continue with the qualitative measurement (step 14).

NOTE: Point-counting techniques to determine percentages of the asbestos minerals are not generally recommended. The point-counting method only produces accurate quantitative data when the material on the slide is homogeneous and has a uniform thickness, which is difficult to obtain [6]. The point-counting technique is, recommended by the EPA to determine the amount of asbestos in bulk [1]; however, in the more recent Asbestos Hazard Emergency Response Act (AHERA) regulations, asbestos quantification may be performed by a point-counting or equivalent estimation method [7].

16. Make a quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. If asbestos fibers are identified, report the material as "asbestos-containing". Asbestos content should be reported as a range of percent content. The range reported should be indicative of the analyst's precision in estimating asbestos content. For greater quantities use Figure 1 in arriving at your estimate.

EVALUATION OF METHOD:

The method is compiled from standard techniques used in mineralogy [8-13], and from standard laboratory procedures for bulk asbestos analysis which have been utilized for several years. These techniques have been successfully applied to the analysis of EPA Bulk Sample Analysis Quality Assurance Program samples since 1982 [1,5]. However, no formal evaluation of this method, as written, has been performed.

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METHOD WRITTEN BY:

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Appendix 15

(EPA 540-R-97-028) Superfund Method for the Determination of Releasable Asbestos in Soil and Bulk Materials

United States
Environmental Protection
Agency

Solid Waste and
Emergency Response

EPA 540-R-97-028
OSWER 9240.1-33
PB97-963503

Superfund

EPA

SUPERFUND METHOD FOR THE DETERMINATION OF RELEASABLE ASBESTOS IN SOILS AND BULK MATERIALS

SUPERFUND METHOD FOR THE DETERMINATION OF RELEASABLE ASBESTOS IN SOILS AND BULK MATERIALS

INTERIM VERSION

each tray into a clean, pre-weighed sample bottle (Figure 8-6) to be weighed (Section 8.2.1) and packaged for shipment to the laboratory (Section 8.4). Be sure that samples are transferred quantitatively from each tray.

NOTE

***NEVER* MAKE A PARTIAL TRANSFER OF MATERIAL FROM A SPLITTER TRAY TO A RECEIVING CONTAINER. THIS WOULD DEFEAT THE PURPOSE FOR HOMOGENIZATION AND SPLITTING BECAUSE IT IS IMPOSSIBLE TO ASSURE THAT ALL OF THE SIZE COMPONENTS OF THE SAMPLE ARE TRANSFERRED PROPORTIONALLY UNDER SUCH CIRCUMSTANCES.**

Clean the body of the splitter, all trays, and any appurtenant equipment (such as a nylon brush) between samples (but *not* between splits of the same sample) with a detergent wash followed by thorough rinsing with distilled, filtered water. Be sure that the splitter, trays, and appurtenant equipment are completely dry before use. These may be dried with forced air that is properly filtered to be free of asbestos.

Option 2: Use of a mixer with coning and quartering. Samples may also be homogenized for this method using any of various sealed, rotating mixers (tumblers). The mixers should contain internal baffles to promote mixing. Such mixers must be sufficiently large to accommodate the largest sample to be homogenized[17] with adequate room to spare so that tumbling is facilitated. The mixers must be sealable to prevent the loss of fines during mixing.

Place the fine fraction of the sample to be homogenized in a clean, dry mixer. Seal the mixer. Tumble the mixer at the manufacturers recommended speed for an amount of time recommended by the manufacturer to assure adequate homogenization. Stop the mixer and allow ample time (approximately 15 minutes) for the fines to settle. Disconnect the mixing container from the rest of the mixer. Open the mixer.

Under this option, a procedure termed coning and quartering is used to split a homogenized sample. Lay out a clean, aluminum plate on a flat surface. Hold the mixer immediately over the center of the plate and rotate the mixer around an axis represented by the lip on one side of its mouth so that the sample material *slowly* pours onto the metal plate forming a symmetrical cone (Figure 8-7). Keep the point at which the poured material impacts the cone at the same spot and slowly raise the mixer as the pouring continues to keep the distance between the mixer lip and the top of the cone approximately constant. When the mixer is fully inverted, tap it vigorously to complete the quantitative transfer.

To halve the cone, hold a second (clean, dry) aluminum plate directly over the apex (top center) of the cone at an angle that is perpendicular to the aluminum plate on which the cone lies. Slowly lower the second plate so that it splits the cone precisely in half (Figure 8-8a). While holding the two plates steady, push one half of the cone off of the original plate and away from the rest of the sample (Figure 8-8b). Brush the area from which this material is

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[17] If samples are to be composited as described in Section 8.3, the mixer may have to be capable of handling samples that range up 40 kg in size.

FIGURE 8-6

LOADING A SAMPLE SPLIT INTO A SAMPLE BOTTLE

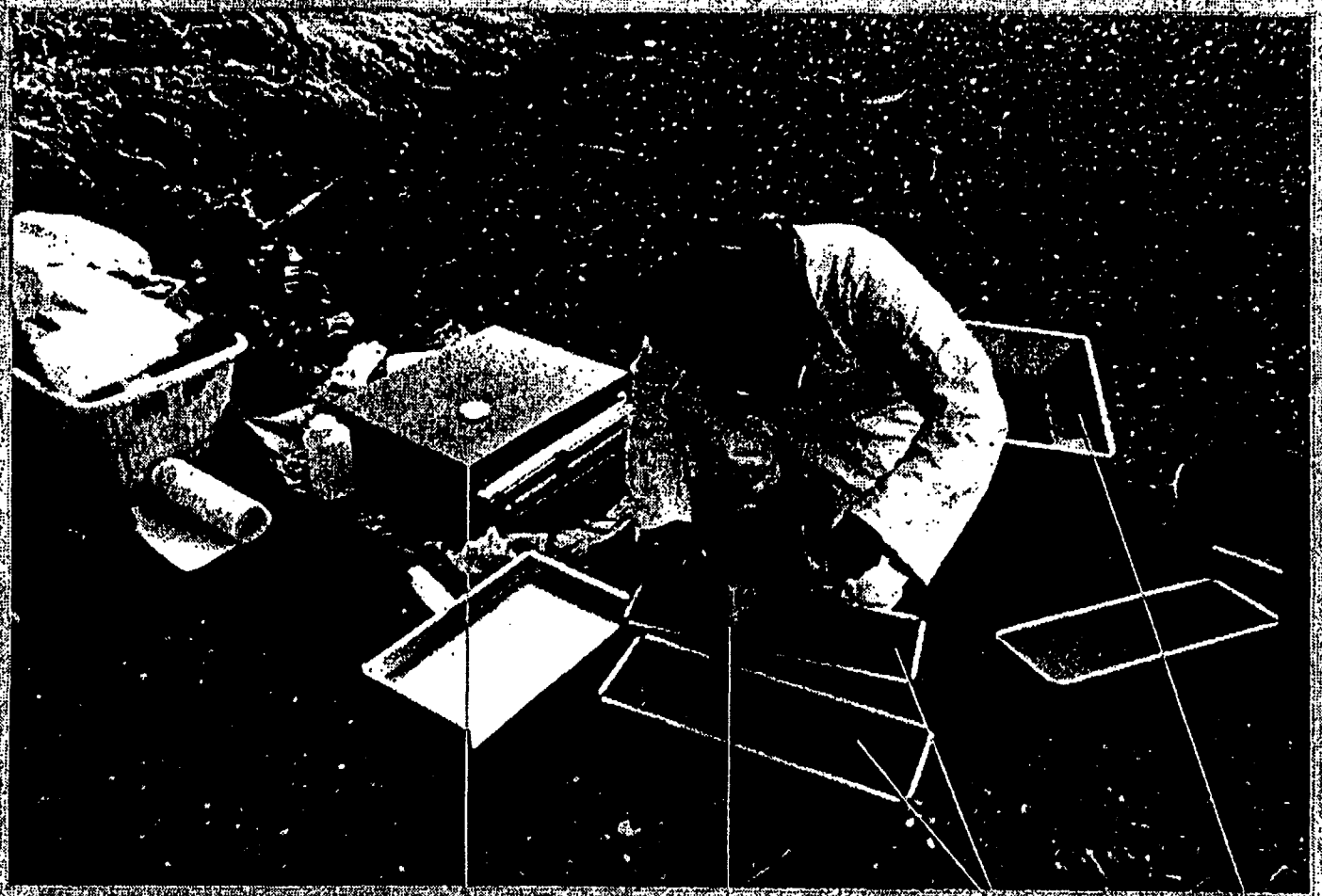


FIGURE 8-7

TRANSFERING SAMPLE FROM A MIXER TO A PLATE FOR CONING AND QUARTERING

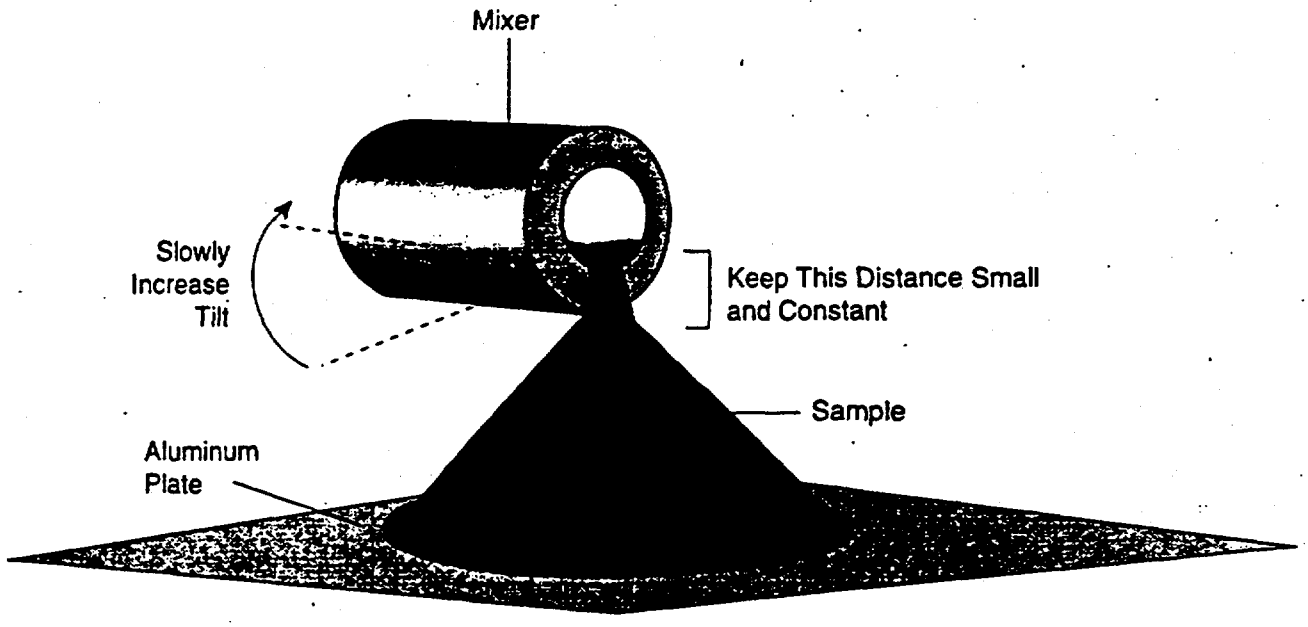
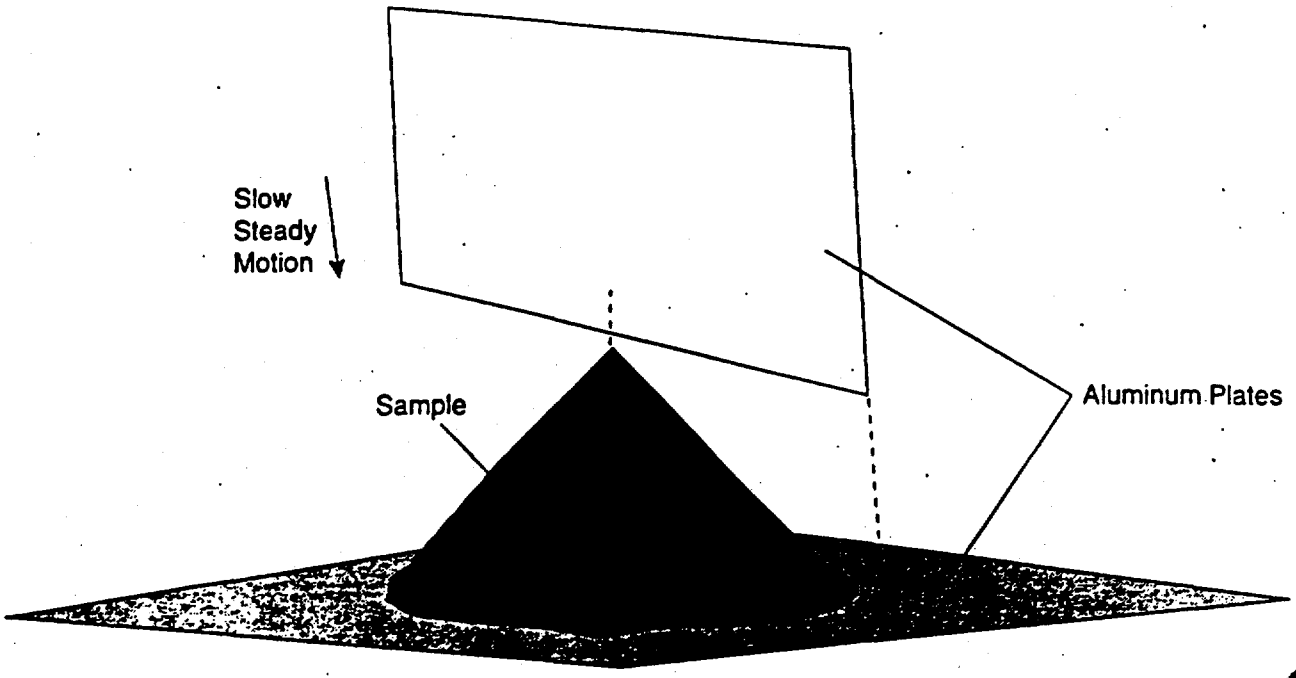
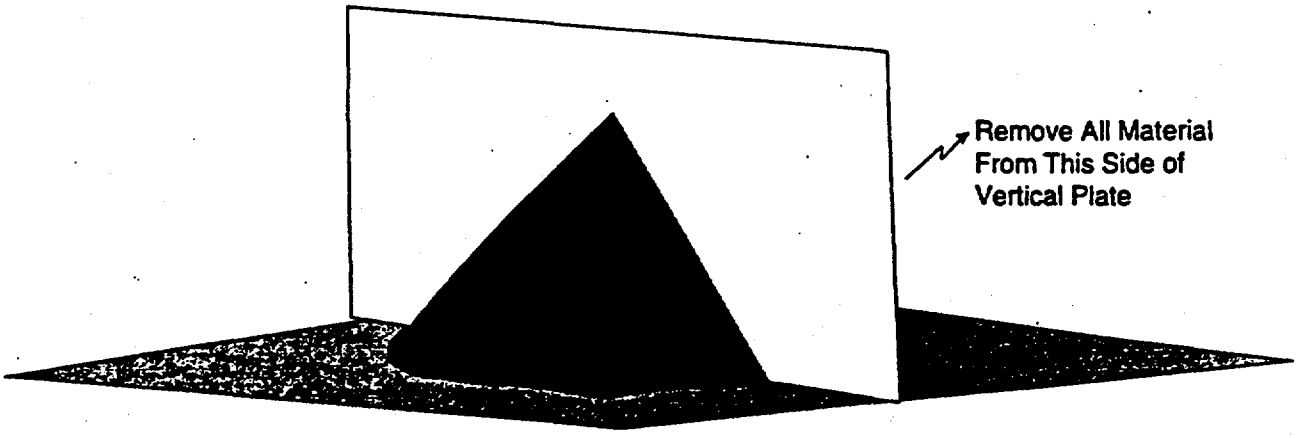


FIGURE 8-8

CONING AND QUARTERING



A. POSITION OF ALUMINUM PLATES IN PREPRATION FOR HALVING SAMPLE



B. POSITION OF ALUMINUM PLATES AFTER HALVING SAMPLE

removed to complete a quantitative transfer, leaving only clean metal. Once half of the sample material has been removed, withdraw the vertical aluminum plate slowly by pulling it upward vertically.

To complete quartering of the cone, rotate the vertical aluminum plate above the cone 90° on a vertical axis. Lower the plate slowly once again so that it splits the remaining portion of the sample cone evenly into two new halves. Once again, quantitatively remove half of the sample material (i.e. remove all of the material from one side of the vertical plate). This process may be repeated by quantitatively transferring the remaining sample material into a clean bucket and pouring the sample onto the clean aluminum plate to form a new cone.

Repeat the coning and quartering process until the remaining quarters (or halves) of the sample at the end of one cycle falls in the range of 50 to 80 g. At that point, carefully transfer the material from each quarter (or half) into a clean, pre-weighed sample bottle to be weighed (Section 8.2.1) and packaged for shipment to the laboratory (Section 8.4).

NOTE

NEVER MAKE A PARTIAL TRANSFER OF MATERIAL FROM ANY PORTION OF THE CONE THAT DOES NOT INCLUDE A WEDGED-SHAPE SLICE THROUGH THE CENTER OF THE CONE OVER ITS ENTIRE THICKNESS (DEPTH). THIS WOULD DEFEAT THE PURPOSE FOR FORMAL CONING AND QUARTERING BECAUSE IT IS IMPOSSIBLE TO ASSURE THAT ALL OF THE SIZE COMPONENTS, WHICH WILL *NOT* BE HOMOGENEOUSLY DISTRIBUTED VERTICALLY THROUGHOUT THE CONE OF THE SAMPLE, ARE TRANSFERRED PROPORTIONALLY.

8.3 COMPOSITING SAMPLES (OPTIONAL)

In many cases, there may be interest in limiting the number of analyses required to characterize a matrix that serves as a potential source *without* sacrificing representativeness. One procedure that may be employed for this purpose is to composite samples in the field. Note however, while compositing can reduce the cost of analysis by reducing the number of samples requiring analysis, also lost is information concerning the spatial variability of the sampled matrix. Therefore, if such information is desired for any particular reason, compositing is not recommended.

Only minor adjustments to the field preparation procedures described above are required to incorporate compositing into this method. First, during planning, group the samples to be collected in the field into sets that are to be composited. For example, there may be a desire to combine all samples from the eastern part of a road into a composite representing the east end of the road. Similarly, samples from the west end might be combined into a west end composite. Alternately, all samples from the road may be combined into a single composite, representing the road as a whole. As another alternative, the composite road sample might also be split into duplicate pairs to allow determination the variability contributed by sample preparation and analysis. Such decisions shall all be determined during planning.

NOTE

It is expected that the same set of locations will be selected for sample collection whether or not compositing is employed; compositing only changes the number of analyses required. This is because, the same number of samples collected from a set of locations selected using the same formal procedures are still required to adequately characterize the sampled matrix, whether or not samples are composited prior to analysis.

When brought to the central location where field preparation is conducted, after the initial weighing, the samples collected from a set that is to be composited can be combined in a common bucket. Modify the procedures described in Sections 8.2.1 and 8.2.2 as follows:

- transport each sample (of a set to be composited) from the point of collection to a central location in a clean bucket;

- weigh the sample and record the weight along with the appropriate sample identifier. Subtract the tare weight of the bucket and record the net weight of the sample;

- sieve the sample and collect the fine fraction from each sample in the set to be composited in a common bucket. Weigh the bucket containing the fines following the addition of the contributions of each sample and subtract the previous weight of the bucket to determine the net weight contributed by each sample. Record the weight with the proper identifier in the field notebook; and

- transfer the coarse fraction from each sample in the set to be composited to a common bucket. Weigh the bucket containing the coarse fraction following the addition of the contributions from each sample and subtract the previous weight of the bucket to determine the net weight contributed by each sample. Record the weight of the coarse fraction with the proper sample identifier in the field notebook.

Once all the samples of the set to be composited have been collected and added, the combined fine fraction from all of the samples (which resides in a common bucket) shall be given a separate identifier representing the intended composite. Record the new identifier in the field notebook. This material can now be treated as a single, composite sample for all remaining steps of field preparation and sample handling, packaging, and shipment to the laboratory. Thus, homogenize and split the sample as described in Section 8.2.3 and package and ship the sample to the laboratory as described in Section 8.4. Record the appropriate weights of the samples to be shipped as described in Section 8.2.1.

8.4 SAMPLE HANDLING AND SHIPMENT

Once their weights and identifiers are recorded, the samples to be shipped to the laboratory must be sealed and labeled. Fill out and apply appropriate labels to each sample bottle. Record the date and time that each sample was created on both the label and the field notebook and be sure that the identification numbers on the label and field notebook match.

Fill out the appropriate chain of custody forms and seal each sample cap with a breakaway label. As indicated in Section 12.2, be sure to complete the field activities report and to include this report in the package with samples sent to the laboratory.

Wipe each sample to be shipped to the laboratory with a clean, asbestos-free cloth and place it in a cooler. Ship samples to the laboratory in a cooler with ice to limit biological growth during shipment. Sufficient ice must be provided to assure that samples remain cold until received and processed by the laboratory.

NOTE

When matrices that are sampled contain a significant fraction of coarse material (i.e. more than 10% by mass), the final determination of the concentration of asbestos in that matrix must be adjusted to account for the fraction of coarse material. This requires determining the ratio of the mass of *fine* material in the sampled matrix to the total mass of material in the sampled matrix to generate a "coarseness adjustment factor". The concentration of asbestos determined for samples sent to the laboratory must then be multiplied by this coarseness adjustment factor to determine the concentration of asbestos in the sampled matrix.

Equations for deriving and using the coarseness adjustment factor are provided in Section 11.4.3. The weights of the coarse and fine fractions of each sample are to be included (along with the appropriate sample identifiers) as part of the field activities report that is to be shipped with the sample to the laboratory (Section 12.2). This assures that individuals responsible for estimating the concentration of asbestos for the project have access to the required field information.

9.0 SAMPLE PREPARATION BY DUST GENERATION

The primary purpose for sample preparation by dust generation that is described in this section is to generate dust-laden filters that can be suitably prepared for analysis by an appropriate method for the determination of asbestos in air[18]. The rate of generation of total respirable dust is also monitored and is used both to estimate the total mass of respirable dust in the original sample and to tie asbestos structure concentrations determined from filters to the mass of the original sample. Such information might also be used in some studies to characterize the releasibility of asbestos (or total dust) from particular sample types.

A detailed description of the apparatus employed for dust generation and its theory of operation is provided in Appendix A. Specifications and construction drawings are also provided.

9.1 SAMPLE RECEIVING AND STORAGE

All samples received from the field are to be wiped clean with a damp cloth prior to storage or other handling. Samples shall be stored at ice temperature (to minimize biological growth) until sample preparation is initiated. To minimize complications from biological agents, once initiated, sample preparation shall be completed expeditiously. In any case, sample preparation shall be completed within 48 hours.

Samples to be prepared using the dust generator are to be inspected for the presence of free water. If a sample contains free water or if the sample appears visibly moist, it shall be dried at low temperature. If time permits, place the sample in an open, shallow container and store it for several days in a desiccator containing *moist* potassium carbonate dihydrate or another salt that corresponds to the salt selected for humidity control (see Section 9.3.2). The type of salt placed in the desiccator is chosen deliberately; rather than to dry the sample completely, the goal is to bring the moisture content of the sample into equilibrium with conditions that will prevail in the dust generator.

If sufficient time is not available to dry the sample in a desiccator, the sample may be oven dried. Dry the sample in an open, shallow container in an oven that is maintained at a temperature *below* 60° C until the sample comes to constant weight. Note that oven-dried samples may require additional time for conditioning (Section 9.4.2) because the moisture content of the sample will need to be increased to bring it into equilibrium with conditions prevailing in the dust generator.

Once dry, samples smaller than 80 g can be loaded directly into the tumbler of the dust generator (Section 9.4.1). Larger samples must be homogenized and split, as described in Section 9.2, prior to being placed in the tumbler of the dust generator.

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[18] The ISO method for the determination of asbestos in air (using either an indirect or a direct filter preparation technique - - Chatfield 1993) is the default method recommended for use in tandem with this method.

9.2 SAMPLE HOMOGENIZATION AND SPLITTING IN THE LABORATORY

Samples received from the field that are larger than approximately 80 g must be dried, as described above, and then homogenized and split as described in this section.

WARNING:

BECAUSE ASBESTOS CONTAINING DUSTS MAY BE GENERATED FROM THE HANDLING AND PREPARATION OF BULK SAMPLES, ALL OF THE FOLLOWING PREPARATION STEPS SHALL BE PERFORMED IN A PROTECTIVE ENCLOSURE (I.E. A HEPA FILTERED GLOVE BOX OR AN APPROVED FUME HOOD THAT IS DESIGNED TO MINIMIZE EXPOSURE TO LABORATORY PERSONNEL[19]). IT IS ALSO NECESSARY THAT ALL HANDLING OF BULK SAMPLES BE CONDUCTED IN A SEPARATE ROOM THAT IS PHYSICALLY ISOLATED FROM THE ROOM(S) IN WHICH AIR SAMPLES ARE HANDLED AND ASBESTOS ANALYSIS IS PERFORMED.

As with field homogenization and splitting (Section 8.2.3), either of two options may be selected for homogenization and splitting in the laboratory. When performed in the laboratory, however, such equipment must fit within an appropriately designed, protective enclosure, which is why field preparation may be cost-effective.

Homogenize large samples in precisely the same manner as described in Section 8.2.3. Once samples are homogenized, split samples in precisely the same manner as described in Section 8.2.3. Continue splitting until a paired set of samples are produced that each contain between 50 and 80 g of material. Record in a laboratory notebook the final weights and identification numbers of the samples homogenized and split.

9.3 DUST GENERATOR SETUP

Prior to using the dust generator, a supply of at least 35 MCE filters must be conditioned and stored for use, the constant humidity chamber must be loaded with the appropriate solution, the scrubber must be primed, and air flow within the dust generator must be calibrated and adjusted.

9.3.1 Conditioning a Stock of Filters

A stock of at least 35 filters (0.45 µm pore size, 25 mm diameter), all from the same filter lot, must be conditioned in a desiccator overnight to bring them into equilibrium with the relative humidity at which they will be used during a run. Place the 35 MCE filters in a desiccator containing *moist* salt of the same variety as that selected to fill the pans in the humidity control chamber of the dust generator (Section 9.3.2). For most applications, this will be potassium carbonate dihydrate (see Appendix A).

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[19] The work should be performed in a Class II biohazard hood as per the specifications of Standard #49 of the National Sanitation Foundation.

After storing the filters overnight in the desiccator, pre-weigh each filter to a minimum precision of ± 0.0002 g[20]. Each filter shall then be placed in a separate, covered Petri dish with its weight marked on the top of the container. The lids shall also be numbered sequentially and the filters shall all be used during the run in the order numbered.

9.3.2 Initiating Humidity Control

Use asbestos-free (filtered, distilled) water to make a 2 L solution of saturated salt. As indicated previously, for most applications, use potassium carbonate dihydrate (to achieve a relative humidity of 43%), but other salts may be used for specific applications (see Appendix A)[21].

Prepare the solutions by placing 1000 g of the anhydrous salt into a one L container and adding distilled water to fill the container. The container should be capped and additional water and salt added as necessary the next day. No water or salt should be added within a day of using the mixture since some time is needed to saturate the salt solution and form a hydrate when adding anhydrous potassium carbonate. However, sufficient salt shall have been added previously to assure that sufficient undissolved potassium carbonate dihydrate has precipitated in the container to form a closely spaced layer of the material on the bottom of the shallow pans that will be covered with a thin layer of the solution (next paragraph).

Open the top of the humidity control chamber and remove the two pans. Fill each pan with the saturated salt solution being sure that a small quantity of excess (undissolved) salt is also transferred to each pan. Replace the pans and seal the top of the plastic enclosure; air should enter the enclosure primarily from the front opening.

9.3.3 Priming the Scrubber

Fill the round bottom flask of the scrubber to about one third full with asbestos-free (i.e. filtered, distilled) water. Initiate the flow of ice water through the entrance and exit condensers. Adjust the variable voltage transformer on the heating mantle so that water in the round bottom flask boils and the rate at which condensate drops back into the flask from the condensers is approximately equal to one drop per second.

9.3.4 Adjusting Initial Air Flow

The air flow within the various components of the dust generator must be adjusted so that flow within the vertical elutriator will properly separate and pass only respirable particles. Based on the discussion presented in Appendix A (Section A.2.3), the proper linear flow rate in the elutriator shall be set at 0.31 cm/s, which is 5% greater than the Stokes' velocity estimated for the largest spherical, respirable particles (i.e. those with a radius of 5 μm).

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[20] Filters to be used to collect samples over the isokinetic sampling tube of the elutriator (see Section 9.4.5) must be weighed to a minimum precision of ± 0.00002 g.

[21] Note that potassium carbonate dihydrate is not the usual form of potassium carbonate sold commercially. The usual commercial forms are the anhydrous and the sesquihydrate. The dihydrate can be made by allowing either of the commercial varieties of the salt to stand in their saturated solution for some extended period of time with some temperature cycling (Berman and Kolk 1994). A week appears to be sufficient but the process can be accelerated by augmenting the temperature cycling.

Next, calculate the required volumetric air flow, V_v, within the elutriator using Equation 9-1:

$$V_v = 81.1 * V_l \qquad (9\text{-}1)$$

where:

V_l is the estimated linear flow rate required to separate respirable particles (i.e. 0.31 cm/s); and

V_v is the corresponding volumetric flow rate (cm^3/s) through the elutriator.

The coefficient, 81.1, in Equation 9-1 corresponds to the cross-sectional area of the elutriator (in cm^2).

To adjust the initial flow valve settings on the pumps, first connect one of the flowmeters such that air flows directly from the exit opening at the top of the elutriator that is *not* articulated with the isokinetic sampling tube[22]. This is the exit that opens directly into the top, tapering portion of the elutriator (see Appendix A) and is labeled the "ME" opening in Figure A-1. A filter cassette containing a filter from the batch of filters to be used for the run shall be placed between the flowmeter and the pump when adjusting the flow valve on this pump.

NOTE

The easiest way to directly connect a flow line to one of the top openings of the elutriator (the ME or the IST opening) is to align the appropriate slide mechanism so that a filter mount is directly over the opening, mount a cassette *without* a filter into the mount, and connect the air flow line to the exit side of the filterless cassette (see Section A.1.6 of Appendix A). During calibration, such a line would then feed sequentially into a flowmeter, a filter-containing cassette, and a pump.

Connect a second flowmeter directly to the exit line of the scrubber (the entrance line of which should already be attached to the two side exit openings on the elutriator) and place a filter cassette containing a filter from the batch of filters to be used for the run between this flowmeter and the pump for the scrubber.

Adjust the flow control valves on both the scrubber pump and the pump to be connected to the ME opening of the elutriator so that flow in both lines are equal and that each flow is set at $0.48 * V_v$.

Connect a third flowmeter to the exit opening on the top of the elutriator that articulates with the isokinetic sampling tube. This is labeled the "IST" opening in Figure A-1.

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[22] To access this opening, it will be necessary to dismount any filter cassettes from the appropriate slide mechanism and to align one of the two openings in the appropriate slide mechanism over the desired opening in the elutriator (see Appendix A).

The flowmeter attached to the IST opening shall also be backed by a filter cassette containing a filter from the batch to be used during the run and adjust the flow in this line so that it is equal to $0.047*V_v$[23]. Due to the low flow required on this line, an auxiliary low flow valve is also attached to this pump and must be adjusted to achieve the desired flow. To optimize conditions, it may be necessary to adjust the flow control valves on the three air lines that exit the elutriator iteratively.

To prepare for a run using the dust generator, disconnect the flowmeters from the slide mechanisms over the top openings of the elutriator (i.e. the ME and IST openings) and mount filters in each of the four cassette holders on the two slide mechanisms that cover the two elutriator openings. Connect one of the air flow lines coming off of the "T" from each pump to each of the two filters on the same slide mechanism for the appropriate opening of the elutriator (i.e. the opening for which the pump had been calibrated). Then adjust each slide so that one filter cassette is aligned directly over each elutriator opening. Be sure that the valves on each "T" are configured so that flow is directed from the filter cassette that is aligned directly over the elutriator opening (see Section A.1.6 of Appendix A).

NOTE

The connections between the exit side of the filter cassettes mounted on the slide mechanisms and the flow control valves on the pumps should now be direct; there should be no second filter cassette in the line.

The flowmeter attached to the exit line of the scrubber may remain attached during a run to monitor air flow through the scrubber. If there is a desire also to monitor airflow through one or both of the filter cassettes mounted over the top openings of the elutriator during a run, flowmeters may now be attached to the downstream side of the filter cassettes (i.e. between the filter cassettes and the pumps). Due to the pressure drop across the filters, however, the readings from these flowmeters must be adjusted using the following equation to provide estimates of the true flow through each elutriator opening:

$$F_t = R_f*(P_f*T_t/P_t*T_f) \qquad (9\text{-}2)$$

where:

F_t is the true flow rate through the elutriator opening (cm/s);

R_f is the flow reading from the flowmeter (cm/s);

P_f is the pressure at the flowmeter (torr);

T_f is the *absolute* temperature at the flowmeter (^oK);

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23 The coefficient, 0.047, used to estimate the volumetric flow rate for air passing through the isokinetic sampler represents the fraction of the cross-sectional area of the elutriator that is subtended by the isokinetic sampling tube and therefore represents the fraction of the total flow that should pass through the tube, assuming that flow in the elutriator has been properly set.

P_t is the pressure at the elutriator opening (torr); and

T_t is the *absolute* temperature at the elutriator opening (^oK).

To use this equation, P_f and P_t will have to have been measured during flow calibration, prior to a run, using mercury manometers or other appropriate pressure measuring devices. Generally, T_t and T_f can be considered equal and will drop out of the equation. However, Equation 9-2 can also be used to adjust flow readings between calibrations and runs that are conducted on different days, such that temperatures may vary between the time during which the calibration was conducted and the time that the run is performed.[24]

9.4 DUST GENERATOR OPERATION

To prepare asbestos samples using the dust generator, load the tumbler, condition the bulk sample, begin the run, monitor the rate of dust generation, and collect appropriately loaded filters for asbestos analysis. Asbestos is also collected in the scrubber. Prior to use, be sure that the dust generator is clean (see Section 9.5).

9.4.1 Loading the Tumbler

Detach the tumbler from its drive motor and the vertical elutriator and remove it from the plastic enclosure at the bottom of the dust generator (see Appendix A). Place the tumbler on a flat surface and open the top for loading. Be sure that the tumbler is clean prior to loading.

Introduce a sample[25] by holding the sample container against the inner lip of the tumbler and tilting the container so that the sample pours smoothly into the tumbler. Move the sample container back and forth along the length of the tumbler to facilitate uniform deposition of the sample in the tumbler. When pouring is complete, tap the sample container vigorously so that the quantitative transfer is complete. The masses of samples introduced into the tumbler shall range between 50 and 80 g. Larger samples shall be homogenized and split prior to loading as described in Section 9.2.

Shake the tumbler gently to assure uniform deposition of the sample within the tumbler, which should be no more than about one third full. Be sure that the rubber gasket on the tumbler is in good repair and properly seated. Replace the gasket if it is worn. Secure the top of the tumbler with 10 screws and replace the tumbler within the plastic enclosure at the bottom of the dust generator. Reattach the elutriator entrance tube and D.C. motor to the tumbler (see Appendix A).

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24 Because the viscosity of air is somewhat temperature dependent, when runs are to be conducted at temperatures that differ by more than a few degrees from room temperature (nominally 20° C), Equation A.10 (in Appendix A) may have to be adjusted to account for the varying viscosity (see Equation A.9) so that the correct flow regime can be established at the new temperature to assure that the elutriator of the dust generator passes only respirable particles.

25 Samples to be introduced into the tumbler shall have been dried per Section 9.1.

9.4.2 Conditioning the Sample

Before conditioning the sample, be sure that the dust generator has been properly set up. This means, check that:

- the pans in the constant humidity chamber have been filled with saturated solution;
- filters have been mounted on each of the four cassette mounts on the slide mechanisms atop the elutriator;
- water is boiling in the scrubber;
- the air flow valves have been properly set; and
- all air lines between the dust generator, flow valves, and pumps are properly configured (see Section A.1.6 of Appendix A).

To condition the sample, turn on all pumps and begin the flow of air through the dust generator. ***DO NOT TURN ON THE TUMBLER MOTOR.*** Allow the flow of air to continue for a minimum of two hours before beginning a run. If the sample was oven dried rather than equilibrated with an appropriate salt in a desiccator (Section 9.1), the sample should be conditioned for a minimum of four hours prior to initiating a run.

9.4.3 Initiating a Run

Once the sample has been conditioned, set the tumbler drive motor to 30 rpm and turn it on. Simultaneously, move the two slide mechanisms at the top of the elutriator so that new, clean filters are now aligned over both the ME and IST openings of the elutriator. Be sure to change the valve orientations on the lines leading to the filters so that air flow is directed through the filter cassettes that are newly aligned with the elutriator openings.

Replace the filters originally aligned over the elutriator openings (but no longer aligned) with clean filters and weigh and store the old filters in labelled Petri dishes. These filters are equipment blanks. After five minutes,[26] move the sliding mechanism again to bring new, clean filters over the ME and IST openings of the elutriator. Immediately after the filters are brought out of alignment, dismount the cassette and turn the potentially dust-laden side of the filter face up before halting the flow of air through the filter (by turning the appropriate valves). Once flow from the dismounted cassettes has been halted (correspondingly, flow will have been re-directed to the cassettes that are currently aligned over the elutriator openings), replace the dismounted filters with clean filters and weigh and store the dismounted filters in labelled Petri dishes. These filters are run blanks.

As the run proceeds, record the times that air flow was started and stopped for each filter, the initial and final weights of each filter, and the identifier of each filter in a log book.

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[26] This interval is selected because, in the absence of channeling, five minutes is just less than the time over which the fastest particles are expected to reach the filter.

9.4.4 Monitoring the Rate of Respirable Dust Generation

The rate of respirable dust generation is monitored during a run by recording the weights of a set of filters that are sequentially changed out of the filter mounts over the ME opening of the elutriator at defined, regular intervals.

Initially, change the filter that is aligned over the ME opening of the elutriator at intervals of five to eight minutes. The change is accomplished by moving the slide mechanism to switch a new filter into alignment at the same time that the old filter is switched out of alignment. Immediately after the filter is brought out of alignment, dismount the cassette and turn the dust-laden side of the filter face up before halting the flow of air through this filter (i.e. by turning the appropriate valves to re-direct air flow to the filter that is newly aligned over the ME opening). Exchange the old filter for a new filter.

NOTE

Because the time during which air flow is directed through a dismounted cassette (rather than the filter that is aligned over the elutriator opening) results in a disturbance in the otherwise smooth flow of air through the elutriator, the changing of filters shall be performed expeditiously. As long as this interval is not more than a few seconds, however, studies indicate that this effect is not significant (Berman and Kolk 1994).

Along with the proper identifier, record the times during which air flow is started and halted for each filter. Weigh each filter after dismounting. Record the initial and final weights of the filter and the net weight of dust deposited on the filter (i.e. the difference between the initial and final weight).

After exchange of the first two or three filters, the interval over which dust is collected on each filter may be optimized. The ideal weight of dust to be deposited on each filter is between 0.01 and 0.03 g (Berman and Kolk 1994). Based on the rate of dust deposition on the first two or three filters, estimate the interval of time required to deposit approximately 0.02 g and exchange later filters at this rate (see Section A.2.2 of Appendix A, Equation A-8)[27]. However, it is important that time be adjusted so that no more than 0.03 g be deposited on each filter because the possibility that a portion of the deposit accidently drops from the filter increases as the weight of the deposit on the filter increases.

NOTE

Until there is a need for generating filters for asbestos analysis (see Section 9.4.5), the filters that get aligned over the IST opening of the elutriator need to be changed only one fifth to one tenth as often as the filters over the ME opening of the elutriator. These filters shall be changed at this lower rate, however, to prevent the potential for a heavy deposit to drop off of the filter and fall back into the elutriator.

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[27] At the beginning of a run the rate of dust deposition on the filters has been observed to be nearly constant with time.

Continue the run at 30 rpm (with continuing exchange of filters) for approximately two hours; this time interval has generally been observed as sufficient to define the rate of respirable particle release at this rotation rate (Berman et al 1994a). Generally, the plot of the release of respirable dust versus time at 30 rpm shows almost no curvature (see Section 11.2).

After completing the run at 30 rpm, select a new, higher rotation rate to continue the run. Generally, the new rotation rate selected shall be 60 rpm, unless the rate of release at 30 rpm was noticeably low in comparison with prior runs on other samples, in which case 90 or 120 rpm shall be used.

NOTE

Use of the highest rotation rates should generally be avoided, unless there is compelling evidence for their efficacy, because they tend to facilitate the transport of non-respirable particles from the tumbler into the bottom of the elutriator and, if such transport is heavy, this may affect results (see Section A.1.3 of Appendix A).

Continue the run at the higher rate of rotation by collecting a minimum of eight additional dust-laden filters. The same procedures outlined above should be continued for collecting data during the run at this higher rotation rate except that the interval between the exchange of filters must be adjusted downward to assure that deposits on these latter filters do not exceed 0.03 g (see Section A.2.2 of Appendix A, Equation A-8, but note that the rate constant, k, is dependent on the rotation rate for the tumbler so that Equation A-8 cannot be extrapolated across runs).

When the run is complete, turn off the tumbler motor but allow the air flow to continue for ten or fifteen additional minutes to empty the elutriator. Be sure to continue the exchange of filters, if necessary to prevent overloading. The air flow pumps may now be shut off.

9.4.5 Generating Appropriately Loaded Filters for Asbestos Analysis

The primary purpose for collecting dust on filters mounted over the exit of the isokinetic sampling tube of the elutriator (the opening labeled "IST" in Figure A-1) is to obtain samples suitable for asbestos analysis using a direct transfer technique, although use of an indirect transfer technique is not precluded. This is an option built into the design of the dust generator as an alternative to preparation of a specimen for asbestos analysis using the liquid from the scrubber, which necessarily mimics an indirect transfer technique because (in the scrubber) the asbestos is captured and suspended in water.

Collect filters for asbestos analysis near the end of each of the two runs (i.e. one run at each of two rotational speeds for the tumbler) that is described in the previous section of this chapter. Filters to be used for asbestos analysis shall be collected at the end of the runs both because this will be the period when the rate of asbestos emission is the lowest and because sufficient time will have elapsed over each run to allow a steady-state distribution of particle sizes to have developed in the dust traversing the elutriator. At the beginning of a run, only the smallest (fastest) respirable particles reach the filters and it takes time for the larger (slower) respirable particles in the air stream to begin reaching the filters in numbers that are proportional to their rate of emission from the tumbler. It takes several tens of minutes for transport of a steady state distribution to develop.

Collect multiple filters during each run that bracket the estimated time during which an optimal loading for analysis of a directly prepared specimen is expected to be achieved. Mount, exchange, dismount, weigh, record, and store filters precisely in the manner described in Section 9.4.4.

Estimate the time required for achieving an optimum loading as follows. The optimal mass loading on a filter to be prepared by a direct transfer technique lies between 1 and 10 µg (see, for example, Berman and Chatfield 1990). Assume a target of 5 µg. However, this may have to be adjusted based on experience with the dust generator. Equation A-7 (Appendix A) can then be re-arranged to estimate the time required to collect 5 µg (or some other defined mass) of dust:

$$\Delta t = \Delta M_f / 0.047 * k * M_s \qquad (9\text{-}3)$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" after the beginning of the run, but it is assumed constant over the short interval of time "Δt" (g);

ΔM_f is the mass of respirable dust collected on a filter over the IST opening during the short time interval "Δt" (as indicated above, assume a target of 5 µg or 5×10^{-6} g);

Δt is a short time interval (no more than several minutes) during which the release of dust is being estimated (s); and

k is the first-order rate constant for the release that is derived from the dust measurements collected during the run (s^{-1}).

The mass of respirable dust remaining in the sample during the time interval of interest (K_s) is estimated using a rearrangement to Equation A-2 (Appendix A):

$$M_s = M_o * \exp(-kt) \qquad (9\text{-}4)$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" (g);

M_o is the mass of respirable dust in the sample at the start of the run (i.e. at time t = 0) (g).

t is the time from the start of the run to the beginning of the time interval "Δt" (s); and

k is the first-order rate constant for the release (s^{-1}).

