

## **SAMPLING AND ANALYSIS SUMMARY REPORT**

### **ROADSIDE AIRBORNE ASBESTOS MONITORING ALONG AN EL DORADO COUNTY SERPENTINE ROADWAY (Initial Study and Post Resurfacing Study)**

Volpe Center IAG VP262  
Sponsor Doc 01-T2226

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Prepared for:

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Approved by:  Date: 01 / 27 / 05

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## SECTION 1. INTRODUCTION

In a previous study conducted in Garden Valley, California, the Department of Toxic Substances Control (DTSC) with support from the United States Environmental Protection Agency (USEPA) collected bulk samples from several roads, bus stops, two quarries and a road cut within the community. The objective of the study was to identify potential sources of NOA releases to the air. Results of this study are found in "Report on Surface Soil Sampling for Naturally Occurring Asbestos, Garden Valley, California" prepared by DTSC and dated October 2002. The report concluded that the primary source of NOA in the community is found in the unpaved roads. The report recommended that further studies be conducted to quantify the asbestos emissions from roads in the community.

In 2002, The DTSC established an Interagency Agreement (IAG) with the U.S. Department of Transportation's (USDOT) John A. Volpe National Transportation Systems Center (Volpe Center). The IAG was established to assist DTSC to address concerns regarding potential exposure of communities to airborne asbestos fibers resulting from vehicular traffic along unpaved roadways known to contain asbestos within the Garden Valley community. Volpe Center support included providing DTSC with a variety of technical and scientific services related to assessing NOA emissions from unpaved roads.

The purpose of this report is to summarize the sampling and analysis activities conducted by DTSC and the Volpe Center for a roadside air monitoring study performed on Slodusty Road. Slodusty Road was an unpaved serpentine road located within the community of Garden Valley in El Dorado County, California. The report also summarizes related findings and recommendations. This report does not include any health assessment analysis of the airborne asbestos concentrations that were identified to be present within proximity of Slodusty Road.

The study involved monitoring of air-entrained asbestos associated with vehicular traffic along Slodusty Road, in Garden Valley, CA. Slodusty Road was selected by DTSC as being representative of unpaved serpentine-surfaced roadways that exist in the region and that are known to contain asbestos within the aggregate road surfacing materials. In cooperation with DTSC, the Volpe Center conducted sampling activities along Slodusty road during July 2002 as part of an Initial Study.

The objectives of the Initial Study were:

- 1) to practice, examine and refine methodologies for collecting airborne asbestos dust samples to refine sampling strategies and protocol; and
- 2) to collect initial airborne asbestos data associated with vehicular traffic.

Following the Initial Study, in early August 2003 DTSC resurfaced Slodusty Road with surfacing materials that did not contain asbestos. Approximately one week later the Volpe Center returned with DTSC to resample along the roadway. The objective of the follow-up sampling was to assess the effectiveness of the resurfacing in reducing airborne asbestos concentrations, near Slodusty Road.

## **SECTION 2. PROJECT BACKGROUND**

### **2.1 Contaminant of Concern: Asbestos**

The only contaminant of concern being investigated at the site is naturally occurring asbestos. Asbestos is a generic term for a group of six naturally occurring, fibrous silicate minerals. Asbestos minerals fall into two groups: serpentine asbestos and amphibole asbestos. Serpentine asbestos, which includes the magnesium silicate mineral chrysotile, possesses relatively long, flexible crystalline fibers. Amphibole asbestos, which includes the silicate mineral series tremolite through actinolite, forms crystalline fibers that are shorter and substantially more brittle than serpentine asbestos. All asbestos fibers are odorless and tasteless. Fibers are microscopic and environmentally persistent, as they are chemically inert and do not evaporate, dissolve, burn or undergo reactions with most chemicals. Separated asbestos fibers are strong and flexible enough to be spun and woven. Due to its resistance to heat and most chemicals, as well as its ability to be “woven “ asbestos fibers have historically been used for a wide range of manufactured products. In 1989 the EPA banned all new uses of asbestos, due to associated health effects. Asbestos has been classified as a known human carcinogen, by State, Federal, and International agencies.

The community of Garden Valley, California, is located between two deposits of serpentinite rock (often called serpentine and referred to in all other sections of this report as serpentine) (DOC 2000, DTSC 2000), a metamorphosed igneous rock composed essentially of the mineral serpentine as well as other ferromagnesium silicate minerals, including naturally occurring asbestos (NOA) minerals. Chrysotile asbestos is often associated with serpentinite deposits, though amphibole asbestos can also be found in some serpentinite deposits (Klein, 1998). Disturbance of serpentinite rock (e.g. through mining or crushing of the serpentinite rock) may release NOA fibers into the air.

### **2.2 Location and Environmental Setting**

The study site is located in the community of Garden Valley, in El Dorado County, California. The study area is within five miles of the South Fork of the American River, which is used extensively for recreational activities during the summer. The City of Sacramento is located approximately 60 miles to the west.

Winters are cool and moist. The average January temperature in Sacramento is 53°Fahrenheit (F) The average January precipitation in Sacramento is 3.6 inches. Summers are hot and dry. The average July temperature is 93°F, and the average July precipitation is less than 0.5 inches based on data from the National Weather Service. The area is occasionally subject to significant winds.

Potential for exposure to NOA, particularly during excavation activities, has been an active environmental health issue in El Dorado County for several years



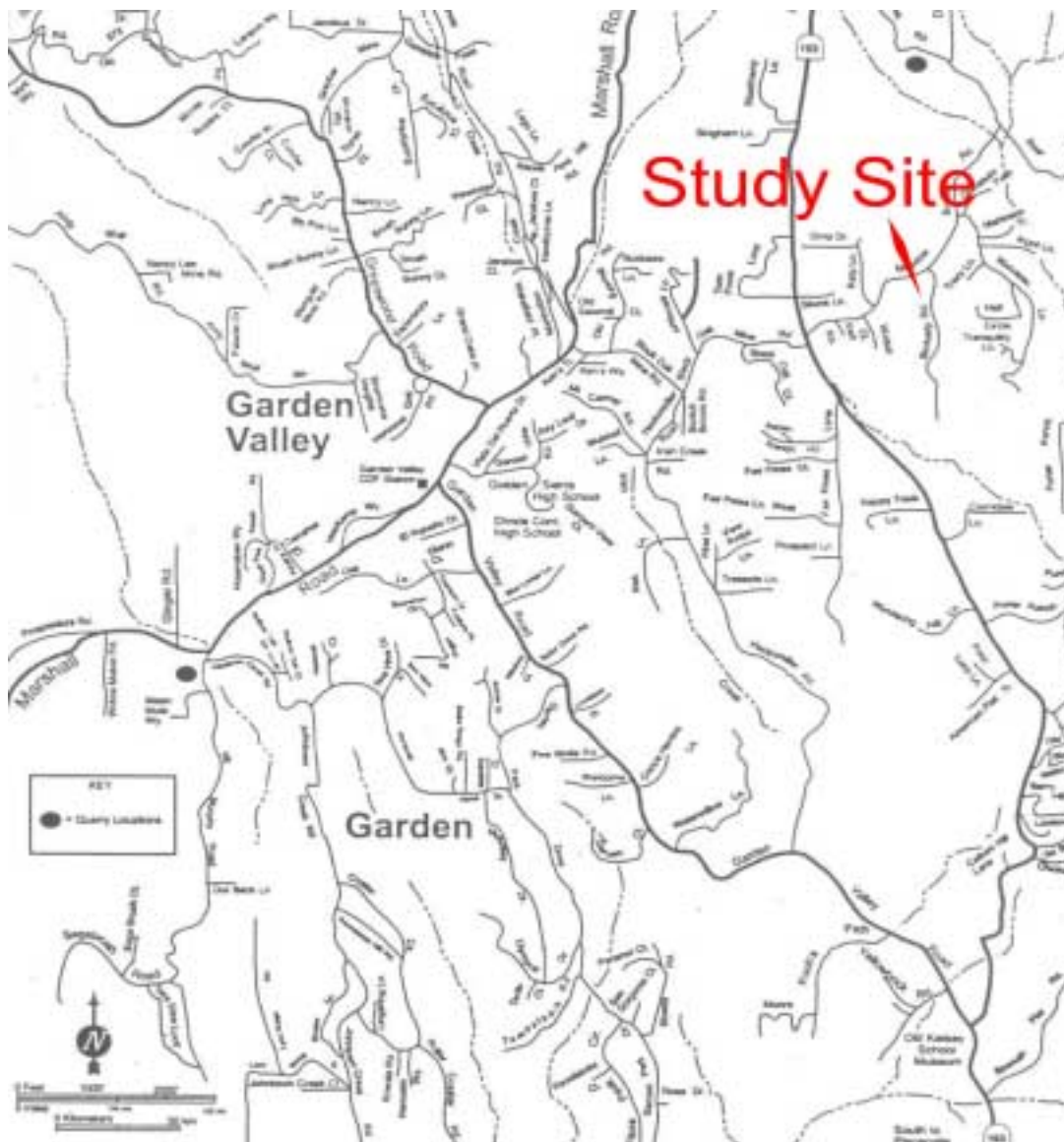
**MAP OF CALIFORNIA SHOWING PRINCIPAL ASBESTOS DEPOSITS**

**EXPLANATION**

- SERPENTINE AND PERIDOTITE
- AMPHIBOLE ASBESTOS
- CHRYSOTILE ASBESTOS
- DEPOSITS OUTSIDE OF SERPENTINE AREAS

**Garden Valley Community**

Slodusty Road is the emission study site selected within Garden Valley (Figure 2-2). Slodusty Road is a north-south trending road that primarily services a small residential population of 35 to 50 people. Vegetation around Slodusty Road is sparse, consisting of manzanita (*Arctostaphylos* spp.), buck brush (*Ceanothus cuneatus*), and digger pines (*Pinus sabiniana*). The straight segment of road is open on either side for a few hundred feet with only a few trees. Thus there is only minimal obstruction of air movement in the vicinity of the road. Residents along the road strongly encourage and have posted a maximum speed limit of ten miles per hour (mph). The test site was an approximately 250 ft. stretch of straight road located about 1/4 mile south of where Slodusty Road meets Meadow Brook Road. At the time of the Initial Study, the roadway was unpaved and surfaced with asbestos-containing serpentine material. In early August 2003 DTSC resurfaced the roadway using surface materials that did not contain asbestos. DTSC's resurfacing approach involved a multi layer approach of compacted  $\frac{3}{4}$  aggregate, with a chipseal barrier and top surface of fine Lime Stone aggregate. The purpose of this multi layer approach was to act as a barrier to the road bed's asbestos-containing serpentine material.



**Figure 2-2. Regional map, with study site (Slodusty Road) highlighted.**

### 2.3 Previous Investigations

The California Air Resources Board (CARB) had conducted air monitoring for NOA at various sites in Garden Valley intermittently from April 1998 through May 2001 (see summarized results in CARB 2001). CARB data showed levels of asbestos in the air above air monitoring results in other areas of the state where NOA is not known to occur. As a result DTSC and USEPA believed that the potential sources of NOA in the community warranted assessment.

DTSC collected soil and bulk road samples in August 2000 (DTSC 2000) at potential asbestos source areas in and around Garden Valley; source areas were selected in part based on their apparent potential contribution to air emissions.

Results indicated the presence of asbestos in the bulk road material used for surfacing roads in the community including Slodusty Road. This study and the analytical results for the soil are provided separately within "Surface Soil Sampling for Naturally Occurring Asbestos, Garden Valley, California", October 2002 prepared by DTSC

## **SECTION 3. FIELD ACTIVITIES AND SAMPLING PROCEDURES**

The purpose of sampling activities along Slodusty Road was to evaluate asbestos emissions from a typical serpentine covered roadway by observing airborne asbestos concentration levels as they vary with distance from the road, traffic frequency, and vehicle speed.

The Volpe Center provided the majority of the necessary materials, equipment, and supplies, as well as the field personnel to conduct all of the sampling activities. DTSC provided some of the air sampling equipment, additional meteorological equipment, the test vehicles and additional field support to assist with conducting the traffic simulations.

This section describes the field activities conducted during both the 2002 (Initial Study) and 2003 (post resurfacing) sampling events. The following field activities were performed:

- Traffic Simulations
- Air Sampling
- Meteorological Monitoring
- Particulate Dust Measurements
- Documentation of Field Activities

In addition (to above) DTSC personnel collected bulk samples from the road material. Sampling methods and results are described in a separate report prepared by DTSC titled "Slodusty Road Bulk Sampling Results"

### **3.1 General Methodology**

This section describes the general methodology used to measure meteorological conditions and asbestos emissions from Slodusty Road during controlled traffic scenarios. Details of sampling methodology are described in subsequent sections on Field Sampling and Instrumentation.

Day 0 and Day 1 (7/15 & 7/16/02) of the Initial Study were used to test equipment, optimize locations for samplers and instruments and define traffic scenarios to test. The selected traffic scenarios were conducted on Day 2 and Day 3 (7/17 & 7/18/02). The Post resurfacing runs include a no vehicle run to determine background and repeating the traffic scenarios conducted during Day 2 and Day 3 of the Initial Study to measure differences in emissions.

#### ***Traffic Scenarios:***

After testing different vehicle speeds and observing traffic patterns during Day 0 and Day 1 of the Initial Study, two mile per hour (mph) and vehicle per hour (vph) traffic scenarios were selected for testing:

1. vehicles traveling at 25 mph at a frequency of 30 vph
2. vehicles traveling at 10 mph at a frequency of 10 vph.

The 25 mph / 30 vph scenario was chosen to represent an extreme worst case traffic condition for the road. While the 10 mph / 10 vph scenario was chosen to represent actual speed and highest vehicle frequency conditions that may be encountered along Slodusty during peak commute.

Two DTSC-owned vehicles, a Dodge Ram 4x4 truck and a Chevy Cavalier compact sedan, alternately drove along the test stretch of road, maintaining the test speed for a distance of 110 feet on either side of the sampling transect (total distance speed was maintained=220 feet). A "traffic controller" noted the time samplers were started and then immediately began the controlled traffic flow via radio communication with the drivers. The test vehicle frequency was maintained as closely as possible, with local traffic asked to either move very slowly (speed <10 mph) through the test site in the interim period between pass-bys of the test vehicles *or* to move through the test site at the test speed, thereby serving as a substitute for the test vehicle during that particular pass-by (whenever a local car passed, it was noted by the traffic controller). DTSC public information personnel, positioned at both ends of the test stretch of road, served as "traffic controllers" and directed local cars by stopping the local drivers, informing them of the study, and, via radio communication with the traffic controller, telling the local drivers when and how fast to proceed through the test section. Each traffic run was conducted for two hours. After the Run was completed all samplers were simultaneously shut down and the filter cassettes from the air monitors were collected for laboratory analysis. If another Run was to take place that day, set-up would then begin with each sampler outfitted with a fresh filter-cassette. If no other Run was to occur that day, then the equipment was torn-down.