Based on the recently completed pilot study for this method (Berman et al. 1994a), a typical rate constant for dust emission from the tumbler is 0.004 min^{-1} (6.7×10^{-5} s^{-1}). Results from this study also suggest that the range in respirable dust content likely to be encountered for samples typically run using the dust generator may vary between 0.5% and 2%. Therefore, given a typical sample mass of 70 g, M_o for the 30 rpm run likely ranges between approximately 0.17 and 0.68 g. Given that a typical 30 rpm run lasts for approximately 3 hours (1.1×10^4 s) and substituting these values for M_o, k, and t into Equation 9-4, and then substituting the subsequent estimate of M_s into Equation 9-3, it appears that between 2.3 and 9 seconds would be required at the end of the 30 rpm run to collect 5 µg of material on a filter loaded over the isokinetic sampler.

Similarly, assuming that the initial mass of respirable dust in a sample at the beginning of a 60 rpm run, $M_{o\text{-}60}$, is equal to the remaining mass at the end of the 30 rpm run (i.e. the "M_s" calculated above), remembering that a 60 rpm run typically lasts 2 hours (7.2×10^3 s), and once again noting the typical value for k indicated above, Equation 9-4 is used to estimate an appropriate M_s for the end of a 60 rpm run. Substituting this new value into Equation 9-3, it appears that between 4 and 15 seconds may be required to collect 5 µg of material on a filter that is loaded over the isokinetic sampler at the end of a 60 rpm run.

Given the above, to properly bracket the optimal loading for a filter to be employed for asbestos analysis using a (direct transfer technique), collect filters over the IST opening of the elutriator that are exposed for periods of 3, 10, and 20 seconds (at both the end of the 30 rpm run and the end of the 60 rpm run).

9.4.6 Obtaining Asbestos Samples from the Scrubber

At the end of all runs for a particular sample (after all pumps have been shut off), turn off the heating mantle to the scrubber and let it cool for 10 to 15 minutes before discontinuing the flow of ice water to the condensers. Disconnect the outlet lines from the elutriator to the scrubber at the elutriator. Samples shall be extracted from the scrubber *expeditiously* to minimize losses to the walls of the glassware and to facilitate cleaning.

To minimize loss, before disconnecting the transfer lines and condensers from the round-bottom flask of the scrubber, pour approximately 100 ml of asbestos-free (filtered, distilled) water down the exit condenser and another 100 ml down the transfer lines and entrance condenser. Such rinsing should be performed in multiple stages, approximately 20 ml at a time. Swirl each condenser (and the transfer lines) as the water drains into the flask. Tap each condenser (and the transfer lines) several times after rinsing to assure a reasonably quantitative transfer.

Detach and remove the round-bottom flask from the condensers and its stand and pour the contents of the flask into a clean, pre-weighed, wide-mouthed 1 L plastic container. Rinse the round bottom flask several times with additional asbestos-free water to assure a quantitative transfer of any residual solids. Reweigh the container and record the net weight as the total weight of suspension. If necessary, the sample may then be stored at ice temperature until it can be prepared. However, preparation shall not be delayed for more than 48 hours.

Immediately prior to filter preparation, add 1.5 g/L of sodium hexametaphosphate to the suspension in the plastic container. Shake the suspension vigorously and divide it

approximately evenly into two (or, if necessary, three) 500 ml Erlenmeyer flasks. Place the flasks on a laboratory shake table for approximately three hours. Quickly re-combine the contents of the two (or three) flasks into a clean, plastic container (with minimal flushing) and place the container in a sonicator. Sonicate the suspension for approximately 1 minute (with the power of the sonicator set at no more than 0.1 W/ml). Withdraw one ml with a disposable pipette from the center of the volume of the suspension in the plastic container and dilute this with asbestos-free water to 100 ml in a clean, volumetric flask.

The mass of respirable dust collected in the scrubber should be equal to the sum of the cumulative mass of dust measured on the filters collected above the ME opening of the elutriator over the entire run(s) during which the scrubber suspension was collected. Use this estimated mass (and account for the 100-fold dilution performed as described in the last paragraph) to estimate the size of aliquots required to produce filterable suspensions containing: 0.5, 2, and 5 µg of respirable material. Dilute each aliquot to a minimum total volume of 20 ml and filter each aliquot in the manner described in Sections 10.34 and 10.35 of the ISO Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993)[28].

The filtered aliquots shall all be prepared as described in Section 10.1 and scanned briefly at low magnification in the TEM to select the optimally loaded specimen for detailed analysis (see Section 11.1.2).

9.5 CLEANING THE DUST GENERATOR

The dust generator is designed for quick and easy assembly and disassembly to facilitate cleaning. Most of the joints are simple friction couplings or ring clamp couplings. To clean the dust generator, disconnect and disassemble the tumbler, remove the bottom cup and dust collector system from the elutriator, decouple the two halves of the elutriator tube, disassemble the slide mechanisms of the dust collector and disconnect the transfer lines to the scrubber. The metal pieces of the dust generator may then be washed with biodegradable detergent, rinsed with asbestos-free water, sonicated briefly, and rinsed again. The pieces may then be left to dry in room air or may be dried with a forced, HEPA-filtered air stream.

The glassware of the scrubber shall also be washed with biodegradable detergent and asbestos-free water, rinsed liberally, and dried in room air or dried with a forced, HEPA-filtered air stream. It is recommend that new transfer lines between the elutriator and the scrubber (constructed of 1.00 in. i.d. Tygon tubing) be cut and installed after each cleaning.

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[28] As indicated in Chatfield (1993), filters to be employed for filtering scrubber suspensions are of a different type than those employed in dust generator mounts. Filters used to filter scrubber suspension are to be the 0.22 µm pore size variety

10.0 PREPARATION OF SPECIMEN GRIDS FOR TEM ANALYSIS

For Superfund applications of this method, asbestos analysis of all samples prepared using the dust generator are to be performed on specimen grids prepared from aliquots of the scrubber suspension. In addition, for a minimum of a subset of 5% (preferably 10%) or 10 samples (whichever is greater), asbestos analysis is also to be performed on specimen grids prepared by a direct transfer technique from filters collected over the IST opening of the elutriator (i.e. the opening over the isokinetic sampling tube). These analyses are then paired with the analyses of asbestos from scrubber suspension collected during the same runs to provide a link between samples prepared by each technique.

The primary reason for preparing 100% of samples from the scrubber suspension is to facilitate identification of distinctions in sample characteristics; samples prepared in this manner are expected to exhibit the best precision among the options for this method. At the same time, when comparing results to published slope factors, the apparent need for normalizing asbestos analyses to counts derived from directly prepared specimens (see, for example, Berman and Crump 1989) is satisfied by providing a subset of samples prepared both ways to allow a regression to be performed linking results from the scrubber suspension samples to specimens prepared by a direct technique. As indicated previously (Section 2.1.3), the recommended procedure is based on a compromise allowing optimum precision for distinguishing among relative measurements (and relative risks) while excepting a small reduction in the precision of estimates of absolute risk.[29]

10.1 PREPARATION OF SPECIMEN GRIDS FROM FILTERED ALIQUOTS OF THE SCRUBBER SUSPENSION

Filters generated from aliquots of the scrubber suspension (as described in Section 9.4.6) shall be prepared using the direct transfer technique that is described in Sections 10.5 of the ISO Method (Chatfield 1993). As indicated previously, multiple aliquots representing a sequence of dilutions are to be prepared to allow selection of the optimally loaded filters (and corresponding set of specimen grids) for final, detailed analysis.

From each filter, prepare a minimum of three specimen grids: one from near the center of the filter, one from a location that is half the distance between the center and the outer edge, and one from near the outer edge of the filter.

10.2 SPECIMEN GRID PREPARATION FROM FILTERS COLLECTED OVER THE IST OPENING OF THE ELUTRIATOR

Although this method specifies that filters collected over the IST opening of the elutriator shall be prepared using a direct preparation technique, an indirect preparation technique is also described, as an option for non-Superfund applications.

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[29] For other applications of this method, options might include preparation of 100% of samples from filters collected over the IST opening of the elutriator and/or preparation of filters collected over this opening using an indirect transfer technique. The method is designed to be flexible.

10.2.1 Specimen Grid Preparation Using a Direct Transfer Technique

Filters collected over the IST opening of the elutriator (as described in Section 9.4.5) shall be prepared using the direct transfer technique that is described in Section 10.5 of the ISO Method (Chatfield 1993). As indicated previously, sections of multiple filters representing a range of loadings are to be prepared to allow selection of the optimally loaded specimen grids for final, detailed analysis.

From each filter that has been collected over the IST opening, prepare four specimen grids from locations on the filter that are each separated by 90° radially. Select two of the locations (from opposing sides of the filter) at points that are about two thirds of the distance from the center to the edge of the filter. The remaining two locations shall be selected at points that are about one third of the distance from the center to the edge of the filter. Such an arrangement will eliminate any effects potentially associated with a linear gradient across the filter that may develop due to the brief time over which the filters are exposed to air flow from the elutriator and, consequently, the potentially significant time during which the filter is being slid in and out of alignment.

10.3 Specimen Grid Preparation Using an Indirect Transfer Technique

As an option to the procedure described in Section 10.2.1 above (for non-Superfund applications *only*), filters collected over the IST opening of the elutriator (as described in Section 9.4.5) may also be prepared using the indirect transfer technique that is described in Sections 10.3 to 10.5 of the ISO Method (Chatfield 1993). For this option, multiple sections of the most highly loaded filter obtained from the dust generator shall be prepared using a range of dilutions to allow selection of the optimally loaded specimen grids for final, detailed analysis.

11.0 PROCEDURES FOR ASBESTOS AND DUST ANALYSIS

11.1 PROCEDURES FOR ASBESTOS ANALYSIS

Specimen grids prepared as described in Chapter 10 are to be analyzed using transmission electron microscopy (TEM). Follow the procedures for analysis described in the ISO Method (Chatfield 1993) including procedures for:

- examining specimen grids to determine acceptability for analysis;
- structure counting by TEM (except determination of the stopping point);
- structure morphological classification;
- structure mineralogical identification; and
- blank and quality control determinations.

The stopping points for the analyses conducted in support of this method are a function of the required sensitivity for the method and are defined in Sections 11.1.1 and 11.1.2 below.

Begin by examining one of each set of specimen grids derived for a defined loading from a particular run of the dust generator and select the optimally loaded set for analysis. Use the criteria for determining the acceptability of specimen grids (from the ISO Method) to define optimal loading.

When performing detailed analysis, be sure to distribute asbestos counts evenly over the entire set of specimen grids prepared from a particular filter at a defined (optimal) loading. Record the morphology and mineral type of asbestos structures as described in the ISO Method (Chatfield 1993). Also as described in the ISO Method, complete separate scans for counts of total structures and, at lower magnification, for counts of structures longer than 5 µm.

11.1.1 Analysis of Specimen Grids Prepared from Filters Collected Over the IST Opening of the Elutriator

Prior to initiating a detailed analysis of specimen grids, the stopping rules for the analysis must be defined. Assuming these specimen grids have been prepared using a direct transfer technique (as discussed in Section 10.2), define the stopping rules for the detailed analysis as follows.

First, calculate the maximum number of grid openings that will have to be scanned during the analysis from the relationship:

$$N_{go} = S_d * A_f * \%RD / (S_{smpl} * A_{go} * 100 * \Delta M_f) \qquad (11\text{-}1)$$

where:

N_{go} is the maximum number of grid openings to be scanned;

S_d is the number of structures required to define detection using the analysis (defined here as 1);

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9;

S_{smpl} is the required analytical sensitivity for the method (s/g);

A_{go} is the area of a single grid opening (mm^2); and

ΔM_f is the mass of respirable dust collected on the filter from which the specimen grids were prepared. It is defined using Equation A-7 of Section A.2.2 of Appendix A (g).

The following are typical values for the above parameters:

S_d = 1;
S_{smpl} = 5×10^7 (for total structures) or
= 3×10^6 (for structures longer than 5 µm)[30];
A_f = 382 mm^2;
A_{go} = 8.1×10^{-3} mm^2;
%RD = between 0.5% and 2%[31]; and
ΔM_f = 5 µg [32];

Given the above values, it is estimated using Equation 11-1 that between 1 and 4 grid openings will typically need to be scanned to derive a count of total structures. However, a minimum of 4 grid openings shall be scanned during any analysis. Based on the above, similarly, between 15 and 62 grid openings will typically need to be scanned to derive a count of structures longer than 5 µm.

The number of grid openings to be scanned for specific analyses shall be determined by substituting case-specific values for the above listed parameters into Equation 11-1.

Stop the counting, characterization, identification, and recording of asbestos structures on a particular analysis when one of the following obtains:

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30 See Section 2.1.1.

31 This is the range of values observed for a diverse variety of samples tested during the pilot study for this method (Berman et al. 1994a).

32 See Sections 9.4.5 and A.2.2 of Appendix A.

- the scan is completed for the grid opening on which the 50th asbestos structure is counted; or

- either 4 grid openings or the maximum number of grid openings (estimated as defined above), whichever is greater, are scanned completely.

These rules are to be applied separately to the scan for total structures and the scan for long structures (i.e. longer than 5 μm) that are described in the ISO Method.

11.1.2 Analysis of Specimen Grids Prepared from Filtered Scrubber Suspension

Prior to initiating a detailed analysis of specimen grids, the stopping rules for the analysis must also be defined in this case. Given that these specimen grids have been prepared as described in Section 10.1, define the stopping rules for the detailed analysis as follows.

First, calculate the maximum number of grid openings that will have to be scanned during the analysis from the relationship:

$$N_{go} = S_d * A_f * \%RD * DF / (S_{smpl} * A_{go} * 100 * M_{scrbr}) \quad (11\text{-}2)$$

where:

N_{go} is the maximum number of grid openings to be scanned;

S_d is the number of structures required to define detection using the analysis (defined here as 1);

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9;

S_{smpl} is the required analytical sensitivity for the method (s/g);

A_{go} is the area of a single grid opening (mm^2);

DF is the dilution factor by which the scrubber suspension had to be diluted to prepare specimen grids that are suitably loaded for analysis; and

M_{scrbr} is the mass of respirable dust collected in the scrubber suspension during the run.

The dilution factor, DF, is simply the product of the individual dilution factors for the two sequential dilutions performed to derive the final volume that is ultimately filtered (per the procedure defined in Section 9.4.6):

$$DF = (V_s/V_{a1})*(V_d/V_{a2}) \quad (11\text{-}3)$$

where:

V_s is the volume of the initial scrubber suspension (which is estimated from the recorded weight of the supernatant assuming a density of 1 g/cm^3) (ml);

V_{a1} is the volume of the first aliquot collected from the scrubber suspension for further preparation (the default defined in Section 9.4.6 is 1 ml);

V_d is the volume into which the first aliquot from the suspension is diluted (the default defined in Section 9.4.6 is 100 ml); and

V_{a2} is the volume of the final aliquot collected from V_d that is ultimately filtered for preparation of the optimally loaded specimen grids (ml).

The mass of respirable dust collected in the scrubber suspension, M_{scrbr}, is derived from the cumulative mass of dust collected during the run on filters that are mounted over the ME opening of the elutriator (defined below as M_f). These differ by the ratio of the air flow into the filters and the air flow into the scrubber:

$$M_{scrbr} = M_f*(F_s/F_c) \quad (11\text{-}4)$$

where:

M_{scrbr} is the mass of respirable dust collected in the scrubber during the run (g);

M_f is the cumulative mass of dust collected on the filters (mounted over the ME opening of the elutriator) during the same run (calculated from Equation A-4 of Section A.2.1 of Appendix A);

F_s is the volumetric air flow rate into the scrubber (cm^3/s); and

F_c is the volumetric air flow rate into the filters over the ME opening of the elutriator (cm^3/s).

Assuming air flow in the elutriator is setup as described in Section 9.3.4, F_s and F_f are equal so that M_{scrbr} simply equals M_f.

Given the typical values for the corresponding parameters provided in Section 11.1.1, assuming a typical value for DF [33] of between 2 x 10^4 and 4 x 10^4, and selecting a range of typical values for M_f (i.e. between 0.1 and 0.2 g) from among the range observed during the pilot study (Berman et al. 1994a), it is estimated using Equation 11-2 that between 1 and 8 grid openings will need to be scanned to derive counts of total structures. Similarly, it is estimated that between 8 and 133 grid openings will need to be scanned to derive counts of long structures (i.e. longer than 5 µm).

[33] This assumes typical values for M_f (between 0.1 and 0.2 g) from among the range of values observed among diverse samples during the pilot study (Berman and Kolk 1994) and further assumes that the dilution factor is selected so as to produce a loading of 5 µg on the filter.

Determine the actual number of grid openings required for a specific analysis by substituting case-specific values for the above parameters into Equations 11-2, 11-3, and 11-4.

Stop the counting, characterization, identification, and recording of asbestos structures on a particular analysis when one of the following obtains:

- the scan is completed for the grid opening on which the 50th asbestos structure is counted; or
- either 4 grid openings or the maximum number of grid openings (estimated as defined above), whichever is greater, are scanned completely.

These rules are to be applied separately to the scan for total structures and the scan for long structures (i.e. longer than 5 µm) that are described in the ISO Method (Chatfield 1993).

11.2 EVALUATING THE RATE OF RELEASE OF RESPIRABLE DUST

The rate of release of respirable dust from a sample prepared using the dust generator is estimated from measurements of the mass of dust collected over time on the set of filters mounted over the ME opening of the elutriator. The measurements used specifically are from those filters that are collected while the tumbler is operating at the *highest* rotation rate employed for the sample (see Section A.2.1 of Appendix A).

Begin by plotting the cumulative mass collected on the filters as a function of time. To derive the cumulative mass for a particular time interval, add the mass of dust measured on the filter collected from that time interval to the sum of the masses measured on the set of filters collected earlier in the run. Typical curves are depicted in Figures 11-1 and 11-2. Next, calculate the cumulative mass released from the sample over time from the cumulative mass collected on filters over time using the relationship developed in Section A.2.1 of Appendix A:

$$M_r = 2.1 * M_f \qquad (11\text{-}5)$$

where:

M_r is the cumulative mass of dust released from a sample between the start of a run and time "t" (g); and

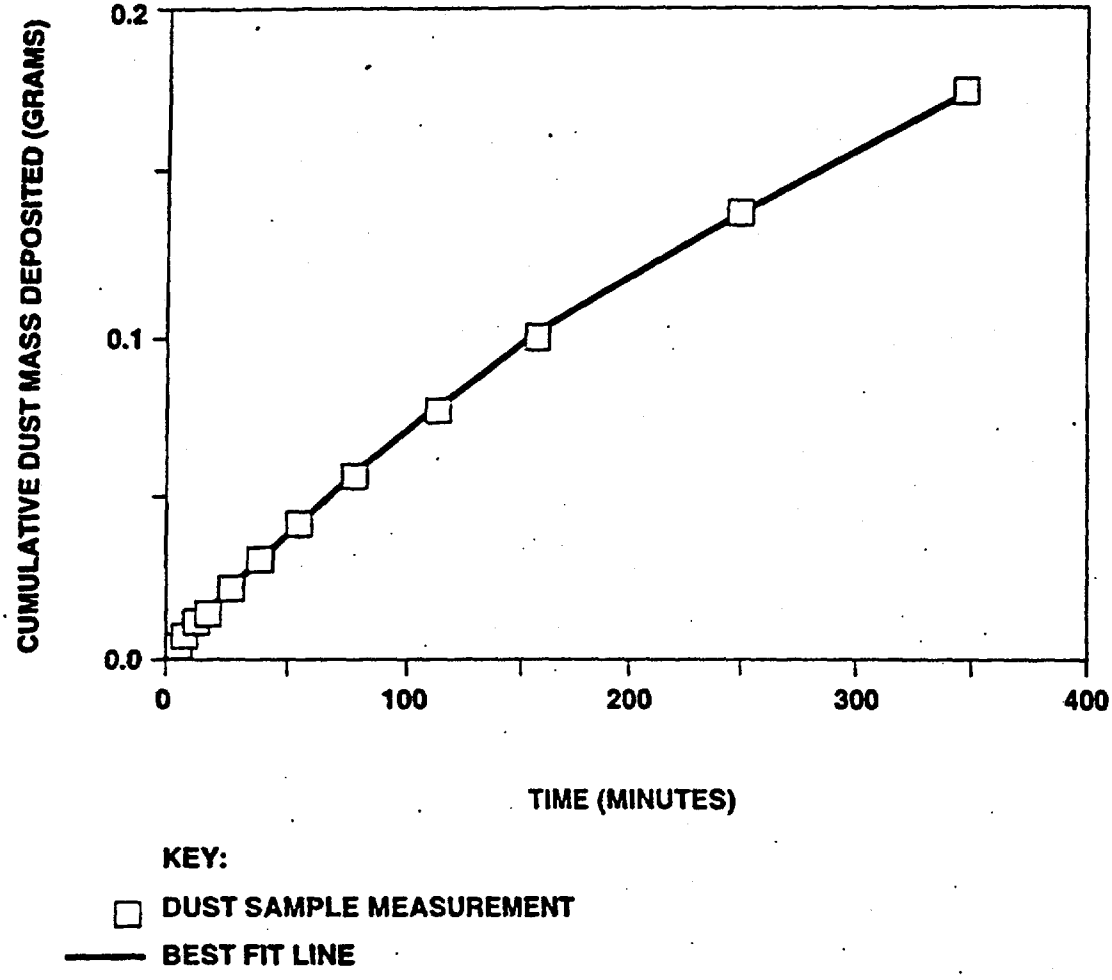
M_f is the cumulative mass collected on filters[34] between the start of a run and time "t" (g).

Equation 11-5 is appropriate to use to relate the mass of dust collected on filters to the mass released from the sample when air flow in the dust generator is setup as indicated in Section 9.3.4. If different air flow conditions are established for a particular experiment, the relationship between M_r and M_f will have to be derived using Equation A-4 from Appendix A.

[34] These are the filters that are mounted over the ME opening of the elutriator.

FIGURE 11-1

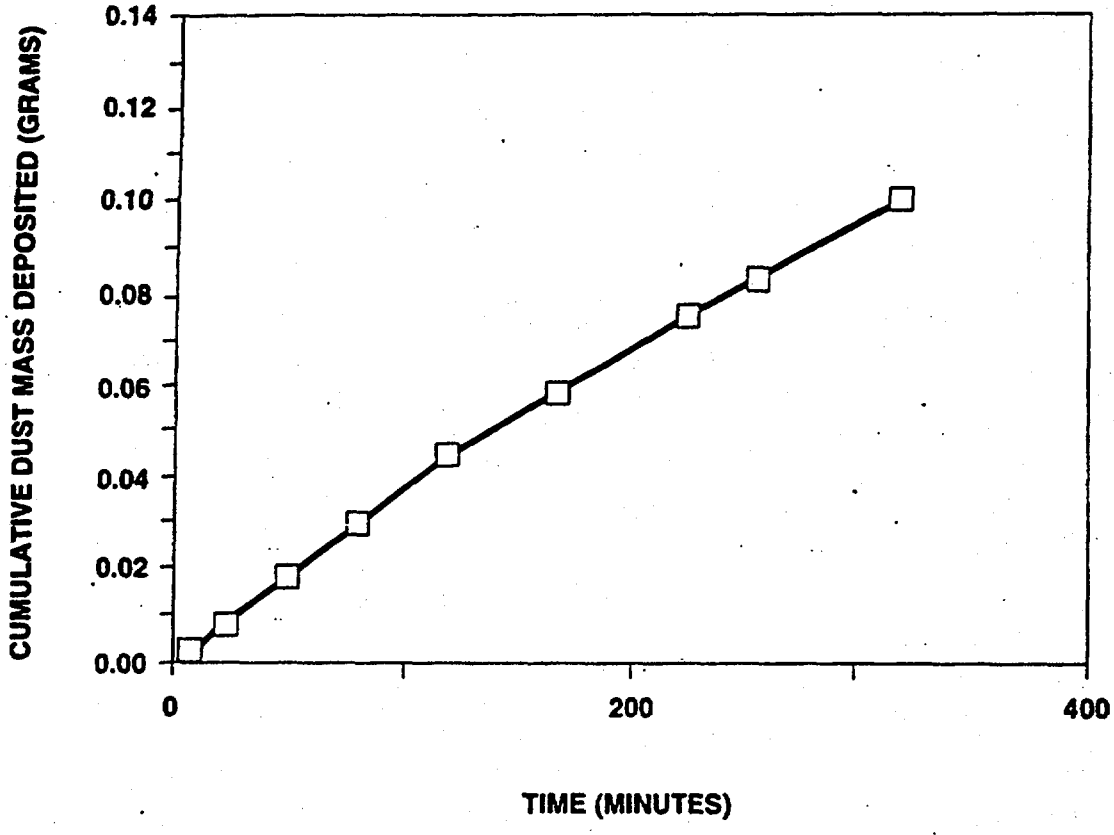
TYPICAL CUMULATIVE MASS RELEASE VERSUS TIME CURVE FOR 30 RPM RUN[a]



[a]FROM THE 30-RPM RUN ON SAMPLE 1-100, WHICH WAS COMPLETED ON 8/27/93 DURING THE PILOT STUDY (BERMAN ET. AL. 1994a).

FIGURE 11-2

TYPICAL CUMULATIVE MASS RELEASE VERSUS TIME CURVE FOR 60 RPM RUN[a]



KEY:

□ **DUST SAMPLE MEASUREMENT**

—— **BEST FIT LINE**

[a]FROM THE 60-RPM RUN ON SAMPLE 1-100, WHICH WAS COMPLETED ON 8/27/93 DURING THE PILOT STUDY (BERMAN ET. AL. 1994a).

The total mass of dust in the sample at the beginning of the run must next be estimated using the relationship developed in Appendix A. Based on the relationship (see Section A.2.1):

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad (11\text{-}6)$$

where:

M_o is the mass of dust in the sample at the start of the run (g);

k is the first-order rate constant for the release of dust from the sample (s^{-1}); and

t is the time since the start of the run (s);

a plot of $\ln(M_o - M_r)$ versus t should be a straight line with a slope equal to the rate constant for the release of dust from the sample and an intercept equal to the natural logarithm of the mass of dust in the sample at the start of the run. Derive estimates of "M_o" and "k" by programming Equation 11-6 into a spreadsheet and running a regression[35].

Input a range of guesses for the value of M_o into the spreadsheet and run a regression to fit a value for k and to calculate a value for the regression coefficient, "r^2" for each value of M_o. Plot the regression coefficient, "r^2" as a function of M_o. An example of such a plot is presented in Figure 11-3. The value of M_o that provides the fit with the largest regression coefficient (i.e. with r^2 closest to 1) shall be reported as the correct value for the mass of dust in the sample at the start of the run and shall be reported with the corresponding k value as the estimated rate constant for dust release from the sample during the run.

11.3 DETERMINING THE CONTENT OF RESPIRABLE DUST

To determine the mass percent of respirable dust in the original sample, first determine the total mass of respirable dust in the sample *at the start of a run* for the last run completed on the sample, which is derived as described in the last section.

Because the dust generator run analyzed as described in Section 11.2 will generally have been preceded by a run with the tumbler speed set at 30 rpm (see Section 9.4.4), to estimate the total mass of dust present in the sample, it is necessary to include the mass released during this first run.

Sum the masses of dust measured on each of the filters collected during the 30 rpm run and designate this sum, "M_{f30}", which is the cumulative mass of dust collected during the 30 rpm run. Using the following equation, estimate the total mass of dust *released* from the sample during the 30 rpm run, "M_{r30}," based on the mass of dust *collected* during that run (see Section A.2.1 of Appendix A):

$$M_{r30} = 2.08 * M_{f30} \qquad (11\text{-}7)$$

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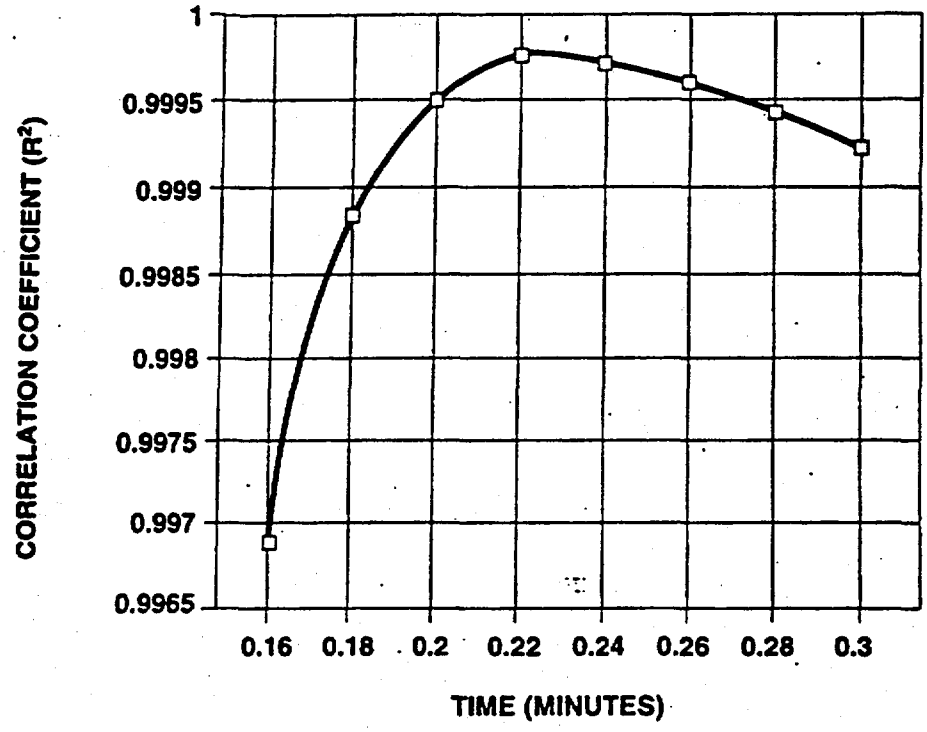
35 Any of several commercial spreadsheet programs (including, for example, LOTUS™) contain the necessary capabilities and may be employed to derive optimum values for "M_o" and "k."

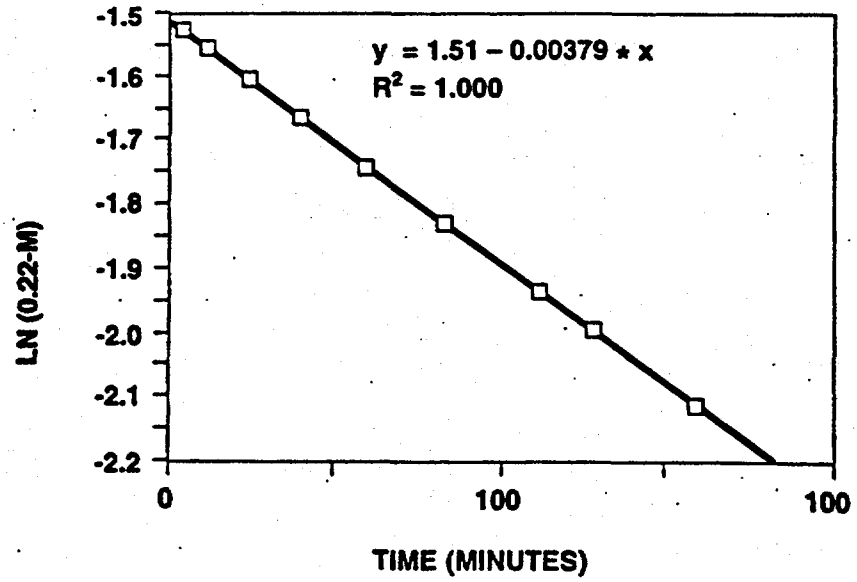
FIGURE 11-3

ILLUSTRATION OF THE OPTIMIZATION
OF THE ESTIMATE OF INITIAL MASS "M_0"

A. TYPICAL PLOT OF CORRELATION COEFFICIENT VERSUS INTITIAL MASS FOR A DUST GENERATOR RUN[a]



B. TYPICAL PLOT OF THE OPTIMIZED FIRST ORDER RATE EQUATION FOR A DUST GENERATOR RUN[a]



[a]FROM THE 60-RPM RUN ON SAMPLE 1-100, WHICH WAS COMPLETED ON 8/27/93 DURING THE PILOT STUDY (BERMAN ET. AL. 1994a).

Calculate the total mass of dust originally present in the sample, M_{tot}, by summing the mass released during the 30 rpm run with the mass of dust estimated to have resided in the sample at the beginning of the higher rpm run, M_o (this is equal to the mass of dust remaining in the sample at the end of the 30 rpm run). M_o will have been derived as described in Section 11.2:

$$M_{tot} = M_{r30} + M_o \qquad (11\text{-}8).$$

Estimate the mass percent of respirable dust in a sample as follows:

$$\%RD = 100 * M_{tot}/M_{sample} \qquad (11\text{-}9)$$

where:

%RD is the mass percent of respirable dust in the sample (%); and

M_{sample} is the mass of the original sample placed in the tumbler (g).

11.4 DETERMINING THE CONTENT OF ASBESTOS

The concentration of asbestos in a sample is determined differently depending on whether asbestos is determined from sampling grids prepared from filters collected over the IST opening of the elutriator or from scrubber suspension.

NOTE

For samples originally containing a significant fraction of coarse material (see Section 8.4), the concentration of asbestos reported as a function of the mass of the sample (specified in this section) must be adjusted for the quantity of coarse material originally measured in the matrix sampled in the field before it can be considered representative of that matrix. A procedure for adjusting asbestos concentrations to account for the coarse fraction of an environmental matrix is presented in Section 11.4.3.

11.4.1 Based on Directly Prepared Filters Collected Over the IST Opening of the Elutriator

Procedures for determining the concentrations of asbestos structures in a sample differ slightly depending on whether the structures of interest are longer or shorter than 5 µm.

Structures that are shorter than 5 µm in length are derived *only* from the high magnification scan of an analysis. Calculate and report the concentration of short asbestos in the original sample based on the counts of asbestos structures that are derived as defined in Section 11.1.1 using the following relationship:

$$C_{smpl} = S_{ch}*A_f*\%RD/(N_{goh}*A_{go}*100*\Delta M_f) \quad (11\text{-}10)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{ch} is the number of structures (of the defined size range or type of interest) counted during the high magnification scan of the analysis;

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9 (%);

N_{goh} is the number of grid openings scanned during the high magnification scan of the analysis;

A_{go} is the area of a single grid opening (mm^2); and

ΔM_f is the mass of respirable dust collected on the filter from which the specimen grids were prepared. It is defined using Equation A-7 (see Section A.2.2 of Appendix A) (g).

Structures that are longer than 5 µm in length are derived from combined counts collected during both the high and low magnification scans of an analysis. Calculate and report the concentration of long asbestos in the original sample based on the counts of asbestos structures that are derived as defined in Section 11.1.1 using the following relationship:

$$C_{smpl} = (S_{ch} + S_{cl})*A_f*\%RD/[(N_{goh} + N_{gol})*A_{go}*100*\Delta M_f] \quad (11\text{-}11)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{cl} is the number of structures (of the defined size range or type of interest) counted during the low magnification scan of the analysis; and

N_{gol} is the number of grid openings scanned during the low magnification scan of the analysis.

Thus, for long structures, it is the total counts of structures observed over both the high and low magnification scans and the total area scanned (over both the low and high magnification scans) that are used to determine concentration.

As an option, asbestos concentrations may also be reported as a function of the mass of respirable dust in a sample using the following relationships. For short structures, use:

$$C_{dust} = S_{ch}*A_f/(N_{goh}*A_{go}*\Delta M_f) \quad (11\text{-}12)$$

where:

C_{dust} is the concentration of asbestos structures (of any defined size range or type) in the respirable dust of the sample (s/g_{dust}); and all other parameters are defined as described above.

Similarly, for long structures, use:

$$C_{dust} = (S_{ch} + S_{cl})*A_f/[(N_{goh} + N_{gol})*A_{go}*\Delta M_f] \quad (11\text{-}13)$$

11.4.2 Based on Specimens Prepared from Scrubber Water

As described in Section 11.4.1, procedures for determining the concentrations of asbestos structures in a sample differ slightly depending on whether the structures of interest are longer or shorter than 5 µm.

Structures that are shorter than 5 µm in length are derived only from the high magnification scan of an analysis. Calculate and report the concentration of short asbestos in the original sample based on the counts of short asbestos structures that are derived from the scrubber suspension as defined in Section 11.1.2 using the following relationship:

$$C_{smpl} = S_{ch}*A_f*\%RD*DF/(N_{goh}*A_{go}*100*M_{scrbr}) \quad (11\text{-}14)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{ch} is the number of structures (of the defined size range or type of interest) counted during the high magnification scan of the analysis;

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9 (%);

N_{goh} is the number of grid openings scanned during the high magnification scan of the analysis;

A_{go} is the area of a single grid opening (mm^2);

DF is the dilution factor by which the scrubber suspension had to be diluted to prepare specimen grids for analysis (derived as defined in Equation 11-3); and

M_{scrbr} is the mass of respirable dust collected in the scrubber suspension during the run (derived as defined in Equation 11-4).

Structures that are longer than 5 µm in length are derived from combined counts collected during both the high and low magnification scans of an analysis. Calculate and report the concentration of long asbestos in the original sample based on the counts of long asbestos structures that are derived from scrubber suspension as defined in Section 11.1.2 using the following relationship:

$$C_{smpl} = (S_{ch} + S_{cl})*A_f*\%RD*DF/[(N_{goh} + N_{gol})*A_{go}*100*M_{scrbr}] \quad (11\text{-}15)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{cl} is the number of structures (of the defined size range or type of interest) counted during the low magnification scan of the analysis; and

N_{gol} is the number of grid openings scanned during the low magnification scan of the analysis.

As an option for analysis of the scrubber suspension, asbestos concentrations may also be reported as a function of the mass of respirable dust in a sample using the following relationships. For short structures, use:

$$C_{dust} = S_{ch}*A_f*DF/(N_{goh}*A_{go}*M_{scrbr}) \quad (11\text{-}16)$$

where:

C_{dust} is the concentration of asbestos structures (of any defined size range or type) in the respirable dust of the sample (s/g_{dust}); and all other parameters are defined as described above.

Similarly, for long structures, use:

$$C_{dust} = (S_{ch} + S_{cl})*A_f*DF/[(N_{goh} + N_{gol})*A_{go}*M_{scrbr}] \quad (11\text{-}17)$$

11.4.3 Procedure for Adjusting Asbestos Concentrations to Account for the Presence of Coarse Material in the Sampled Matrix

As indicated in Section 8.4, due to the need to incorporate field data into this calculation, a formal protocol designating who is to perform this calculation and how that individual is to obtain the needed field information must be defined at the start of a study using this method.

To provide a representative measure of the concentration of asbestos in an environmental matrix in which significant coarse material is found (i.e. more than 10% by weight), first derive an appropriate coarseness adjustment factor:

$$CF = M_{fine}/(M_{fine} + M_{coarse}) \qquad (11\text{-}18)$$

where:

- CF is the coarseness adjustment factor for a particular sample (dimensionless);
- M_{fine} is the mass of fine material measured immediately after sieving the sample in the field (g); and
- M_{coarse} is the mass of coarse material measured immediately after sieving the sample in the field (g).

Then, to determine the concentration of asbestos in the environmental matrix that was sampled, perform the following adjustment:

$$C_{mtrx} = CF*C_{smpl} \qquad (11\text{-}19)$$

where:

- CF is the coarseness adjustment factor for a particular sample (derived as defined above);
- C_{mtrx} is the concentration of asbestos structures (of a defined size range or type) in the field matrix sampled (s/g); and
- C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the sample sent to the laboratory (s/g). This is determined as described in Sections 11.4.1 and 11.4.2 above.

12.0 PERFORMANCE CHARACTERISTICS AND QUALITY CONTROL/QUALITY ASSURANCE REQUIREMENTS

12.1 METHOD PERFORMANCE

The method defined in this document achieves the target performance requirements defined in Section 2.1 at a cost that should be competitive with other procedures that might be designed to produce comparable information (Section 2.4).

12.1.1 Analytical Sensitivity

As indicated in Section 11.1, depending on sample characteristics, between 1 and 8 grid openings will likely have to be scanned at high magnification (20,000x) to achieve the target analytical sensitivity for total asbestos structures of $5x10^7$ s/g_{solid}. Similarly, between 15 and 133 grid openings will likely have to be scanned at low magnification (10,000x) to achieve the target analytical sensitivity for long asbestos structures (longer than 5 µm) of $3x10^6$ s/g_{solid}.

The defined sensitivities for total and long structures were easily achieved during the pilot study for the method (Berman et al 1994a).

12.1.2 Precision

Results of the pilot study for this method (Berman et al 1994a) indicate that, when 50 structures are counted, the average relative percent difference observed among eight sets of duplicate samples (four sets of duplicate samples analyzed by each of two laboratories) is 20%.

The precision of an asbestos measurement (in this case, expressed as the relative percent difference) should be inversely proportional to the square root of the number of structures counted. Given that 10 structures are likely to be counted at the target concentrations for this method (see Section 2.1.1) and, based on the precision observed in the pilot study when 50 structures are counted, it is expected that the average relative percent difference achievable for this method should be 43% at the target concentrations listed in Section 2.1.1. Thus, given the analytical sensitivities defined in Section 2.1.1 and the stopping rules defined in Section 11.1, this method is capable of achieving a level of precision that is comparable with the guidelines recommended in the CLP for the analysis of other analytes in soils and defined for this method in Section 2.1.2.

The precision observed during the pilot study for this method is based on specimen grids prepared from the suspension collected in the scrubber of the elutriator (Appendix A). It is expected that the precision achieved for analyses derived from scans of specimen grids derived by direct preparation of filters collected over the isokinetic sampling tube of the dust generator (see Appendix A) will be significantly worse. Procedures are therefore incorporated into this method in which the majority of samples will be analyzed based on preparation of specimen grids from filtered scrubber suspension with a subset of 5 to 10% simultaneously analyzed using specimen grids prepared by a direct transfer technique from filters collected over the isokinetic sampling tube of the elutriator (see Chapter 10). The latter is required to develop a regression relating these two types of preparations so that measurements can be evaluated using existing dose-response factors for asbestos (see Section 2.1.3); existing

dose-response factors tend to be based on the analysis of samples whose preparation corresponds most closely to a direct preparation.

NOTE

It is anticipated that a direct, linear relationship will be observed between asbestos concentrations derived, respectively, from scrubber suspension and from filters collected over the IST opening of the elutriator for samples collected from the same environmental matrix. However, there is little reason to expect that such a relationship will hold across samples collected from different environmental matrices. Therefore, samples to be employed to determine the relationship between asbestos measured from scrubber suspension and from filters mounted over the IST opening of the elutriator should be selected and evaluated separately for each environmental matrix sampled during a study for analysis using this method.

It must be recognized that compromises are required when developing a method of this type. The proposed reporting of samples prepared primarily by an indirect technique with a small subset of directly prepared samples (the latter used primarily to provide a link between the reported measurements and existing dose-response factors) represents such a compromise.

The proposed procedure allows for the determination of *relative* concentrations with maximum precision so that arbitrarily small differences in concentrations can be easily distinguished. Among other things, this will facilitate distinguishing upwind and downwind concentrations by a source. At the same time, somewhat lower precision is considered acceptable for estimating the *absolute* risk associated with any particular measurement.

NOTE

The uncertainty of the proposed regression to link results from directly and indirectly prepared samples is expected to be attributable almost exclusively to the limited precision of the directly prepared samples. Therefore, use of the proposed regression for estimating risks may not significantly increase the uncertainty of such a risk analysis beyond what would be associated with assigning an estimated risk to an asbestos concentration derived from a directly prepared sample in any case.

It is also assumed that a sufficient number of samples (i.e. a minimum of approximately 10) will be co-prepared and analyzed to allow reasonable confidence in the results of the regression.

12.1.3 Accuracy

Based on the results of the pilot study (Berman et al. 1994a), there appears to be a problem with laboratory bias during the counting of asbestos structures in support of this method. The bias is introduced specifically during the analysis of specimen grids because duplicate samples prepared by the same laboratory using identical procedures nonetheless yield significantly different counts by analysts from each of two laboratories.

Given that analysts within a single laboratory appear to be capable of achieving good agreement on counting (also demonstrated in the pilot study), the problem of between-laboratory bias will need to be addressed by the implementation of an aggressive inter-laboratory quality control (QC) program in which samples are regularly shared among laboratories and a procedure for verified counting is instituted. It may also be useful to promote meetings with discussion sessions in which analysts from different laboratories discuss the interpretation of structures viewed simultaneously (either directly from a microscope or from a video).

12.1.4 Asbestos Characteristics

As was observed during the pilot study (Berman et al. 1994a), this method is easily capable of preserving information concerning the distribution of the sizes and shapes of asbestos structures that are likely to be released from environmental matrices that are disturbed by natural or anthropogenic forces. As indicated in Section 2.1.3, such information is critical to evaluating the potential health effects of the asbestos dusts generated by releases from asbestos-containing matrices (see, for example, Berman and Crump 1989).

Interferences and limitations concerning the ability to identify and characterize asbestos structures using the counting and identification rules of the ISO Method, as adopted for this method (Section 11.1), are described in the ISO Method (Chatfield 1993).

12.1.5 Reporting Requirements

As indicated in Section 11.4, the concentrations of asbestos structures (of any defined size or type) that are measured using this method can be easily reported either as a function of the mass of the sampled material or as a function of the mass of respirable dust in the sampled material. This should allow sufficient flexibility to facilitate use of results from this method in concert with any of the fate and transport models that may be employed to predict exposure (Section 2.1.4).

12.2 QUALITY CONTROL REQUIREMENTS

The quality assurance/quality control (QA/QC) requirements indicated in the ISO Method (Chatfield 1993) shall be considered relevant and appropriate when using this method. In addition, the following blank and duplicate/replicate schedule shall be employed when running samples using this method.

12.2.1 Blanks

The following blanks shall be collected routinely in concert with use of this method:

- lot blanks or filter blanks. Two filters from each lot of 50 filters obtained from the manufacturer shall be prepared using a direct transfer procedure and analyzed to assure that background contamination on the filters does not exceed 10s/mm^2 (Section 6.6). Only filters from lots whose blanks pass the defined criterion shall be used in support of this method;

- laboratory blanks. A sufficient number of laboratory blanks shall be collected, prepared using a direct transfer technique and analyzed to show that the room in

which bulk samples are handled and prepared satisfy the requirements defined in Section 10.6 of Chatfield and Berman (1990). When laboratory blanks indicate that room air is out of compliance with the stated criterion, use of this method is to cease until appropriate corrective actions are completed;

- field blanks. Field blanks shall be collected during any sample collection activities performed in association with use of this method. The number of such blanks to be collected and the schedule for their analysis shall be determined based on the complexity of the anticipated sampling scheme and shall be defined as part of the sampling plan for the site. QC criteria for field blanks will also be set as part of the planning for the study;

- equipment blanks. Equipment blanks are collected at the beginning of each run of the dust generator, as described in Section 9.4.3. Equipment blanks do not generally need to be analyzed on a regular basis but shall be stored in case their use is required to help determine the source of contamination that may be discovered by some other means;

- run blanks. Run blanks are also collected at the beginning of each run of the dust generator, as described in Section 9.4.3. One run blank shall be analyzed routinely for the first run completed on any particular sample. The remaining run blanks generated in association with a particular sample shall be stored in case their use is required to help determine the source of contamination that may be discovered by some other means.

  Should asbestos structure counts on run blanks exceed the target criterion of 10 s/mm^2, use of this method shall cease until appropriate corrective actions have been completed and new run blanks are shown to achieve the stated criterion; and

- scrubber blanks. Scrubber blanks shall be derived by collecting a 1 ml aliquot of scrubber liquid (after the scrubber is loaded and assembled for a run but before heating of the scrubber is initiated), diluting the aliquot to 10 ml, and completing the preparation of the diluted aliquot as described in Section 9.4.6. Scrubber blanks do not need to be analyzed routinely but shall be stored in

  case their use is required to help determine the source of contamination that may be discovered by some other means.

In addition to the above listed blanks that must be collected routinely in association with use of this method, the following may prove helpful for identifying the source of any contamination that might be detected in association with use of the dust generator:

- modified run and scrubber blanks. Modified run and scrubber blanks may be generated by setting up and operating a clean dust generator *without* sample. Filters may be collected from either the ME or the IST opening of the dust generator at any point of such a run. Similarly, aliquots of scrubber liquid may be withdrawn from the scrubber at any point. Such blanks may prove useful for determining whether contamination is being introduced by any of the components of an operating dust generator (including, for example, the constant

humidity solution, the rotating tumbler, the elutriator tube, the air transfer lines, and/or the glassware or liquid of the boiling scrubber); and

- post-run scrubber blanks. A post-run scrubber blank may be generated by repeating the rinse of the transfer lines, condensers, and scrubber flask immediately after the quantitative rinse conducted for a particular sample (see Section 9.4.6). The resulting liquid must then be weighed, diluted quantitatively, and prepared in the same manner as described for the scrubber suspension of a sample (Section 9.4.6). The normalized concentration of asbestos structures found in such a blank shall represent no more than 10% of the concentration of asbestos structures observed in the sample prepared immediately prior to collecting the post-run blank. Higher blank concentrations shall be considered unacceptable. If blank concentrations are observed, procedures for quantitative rinsing shall be reviewed, modified, and tested until losses can be shown to be acceptable.

NOTE

Asbestos observed in post-run blanks constitute asbestos that is lost from a sample during preparation.

12.2.2 Duplicates/Replicates

A fixed fraction (5 to 20%) of the samples collected in the field in support of this method shall be collected as spatial duplicates (two samples collected at immediately adjacent locations). These shall be labeled and sent to the laboratory in such a manner so as to assure that laboratory personnel cannot identify them as duplicates. The frequency of collection of spatial duplicates shall be defined as part of the sampling plan for the site. Comparison of the results of the analysis of such samples provides a measure of all of the components of total precision except population variability.

As indicated previously (Section 8.2.1), 100% of the samples shipped from the field are to be shipped as duplicate pairs. The laboratory shall randomly select 10% of the duplicate samples shipped from the field and shall analyze both samples of the pairs so selected. Comparison of the results of the analysis of such samples, which are homogenized splits of the same sample, provides an indication of the precision achieved by sample preparation and analysis.

Should analysis of duplicate pairs indicate an unacceptable degree of variability (i.e. a relative percent difference greater than 50%), replicate counts shall be performed on designated samples by multiple analysts in the laboratory (or by the same analyst on different days). Laboratory management shall assign such counts so as to assure that analysts cannot determine which counts are replicates. Results of such replicate counts shall serve to distinguish whether the major source of variability observed among duplicate pairs is due to analysis or to sample preparation. Appropriate corrective actions may then be devised.

13.0 REPORTING REQUIREMENTS

13.1 FIELD AND LABORATORY NOTEBOOKS

Over the course of the project, information critical to the proper reporting and interpretation of each sample analysis will be developed both in the field and in the laboratory. Formal procedures are required to preserve such information and to allow for the documentation of attendant information that, while not employed directly in the calculation of results, may provide insight into the interpretation of such results.

13.1.1 Field Notebooks

During sample collection, detailed notes will be kept in a standard field or laboratory notebook that is bound and pre-paginated. All entries to the notebook are to be dated and initialed. Information to be recorded during sample collection should include (but is not limited to):

(a) reference to this method;

(b) the project title, identification of the site, and the names and titles of field personnel participating in the sampling effort;

(c) identification of each sample or fraction of each sample handled in the field;

(d) the date and time that each sample was collected and the time interval during which each sample was prepared in the field;

(e) the location from which each sample was collected and the manner in which sample locations were selected (including reference to the sampling plan under which sampling was conducted);

(f) a general description of the physical appearance of the materials sampled;

(g) the types of equipment and the procedures employed to collect each sample;

(h) a summary of the procedure employed for field preparation of each sample (including, for example, the number of passes through a riffle splitter employed for homogenization, and the number of splits required to reduce the sample to the size required);

(i) the relevant identities and weights of each fraction of sample handled during field preparation (including specifically the weights of the coarse and fine fractions separated from each sample during sieving);

(j) other relevant field observations (including, for example, the meteorological conditions under which sampling was conducted); and

(k) the identification, weight, and intended destination of each sample shipped from the field.

13.1.2 Laboratory Notebooks

During sample handling, preparation, and analysis, detailed notes will be kept in a laboratory notebook that is bound and pre-paginated. All entries to the notebook are to be dated and initialed. Information to be recorded during sample preparation and analysis should include (but is not limited to):

(a) reference to this method;

(b) identification of each sample or fraction of each sample received from the field and the date and time that each sample was received;

(c) identification of all laboratory personnel who participate in the preparation and analysis of samples and the specific operations performed by each;

(d) identification of reagents, equipment, and supplies employed during sample handling, preparation, and analysis;

(e) relevant material weights and/or volumes;

(f) the lot number and manufacturer of the filters employed during sample preparation;

(g) a summary of the procedure employed for laboratory preparation of each sample (including, for example, the number of passes through a riffle splitter employed for homogenization and the number of splits required to reduce the sample to the size required);

(h) the relevant identities and weights of each fraction of sample handled during laboratory preparation;

(i) the setup conditions employed for the dust generator (including, for example, the identity of the salt employed for humidity control, air flow conditions, and the setting on the variable voltage transformer employed for the scrubber);

(j) the date and starting time, description (including, primarily, the tumbler rotational speed), and sample identification for any run conducted on the dust generator;

(k) the weight and identity of the sub-sample placed in the dust generator;

(l) the identities, time intervals of collection, the starting and ending weights, and the net weights of filters collected during a run on the dust generator that are to be used to determine the rate of dust generation;

(m) the figures and calculations employed to determine the rate of dust generation and the quantity of dust in the sample;

(n) the identity, time interval of collection, starting and ending weights, and the net weights of filters collected during a run on the dust generator that are to be used to generate specimen grids for asbestos analysis;

(o) the identity, time interval of collection, the weight/volume, dilution factor, aliquot identification, and filter identification for the collection, handling, and filtering of scrubber suspension;

(p) the identity of all filter sections used for preparation of specimen grids and the identity of the specimen grids (include a description of the sector and radial distance from the center of the filter represented by each section);

(q) energy levels and settings for instruments employed for analysis;

(r) flow rates, pressures, temperatures, and other relevant physical parameters that potentially impact procedures;

(s) room conditions (i.e. temperature, relative humidity, and ventilation rates) prevailing during sample preparation;

(t) other relevant observations (including, for example, any difficulties encountered during preparation and analysis and any procedural changes incorporated into the method that are necessitated by such difficulties);

(u) documentation of all calculations performed in support of preparation and analysis;

(v) all relevant QA/QC measurements (including the results of the analysis of all blanks, duplicates, and replicates--see Chapter 12); and

(w) a detailed time log of events including the time that particular procedures are initiated and completed for each sample.

13.2 FIELD ACTIVITIES REPORT

To assure that the field information required to complete estimation of dust and asbestos concentrations and release rates are provided to the data users, a field activities report must be completed and must be submitted to the laboratory along with the corresponding samples. Laboratory personnel are then to attach this report directly to their batch report, which shall cover the corresponding batch of samples.

The field activities report shall include the following for each sample batch, at a minimum:

(a) the project title and the identification of the site;

(b) reference to this method;

(c) reference to the sampling and analysis plan under which samples were collected;

(d) a brief description of the objectives for sampling;

(e) a brief description of the procedures employed for selecting sampling locations and the motivation for employing such procedures;

(f) for each sample in the batch accompanying the report:

- the identifier for each sample fraction submitted to the laboratory;
- the identifier for the original sample from which each submitted sample fraction was derived;
- the type of equipment and reference to (or a brief description of) the procedures employed for collection of the original sample;
- the coordinates at which each original sample was collected[36];
- the total mass of the original sample from which each sample fraction originated and the masses of the coarse and fine fractions separated during sieving of the sample in the field;
- a brief description of the procedures employed for sample homogenization and for sample splitting;
- the date and time that each sample was collected and the date and time that each sample fraction was prepared in the field; and
- a brief discussion of any deviation from the sampling and analysis plan not covered in e.

An example of the format to be employed for a field activities report is provided in Figure 13-1.

13.3 SAMPLE ANALYSIS REPORT

The sample analysis report for each sample shall include the following at a minimum:

(a) reference to this method;

(b) reference to the sample identification and batch number for the sample;

(c) the date and site from which the sample was collected;

(d) the weights and identities of the coarse and fine fractions of the sample and the sub-sample of the fine fraction sent for analysis;

(e) the weights and identities of any splits or other fractions of the sample generated during laboratory preparation;

(f) the weight and identity of the sub-sample placed in the dust generator;

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[36] If the submitted sample fraction is a sub-sample of a composite, what should be described here is the specific area of concern (or portion thereof) that is intended to be represented by the composite).

FIGURE 13-1
FORMAT FOR THE FIELD ACTIVITIES REPORT

Name of field activities contractor
Address of contractor
Contact Name
Telephone Number

Report Title:
Report Number:

Date

PROJECT/SITE:
Name
Address

METHODS AND PROCEDURES:
Field Investigation Design: *(Reference the sampling plan)*
Sample Collection and Handling: *(Reference this method)*

SAMPLING OBJECTIVES:
(Complete a brief description here)

SAMPLING LOCATION SELECTION PROCEDURE:
(Complete a brief description here)

SAMPLE DATA

Field Sample Number	Location Identifiers	Mass of Sample	Mass Fine Fraction	Mass Coarse Fraction	Mass Sample Split	Mass Duplicate Split	Sample Split ID	Duplicate Split ID	Date Sampled	Time Sampled	Comments: *(Include identification of all field sampling and field preparation procedures employed)*

An example of the format to be employed for a sample analysis report is presented in Figure 13-2.

13.4 SAMPLE BATCH REPORTS

In addition to the sample analysis report for each sample, provide a summary page for each batch of samples representing an entire project. The summary sheet shall include:

(a) the project title;

(b) reference to this method;

(c) the date that samples were collected, the date they were received by the laboratory, and the date they were analyzed;

(d) a summary listing of sample results including:

- the sample number;
- the estimated concentration of respirable dust in the sample;
- the analytical sensitivity achieved for each size/type category of interest;
- the total number of structures of each size/type category of interest counted;
- the concentration of asbestos structures of each size/type category of interest in the sample and in the respirable dust of the sample (both reported along with corresponding 95% confidence limits); and
- the concentration of asbestos structures of each size/type category of interest estimated for the environmental matrix that was sampled in the field (reported along with corresponding 95% confidence limits).

An example of the format to be employed for sample batch reports is presented in Figure 13-3.

FIGURE 13-2
SAMPLE ANALYSIS REPORT FORMAT

Laboratory Name
Laboratory Address
Laboratory Contact
Telephone Number

Report Date
Project Name *(Optional)*

METHODS:
(reference this method)

Date Analysis Started (M/D/Yr)
Date Analysis Completed (M/D/Yr)
Analyst(s) Initials

Laboratory Sample No.
Field Sub-Sample Identification No.
Field Preparation Technique **(Attach a Copy of the Relevant Field Activities Report)**
Additional Laboratory Preparation Procedures *(describe any employed)*
Sample Drying
Sample Splitting
Other

TEM Analysis:
Effective Area of Analytical Filter (sq mm)
(Indicate whether from Scrubber or from IST Opening)
Magnification
Grid Opening Area (sq mm)
Number of G.O. Scanned
Asbestos Structure Size and Type Categories of Interest *(see Chatfield 1993)*
Minimum Acceptable Structure Identification Category *(see Chatfield 1993)*

Dust Generator
Mass of Sample Tumbled (g)
Air Flow Rate Through ME Opening of Dust Generator (ml/min)
Air Flow Rate Through IST Opening of Dust Generator (ml/min)
Air Flow Rate Through Scrubber (ml/min)
Estimated Total Air Flow Rate Through Elutriator (ml/min)

Total Mass of Dust Collected on Dust Filters (g)
Time of Dust Collection *(24 hour clock)* at 30 rpm
Start:
Stop:
Time of Dust Collection *(24 hour clock)* at 60 rpm
Start:
Stop:
Estimated first-order rate constants for dust generation (min^{-1})
At 30 rpm:
At 60 rpm:
Estimated Starting Mass of Respirable Dust in Sample (g)
(Attach time plots and calculations).

Samples from the Scrubber Suspension
Total Volume of Scrubber Suspension (ml)
Estimated Mass of Dust Collected in Scrubber Suspension (g)
Volume of Aliquot Withdrawn from Scrubber Suspension (ml)
Volume into which Scrubber Aliquot Diluted (ml)
Dilution Factor (dimensionless)
Volume of Aliquot Filtered *(from Diluted Suspension)* (ml)

Samples from the Isokinetic Sampling Tube (IST) Opening of the Dust Generator
(indicate whether 30 or 60 rpm run)
Time of Collection *(24 hour clock)*
Start:
Stop:
Estimated Mass of Dust Collected on Filter

FIGURE 13-2
SAMPLE ANALYSIS REPORT FORMAT (Cont.)

Laboratory Name

Report Date

Laboratory Sample No.

	Low Magnification	High Magnification
Chrysotile Asbestos Analysis Results:		
No. of Total Chrysotile Asbestos Structures	XXX	
No. of Long (> 5 um) Chysotile Asbestos Structures		
No. of Total Chrysotile Asbestos Fibers/Bundles	XXX	
No. of Long (> 5 um) Chysotile Asbestos Fibers/Bundles		

	Low Magnification	High Magnification
Amphibole Asbestos Analysis Results:		
No. of Total Amphibole Asbestos Structures	XXX	
No. of Long (> 5 um) Amphibole Asbestos Structures		
No. of Total Amphibole Asbestos Fibers/Bundles	XXX	
No. of Long (> 5 um) Amphibole Asbestos Fibers/Bundles		

(Indicate Amphibole Mineral Type)

ESTIMATED CONCENTRATIONS OF RELEASABLE ASBESTOS IN SAMPLE

	Conc.	95%UCL
Total Chrysotile Structures per g Sample:		
Total Amphibole Structures per g Sample:		
Total Asbestos Structures per g Sample:		
Long Chrysotile Structures per g Sample:		
Long Amphibole Structures per g Sample:		
Long Asbestos Structures per g Sample:		

Estimated Analytical Sensitivity: *(structures/g sample)*

ESTIMATED CONCENTRATIONS OF RELEASABLE ASBESTOS IN RESPIRABLE DUST OF SAMPLE

	Conc.	95%UCL
Total Chrysotile Structures per g Dust:		
Total Amphibole Structures per g Dust:		
Total Asbestos Structures per g Dust:		
Long Chrysotile Structures per g Dust:		
Long Amphibole Structures per g Dust:		
Long Asbestos Structures per g Dust:		

Estimated Analytical Sensitivity: *(structures/g dust)*

(Attach a Copy of the TEM Raw Data Sheets)

FIGURE 13–3
SAMPLE BATCH REPORT FORMAT

Laboratory Name
Laboratory Address
Laboratory Contact
Telephone Number

Report Date
Project Name *(Optional)*

METHODS:
(reference this method)

RELEASABLE ASBESTOS IN RESPIRABLE DUST

Laboratory Sample I.D.	Respirable Dust Conc (g/g smpl)	Total Asbestos Analytical Sensitivity (s/g dust)	Total Asbestos Conc (s/g dust)	Total Asbestos 95% UCL (s/g dust)	Long Asbestos Analytical Sensitivity (s/g dust)	Long Asbestos Conc (s/g dust)	Long Asbestos 95% UCL (s/g dust)	Dust Generation Rate (at 30 rpm) (min^{-1})	Dust Generation Rate (at 60 rpm) (min^{-1})	Comments:

RELEASABLE ASBESTOS IN LABORATORY SAMPLES

Laboratory Sample I.D.	Respirable Dust Conc (g/g smpl)	Total Asbestos Analytical Sensitivity (s/g smpl)	Total Asbestos Conc (s/g smpl)	Total Asbestos 95% UCL (s/g smpl)	Long Asbestos Analytical Sensitivity (s/g smpl)	Long Asbestos Conc (s/g smpl)	Long Asbestos 95% UCL (s/g smpl)	Comments:

RELEASABLE ASBESTOS IN FIELD SAMPLE MATRICES

Laboratory Sample I.D.	Field Sample I.D.	Mass Fine Fraction (g)	Mass Coarse Fraction (g)	Adjusted Respirable Dust Conc (g/g mtrx)	Adjusted Total Asbestos Conc (s/g mtrx)	Adjusted Total Asbestos 95% UCL (s/g mtrx)	Adjusted Long Asbestos Conc (s/g mtrx)	Adjusted Long Asbestos 95% UCL (s/g mtrx)	Comments:

14.0 REFERENCES

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APPENDIX A:

CONSTRUCTION AND OPERATION OF A DUST GENERATOR

APPENDIX A:
CONSTRUCTION AND OPERATION OF A DUST GENERATOR FOR ISOLATING THE RESPIRABLE FRACTION OF MATERIAL FROM SOILS OR OTHER BULK SAMPLES

The dust generator incorporated into this method for the determination of asbestos in soils and bulk materials is designed to isolate the respirable fraction of material (including the releasable fraction of respirable asbestos) that is present within the matrix of the parent sample. A description of the apparatus is provided below along with a brief discussion of the theory of its operation. Figures depicting design of the prototype are also provided in the last section of this appendix to facilitate design and construction of similar equipment.

A.1 DUST GENERATOR DESCRIPTION

The dust generator is composed of a tumbler, a vertical elutriator, a dust collection system, and a scrubber. A schematic diagram of the apparatus is presented in Figure A-1 and a photograph of the apparatus is shown in Figure A-2. The appurtenant equipment required to operate the dust generator is also shown in Figure A-2. This includes:

- a DC motor to drive the tumbler;
- a constant humidity chamber to control the humidity of the air that flows into the dust generator. This is the clear plastic box enclosing the tumbler in the photograph;
- the pumps required to create an air flow through the dust generator and the flow controllers required to monitor and apportion air flow through the various filters and the scrubber; and
- the heating mantle, variable voltage transformer, and cooling towers required for the scrubber.

A.1.1 The Tumbler

The tumbler is located in the clear plastic enclosure at the bottom of the dust generator (Figures A-2 and A-3). It is a long shallow tube of square cross section that is approximately 1 and 1/8 inches in height and width and has an overall length of 10 inches (Figure A-4).

The tumbler is driven by a variable speed DC motor, which rotates the tumbler around its long axis. The tumbler's rotation rate can be varied over the range of 10 to 150 revolutions per minute (rpm). The DC motor is attached to the tumbler using a slip-on type flexible coupling so that they can be readily detached simply by pulling the motor away from the tumbler. The tumbler can also be readily detached from the connection tube to the elutriator; they are connected with a slip fit over the outer race of the ball bearing assembly that is welded to the end of the tumbler (Figure A-4).

FIGURE A-1

SCHEMATIC OF DUST GENERATOR

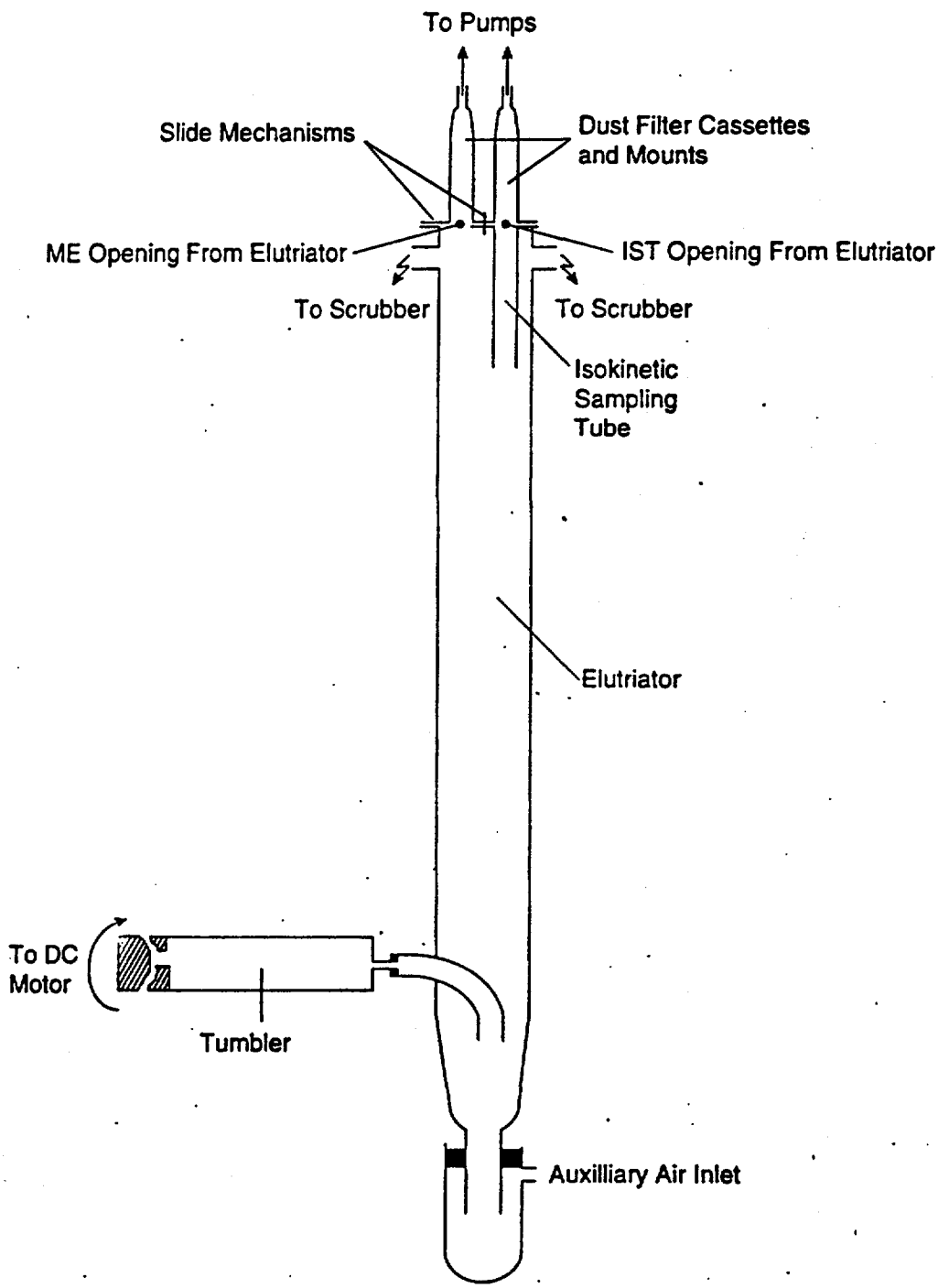


FIGURE A-2

THE DUST GENERATOR

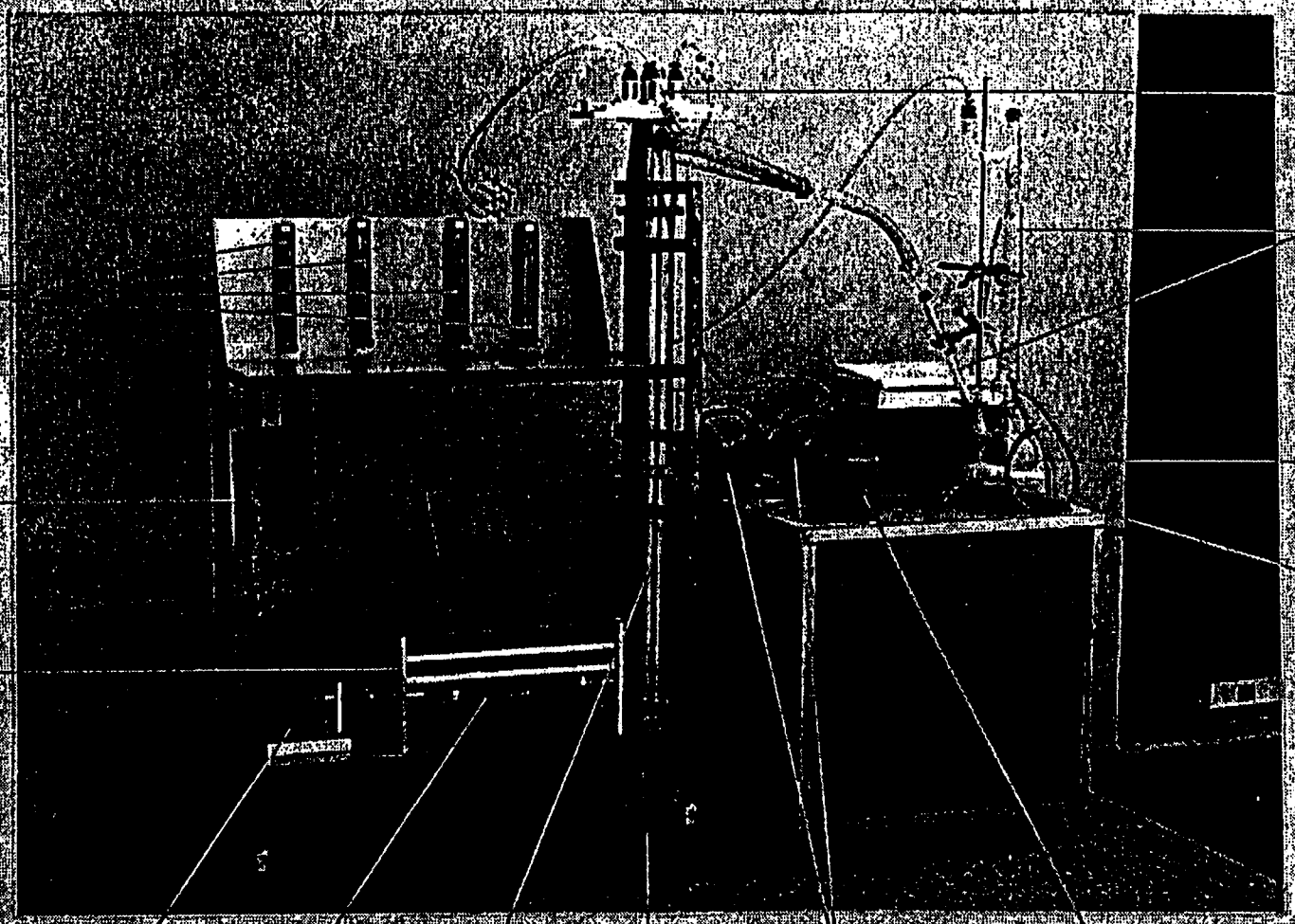


FIGURE A-3

TUMBLER IN CONSTANT HUMIDITY CHAMBER

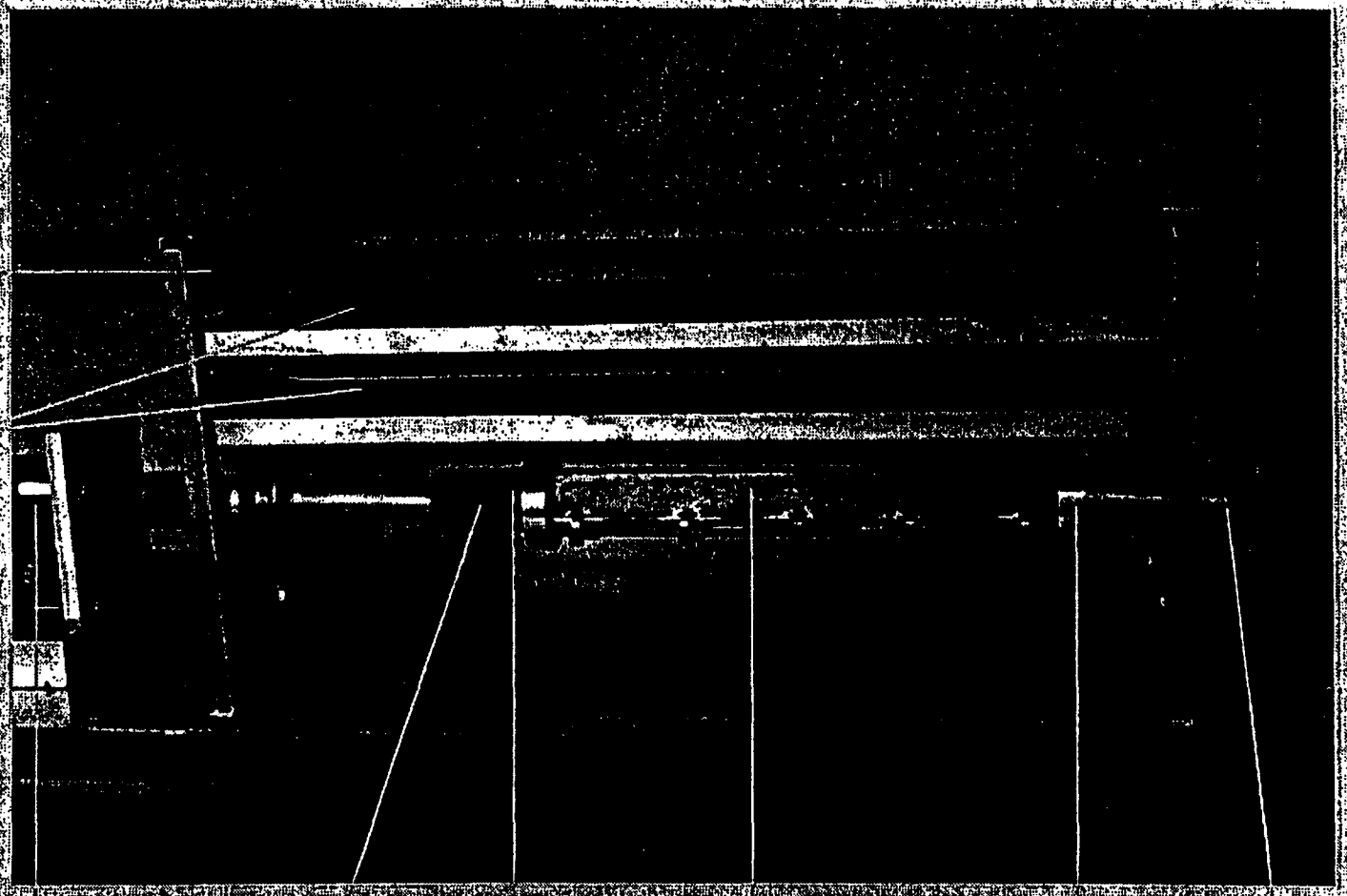
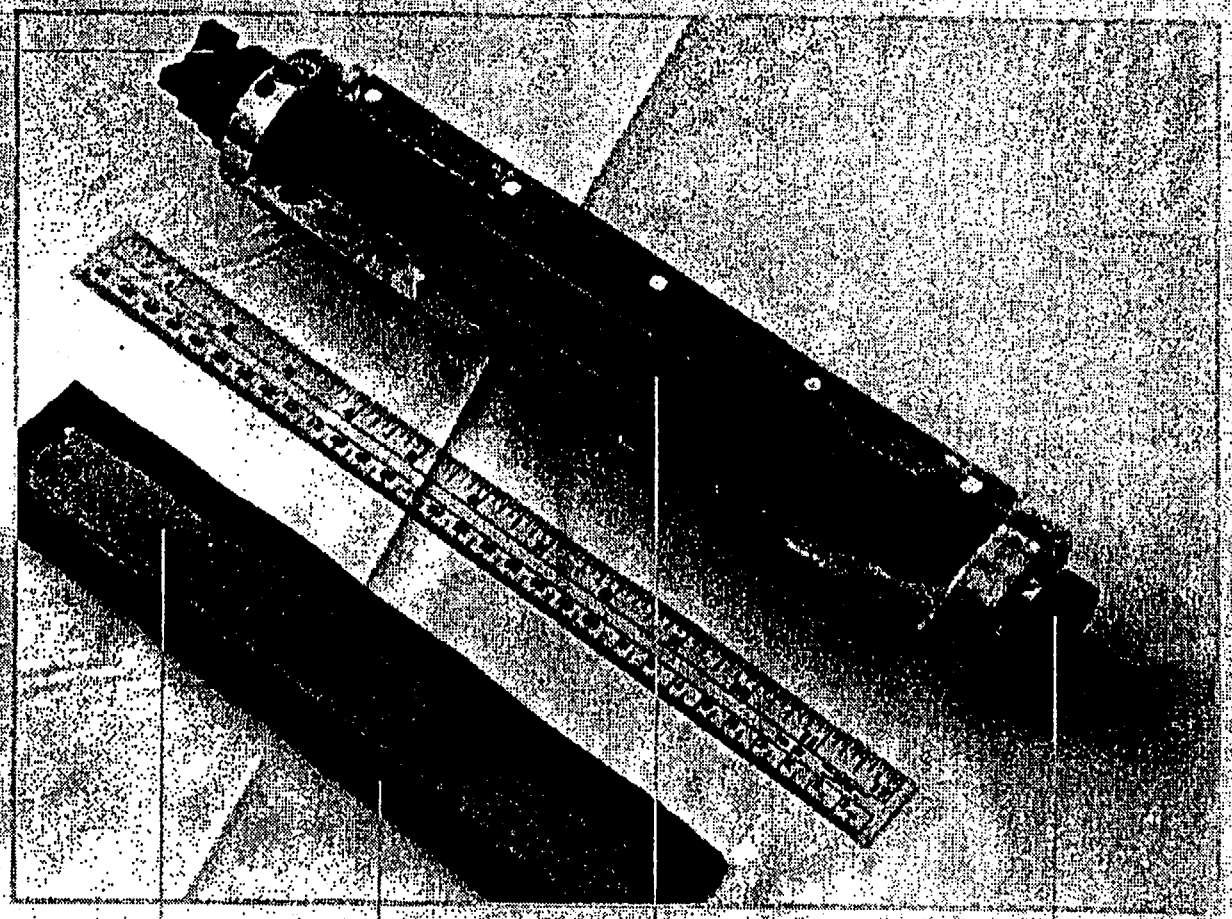


FIGURE A-4

TUMBLER ASSEMBLY



NOTE

The seal between the tumbler and elutriator need not be air tight. In fact, a *small* leak at this fitting will reduce the chance that particles discharged from the tumbler will settle in the entrance tube to the elutriator. However, such a leak must not be more than 10% of the total airflow through the tumbler.

Once the tumbler has been detached from the motor and elutriator, it can simply be lifted out of the constant humidity chamber.

The top cover of the tumbler is secured with 10 screws and can be removed so that the tumbler can be loaded or cleaned (Figure A-4). A rubber gasket is employed to assure an airtight seal between the tumbler cover and body.

A.1.2 The Constant Humidity Chamber

The plastic enclosure that houses the tumbler and isolates it from outside air, except for an opening at the top front of the enclosure, is designed as a constant humidity chamber (Figures A-2 and A-3). Air drawn into the front of the enclosure flows successively over two trays (located on shelves at the top of the enclosure) before being drawn into the tumbler. The trays are designed to hold a saturated salt solution that is selected to exhibit a vapor pressure equal to the relative humidity desired for running the dust generator. The path length over the two trays is designed to assure that air drawn through the device will come to equilibrium at a relative humidity within a few percent of the desired value (assuming that the outside air exhibits a starting humidity within the range common to Pasadena, California[1]); the path length must be sufficient to allow adequate exchange of vapor between the salt solution and the moving air.

For most studies, a relative humidity of approximately 50% should be employed because this is the humidity at which emission of asbestos is expected to be maximum (Zimon, A.D. 1982). At higher humidities, air moisture tends to wet surfaces so that aggregation decreases particle release. At lower humidities, electrostatic effects tend also to cause aggregation and, therefore, also to decrease emissions. A saturated solution of potassium carbonate dihydrate is recommended for most applications because such a solution maintains equilibrium with air at 43% relative humidity at room temperature (20° C) with less than 1% change in humidity over a range of temperatures varying by several degrees on either side of room temperature.

For special applications of this method, where dust generation is to be run at a different relative humidity than that produced over a saturated solution of potassium carbonate dihydrate, the conditioning pans in the dust generator may be filled with a saturated solution of another salt. Such salts may be selected to yield any of a broad range of conditions. For example, the International Critical Tables provides a list of saturated salt solutions that maintain equilibrium with a broad range of relative humidities in air.

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1 The prototype device was constructed and tested in Pasadena. The design of the constant humidity chamber may have to be modified slightly (e.g. by increasing the number of trays) for operation in other locales.

A.1.3 The Vertical Elutriator

The vertical elutriator is the tall metal cylinder visible in Figure A-2. Its dimensions were selected to assure that the path length traversed by particles in the elutriator is at least 10 times the diameter of the elutriator (in the direction perpendicular to the air flow). Thus, channeling is expected to be minimized (Dennis, R 1976).

The elutriator separates respirable from non-respirable particles and respirable particles are passed on to dust collection filters and the scrubber. Air flow within the elutriator is adjusted to a velocity such that only those particles in the respirable size range will be lifted to the top. The velocity of air selected for the elutriator determines the rate of air flow through all of the other parts of the dust generator.

The main body of the elutriator is composed of two, 20 in. sections of 4-in. i.d. stainless steel tubing with a tapered, o-ring flange welded to each end of each tube (Figure A-5a and b). These flanges allow the two halves of the main body to be connected to each other and to the top and bottom assemblies of the elutriator using quick-release, pressure clamps (Figure A-5a).

The entrance tube to the elutriator (from the tumbler) is pointed downward at its exit point in the bottom of the elutriator (Figures A-1 and A-5b). This geometry was selected to prevent introduction of a large concentration of particles into the bottom center of the elutriator. This geometry should also promote a flatter velocity profile in the air traveling up the elutriator. It is expected that respirable particles will make the upward turn because the taper at the bottom of the elutriator promotes a higher velocity profile at this location than in the main body of the elutriator (where the air velocity is just sufficient to lift particles in the respirable size range but no larger).

The bottom assembly of the elutriator has also been designed to allow the removal of coarse particles while assuring that respirable particles remain in the elutriator air stream (Figure A-6a and b). This is to minimize buildup of a zone at the bottom of the elutriator of a high concentration of coarse particles through which respirable particles would have to pass before moving up the elutriator to the dust collection system. Thus, it is expected that collisions between respirable particles and coarse particles (with subsequent loss of respirable particles due to aggregation) are minimized.

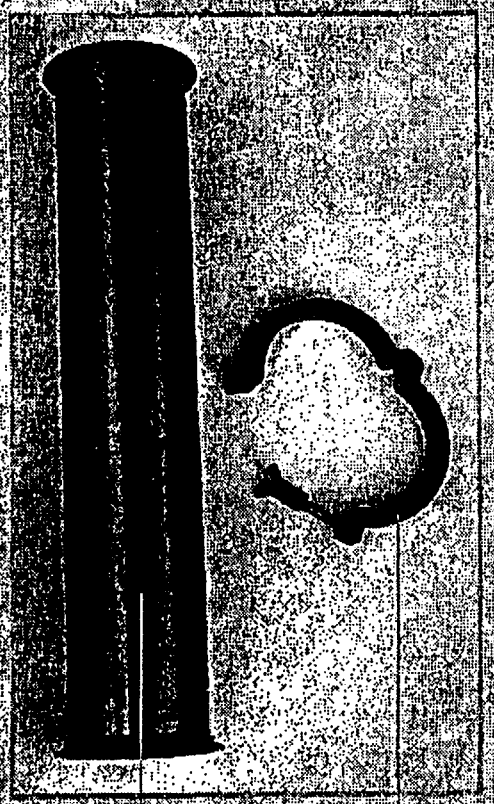
An opening at the bottom of the elutriator allows coarse particles to pass into a removable, glass cup (Figures A-1 and A-6a and b). The glass cup has a small side arm that is connected to a two ft. piece of one quarter in. i.d. Tygon tubing. The far end of the Tygon tube is open to the air. It has been found that, as long the Tygon tube is not crimped or otherwise blocked, air drawn through this tube is just sufficient to prevent respirable particles from passing through the bottom opening of the dust generator into the glass cup while allowing larger particles to do so.