***Instrumentation:***

Air samplers were installed on both (east and west) sides of the road, along a transect that was approximately perpendicular to the center-point of the test stretch of road. Each sampler was outfitted with one 25 mm mixed cellulose ester (MCE) membrane filter (0.45  $\mu$ m pore size). Additionally, meteorological (MET) station(s) monitoring wind speed, wind direction, air temperature and humidity were installed on site. MET instruments were changed during the course of the testing to obtain more accurate measurements. Discussions of these changes are described in section 4.1.

During the entire Run, flow rates on the samplers were monitored to ensure that air flow was steady. Flow rates were set and monitored using a rotometer attached to the sampler. A digital flowmeter was used to set the flow rates and calibrate the flow meters before and after each test run. The pre and post flow rates were averaged and used to calculate the actual volume of air sampled.

Quality Assurance and Quality control samples were also collected and are further described in the sections 5.1.2 and 5.2.2.

All sampling activities were documented with detailed notes and digital photographs. Additionally, video footage of all traffic simulations was collected. Refer to Appendix A for additional details regarding air sampling procedures. Representative field pictures are included within Appendix G of this report.

### **3.2 Initial Study**

In cooperation with DTSC, the Volpe Center conducted the Initial Study sampling along Slodusty Road between July 15<sup>th</sup> and 18<sup>th</sup> 2002. The goals of the Initial Study included the following:

- 1) determine the maximum volume of air that may be collected at various distances from the roadway without overloading the samples with particulate (this is an important consideration for laboratory analysis of the field samples, i.e., samples do not require an indirect prep unless they are overloaded);
- 2) determine spatial sampler locations adequate to characterize the dispersion of asbestos from the road bed; and
- 3) determine simulated traffic frequencies and speed necessary to provide sufficient data for development of the relationship between vehicular traffic on unpaved asbestos-containing roadways and the resulting airborne asbestos fibers.
- 4) collect initial airborne asbestos data.

The sampling team included the Volpe Center and DTSC project managers, four Volpe Center personnel and up to eight DTSC field personnel. Five types of sampling data were collected as part of the Initial Study:

- 1) Stationary air samples were established at various distances perpendicular to the roadway (see Section 3.2.1 below), which were used to observe the dispersion of asbestos from the roadbed;
- 2) Personal samples were collected to monitor worker exposure (one representative sample was taken for each task); and
- 3) MET station(s) were established to monitor wind speed, wind direction, air temperature and humidity.
- 4) In addition DTSC performed air monitoring of particulates, via spot measurements.
- 5) DTSC staff collected bulk samples from the road. The bulk sample collection and results are described in a separate report titled "Slodusty Road Bulk Sampling Results" prepared by DTSC.

***Objectives for each day:***

Day 0 (7/15/02) objectives were to:

- (1) Practice all aspects of conducting a sampling “Run” and determine maximum appropriate vehicle speed on Slodusty Road Rd.;
- (2) Test samplers, MET stations, and other equipment.

Day 1 (7/16/02) objectives were to:

- (1) Determine maximum achievable flow rates by collocating multiple samplers;
- (2) Test the utility of using 47 mm filters (to see if they don't get overloaded as quickly as do the 25 mm filters)

Day 2 (7/17/02) objectives were to:

- (1) Gauge maximum flow rates at all distances,
- (2) Determine the distance from the source where asbestos fibers approach background levels, and
- (3) Establish detailed hi-vol flow rate calibration curves (a primary standard flowmeter was unavailable for day 1 & day 2).

Sample flow rates and distances for day 2 were adjusted based on results from the previous days sampling. Day 1 showed that many samples were overloaded and that asbestos fibers were present at all distances, including the 100 foot locations. Two runs were conducted, Run 02-01 & Run 02-02 (see characteristics below).

Day 3 (7/18/02) objective was to:

- (1) Gauge “typical” dispersions that may be encountered along Slodusty Road by simulating a vehicle speed and frequency that is representative of actual speed and highest vehicle frequency conditions along Slodusty Road during peak commute. Based on observations from the previous three days of sampling; vehicle frequencies were observed to be less than 10 vph, so 10 vph was used as a conservative estimate. The posted vehicle speed on Slodusty Road is 10 mph, and it was observed that the traffic was mostly residential and the residents respected the low speed limit.

**3.2.1 Initial Study Sample Station Locations**

Air samplers were set up on both the east and the west sides of the road along a transect that was approximately perpendicular to the center-point of the test stretch of road (Figure 3-1). Samplers were generally placed at 5, 10, 30, 50 80, 100, 130 160, and 190 feet from the road. Prior to each test run, each sampler was outfitted with one filter of either 25 or 47 millimeters (mm) in diameter. On 07/15/02 (Day 0 of study), polycarbonate filters (0.4 micrometer (µm) pore size) were used while mixed cellulose ester (MCE) membrane filters (0.45 µm pore

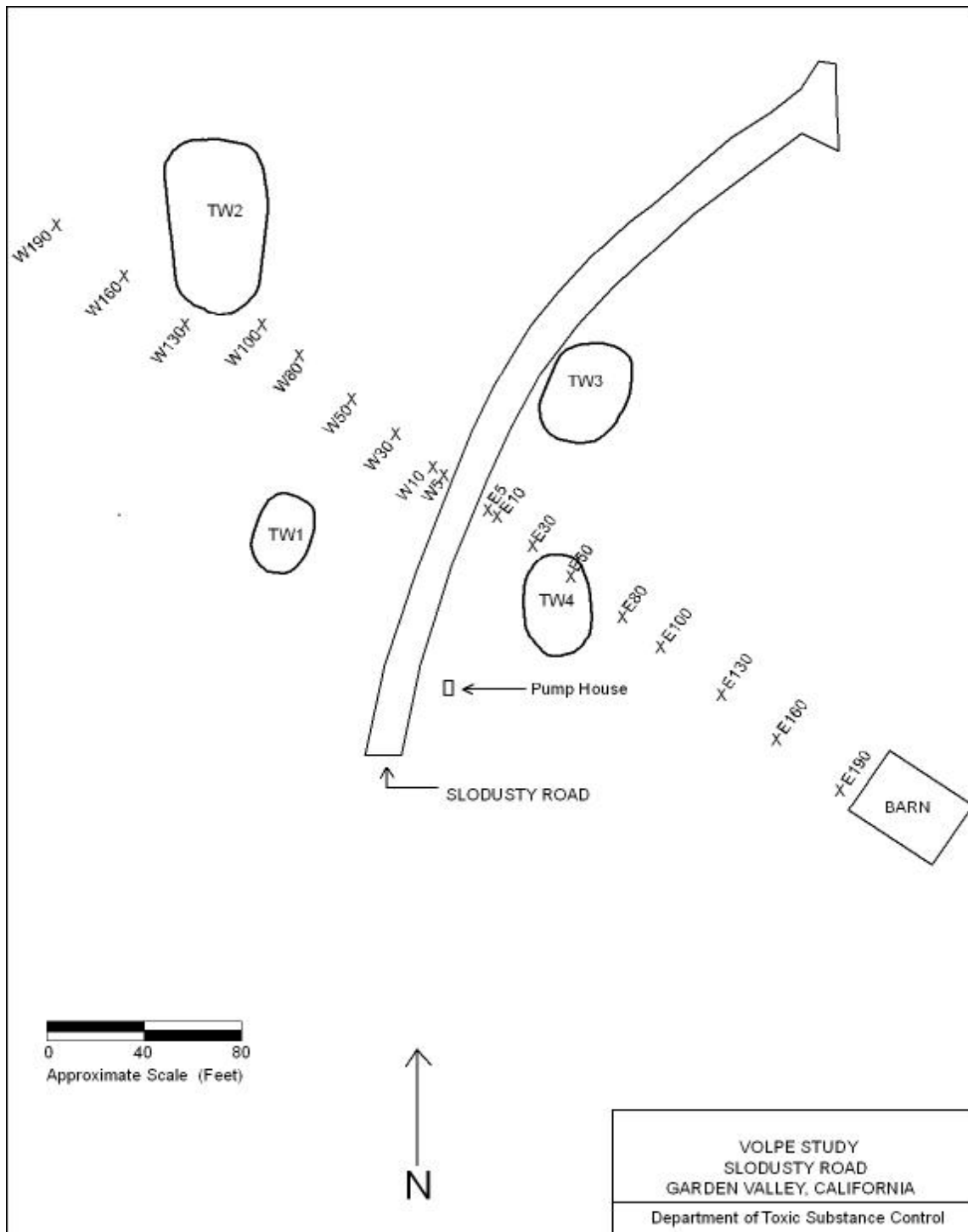


size) were used on all other days. Additionally, MET station(s) monitored wind speed, wind direction, air temperature and humidity. MET stations were placed at varying locations in order to verify meteorological conditions were consistent throughout the test area.

#### MET Station Locations

<b>STUDY</b>	<b>DAY</b>	<b>MET Station Locations</b>
Initial Study	Day 0	1 MET Sta. on each side at 30 ft
Initial Study	Day 1	2 MET Sta. on each side at 25 ft and 90 ft
Initial Study	Day 2	2 MET Stations on the west side at 25 ft and 95 ft, a third at 100 ft on the east side, and a fourth was located approximately 100 yds NE of the sampling area (adjacent to the video camera)
Initial Study	Day 3	1 MET Sta. on each side at 30 ft
Post Resurfacing	Day 1	1 MET Sta. on each side at 50 feet
Post Resurfacing	Day 2	1 MET Sta. on each side at 50 feet
Post Resurfacing	Day 3	1 MET Sta. on each side at 50 feet

**Figure 3-1. Map of Initial Study site, (TW=tree, X=air sampler (E or W of road and distance (feet) away from road sampler was located))**



### 3.2.2 Sampling Activities

Below is a summary of the Initial Study sampling activities that occurred between July 15 and July 18, 2002. Individual sample data is presented in Appendix B.

#### ***Day 0 (07/15/02) - Setup***

Test vehicles were driven at different speeds over the road segment to determine the maximum safe speed for the road that may be encountered. The maximum speed deemed to be safe was 25 miles per hour. Slodusty Road posted speed limit is 10 mph. These speeds were selected for the test runs.

Air monitors and MET stations were setup at locations described below. At each location, one hi-vol sampler with a 25-mm filter was set up; additionally, at the 30 ft. locations, a mini-vol sampler with a 47-mm filter was also set up. Following completion of site set-up activities one sampling run was performed, Run 00-01 (see characteristics below).

Two personal samples were collected, one on the sedan driver and one on the traffic controller.

#### Run 00-01 Characteristics

Vehicle Frequency	30 vehicles per hour (vph)
Vehicle Speed	25 mph (determined to be max speed)
Distances Sampled	5, 10, 30, 50, 80, and 100 ft
Target Flow Rates	5, 6, 8, 10, and 15 L / min (depending on distance)
MET Stations	1 MET Sta. on each side at 30 ft
Duration	2 hrs

#### ***Day 1 (07/16/02)***

Several air samplers were collocated to test reproducibility and maximum flow rates of sample collection instrumentation. Also, some samplers were equipped with 47 mm diameter filters to test whether the 47 mm get overloaded as quickly as the 25 mm filters. Two runs were conducted, Run 01-01 & Run 01-02 (see characteristics below). At each location, 1-3 hi-vol sampler(s) with either a 25-mm filter or 47-mm filter were set up.

Three personal samples were collected, one in the pick-up truck, one field personnel and one on the traffic controller.

In addition, for both Runs DTSC personnel collected spot measurements of particulate concentrations in the air at the various sample locations west of the roadway.

Run 01-01 Characteristics

Vehicle Frequency	10 vph
Vehicle Speed	25 mph
Distances Sampled	5, 10, 30, 50, 80, and 100 ft
Target Flow Rates	6, 8, 10, 12 and 15 L/min (depending on distance)
MET Stations	2 MET Sta. on each side at 25 ft and 90 ft
Duration	2 hrs

Run 01-02 Characteristics

Identical to Run 01-01 except the Run was conducted with a higher vehicle frequency (30 vph vs. 10 vph in Run 01-01).

**Day 2 (07/17/02)**

Day 1 showed that many sample filters were overloaded and that asbestos fibers were present at all distances, including the 100 foot locations. Sample flow rates and distances for Day 2 were adjusted based on results from the previous days sampling as described below. All samplers were set-up with 25mm filters since early analytical results indicated that the loading affects were similar between the 25mm and 47 mm filters. Also, laboratory observations indicated the 47mm filter loadings had a tendency to be more centralized rather than uniformly distributed across the filter. Because the asbestos was detected in the 100 foot sampler, additional sample stations were added at 130, 160 and 190 feet from the road.

A vehicle frequency of 30 vehicles per hour was selected to represent a reasonable upper bound maximum number of vehicles per hour that traveled on Slodusty Road. As such the test run scenario of 30 vehicles per hour at 25 mph was determined to be the reasonable maximum scenario for traffic for this study.

Two runs were conducted, Run 02-01 & Run 02-02 (see characteristics below). At each location, one or two hi-vol sampler(s) with 25-mm filters were positioned. Three personal samples were collected, two field personnel and one on the traffic controller.

Run 02-01 Characteristics

Vehicle Frequency	30 vph
Vehicle Speed	25 mph
Distances Sampled	5, 10, 30, 50, 80, 100, 130, 160 and 190 ft
Target Flow Rates	2, 4, 6, 8, 10 and 12 L/min (depending on distance)
MET Stations	2 MET Stations on the west side at 25 ft and 95 ft, a third at 100 ft on the east side, and a fourth was located approximately 100 yds NE of the sampling area (adjacent to the video camera)

Duration	2 hrs
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**Run 02-02 Characteristics**

This run was identical to Run 02-01, except that a primary standard flowmeter was used to set the flow rates. Flow rates for all previous Runs were set and monitored through the use of a rotometer attached to the sampler. Due to equipment failure, a primary flowmeter was not available to calibrate or set flow rates until Days 2 and 3 of the study. It was discovered through using the flowmeter that the rotometer was often quite inaccurate. The flow rates used in Days 0 and 1 were suspect because it was unknown whether the “dialed in” rotometer flow rates were the true flow rates at which the samplers were operating. In order to estimate the actual volumes collected during Day 0 and Day 1 a five-point calibration curve was developed for all samplers using the primary flow meter. Based on this curve, an actual flow rate ( $Q_{act}$ ) was gauged on Day 2, and applied to the earlier runs in order to calculate a total volume sampled. This volume is termed "Estimated Total Volume" (see Appendix B). For Day 2 Run 02-02, Day 3 and all Post Resurfacing Runs, flowmeters were used to calibrate and set flow rates to the true desired flow rate at the beginning of each Run. At the end of each Run, the flow rate was measured again. The beginning and end flow rates were then averaged and used to calculate the actual volume of air sampled.