There are four openings where air may exit from the top assembly of the elutriator (Figures A-1 and A-7a and b). The two side openings lead into the scrubber (described below). The two top openings lead into the air filter cassettes of the dust collection system through two different paths; one draws air isokinetically from the uniform portion of the elutriator through a long, thin-walled tube and the other draws air from the top, tapered part of the elutriator (Figures A-1 and A-7a and b). For ease of reference, the opening in the elutriator that draws

FIGURE A-5

MAIN BODY OF VERTRICAL ELUTRIATOR

a. Top Section



b. Bottom Section

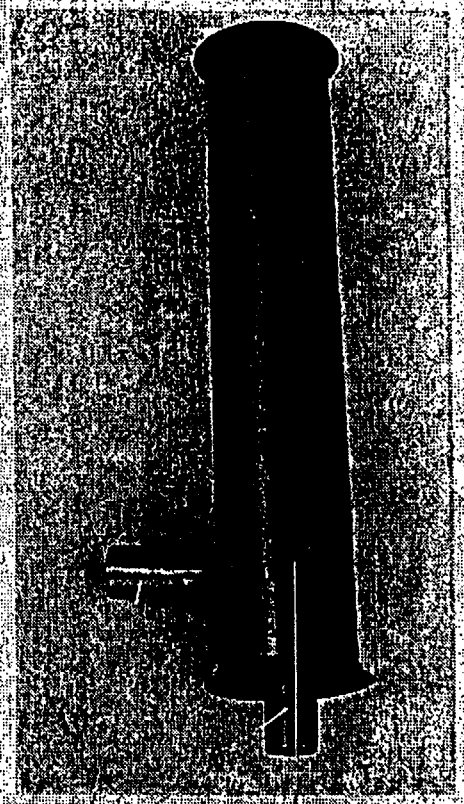


FIGURE A-6

BOTTOM ASSEMBLY OF VERTICAL ELUTRIATOR

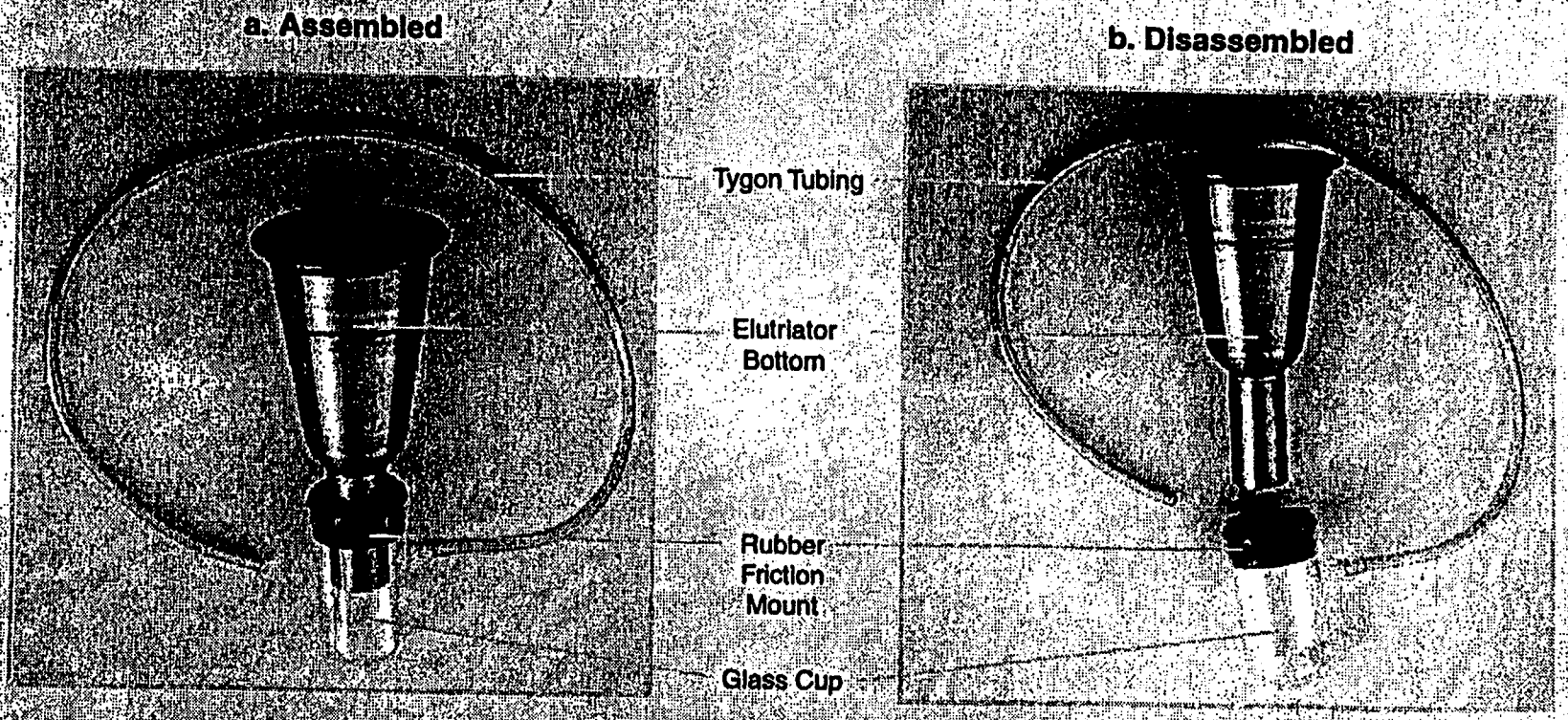
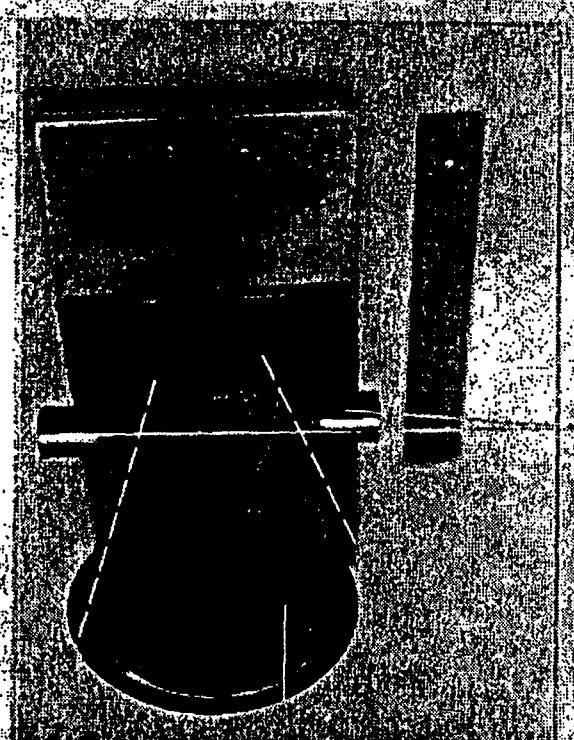
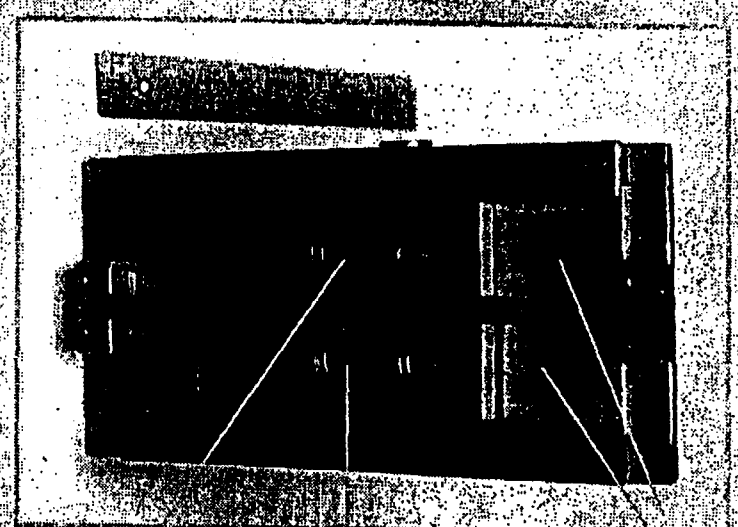


FIGURE A-7

TOP ASSEMBLY OF VERTICAL ELUTRIATOR





air through the isokinetic sampling tube is labeled the "IST" opening in this document. The opening that draws air from the top of the main body of the elutriator is labeled the "ME" opening. The dust collection system mounted over both of the top openings of the elutriator is described below.

A.1.4 The Dust Collection System

The dust collection system consists of two mounts for filter cassettes welded to each of two sliding mechanisms, which in turn are mounted directly over the exit openings of the top assembly of the vertical elutriator (Figure A-8a and b). An underside view of the tapered top piece of the elutriator was also shown in Figure A-7a in which the isokinetic sampling tube is visible.

The sliding mechanisms permit the filter cassettes aligned over the ME and the IST openings of the elutriator, respectively, to be changed with minimal disturbance of the air flow; at either of the two extreme positions in the travel of each slide mechanism, one of the two cassette mounts are aligned over the corresponding exit opening of the elutriator and the other is effectively isolated from the air flow. Details of the sliding mechanisms and filter mounts are shown in Figures A-7b and A-8a and b.

NOTE

The slides are sealed against the elutriator with o-rings. These must be inspected periodically for wear and replaced if worn.

The design of the filter mounts in the dust collection system proved to be important to the performance of the dust generator; it is critical that these mounts be leak tight. The design that was ultimately adopted is depicted in Figure A-9a and b. The filter mounts each consist of a tapered aluminum base that is glued to the bottom half of a commercially available filter cassette.

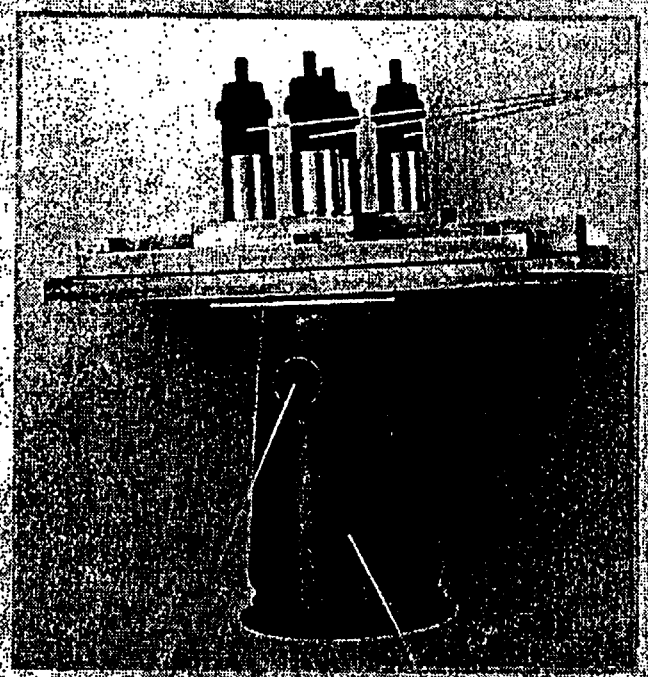
The aluminum base of a filter mount is sealed in each of the openings of the slide mechanisms with the o-rings that are visible in Figure A-7b. The bottom half of the plastic, 25 mm filter cassette is glued into the inside taper of the aluminum base. Filters are mounted in the traditional manner between the top and bottom halves of the plastic filter cassette, which are then pressure sealed. To further assure a good seal, pressure tape is also applied to the outside of each filter cassette at the seam where the filters are mounted.

A.1.5 The Scrubber

The scrubber, which can be seen sitting on the table to the right of the main body of the dust generator in Figure A-2, is constructed from ordinary laboratory glassware. In the scrubber, water is boiled in the bottom of a 1 liter, round bottom flask (shown seated within a heating mantle). A straight-jacketed, cold-water condenser is incorporated along the entrance line to the scrubber and a spiral condenser is incorporated along the exit line from the scrubber. An immersion pump circulates water between a cooler containing a water-ice mix and the condensers. Water flowing into the condensers is maintained at approximately 0° C to prevent moisture from leaving the scrubber.

FIGURE A-8

DUST COLLECTION SYSTEM



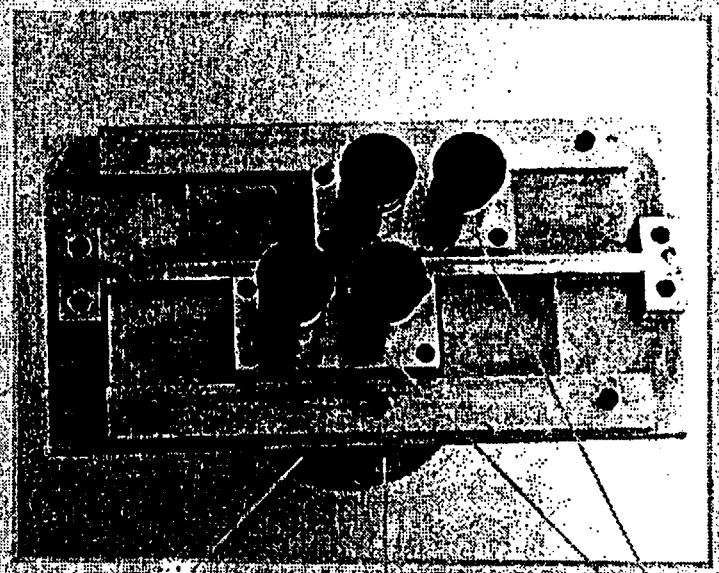
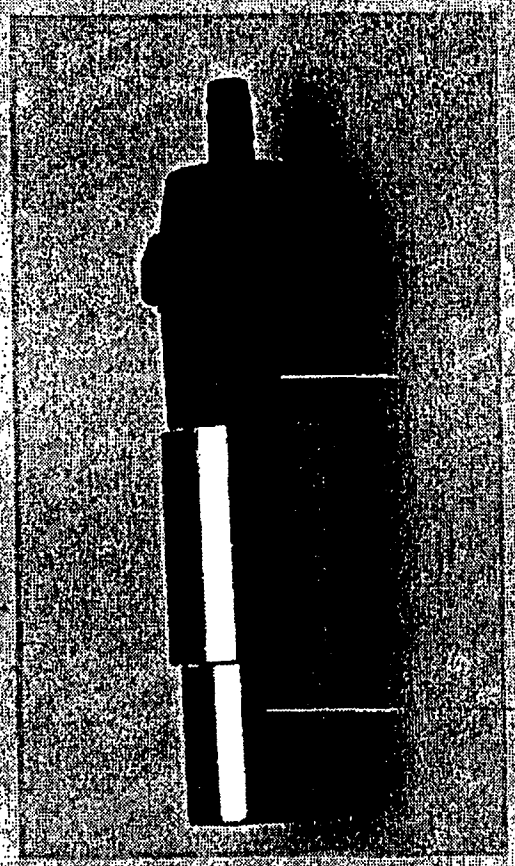


FIGURE A-9

FILTER MOUNTS

a. Assembled Filter Mount



b. Disassembled Filter Mount

Commercially Available 25mm Filter Cassette

Aluminum Base

Particles in the air stream entering the scrubber serve as nucleation centers around which the steam in the scrubber condenses. The resulting water droplets eventually fall back into the water reservoir at the bottom of the scrubber so that the trapped particles are collected in this reservoir.

A flowmeter and a filter cassette are also placed in the air flow line that exits the scrubber (between the scrubber and the vacuum pump). The primary reason for including the filter cassette is to make the pressure drop through this path approximately equal to the pressure drop through the other filter cassettes mounted on top of the elutriator (the dust collection system). The pressure drop through the elutriator and scrubber is small enough not to affect the flowmeter operation.

A.1.6 The Configuration of Air Transfer Lines

A schematic indicating the configuration of air transfer lines leading to and from the scrubber is presented in Figure A-10. As indicated in the figure, the two side exits from the elutriator are each attached to one ft sections of 1.00 in. i.d. Tygon tubing, which then join at a glass "Y" connector. The stem end of the "Y" connector is attached (with a one ft section of 1.00 in. i.d. Tygon tubing) to a diameter reducing piece of glass that feeds into the entrance condenser to the scrubber through a rubber stopper. The large tubing is held in place at connections by ring clamps (Figure A-2).

Also as indicated in Figures A-10 and A-2, the exit condenser of the scrubber is connected to a flow control valve with 0.25 in. i.d. Tygon tubing. The exit side of the flow control valve is connected first to a 25 mm filter cassette and then to a vacuum pump. Both of these connections also use 0.25 in. i.d. Tygon tubing.

A schematic indicating the configuration of air transfer lines leading from the filter cassette mounts of the dust collection system is depicted in Figure A-11. A photograph of the transfer line connections is also presented in Figure A-12. As indicated in the figures, a 0.25 in. i.d. Tygon line leads from the exit side of each filter cassette to a plastic, stop cock valve. Another Tygon line then leads from each valve to one of two plastic "T" connectors so that the pair of filter cassettes mounted on the slide mechanism over the IST opening of the elutriator are joined (beyond the stop cock valves) and the pair of filter cassettes mounted on the slide mechanism over the ME opening are also joined (beyond the stop cock valves). The common line from the exit side of *each* "T" connector is then connected first to a flowcontrol valve and then to a vacuum pump. All such connections use 0.025 in. i.d. Tygon tubing.

NOTE

This is the configuration of air transfer lines that is appropriate during the operation of the dust generator. The configuration that is appropriate during calibration of air flow is discussed in Section 9.3.4 of the main text.

As indicated previously, the side arm on the bottom cup of the elutriator is connected to a two ft section of 0.25 in. i.d. Tygon tubing that is simply allowed to hang free (Figure A-6a).

FIGURE A-10

TUBING CONNECTIONS FOR THE SCRUBBER OF THE DUST GENERATOR

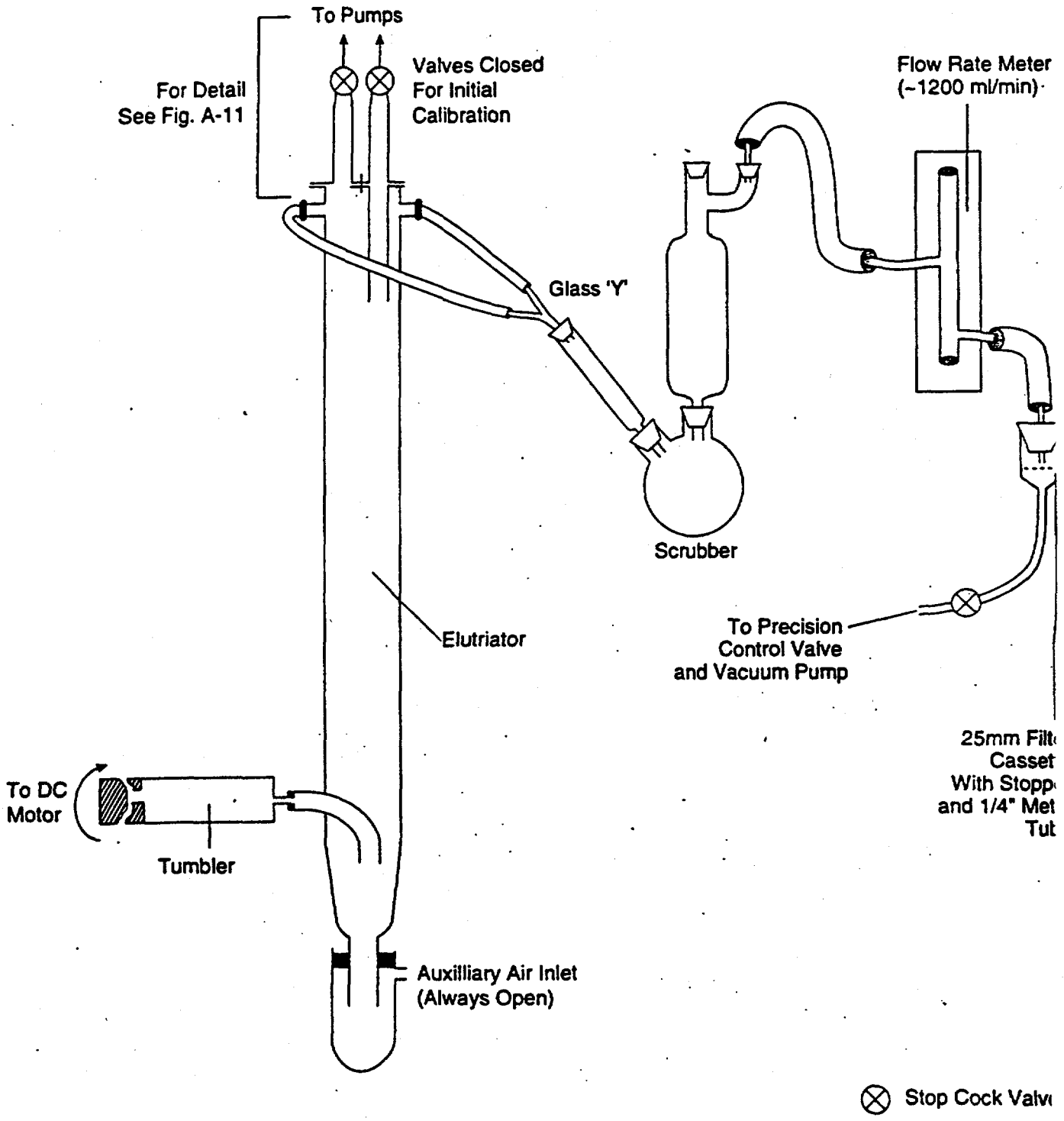
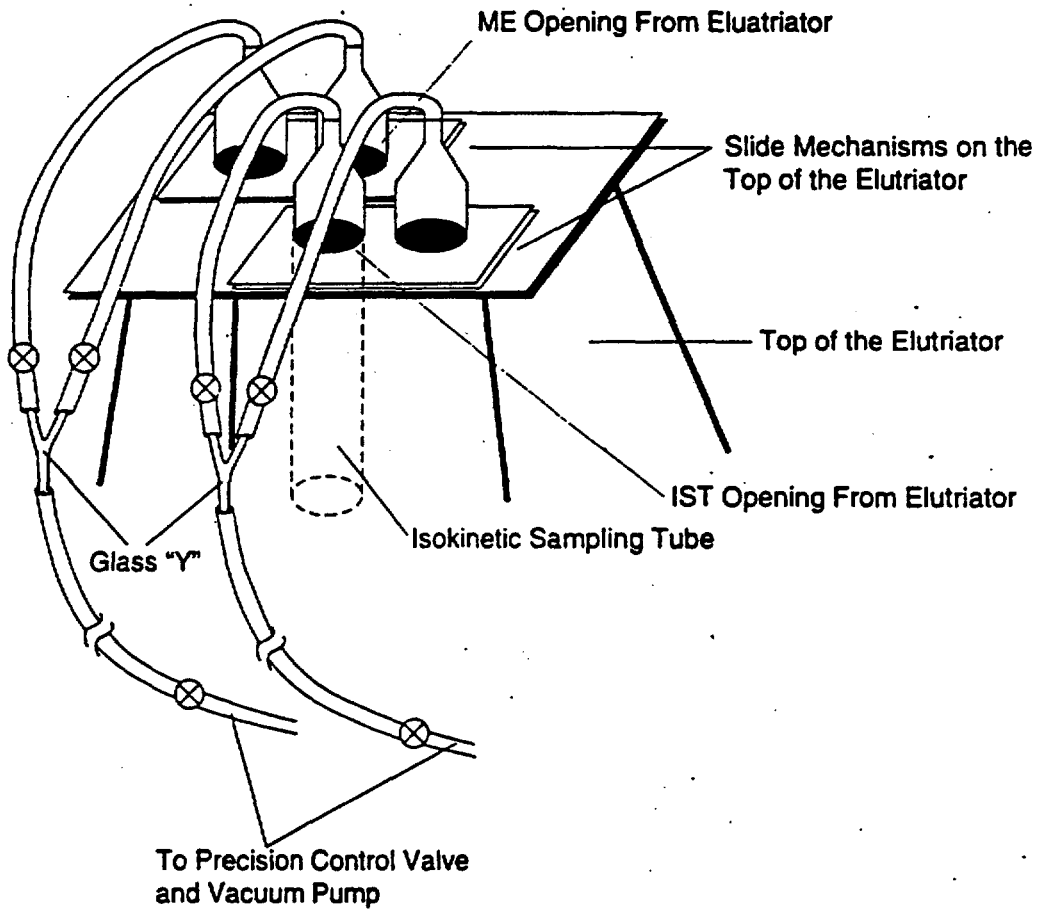


FIGURE A-11

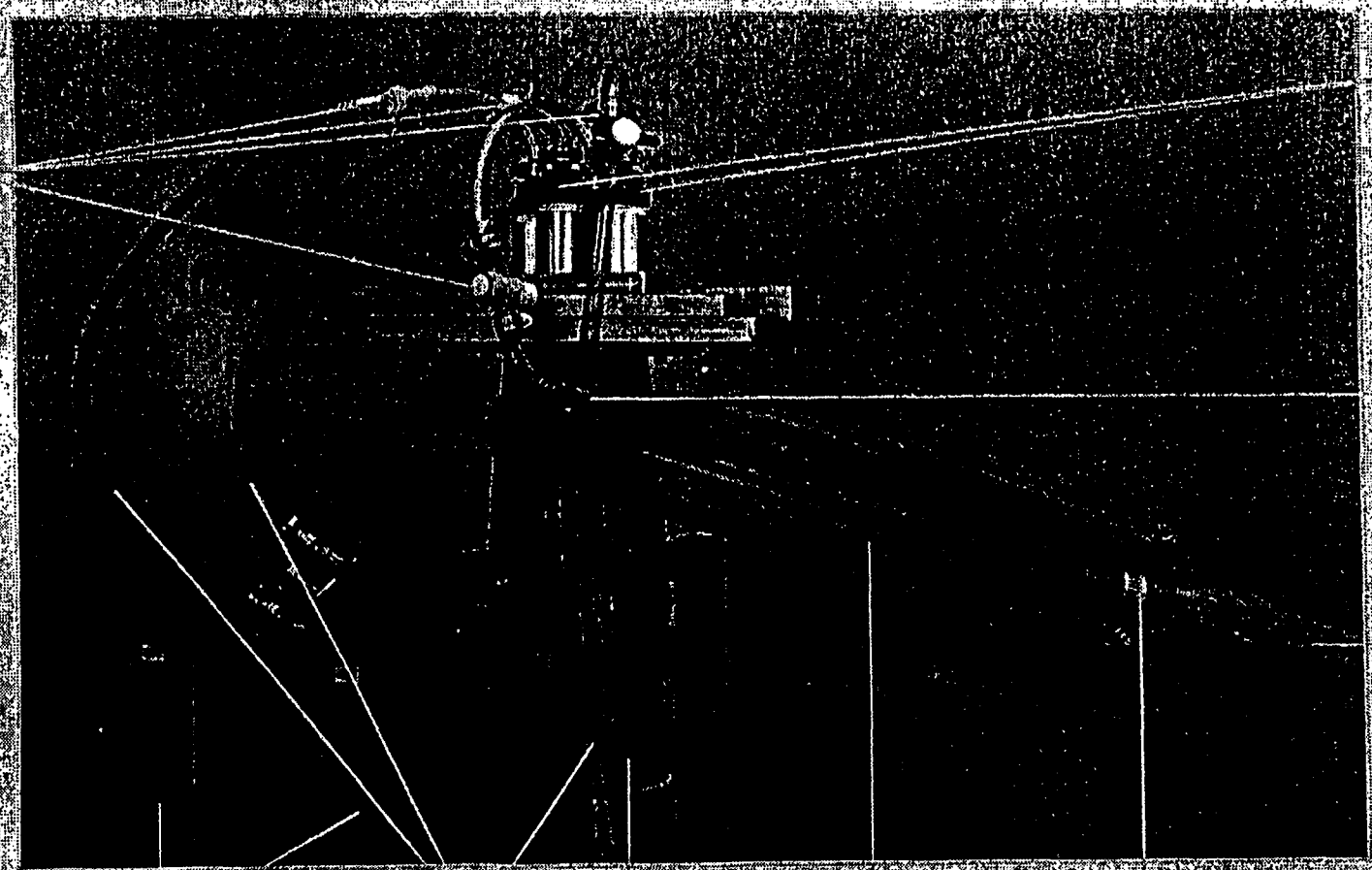
TUBING CONNECTIONS FOR FILTER CASSETTES MOUNTED ON THE ELUTRIATOR OF THE DUST GENERATOR



⊗ Stop Cock Valves

FIGURE A-12

DETAIL OF TUBING CONNECTIONS AT THE TOP OF THE VERTICAL ELUTRIATOR



A.2 THEORY OF OPERATION

A.2.1 The Dynamics of Dust Generation

The dynamics of the release of dust from a sample during a run using the dust generator have been evaluated so that the rate of release and mass of dust in the sample can be derived from measurements of the mass of dust deposited over time on the set of filters collected over the ME opening of the elutriator. Analysis of data obtained from several different types of samples during the pilot study for this method (Berman et al., 1994) indicate that the rate of release of mass from a sample in the dust generator is well described by a first-order rate equation:

$$-dM_s/dt = k*M_s \qquad \text{(A-1)}$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" (g);

t is the time since the start of the run (s); and

k is the first-order rate constant for the release (s^{-1}).

The minus sign in this equation indicates that mass is lost with time.

Equation A-1 can be integrated to yield:

$$\ln(M_s) = \ln(M_o) - kt \qquad \text{(A-2)}$$

where:

M_o is the mass of respirable dust in the sample at the start of the run (i.e. at time t = 0) (g).

Given that "M_s" can also be expressed as the difference between "M_o" and "M_r" the cumulative mass released up to time "t," Equation A-2 can also be expressed as:

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad \text{(A-3)}$$

where:

M_r is the cumulative mass released between the start of a run and time "t" (g).

The relationship presented in Equation A-3 indicates that a plot of the natural logarithm of the quantity (M_o -M_r) versus time should be a straight line with a slope equal to the rate constant for dust release, k, and an intercept equal to the initial mass of dust in the sample at the start

of the run, M_o. The cumulative mass of dust released from the sample over time, M_r, can be derived from measurements of dust collected on filters during the run. However, because M_o also appears as part of one of the parameters that must be plotted to evaluate the relationship expressed in Equation A-3, the value of M_o must be optimized using regression, as described in Section 11.2 of the main text of this method.

The cumulative mass released from a sample at time "t" during a run, "M_r" is directly proportional to the cumulative mass measured on filters collected during the run:

$$M_r = M_f*(F_s + F_d + F_c)/F_c \qquad \text{(A-4)}$$

where:

M_f is the cumulative mass measured on filters collected from filters mounted over the top of the elutriator up to time "t" (g);

F_s is the rate of airflow through the scrubber (cm^3/s);

F_d is the rate of airflow through the IST opening of the elutriator (cm^3/s); and

F_c is the rate of airflow through the ME opening of the elutriator (cm^3/s).

Because F_s and F_c will typically have been set to $0.48*V_v$ and F_d will typically have been set to $0.047*V_v$ during the initial setup of the dust generator (see Section 9.3.4), for most applications, Equation A-4 reduces to:

$$M_r = 2.1*M_f \qquad \text{(A-5)}$$

As indicated above, values for M_o must be derived by performing a regression analysis of the relationship described by Equation A-3. This can be accomplished by using any of several commercially available spreadsheet programs (such as, for example, LOTUS™). The procedure to be followed to derive estimates of M_o and k are described in Section 11.2.

A.2.2 The Time Dependence of Dust Collection

As indicated in Section A.2.1 above, the generation of dust from the tumbler is well described by the first order rate equation:

$$-dM_s/dt = k*M_s \qquad \text{(A-1)}$$

However, experience gained during the pilot study for this method (Berman et al. 1994) further indicates that the rate of change of M_s is sufficiently slow in most cases such that, for periods of no more than 5 to 10 minutes, M_s can be considered constant. Thus, for

estimating such things as the time required to load individual filters in the dust generator, a simpler form of Equation A-1 can be used (in which M_s is considered constant):

$$\Delta M_s = k*M_s*\Delta t \qquad \text{(A-6)}$$

where:

M_s is still the mass of respirable dust remaining in the sample at time "t" but it is assumed constant over the short interval of time "Δt" (g);

ΔM_s is the mass of respirable dust released from the sample over the short time interval "Δt" (g);

Δt is a relatively short time interval (no more than ten minutes) during which the release of dust is being estimated (s); and

k is still the first-order rate constant for the release (s^{-1}).

Based on Equation A-6, the mass of respirable dust deposited on a filter in the dust generator, call this ΔM_f, is simply the product of the dust released from the sample, ΔM_s, and the fraction of the flow through the dust generator that is also directed through that filter. Thus, for filters collected over the isokinetic sampling tube (the IST opening of the elutriator):

$$\Delta M_f = 0.047* k*M_s*\Delta t \qquad \text{(A-7)}$$

or for filters collected over the ME opening of the elutriator:

$$\Delta M_f = 0.48* k*M_s*\Delta t \qquad \text{(A-8)}$$

The correct value for M_s to be used with Equations A-7 and A-8 is the value estimated by the relationship provided in Equation A-2 of Section A.2.1 where the "t" in Equation A-2 is the time that has elapsed from the beginning of the run to the start of the interval of interest, "Δt".

NOTE

In deriving Equations A-7 and A-8, it was assumed that air flow within the dust generator was setup as described in Section 9.3.4.

A.2.3 Size Separation Using the Vertical Elutriator

Separation of the respirable fraction of a particulate matrix can be accomplished by exploiting differences in the settling velocities of particles of different sizes when such particles are suspended in either a liquid or gaseous medium. However, air was selected as the medium into which samples would be suspended in this method to avoid changes in the characteristics of asbestos that typically occur when asbestos samples are placed in water[2]. The force on a particle suspended in a moving fluid is given by Stoke's Law (Fuchs, N.A. 1964). When such a particle is suspended in a fluid that is moving upward such that the force of the fluid and the force of gravity on the particle just balance and the particle remains motionless, Stoke's Law indicates that the following relationship holds:

$$4/3*\pi*r^3*d*g = -6*\pi*\eta*r*V_l \qquad \text{(A-9)}$$

where:

- r is the radius of the particle (cm);
- d is the density of the particle (g/cm^3);
- g is the acceleration due to gravity (cm/s^2);
- η is the dynamic viscosity of the fluid (g/cm*s); and
- V_l is the linear velocity of the fluid (cm/s).

The velocity estimated in Equation A-9 is termed the Stoke's velocity, V_s.

By substituting the viscosity of air at room temperature (i.e. 20° C) and the acceleration due to gravity into Equation A-9, the value of the Stoke's velocity of a particle is estimated as follows:

$$V_s = 1.18*10^6*r^2*d \qquad \text{(A-10)}$$

where "r" is the radius of the particle and "d" is the density of the particle. Because respirable particles are generally defined as those exhibiting an "aerodynamic equivalent diameter" of 10 µm, where an aerodynamic equivalent diameter is the diameter of a particle of unit density that settles at the same rate as the particle of interest, Equation A-10 can be used to find the Stoke's velocity of the largest respirable particles in the elutriator (i.e. by substituting a radius of 5 µm and assuming a density of 1):

$$V_s = 0.295 \text{ cm/s}$$

---

[2] It is well documented that the size distribution of asbestos structures in a sample change when such a sample is placed in water. For an overview of such documentation, see Berman and Chatfield (1990). Typically, the number of small fibers and bundles increases and the number and complexity of clusters and matrices decrease when asbestos samples are placed in water.

The Stoke's velocity for a particle is also equal to the velocity of the fluid stream that will just hold a particle motionless against gravity. Because the goal of the dust generator is to capture all particles that are potentially respirable, the velocity of air within the elutriator should be set so that it is just slightly larger (i.e. by 5%) than the Stoke's velocity estimated above (for the largest respirable particles) so that all respirable particles entering the elutriator will be imparted with an upward velocity and will be carried along with the air stream so that they are, ultimately, either deposited on a filter or passed into the scrubber. Therefore, airflow within the dust generator should be set so that the velocity of air within the vertical elutriator is 5% greater than 0.295:

$$V_l = 0.31 \text{ cm/s.}$$

A.3 DUST GENERATOR PROTOTYPE DESIGN FIGURES AND CONSTRUCTION GUIDELINES

The figures of the prototype dust generator that are included in this package are intended to be suggestive and not meant to be followed in exact detail. However, the dimensions of the various cross-sectional areas of different parts of the dust generator, which affect the relative air flows in various places, need to be followed closely for the dust generator to perform as intended. Other design features, such as couplings, clamps, and seals are intended more to be illustrative; alternate designs can be equally effective.

In spite of the low pressure differentials developed during operation in the prototype, performance was found to be very sensitive to leaks, especially leaks occurring in the isokinetic sampler filter mounts. Design features associated with the dust collection system should therefore be selected so as to minimize the potential for leaks in this area. The air flow path in the elutriator of any dust generator that is constructed for use with this method should be tested for leaks when construction is completed and periodically thereafter.

So that dust generator equipment constructed for use with this method will perform adequately, the following requirements must be incorporated into its design:

- the tumbler must be designed to hold a minimum of approximately 100 g of sample (with ample space left over to allow adequate tumbling) so that field homogenization requirements are not compromised. It should also be designed to assure a reasonably long pathlength over which air passes through the sample and baffles (or square corners) should be incorporated to assure adequate tumbling action and thorough mixing of sample and air;

- the pathlength of the elutriator should be a minimum of 10 times its diameter to minimize the possibility of channeling and the diameter should be large enough to assure a cross-sectional area that is at least 10 times that of the tumbler. This latter requirement is to assure adequate air flow in the tumbler that will effect efficient transfer of sample while flow throughout the device is limited to allow flow in the elutriator to be set at 1.05 times the settling velocity of the largest respirable particle of interest;

- the entrance tube and bottom of the elutriator should be shaped so that sufficient air velocities are maintained in this part of the device to assure a smooth transition (with efficient sample transfer) between flow in the tumbler and flow in the elutriator. It is also recommended that the entrance tube to the elutriator be pointed downward as a further hindrance to channeling in the elutriator; and

- filter mounts in the dust collection system need to allow for ready, facile exchange of filters while minimizing the potential for air leaks in this area. A modification incorporated into the prototype device to achieve both requirements was to design aluminum mounts that are tapered such that the shaved bottom half of commercially available filter cassettes fit snugly and can be glued in place (to prevent air leaks at this joint). These can then be fitted with filters and joined to the matching top halves of commercially available cassettes. This feature takes good advantage of the fit between the two plastic halves of a commercial filter cassette, which are designed to join with minimal leakage while allowing for rapid exchange of filters. The bottom half of the aluminum filter mounts are sealed into the slide mechanisms on the prototype apparatus with o-rings.

The following figures are included in this package:

- Figure A-13 indicates the overall assembly of the prototype dust generator;

- Figure A-14 indicates the details of the prototype tumbler assembly;

- Figure A-15 indicates the details of the prototype vertical elutriator;

- Figure A-16 is a vertical cross-section of the top of the prototype vertical elutriator indicating the relationship between the elutriator openings, the isokinetic sampling tube, and the slide mechanisms of the dust collection system;

- Figure A-17 indicates the details of the isokinetic sampling tube;

- Figure A-18 indicates the details of the prototype slide mechanism of the dust collection assembly; and

- Figure A-19 indicates the details of the prototype filter cassette mounts.

FIGURE A-13 OVERALL PROTOTYPE DUST GENERATOR ASSEMBLY

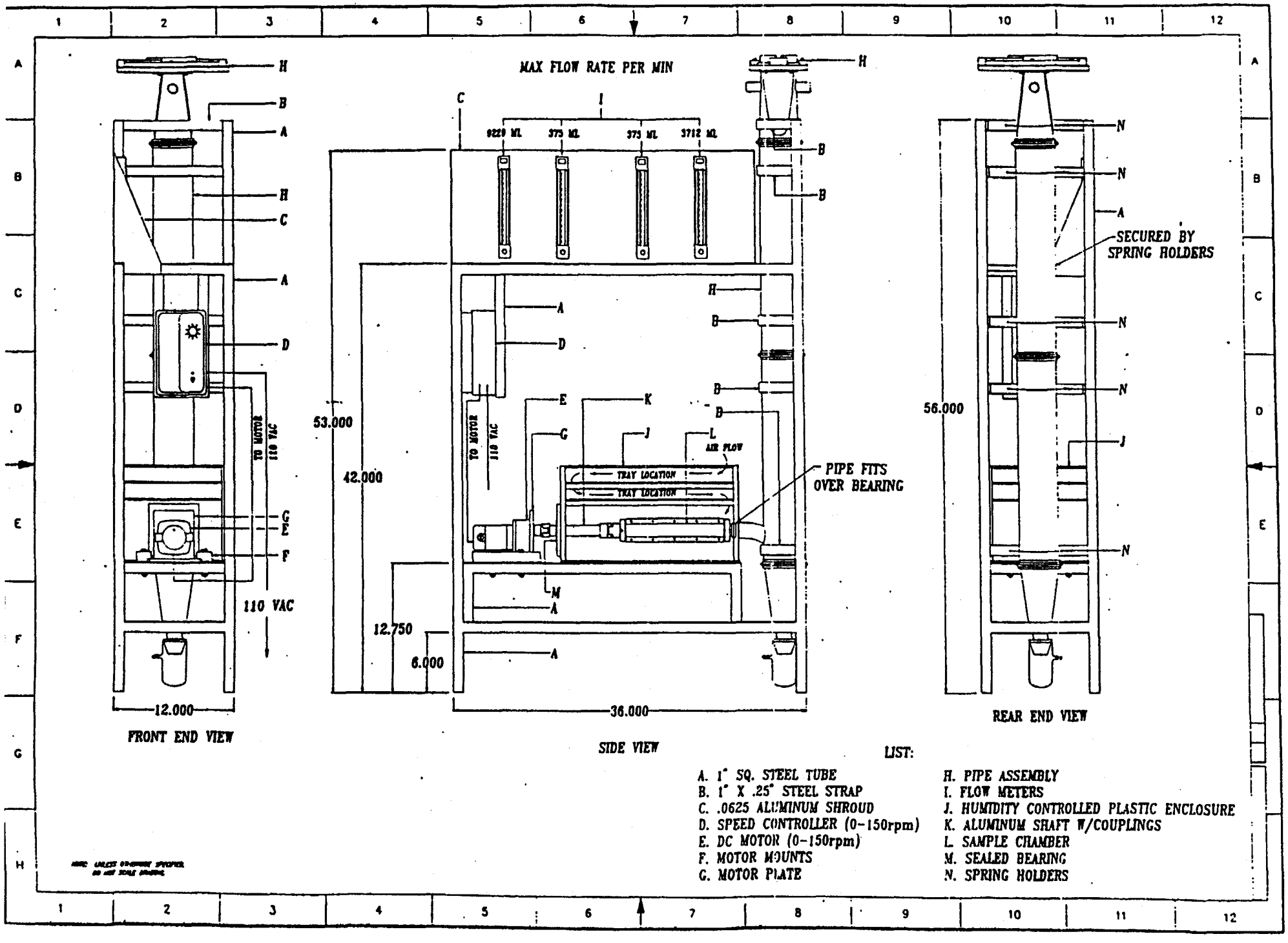


FIGURE A-14 PROTOTYPE TUMBLER ASSEMBLY DETAIL

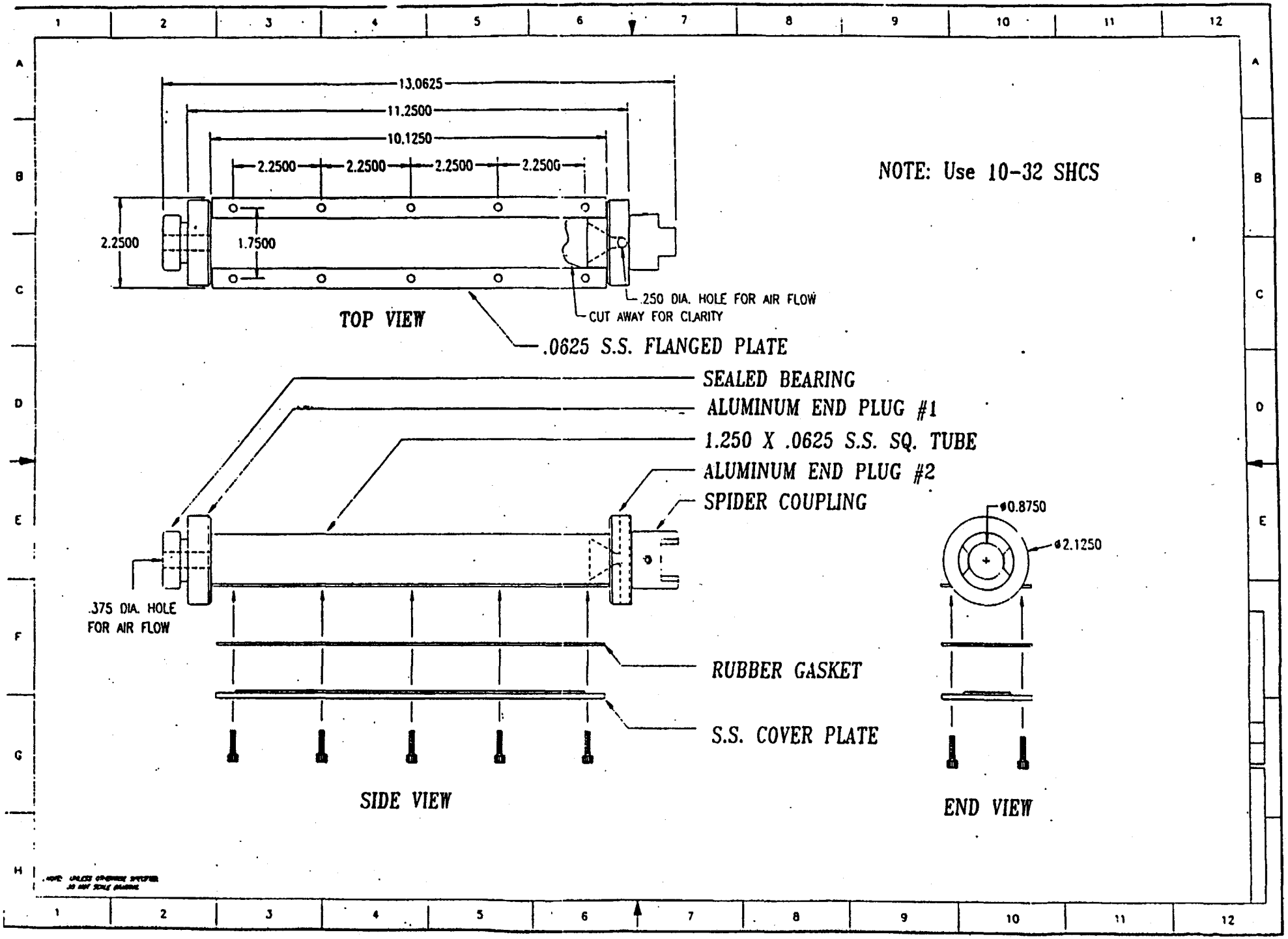


FIGURE A-15 PROTOTYPE VERTICAL ELUTRIATOR DETAIL

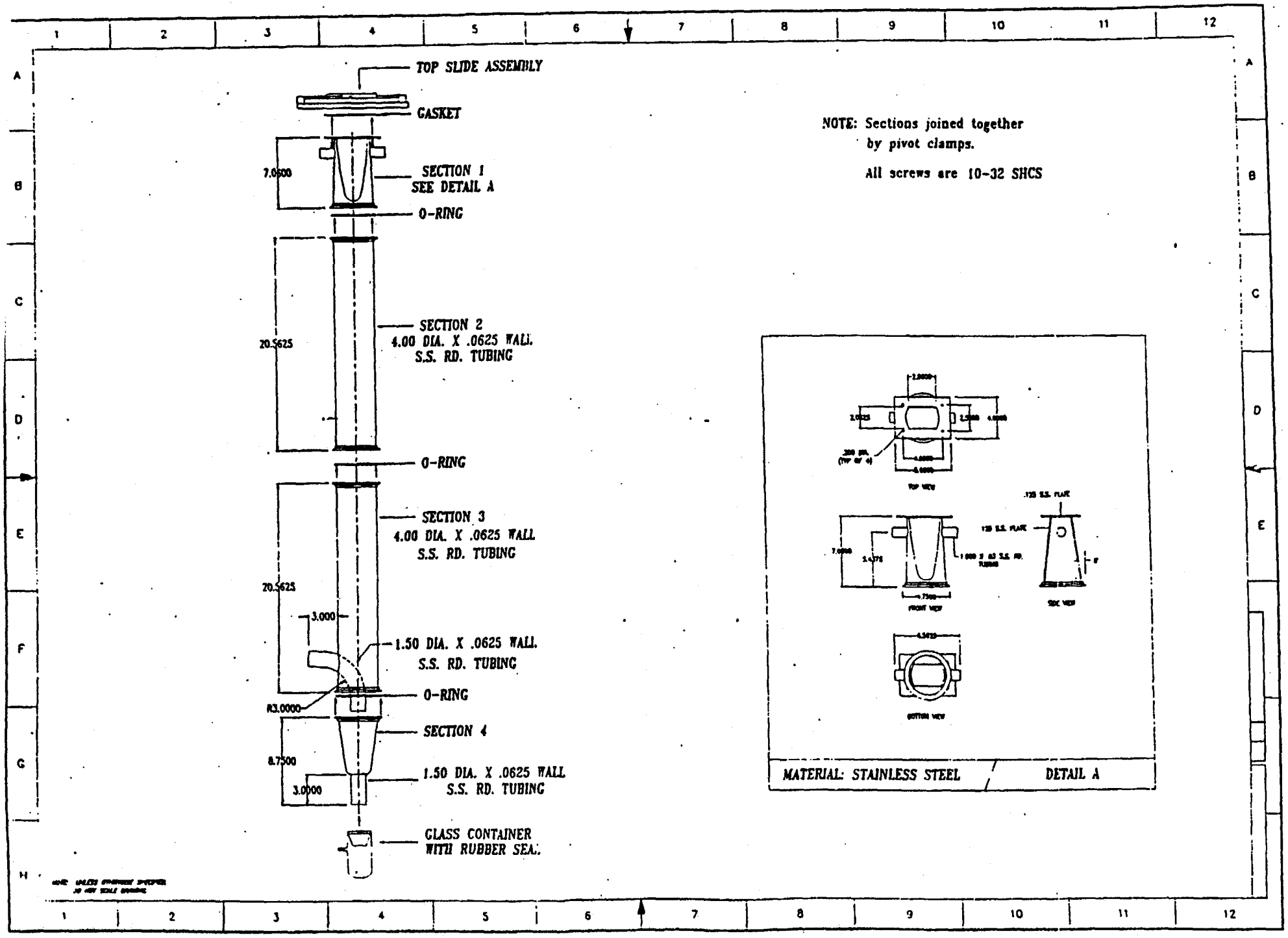


FIGURE A-16

SECTION OF ELUTRIATOR TOP WITH EXTENSION TUBE

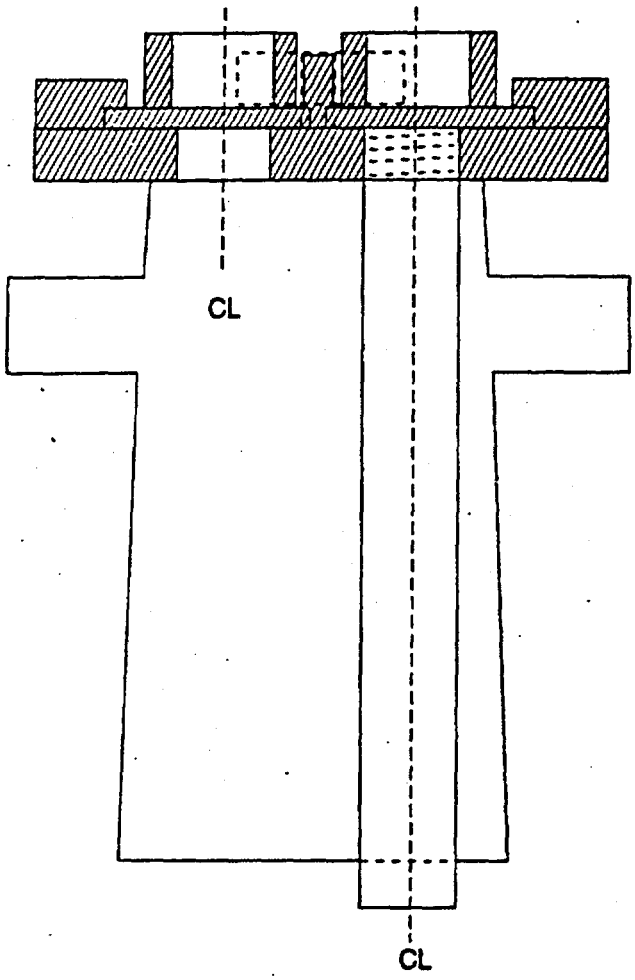


FIGURE A-17

LONGITUDINAL & TRANSVERSE SECTIONS OF ISOKINETIC SAMPLING TUBE

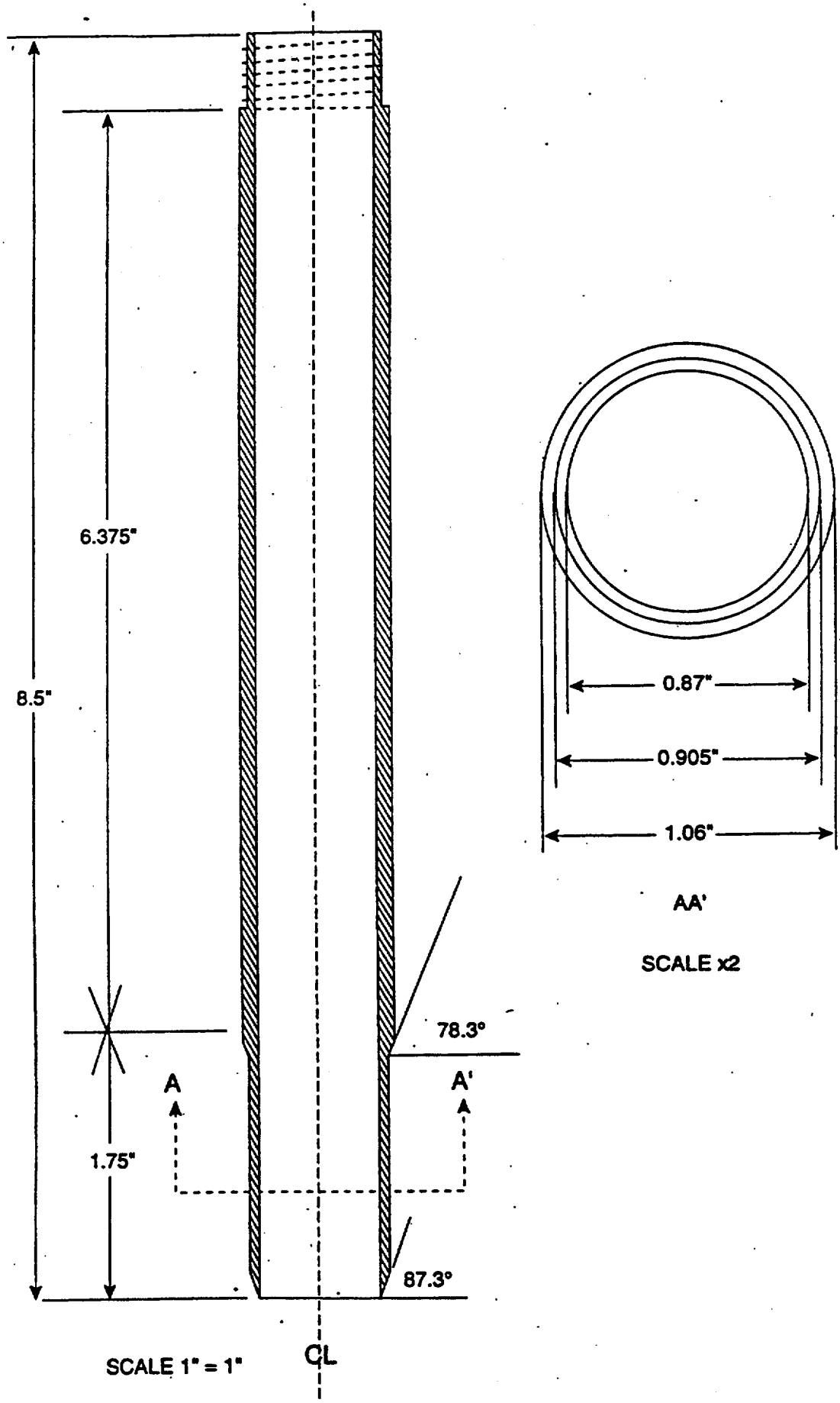
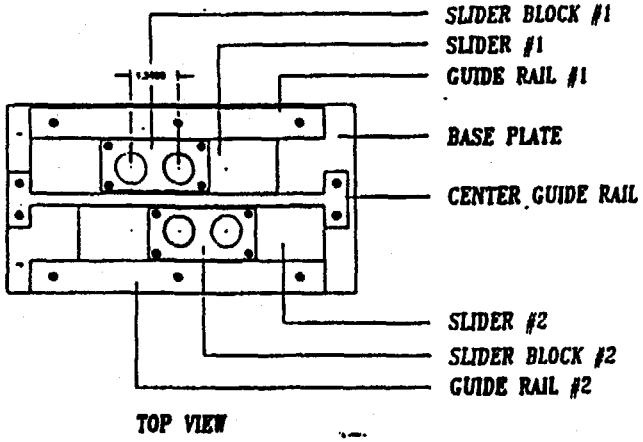
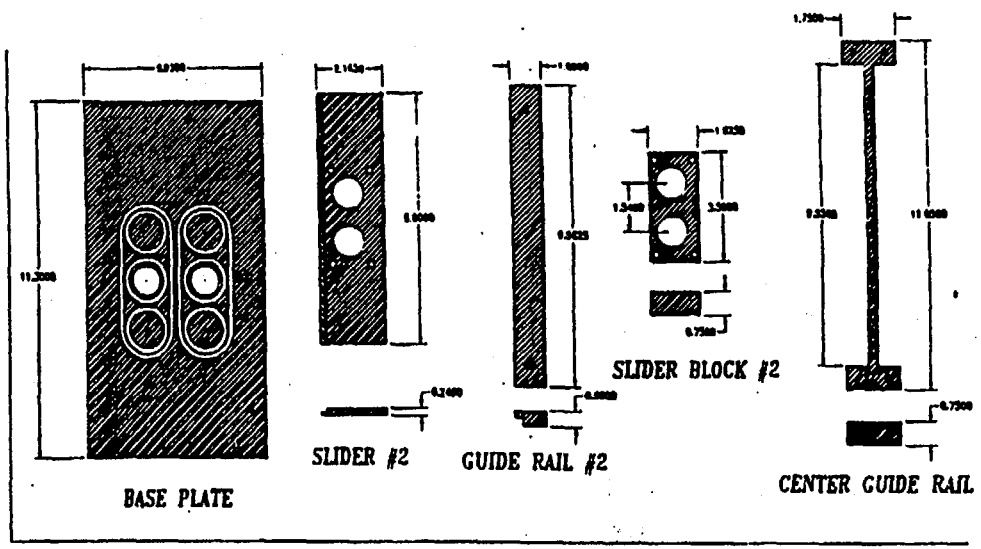
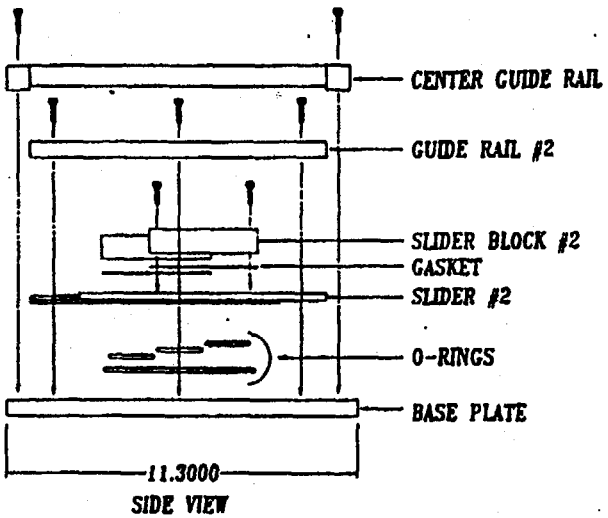
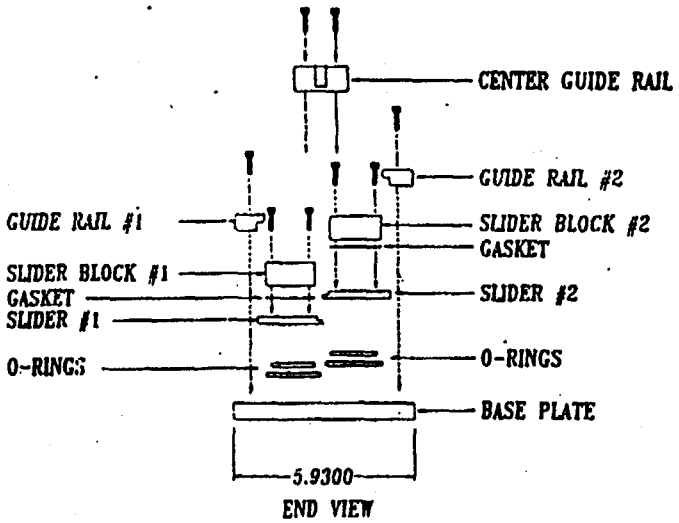


FIGURE A-18 PROTOTYPE SLIDE MECHANISM DETAIL







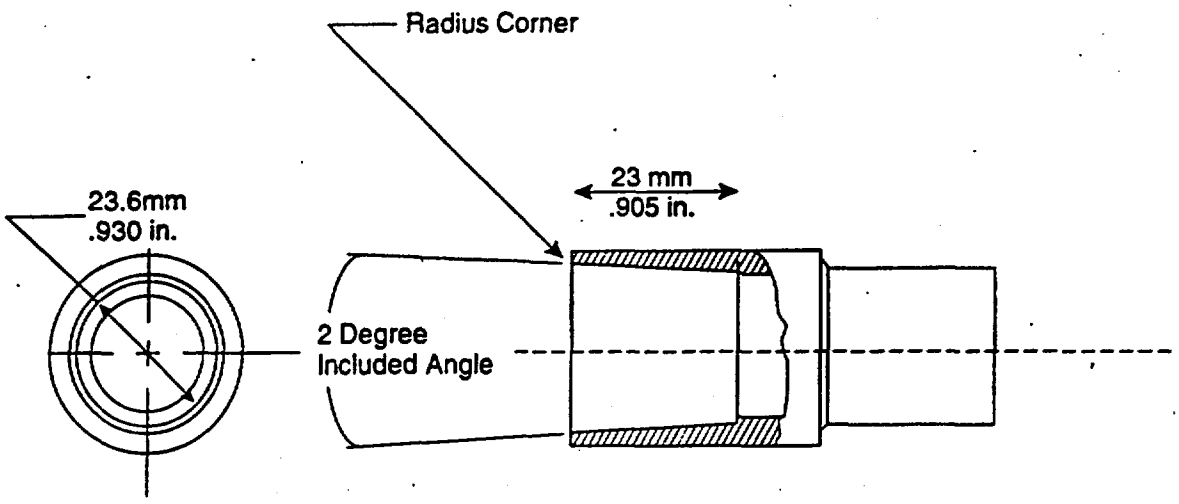


TOP SLIDE ASSEMBLY

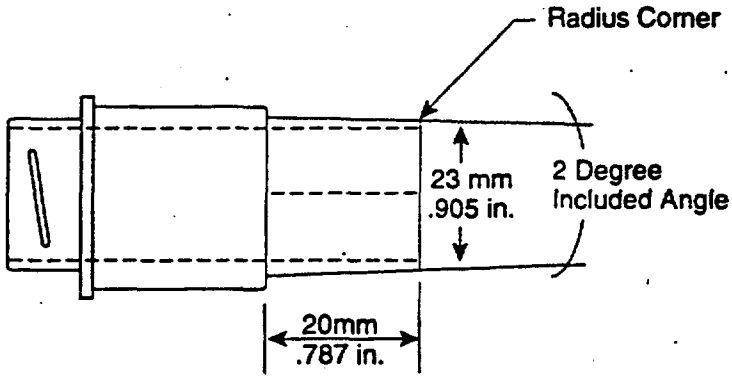
MATERIAL: ALUMINUM

FIGURE A-19

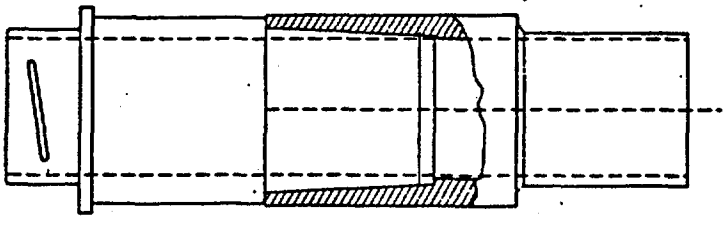
DETAIL OF PROTOTYPE FILTER CASSETTE MOUNTS



ALUMINUM CASSETTE HOLDER, TOP MODIFIED WITH INTERNAL TAPER AS SHOWN



25MM MILLIPORE CASSETTE, BOTTOM MODIFIED WITH EXTERNAL TAPER AS SHOWN



CASSETTE AND HOLDER SHOWN JOINED TO CREATE A TAPER LOCK SEAL

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Appendix 17

(EPA 600/4-84-043) Analytical Method for Determination of Asbestos Fibers in Water

PB83-260471

ANALYTICAL METHOD FOR DETERMINATION OF ASBESTOS FIBERS IN WATER

by

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Contract 68-03-2717

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16. ABSTRACT

An analytical method for measurement of asbestos fiber concentration in water samples is described. Initially, the water sample is treated with ozone gas and ultraviolet light to oxidize suspended organic materials. The water sample is then filtered through a 0.1 micrometer pore size capillary-pore polycarbonate filter, after which the filter is prepared by carbon extraction replication for examination in a transmission electron microscope (TEM). Fibers are classified using selected area electron diffraction and energy dispersive X-ray analysis. Measurement of characteristic features on a recorded and calibrated selected area electron diffraction pattern is specified for precise identification of chrysotile. Quantitative determination of the chemical composition, and quantitative interpretation of at least one calibrated zone azis selected area diffraction pattern are specified for precise identification of amphibole. Amphibole identification procedures and generation of the standard reporting format specified for the fiber count results are achieved using two computer programs that are integral to the analytical method.

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FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure specific pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of environmental contaminants, the Analytical Chemistry Branch develops and assesses new techniques for identifying and measuring chemical constituents of water and soil.

A 3-year study was conducted to develop improvements in the analytical method for determination of asbestos fiber concentrations in water samples. The research produced an improved sample preparation and analysis methodology, a rapid screening technique to reduce analysis cost, and a new reference analytical method for asbestos in water. The analytical method for determining asbestos fibers in water is perceived as representing the current state-of-the-art.

William T. Donaldson
Acting Director
Environmental Research Laboratory
Athens, Georgia

PREFACE

The Preliminary Interim Method for Determining Asbestos in Water was issued by the U.S. Environmental Protection Agency's Environmental Research Laboratory in Athens, Georgia. The method was based on filtration of the water sample through a sub-micrometer pore size membrane filter, followed by preparation of the filter for direct examination and counting of the fibers in a transmission electron microscope. Two alternative techniques were specified: one in which a cellulose ester filter was prepared by dissolution in a condensation washer; and another known as the carbon-coated Nuclepore[R] technique which used a polycarbonate filter. In January 1980 the method was revised (EPA-600/4-80-005) to eliminate the condensation washer approach, and a suggested statistical treatment of the fiber count data was incorporated.

The analytical method published here is a further refinement of the revised interim method. Major additions include the introduction of ozone-ultraviolet light oxidation prior to filtration, complete specification of techniques to be used for fiber identification and fiber counting rules, and incorporation of reference standard dispersions. A standardized reporting format has also been introduced. The major deletion is the low temperature ashing technique for samples high in organic material content; ashing is not required for the analysis of drinking water and drinking water supplies when samples are treated using the ozone-ultraviolet oxidation technique. The "field-of-view" approach for examination also has been deleted from the method. If a sample is too heavily loaded for examination of entire grid openings, a more reliable result is obtained by preparation of a new filter using a smaller volume of water.

ABSTRACT

An analytical method for measurement of asbestos fiber concentration in water samples is described. Initially, the water sample is treated with ozone gas and ultraviolet light to oxidize suspended organic materials. The water sample is then filtered through a 0.1 μm pore size capillary-pore polycarbonate filter, after which the filter is prepared by carbon extraction replication for examination in a transmission electron microscope (TEM). Fibers are classified using selected area electron diffraction (SAED) and energy dispersive X-ray analysis (EDXA). Measurement of characteristic features on a recorded and calibrated SAED pattern is specified for precise identification of chrysotile. Quantitative determination of the chemical composition, and quantitative interpretation of at least one calibrated zone axis SAED pattern are specified for precise identification of amphibole. Amphibole identification procedures and generation of the standard reporting format specified for the fiber count results are achieved using two computer programs which are integral to the analytical method.

This analytical method is a further development of the interim method issued in 1980, and incorporates results of research performed under Contract 68-03-2717 under sponsorship of the U.S. Environmental Protection Agency. This report covers a period from October 1978 to September 1981 and the work was completed as of September 1981.

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ANALYTICAL METHOD FOR DETERMINATION OF ASBESTOS FIBERS IN WATER

1. SCOPE AND APPLICATION

1.1 This method is applicable to drinking water and drinking water supplies, and should be used when the best available analytical procedure is required.

1.2 The method determines the numerical concentration of asbestos fibers, the length and width of each fiber, and the estimated mass concentration of asbestos in the water. Fiber size and aspect ratio distributions are also determined.

1.3 The method permits, if required, identification of all mineral fibers found in water. In particular, chrysotile can be distinguished from the amphiboles, and fibers of specific amphiboles can be identified.

1.4 The analytical sensitivity which can be achieved depends primarily on the amount of other particulate matter which is present in the sample. This limits the proportion of the sample which can be mounted for examination in the electron microscope. In drinking water which meets the AWWA turbidity criterion of 0.1 NTU, an asbestos concentration of 0.01 million fibers per liter (MFL) can be detected. The contamination level in the laboratory environment may degrade the sensitivity. The analytical sensitivity for the determination of mass concentration is a function of the preceding parameters and also depends on the size distribution of the fibers. In low turbidity drinking water the analytical sensitivity is usually of the order of 0.1 nanogram per liter (ng/L).

1.5 It is beyond the scope of this document to provide detailed instruction in electron microscopy, electron diffraction, crystallography or X-ray fluorescence techniques. It is assumed that those performing this analysis will be sufficiently knowledgeable in these fields to understand the specialized techniques involved.

2. SUMMARY OF METHOD

Water collected in a polyethylene or glass container is treated with ozone and ultraviolet light to oxidize organic matter. After mild ultrasound treatment to disperse the fibers uniformly, a known volume of the water is filtered through a 0.1 micrometer (μm) pore size Nuclepore[R] polycarbonate filter. A carbon coating is then applied in

vacuum to the active surface of the filter. The carbon layer coats and retains in position the material which has been collected on the filter surface. A small portion of the carbon-coated filter is placed on an electron microscope grid and the polycarbonate filter material is removed by dissolution in an organic solvent. The carbon film containing the original particulate, supported on the electron microscope grid, is then examined in a transmission electron microscope (TEM) at a magnification of about 20,000. In the TEM, selected area electron diffraction (SAED) is used to examine the crystal structure of a fiber, and its elemental composition is determined by energy dispersive X-ray analysis (EDXA).

Fibers are classified according to the techniques which have been used to identify them. A simple code is used to record for each fiber the degree to which the identification attempt was successful. The fiber classification procedure is based on successive inspection of the morphology, the selected area electron diffraction pattern, and the qualitative and quantitative energy dispersive X-ray analyses. Confirmation of the identification of chrysotile is only by quantitative SAED, and confirmation of amphibole is only by quantitative EDXA and quantitative zone axis SAED.

Several levels of analysis are specified, three for chrysotile and four for amphibole, defined by the most specific fiber classification to be attempted for all fibers. The procedure permits this target classification to be defined on the basis of previous knowledge, or lack of it, about the particular sample. Attempts are then made to raise the classification of all fibers to this target classification, and to record the degree of success in each case. The lengths and widths of all identified fibers are recorded. The number of fibers found on a known area of the microscope sample, together with the equivalent volume of water filtered through this area, are used to calculate the fiber concentration in MFL. The mass concentration is calculated in a similar manner by summation of the volume of the identified fibers, assuming their density to be that of the bulk material.

3. DEFINITIONS, UNITS AND ABBREVIATIONS

3.1 Definitions

Acicular - The shape shown by an extremely slender crystal with small cross-sectional dimensions.

Amphibole - A group of rock-forming ferromagnesian silicate minerals, closely related in crystal form and composition and having the general formula: $A_{2-3}B_5(Si,Al)_8O_{22}(OH)_2$, where A = Mg, Fe^{+2}, Ca, Na or K, and B = Mg, Fe^{+2}, Fe^{+3} or Al. Some of these elements may also be substituted by Mn, Cr, Li, Pb, Ti or Zn. It is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in

two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

Amphibole Asbestos - Amphibole in an asbestiform habit.

Analytical Sensitivity - The calculated concentration in MFL equivalent to counting of one fiber.

Asbestos - A commercial term applied to a group of silicate minerals that readily separate into thin, strong fibers that are flexible, heat resistant and chemically inert.

Aspect Ratio - The ratio of length to width in a particle.

Camera Length - The equivalent projection length between the sample and its electron diffraction pattern, in the absence of lens action.

Chrysotile - A mineral of the serpentine group: $Mg_3Si_2O_5(OH)_4$. It is a highly fibrous, silky variety of serpentine, and constitutes the most important type of asbestos.

Cleavage - The breaking of a mineral along its crystallographic planes, thus reflecting crystal structure.

Cleavage Fragment - A fragment of a crystal that is bounded by cleavage faces.

d-Spacing - The separation between identical adjacent and parallel planes of atoms in a crystal.

Diatom - A microscopic, single-celled plant of the class Bacillariophyceae, which grows in both marine and fresh water. Diatoms secrete walls of silica, called frustules, in a great variety of forms.

Electron Scattering Power - The extent to which a thin layer of a substance scatters electrons from their original path directions.

Energy Dispersive X-ray Analysis - Measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyzer system.

Eucentric - The condition when an object is placed with its center on a rotation or tilting axis.

Fibril - A single fiber, which cannot be separated into smaller components without losing its fibrous properties or appearances.

Fiber - A particle which has parallel or stepped sides, an aspect ratio equal to or greater than 3:1, and is greater than 0.5 μm in length.

Fiber Aggregate - An assembly of randomly oriented fibers.

Fiber Bundle - A fiber composed of parallel, smaller diameter fibers attached along their lengths.

Habit - The characteristic crystal form or combination of forms of a mineral, including characteristic irregularities.

Miller Index - A set of three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes.

Replication - A procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

Selected Area Electron Diffraction - A technique in electron microscopy in which the crystal structure of a small area of a sample may be examined.

Serpentine - A group of common rock-forming minerals having the formula: $(Mg,Fe)_3\ Si_2O_5(OH)_4$.

Unopened Fiber - Large diameter asbestos fiber which has not been separated into its constituent fibrils.

Zone Axis - That line or crystallographic direction through the center of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone.

3.2 Units

eV	- electron volt
g/cm^3	- grams per cubic centimeter
kV	- kilovolt
μg/L	- micrograms per liter (10^{-6} grams per liter)
μm	- micrometer (10^{-6} meter)
MFL	- Million Fibers per Liter
ng/L	- nanograms per liter (10^{-9} grams per liter)
nm	- nanometer (10^{-9} meter)

NTU - Nephelometric Turbidity Unit

ppm - parts per million

3.3 Abbreviations

AWWA - American Water Works Association

EDXA - Energy Dispersive X-ray Analysis

HEPA - High Efficiency Particle Absolute

SAED - Selected Area Electron Diffraction

SEM - Scanning Electron Microscope

STEM - Scanning Transmission Electron Microscope

TEM - Transmission Electron Microscope

UICC - Union Internationale Contre le Cancer (International Union Against Cancer)

UV - Ultraviolet

4. EQUIPMENT AND APPARATUS

4.1 Specimen Preparation Laboratory

Asbestos, particularly chrysotile, is present in small quantities in practically all laboratory reagents. Many building materials also contain significant amounts of asbestos or other mineral fibers which may interfere with analysis. It is therefore essential that all specimen preparation steps be performed in an environment where contamination of the sample is minimized. The primary requirement of the sample preparation laboratory is that a blank determination using known fiber-free water must yield a result which will meet the requirements specified in Section 6.8.1. Preparation of samples should be carried out only after acceptable blank values have been demonstrated.

The sample preparation areas should be a separate clean room with no asbestos-containing materials such as flooring, ceiling tiles, insulation and heat-resistant products. The work surfaces should be stainless steel or plastic-laminate. The room should be operated under positive pressure and have absolute (HEPA) filters, electrostatic precipitation, or equivalent, in the air supply. A laminar flow hood is recommended for sample manipulation. It is recommended that a supply of disposable laboratory coats and disposable overshoes be obtained to be worn in the clean room. This will reduce the levels of dust, and particularly asbestos, which might be transferred inadvertently by the operator into the

clean area. Normal electrical and water services are required. An air extract (fume hood) is required to remove surplus ozone from the area near the ozone generator.

4.2 Instrumentation Requirements

4.2.1 Transmission Electron Microscope

A transmission electron microscope having an accelerating potential of a minimum of 80 kV, a resolution better than 1.0 nm, and a magnification range of 300 to 100,000 is required. The ability to obtain a direct screen magnification of at least 20,000 is necessary. An overall magnification of about 100,000 is necessary for inspection of fiber morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. It is also required that the viewing screen be calibrated (as shown in Figure 1) with concentric circles and a millimeter scale such that the lengths and widths of fiber images down to 1 mm width can be measured in increments of 1 mm.

For Bragg angles less than 0.01 radians the instrument must be capable of performing selected area electron diffraction from an area of 0.6 μm^2 or less, selected from an in-focus image at a screen magnification of 20,000. This performance requirement defines the minimum separation between particles at which independent diffraction patterns can be obtained from each. The capability of a particular instrument may normally be calculated using the following relationship:

$$A = \frac{\pi}{4}\left(\frac{D}{M} + 2000\ C_s\theta^3\right)^2$$

where: A = Effective SAED area in μm^2

D = Diameter of SAED aperture in μm

M = Magnification of objective lens

C_s = Objective lens spherical aberration coefficient in mm

θ = Maximum Bragg angle in radians

Although almost all instruments of current manufacture meet these requirements, many older instruments which are still in service do not. It is obviously not possible to reduce the area of analysis indefinitely by use of apertures.

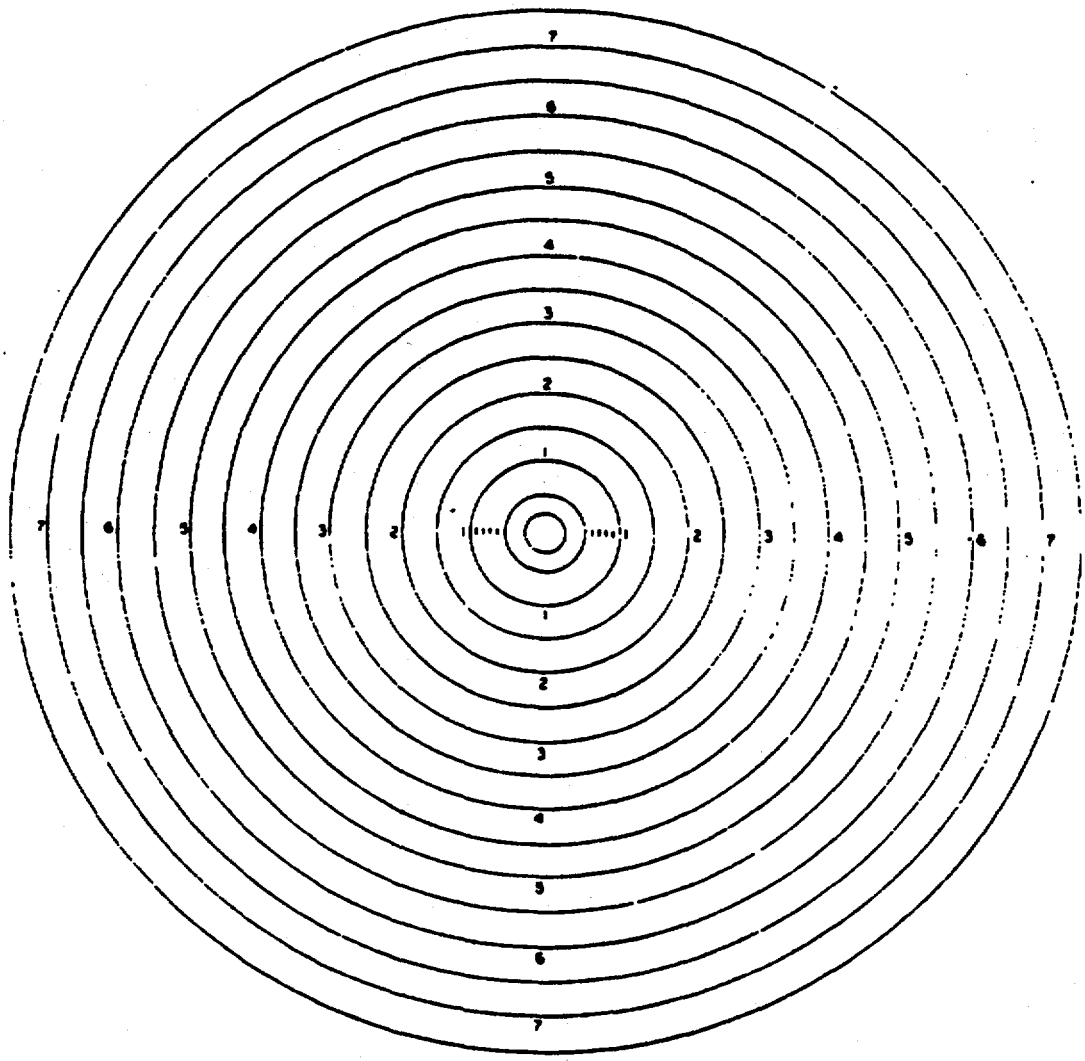


Figure 1. Calibration Markings on TEM Viewing Screen.

smaller in diameter than those specified by the manufacturer, since there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

If zone axis SAED analyses are to be performed, it is required that the electron microscope be fitted with a goniometer stage which permits either a 360° rotation combined with tilting through at least $+30^{\circ}$ to -30°, or tilting through at least $+30^{\circ}$ to -30° around two perpendicular axes in the plane of the sample. The work is greatly facilitated if the goniometer permits eucentric tilting.

It is also essential that the electron microscope have an illumination and condenser lens system capable of forming an electron probe smaller than 100 nm in diameter.

Use of an anti-contamination trap around the specimen is recommended if the required instrumental performance is to be obtained.

4.2.2 Energy Dispersive X-ray Analyzer

An energy dispersive X-ray analyzer is required. Since the performance of individual combinations of equipment is critically dependent on a number of geometrical factors, the required performance of the combination of electron microscope and X-ray analyzer is specified in terms of the measured X-ray intensity from a small diameter fiber, using a known electron beam diameter. X-ray detectors are generally least sensitive in the low energy region, and so measurement of sodium in crocidolite is selected as the performance criterion. The combination of electron microscope and X-ray analyzer must yield a background-subtracted NaKα peak integral count rate of more than 1 count per second (cps) from a 50 nm diameter fiber of UICC crocidolite irradiated by a 100 nm diameter electron probe at an accelerating potential of 80 kV. The equivalent peak/background ratio should exceed 1.0.

The EDXA equipment must provide the means for subtraction of the background, identification of elemental peaks, and calculation of net peak areas.

4.2.3 Computer

Many repetitive numerical calculations are necessary, and these can be performed conveniently by relatively simple computer programs. For analyses of zone axis diffraction pattern measurements, a computer facility with minimum available memory of 64K words is required to accommodate

the more complex programs involved. Suggested program listings for standardized data reporting and fiber identification routines are included as part of this analytical procedure. (Appendices A and B).

4.2.4 Vacuum Evaporator

A vacuum evaporator capable of producing a vacuum better than 10^{-4} Torr (0.013 Pa) is required for vacuum deposition of carbon on to the polycarbonate filters. A sample holder is desirable which allows a 51 x 75 mm glass microscope slide to be tilted and rotated during the coating procedure. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum evaporator may also be used for deposition of the thin film of gold, or other reference material, required on electron microscope samples for calibration of electron diffraction patterns. For gold deposition, a sputter coater may allow better control of the process, and is therefore recommended.

4.2.5 Ozone Generator

An ozone generator, in combination with ultraviolet light irradiation, is used for the oxidation of organic material in water samples. This procedure is necessary on all water samples. The generator should be capable of generating at least 400 g of ozone per day at a concentration of at least 1% by weight when supplied with dry oxygen. The ozone generator Model GL-1 (PCI Ozone Corporation, 1 Fairfield Crescent, West Caldwell, New Jersey 07006) or equivalent has been found to meet the requirements of this analytical technique.

4.3 Apparatus, Supplies and Reagents

4.3.1 Gas Supply to Ozone Generator

The ozone generator can be supplied by either compressed air or oxygen. The input gas must be regulated to the pressure specified by the generator manufacturer. It is recommended that oxygen be provided in order to reduce the possibility of acid formation in the sample.

4.3.2 Gas-Line Drying Tube

The ozone generator operates more efficiently when supplied with dry oxygen. An in-line drying tube. filled with a desiccant, followed by a 0.2 µm pore size polytetrafluoroethylene filter to prevent particulate from the desiccant entering the ozone generator is recommended.

A stainless steel pressure filtration assembly (Millipore Corporation, Bedford MA 01730, Cat. No. XX40 047 00) with a 0.2 µm pore size Fluoropore[R] filter (Millipore Corporation, Cat. No. FGLP 047 00) in the normal filter position and silica gel in the reservoir have been found to be satisfactory for this purpose.

4.3.3 In-Line Gas Filtration Assembly

A filter is placed in the ozone line immediately before the gas enters the sample. A 25 mm stainless steel gas line filter holder (Millipore Corporation, Cat. No. XX40 025 00) or equivalent with a 0.2 µm pore size Fluoropore filter (Millipore Corporation, Cat. No. FGLP 025 00) or equivalent is used in each ozone supply line to ensure that the ozone entering the sample is particle-free.

4.3.4 Ultraviolet Lamp

A submersible short wavelength (254 nm) ultraviolet lamp is required for the ozone-UV oxidation treatment of water samples. A 6 inch Pen-Ray[R] ultraviolet lamp (Part No. 90-0004-11) and power supply model SCT-4 (Ultra-Violet Products Inc., 5100 Walnut Grove Avenue, San Gabriel, California 91778) or equivalent have been found to meet the requirements of this analytical technique.

4.3.5 Source of Known Fiber-Free Water

For blank determinations, final washing of analytical equipment, and dilution of some samples, a source of water which is free of both particles and fibers is required. Fresh double-distilled water from a glass distillation apparatus (MEGA-PURE[TM] manufactured by Corning and available from all authorized Corning Laboratory Supply Dealers) or equivalent is preferable, and has been found to meet this requirement. De-ionized water, filtered through a 0.1 µm pore size Nuclepore polycarbonate filter has also been found to be satisfactory, but the filtration assembly itself tends to contribute some particles to the filtrate.

4.3.6 Filtration Apparatus

The water sample is filtered through a membrane filter of either 47 mm diameter or 25 mm diameter. The filtration assembly should be chosen to suit the size of filter in use. A glass frit support is required in order to obtain a uniform deposit on the filter. The reservoir must be easily cleaned in order to prevent sample cross-contamination. A 47 mm analytical filter holder (Millipore Corporation, Cat. no. XX10 047 00) or a 25 mm analytical filter holder (Millipore Corporation, Cat. No. XX10 025 00)

or equivalent has been found to be suitable. When using the larger diameter equipment it is necessary to filter proportionately larger volumes of water.

4.3.7 Filtration Manifold

When a number of samples are to be filtered, several filtration units can be operated simultaneously from a single vacuum source by using a multiple port filtration manifold (Millipore Corporation, Cat. No. XX26 047 35) or equivalent. The manifold should include valves to permit each port to be opened or closed independently.

4.3.8 Vacuum Pump

A pump is required to provide a vacuum of 20 kPa for the filtration of water samples. A water jet pump (Edwards High Vacuum Inc., Grand Island, NY 14072, Cat. No. 01-C046-01-000-female connection or 01-C039-01-000-male connection) or equivalent has been found to provide sufficient vacuum for a 3-port filtration manifold and also incorporates a non-return valve to prevent back-streaming.