**Day 3 (07/18/02)**

Ten vehicles per hour traveling at 10 mph was determined to be the “typical” scenario for this study. The posted speed limit is 10 mph and informal traffic observations during the study showed that 10 vehicles per hour approximated a conservative average of the traffic on Slodusty Road.

At each location, one hi-vol sampler with a 25-mm filter was set up. Three personal samples were collected, two field personnel and one on the sedan driver.

**Run 03-01 Characteristics**

Vehicle Frequency	10 vph
Vehicle Speed	10 mph
Distances Sampled	5, 10, 30, 80, and 130 ft
Target Flow Rates	8, 10 and 12 L/min (depending on distance)
MET Stations	1 MET Sta. on each side at 30 ft
Duration	2 hrs

### 3.3 Post Resurfacing Sampling

DTSC completed resurfacing Slodusty Road in early August 2003. Approximately 1 week following resurfacing (Aug 18 – 20, 2003) the Volpe Center returned with DTSC to resample along the roadway. The goal of the follow-up sampling was to assess the effectiveness of the resurfacing in reducing airborne asbestos concentrations, near Slodusty Road.

The sampling team consisted of Volpe Center staff, the DTSC project manager and four DTSC support field personnel. Four types of sampling data were collected by Volpe and DTSC personnel, as part of the Post Resurfacing sampling activities (following procedures established by the Initial Study):

- 1) Stationary air samples were established at various distances perpendicular to the roadway (see Section 3.3.1 below), which were used to observe the dispersion of asbestos from the roadbed;
- 2) Personal samples were collected to monitor worker exposure (one representative sample was taken for each task);
- 3) MET stations were established to monitor wind speed, wind direction, and air temperature.
- 4) DTSC performed air monitoring of particulates, as well as collected additional data on air temperature and humidity.

#### ***Objectives for each day:***

##### Day 1 (8/18/03) objective was to:

Set-up site equipment and collect background and site samples representative of actual field conditions

##### Day 2 (8/19/03) objective was to:

Repeat the traffic scenarios used during the Initial Study of 10 vehicles per hour traveling at 10 mph and 30 vehicles per hour traveling at 25 mph.

##### Day3 (8/20/03) objective was to:

Repeat the traffic scenarios used during the Initial Study of 10 vehicles per hour traveling at 10 mph and 30 vehicles per hour traveling at 25 mph.

#### **3.3.1 Post Resurfacing Sample Station Locations**

In order to directly compare sampling results collected during pre and post resurfacing activities the air samplers were positioned at the same locations as during the Initial Study, with two exceptions. A 19<sup>th</sup> air station was established 300 ft perpendicular to the roadway, west of the road. A similar station was not established on the east side due to physical barriers. In addition a 20<sup>th</sup> air station was established at a residence on Bayleaf Drive approximately 1½ miles from the site to serve as a ambient background sample.

The location on Bayleaf Drive was selected by DTSC because it did not have significant traffic and because it was located on top of a hill in close proximity to

Golden Sierra High School. The Bayleaf Drive location was thought to represent regional background concentrations for asbestos in the Garden Valley area. The Bayleaf Drive location was sampled only on Day 1. Each sampler was outfitted with a mixed cellulose ester (MCE) membrane filter (0.45 µm pore size, 25mm diameter). Additionally, MET stations monitored wind speed, wind direction, and air temperature were installed on site.

### 3.3.2 Sampling Activities

Below is a summary of the Post Resurfacing sampling activities that occurred between August 18 and August 20, 2003. Individual sample data is presented in Appendix B. The same DTSC test vehicles that were used during the Initial Study were also used for post resurfacing traffic simulations.

#### ***Day 1 (08/18/03) – Setup & Monitor Actual Conditions***

The stationary air samplers were positioned at the same locations as on Days 2 & 3 of the Initial Study. Additional samplers were also positioned at distances greater than those sampled during the Initial Study. MET stations were positioned at 50 ft on each side of the road. A distance of 50 ft was chosen based on a review of the Initial Study MET data that indicated there was no discernable difference between the MET data collected as close as 25 ft and as distant as 90 ft.

Air samples were collected at the site over two separate two hour sampling periods. No traffic simulation activities were performed during the sampling (see characteristics below). A background sample was also collected at a residence on Bayleaf Drive approximately 1½ miles from the site. Personal samples were collected from two field personnel. DTSC also performed particulate monitoring concurrent with the personal samples. The particulate monitor (Data RAM) was located approximately 5 ft east of the roadway (positioned on adjacent fence post).

#### Characteristics

Vehicle Frequency	N/A
Vehicle Speed	N/A
Distances Sampled	5, 10, 30, 50, 80, 100, 130, 160, 190 (both sides) 300 feet (west side only)
Target Flow Rates	varied from 7.2 to 12.1 L/min (depending on distance)
MET Stations	1 MET Sta. on each side at 50 feet
Duration	2 hrs

An AALBORG mass flow meter was used to measure the start flow rates. However, when measuring the stop flow rates with both a Dry Cal primary flow meter and the AALBORG mass flow meter inconsistencies in the measurements were observed. It was determined that the Dry Cal values

were more reliable. The Start Flow rate for the collected samples was estimated based on the average difference between Dry Cal (primary standard flowmeter) start and final flow measurements observed on 8/19/03 and 8/20/03 (excluding outliers). The Dry Cal flow meters were used for all other sampling.

### ***Day 2 (08/19/03)***

Two runs were conducted, Run 02-01 & Run 02-02 (see characteristics below). Three personal samples were collected, two field personnel and one on the truck driver. DTSC also performed particulate monitoring concurrent with the personal samples. The Data RAM was located approximately 5 ft east of the roadway (positioned on adjacent fence post).

#### Run 02-01 Characteristics

Vehicle Frequency	10 vph
Vehicle Speed	10 mph
Distances Sampled	5, 10, 30, 50, 80, 100, 130, 160, 190 (both sides) 300 feet (west side only)
Target Flow Rates	varied from 7.5 to 11.8 L/min (depending on distance)
MET Stations	1 MET Sta. on each side at 50 feet
Duration	2 hrs

#### Run 02-02 Characteristics

Vehicle Frequency	30 vph
Vehicle Speed	25 mph
Distances Sampled	5, 10, 30, 50, 80, 100, 130, 160, 190 (both sides) 300 feet (west side only)
Target Flow Rates	varied from 3.8 to 10 L/min (depending on distance)
MET Stations	1 MET Sta. on each side at 50 feet
Duration	2 hrs

### ***Day 3 (08/20/03)***

Two runs were conducted, Run 03-01 & Run 03-02 (see characteristics below). Three personal samples were collected, two field personnel and one on the truck driver.

#### Run 03-01 Characteristics

Vehicle Frequency	10 vph
Vehicle Speed	10 mph
Distances Sampled	5, 10, 30, 50, 80, 100, 130, 160, 190 (both sides) 300 feet (west side only)
Target Flow Rates	varied from 7.4 to 10.3 L/min (depending on



	distance)
MET Stations	1 MET Sta. on each side at 50
Duration	2 hrs

Run 03-02 Characteristics

Vehicle Frequency	30 vph
Vehicle Speed	25 mph
Distances Sampled	5, 10, 30, 50, 80, 100, 130, 160, 190 (both sides) 300 feet (west side only)
Target Flow Rates	varied from 4.0 to 10.6 L/min (depending on distance)
MET Stations	1 MET Sta. on each side at 50 feet
Duration	2 hrs

## SECTION 4. SAMPLING RESULTS

This section describes the sampling results from both the 2002 (Initial Study) and 2003 (Post Resurfacing Study) sampling events. The results provided in the following sections relate to four types of sampling that were performed.

- Meteorological
- Particulate
- Stationary Air
- Personal Air

### 4.1 Meteorological Data

Meteorological measurement and data recording equipment were used to record wind speed, direction, temperature and humidity at the test site during sampling. Meteorological information was used to evaluate the effect of wind speed and direction on asbestos concentrations measured in the air samplers

MET data was sampled at a 1Hz sample rate and recorded in ASCII text file format. As described below, different MET station sensors were used for the Initial Study and the Post Resurfacing Study. The sensor used for the Post Resurfacing Study is more sensitive at lower wind speeds. Both sensors collect the same types of meteorological information.

Specific sampling details are described below.

#### ***Initial Study***

The Initial Study used MET stations called Transportable Automated MET Station (TAMS). The TAMS units are cup anemometer based weather stations that provide the following information:

##### **TAMS sensors include:**

- wind speed and gust
- wind direction and variable wind direction
- relative humidity
- temperature
- dew point
- atmospheric pressure
- density altitude
- barometric pressure

A picture of a typical TAMS unit (with tripod) is shown in Appendix G of this report. The TAMS units have cup anemometers and can operate in the wind speed range of 2-70 knots (2.3-80.6 mph). Since they are cup anemometers

they generally become less accurate at low wind speeds. Data was recorded at a 1Hz sample rate and stored to a personal computer for post sampling analysis. The units were mounted on tripods at a height of approximately 5 ft. above ground surface.

The TAMS units were placed at different locations during Day 0 and Day 1 of the Initial Study. One or two units were placed on either side of Slodusty Road at various distances (either 25 ft, 30 ft, 95 ft, or 100 ft). On Day 2 a unit was also located approximately 100 yards from the sampling area, adjacent to the video camera. Specific locations for each run, of each day are identified within section 3.2.2.

### ***Post Resurfacing Study***

The Post Resurfacing study used Gill WindObserver II Ultra Sonic anemometers which are much more accurate at low wind speeds because they operate on the principle of ultra sonic sound wave travel time versus changes in the medium of transport, rather than the mechanical cup anemometer approach. The ultra sonic anemometers have a wind speed resolution 0.01 m/s (0.02 mph) and range of 0-145 mph. During all sampling activities the Ultra sonic anemometers were placed in a symmetric fashion, fifty feet from the road edge on the east and west side of Slodusty Road. The units were mounted on tripods at a height of approximately 5 ft. Data from the ultra sonic anemometers was recorded using two Campbell Scientific dataloggers at a 1 Hz sample rate. A picture of a typical Gill WindObserver II Ultra Sonic unit (with tripod) is shown in Appendix G of this report.

Ambient temperature and humidity were measured with DTSC instrumentation. The data was transferred to personal computers for post processing after measurements were completed. Temperature and humidity data were collected using Davis Instruments Weather Link II Air Monitoring Stations. The Weatherlink system has the ability to log data to be able to transfer it to a laptop portable computer.

One Gill WindObserver II Ultra Sonic unit was located on each side of the road at a distance of 50 ft during all Post Resurfacing sampling activities. Also one Davis Instruments Weather Link II Air Monitoring station was located on the west side of the road at 50 ft.

### **4.1.2 Initial Study & Post Resurfacing Meteorological Results**

Wind roses and data summary tables were generated from the data collected during both studies. Winds were predominantly out of the west and northwest direction and were generally less than 8 mph for both the Initial Study and Post Resurfacing Study. As indicated by Tables 4-1 and 4-2 below, the wind conditions during both studies were on average very consistent between the two studies and with little fluctuation during the day. The relative humidity readings

during the Initial Study were higher and the temperatures slightly lower than during the Post Resurfacing sampling.

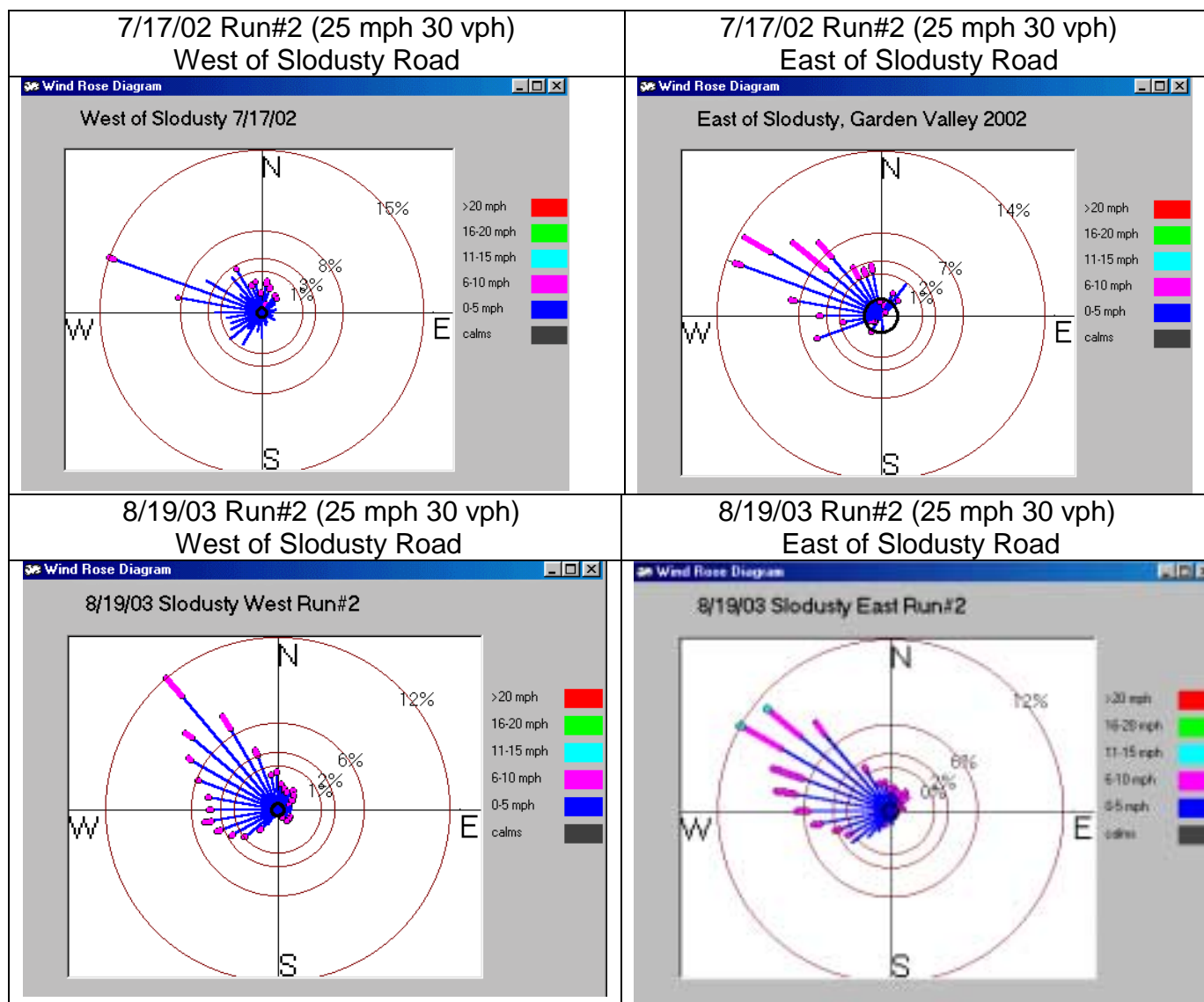
**Table 4-1 Meteorological Conditions Comparison (25 mph / 30 vph)**