4.3.9 Membrane Filters

The diameters of the membrane filters should be matched to the diameters of the filtration apparatus in use. For filtration of water samples, two types of filters are required:

- polycarbonate capillary-pore membrane filters, 0.1 µm pore size (Nuclepore Corporation, 7035 Commerce Circle, Pleasanton, California 94566) or equivalent, are used to collect the suspended material from a water sample.

- mixed esters of cellulose membrane filters, 0.45 µm pore size Type HA (Millipore Corporation, Bedford, MA 01730) or equivalent, are used as a support filter placed between the glass frit of the filtration apparatus and the polycarbonate filter.

4.3.10 Jaffe Washer

A Jaffe Washer is used for dissolution of Nuclepore filters. Several designs of Jaffe Washer have been used which are modifications of the original design. Provided that the polycarbonate filter can be completely dissolved, and that the materials used in the different designs of washer are demonstrably free of mineral fiber contamination, the precise design is not considered

important. Because of recent changes in the formulation of Nuclepore polycarbonate filters which have degraded their solubility in chloroform, a more complex dissolution procedure may be required. The additional steps in the preparation are more easily completed if the original washer design is followed. This original design is illustrated in Figure 5A. Figure 5B shows samples being placed on a Jaffe Washer of this design. Alternatively, methylene chloride may be substituted for chloroform, but because this has a higher vapor pressure it is then necessary to ensure that the Jaffe Washer is tightly sealed to avoid excessive evaporation.

4.3.11 Condensation Washer

A condensation washer may be useful if TEM specimens are required more quickly than is possible if the Jaffe Washer is used alone to dissolve some batches of Nuclepore polycarbonate filters. A condensation washer consists of a system with controlled heating, controlled refluxing, and a cold finger for holding the electron microscope sample grids. Figure 6 shows one model of the condensation washer (Cat. No. 16950, Ladd Research Industries, Inc., P.O. Box 901, Burlington, Vermont 05401) which has been found satisfactory.

4.3.12 Electron Microscope Grids

Specimen grids of 200 mesh and 3 mm diameter are required in both copper and gold. The grid openings should be approximately 80 μm square. The fiber count result obtained is proportional to the mean area of the openings examined. Therefore, it is important that an accurate measurement of the dimensions of each grid opening can be obtained. Since there is a wide range of quality in the available copper specimen grids, these should be examined carefully to establish the degree of uniformity of both the grid openings and the grid bars. Copper specimen grids Cat. No. SPI #3020C and 3020T, SPI Supplies Division of Structure Probe, Inc., P.O. Box 342, West Chester, PA 19380, or equivalent, have been found to meet the requirements. In addition, these grids have a mark at the center opening. This reference can be used to indicate the location of openings which have been examined. Alternatively, finder grids may be substituted if re-examination of specific grid openings is to be required. Gold specimen grids Cat. No. 21612, Ernest F. Fullam, Inc., P.O. Box 444, Schenectady, N.Y. 12301, or equivalent, have been found to meet the requirements for gold grids.

4.3.13 Ultrasonic Bath

An ultrasonic bath is required for dispersing particulate in sample containers and for general cleaning of equipment. The size of unit selected is unimportant, and should be related to the volume of work in progress. Bransonic Model B-52 (Branson Cleaning Equipment Company, Parrott Drive, Shelton Connecticut 06484) has a power of 200 watts at a frequency of 50 kHz and has been found to meet the requirements.

4.3.14 Carbon Rod Electrodes

Spectrochemically pure carbon rods are required for use in the vacuum evaporator during carbon coating of filters. Type AGKSP, National Spectroscopic Electrodes, manufactured by Union Carbide, or equivalent, have been found to meet the requirements.

4.3.15 Carbon Rod Sharpener

This device is used to sharpen the carbon rods to a neck of 3.6 mm long and 1.0 mm diameter. The use of necked rods, or equivalent, allows the carbon layer to be applied with a minimum of heating of the polycarbonate membrane. The sharpener, Cat. No. 1204, Ernest F. Fullam, Inc., Schenectady, N.Y. 12301, or equivalent, meets the requirements.

4.3.16 Standards

a) Reference Standard Fiber Suspensions. Glass ampoules of stable concentrated chrysotile or amphibole fiber dispersions, (Electron Optical Laboratory, Ontario Research Foundation, Sheridan Park, Mississauga, Ontario, Canada L5K 1B3) can be used to establish quality assurance in analytical programs. The reference suspensions of known mass and numerical fiber concentrations are used to generate control samples for inclusion in analytical programs.

b) Reference Silicate Mineral Standards on TEM Grids. For calibration of the EDXA system, reference silicate mineral standards are required (Electron Optical Laboratory, Ontario Research Foundation, Sheridan Park, Mississauga, Ontario, Canada L5K 1B3).

c) Asbestos Bulk Material. Chrysotile (Canadian), Chrysotile (Rhodesian), Crocidolite, Amosite. UICC (Union Internationale Contre le Cancer) Standards. Available from Duke Standards Company, 445 Sherman Avenue, Palo Alto, CA 94306.

4.3.17 Carbon Grating Replica

A carbon grating replica with about 2000 parallel lines per mm (Cat. No. 10020, Ernest F. Fullam, Inc., Schenectady, N.Y. 12301) or equivalent is required for calibration of the magnification of the TEM.

4.3.18 Chloroform

Spectrograde chloroform, distilled in glass (preserved with 1% (v/v) ethanol, Burdick & Jackson Laboratories Inc., Muskegon, Michigan 49442) or equivalent, is required for the dissolution of the polycarbonate filters.

4.3.19 Petri Dishes

Disposable plastic petri dishes (Millipore Corp. Cat. No. PD 10 047 00) or equivalent, are useful for storage of sample filters and specimen grids. If charge build-up on these dishes is experienced, it has been found that rinsing them with a weak detergent solution will reduce the problem.

4.3.20 Quartz Pipets

Quartz pipets are used to bubble ozone through the liquid sample. These pipets are formed by heating quartz tubing and drawing it to a tip of approximately 0.35 mm inside diameter. The pipet should be sufficiently long to reach within 1 inch of the bottom of the sample bottle, to create good mixing of the liquid during oxidation.

4.3.21 Mercuric Chloride Solution

A 0.01 molar solution of mercuric chloride may be required for preservation of water samples. This is prepared by dissolving 2.71 g of reagent grade mercuric chloride in 100 mL of fiber-free water. The solution is then filtered twice through the same 0.1 µm pore size Nuclepore filter, using the filtration apparatus described in Section 4.3.6 and a conventional filtration flask.

4.3.22 Routine Electron Microscopy Preparation Supplies

Electron microscopy preparation supplies such as scalpels, disposable scalpel blades (curved cutting edge), double-sided adhesive tape, sharp point tweezers and specimen scissors are required. These items are available from most EM supply houses.

4.3.23 Routine Laboratory Supplies

Routine laboratory supplies and labware are required. The general supplies include a detergent for cleaning apparatus, marking pens for labelling glass and plastic apparatus, glass microscope slides, lens paper (for preparation of Jaffe Washer and lining of TEM grid storage dishes), lint free tissues. General labware includes such items as graduated cylinders, beakers of several sizes, pipets. Whenever possible, disposable plastic labware is recommended to avoid the problems of contamination from new glassware and cross-contamination between samples.

5. SAMPLE COLLECTION AND PRESERVATION

5.1 Sample Container

The sample container will be an unused, pre-cleaned, screw-capped bottle of glass or low density (conventional) polyethylene and capable of holding at least 1 liter. It is recommended that the use of polypropylene bottles be avoided since problems of particulate being released into water samples have been observed.

Ideally, water samples are best collected in glass bottles. However, glass can have significant levels of asbestos on the surfaces and therefore requires careful cleaning before use. Glass is also difficult to ship because of possible breakage through dropping or freezing. Because of these disadvantages, polyethylene bottles are more convenient to use and therefore are recommended.

The bottles should first be rinsed twice by filling approximately one third full with fiber-free water and shaking vigorously for 30 seconds. After discarding the rinse water, the bottles should then be filled with fiber-free water and treated in an ultrasonic bath for 15 minutes, followed by several rinses with fiber-free water.

It is recommended that blank determinations be made on the bottles before sample collection. The following method has been found satisfactory for these determinations. A pre-washed bottle containing approximately 800 milliliters of fiber-free water is processed as described for preparation of samples, including ozone-UV and ultrasonic treatments. When using polyethylene bottles, 1 bottle in each batch or a minimum of 1 bottle in each 24 is tested for background level. When using glass bottles, the risk of asbestos contamination from the bottle is greater and a minimum of 4 bottles in each 24 are examined for background level. Additional blanks may be desirable when sampling waters suspected of containing very low levels of asbestos, or when additional confidence in the bottle blanks is desired.

5.2 Sample Collection

It is beyond the scope of this procedure to furnish detailed instructions for field sampling; the general principles of obtaining water samples apply. However, some specific considerations apply to asbestos fibers because they are a special type of particulate matter. These fibers are small, and in water range in length from 0.1 μm to 20 μm or more.

Because of the range of sizes there may be a vertical distribution of particle sizes in large bodies of water. This distribution may vary with depth depending upon the vertical distribution of temperature, the water current pattern and the local meteorological conditions. Sampling should take place according to the objective of the analysis. If a representative sample of a water supply is required, a carefully designated set of samples should be taken representing the vertical as well as the horizontal distribution and these samples should be composited for analysis.

When sampling from a faucet, remove all hoses or fittings and allow the water to run to waste for a sufficiently long period to ensure that the sample collected is representative of fresh water. Faucets or valves should not be adjusted until all samples have been collected. If possible, sampling at hydrants and at the ends of distribution systems should be avoided.

As an additional precaution against contamination, before collection of the sample, each bottle may be rinsed several times in the source water being sampled. In the case of depth sampling in bodies of water, this rinsing may compromise the results and should be omitted.

5.3 Quantity of Sample

Two separate samples of approximately 800 milliliters each are required. An air space must be left in the bottle to allow efficient redispersal of settled material before analysis. The second bottle is stored for analysis if confirmation of the results obtained from the analysis of the first bottle is required.

5.4 Sample Preservation and Storage

Samples must be transported to the analytical laboratory as soon as possible after collection. No preservatives should be added during sampling; the addition of acids should be particularly avoided.

If the sample cannot be given ozone-UV treatment and filtered within 48 hours after arrival at the analytical laboratory, amounts (1 milliliter per liter of sample) of a pre-filtered 2.71% solution of mercuric chloride sufficient to give a final concentration of 20 ppm of mercury may be added, to prevent bacterial growth. Appropriate care should be taken when handling mercury compounds.

At all times after collection, it is recommended that the samples should be stored in the dark and refrigerated at about 5°C in order to minimize bacterial and algal growth. The samples should not be allowed to freeze, since the effects on asbestos fiber dispersions are not known.

Before the sample bottles are opened, the exterior surfaces should be thoroughly washed and then rinsed in fiber-free water to avoid inadvertent contamination of the sample by material which may be attached to the bottles.

6. PROCEDURE

6.1 Cleanliness and Contamination Control

It is most important that all glassware and apparatus be cleaned thoroughly in order to minimize the possibility of specimen contamination. All phases of the specimen preparation should be conducted in the clean room facilities or in a laminar flow hood. Glassware should be cleaned in an ultrasonic bath using a detergent solution. After this, it should be rinsed three times using fiber-free water. After drying, equipment should be stored in clean containers and covered using aluminum foil or parafilm. All glassware must be washed by the above procedure before each use.

6.2 Oxidation of Organics

Oxidation of the high molecular weight organic components in water samples prior to filtration has been found necessary if precise results are to be obtained. Asbestos fibers have an affinity for these organic materials. Three separate effects have been identified which result from this affinity and which give rise to serious errors if this oxidation is not carried out:

a) asbestos fibers associated with organic materials tend to adhere to the container walls;

b) asbestos fibers tend to aggregate with organic materials;

c) fibers embedded in organic material are not transferred to the TEM specimen.

All three effects give rise to low results. Before sub-samples are taken from the bottle it is necessary to ensure that all the particulate material is in suspension. The organic material and associated fibers must be released from the container walls. This can be achieved by treating the water sample in the original collection container using the ozone-ultraviolet (ozone-UV) technique to oxidize the organic materials. However, if a sample is known to be free of organic interferences the ozone-UV oxidation may not be required.

The equipment should be assembled as shown in Figures 2 and 3.

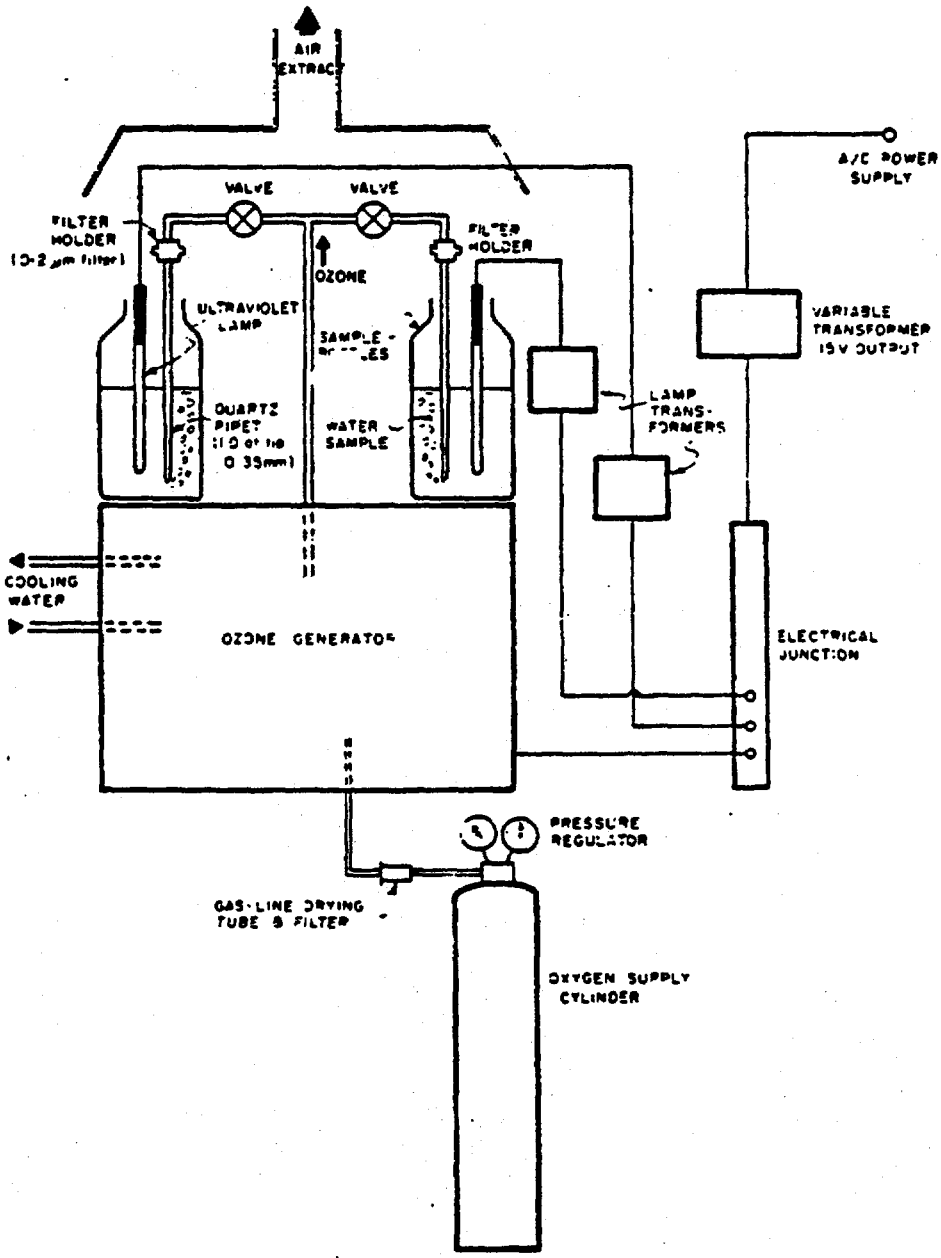


Figure 2. Diagram of Ozone-UV Equipment.

Figure 3. Ozone-UV Oxidation of Water Samples in Glass Bottles. The ozone supply line has been split into two lines to permit simultaneous oxidation of two samples. A valve and a filter holder are incorporated in each of the supply lines to the samples.

An air extract to remove surplus ozone is required. If it is necessary to check that the ozone generator is functioning within the specifications, the output can be verified by normal chemical methods. A suitable technique is to bubble the ozone through a solution of potassium iodide and to titrate the displaced iodine with sodium thiosulfate solution, using starch as an indicator.

Before the ozone-UV treatment, place each polyethylene or glass bottle containing the water sample in the ultrasonic bath for a period of 15 minutes. Mark the level of the liquid in the sample bottle using a waterproof felt marker. The quartz pipets should be thoroughly washed before each use, and installed on the ozone supply as indicated so that the tip is close to the bottom of the sample bottle. The UV lamp is also thoroughly washed and then immersed in the sample and switched on.

At an ozone concentration of 4% in oxygen, treat each sample with about 1 liter/minute of gas for approximately 3 hours. At other ozone concentrations, adjust the oxidation time so that each sample receives about 10 grams of ozone. The gas flow rate should be

sufficient to produce a mixing action in the liquid but should not splash sample out of the container. It is not easy to indicate when oxidation is complete, but this treatment as described has been found to be adequate for all water samples so far handled. When oxidation is complete, remove the UV lamp and quartz pipet, re-cap the bottle and place it in the ultrasonic bath for a period of 15 minutes. This allows particulate released from the oxidized organic materials and the container surfaces to be uniformly dispersed throughout the sample.

The water level in the bottle may have fallen, due to evaporation during the oxidation procedure. The loss of volume should be noted and can be accounted for if it is significant. The sample should be filtered immediately after it is removed from the ultrasonic bath.

6.3 Filtration

6.3.1 General

The separation of suspended particulate by filtration of the sample through a membrane filter is a critical step in the analytical procedure. The objective is to produce a Nuclepore filter on which the suspended solids from the sample are distributed uniformly, with a minimum of overlapping of particles. The volume to be filtered depends on the diameter of the filtration equipment in use, the total suspended solids content of the sample, and in some samples the volume depends on the fiber concentration present.

Table 1 shows the limitation of the analytical sensitivity as a function of the volume of water filtered. In practice, it is usually found that the concentration of suspended solids limits the filtration volume. The maximum particulate loading on the filter which can be tolerated is about 20 $\mu g/cm^2$, with an optimum value of about 5 $\mu g/cm^2$. Where the concentration of suspended solids is known, the maximum volume which can be used may be estimated. Usually, however, nothing is known about the sample and the best procedure is to prepare several filters using different volumes of the sample. It has been found that suitable filter samples display a faint coloration of the surface, and with experience over-loaded filters usually can be recognized. The determination of a suitable volume to filter is usually a matter of trial and error in the analysis of samples of relatively low total suspended solids but high asbestos concentration.

No attempt should be made to filter sample volumes less than 10 mL for 25 mm diameter equipment, and 50 mL for 47 mm diameter equipment. If smaller volumes are filtered

TABLE 1. LIMITATION OF ANALYTICAL SENSITIVITY BY VOLUME OF WATER SAMPLE FILTERED

Volume Filtered (mL)		Analytical Sensitivity[1] (Fibers/Liter)
Using 25 mm Diameter Filter[2]	Using 47 mm Diameter Filter[3]	
0.1	0.6	1.5×10^7
0.5	2.8	3.0×10^6
1.0	5.7	1.5×10^6
2.0	11	0.8×10^6
5.0	28	3.0×10^5
10	57	1.5×10^5
25	142	6.0×10^4
50	285	3.0×10^4
100	570	1.5×10^4

[1]Concentration corresponding to 1 fiber detected in 20 grid openings of nominal 200 mesh grid (approximately 80 μm square grid openings)

[2]Assuming Active Filter Area of 1.99 cm^2

[3]Assuming Active Filter Area of 11.34 cm^2

it is difficult to ensure that a uniform deposit of particulate will be obtained on the filter. Samples of high solids content, or of high fiber content, may require filtration of volumes less than these. Such samples should be diluted with fiber-free water so that the volumes filtered exceed the minima specified. Dilutions should be made by transferring a known volume of the sample to a disposable plastic beaker and making up to a known volume with fiber-free water. The mixture should be stirred vigorously before sub-sampling takes place.

6.3.2 Filtration Procedure

a) The sample must be filtered immediately after the ozone-UV and ultrasonic bath treatment. If for any reason the sample has been stored for more than a few hours after these treatments, it is recommended that ozone-UV oxidation be repeated for a short period of about 15 minutes, followed by an additional 15 minutes in the ultrasonic bath.

b) Assemble the filtration base and turn on the vacuum. The upper surface of the filtration base (both the glass frit and the ground mating surface) must be dry before the membrane filters are installed. Place a 0.45 μm pore size type HA Millipore filter on the glass frit. If the filter appears to become wet by capillary action on residual water in the glass frit it must be discarded and replaced by another filter. Place a 0.1 μm pore size Nuclepore filter, shiny side up, on top of the Millipore filter. If the Nuclepore filter becomes folded it must be discarded and replaced. The mating surface of the reservoir component of the filtration apparatus (the funnel) should be dried by shaking off any surplus water and draining on paper towel or tissue. The funnel should be positioned on the filters and firmly clamped, taking care not to disturb the filters. The vacuum should not be released until the filtration has been completed.

It is necessary to comment on the use of filtration equipment which is still wet after washing, since improper procedures at this point can very seriously compromise the results. If the glass frit is wet when the Millipore filter is applied to it, capillary action will result in some areas of the Millipore filter structure being filled by water. When the Nuclepore filter is applied to the surface of the Millipore filter

and the vacuum is applied, the differential pressure across the Millipore filter will be insufficient to overcome the surface tension of the water in the filled areas. Thus no filtration will take place through the corresponding areas of the Nuclepore filter, and a grossly non-uniform deposit of particulate will be obtained.

c) Add the required volume of sample water to the filtration funnel. Disposable plastic beakers and pipets provide a means of measuring the required sample volume without introducing problems of sample cross-contamination. The reservoir may not be sufficiently large to accommodate the total volume to be filtered. In this case more of the sample may be added during the filtration, but this should be done carefully and only when the reservoir is more than half full. In this way the addition will not disturb or affect the uniformity of particulate already deposited on the Nuclepore filter. Do not rinse the sides of the funnel, and avoid other manipulations which may disturb the particulate deposit on the filter.

d) Disassemble the filtration unit, and transfer the Nuclepore filter to a labelled, clean petri dish. Since the Nuclepore filters are more easily handled while they are still wet, it is recommended that the strip of filter to be used for TEM sample preparation should be cut as described in Section 6.4.2 before the filter is dried. Place the cover loosely over the dish to limit any deposition of dust onto the filter. Dry the filter under an infra-red heat lamp for a short time before closing the petri dish completely. Discard the Millipore filter.

6.4 Preparation of Electron Microscope Grids

Preparation of the grid for examination in the electron microscope requires a high degree of manual dexterity and is a critical step in the procedure. The objective is to replicate the filter surface by deposition of a carbon film and then to dissolve away the filter itself with a minimum of particle movement and breakage of the carbon film. The filter dissolution procedure is illustrated in Figure 4.

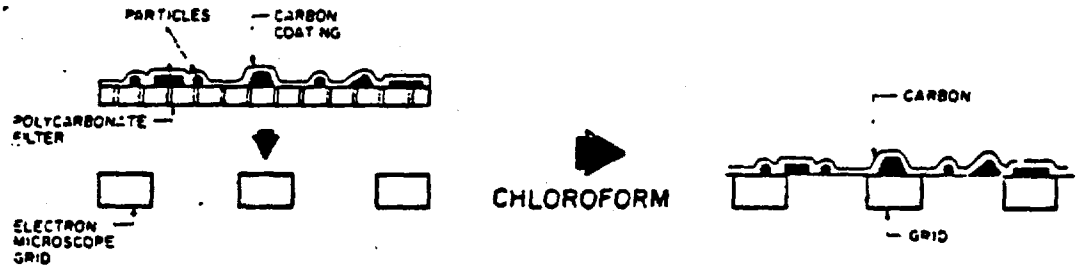


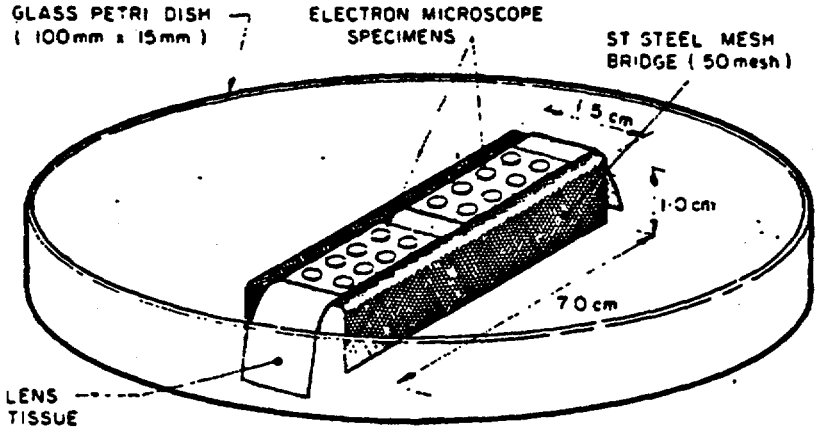
Figure 4. Nuclepore Dissolution Technique

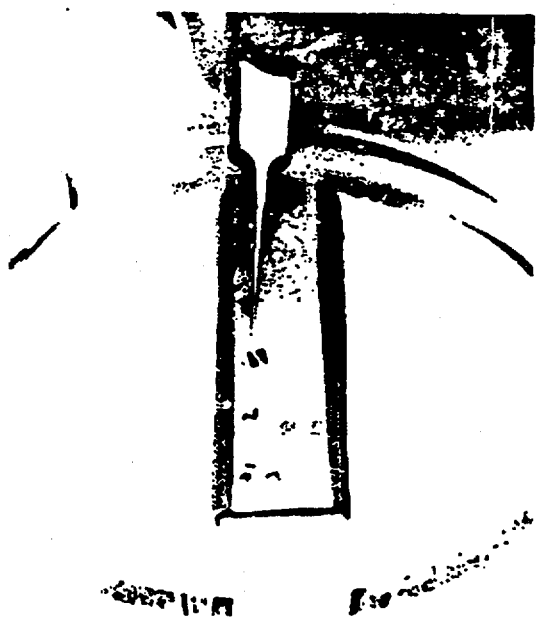
6.4.1 Preparation of Jaffe Washer

Prepare the Jaffe Washer as illustrated in Figure 5A. The stainless steel mesh is formed into a bridge slightly less than 1 cm high, and placed in a 10 cm diameter glass petri dish with a tight fitting lid. A narrow strip of lens cleaning tissue is placed over the bridge with each end of the tissue extending beyond the bridge to the base of the petri dish. The other dimensions of the stainless steel bridge and the length of the lens tissue are not critical, but those specified in Figure 5A have been found to be satisfactory. After the assembly is complete, fill the petri dish with chloroform to a level just below that of the horizontal surface of the stainless steel bridge. It may be found that the chloroform contacts the underside surface of the stainless steel mesh; this is not critical. Cover the petri dish with the lid and the Jaffe Washer is ready for use. Each time the Jaffe Washer is used, the lens tissue and solvent should be discarded and replaced with new lens tissue and fresh solvent. Appropriate precautions should be taken when handling chloroform.

6.4.2 Selection of Filter Area for Carbon Coating

Polycarbonate filters are easily stretched during handling, and cutting of areas for further preparation must be performed with great care. The best method is to use a curved edge scalpel blade to cut the filter while it is in


GLASS PETRI DISH
(100mm x 15mm)
ELECTRON MICROSCOPE
SPECIMENS
ST STEEL MESH
BRIDGE (50 mesh)
1.5 cm
1.0 cm
7.0 cm
LENS
TISSUE



the plastic petri dish. Press the scalpel point on the filter at the beginning of the desired cut, and rock the blade downwards while maintaining pressure. It will be found that a clean cut is obtained without stressing of the filter. The process should be repeated along all four directions to remove a rectangular portion from the active filtration area of the filter. This filter portion should be selected from along a diameter of the filter, and should be about 3 mm wide by a minimum of 15 mm long. Areas close to the perimeter of the active filtration area should be avoided.

6.4.3 Carbon Coating of the Nuclepore Filter

The ends of the selected filter strips should be attached to a glass microscope slide using double-sided adhesive tape. This must be performed carefully to ensure that the filter strips lie flat on the slide and are not stretched. The filter strips can be identified by using a wax pencil on the glass slide. After inserting the necked carbon rods into the vacuum evaporator, place the glass slide on the sample rotation and tilting device. The separation between the sample and the tips of the carbon rods should be about 7.5 cm to 10 cm.

If desired, the amount of carbon to be evaporated can be monitored instrumentally so that a thickness of about 30 nm to 50 nm is deposited on the filter strips. Alternatively, a porcelain fragment will serve as a simple carbon deposition monitor. Place a small drop of silicone diffusion pump oil on the surface of a clean fragment of white glazed porcelain. Locate the porcelain in the evaporation chamber with the oil droplet towards the carbon rods and at a distance from the carbon rods equal to that separating the rods from the filter strips. Carbon will not deposit on the oil drop whereas it does on the other areas of the porcelain. With experience, the correct thickness can be monitored visually by observation of the contrast between the darkened areas of the porcelain and the uncoated areas under the oil drop.

Pump down the evaporation chamber to a vacuum better than 10^{-4} Torr (0.013 Pa). Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surfaces by oil from the pumping system. Continuously rotate and tilt the glass slide holding the filter strips, while the carbon is evaporated in intermittent bursts, allowing the rods to cool between each evaporation. This procedure is necessary to avoid overheating of the filter strips. Overheating tends to cross-link the polycarbonate which then becomes difficult to dissolve in chloroform.

6.4.4 Transfer of the Filter to Electron Microscope Grids

Remove the glass slide carrying the filter strips from the evaporator, and using the technique described in 6.4.2 cut four pieces slightly less than about 3 mm x 3 mm in size from each filter strip. The square of filter should fit within the circumference of an electron microscope grid. Three of the filter pieces are to be prepared on 200 mesh copper grids, and unless the analysis is to be for chrysotile only, a fourth piece should be prepared on a 200 mesh gold grid. The specimens prepared on copper grids are used for fiber counting and most EDXA examinations. The preparation on the gold grid is intended for EDXA work on fibers containing sodium.

Place a piece of the carbon-coated filter, carbon side up, on to the shiny side of an electron microscope grid. Using fine tweezers, pick up the grid and filter together and place quickly on to the chloroform-saturated lens tissue in the Jaffe Washer, as shown in Figure 5B. It is important that the sample be placed on the lens tissue quickly, since hesitation while the sample is exposed to chloroform vapor will cause it to curl. This is a simplified technique which does not involve dropping of chloroform on to the samples.

Some components of the polycarbonate filters now available dissolve in chloroform only very slowly. Consequently, the grids must be left in the Jaffe Washer for longer than 4 days, and the solvent must be replaced every day. Depending on the particular lot number of the filters, even this period may be insufficient to yield satisfactory grids clear of undissolved plastic. In this event, or if a more rapid sample preparation is desired, after a minimum period of 30 minutes in the Jaffe Washer the lens paper supporting the grids may be transferred to the condensation washer as illustrated in Figure 6. The condensation washer should then be operated for a period of between 30 and 60 minutes, after which the grids will have been cleared of residual plastic. The rate of condensation in the washer is not critical, provided that chloroform drips rapidly from the cold finger for the whole of the washing period and the condensation level is above the samples.

During the dissolution, it is recommended that the grids not be allowed to dry since this has been found to greatly increase the time required for complete dissolution of the polycarbonate.

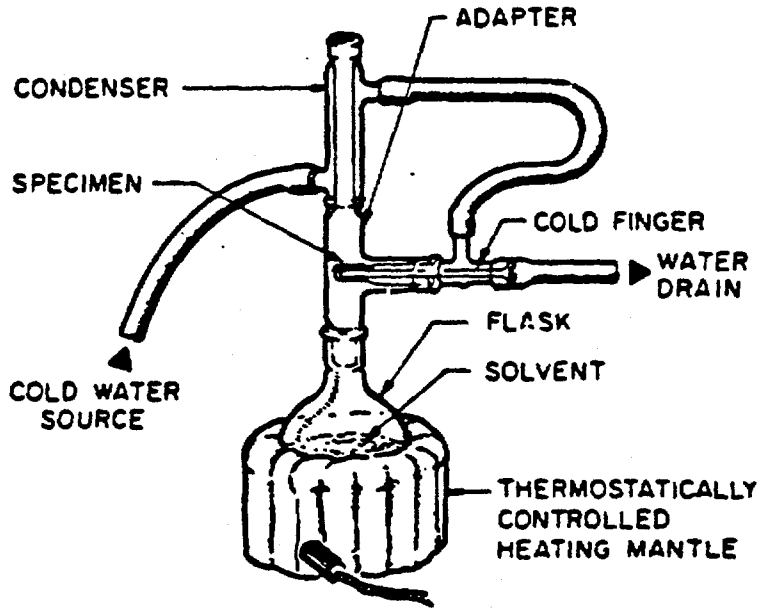


Figure 6. Condensation Washer.

6.5 Examination by Electron Microscopy

6.5.1 Microscope Alignment and Magnification Calibration

Align the electron microscope according to the specifications of the manufacturer. Initially, and at regular intervals, carry out a calibration of the two magnifications used for the analysis (approximately 20,000 and 2,000) using a diffraction grating replica. The calibration should always be repeated after any instrumental maintenance or change of operating conditions. The magnification of the screen image is not the same as that obtained on photographic plates or film. The ratio between these is usually a constant value for the instrument. It is most important that before the magnification calibration is carried out the sample height is adjusted so that the sample is in the eucentric position.

6.5.2 Calibration of EDXA System

The purpose of the calibration is to enable quantitative composition data, at an accuracy of about 10% of the elemental concentration, to be obtained from EDXA spectra of silicate minerals involving the elements sodium, magnesium, aluminum, silicon, potassium, calcium, manganese and iron. If quantitative determinations are required for minerals containing other elements, suitable calibration information may be incorporated in the computer analysis. The well-characterized standards recommended permit calibration of any TEM-EDXA combination which meets the instrumental specifications of Section 4.2, so that data from different instruments can be compared. The standards used for calibration, and the elements which they represent, are shown in Table 2.

TABLE 2. SILICATE MINERAL STANDARDS

Elements	Mineral Standard
Na, Fe, Si	Riebeckite
Mg, Si	Chrysotile
Al, Si	Halloysite
K, Si	Phlogopite
Ca, Si	Wollastonite
Mn, Si	Bustamite

The compositions of these standards have been determined by microprobe analysis, and the TEM grids were prepared from fragments of the same selected mineral specimens. They permit the computer program of Appendix A to be used with any TEM-EDXA system.

Place the first grid into the microscope, form an image at the calibrated higher magnification of about 20,000, and adjust the specimen height to the eucentric point. Tilt the specimen towards the X-ray detector as required by the instrument geometry. Select an isolated fiber or particle less than 0.5 μm in width, and accumulate an EDXA spectrum using an electron probe of suitable diameter. When a well defined spectrum has been obtained, perform an appropriate background subtraction and obtain the net peak areas for each element listed, using energy windows centered on the

peaks and about 130 eV wide. Compute the ratio of the peak area for each specified element relative to the peak area for silicon. Repeat the procedure for about 20 particles of each mineral standard. Analyses of any obvious foreign particles should be rejected, and the data from any one standard should be reasonably self-consistent. Calculate the arithmetic mean peak area ratios for each specified element of each mineral standard. These values are required initially as input for the fiber identification program, and apart from occasional routine checks to ensure that there has been no degradation of the detector resolution, the calibration need not be repeated unless there has been a change of instrumental operating conditions.

6.5.3 Grid Preparation Acceptability

Insert the specimen grid into the electron microscope and adjust the magnification to a value sufficiently low (300 - 1000) so that complete grid openings can be inspected. Examine at least 10 grid openings to evaluate the fiber and total particulate loadings, the uniformity of the particulate deposit, and the extent to which the carbon film is unbroken. The grid must be rejected from further analysis if:

a) the grid is too heavily loaded with fibers to perform an accurate count. Accurate counts cannot be performed if the grid has more than about 50 fibers per grid opening. A new grid preparation must be made using either a smaller volume of water or a suitable volume of the water diluted with fiber-free water;

b) the overall distribution of the deposited debris is noticeably non-uniform. A new grid preparation must be made, paying particular attention to proper particulate dispersal and filtration procedures;

c) the grid is too heavily loaded with debris to allow examination of individual particles by SAED and EDXA. A new grid preparation must be made using either a smaller volume of water or a dilution of the original water sample;

d) a large proportion of the grid openings have broken carbon film. Since the breakage is usually more frequent in areas of heavy deposit, counting of the intact openings could lead to biased results. Therefore, a new grid preparation must be made from a more completely dispersed sample, a reduced volume of sample, or alternatively, a thicker carbon film may be necessary to support the larger particles.

6.5.4 Procedure for Fiber Counting

The number of fibers to be counted depends on the statistical precision desired. In the absence of fibers, the area of the electron microscope grids which must be examined depends on the analytical sensitivity required. For statistical reasons, discussed in Section 7.2, the fibers on a minimum of 4 grid openings must be counted. The precision of the fiber count depends not only on the total number of fibers counted, but also on their uniformity from one grid opening to the next. In practice, it has been found that termination of the fiber count at a minimum of 100 fibers or 20 grid openings, whichever occurs first, yields results which usually require no further refinement. Additional fiber counting will be necessary if greater precision is required.

At least three grids prepared from the filter must be used in the fiber count. Several grid openings are to be selected from each grid, and the data are all incorporated in the calculation of the results. This permits the measurements to be spread across a diameter of the original filter, so that any gross deviations from a uniform deposition of fibers should be detected.

Figures 7 and 8 show specimen fiber counting raw data sheets which represent the minimum standard of data reporting for this analytical procedure. Figure 7 shows page 1 of the raw data tabulation, which contains all specimen preparation details. Figure 8 is a continuation sheet for the fiber classification and measurement data; several of these sheets may be required for analysis of a sample.

Select a typical grid opening from one of the grids. Set the magnification to the calibrated higher value (about 20,000). Adjust the sample height until the features in the center of the screen are at the eucentric point. Check that the goniometer tilt is set at zero. Reduce the magnification to the lower calibrated value of about 2,000. Measure both dimensions of the grid opening image in millimeters, using the markings on the fluorescent screen. In columns 1 and 2 specify the sequential number of the grid opening, and its dimensions. These two columns are not used again until fiber counting is commenced in the next grid opening to be examined. Adjust the magnification to the upper calibrated value, close to 20,000, and position the grid opening so that one corner is visible on the screen. Move the image by adjustment of only one translation control, carefully examining the sample for fibers, until the opposite side of the opening is encountered. Move the image by one screen width using the

ASBESTOS ANALYSIS - WATER SAMPLE DATA

SEQ:	SAMPLE:	CODE
JOB:		

PREP: By ____ Date - -	COUNT: By ____ Date - -	PROCESS: By ____ Date - -
INSTRUMENT:	MAGNIFICATIONS: Grid _____	Count _____

DILUTIONS: 0

1 Volume Taken (mL) _____ Final Volume (mL) _____

2 Volume Taken (mL) _____ Final Volume (mL) _____

FINAL PREPARATION FILTRATION: Vol. Filtered (mL) _____ Active Area (cm^2) _____

COMMENTS: (for inclusion in computer print-out; format in 5 lines of 60 characters)

_____

_____

_____

_____

_____

FIBER CLASSIFICATIONS:

COUNT. NAM TM CM CD CQ CMQ CDQ UF AD AX ADX AQ ADQ AZQ AZZ

PROCESS:

FIBER TYPE	CLASSIFICATION	FIBER TYPE	CLASSIFICATION

NOTES: Preparation: _____

_____

_____

_____

_____

Examination: _____

_____

_____

_____

_____

_____

_____

Figure 7. Sheet for Recording Water Sample Data.

ASBESTOS ANALYSIS - FIBER CLASSIFICATION

SAMPLE:

Page ____ of ____

Grid Opening		Fiber Classification															Fiber Size		EDXA Peak Areas	Comments
No.	Dimensions (mm)	NAM	TM	CM	CD	CQ	CMQ	CDQ	UF	AD	AX	ADX	AQ	ADQ	AZQ	AZZ	Length (mm)	Width (mm)		
	x																			
	x																			
	x																			
	x																			
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Figure 8. Sheet for Recording Fiber Classification and Measurement Data.

Continued on Page ____

other translation control, and then scan the image in the reverse direction. Continue in this manner until the entire grid opening has been inspected. When a fiber is detected, classify it according to the procedures described in Section 6.7, and then insert the appropriate classification on the data sheet. Measure the length and width of the fiber image in millimeters and record these in the appropriate columns of the data sheet. Do not record fibers of obvious biological origin or diatom fragments. Continue the examination until 100 fibers have been recorded in all classification categories of interest, or until 20 grid openings have been inspected. The data should be drawn approximately equally from the three grids. In all samples, fibers on a minimum of 4 grid openings must be counted. Fibers less than 0.5 μm in length will not be incorporated in the fiber concentration calculation.

6.5.5 Estimation of Mass Concentration

If the primary objective of the analysis is to determine the mass concentration, the fiber counting should be approached in a different manner. The number of fibers which must be counted in order to achieve a reliable estimate of the mass concentration depends primarily upon the range of the fiber diameter distribution. The mass concentration measurement is most sensitive to fibers of large diameter, which unfortunately are among those which occur infrequently. When the diameter distribution is narrow, such as that found in the case of chrysotile fibrils, then the mass concentration has approximately the same precision as that of the number concentration. However, the mass concentration may be actually meaningless when calculated from a low number of fibers observed during a routine fiber count, if these fibers have a broad distribution of widths.

If the mass concentration is the primary interest, and the precision required is greater than is possible from the normal fiber count, a different approach to the fiber count must be used. Initially, establish the largest width of fiber which can be detected on the grid by a cursory survey, at a reduced magnification, of a large number of grid openings (about 50). Calculate the volume of this fiber. Adjust the magnification to a value such that a width of 1 mm on the screen corresponds to 10% of the width of the previously selected large fiber. Carry out a routine fiber count for a minimum of 100 fibers, recording only fiber images greater than 1 mm in width. Continue counting until the total volume of fibers is at least 10 times the volume of the originally selected large fiber. The precision and accuracy of this technique has not been

investigated fully, but for samples with broad width distributions it is capable of yielding significantly more precise mass determinations than are obtainable by the conventional fiber count.

The remaining problem concerns the assumption that the widths also represent the thicknesses of the fibers. Measurements of particle thicknesses can be made separately, using the shadow casting technique. Before the filter is carbon coated, apply a vacuum coating of platinum-carbon or gold to the active surface of the Nuclepore filter at an angle of 45°. In the TEM, the fibers will then display shadows on the carbon film which approximate to their thicknesses. Suitable techniques for shadowing are described in the paper by D.E. Bradley included in the Selected Bibliography.

6.6 Fiber Counting Criteria

6.6.1 Fiber Counting Method

Fiber counting with this analytical method will be performed only by the grid opening technique. If a specimen grid is too heavily loaded for examination of entire grid openings, a more reliable result is obtained by preparation of a new filter, using a smaller volume of sample.

6.6.2 Fibers Which Touch Grid Bars

A fiber which intersects a grid bar will be counted only for two sides of the grid opening, as illustrated in Figure 9. The length of the fiber will be recorded as twice the visible length. Fibers intersecting either of the other two sides will not be included in the count.

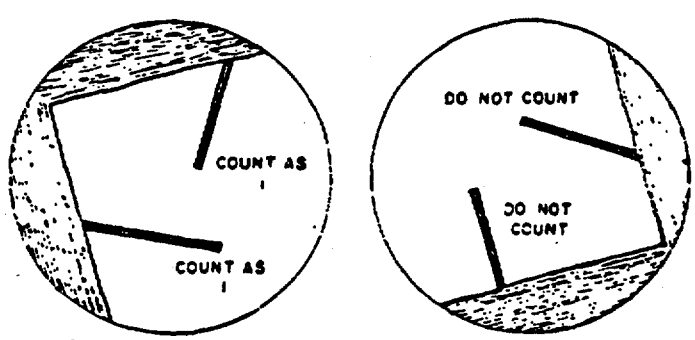

Figure 9. Counting of Fibers Which Overlap Grid Bars.

This procedure ensures that the numerical count will be accurate, and that the best average estimate of length has been made.

6.6.3 Fibers Which Extend Outside the Field of View

During scanning of a grid opening, fibers which extend outside of the field of view must be counted systematically to avoid double-counting. In general, a rule must be established so that fibers extending outside the field of view in only two quadrants are counted. Fibers without terminations in the field of view must not be counted. The procedure is illustrated by Figure 10. The length of each fiber counted is established by moving the sample, and then returning to the original field of view before scanning is continued.

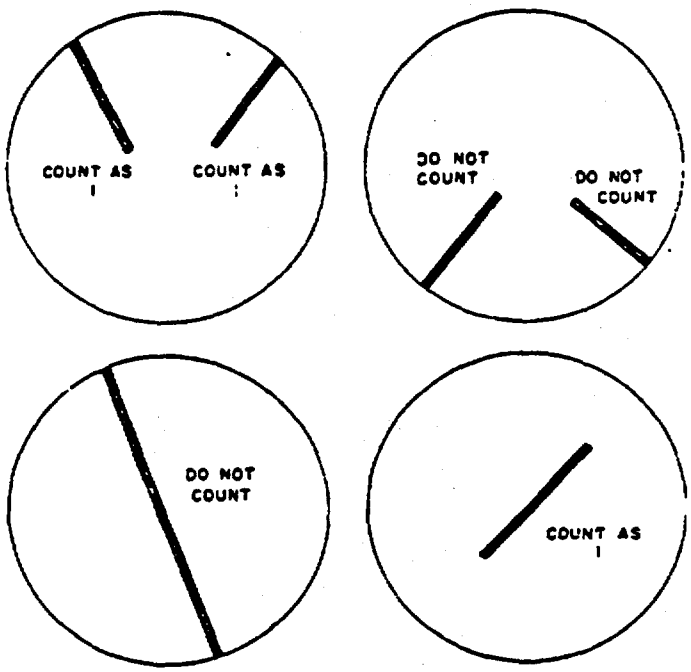

Figure 10. Counting of Fibers Which Extend Outside the Field of View.

6.6.4 Fibers with Stepped Sides

A fiber with stepped sides will be assigned a width mid-way between the minimum and maximum widths.

6.6.5 Fiber Bundles

A fiber bundle composed of many parallel fibers will be counted as a single fiber of a width equal to an estimate of the mean bundle width. Figure 11 shows examples of the procedure.

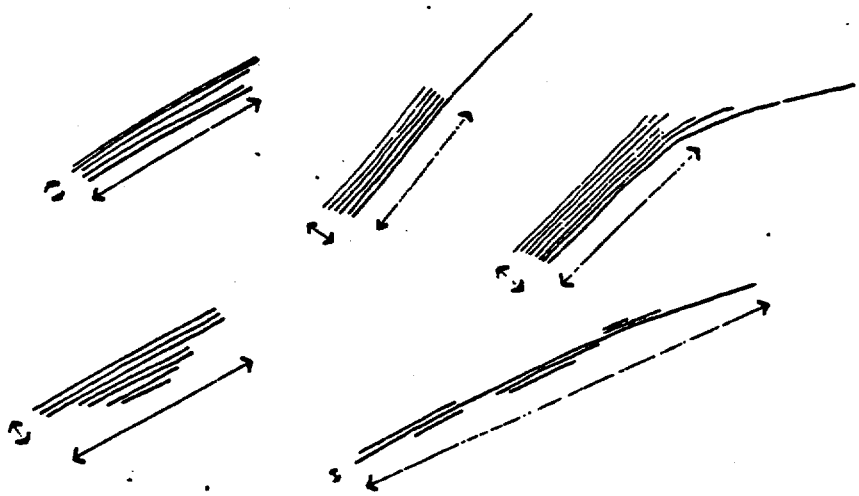

Figure 11. Counting and Measurement of Fiber Bundles. Each bundle to be counted as one fiber with dimensions as indicated by arrows.

6.6.6 Aggregates of Randomly Oriented Fibers

The structure of an aggregate of randomly oriented fibers may be sufficiently visible that the constituent fibers can be counted. This is illustrated in Figure 12. In this

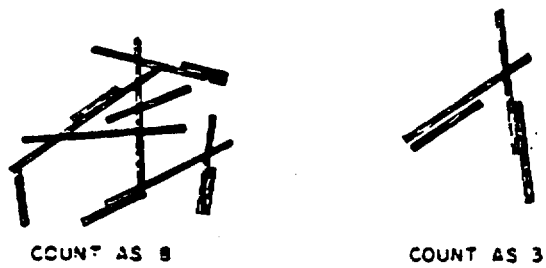

Figure 12. Counting of Fiber Aggregates.

case individual fibers will be recorded. Where the fiber aggregate is too large and complex to count each individual fiber, the identification and aggregate dimensions will be recorded, but it will not be incorporated in the fiber count and mass calculations.

6.6.7 Fibers Attached to Non-Fibrous Debris

A fiber may be attached to, or partially concealed by, a particle of non-fibrous debris. If two ends are visible which appear to be the ends of a single fiber, the fiber will be counted. Where only one end of a fiber is visible, the fiber will be counted as a single fiber having a length equal to twice the visible length, except where this would place the concealed end outside of the particle. In this case the length will be recorded as the visible length plus the extension of it to the opposite side of the particle. Examples of the procedure are shown in Figure 13. There may be more than one fiber attached to a single particle of debris; each one should be counted. If an assembly of fibers and particles is too complex to treat in this way, the overall dimensions should be recorded, but the assembly should not be incorporated in the fiber count and mass calculations.

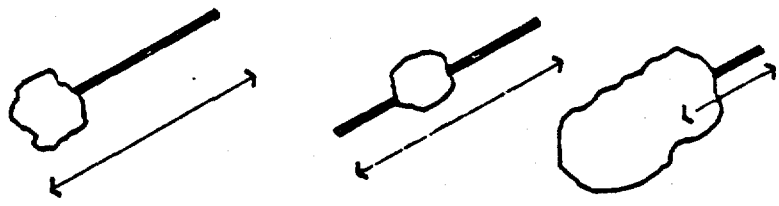

Figure 13. Counting and Measurement of Fibers Attached to Non-Fibrous Debris.

6.7 Fiber Identification Procedures

6.7.1 General

Before it is incorporated into the fiber count, each particle with an aspect ratio of 3 to 1 or greater and not of obviously biological origin must be identified according to defined criteria. It is recognized that economic considerations usually preclude unequivocal identification of every fiber reported. In this analytical method, the

requirement for unequivocal identification is limited to a small proportion of the fibers in order to demonstrate the presence of the particular species. The proportion of fibers examined for unequivocal identification will be stated in the analytical result. The remainder of the fibers are then classified on the basis of crystallographic or chemical similarity, or both, to the identified fibers. If on later examination it is considered necessary to perform a more complete and rigorous identification, additional fibers may be examined in more detail to confirm conclusions based on the fiber classification data.

In general, it will be found that for various instrumental reasons it may be impossible to identify a specific fiber completely, even though the fiber may be of a well-characterized variety. It is, nevertheless, important to record the degree to which the procedures were successful in classification or identification of a particular fiber.

6.7.2 SAED and EDXA Techniques

Fibers are initially classified into two categories on the basis of morphology: those fibers with tubular morphology, and those fibers without tubular morphology. Further analysis of each fiber is conducted using SAED and EDXA methods. Although the precise techniques and classification procedures are specified in Sections 6.7.4 and 6.7.5, some general guidance on the use of SAED and EDXA methods is given here.

The crystal structure of some mineral fibers, such as chrysotile, is easily degraded by the high current densities required for EDXA examination. Therefore, SAED investigation of these sensitive fibers must be completed before attempts are made to obtain EDXA spectra. When examining more stable fibers, such as the amphiboles, the order of work is unimportant.

The SAED technique can be either qualitative or quantitative. Qualitative SAED consists of visual examination of the pattern obtained on the microscope screen from a randomly oriented fiber. SAED patterns obtained from fibers with cylindrical symmetry, such as chrysotile, are an exception since they are not sensitive to axial tilt, and patterns from randomly oriented fibers can be interpreted quantitatively. For non-cylindrical fibers, quantitative (zone axis) SAED requires alignment of the fiber so that a principal crystallographic axis is parallel to the electron beam. The pattern is then recorded and its consistency with known mineral structures is checked by a computer program. The SAED pattern obtained from one zone axis may not be sufficiently

specific to identify the mineral fiber, but it is often possible to tilt the fiber to another angle and to record a different zone axis pattern. The angle between the two axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the SAED pattern, the camera length of the TEM should be set to a low value and the SAED pattern then should be viewed through the binoculars. This procedure minimizes the irradiation and possible degradation of the fiber. However, the pattern is distorted by the tilt angle of the viewing screen. For recording purposes, a camera length of at least 2 meters must be used if accurate measurement of the pattern is to be possible. It is of extreme importance that, when obtaining an SAED pattern for either recording or visual evaluation, the sample height be properly adjusted to the eucentric point and the image be focussed in the plane of the selected area aperture. If this is not done there may be some components of the SAED pattern which do not originate from the selected area. It will be found in general that the smallest SAED aperture will be necessary.

For accurate measurements of the SAED pattern, an internal calibration standard is required. A thin coating of gold, or other calibration material, must be applied to the underside of the TEM specimen. This coating can be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every SAED pattern and these rings provide the required calibration information.

To form an SAED pattern, move the image of the fiber to the center of the screen and insert a suitable selected area aperture into the electron beam so that the fiber, or a portion of it, is in the illuminated area. The size of the aperture and the portion of the fiber should be such that particles other than the one to be examined are excluded from the selected area. Observe the diffraction pattern with the binocular attachment. If an incomplete diffraction pattern is obtained, move the particle around in the selected area to attempt to get a clearer diffraction pattern or to eliminate possible interferences from neighboring particles.

If a zone axis SAED analysis is to be attempted on the fiber, the sample must be in the appropriate holder. The most convenient holder allows complete rotation of the sample and single axis tilting. Rotate the sample until the fiber image indicates that the fiber is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fiber is at the

eucentric position. Tilt the fiber until a pattern appears which is a symmetrical, two dimensional array of spots. The recognition of zone axis alignment conditions requires some experience on the part of the operator. During tilting of the fiber to obtain zone axis conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of multiple diffraction exists, and some caution should be exercised in selection of diffraction spots for measurement. A full discussion of electron diffraction and multiple diffraction can be found in the references by J.A. Gard, P.B. Hirsch et al, and H.R. Wenk, included in the Selected Bibliography. Not all zone axis patterns which can be obtained are useful or definitive. Only those which have closely-spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d-spacings are less than about 0.3 nm are not useful and are usually very wasteful in computer time. A useful guideline is that the lowest angle reflections should be within the radius of the first gold diffraction ring (111), and that patterns with smaller distances between reflections are usually the most definitive.

Five spots, closest to the center spot, along two intersecting lines of the zone axis pattern must be selected for measurement, as illustrated in Figure 14.

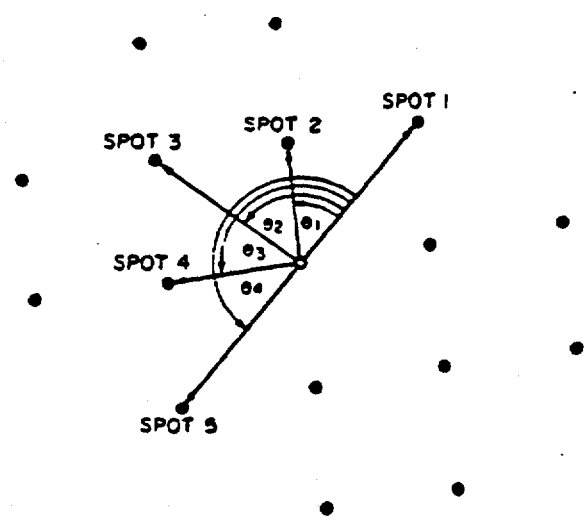

Figure 14. Measurement of Zone Axis SAED Patterns.

The distances of these spots from the center spot and the four angles shown are the input for the computer program. Since the center spot is usually very over-exposed, it does not form a suitable origin for measurement. The required distances must therefore be obtained by measuring between pairs of spots symmetrically disposed about the center spot, preferably separated by several repeat distances. The distances must be measured with a precision of better than 0.3 mm, and the angles better than 2.5°. The diameter of the first or second ring of the calibration pattern (111 and 200) must also be measured with the same precision.

The camera constant (λL) required for the computer program is given by:

$$\lambda L = \frac{aD}{\sqrt{h^2 + k^2 + l^2}}$$

where:

λ = Wavelength of the incident electrons

L = Effective camera length in mm

a = Unit cell dimension in Ångstroms

D = Diameter of the (h, k, l) diffraction rings in millimeters

h, k, l = Miller indices of the scattering plane of the crystal.

Using gold, the camera constant is given by:

$$\lambda L = 2.3548\ D \text{ (first ring)}$$

$$\lambda L = 2.0393\ D \text{ (second ring)}$$

Analysis of a fiber by EDXA is required in this analytical procedure. Interpretation of the EDXA spectrum may be either qualitative or quantitative. For qualitative interpretation of a spectrum, the elements originating from the fiber are recorded. For quantitative interpretation, the net peak areas, after background subtraction, are obtained for the elements originating from the fiber. As discussed in Section 6.5.2, this method provides for quantitative interpretation for those minerals which contain silicon.

To obtain an EDXA spectrum move the image of the fiber to the center of the screen and remove the objective aperture. Select an appropriate electron beam diameter and deflect the spot to impinge on the fiber. Depending on the instrumentation, it may be necessary to tilt the sample and in some instruments to use Scanning Transmission Electron Microscopy (STEM) mode of operation.

The time for acquisition of a suitable spectrum varies with the fiber diameter, and also with instrumental factors. For quantitative interpretation, spectra should have a statistically valid number of counts in each peak. Analyses of small diameter fibers which contain sodium are the most critical, since it is in the low energy range that the X-ray detector is least sensitive. Accordingly, it is necessary to acquire a spectrum for a sufficiently long period that the presence of sodium can be detected in such fibers. It has been found that satisfactory quantitative analyses can be obtained if acquisition is continued until the background-subtracted silicon Kα peak integral exceeds 10000 counts. The spectrum should then be manipulated to subtract the background and to obtain the net areas of the elemental peaks.

After quantitative EDXA classification of some fibers by computer analysis of the net peak areas, it may be possible to classify further fibers in the same sample on the basis of comparison of spectra at the instrument. Frequently, visual comparisons can be made after somewhat shorter acquisition times.

6.7.3 Analysis of Fiber Identification Data

Since the fiber identification procedure can be involved and time-consuming, a Fortran computer program has been provided, the listing of which is given in Appendix A. This program permits the EDXA and zone axis SAED measurements to be compared against a library of compositional and structural data for 226 minerals. The mineral library includes fibrous species which have been listed by several authors, together with other minerals which are known to be similar to amphibole in either their compositions or some aspects of their crystallography. Additional minerals may be added to the library if they are thought to be of concern in particular situations. Rejection of a mineral by the program indicates that either the compositional or crystallographic data for the mineral in the library are inconsistent with the measurements made on the unknown fiber. Demonstration that the measurements are consistent with the data for a particular test mineral does not uniquely identify the unknown, since the possibility exists that data from other minerals may also

be consistent. It is, however, very unlikely that a mineral of another structural class could yield data consistent with that from an amphibole fiber identified uniquely by quantitative EDXA and two zone axis SAED patterns.

The computer program classifies fibers initially on the basis of chemical composition. Either qualitative or quantitative EDXA information may be entered. The procedure using qualitative EDXA consists of entering the list of elements which originate from the particle. For quantitative EDXA (silicon-containing minerals only), the list of elements and the areas under the corresponding X-ray emission peaks, after background correction, form the input data for the computer program. The width of the fiber is also required as input into the program. The program will select from the file a list of minerals which are consistent in composition with that measured for the unknown fiber. To proceed further, it is necessary to obtain the first zone axis SAED pattern, according to the instructions in Section 6.7.2.

It would be attractive to specify a particular zone axis pattern to be obtained for confirmation of amphibole, particularly if such a pattern could be considered characteristic. Unfortunately, for a fiber with random orientation on the grid, no specimen holder and goniometer currently available will permit convenient and rapid location of two pre-selected zone axes. The most practical approach has been adopted, which is to accept those low index patterns which are easily obtained, and then to test their consistency with the structures of the minerals already pre-selected on the basis of the EDXA data. Even the structures of non-amphibole minerals in this pre-selected list must be tested against the zone axis data obtained for the unknown fiber, since non-amphibole minerals may yield similar patterns consistent with amphibole structures in some orientations.

The zone axis SAED interpretation part of the program will consider all minerals previously selected from the file as being chemically compatible with the EDXA data. It will then return a second and usually reduced list of minerals for which solutions have been found. A second set of zone axis data from another pattern obtained on the same fiber can then be processed either as further confirmation or to attempt elimination of an ambiguity. In addition, the angle measured between the orientations of the two zone axes can be entered into the computer to be checked for consistency with the structures of minerals. Caution should be exercised in rationalizing the inter-zone axis angle, since if the fiber contains c-axis twinning the two zone axis SAED patterns may originate from the separate twin crystals.

In practice, the full program will normally be applied to very few fibers, unless precise identification of all fibers is required.

6.7.4 Fiber Classification Categories

It is not always possible to proceed to a definitive identification of a fiber; this may be due to instrumental limitations or to the actual nature of the fiber. In many analyses a definitive identification of each fiber may not actually be necessary if there is other knowledge available about the sample, or if the concentration is below a level of interest. The analytical procedure must therefore take account of both instrumental limitations and varied analytical requirements. Accordingly, a system of fiber classification has been devised to permit accurate recording of data. The classifications are shown in Tables 3 and 4, and are directed towards identification of chrysotile and amphibole respectively. Fibers will be reported in these categories.

The general principle to be followed in this analytical procedure is first to define the most specific fiber classification (target classification) which is to be attempted. Then, for each fiber examined, the classification which is actually achieved is recorded. Depending on the intended use of the results, criteria for acceptance of fibers as "identified" can then be established at any time after completion of the analysis.

In an unknown sample, chrysotile will be regarded as confirmed only if a recorded, calibrated SAED pattern from one fiber in the CD category is obtained. Amphibole will be regarded as confirmed only by obtaining recorded data which yields exclusively amphibole solutions for fibers classified in the AZQ, AZZ or AZZQ categories.

6.7.5 Procedure for Classification of Fibers With Tubular Morphology, Suspected to be Chrysotile

Many fibers are encountered which have tubular morphology similar to that of chrysotile, but which defy further attempts at characterization by either SAED or EDXA. They may be non-crystalline, in which case SAED techniques are not useful, or they may be in a position on the grid which does not permit an EDXA spectrum to be obtained. Alternatively, the fiber may be of organic origin, but not sufficiently definitive that it can be disregarded.

Classification attempts will meet with various degrees of success. Figure 15 shows the classification procedure to be used for fibers which display any tubular morphology.

TABLE 3. CLASSIFICATION OF FIBERS WITH TUBULAR MORPHOLOGY

TM	-	Tubular Morphology not sufficiently characteristic for classification as chrysotile
CM	-	Characteristic Chrysotile Morphology
CD	-	Chrysotile SAED pattern
CQ	-	Chrysotile composition by Quantitative EDXA
CMQ	-	Chrysotile Morphology and composition by Quantitative EDXA
CDQ	-	Chrysotile SAED pattern and composition by Quantitative EDXA
NAM	-	Non-Asbestos Mineral

TABLE 4. CLASSIFICATION OF FIBERS WITHOUT TUBULAR MORPHOLOGY

UF	-	Unidentified Fiber
AD	-	Amphibole by random orientation SAED (shows layer pattern of 0.53 nm spacing)
AX	-	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	-	Amphibole by random orientation SAED and Qualitative EDXA
AQ	-	Amphibole by Quantitative EDXA
AZ	-	Amphibole by one Zone Axis SAED
ADQ	-	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	-	Amphibole by one Zone Axis SAED pattern and Quantitative EDXA
AZZ	-	Amphibole by two Zone Axis SAED patterns with consistent inter-axial angle
AZZQ	-	Amphibole by two Zone Axis SAED patterns, consistent inter-axial angle and Quantitative EDXA
NAM	-	Non-Asbestos Mineral

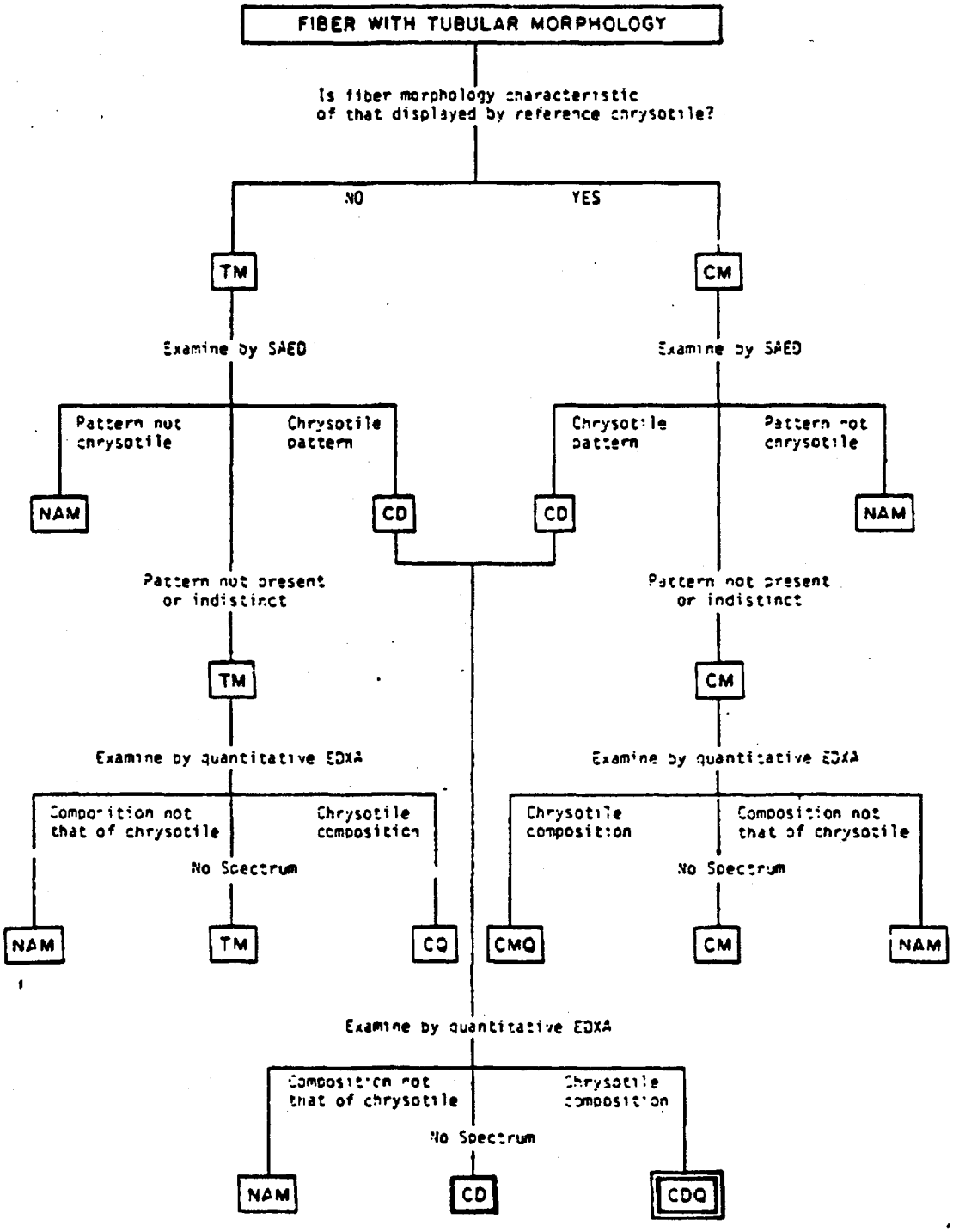

Figure 15. Classification Chart for Fiber With Tubular Morphology.

The chart is self explanatory, and essentially every fiber is either rejected as a non-asbestos mineral (NAM), or classified in some way which could still contribute to the chrysotile fiber count.

Morphology is the first consideration, and if this is not similar to that usually seen in chrysotile standard samples, the initial classification is TM. Regardless of the doubtful morphology, the fiber will still be examined by SAED and EDXA methods according to Figure 15. Where the morphology is more definitive, it may be possible to classify the fiber as having chrysotile morphology (CM).

The morphological characteristics required will be:

a) the individual fibrils should have high aspect ratios exceeding 10:1 and be about 40 nm in diameter;

b) the electron scattering power of the fiber at 60 to 100 kV accelerating potential should be sufficiently low for internal structure to be visible; and

c) there should be some evidence of internal structure suggesting a tubular appearance similar to that shown in Figure 16A, which may degrade in the electron beam to the appearance shown in Figure 16B.

Every fiber having these morphological characteristics will be examined by the SAED technique, and only those which give diffraction patterns with the precise characteristics of Figure 17 will be classified as chrysotile by SAED (CD). The relevant features in this pattern for identification of chrysotile are indicated. The (002) reflections should be examined to determine that they correspond approximately to a spacing of 0.73 nm, and the layer line repeat distance should correspond to 0.53 nm. There should also be "streaking" of the (110) and (130) reflections. Using the millimeter calibrations on the microscope viewing screen, these observations can readily be made at the instrument. A TEM micrograph of at least one representative fiber will be recorded, and its SAED pattern will also be recorded on a separate film or plate. This plate will also carry calibration rings from a known polycrystalline substance such as gold. This calibrated pattern is the <u>only</u> documentary proof that the particular fiber is chrysotile and not some other tubular or scrolled species such as halloysite, palygorskite, talc or vermiculite. The proportion of fibers which can be successfully identified as chrysotile by SAED is variable, and to some extent dependent on both the instrument and the procedures of the operator. The fibers that fail to yield an identifiable SAED pattern will remain in the TM or CM categories unless they are examined by EDXA.

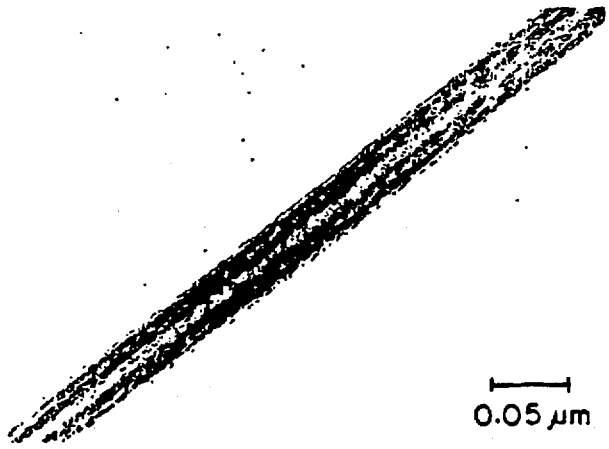

Figure 16A. TEM Micrograph of Chrysotile Fibril, showing Morphology.

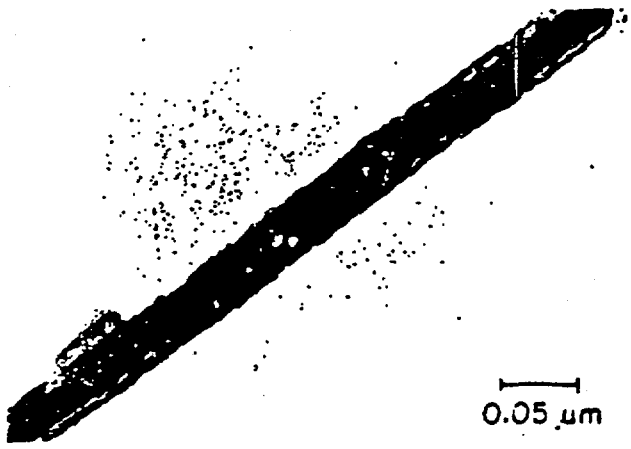

Figure 16B. TEM Micrograph of UICC Canadian Chrysotile Fiber after Thermal Degradation by Electron Beam Irradiation.

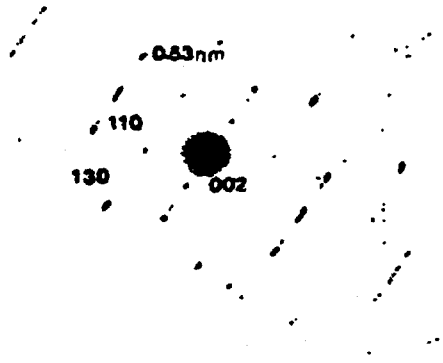

Figure 17. SAED Pattern of Chrysotile Fiber with Diagnostic Features Labelled. Necessary criteria are the presence of 0.73 nm spacing for the 002 reflections, 0.53 nm spacing for the layer line repeat and characteristic streaking of the 110 and 130 reflections.

In the EDXA analysis of chrysotile there are only two elements which are relevant. For fiber classification, the EDXA analysis must be quantitative. If the spectrum displays prominent peaks from magnesium and silicon, with their areas in the appropriate ratio, and with only minor peaks from other elements, the fiber will be classified as chrysotile by quantitative EDXA, in the categories CQ, CMQ or CDQ, as appropriate.

For chrysotile analyses there are essentially three possible levels of analysis:

1. morphological and SAED discrimination only (Target classification CD);

2. in addition, EDXA of only those fibers unclassified by SAED (Target classification CD);

3. EDXA in addition to SAED on all fibers (Target classification CDQ).

6.7.6 Procedure for Classification of Fibers Without Tubular Morphology, Suspected to be Amphibole

Every particle without tubular morphology and which is not obviously of biological origin, with an aspect ratio of 3 to 1 or greater and having parallel or stepped sides, will be considered as a suspected amphibole fiber. Further examination of the fiber by SAED and EDXA techniques will

meet with a variable degree of success, depending on the nature of the fiber and on a number of instrumental limitations. It will not be possible to identify every fiber completely, even if time and cost were of no concern. Moreover, confirmation of the presence of amphibole can be achieved only by quantitative interpretation of zone axis SAED patterns, a very time-consuming procedure. Accordingly, for routine samples from unknown sources, this analytical procedure limits the requirement for zone axis SAED work to a minimum of one fiber representative of each compositional class reported. In some samples, it may be necessary to identify more fibers by the zone axis technique. When analyzing samples from well-characterized sources, the cost of identification by zone axis methods may not be justified.

The 0.53 nm layer spacing of the random orientation SAED pattern is not by itself diagnostic for amphibole. However, the presence of c-axis twinning in many fibers leads to contributions to the layers in the patterns by several individual parallel crystals of different axial orientations. This apparently random positioning of the spots along the layer lines, if also associated with a high fiber aspect ratio, is a characteristic of amphibole asbestos, and thus has some limited diagnostic value. If a pattern of this type is not obtained, the identity of the fiber is still ambiguous, since the absence of a recognizable pattern may be a consequence of an unsuitable orientation relative to the electron beam, or the fiber may be some other mineral species.

Figure 18 shows the fiber classification chart for suspected amphibole fibers. This chart shows all the classification paths possible in analysis of a suspected amphibole fiber, when examined systematically by SAED and EDXA. Initially two routes are possible, depending on whether an attempt to obtain an EDXA spectrum or a random orientation SAED pattern is made first. The normal procedure for analysis of a sample of unknown origin will be to examine the fiber by random orientation SAED, qualitative EDXA, quantitative EDXA, and zone axis SAED, in this sequence. The final fiber classification assigned will be defined either by successful analysis at the target level or by the instrumental limitations. The maximum classification achieved for each fiber will be recorded on the counting sheet in the appropriate column. The various classification categories can then be combined in any desired way for calculation of the fiber concentration, and a complete record of the results from each fiber is maintained for reassessment of the data if necessary.

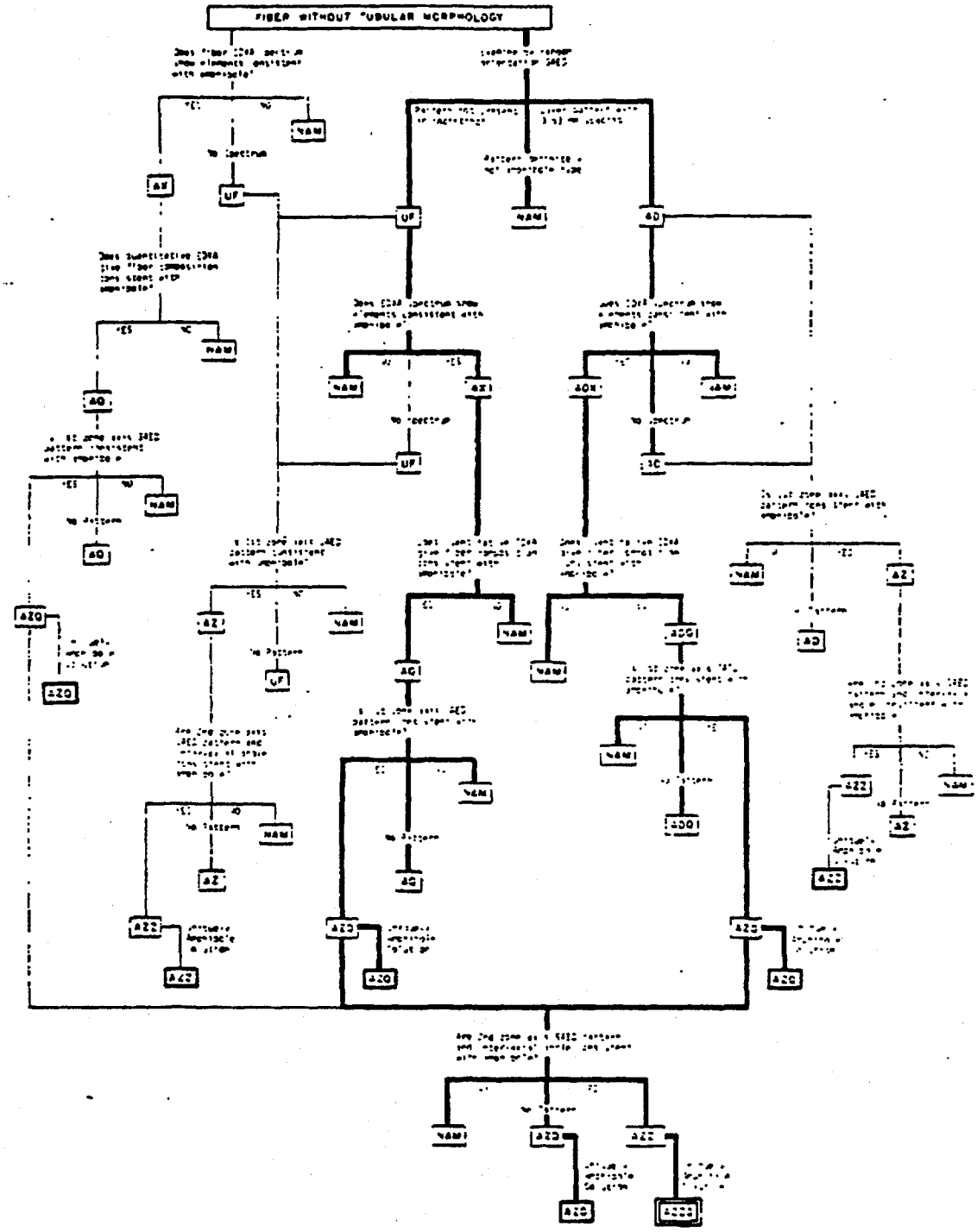

Figure 18. Classification Chart for Fiber Without Tubular Morphology. Bold Lines indicate the Preferred Paths.

Depending on the particular situation, four levels of analysis can be defined in this analytical procedure, and these are shown in Table 5.

In the routine unknown sample, a level 3 analysis will be required if the presence of amphibole is to be confirmed. For this level of analysis, attempts will be made to raise the classification of every fiber to the ADQ category. In addition, at least one fiber from each type of suspected amphibole found will be examined by zone axis SAED methods to confirm the identification.

TABLE 5. LEVELS OF ANALYSIS FOR AMPHIBOLE

Level of Analysis	Application	Target Classification for all Fibers	Required Classification for Confirmation of Amphibole in a Proportion of the Fibers
1	Routine monitoring of known and well-characterized sources for one mineral fiber type.	ADX	Not Applicable
2	Routine monitoring of known and well-characterized sources where discrimination between two or more amphibole fiber types is required.	ADQ	Not Applicable
3	Routine samples from uncharacterized sources in which presence or absence of amphibole is to be confirmed.	ADQ	AZZ, AZQ or AZZQ - Solutions must include only amphiboles.
4	Samples where precise identification of all amphibole fibers is an important issue.	AZQ	AZZQ - Solutions must include only amphiboles.

6.8 Blank and Control Determinations

To ensure that contamination by extraneous fibers during sample preparation is insignificant compared with the results reported on samples, it is necessary to establish a continuous program of blank measurements. Initially, and at intervals during an analytical program, it is also necessary to ensure that samples of known fiber concentrations can be analyzed satisfactorily.

6.8.1 Blank Determinations

At least one blank determination will be made along with every group of samples prepared at any one time. For the blank determination, a 0.1 μm Nuclepore filter will be prepared by filtration of 100 mL of ozone-UV treated fiber-free water if using 25 mm diameter equipment, and 500 mL treated water if using 47 mm diameter equipment. If the samples have been preserved with mercuric chloride, an equivalent amount of the solution should be added to the water used for the preparation of the blank. This blank filter will be carbon coated at the same time as the group of samples, and solvent extracted in the same Jaffe Washer. All aspects of the sample preparation will then be identical to those for the actual samples. All fibers on 20 grid openings of the blank sample will be recorded. The mean fiber concentration for the blank must be less than 0.05 MFL or less than 1% of the lowest individual value reported in the samples concerned, whichever is the greater value. If a value higher than these criteria is encountered, satisfactory blank values must be demonstrated before further analyses are carried out. If it is suspected that samples could have been contaminated during the original preparation, the duplicate bottles should be used for the repreparation of the samples concerned.

6.8.2 Control Samples

Control samples must be incorporated into sample analysis programs in order to demonstrate that the expected results can be produced from samples of known fiber concentration. Such reference suspensions can be prepared using ampoules of stable fiber dispersions listed in Section 4.3.16. It is recommended that the range of fiber concentrations found in the real samples should be simulated using the reference suspensions. The sealed ampoules of fiber dispersions become unstable when they are opened, and the fiber concentration value should not be relied upon for more than 8 hours after opening. Accordingly, it is recommended that, upon opening a dispersion concentrate ampoule, several reference suspensions of different fiber concentrations be prepared in sample bottles. These

bottles can then be stored for preparation and analysis along with water samples of unknown fiber concentrations.

7. CALCULATION OF RESULTS

The results are conveniently calculated using a computer program, the listing of which is provided in Appendix B. The methods by which the calculations are made are described below.

7.1 Test for Uniformity of Fiber Deposit on Electron Microscope Grids

A check must be made using the chi-square test, to determine whether the fibers found on individual grid openings are randomly and uniformly distributed among the grid openings. If the total number of fibers found in k grid openings is n, and the areas of the k individual grid openings are designated A_1 to A_k, then the total area examined is

$$A = \sum_{i=1}^{i=k} A_i$$

The fraction of the total area examined which is represented by the individual grid opening area, p_i, is given by A_i/A. If the fibers are randomly and uniformly dispersed over the k grid openings counted, the expected number of fibers falling in one grid opening with area A_i is np_i. If the observed number found on that grid opening is n_i, then:

$$\chi^2 = \sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

This value is compared with significance points of the χ^2 distribution, having (k - 1) degrees of freedom. Significance levels lower than 0.1% are cause for the sample analysis to be rejected, since this corresponds to a very inhomogeneous deposit. If this occurs, a new filter should be prepared, paying more attention to both uniform dispersal of the suspension and the filtration procedure as described in Section 6.3.2.

7.2 Calculation of the Mean and Confidence Interval of the Fiber Concentration

In the fiber count, a maximum of 20 grid openings have been sampled from a population of grid openings, and it is required to determine the mean grid opening fiber count for the population on the basis of this sampling. The interval about the sample mean, which, with 95% confidence, contains the population mean, is also required.

The distribution of fibers on the grid openings should theoretically approximate a Poisson distribution. Because of fiber aggregation and size-dependent identification effects, the fiber count data often do not conform to the Poisson distribution, particularly at high fiber counts. Simple assumption of a Poisson distribution may therefore lead to confidence intervals narrower than are justified by the data. Moreover, if a Poisson distribution is assumed, the variance is fixed in relation to the total number of fibers counted. Thus a particular fiber count conducted on one grid opening is considered to have the same confidence interval as that for the same number of fibers found on many grid openings. However, the area of sample actually counted is very small in relation to the total area of the filter, and for this reason fibers must be counted on a minimum of 4 grid openings taken from different areas of the filter in order to ensure representative evaluation of the deposit.

At high fiber counts, where there are adequate numbers of fibers per grid opening to allow a sample estimate of the variance to be made, the distribution can be approximated to Gaussian, with independent values for the mean and variance. Where the sample estimate of variance exceeds that implicit in the Poissonian assumption, use of Gaussian statistics with the variance defined by the actual data is the most conservative approach to calculation of confidence intervals.

At low fiber counts, it is not possible to obtain a reliable sample estimate of the variance, and the distribution also becomes asymmetric, but not necessarily Poissonian. For 30 fibers and below, the distribution becomes sufficiently asymmetric that the Gaussian fit is no longer a reasonable one, and sample variance estimates are unreliable. Accordingly, for fiber counts below 31 fibers, the assumption of a Poisson distribution must be made for calculation of the confidence intervals.

For total fiber counts less than 5, the lower 95% confidence value corresponds to one fiber or less, and in addition, the upper 95% confidence value corresponding to a fiber count of zero is 3.69 fibers. Therefore, it is not meaningful to quote lower confidence interval points for fiber counts of less than 5, and the result should be specified as "less than" the corresponding Poisson upper 95% confidence value.

For fiber counts higher than 30, the sample estimate of variance is also calculated, and the larger of the two confidence intervals is selected. For calculation of Poisson 95% confidence intervals, Table 40 of the reference by E.S. Pearson and H.O. Hartley should be used, with an extension to an expectation of 100. For more than 100 fibers, the Poisson distribution can be accurately approximated by a Gaussian distribution, still using the Poisson variance estimate. For counts of more than 30 fibers, the 95% confidence interval based on a sample estimate of variance is calculated using the Student's "t" distribution. For the two-sided Student's "t" calculation, k values of grid opening fiber count are compared with the expected values for the areas of the grid openings concerned.

In summary, fiber counting data will be reported as follows:

<u>No fibers detected</u>

The value will be reported as less than 369/ of the concentration equivalent to one fiber.

<u>1 to 4 fibers</u>

When 1 to 4 fibers are counted, the result will be reported as less than the corresponding upper 95% confidence limit (Poisson).

<u>5 to 30 fibers</u>

Mean and 95% confidence intervals will be reported on the basis of the Poisson assumption.

<u>More than 30 fibers</u>

When more than 30 fibers are counted, both the Gaussian 95% confidence interval and the Poisson 95% confidence interval will be calculated. The larger of these 2 intervals will be selected for data reporting. When the Gaussian 95% confidence interval is selected for data reporting, the Poisson interval will also be noted.

Fiber counts performed on less than 4 grid openings yield very wide 95% confidence intervals when using Gaussian statistics. This is because the value of Student's "t" is very large for 1 and 2 degrees of freedom. Accordingly, fiber counts must not be made on less than 4 grid openings.