Study	Initial Study		Post Resurfacing	
Time	Afternoon		Afternoon	
Met. Station Location	West	East	West	East
Wind speed (mph)	2.8	3.4	2.9	3.5
Wind direction (degrees)	324	315	308	298
Relative Humidity (%)	27.6		23.3	
Temperature (Fahrenheit)	88.6		89.8	

**Table 4-2 Meteorological Conditions Comparison (10 mph / 10 vph)**

Study	Initial Study		Post Resurfacing	
Time	Morning		Morning	
Met. Station Location	West	East	West	East
Wind speed (mph)	2.6	2.6	2.5	3.0
Wind direction (degrees)	80	268	352	320
Relative Humidity (%)	49.5		26.0	
Temperature (Fahrenheit)	82.4		90.0	

Example wind roses are shown in the figures below comparing wind conditions during a 25 mph (30 vph) traffic scenario conducted during the Initial Study and Post Resurfacing sampling. The length of each spoke around the circle shows the percent of time the wind blew from that direction. The spokes are color coded indicating the magnitude of the wind speed for each direction. Additional summary tables and wind roses for individual runs from both the Initial Study and Post Resurfacing sampling events are provided in Appendix F.



## 4.2 Air Particulate Data

Air particulate data was collected using an MIE, model pDR-1000AN (personal Data RAM), aerosol monitor. This instrument provides direct instantaneous measurement readings of airborne dust and mist in concentrations between 0.001 mg/m<sup>3</sup> and 400 mg/m<sup>3</sup>. It monitors passively using an optical feedback-stabilized sensing system, and provides data logging capability with storage for up to 13,000 data points.

Table 4-3 below summarize the particulate measurements collected by DTSC during the Initial Study. All measurements were taken on the west side of the road at varying distances during both of the Day 1 traffic scenarios. All Post Resurfacing measurements were collected on the east side of the road during

Days 1 and 2 (see Table 4-4 below). Due to a significant reduction in visible dust emissions all Post Resurfacing particulate measurements were collected approximately 5 ft east of the roadway (positioned on adjacent fence post), which was considered to be representative of peak particulate emissions.

**Table 4-3 Initial Study Particulate Data**

	<b>RUN 01-01</b> <b>(10 vph / 25 mph)</b>	<b>RUN 01-02</b> <b>(30 vph / 25 mph)</b>
<b>Distance</b> <b>(ft)</b>	<b>Dust Load</b> <b>(mg / m<sup>3</sup>)</b>	<b>Dust Load</b> <b>(mg / m<sup>3</sup>)</b>
5, W	1.01, 2.54	1.60
<b>10, W</b>	3.47, 3.48	0.70
30, W	1.00, 0.20	0.03
50, W	1.10, 0.70	0.02
80, W	0.22	0.03
100, W	0.02	0.02

Note: the particulate measurements were spot measurements taken on July 16, 2002 by DTSC.

**Table 4-4 Post Resurfacing Particulate Data**

	<b>DAY 1 (8/18/03)</b> <b>No traffic simulation</b> <b>performed</b> <b>(actual field cond.)</b>	<b>DAY 2 (8/19/03)</b> <b>During Simulated</b> <b>traffic</b>
<b>Distance (ft)</b>	<b>Dust Load (mg / m<sup>3</sup>)</b>	<b>Dust Load (mg / m<sup>3</sup>)</b>
5, E (on fence post)	Avg. Conc.= 0.015 TWA = 0.019	Avg. Conc.= 0.005 TWA = 0.020

Note: the particulate measurements were collected concurrent with the personal samples. The Data RAM was located approximately 5 ft east of the roadway (positioned on adjacent fence post).

### 4.3 Stationary Air Sample Analytical Results

This section describes the stationary air sampling results from both the Initial Study and Post Resurfacing Study. A data quality assessment of the analytical results are provided in section 5.2. Copies of the laboratory analytical methods are provided in Appendices J, K, and L of this report (Appendix J – TEM AHERA, Appendix K – TEM ISO, Appendix L = PCM).

All analysis were performed by laboratories that:

- Are accredited under the Laboratory Accreditation Program as sponsored by the American Industrial Hygiene Association;
- Actively participate in the National Institute for Occupational Safety and Health (NIOSH) “Proficiency Analytical Testing Program for Laboratory Quality Control” for asbestos;
- Are fully accredited for PCM and TEM analysis under the National Voluntary Laboratory Accreditation Program as sponsored by the National Institute of Standards and Technology (NIST).

#### ***Initial Study***

During the course of the Initial Study 141 air samples were collected including field, blank and personal samples. The original objective of the Initial Study was to refine sampling methodologies and collect initial airborne asbestos data. Because sample objectives were met on Day 0 and Day 1, Initial Study traffic runs were conducted on Day 2 and Day 3.

In order to refine sampling methodologies it was necessary to determine:

- (1) the maximum pump flow rate for each traffic scenario, at various distances, that will not result in overloading the samples with particulate (so that when samples are analyzed they won't require an indirect prep); and
- (2) the maximum distance asbestos fibers are being transported for each traffic scenario, in order to ensure sample stations are located appropriately.

During the Initial Study a total of 128 stationary samples were collected. At the conclusion of each day a select set of samples were chosen for immediate, limited analysis by the laboratory. These limited analyses were performed concurrent with field activities. The samples were selected in a phased approach based on flow rate and distance for the purpose determining which filters were overloaded and / or for the mere presence of asbestos (not concentration data). The results of these limited analyses were used to refine each day's sampling methodology in regards to maximizing the flow rate and determining optimum locations of the stationary samplers.

Initially, a second small set of nine samples were selected to undergo TEM analysis for the purpose of obtaining an initial understanding of asbestos concentrations in the vicinity of the site during each traffic scenario. Eight samples were selected from Day 2 (methodologies were refined during Days 0 and 1). All eight of the samples were collected during Run 02-02 (25 mph / 30 vph scenario) from both sides of the road. The eight were selected to be representative of the varying incremental distances sampled during the study. The eight were collected at distances of either 10, 30, 50, 80, 100, 130, or 160 feet. A ninth sample from Run 03-01 of Day 3 (10 mph /10 vph scenario) was selected based on its close proximity to the road during traffic conditions that were considered to be representative of actual speeds and the peak frequency of Slodusty Road. Based on levels from these initial TEM results it was determined to analyze all of the samples collected during Runs 02-02 and 03-01 (42 samples), which were the only runs for which a primary standard flowmeter was used to verify the actual start and stop flow rates. Because of equipment failure, a digital flowmeter was not used to calibrate or set flow rates until 07/17/02 and 07/18/02 (Days 2 and 3 of study), therefore the volume for these samples could only be estimated and consequently only a limited analysis was performed on these samples. The results for the stationary samples collected during Runs 02-02 and 03-01 (42 are summarized in section 4.3.1 below and are provided in Appendix C.

Copies of the laboratory analytical methods are provided in Appendices J, K, and L of this report (Appendix J – TEM AHERA, Appendix K – TEM ISO, Appendix L = PCM).

### ***Post Resurfacing***

During the course of the post resurfacing sampling 108 air samples were collected including all field, blank and personal samples. The objective of the Post Resurfacing sampling was to assess the effectiveness of the resurfacing activities in reducing airborne asbestos concentrations, near Slodusty Road.

Stationary air samples were analyzed via TEM in order to be directly compared to the results from Initial Study Runs 02-02 and 03-01. Samples were selected for analysis in a phased approach based on distance and traffic scenario. Ultimately, from the total 96 stationary samples collected 35 samples were selected for TEM analysis and determined to be representative of all sampling activities. Of the 35 samples analyzed, eight were selected from Day 1 and were considered to be representative of the range of distances sampled during the days “No Simulation Run” and included the background sample from Balyleaf Drive. Fifteen additional samples were collected during Day 2 and twelve from Day 3. The twenty seven samples from Days 2 and 3 were selected to be representative of the range of distances sampled during both traffic scenarios over the period of both days (13 from the 10 mph and 14 from 25 mph scenario).



The results for the stationary samples are summarized in section 4.3.1 below and are provided in Appendix C.

All post resurfacing personal samples were analyzed via TEM, see Section 4.4 and Appendix C.

#### **4.3.1 TEM Results**

Initially, TEM analysis of the Initial Study samples was limited to just a few samples that were analyzed following the ISO 10312 TEM (ISO) method. The ISO 10312 method was selected in order to be able to gain a better understanding of the fiber distribution within the samples. Unlike other TEM analysis methods the counting rules under the ISO 10312 method requires the analyst to record the individual countable fibers that are part of a complex structure (such as a cluster or matrix of fibers). For example, under ISO in the case of a complex structure such as a “cluster” that is comprised of 4 individual countable fibers the analyst would record and count all four fibers individually, while under another TEM method such as TEM-AHERA (AHERA) the same cluster of fibers would be recorded and counted as one structure (assuming it was countable under the rules of the method). Initial ISO results indicated a fiber distribution of numerous, primarily complex chrysotile asbestos structures less than 5um in length with a few free scattered chrysotile fibers. Also, many of the structures were observed to have splayed ends (“weathered”) and some of them also were observed to have a tubular appearance. Based on these observations the first lab analyzing the samples classified many of the chrysotile structures as “scrolled lizardite”. However, based on further evaluation and discussion among several experienced microscopists it was determined that these structures should be classified as chrysotile structures. Refer to section 5.2 below for additional information on the “scrolled lizardite” evaluation.

Based on the high asbestos levels from these first ISO results it was determined to analyze all of the samples collected during Runs 02-02 and 03-01 (33 stationary samples). These were the only runs where a primary standard flowmeter was used to verify the actual start and stop flow rates. The 33 samples from these runs were all analyzed via a modified AHERA method. AHERA was chosen over the ISO method because we already had acquired an understanding of the fiber distribution based on the prior ISO analysis. The AHERA method requires less time for analyzing each filter. The samples from the Initial Study were heavily loaded with asbestos structures and debris. As a result of these heavy loadings of asbestos the AHERA stopping rules were modified to allow counting less than 4 grid openings. Although in all cases the analyst examined two sample grids to assure even loading between grids. In addition the grid rejection criteria was increased to 25 % and non-asbestos structures were not counted or recorded. The project modifications to the AHERA method are further described in Appendix H of this document. No negative implications to these modifications are anticipated. Positive implications

are documentation of procedures and increased efficiency in completion of analysis.

The project labs were required to record all TEM analytical results into a project specific electronic data deliverable (EDD). The EDD is a modified version of an EDD that was developed by EPA Region 8 as part of asbestos investigative and Superfund activities in Libby, MT. An example of the project TEM EDD is provided in Appendix I of this report. The EDD requires the lab to record sample preparation information, and describe the size (length, thickness) and type (chrysotile, amphibole, non-asbestos) of each structure that is characterized in accord with the applicable counting rules, as well as any relevant analytical comments. The purpose of the EDD is to electronically capture counting information in order to accommodate potential future changes in regulatory and health classification requirements, enabling samples to be re-evaluated in the future without needing to reanalyze the actual samples. Also, by recording each type of structure encountered (chrysotile, fiber, matrix, cluster, etc.) will assist with making additional determinations regarding the asbestos distribution. The asbestos concentrations were based solely on the rules of the applicable analytical method. However, the laboratories were required to include information on all structures observed.

All of the Initial Study and Post Resurfacing TEM analyses were performed following a direct sample preparation technique.

Tables 4-5 & 4-6 below summarize the average AHERA results for both the 33 Initial Study (Runs 02-02 and 03-01) and 35 Post Resurfacing stationary samples analyzed, including information regarding the distribution of the asbestos structures. The values presented are the average of all samples taken for each distance, including for both sides of the road, during each traffic scenario. Consistent with the early Initial Study ISO results, the average AHERA results indicate approximately 90 % of the structures observed during both sampling events are less than (<) 5.0 um in length and 10 % are greater than (>) or equal (=) to 5.0 um in length. Tables 4-7 & 4-8 and Figures 4-1 to 4-4 provide a summary comparison of the average results. They compare the average Initial Study 10 mph (10 vph) and 25 mph (30 vph) traffic scenarios to those collected during the Post Resurfacing sampling, including the no simulation scenario and the background sample. Additional plots and all of the individual sample results are provided within Appendix D of this report.

Plots of the average Initial Study results indicate that during both the 10 mph (10 vph) and 25 mph (30 vph) traffic scenarios the asbestos concentrations are significantly higher 5 ft from the roadway, and the concentration at 10 ft was typically 64 % to 70 % lower. As the distance increases from 10 ft away the concentration reduction is much more gradual, dropping by approximately 40 % every 20 ft. In addition to distance, traffic conditions appear to significantly affect the levels of asbestos that were emitted from the road. The concentrations for

the Initial Study measured at each distance are approximately an order of magnitude higher for the 25 mph (30 vph) scenario when compared to the 10 mph (10 vph) scenario. For example for the 25 mph (30 vph) scenario, at 5 ft the average asbestos concentration was 6.300 s/cc compared to 0.7550 s/cc. Also, at a distance of 130 ft from the road the average concentration was 0.0477 s/cc for the 10 mph scenario, while at the same distance the average concentration was still 0.5050 s/cc for the 25 mph (30 vph) scenario. In fact at 190 ft (furthest sampling location collected during the Initial Study) the average concentration was still 0.1870 s/cc for the 25 mph (30 vph), significantly above the 0.0047 s/cc background sample. The difference is less pronounced when comparing the 130 ft location from the 10 mph (10 vph) scenario (furthest location sampled) to the background sample. The average concentration for the 130 ft 10 mph (10 vph) location was 0.0477 s/cc, which is still above the 0.0047 s/cc background sample. When comparing the background sample to other average results it is important to note that except for at immediately adjacent to the road (at 5 ft) the average concentrations associated with the no simulation scenario tend to be approximately equal to the background sample and the results from Bayleaf Drive during the Post Resurfacing Study (see Table 4-9 & Figure 4-5).

Plots of the average Post Resurfacing results indicate an average 94 % reduction in asbestos concentrations during the 10 mph (10 vph) scenario and an average 98 % reduction during the 25 mph (30 vph) traffic scenario compared to the Initial Study average results (see Tables 4-7 & 4-8). The average Post Resurfacing results for the 10 mph (10 vph) scenario are approximately an order of magnitude lower than those of the Initial Study average results. While the average Post Resurfacing results for the 25 mph (30 vph) scenario are approximately two orders of magnitude lower than those of the Initial Study average results. Unlike during the Initial Study, there is no apparent trend to the asbestos concentrations in regards to distance for the 10 mph (10 vph) scenario. This is also true for the 25 mph (30 vph) scenario, with the exception being at 5 ft, which had an elevated average result compared to sampling stations further from the roadway. Also, there is no apparent trend to the average Post Resurfacing results in regards to traffic conditions (see Figure 4-5). Although closer to the road (5 ft & 30 ft) the average results of the 10 mph (10 vph) scenario are lower than the 25 mph (30 vph) scenario, they were higher further from the road (80 ft & 300 ft). The only Post Resurfacing sampling trend associated with traffic conditions is that the average concentrations associated with simulated traffic tend to be slightly higher than the no simulation scenario and the background sample.

**Table 4-5 Initial Study Average Results (Stationary Samples)**

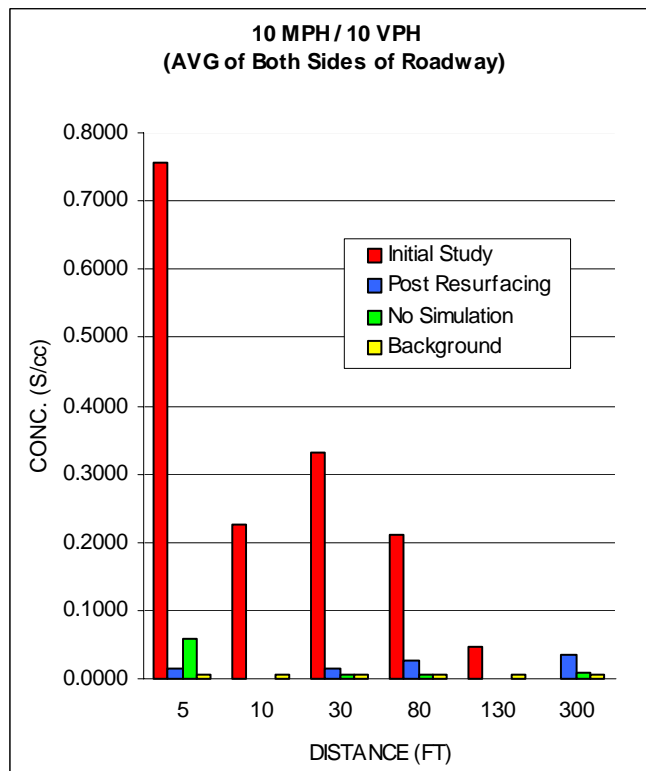
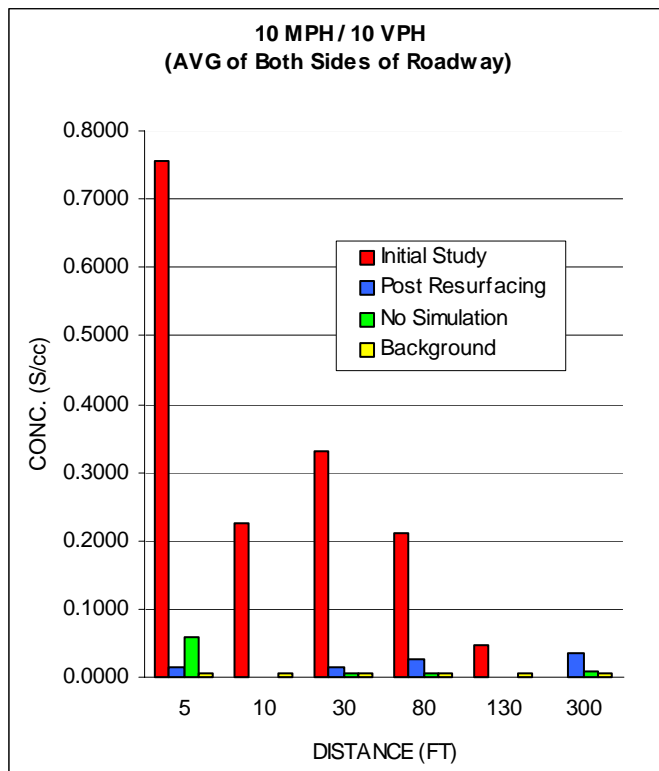
LOCATION / SCENARIO		AHERA ASBESTOS RESULTS						
		# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (s/cc)	Analytical Sensitivity (s/cc)
Scenario	Distance (ft)							
(10 mph / 10 vph)	5	8	49	2	51	3	0.7550	0.0145
(25 mph / 30 vph)	5	7	60	6	66	1	6.3000	0.1015
(10 mph / 10 vph)	10	4	37	8	45	6	0.2250	0.0051
(25 mph / 30 vph)	10	17	41	5	46	1	2.2750	0.0478
(10 mph / 10 vph)	30	12	46	7	53	4	0.3300	0.0063
(25 mph / 30 vph)	30	13	39	5	44	1	1.5350	0.0350
(25 mph / 30 vph)	50	6	63	5	68	3	0.9100	0.0108
(10 mph / 10 vph)	80	5	25	2	27	4	0.2115	0.0071
(25 mph / 30 vph)	80	12	42	5	46	4	0.7100	0.0162
(25 mph / 30 vph)	100	6	21	3	23	4	0.4265	0.0169
(10 mph / 10 vph)	130	5	17	1	18	8	0.0477	0.0036
(25 mph / 30 vph)	130	5	25	1	26	3	0.5050	0.0154
(25 mph / 30 vph)	160	12	35	2	37	3	0.3500	0.0089
(25 mph / 30 vph)	190	8	28	7	35	5	0.1870	0.0052
		90 %		10 %				

**Table 4-6 Post Resurfacing Average Results (Stationary Samples)**

LOCATION / SCENARIO		AHERA ASBESTOS RESULTS						
		# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5 um)	# AHERA Structures (>= 5 um)	# AHERA Structures (total)	#GOs	[Asbestos] (s/cc)	Analytical Sensitivity (s/cc)
Scenario	Distance (ft)							
Background sample taken at Residence (350 Bayleaf Drive, owner = Vance Fellows)	Background	0	2	0	2	10	0.0047	0.0023
No Simulation	5	1	13	1	14	9	0.0585	0.0044
(10 mph / 10 vph)	5	0	3	1	4	9	0.0155	0.0043
(25 mph / 30 vph)	5	0	8	1	9	10	0.0654	0.0071
No Simulation	30	0	1	1	2	8	0.0069	0.0047
(10 mph / 10 vph)	30	0	2	1	3	10	<0.0139	0.0040
(25 mph / 30 vph)	30	0	5	0	5	9	0.0218	0.0041
No Simulation	80	0	1	0	1	7	0.0046	0.0045
(10 mph / 10 vph)	80	0	6	0	6	9	0.0250	0.0038
(25 mph / 30 vph)	80	0	3	0	3	9	0.0076	0.0038
(25 mph / 30 vph)	100	0	3	0	3	7	0.0130	0.0044
(25 mph / 30 vph)	160	0	0	0	0	7	<0.0046	0.0046
(25 mph / 30 vph)	190	0	2	0	2	7	0.0090	0.0045
No Simulation	300	0	1	2	3	10	0.0091	0.0030
(10 mph / 10 vph)	300	0	7	0	7	6	0.0360	0.0052
(25 mph / 30 vph)	300	0	0	0	0	7	<0.0043	0.0043
		90 %		9 %				

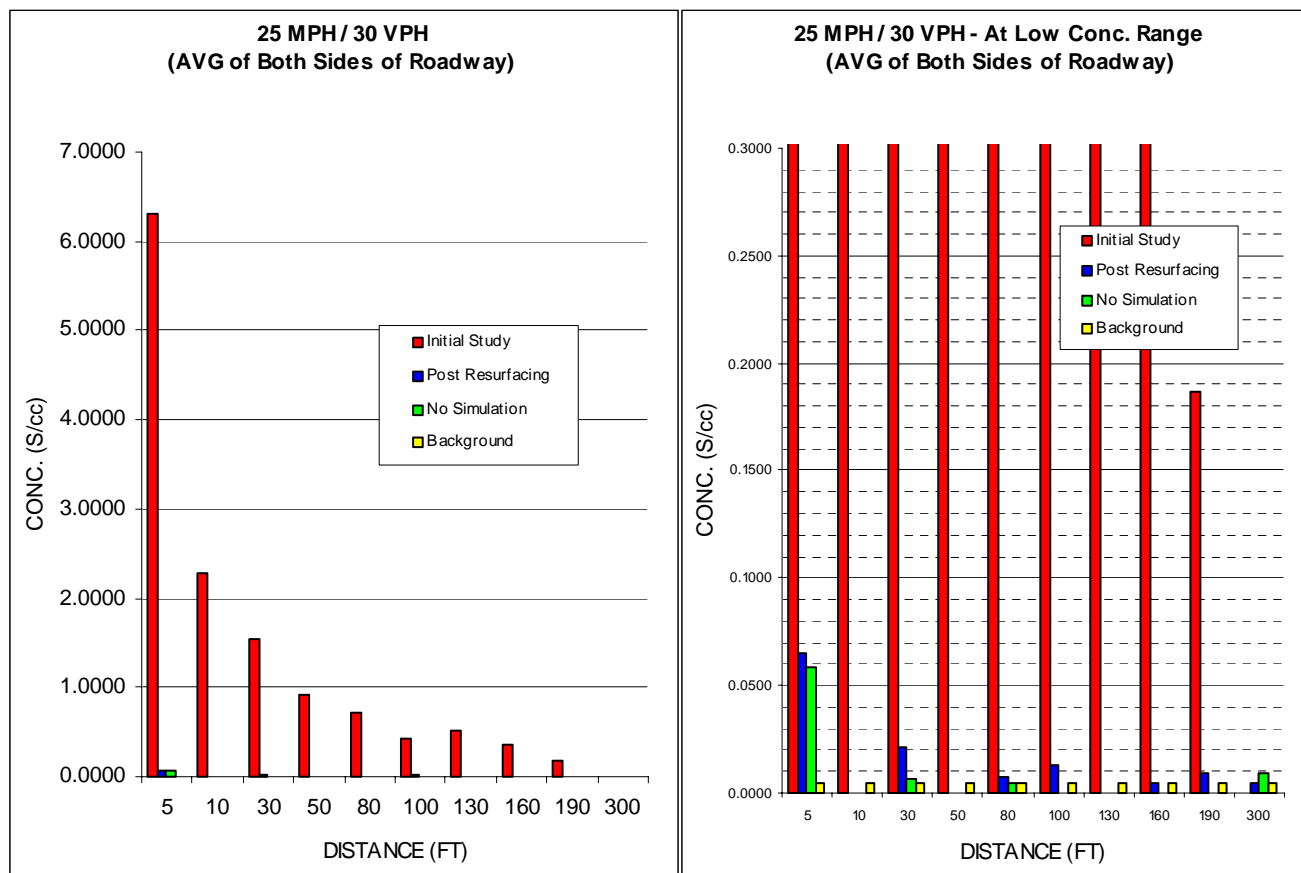
**Table 4-7 Comparison of Average Results (10 mph / 10 vph)**

Distance	Initial (s/cc)	Post (s/cc)	% Diff (Pre & Post)	No Simulation (s/cc)	Background (s/cc)
5	0.7550	0.0155	98 %	0.0585	0.0047
10	0.2250	NA		NA	0.0047
30	0.3300	< 0.0139	96 %	0.0069	0.0047
50	nm	NA		NA	0.0047
80	0.2115	0.0250	88 %	0.0046	0.0047
130	0.0477	NA		NA	0.0047
160	nm	NA		NA	0.0047
190	nm	NA		NA	0.0047
300	nm	0.0360		0.0091	0.0047
<b>AVG</b>			<b>94 %</b>	NA	Not Analyzed
				nm	Not Measured

**Figures 4-1 & 4-2 Comparison Average Results (10 mph / 10 vph)**

**Table 4-8 Comparison of Average Results (25 mph / 30 vph)**

Distance	Initial (s/cc)	Post (s/cc)	% Diff (Pre & Post)	No Simulation (s/cc)	Background (s/cc)
5	6.3000	0.0654	99 %	0.0585	0.0047
10	2.2750	NA		NA	0.0047
30	1.5350	0.0218	99 %	0.0069	0.0047
50	0.9100	NA		NA	0.0047
80	0.7100	0.0076	99 %	0.0046	0.0047
100	0.4265	0.0130	97 %	NA	0.0047
130	0.5050	NA		NA	0.0047
160	0.3500	< 0.0046	99 %	NA	0.0047
190	0.1870	0.0090	95 %	NA	0.0047
300	nm	< 0.0043		0.0091	0.0047
<b>AVG</b>			<b>98 %</b>	NA	Not Analyzed
				nm	Not Measured