The sample estimate of variance S^2 is first calculated:

$$S^2 = \frac{\sum_{i=1}^{i=k} (n_i - np_i)^2}{(k - 1)}$$

where:

n_i = Number of fibers on the i'th grid opening

n = Total number of fibers found in k grid openings

p_i = Fraction of the total area examined represented by the i'th grid opening

k = Number of grid openings

For the 95% confidence interval, the value of $t_{0.975}$ is obtained from tables for (k - 1) degrees of freedom. If the mean value of fiber count is calculated to be $\bar{n}$, the upper and lower values of the 95% confidence interval are given by:

$$n_U = \bar{n} + \frac{ts}{\sqrt{k}}$$

$$n_L = \bar{n} - \frac{ts}{\sqrt{k}}$$

where:

n_U = Upper 95% confidence limit

n_L = Lower 95% confidence limit

$\bar{n}$ = Mean number of fibers per grid opening

s = Standard deviation (square root of sample estimate of variance)

k = Number of grid openings

The fiber concentration in MFL which corresponds to counting of one fiber is given by:

$$C = \frac{A_f \times R_D}{A \times V \times 1000}$$

where:

A_f = Effective filtration area of filter membrane in mm^2 used for filtration of liquid sample

A = Total area examined in mm^2

V = Original volume of sample filtered (mL)

R_D = Dilution ratio of original sample

The mean concentration in MFL is obtained by multiplying the mean number of fibers per grid opening by kC. To obtain the upper and lower 95% confidence limits for the concentration (in MFL) multiply the values n_U and n_L by kC.

7.3 Estimated Mass Concentration

The mass of each amphibole fiber in micrograms is calculated using the relationship:

$$M = L \times W^2 \times D \times 10^{-6}$$

where:

M = Mass in micrograms

L = Length in µm

W = Width in µm

D = Density of fiber in g/cm^3

For chrysotile, the mass may be calculated using the relationship for a cylinder:

$$M = \frac{\pi}{4} \times L \times W^2 \times D \times 10^{-6}$$

The estimated mass concentration is then given by:

$$M_c = C \times \sum_{i=1}^{i=n} M_i \times 10^6$$

where:

M_c = Mass concentration in µg/L

C = fiber concentration in MFL, which corresponds to counting of one fiber

M_i = Mass of the i'th fiber, in micrograms

n = Total number of fibers found in k grid openings

The densities to be assumed are as follows:

Chrysotile	2.55	g/cm^3
Crocidolite	3.37	g/cm^3
Cummingtonite	3.28	g/cm^3
Grunerite	3.52	g/cm^3
Amosite	3.43	g/cm^3
Anthophyllite	3.00	g/cm^3
Tremolite	3.00	g/cm^3
Actinolite	3.10	g/cm^3
Unknown Amphibole	3.20	g/cm^3

7.4 Fiber Length, Width, Mass and Aspect Ratio Distributions

The distributions all approximate to logarithmic-normal, and so the size range intervals for calculation of the distribution must be spaced logarithmically. The other characteristics required for the choice of size intervals are that they should allow for a sufficient number of size classes, while still retaining a statistically-valid number of fibers in each class. Interpretation is also facilitated if each size class repeats at decade intervals. A ratio from one class to the next of 1.468 satisfies all of these requirements. The other constraints are that the length distribution should include 0.5 um as one interval point, since this is the minimum length to be counted in the method, and the minimum aspect ratio is by definition 3.0. The resulting size classes for the various distributions can be seen in the example shown in Appendix B. The distributions, being approximately logarithmic-normal, must be plotted using a logarithmic ordinate scale and a Gaussian abscissa.

7.4.1 Fiber Length Cumulative Number Distribution

This distribution allows the fraction of the total number of fibers either shorter or longer than a given length to be determined. It is calculated using the relationship:

$$C_{(N)k} = \frac{\sum_{i=1}^{i=k} n_i}{\sum_{i=1}^{i=N} n_i} \times 100$$

where:

$C_{(N)k}$ = Cumulative number percentage of fibers which have lengths less than the upper bound of the k'th class

n_i = Number of fibers in the i'th length class

N = Total number of length classes

7.4.2 Fiber Width Cumulative Number Distribution

This distribution allows the fraction of the total number of fibers either narrower or wider than a given width to be determined. It is calculated in a similar way to that used in 7.4.1 for the length distribution.

7.4.3 Fiber Length Cumulative Mass Distribution

This distribution allows the fraction of the total mass incorporated in fibers either shorter or longer than a given length to be determined. It is computed using the relationship:

$$C_{(M)k} = \frac{\sum_{i=1}^{i=k} \sum_{j=1}^{j=n_i} l_j w_j^2}{\sum_{i=1}^{i=N} \sum_{j=i}^{j=n_i} l_j w_j^2} \times 100$$

where:

$C_{(M)k}$ = Cumulative mass percentage of fibers which have lengths less than the upper bound of the k'th class

n_i = Number of fibers in the i'th length class

l_j = Length of the j'th fiber in the i'th length class

w_j = Width of the j'th fiber in the i'th length class

N = Total number of length classes

7.4.4 Fiber Aspect Ratio Cumulative Number Distribution

This distribution allows the fraction of the total number of fibers which have aspect ratios either smaller or larger than a given aspect ratio to be determined. It is

calculated in a similar way to that used in 7.4.1 for the length distribution.

7.4.5 Fiber Mass Cumulative Number Distribution

This distribution allows the fraction of the total number of fibers which have masses either smaller or larger than a given mass to be determined. It is calculated by placing the fibers into logarithmically-spaced mass categories, after which the cumulative frequency distribution is obtained in a similar way to that used in 7.4.1 for the length distribution.

7.5 Index of Fibrosity

It is possible to discriminate between amphibole asbestos fibers and amphibole cleavage fragments on the basis of the distribution of their aspect ratios. The concept of fibrosity in a mineral embodies a high median aspect ratio, together with a large spread of aspect ratios above the median value. A single number can be used to describe the fibrosity of a mineral fiber dispersion, and in many cases the value can be used to state if the material is or is not asbestos. The fibrosity index can be defined thus:

$$F = R^g$$

where R is the median of the aspect ratio distribution and g is the geometric standard deviation of the aspect ratio distribution above the median. The value of g is obtained from that portion of the distribution lying between one and two geometric standard deviations above the median. Meaningful values of the index of fibrosity can be obtained for most waterborne fiber dispersions if more than 50 fibers have been measured.

The fibrosity index as defined above has values exceeding 100 for waterborne dispersions of asbestos. Values below 50 indicate a distribution characteristic of cleavage fragments, or one from which the high aspect ratio fibers have been selectively removed.

8. REPORTING

The computer program provided in Appendix B satisfies all of the reporting requirements for this analytical method, and it is recommended that this format be used. The size classifications used must be the same as those in Appendix B.

8.1 Before the fiber count data can be processed to give concentration values, a decision must be made as to which fiber classifications are to be considered adequate as identification of the fiber species in question. This decision will depend on how much is known about the particular source from which the sample was collected.

For a sample from a completely uncharacterized source, the following procedure will be used to accumulate the classified fibers:

a) Confirmed Amphibole: AZZQ + AZQ + AZZ (solutions must include only amphiboles)

b) Amphibole Best Estimate*: AZZQ + AZQ + AZZ + AZ + ADQ + AQ

c) Suspected Amphibole: ADX + AX + AD

d) Confirmed Chrysotile: CDQ + CD

e) Chrysotile Best Estimate*: CDQ + CD + CMQ + CQ

f) Suspected Chrysotile: CM

*NOTE: Best estimate can be reported only if some fibers are also reported in the confirmed category, otherwise all fiber classifications must be reported as suspected amphibole or chrysotile.

8.2 The concentration in MFL, together with 95% confidence intervals, will be reported for the groupings in Section 8.1 (a) to (f).

8.3 Two significant figures will normally be used for concentrations greater than 1 MFL, and one significant figure for concentrations less than 1 MFL.

8.4 For confirmation of chrysotile, a micrograph and a calibrated diffraction pattern will be provided from a typical fiber. The identification features in Figure 17 must be visible on the diffraction pattern.

For confirmation of amphibole, either (1) or (2) or (3) below must be provided for a typical fiber of each amphibole variety reported. The data provided must yield solutions which include only amphibole.

1) A micrograph, a calibrated zone axis SAED pattern, and an EDXA spectrum together with peak area measurements and EDXA calibration data;

2) A micrograph, and two calibrated zone axis SAED patterns with a measurement of the angular rotation between the two patterns;

3) A micrograph, two calibrated zone axis SAED patterns with a measurement of the angular rotation between the two patterns, and an EDXA spectrum together with peak area measurements and EDXA calibration data.

8.5 Tabulate the length, width and aspect ratio distributions.

8.6 Report the estimated mass concentration in ug/L for each of the groupings in Section 8.1 (a) to (f).

8.7 One significant figure will normally be used for reporting mass concentration.

8.8 Report the concentration in MFL corresponding to one fiber detected.

8.9 Report the total number of fibers counted in each of the groupings in Section 8.1 (a) to (f).

8.10 Report the X^2 value for each of the groupings in Section 8.1 (a) to (f).

8.11 Report the number of fiber aggregates not included in the fiber count

8.12 Report any special circumstances or observations such as aggregation, presence of organic materials, amount of debris, presence of other fibers and their probable identity if known.

9. LIMITATIONS OF ACCURACY

9.1 Errors and Limitations of Identification

Complete identification of every chrysotile fiber is not possible, due to both instrumental limitations and the nature of some of the fibers. The requirement for a calibrated SAED pattern eliminates the possibility of an incorrect identification of the fiber selected. However, there is a possibility of misidentification of other chrysotile fibers for which both morphology and SAED pattern are reported on the basis of visual inspection only. The only significant possibilities of misidentification occur with halloysite, vermiculite scrolls or palygorskite, all of which can be discriminated from chrysotile by the use of EDXA and by observation of the 0.73 nm (002) reflection of chrysotile in the SAED pattern.

As in the case of chrysotile fibers, complete identification of every amphibole fiber is not possible due to instrumental

limitations and the nature of some of the fibers. Moreover, complete identification of every amphibole fiber is usually not practical due to limitations of both time and cost. Particles of a number of other minerals having compositions similar to those of some amphiboles could be erroneously classified as amphibole when the classification criteria do not include zone axis SAED techniques. However, the requirement for quantitative EDXA measurements on all fibers as support for the random orientation SAED technique makes misidentification very unlikely, particularly when other similar fibers in the same sample have been identified as amphibole by zone axis methods. The possibility of misidentification is further reduced with increasing aspect ratio, since many of the minerals with which amphibole may be confused do not display its prominent cleavage parallel to the c-axis.

9.2 Obscuration

If large amounts of other materials are present, some asbestos fibers may not be observed because of physical overlapping. This will result in low values for the reported asbestos content.

9.3 Inadequate Dispersion

If the initial water sample contains organic material which is incompletely oxidized in the ozone-UV treatment, it will not be possible to disperse any fibers associated with the organic material. This may lead to adhesion of some fibers to the container walls and aliquots taken during filtration will then not be representative. It may also lead to a large proportion of fiber aggregates which are either not transferred during the replication and filter dissolution step or which cannot be counted during the sample examination. The result obtained from such an analysis will be low. The sample will also be inadequately dispersed if it is not treated in an ultrasonic bath prior to filtration, and therefore instructions regarding this treatment must be followed closely.

9.4 Contamination

Contamination by introduction of extraneous fibers during the analysis is an important source of erroneous results, particularly for chrysotile. The possibility of contamination, therefore, should always be a consideration.

9.5 Freezing

The effect of freezing on asbestos fibers is not known but there is reason to suspect that fiber breakdown could occur and result in a higher fiber count than was present in the original sample. Therefore, the sample should be transported to the laboratory and stored under conditions that will avoid freezing.

10. PRECISION AND ACCURACY

10.1 General

The precision that can be obtained is dependent upon the number of fibers counted, and on the uniformity of particulate deposit on the original filter. If 100 fibers are counted and the loading is at least 3.5 fibers/grid square, computer modeling of the counting procedure shows that a relative standard deviation of about 10% can be expected. As the number of fibers counted decreases, the precision will also decrease approximately as $\sqrt{N}$ where N is the number of fibers counted. In actual practice, some degradation from this precision will be observed. This degradation is a consequence of sample preparation errors, non-uniformity of the filtered particulate deposit, and fiber identification variability between operators and between instruments. The 95% confidence interval about the mean for a single fiber concentration measurement using this analytical method should be about ±25% when about 100 fibers are counted over 20 grid openings. For these conditions the precision of the computed mass concentration is generally lower than the precision for the fiber number concentration. The precision to be expected for a single determination of mass concentration is critically dependent on the fiber width distribution. For a result based on measurement of a minimum of about 100 fibers, the 95% confidence interval about the mean computed mass concentration may vary between ±25% and ±60%. If better precision is required for a mass determination, the alternative counting method described in Section 6.5.5 should be used.

10.2 Precision

10.2.1 Intra-Laboratory Comparison Using Environmental Water Sources

Table 6 shows the results obtained from analysis of 10 replicate samples from each of 8 water sampling locations. Four of these locations were associated with a source of chrysotile and four associated with a source of amphibole. It can be seen that the relative standard deviations of the number concentrations range between 13% and 22%. The corresponding relative standard deviations for the mass concentrations range between 29% and 69%.

10.2.2 Inter-Laboratory Comparison of Filters Prepared Using Standard Dispersions and Environmental Water Sources

Tables 7 and 8 show the fiber counting results obtained when sectors of filters prepared in the ORF Laboratory were distributed to six laboratories considered experienced in asbestos analysis by the identification and counting techniques incorporated in this manual. The samples as

TABLE 6. INTRA-LABORATORY COMPARISON OF ENVIRONMENTAL WATER SAMPLES

Type of Fiber in Water Source	Sampling Location	Number of Samples	Number Concentration Results			Mass Concentration Results			Mean Number of Fibers Counted per sample
			Fiber Concentration, f/L		Relative Standard Deviation	Mass Concentration, µg/L		Relative Standard Deviation	
			Mean	95% Confidence Interval		Mean	95% Confidence Interval		
Chrysotile	Domestic Faucet	10	44.3	39.7 - 48.9	15%	0.27	0.19 - 0.35	41%	140
	Domestic Faucet	10	39.7	34.6 - 44.8	18%	0.25	0.19 - 0.32	37%	144
	River	10	15.7	14.0 - 17.3	15%	0.14	0.07 - 0.20	68%	29
	Treatment Plant	10	24.9	22.5 - 27.3	14%	0.17	0.13 - 0.20	29%	93
Amphibole	Domestic Hose	10	16.6	15.1 - 18.1	13%	12.4	9.0 - 15.8	38%	190
	Domestic Hose	10	9.73	8.93 - 10.5	12%	7.8	5.21 - 10.4	46%	110
	Lakeside	10	5.25	4.72 - 5.79	14%	6.55	3.32 - 9.78	69%	38
	Treatment Plant	10	3.42	2.88 - 3.97	22%	2.18	1.45 - 2.92	47%	44

TABLE 7. INTER-LABORATORY COMPARISON: STANDARD DISPERSIONS

Laboratory	Sample 1 Concentration, MFL			Sample 2 Concentration, MFL			Sample 3 Concentration, MFL		
	Mean	95% Confidence Interval	Number of Fibers Counted	Mean	95% Confidence Interval	Number of Fibers Counted	Mean	95% Confidence Interval	Number of Fibers Counted
1	9.16	7.02 - 11.3	81	28.2	20.1 - 36.3	300	1.58	1.17 - 1.99	69
2	9.48	4.79 - 14.2	66	32.3	Not Available	75	2.67	1.82 - 3.52	56
3	12.1	9.20 - 15.0	86	27.0	19.6 - 34.4	186	1.68	1.17 - 2.19	55
4	9.26	7.12 - 11.4	96	20.3	11.2 - 29.4	150	1.76	0.97 - 2.55	32
5	9.66	7.37 - 11.9	85	34.0	28.9 - 39.1	284	1.18	0.85 - 1.51	42
6	8.99	1.22 - 16.7	55	16.2	3.27 - 29.1	202	1.90	1.14 - 2.65	[illegible]
Mean Concentration	9.74			26.3			1.79		
Standard Deviation	0.32			6.89			0.49		
Relative Standard Deviation	8.4%			26%			27%		
Fiber Species Reported	Chrysotile			Chrysotile			Amphibole		

TABLE 8. INTER-LABORATORY COMPARISON: ENVIRONMENTAL WATER SAMPLES

Laboratory	Sample 4			Sample 5		
	Concentration, MFL		Number of Fibers Counted	Concentration, MFL		Number of Fibers Counted
	Mean	95% Confidence Interval		Mean	95% Confidence Interval	
1	1.71	1.19 - 2.23	48	10.6	9.06 - 12.1	113
2	1.47	0.61 - 2.33	27	5.87	3.30 - 8.44	41
3	2.11	1.36 - 2.86	29	6.10	4.92 - 7.28	76
4	2.50	2.01 - 2.99	35	6.74	5.22 - 8.26	79
5	1.31	0.63 - 1.09	19	5.17	4.14 - 6.20	72
6	1.53	0.62 - 2.44	30	6.27	0.72 - 11.8	78
Mean Concentration	1.77			6.79		
Standard Deviation	0.45			1.94		
Relative Standard Deviation	25%			29%		
Fiber Species Reported	Amphibole			Chrysotile		

distributed were identified by number only. In Table 7 it can be seen that the relative standard deviations for the six results on each of the standard dispersion filters did not exceed 27%. In Table 8, the environmental water sources used to prepare the filter samples contained similar types of suspended materials as those used to generate the intra-laboratory results in Table 6. The relative standard deviations do not exceed 29%, which appears higher than the values obtained for the intra-laboratory results. However, when the 6 inter-laboratory results are compared with the 10 intra-laboratory values, there is no statistically significant difference to indicate that there has been any degradation of precision.

10.3 Accuracy

10.3.1 Intra- and Inter-Laboratory Comparison of Standard Dispersions of Asbestos Fibers

Tables 9 and 10 show the results obtained between two laboratories when stable aqueous fiber dispersions of known mass concentrations were analyzed. The fiber concentrations reported displayed no significant difference between values from the two laboratories. The relative standard deviation of the mean fiber concentration was 17% for chrysotile and 16% for crocidolite. The corresponding relative standard deviations for the mass concentration were 16% for chrysotile, and 37% for crocidolite. The higher variability for crocidolite is a consequence of the low statistical reliability of the large diameter fiber counts. The computed mean mass concentration for chrysotile was about 46% higher than the known mass concentration. This may be a consequence of the difficulty of diameter measurement for single chrysotile fibrils or the assumption of the bulk value for the density. The computed mean value for mass concentration for the crocidolite sample was 67.4 µg/L, which is very close to the known concentration of 50 µg/L.

TABLE 9. INTER- AND INTRA-LABORATORY COMPARISON: CHRYSOTILE

ANALYSIS OF REPLICATES OF A 1120 ng/L CHRYSOTILE FIBER DISPERSION

Laboratory	Sample	Fiber Concentration, MFL		Estimated Mass Concentration, ng/L	Number of Fibers Counted
		Mean	95% Confidence Interval		
1	1C	210	165 - 255	1792	147
	2C	199	149 - 248	1435	147
	3C	196	166 - 226	1352	141
2	4C	132	89 - 176	1600	50
	5C	172	146 - 198	1980	79
Mean Concentration		182		1632	
Standard Deviation		31		257	
Relative Standard Deviation		17%		16%	

TABLE 10. INTER- AND INTRA-LABORATORY COMPARISON: CROCIDOLITE

ANALYSIS OF REPLICATES OF A 50 μg/L CROCIDOLITE FIBER DISPERSION

Laboratory	Sample	Fiber Concentration, MFL		Estimated Mass Concentration, μg/L	Number of Fibers Counted
		Mean	95% Confidence Interval		
1	1A	148	112 - 183	94.0	218
	2A	143	123 - 163	89.4	195
	3A	136	98 - 175	66.4	195
2	4A	188	132 - 243	33.0	73
	5A	189	149 - 229	54.1	178
Mean Concentration		161		67.4	
Standard Deviation		26		25.3	
Relative Standard Deviation		16%		37%	

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Appendix 18

(ASTM D 3195-90) Standard Practice for Rotameter Calibration

Designation: D 3195 – 90 (Reapproved 1998)$^{\epsilon 1}$

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Rotameter Calibration[1]

This standard is issued under the fixed designation D 3195; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—Paragraph X1.5 was corrected editorially in October 1998.

1. Scope

1.1 This practice covers the calibration of variable-area flowmeters (rotameters) used to determine air sample volumes at or close to ambient conditions of pressure and temperature, in the analysis of atmospheres for pollutant content.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples[2]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[3]

D 3631 Test Methods for Measuring Surface Atmospheric Pressure[3]

E 1 Specification for ASTM Thermometers[4]

E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice, refer to Terminology D 1356.

3.1.2 *standard conditions* are taken as 25°C and 101.3 kPa (760 mm Hg) at existing ambient humidity. This conforms to most of the ASTM methods for atmospheric sampling and analysis that involve volumetric corrections. Absolute temperature scales are to be used when substituting values into the formulae used in this procedure.

4. Summary of Practice

4.1 Two alternative methods of performing the required volume determinations for rotameter calibration are described:

4.1.1 Using the water-sealed rotating drum meter (wet test meter). See Section 7.

4.1.2 Using the volumetric gasometer (bell prover). See Section 8.

5. Significance and Use

5.1 Choice of method depends primarily on which equipment is available. Higher accuracy is possible with the gasometer. The accuracies of the methods of atmospheric analysis, for which the calibration procedure is intended, do not warrant the very highest possible accuracy in flow measurement.

6. Apparatus

6.1 *Wet Test Meter, or Volumetric Gasometer*, with water seal and equipped with a water manometer on the inlet.

6.2 *Counter Balance Weights*, for gasometer.

6.3 *Mercury Barometer*—See Test Methods D 3631.

6.4 *Psychrometer*, (if room air is used for calibration gas). See Test Method E 337.

6.5 *Thermometer*, to measure ambient temperature. See Specification E 1.

6.6 *Stopwatch.*

6.7 *Air Supply*, either a cylinder of compressed air or a diaphragm type pump of adequate capacity and a ballast volume or restrictor to eliminate pulsations.

6.8 *Needle Valve.*

7. Procedure Using Wet Test Meter

7.1 Unless it was already calibrated within the previous 3 months, calibrate the wet test meter by Test Methods D 1071. The method described in Section 19 is recommended for highest accuracy.

7.2 Set up the apparatus as shown in Fig. 1, making connections as short as possible and large enough inside diameter to avoid any appreciable pressure drops.

7.3 Before and after the complete calibration run, record room temperature, barometric pressure in accordance with Test Methods D 3631, and relative humidity (when room air is used for calibrating gas) in accordance with Test Method E 337. Use

[1] This practice is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheresand is the direct responsibility of Subcommittee D22.01on Quality Control.

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

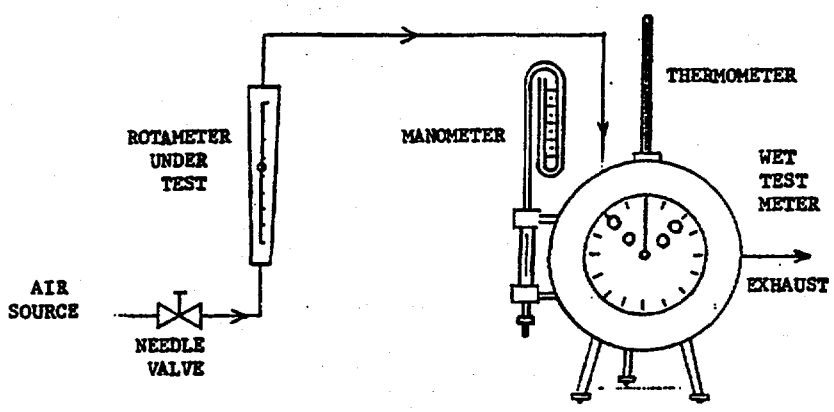

FIG. 1 Calibration Assembly Using Wet Test Meter

average values for subsequent calculations.

7.4 Start air flowing through the rotameter and wet test meter. Adjust the flow to the desired rate with the needle valve. Take a pair of timed readings on the wet test meter, under steady flow, for each of five or more uniformly spaced points on the rotameter scale, going from low values to high values. Repeat, going from high to low. Note the manometer reading and meter water temperature for each meter reading.

8. Procedure Using Gasometer

8.1 Unless it was already calibrated within the previous six months, in the same location, calibrate the gasometer by Test Methods D 1071.

8.2 Set up the apparatus as shown in Fig. 2, making connections as short as possible and large enough inside diameter to avoid any appreciable pressure drops.

8.3 Before and after the complete calibration run, record the room temperature, barometric pressure, and relative humidity (when room air is used for calibrating gas). Use average values for subsequent calculations.

8.4 Start air flowing through the rotameter and into the gasometer. Adjust the flow to the desired value with the needle valve.

8.5 Adjust eight counterbalance weights on the gasometer as required to maintain no greater than 2 in. of water back-pressure when operating.

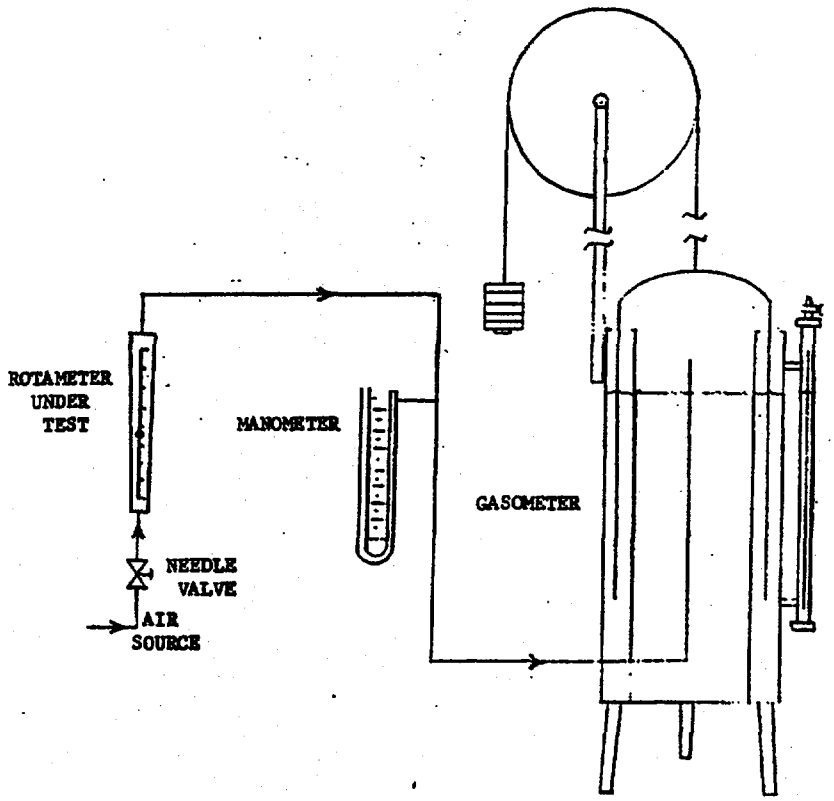

FIG. 2 Calibration Assembly Using Gasometer

8.6 Take a pair of timed readings on the gasometer scale, under steady flow, for each of five or more uniformly spaced points on the rotameter scale going from low values to high values. Repeat, going from high to low.

9. Calculations

9.1 Convert all temperature and pressure readings to absolute units, as follows:

$$°F + 460 = °R \quad (1)$$

$$°C + 273 = K$$

$$\text{in. of water} \times 0.249 = \text{kPa}$$

$$\text{in. of water} \times 0.0737 = \text{in. Hg}$$

$$\text{in. of water} \times 1.87 = \text{mm Hg}$$

9.2 Calculate the indicated flow readings for all recorded rotameter points by dividing the indicated delta volumes by the time.

9.3 Using the following equation, convert these indicated flow readings to actual flows that would be indicated by the rotameter if it were calibrated for air at the standard conditions stated in 5.2:

$$Q_1 = Q(P_m - D)T_s/P_m T_m \sqrt{T_s/T_a} \quad (2)$$

where: (see Appendix)

Q_1 = flow rate rotameter should indicate,
Q = flow rate indicated by wet test meter or gasometer,
T_s = standard temperature (298 K or 537°R),
T_m = meter temperature (water temperature for wet test meter; room temperature for gasometer),
T_a = room temperature,
P_m = gas pressure during calibration (inlet pressure for wet test meter; barometric pressure for gasometer) (kPa, in water, mm Hg), and
D = vapor pressure of water in the calibrating gas (kPa, in water, mm Hg).

9.4 Prepare the calibration curve by best fit to all points. It should be labeled "... at 25°C and 101.3 kPa (760 mm Hg)."

10. Keywords

10.1 calibration; flowmeter; rotameter

APPENDIX

(Nonmandatory Information)

X1. DERIVATION OF FLOWMETER EQUATION

X1.1 The equation is based on the premise that the calibrated rotameter should read air flow correctly at standard conditions, as defined. Therefore, in order to prepare the calibration curve it is first necessary to convert the wet test meter or gasometer readings to the values that would be indicated by the rotameter if it had been calibrated under standard conditions. This can be done logically in several steps. First, the indicated values are corrected for the water vapor added by the wet test meter or gasometer, assuming saturation, by operating with the factor:

$$P_m - D/P_m \quad (X1.1)$$

where:

P_m = gas pressure during calibration (inlet pressure for wet test meter; barometric pressure for gasometer), and
D = vapor pressure of water at 100 % R.H. and temperature T_m, minus the vapor pressure of water in the calibrating gas.

X1.2 Next, the volume measured in the wet test meter or gasometer is corrected to what it was in the rotameter. This factor is:

$$T_a/T_m \quad (X1.2)$$

where:

T_a = room temperature, and
T_m = meter temperature (water temperature for wet test meter; room temperature for gasometer).

X1.3 This is what the rotameter should read but if it were calibrated with air at standard temperature and used to measure air at this different temperature, the viscosity effect would cause a slightly different reading. Since viscosity is proportional to the square root of absolute temperature (independent of pressure up to 10 atmospheres), the indicated flow would differ from actual flow by the factor:

$$\sqrt{T_a/T_s} \quad (X1.3)$$

where:

T_s = standard temperature (298 K or 537°R)

X1.4 Putting these elements all together gives us the equation indicated in the body of the method.

X1.5 Subsequent use of the rotameter normally involves taking the indicated flow off the curve and correcting it to standard conditions. The factor for this correction is:

$$T_s P_r / T_a P_s \sqrt{T_s/T_a} \quad (X1.4)$$

where:

P_r = rotameter pressure, and
P_s = standard pressure (760 mm Hg or 101.3 kPa)

NOTE X1.1—In many cases, P_r is, or can be assumed to be, the same as barometric pressure. However, when any question exists, and for highest accuracy, a water manometer should be used, just downstream of the rotameter.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

Appendix 19

(ISSI-Libby-02) Reflectance Spectroscopy Screening for Asbestos in Soil

Date: January 5, 2000 (Rev. # 0) SOP No. ISSI-LIBBY-02

Title: REFLECTANCE SPECTROSCOPY SCREENING FOR ASBESTOS IN SOIL

APPROVALS:

Author _____ ISSI Consulting Group, Inc. Date: January 5, 2000

SYNOPSIS: A standardized method for spectral screening for asbestos in soil samples is described. Protocols for sample handling and analysis are provided.

Received by QA Unit:

REVIEWS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8		
ISSI Consulting Group, Inc.		

Revision Date	Reason for Revision

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for analyzing soil samples for asbestos-form mineral occurrence, to be used by employees of USEPA Region 8, or contractors/subcontractors supporting USEPA Region 8 projects and tasks. This technique will be used as a screening tool to identify samples that contain asbestos-form minerals. The spectral identification of asbestos-form minerals is limited by the analytical instrumentation, the abundance of asbestos-form materials present in each sample, and the physico-chemical (e.g., composition, moisture content, grain size distribution) state of the material containing the asbestos-form minerals.

This SOP describes the equipment and operations used for analyzing asbestos in surface soils, which will produce data that can be used to support risk evaluations. Site-specific deviations from the procedures outlined in this document must be approved by the USEPA Region 8 On Scene Coordinator, or Science Support Coordinator prior to initiation of the analysis.

It should be noted that this procedure is continually being developed, and may be modified or revised as needed. Any revisions to this SOP will be provided in the final report.

2.0 RESPONSIBILITIES

The Field Project Leader (FPL) may be an USEPA employee or contractor who is responsible for performing reflectance spectroscopy analysis of asbestos in soil. The FPL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to communicate with the Field Personnel regarding specific analysis objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the FPL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (On Scene Coordinator or Science Support Coordinator).

All personnel performing reflectance spectroscopy analysis are responsible for adhering to the applicable tasks outlined in this procedure while analyzing samples.

3.0 EQUIPMENT

- Portable Spectrometer - Various models are acceptable and selection of the specific brand and model will be recommended by the contractor performing the analysis (CDM Federal). The instrument must be able to detect spectral effects in the 0.5 - 2.5 micron wavelength region, at a resolution of 8 - 11 nanometers.

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Personal Protective Equipment - as specified in the contractor's Health and Safety Plan.
- Field notebook -used to record progress of analysis effort and record any problems and field observations.
- Three-ring binder book - to store necessary forms used to record and track samples and analysis results. Binders will contain sample logbook pages for tracking samples, and data analysis sheets that record reflectance spectroscopy results for each sample.

4.0 INSTRUMENTATION

A hand-held portable spectrometer will be used to identify asbestos-form minerals. This instrument must be able to detect spectral effects in the 0.5 - 2.5 μm wavelength region, at a resolution of 8 - 11 nanometers, using a halogen light source. Reflectance data of the samples of interest are compared to spectral standards in the United States Geological Survey (USGS) spectral library, using modified least-squares analysis techniques.

5.0 PRECISION AND ACCURACY

The precision and accuracy of hand-held portable spectrometers is determined by the calibration procedure and post-processing computer program selected by the operator. Precision and accuracy requirements should be determined prior to analyzing any samples. The procedure for optimizing resolution and decreasing signal noise and interference is described in the manufacturer's operating manual.

6.0 METHODOLOGY

Samples are analyzed using a general purpose spectroradiometer that measures either relative or absolute light energy. Inside the instrument, light is projected from the fiber optics onto a holographic diffraction grating where the wavelength components are separated and reflected for independent collection by the detector. Spectral data are generated by converting incident photons into electrons that are stored or integrated for read-out. At read-out time, the photoelectric current for each detector is converted to a voltage and then digitized. The digital data is then transferred directly to the instrument's computer memory. Spectral data for each sample is then compared to a library of spectra that have been generated for various minerals and mineral classes.

6.1 Sample Preparation

All samples will be prepared according to SOP #ISSI-LIBBY-01. Following preparation, soil samples will be analyzed using a hand-held portable spectrometer with a halogen light source.

6.2 Analytical Procedure

The specific steps in collecting reflectance measurements are outlined in the manufacturer's operating manual. The following general steps should be followed:

1. Turn on the instrument
2. Turn on the computer
3. At the prompt, enter the controlling software
4. Enter experimental conditions. Position the instrument probe to view the radiant source/sample that is being examined. The source must fill the field of view of the probe.
5. Optimize the unit for the lighting conditions, and inform the software of the foreoptic being used, if any.
6. Viewing the radiant source/sample, collect a white reference (e.g., Spectralon®) measurement, and view the sample as if in the reflectance mode. If the signal/noise ratio is not acceptable, increase the spectrum averaging.
7. Set up the spectrum averaging to fit the operating conditions. When the signal/noise ratio is acceptable, go back to the raw data collection mode.
8. When all setup procedures are complete, and the unit is collecting spectra, save data regularly by pressing the Spacebar on the keyboard.
9. Periodically collect Dark Current measurements, as described in the manufacturer's operating manual. Reoptimize in varying light conditions, or if any data appear "truncated", i.e., if any spectral humps have flat tops.

Raw data can be processed into radiance data with the postprocessing utility, included in the software package for each instrument. Correct setup procedures must be followed in order to calculate accurate data. Foreoptic attachment selection, for example, **cannot** be forgotten or misstated, as this program will not work if the unit is not calibrated for the foreoptic selected.

Following analysis, record results for each sample on the data analysis sheets.

6.3 Data Presentation

Sample results will include sample identification numbers, a copy of the spectrum generated for each sample, and a qualitative (e.g., presence or absence) or semi-quantitative (e.g., relative abundance) indication of asbestos-form minerals.

6.4 Instrument Calibration and Standardization

All spectra must be fully corrected to absolute reflectance before any analysis can be performed. Reflectance measurements are made relative to a "white" reference material, such as Spectralon®.

Reflectance is derived by the ratio of the sample to the reference material. Calibration procedures are described in the manufacturer's operating manual. The instrument must be calibrated at the beginning of each sample analysis run, and repeated according to the manufacturer's recommendation.

7.0 PERSONAL HEALTH AND SAFETY

The contractor responsible for performing reflectance spectroscopy analysis must have a Health and Safety Plan in place prior to analyzing any samples. All personnel are required to follow the procedures described in the Health and Safety Plan.

8.0 SAMPLE LABELING AND DOCUMENTATION

Each sample must be labeled with a unique identification number, as described in the Project Plan. All samples must be logged into the sample logbook prior to analysis, and results must be recorded on the data analysis sheets. Sample logsheets, and data analysis sheets will be maintained in a three-ring binder.

In addition, a field notebook should be maintained by each individual or team that is analyzing samples. The following information must be recorded for each day that samples are analyzed.

1. date
2. time
3. personnel
4. calibration results, including what reference materials were used
5. identification numbers for each sample analyzed
6. descriptions of any deviations to the Project Plan or this SOP and the reason for the deviation

9.0 REFERENCES

Clark, Roger N. Spectroscopy of rocks and minerals, and principles of spectroscopy. USGS, MS 954. 1999

Appendix 19

(ISSI-Libby-02) Reflectance Spectroscopy Screening for Asbestos in Soil

Date: January 7, 2000 (Rev. # 1) SOP No. ISSI-LIBBY-02

Title: REFLECTANCE SPECTROSCOPY SCREENING FOR ASBESTOS IN SOIL

APPROVALS:

Author Sam Vance USGS Date: January 7, 2000

SYNOPSIS: A standardized method for spectral screening for asbestos in soil samples is described. Protocols for sample handling and analysis are provided.

Received by QA Unit:

REVIEWS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8		1/7/00
ISSI Consulting Group, Inc.	W J Battles	1/7/00

Revision Date	Reason for Revision
1/7/00	Incorporated comments from USGS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for analyzing soil samples for asbestos-form mineral occurrence, to be used by employees of USEPA Region 8, or contractors/subcontractors supporting USEPA Region 8 projects and tasks.

This technique will be used as a screening tool to identify samples that contain asbestos-form minerals. The spectral identification of asbestos-form minerals is limited by the analytical instrumentation, the abundance of asbestos-form materials present in each sample, and the physico-chemical (e.g., composition, moisture content, grain size distribution) state of the material containing the asbestos-form minerals.

This SOP describes the equipment and operations used for analyzing asbestos in surface soils, which will produce data that can be used to support risk evaluations. Site-specific deviations from the procedures outlined in this document must be approved by the USEPA Region 8 On Scene Coordinator, or Science Support Coordinator prior to initiation of the analysis.

It should be noted that this procedure is continually being developed, and may be modified or revised as needed. Any revisions to this SOP will be provided in the final report.

2.0 RESPONSIBILITIES

The Field Project Leader (FPL) may be an USEPA employee or contractor who is responsible for performing reflectance spectroscopy analysis of asbestos in soil. The FPL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to communicate with the Field Personnel regarding specific analysis objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the FPL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (On Scene Coordinator or Science Support Coordinator).

All personnel performing reflectance spectroscopy analysis are responsible for adhering to the applicable tasks outlined in this procedure while analyzing samples.

3.0 EQUIPMENT

- Portable Spectrometer - Various models are acceptable and selection of the specific brand and model will be recommended by the contractor performing the analysis (U. S. Geological Survey Spectroscopy Lab). The instrument must be able to detect spectral effects in the 0.4 - 2.45 micron wavelength region. Over the 0.4 to 2.35 micron wavelength range a bandpass of better than 14 nanometers and spectral wavelength sampling better than 14 nanometers is required. The bandpass and sampling may

increase at wavelengths from 2.35 to 2.45 microns without detriment to mineral identification, if both bandpass and sampling remain less than 35 namometers.

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Personal Protective Equipment - as specified in the contractor's Health and Safety Plan.

- Field notebook -used to record progress of analysis effort and record any problems and field observations.

- Three-ring binder book - to store necessary forms used to record and track samples and analysis results. Binders will contain sample logbook pages for tracking samples, and data analysis sheets that record reflectance spectroscopy results for each sample.

4.0 INSTRUMENTATION

A reflectance spectrometer will be used to identify asbestos-form minerals. This instrument must be able to detect spectral effects in the 0.4 - 2.45 micron wavelength region, at a bandpass and spectral wavelength sampling as specified in #3.0 above, using a light source that covers the spectral range. Reflectance data of the samples of interest are compared to spectral standards in the United States Geological Survey (USGS) spectral library, using standard spectroscopic analysis methods.

5.0 PRECISION AND ACCURACY

The precision and accuracy of spectrometers is determined by the calibration procedure and post-processing computer program selected by the operator. Precision and accuracy requirements should be determined prior to analyzing any samples. The procedure for optimizing resolution and decreasing signal noise and interference is described in the manufacturer's operating manual.

6.0 METHODOLOGY

Samples are analyzed using a general purpose spectroradiometer that measures either relative or absolute light energy as a function of wavelength. Spectral data are generated by converting incident photons into electrons that are stored or integrated for read-out. At read-out time, the photoelectric current for each detector is converted to a voltage and then digitized. The digital data is then transferred to the instrument's computer memory. Spectral data for each sample is then compared to a library of spectra that have been generated for various minerals and mineral classes.

6.1 Sample Preparation

All samples will be prepared according to SOP #ISSI-LIBBY-01. Following preparation, soil samples will be analyzed using a spectrometer.

6.2 Analytical Procedure

The specific steps in collecting reflectance measurements are outlined in the manufacturer's operating manual. The following general steps should be followed:

1. Turn on the instrument
2. Turn on the computer
3. At the prompt, enter the controlling software.
4. Enter experimental conditions.
5. Position the light source to illuminate the sample/reflectance standard.
6. Position the instrument probe to view the sample/reflectance standard that is being examined. The sample/reflectance standard must fill the field of view of the probe.
7. Optimize the unit for the lighting conditions, as described in the manufacturer's operating manual
8. Viewing the reflectance standard, collect a white reference (e.g., Spectralon®) measurement. If the signal/noise ratio is not acceptable, increase the spectrum averaging, or integration time.
9. When all setup procedures are complete, and the unit is collecting spectra, save data as described in the manufacturer's operating manual.
10. **Collect spectra of wavelength standards (e.g. as supplied by the manufacturer), and reference minerals appropriate to the project, such as a tremolite for asbestos form mineral identification.**
11. Periodically collect Dark Current measurements and white reference measurements, as described in the manufacturer's operating manual.
12. All spectra must be post processed to remove spectral effects of the white reference standard, e.g. as described in Clark *et al.*, 1990.

Raw data can be processed into reflectance data with a postprocessing utility such as commercial software, spreadsheets or other established public software.

6.3 Data Presentation

Sample results will include sample identification numbers, a copy of the spectrum generated for each sample, and a qualitative (e.g., presence or absence) or semi-quantitative (e.g., relative abundance) indication of asbestos-form minerals.

6.4 Instrument Calibration and Standardization

All spectra must be fully corrected to absolute reflectance before any analysis can be performed. Reflectance measurements are made relative to a "white" reference material, such as Spectralon®. The reflectance of the standard must be removed from the sample spectra, as described in Clark *et al.*, 1990.

Reflectance is derived by the ratio of the sample to the reference material, and is usually performed inside the instrumentation of modern commercial spectrometers. Calibration procedures are described in the manufacturer's operating manual for both wavelength and intensity. The instrument must be calibrated at the beginning of each sample analysis run, and repeated according to the manufacturer's recommendation.

7.0 PERSONAL HEALTH AND SAFETY

The contractor responsible for performing reflectance spectroscopy analysis must have a Health and Safety Plan in place prior to analyzing any samples. All personnel are required to follow the procedures described in the Health and Safety Plan.

8.0 SAMPLE LABELING AND DOCUMENTATION

Each sample must be labeled with a unique identification number, as described in the Project Plan. All samples must be logged into the sample logbook prior to analysis, and results must be recorded on the data analysis sheets. Sample logsheets, and data analysis sheets will be maintained in a three-ring binder.

In addition, a field notebook should be maintained by each individual or team that is analyzing samples. The following information must be recorded for each day that samples are analyzed.

1. date
2. time
3. personnel
4. calibration results, including what reference materials were used
5. identification numbers for each sample analyzed
6. descriptions of any deviations to the Project Plan or this SOP and the reason for the deviation

9.0 REFERENCES

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