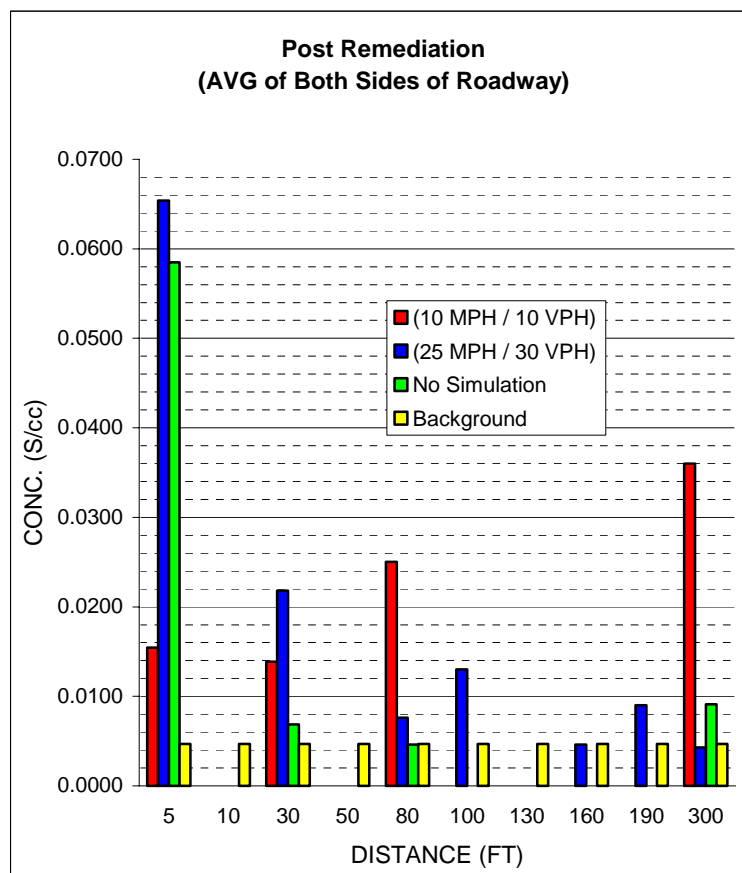
**Figures 4-3 & 4-4 Comparison Average Results (25 mph / 30 vph)**

**Table 4-9 Comparison of Average Results (Post Resurfacing)**

Distance	Post 10 mph / 10vph (s/cc)	Post 25 mph / 30 vph (s/cc)	No Simulation (s/cc)	Background (s/cc)
5	0.0155	0.0654	0.0585	0.0047
10	NA	NA	NA	0.0047
30	< 0.0139	0.0218	0.0069	0.0047
50	NA	NA	NA	0.0047
80	0.0250	0.0076	0.0046	0.0047
100	NA	0.0130	NA	0.0047
130	NA	NA	NA	0.0047
160	NA	0.0046	NA	0.0047
190	NA	0.0090	NA	0.0047
300	0.0360	0.0043	0.0091	0.0047

NA

Not Analyzed

**Figure 4-5 Comparison Average Results (Post Resurfacing)**

#### 4.4 Personal Air Sample Results

This section describes the personal air sampling results from both the Initial and Post Resurfacing Studies. A data quality assessment of the analytical results is provided in section 5.2. Copies of the applicable laboratory analytical methods are provided in Appendices J and L of this report (Appendix J – TEM AHERA, Appendix L = PCM).

All analyses were performed by laboratories that:

- are accredited under the Laboratory Accreditation Program as sponsored by the American Industrial Hygiene Association;
- actively participate in the National Institute for Occupational Safety and Health (NIOSH) “Proficiency Analytical Testing Program for Laboratory Quality Control” for asbestos;
- are fully accredited for PCM and TEM analysis under the National Voluntary Laboratory Accreditation Program as sponsored by the National Institute of Standards and Technology (NIST).

Personal samples were collected to monitor worker exposure. One representative sample was taken for each task. These tasks were categorized as “Field Personnel”, “Traffic Controller”, and “Driver”. The “Field Personnel” category included all sampling and support type staff. The duties of field personnel included all aspects of sample collection such as set-up, operation, monitoring and tear down of all sampling equipment. The “Driver” category included the drivers of the test vehicles (sedan and pick-up truck). The duties of the “Traffic Controller” category were to coordinate traffic simulation activities with field personnel and drivers, as well as local traffic. A traffic controller was positioned at each end of the test stretch of road. Details of sampling and traffic simulation activities are provided in Appendix A of this report. Representative samples were collected from both Volpe and DTSC personnel for all days of each sampling event. All personal samples were collected using SKC Universal Sampling Pumps (Model #224-44XR) calibrated with a SKC DryCal Primary Flow Meter (Model Cat.#717-05).

A total of 19 personal samples were collected during both events (11 Initial and 8 Post). All personal samples were analyzed via both Phase Contrast Microscopy (PCM) and Transmission Electron Microscopy (TEM-AHERA analysis). A limitation of PCM technology is that it is unable to distinguish between asbestos fibers and non-asbestos fibers. Due to this known limitation the personal samples were also analyzed via TEM-AHERA, in order to supplement the PCM results (required by OSHA) for making personnel safety decisions in the field.



#### 4.4.1 PCM Results

The Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) is based on PCM analytical results. The OSHA 8 hr time-weighted-average (TWA) PEL is 0.1 f/cc. As indicated by Table 4-10 and Table 4-11 below, all of the Initial Study and Post Resurfacing PCM TWA concentrations are significantly below the OSHA PEL. Based on these PCM results it was not necessary to upgrade or modify safety procedures during either event.

**Table 4-10 Summary of Initial Study Personal Sample Results**

Index IDs	Date collected	Notes	Estimated Exposure Period (hr)	TEM TWA (s/cc)	LAB	PCM TWA (f/cc)	LAB
P0-00016	07/15/02	Sedan Driver (DTSC personnel)	3.0	0.0056	EMSL++	could not analyze, filter type is PC EMSL	
P0-00017	07/15/02	Traffic Controller	3.0	0.2325	EMSL++	could not analyze, filter type is PC EMSL	
P1-00060	07/16/02	In pick-up truck	6.5	0.2844	EMSL++	0.0130	EMSL
				0.2681	RESI		
P1-00061	07/16/02	Field Personnel	6.5	0.2275	EMSL++	0.0049	EMSL
P1-00062	07/16/02	Traffic Controller	6.5	0.0114	EMSL++	< 0.0033	EMSL
P2-00002	07/17/02	Field Personnel	6.5	0.2763	EMSL++	0.0049	EMSL
				0.3819	RESI		
P2-00003	07/17/02	Traffic Controller	6.5	0.0496	EMSL++	< 0.0033	EMSL
				0.0496	RESI		
P2-00004	07/17/02	Field Personnel	6.5	0.3331	EMSL++	< 0.0033	EMSL
				0.4469	RESI		
				0.2113	RESI QC-RPs		
P3-00002	07/18/02	Field Personnel	3.0	0.1538	RESI	< 0.0034	EMSL
				0.0788	EMSL++		
P3-00003	07/18/02	Field Personnel	3.0	1.6875	RESI	0.0045	EMSL
				0.0525	EMSL++		
P3-00004	07/18/02	Sedan Driver	3.0	0.0413	RESI	< 0.0034	EMSL
				0.0563	EMSL++		
11	<b>Total Personal Samples</b>	<b>AVERAGE</b>		<b>0.2552</b>		<b>&lt; 0.0049</b>	

**NOTE:** "++" EMSL concentrations excluded structures that appeared as "scrolled lizardite", concentrations were determined by EMSL prior to reaching a consensus among laboratories that structures that appear as "scrolled lizardite"

**Table 4-11 Summary of Post Resurfacing Personal Sample Results**

Index ID	Date collected	Notes	Estimated Exposure Period (hr)	TEM TWA (s/cc)	PCM TWA (f/cc)	LAB
F1-00024	08/18/03	Field Personnel - No Simulation Run	4.0	0.0205	0.0060	RESI
F1-00025	08/18/03	Field Personnel - No Simulation Run	3.0	0.0450	0.0045	RESI
F2-00042	08/19/03	Field Personnel battery failure at 31min	6.0	0.3825	0.0593	RESI
F2-00043	08/19/03	Sedan Driver battery failure at 69min	5.0	0.0156	<0.0125	RESI
F2-00044	08/19/03	Field Personnel	6.0	<0.0044	0.0135	RESI
F3-00001	08/20/03	Field Personnel	6.0	0.0630	0.0158	RESI
F3-00002	08/20/03	Field Personnel	6.0	0.0255	0.0120	RESI
F3-00003	08/20/03	Truck Driver	6.0	0.0293	0.0090	RESI

<b>8</b>	<b>Total Personal Samples</b>	<b>AVERAGE</b>	<b>&lt; 0.0313**</b>	<b>&lt; 0.0101*</b>
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**NOTE :** \*\* The average concentration excludes samples F2-00042 and F2-00043, due to equipment failure

#### 4.4.2 TEM Results

Although OSHA regulations require and are based on PCM analysis, the capabilities of PCM are limited. PCM technology is unable to distinguish between asbestos fibers and non-asbestos fibers. Due to this known limitation project personal samples were also analyzed via TEM-AHERA. The TEM-AHERA results were to be used to supplement the information provided by the PCM results. The TEM AHERA results were not used to determine adherence to OSHA regulations, and OSHA related personnel safety decisions in the field. Because the OSHA levels are based on occupational exposures assessed using the PCM technology, it is not appropriate to use TEM results for determining whether or not OSHA requirements have been met.

During the Initial Study, as a result of logistical constraints regarding shipping and the additional time required to complete TEM analysis, the first two TEM results weren't available in the field until prior to the last day of sampling. The TEM TWA results for these two Day 1 samples (P0-00016 & P0-00017) were reported as 0.0056 s/cc and 0.2325 s/cc. Although one of the two TEM results was slightly above the OSHA PCM PEL of 0.1 f/cc, all previous PCM results were significantly below the OSHA PEL. Based on a combination of these results, the last day's reduced field time and expected reduction in road emissions (only traffic simulation being tested was the 10 mph scenario) it was determined to not be necessary to modify any established safety practices. The initial TEM TWA results for the last day's activities confirmed this decision, ranging in concentration from 0.0525 s/cc to 0.0788 s/cc (both below OSHA's PCM PEL of 0.1 f/cc). In the weeks following the completion of the Initial Study the laboratories reached consensus regarding the "scrolled lizardite" issue (see sections 4.3.1 and 5.2), which resulted in an increase in the TEM results initially reported. As indicated by Table 4-10, the updated TEM TWA results indicated that 7 of the 11 Initial Study personal samples exceeded the OSHA PCM PEL 0.1 s/cc, ranging in concentration from 0.0413 s/cc to 1.6875 s/cc (initially reported at 0.0525 s/cc). A comparison of the Initial Study PCM and TEM results indicated no apparent trend between the results other than the TEM results being higher in concentration.

As indicated by Table 4-11 all of the Post Resurfacing personal TWA concentrations were significantly below the OSHA PCM PEL of 0.1 f/cc (ranging from <0.0044 s/cc to 0.045 s/cc, with the exception of one sample (F2-00042) which experienced equipment failure during collection and is therefore considered unreliable. The counter on sample F2-00042 indicated 31 minutes of sampling prior to failure. If this counter reading was considered to be accurate the sample would be considered an excursion type sample and its TEM TWA of 0.3825 s/cc would be lower than OSHA PCM excursion regulations (1.0 f/cc). Unlike the Initial Study, a comparison of the Post Resurfacing PCM and TEM results indicated the TEM results being only slightly higher in concentration, with average TWAs of < 0.0313 and < 0.0101 respectively.

## SECTION 5. DATA QUALITY ASSESSMENT

This section provides a data quality assessment of field activities and analytical results as they relate to the 2002 (Initial Study) and 2003 (Post Resurfacing) objectives below.

Initial Study (initial) - The initial objectives of the Initial Study were: (1) to practice, examine and refine methodologies for collecting airborne asbestos dust samples so as to better develop a protocol for a full-scale study; and (2) to collect initial airborne asbestos data.

Post Resurfacing - The objective of the follow-up sampling was to assess the effectiveness of the resurfacing activities in reducing airborne asbestos concentrations, near Slodusty Road.

The assessment below is based on a review of field activities, laboratory results and Quality Control (QC) sample results. QC samples are used to identify problem areas and isolate the cause of the contamination. Both field and laboratory-based QC samples were collected / analyzed as part of the project. Field-based QC samples are samples that are prepared in the field and submitted blind to the laboratory. Laboratory-based QC samples are samples that are prepared in or re-analyzed by the laboratory.

### 5.1 Field Activities - Data Quality Assessment

A quality assessment of the project's field activities includes reviewing the objectives of each activity against actual outcomes, any associated field observations, and the results of field based QC samples.

#### 5.1.1 Field Procedures

Field procedures conducted during the 2002 and 2003 sampling events related to four activities: (1) stationary air sampling, (2) personal air sampling, (3) traffic simulations, and (4) meteorological sampling. A review of each of these field activities was conducted and is summarized below.

##### ***Stationary Air Sampling***

Field activities included collecting stationary air samples at various distances to monitor for air-entrained asbestos associated with vehicular traffic along Slodusty Road. A copy of the sampling procedures is provided in Appendix A, as well as a summary of the methodology within section 3.1. The initial objectives of the Initial Study air sampling were (1) to refine sampling methodologies and (2) to collect initial airborne asbestos data. The objective of the Post Resurfacing sampling was to duplicate the sampling activities refined during the 2002 Initial Study, in order to assess the effectiveness of the resurfacing activities in reducing airborne asbestos concentrations near Slodusty Road.

A total of 120 stationary air samples were collected during the Initial Study. Of these 120 samples, 86 were collected between Runs 00-01, 01-01, 01-02 and 02-01. During these four runs flow rates were set and monitored through the use of a rotometer attached to the sampler. Due to equipment failure, a primary flowmeter was not used to calibrate or set flow rates until the second Run (02-02) of 07/17/02, as was required by the sampling procedures. Although there are established sampling methods that allow for rotometers to be used as the primary control of pump flows it was determined via field comparison with a primary standard flowmeter that the rotometer was often quite inaccurate. Consequently the flow rates used to collect the 86 samples were suspect as to whether the "dialed in" rates were the true flow rates at which the samplers were operating.

For Run 02-02 a primary standard flowmeter was used to set the flow rates. A five-point calibration curve was developed using the data from Run 02-02 to estimate an actual flow rate for the previous runs in order to calculate a total volume sampled. This volume was termed "Estimated Total Volume" (see Appendix B). Although these estimated volumes are believed to be representative of the true volumes the calculated concentrations associated with the 86 affected samples should be used only for decisions regarding general data trends and refinements to the sampling methodology. It is for this reason that very limited analysis was performed on these samples. The results for these samples should not be used for health risk-based decisions.

In addition, while collecting sample P3-00007 during run 03-01 of the Initial Study a sampling tube collapsed under vacuum causing a 7.9 L/min drop between the recorded initial and final flow rates. The sample was not analyzed for this reason. The tubes for all samplers were replaced with heavy gauge tubing prior to the Post Resurfacing Sampling. Despite the different tubing fluctuations greater than 10 % between the initial and final flow rates were observed in 8 of the 96 Post Resurfacing stationary samples collected. For four of the eight samples the difference exceeded 20 %. With the exception of sample F2-00035 that had a ruptured filter (according to the lab) there was no apparent explanation for the fluctuations. The seven samples were collected with four different DTSC sampling pumps and three different Volpe pumps. The Post Resurfacing samples were analyzed in a phased approach and as such of the eight samples only sample F3-00013 was analyzed and included in the calculated averages. Sample F3-00013 was collected during Run 3-01 and had a difference of 16.3 % between its initial and final flow rates. The sample was selected for analysis because it was one of two samples collected during the scenario immediately adjacent to the road (within 5 ft). A review of the sample's results indicated they are comparable to three other samples that were collected at the same distance during similar runs. These other three samples had flow fluctuations of 1.3 % to 3.9 %.

For the twenty stationary samples collected during the no-simulation scenario the initial start flow rate was calibrated using an AALBORG mass flow meter. However subsequent to starting the simulation inconsistencies were noticed between AALBORG mass flow meter readings and those taken with a Dry Cal DC flow meter. Unlike the Dry Cal, inconsistencies were also noticed between AALBORG mass flow readings and as a result the Dry Cal flow meter was determined to be more accurate. The Dry Cal flow meter was used for all subsequent flow rate calibrations, including measuring the final flow rates for the twenty samples initially calibrated with the AALBORG meter. Due to the AALBORG's poor precision and comparison to the Dry CAL it was determined rather than using the AALBORG start rates, the start rates were estimated from the average observed difference (excluding outliers) between the start and final Dry Cal measured rates collected from 70 stationary samples collected during the Post Resurfacing event. The data used to calculate these estimated start flow rates is presented in Appendix B. The volumes calculated from the average estimated flow rate are considered to be representative of the true volumes and results are appropriate for use in this report.

The AHERA method used for analysis of the stationary samples requires that for each sample a minimum of 580 L be collected and that the analytical sensitivity be no greater than 0.005 s/cc. One of the purposes of the Initial Study was to maximize the flow rate for each sample location during each traffic scenario in order to collect the largest volume possible without overloading the sample with particulate. Due to the experimental nature of the Initial study twelve samples were collected with volumes less than the 580 L required. All twelve samples were collected within 10 ft of the road during 25 mph (30 vph) scenarios, which were observed to generate greater amounts of particulate requiring reduced flow rates within immediate proximity to the road. Based on the Initial Study samples the flow rates were adjusted during the Post Resurfacing sampling and consequently only four of the 96 samples were collected with volumes less than the AHERA 580 L requirement. All four samples were collected within 5 ft of the road during 25 mph (30 vph) scenarios with volumes ranging from 462 L to 494 L and sensitivities ranging from 0.0069 s/cc to 0.0074 s/cc. In the case of each of the four samples the asbestos concentration exceeded the analytical sensitivity. As indicated by the laboratory modification included in Appendix H, for the project a maximum of ten grid openings are analyzed for an AHERA analysis, regardless of sample volume and as a result in some instances the specified analytical sensitivity of 0.005 s/cc was not reached. A stopping rule of ten grid openings was established in order to assure analytical efficiencies of resources, time, production and cost.

A review of field data indicated all required information was recorded appropriately. Stationary sampling data is provided in Appendix F and associated results are in Appendix C. A review of the stationary sampling data indicated that the results from the 86 Initial Study stationary samples collected during Runs 00-01, 01-01, 01-02 and 02-01 should be used only for decisions

regarding general data trends and refinements to the sampling methodology and not for health risk-based decisions. All other samples excluding P3-00007 and the four Post Resurfacing samples with flow rate fluctuations exceeding 20 % do not warrant limitations with the intended use of the data towards project objectives.

### ***Personal Air Sampling***

Field activities during both the Initial and Post Resurfacing events included collecting personal samples with the objective of monitoring worker exposure. Personal samples were to be collected each day with a representative sample for each day's tasks. Representative samples were collected from both Volpe and DTSC personnel for all days of each sampling event. However, during the Initial Study on 7/17/02 while personal samples were collected from three field personnel a representative sample was not collected for the drivers of the test vehicles.

All personal samples were analyzed via both Phase Contrast Microscopy (PCM) and Transmission Electron Microscopy (TEM-AHERA analysis). A limitation of PCM technology is that it is unable to distinguish between asbestos fibers and non-asbestos fibers, due to this known limitation the personal samples were also analyzed via TEM-AHERA in order to supplement the information provided by the PCM results. The PCM results are required by OSHA for use in monitoring worker exposures and making personnel safety decisions in the field. Because the OSHA levels are based on occupational exposures assessed using the PCM technology, it is not appropriate to use TEM results for determining whether or not OSHA requirements have been met.

The TEM-AHERA method requires that for each sample a minimum of 580 L be collected and that the analytical sensitivity be no greater than 0.005 s/cc. Two of the Post Resurfacing personal samples had low volumes (62 L and 138 L) as a result of battery failures. Of the other seventeen personal samples collected during the project, seven (2 post and 5 initial) samples were collected with volumes less than the 580 L required by AHERA. The volumes of the seven samples ranged from 299 L to 422 L with sensitivities ranging from 0.003 s/cc to 0.098 s/cc. In the case of each of the seven samples the asbestos concentration exceeded the analytical sensitivity. As indicated by the laboratory modification included in Appendix H, for the project a maximum of ten grid openings are analyzed for an AHERA analysis, regardless of sample volume and as a result in some instances the specified analytical sensitivity of 0.005 s/cc was not reached. A stopping rule of ten grid openings was established in order to assure analytical efficiencies of resources, time, production and cost.

During the Initial Study there was one sample (P3-00003) that had a TEM result that was an order of magnitude higher than the others collected during the event. The significant difference in concentration does make the sample suspect.

However, the field notes do not indicate any unusual circumstances associated with the sample such as either an inaccurate volume being reported or the cassette being accidentally contaminated (dislodged or dropped).

A review of field data indicated all required information was recorded appropriately. Personal sampling data is provided in Appendix F and associated results are in Appendix C. A review of the personal sampling data did not indicate any issues associated with sample collection activities that warrant any limitations with the intended use of the data towards project objectives.

### ***Traffic Simulations***

Field activities included conducting traffic simulations, at different speeds and frequencies, to provide data that could be used to associate monitoring of air-entrained asbestos with vehicular traffic along Slodusty Road. A summary of the methodology used to simulate traffic is provided within section 3.1. The initial objectives of the Initial Study simulations were (1) to refine field methodologies and (2) to determine appropriate frequency and speeds for performing a more comprehensive study. The objective of the Post Resurfacing traffic simulations was to duplicate the representative traffic conditions determined and conducted during the 2002 Initial Study, in order to assess the effectiveness of the resurfacing activities in reducing airborne asbestos concentrations near Slodusty Road.

No issues were encountered during the Initial Study that required the field procedures to be modified. For consistency the same vehicles were used during both studies (a Dodge 4x4 truck and a Chevy Cavalier compact sedan). Communication and controls between vehicles and the traffic controller were clear and vehicle frequency was well controlled and coordinated with local traffic. One difference between the two events is how local traffic was counted in regards to the target vehicle frequency. The methodology allowed for local vehicles to either be counted towards the target vehicle frequency or for the local vehicles to not be counted, but allowed to travel slowly through the test area (minimizing additional dust emissions). During the Initial Study with the exception of two vehicles (Run 01-01 & Run 03-01), vehicles were consistently not counted as part of the target frequency and were allowed to pass slowly through the test area. The local traffic frequency ranged from 0 to as many as 5 additional vehicles above the target frequency. Although local vehicles traveled slowly through the test area it is possible that the cumulative affect of the additional vehicles may have resulted in emissions being slightly overestimated for the test speed, especially for the 10 vph scenarios. Out of this concern during the Initial Study 10 mph (10 vph) scenario a scheduled simulated run was skipped following an unusual high frequency of local traffic (3 vehicles in 3 minutes). During the Post Resurfacing sampling local vehicles were counted towards the target frequency during all simulations, including during run 3-02 (30 vph scenario) when 3 local vehicles traveled consecutively (2:42pm) through the test area. Although it is possible that the cumulative affect of the consecutive



vehicles may have resulted in emissions being slightly overestimated for the test run the data shows that this is not the case. The run's air sampling results immediately adjacent to the road (at 5 ft) are approximately equal to those taken at the same location during the same scenario of the previous day (run 2-02), which did not include any consecutive traffic. The local traffic was requested to travel at the test speed, however based on field observations (not measurements) the vehicles appeared to in some cases either travel below the 25 mph test speed or above the 10 mph test speed. A review of field data indicated all required information was recorded appropriately. A summary of all traffic data is provided in appendix E.

Based on observations made during the Initial Study the 10 mph / 10 vph scenario was chosen to represent posted speed and highest vehicle frequency conditions that may be encountered along Slodusty Road during peak commute. Local traffic was observed at an average frequency of 5 vph during both the afternoon and morning scenarios, ranging in frequency from 0 to 9 vph. Based on this data a test frequency of 10 vph is representative of the highest vehicle frequency conditions that may be encountered along Slodusty Road during peak commute. No issues identified during the traffic simulation data review warrant any limitations associated with the intended use of the data towards project objectives.

### ***Meteorological Sampling***

Field activities included conducting meteorological sampling as needed to document conditions encountered during monitoring of air-entrained asbestos with vehicular traffic along Slodusty Road. The initial objectives of the Initial Study meteorological sampling were (1) to refine field methodologies and (2) to document meteorological conditions during the Initial Study air sampling. The objective of the Post Resurfacing meteorological sampling was to document meteorological conditions during air sampling as compared to the 2002 event, as needed, in assessing the effectiveness of the resurfacing activities in reducing airborne asbestos concentrations.

During the Initial Study MET units were placed at different locations during different days. The purpose of varying the locations were to determine if recorded conditions varied as a result of distance from the roadway, and / or were being affected by site geographic or vegetative characteristics. Specific Initial Study MET locations are identified within section 3.2.2. A review of the Initial Study data determined that there was some variation between the units placed on opposing sides of the roadway (east units compared to west), however the distance along the tangent did not have a similar affect. Based on this data during all Post Resurfacing sampling activities units were placed in a symmetric fashion, fifty feet from the road edge on the east and west side of Slodusty Road.

In addition to utilizing slightly, but similar spatial locations, different types of MET units were used during each sampling event. The Initial Study used MET

stations called Transportable Automated MET Station (TAMS), which have cup anemometers and can operate in wind speeds ranging from 2-70 knots (2.3-80.6 mph). Cup anemometers generally become less accurate at low wind speeds. Based on the low wind speeds recorded during the Initial Study, Gill WindObserver II Ultra Sonic anemometers were used instead of the TAMS during the Post Resurfacing sampling. The Gill ultra sonic anemometers are much more accurate at low wind speeds having a wind speed resolution of 0.01 m/s (0.02 mph) and a range of 0-145 mph. Although different sensors were used during the two events common data was collected and for both units data was recorded at a 1Hz sample rate and stored to a personal computer for post sampling analysis. A review of both units' data indicated similar wind speeds were recorded and that the wind speeds were within the range of both units. Based on this review although the Gill units are more sensitive at lower wind speeds the data from both events are comparable.

A review of field data indicated all required information was recorded appropriately. A summary of all MET data is provided in Appendix F. No issues identified during the meteorological sampling data review warrant any limitations associated with the intended use of the data towards project objectives.

### 5.1.2 Field Quality Control Samples

Three types of field QC samples, Field Blanks, Lot Blanks, and Sealed Blanks were collected during both the 2002 and 2003 sampling events,. See Table 5-1 below.

**Table 5-1 Types of Field-Based Quality Control Samples**

Type of QC Sample	Description
<b>Field Blanks</b>	This is a filter cassette that is handled in the same manner as air samples, however no air is drawn through the cassette. Instead the caps are removed and the cassette is held open for approximately 30 seconds and then resealed. Field blanks are stored and shipped with sample cassettes.
<b>Lot Blanks</b>	This is an unused filter cassette taken from each new batch of cassettes. A batch may contain multiple boxes of cassettes (50 cassettes in one box). The filters are to be analyzed for asbestos fibers by the same method as will be used for the field samples.
<b>Sealed Blanks</b>	This is an unused filter cassette that is carried and shipped with each sample set. This representative cassette is NOT opened in the field. The filters are to be analyzed for asbestos fibers by the same method as will be used for the field samples.

### ***Data Quality Objectives – Field QC Samples***

The purpose of field blanks is to determine if any asbestos contamination has occurred during sample handling activities. For samples analyzed by AHERA the

acceptable filter background level is 70 structures / mm<sup>2</sup>. The filter background level is the concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank. (TEM AHERA) The purpose of lot blanks is to verify that cassettes, as provided by the manufacturer, are “clean” of asbestos by determining the background asbestos structure concentration. The purpose of sealed blanks is determine if any asbestos contamination has occurred during sample storage and shipping. AHERA requires for TEM analysis field blanks be collected at a frequency of two per I/O site (inside / outside) and sealed blanks at a frequency of one per I/O site.

### ***Data Results - Field QC Samples***

During the project a total of 14 field QC samples (seven field blanks and seven lot / sealed blanks) were collected. A total of ten QC samples (four field blanks and six lot / sealed blanks) were collected during the Initial Study and four (three field blanks and one lot / sealed blank) during the Post Resurfacing event. Because they were unopened lot blanks also served as Sealed Blanks. A lot blank was collected to represent every 50-cassettes (or box) used during the study (note: there were several boxes, all from the same lot). Field blanks were also collected at a minimum of once-per day. Three field personnel (at least one of which was a driver) were monitored for asbestos during each day's site activities.

A higher number of QC samples were collected during the Initial Study due to the additional representative lot blanks needed for the different types of filters / cassettes used while refining the methodology. Eight of the fourteen QC field samples were analyzed. Not all filter types (poly cartridge, 47mm cartridge, 47mm filters) were relevant for the project; therefore, not all lot samples required analysis. QC field sample results are summarized in Table 5-2 below.

**Table 5-2 Field QC Sample Results for Initial Study Runs (02-02 & 03-01) and Post Resurfacing Sampling**

Index ID	Date collected	QC Type	Filter Type	LOT #	ANALYSIS		
					s/cc	Lab	Method
F2-00021	08/19/03	Field Blank	MCE (25mm, 0.45u)	Lot#H3EN2781	Non-Detect 10 GOs (9.1 s/mm2)	RESI	AHERA
F3-00042	08/20/03	Field Blank	MCE (25mm, 0.45u)	Lot#H3EN2781	Non-Detect 10 GOs (9.1 s/mm2)	RESI	AHERA
P1-00001	7/16/02	Field Blank	MCE (25mm, 0.45u)	Lot#410FKA-2135	Non-Detect 10 GOs (7.9 s/mm2)	EMSL	ISO
P2-00001	07/17/02	Field Blank	MCE (47mm, 0.45u)	Lot#H2BN10114-3499	Non-Detect 10 GOs (8.6 s/mm2)	RESI	AHERA
					Non-Detect 10 GOs (8.6 s/mm2)	RESI-RS	AHERA
P3-00001	07/18/02	Field Blank	MCE (25mm, 0.45u)	Lot#410FKA-2135	Non-Detect 10 GOs (8.6 s/mm2)	RESI	AHERA
F1-00023	08/18/03	Lot / Sealed Blank	MCE (25mm, 0.45u)	Lot#H3EN2781	Non-Detect 10 GOs (9.1 s/mm2)	RESI	AHERA
PL-00004	7/16/02	Lot / Sealed Blank	MCE (47mm, 0.45u)	Lot#H2BN10114-3499	Non-Detect 10 GOs (7.9 s/mm2)	EMSL	ISO
PL-00003	07/16/02	Lot / Sealed Blank	MCE (25mm, 0.45u)	Lot#410FKA-2135	Non-Detect 10 GOs (7.9 s/mm2)	EMSL	ISO

A review of the project field QC data indicates field blanks were collected within the AHERA required frequency of two per I/O site (7 were collected and two were analyzed per event) and within the required frequency for lot blanks and sealed blanks (7 were collected and 3 were analyzed, one for each batch of samples analyzed). The results of all QC blank samples were non-detect and well within the required AHERA filter background level of 70 structures / mm2, as indicated in Table 5-2 above.

## 5.2 Analytical Results - Data Quality Assessment

A quality assessment of the project's analytical results includes not only a review of all individual results but also a comparison of results between different laboratories and within the same laboratory (Lab-based QC samples). Reservoirs Environmental Inc. (RESI) was the primary TEM laboratory on the project, with EMSL Analytical Inc. (EMSL) conducting all PCM analysis and providing initial TEM analysis of the Initial Study samples. Reservoirs also used the National Environmental Reference Laboratory at the Denver Federal Center as part of their laboratory QC program (for inter-lab and verified analysis, see Section 5.2.2 below).

### 5.2.1 Analytical Results Between Laboratories

Initial analysis of the Initial Study samples by EMSL Analytical, Inc. raised questions regarding how to characterize many complex and single structures that were being observed. Numerous structures were observed to have splayed ends (“weathered”) and some structures to have a tubular appearance. Based on these observations, questions were raised regarding if the structures should be classified as “scrolled lizardite”, rather than chrysotile asbestos. Serpentine rock is primarily composed of one or more of three magnesium silicate minerals: lizardite, chrysotile, and antigorite. Chrysotile and lizardite have the same chemical composition, however have a different crystal lattice. Chrysotile in fibrous form is the most common type of asbestos. Asbestos is a group of silicate minerals that readily separates into thin, strong flexible fibers that are heat resistant. Lizardite and antigorite don’t form asbestos fibers and instead are plate-like.” (CGS 2002) To determine how to best classify these structures the samples and the TEM grids that were prepared and analyzed by EMSL were sent to Reservoir Environmental Inc. for further evaluation. Images / micrographs of the structures were shared between the two laboratories for discussion. Reservoir also obtained independent opinions from two other outside laboratories. The consensus between the laboratories was that the structures in question meet appropriate criteria as defined by the analytical methods and should be classified as chrysotile asbestos. As indicated in Table 56-3 below, classifying the structures as chrysotile asbestos rather than “scrolled lizardite” resulted in stationary sample TEM concentrations being approximately two orders of magnitude higher than originally indicated. However, the decision to classify the structures as chrysotile asbestos did not have a similar affect regarding initial personal sample TEM results, except in the case of sample P3-00003. See Appendix C and Table 5-3 below. All observations, comparisons, average results, findings and recommendations within this report are based on analytical results that classified the structures as chrysotile asbestos rather than “scrolled lizardite” (i.e., includes RESI’s 98 AHERA results, excludes the 20 AHERA results initially completed by EMSL, prior to reaching a consensus).

**Table 5-3 Chrysotile Asbestos vs. “Scrolled Lizardite”**

Sample	Category (Field=F, Personal=P)	Classified as “Scrolled Lizardite” i.e. Not Counted (initial EMSL AHERA) s/cc	Classified as Chrysotile Asbestos (RESI AHERA) s/cc	Notes
P3-00011	F	0.0085	0.1900	At 10 ft for Run 03-01 (10 mph / 10 vph)
P2-00044	F	0.0240	2.2000	At 10 ft for Run 02-02 (25 mph / 30 vph)
P2-00034	F	0.0320	0.8700	At 30 ft for Run 02-02 (25 mph / 30 vph)
P2-00035	F	0.0510	1.6000	At 50 ft for Run 02-02 (25 mph / 30 vph)
P2-00036	F	0.0510	1.2000	At 80 ft for Run 02-02 (25 mph / 30 vph)
P2-00037	F	0.0044	0.7600	At 100 ft for Run 02-02 (25 mph / 30 vph)
P2-00038	F	0.0091	0.9300	At 130 ft for Run 02-02 (25 mph / 30 vph)

P2-00039	F	0.0220	0.5300	At 160 ft for Run 02-02 (25 mph / 30 vph)
P2-00046	F	0.0320	2.3000	At 30 ft for Run 02-02 (25 mph / 30 vph)
P1-00060	P	0.3500	0.3300	In pick-up truck
P2-00002	P	0.3400	0.4700	Field Personnel (DTSC personnel)
P2-00003	P	0.0610	0.0610	Traffic Controller (DTSC personnel)
P2-00004	P	0.4100	0.5500 & 0.2600 (QC-RPS)	Field Personnel (Volpe personnel)
P3-00002	P	0.2100	0.4100	Field Personnel (Volpe personnel)
P3-00003	P	0.1400	4.5000	Traffic Controller (DTSC personnel)
P3-00004	P	0.1500	0.1100	Sedan Driver (Volpe personnel)

**16 Total Samples were analyzed via AHERA by BOTH EMSL & RESI**

(EMSL analyzed an additional 4 Initial Study samples from earlier runs (not Runs 02-02 or 03-01), that were not analyzed by RESI)

**NOTE: Above Personal Sample Results have NOT been converted to TWA concentrations**

### 5.2.2 Lab-Based Quality Control Samples

There are six types of laboratory based QC samples for transmission electron microscopy (TEM) or phase-contrast microscopy (PCM). See Table 5-4 below.

**Table 5-4 Types of Laboratory-Based Quality Control Samples**

Type of QC Sample	Description
<b>Lab Blank</b>	Laboratory blanks are samples that are collected at the laboratory to determine if contamination is being introduced during analysis.
<b>Recount –Same (RS)</b>	This is a TEM grid or a PCM slide that is re-examined by the same microscopist who performed the initial examination. In the case of a TEM grid, the microscopist returns to the same grid openings as were counted in the original examination. In the case of PCM, the microscopist simply re-counts the slide at randomly selected fields.
<b>Recount Different (RD)</b>	This is a TEM grid or a PCM slide that is re-examined by a different microscopist in the same laboratory than the individual who performed the initial examination. In the case of a TEM grid, the microscopist returns to the same grid openings as were counted in the original examination. In the case of PCM, the microscopist re-counts the slide at randomly selected fields.
<b>Re-preparation (RP)</b>	This is a grid of a slide that is prepared from a new aliquot of the same field sample as was used to prepare the original grid or slide. This is often referred to as a laboratory duplicate. Typically this is done within the same lab as did the original analysis, but a different lab may also prepare grids from a new piece of filter. If the re-preparation is done within a laboratory, the re-preparation and the re-analysis should be done by a different person than did the original, whenever possible.
<b>Verified Analysis VA</b>	This is a re-count of a TEM grid (same openings) or a PCM slide (random fields) by a different laboratory OR within the

	same laboratory but a different analyst as performed the original analysis. Although similar to a RD it has different requirements in regards to documentation and acceptability. A detailed protocol for verified analysis is provided in NIST (1994).
<b>Inter-Lab Analysis (IL)</b>	This is a re-count of a TEM grid (same openings) or a PCM slide (random fields) by a different laboratory than performed the original analysis. A detailed protocol for verified analysis is provided in NIST (1994).

### ***Data Quality Objectives – Lab-based QC Samples***

As indicated in section 4.3 all analysis was performed at laboratories that are fully accredited for PCM and TEM analysis under the National Voluntary Laboratory Accreditation Program (NVLAP), as sponsored by the National Institute of Standards and Technology (NIST). Under NVLAP, laboratory-based QC samples are required to be analyzed at a frequency of 4 % for LBs, 1 % for VA, 0.5 % for IL, and an additional 4.5 % comprising a combination of RP, RS, and RD (distribution to be at the discretion of the laboratory).

Performing laboratory QC sample analysis within established frequency requirements in and of itself is not sufficient to validate lab-based QC samples and the set of analytical results they represent to be acceptable. In addition to reviewing the individual results and raw data for acceptability the results and raw data of the QC samples and their respective original analysis need to be compared and evaluated against acceptance criteria.

### ***Data Results - Lab-Based Quality Control Samples***

The lab-based QC sample results for both project laboratories are summarized in the sections below.

#### Reservoir Environmental Inc. (RESI)

RESI performed a total of 98 AHERA analyses during the project including all QC analysis. Table 5-5 summarizes the corresponding NVLAP required frequency for each type of lab-based QC samples. As indicated below, RESI performed a total of nine QC analysis or 10 %, which exceeds the NVLAP requirement of 8 samples. RESI performed an additional inter-lab analysis. An additional verified analysis was performed, which also meets the requirements and could have been counted as such. Table 5-6 below summarizes the results of RESI's QC analysis.

**Table 5-5 RESI Frequency - Laboratory-based QC Samples**

	NVLAP		RESI	
	%	# Required	# Analyzed	%
LAB BLANKS (1 per 25)	4.0 %	3	3	3.1 %
VERIFIEDS	1.0 %	1	2	2.0 %
INTER-LAB	0.5 %	0	1	1.0 %
RECOUNT-SAME	*	4	1	3.1 %
REPREP	*		1	
RECOUNT-DIFF	*		1	

NOTE: For "\*" % NVLAP allows the lab to use their own discretion as long as the combination of RS, RP and RD percentages equal 4.5%

10 %                      8                      9                      10.1 %

**Table 5-6 RESI Results - Laboratory-based QC Samples**

Index ID	Date collected	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Non QA Analysis (TEM-AHERA)  s/cc	QA Analysis (TEM-AHERA)  s/cc	QC TYPE
F1-00019	08/18/03	F	0.0091	0.0091	VA
F2-00031	08/19/03	F	0.0920	0.0850	IL
F3-00015	08/20/03	F	4.40E-03	4.40E-03	VA
F3-00027	08/20/03	F	9.20E-03	9.20E-03	RD
P2-00001	07/17/02	FB	Non-Detect 10 GOs (8.6 s/mm <sup>2</sup> )	Non-Detect 10 GOs (8.6 s/mm <sup>2</sup> )	RS
LB 91659	NA	LAB Blank	NA	Non-Detect 10 GOs (9.1 s/mm <sup>2</sup> )	LB
LB 96820	NA	LAB Blank	NA	Non-Detect 10 GOs (9.1 s/mm <sup>2</sup> )	LB
LB 96823	NA	LAB Blank	NA	Non-Detect 10 GOs (9.1 s/mm <sup>2</sup> )	LB
P2-00004	07/17/02	P	0.5500	0.2600	RPS



As indicated in Table 5-6, all lab blank (LB) analyses were non-detect and acceptable, with detection limits well below the AHERA requirement of 70 structures / mm<sup>2</sup>.

A review of the raw data for all re-analyses (VA, RS, and RD) and their associated field samples indicated that they are all acceptable. In each case the analytical data was complete, all the same grid openings were analyzed, no additional structures were identified and all structures originally identified were reported with the same structure type, mineral class and within acceptable dimensions.

A review of the raw data for the re-preparation (RPS) analysis and the associated field sample analysis indicated that the analyses are acceptable. For both analyses the analytical data was complete and it was determined that the two results are not statistically different (the field sample analysis identified a total of 24 structures and the RPS analysis identified 23 structures).

A review of the raw data for the inter-lab analysis (IL) and the associated field sample analysis indicated that they are acceptable. For both analyses the analytical data was complete and all the same grid openings were analyzed. The IL analysis was however unable to locate a single small fiber that was originally identified (12 of 13 structures were verified). All other originally identified structures were reported with the same structure type, mineral class and within acceptable dimensions. The lab was unable to perform a reconciliation analysis for the unidentified fiber.

RESI's lab-based QC sample results and the data results they represent were determined to be acceptable according to frequency requirements and acceptance criteria.

#### EMSL Analytical, Inc.

EMSL performed initial analyses of the Initial Study samples, verifying particulate loading of 73 samples and presence of asbestos on 26 of the 72 samples. EMSL only performed twenty AHERA, five ISO, and twelve PCM analyses. EMSL is a documented NVLAP accredited laboratory, however due to the limited number of formal project analyses EMSL did not perform and project specific QC analysis. The project samples counted as part of EMSL's overall QC program and their QC sample analyses were performed on non-project specific samples, which were not evaluated under this project.

## SECTION 6. SUMMARY OF FINDINGS

During the Summers of 2002 (Initial study) and 2003 (Post Resurfacing) the Volpe Center and DTSC performed two sampling events involving monitoring of air-entrained asbestos associated with vehicular traffic along Slodusty Road, in Garden Valley, CA. The initial objectives of the Initial Study were: (1) to practice, examine and refine methodologies for collecting airborne asbestos dust samples so as to better develop a protocol for a full-scale study; and (2) to collect initial airborne asbestos data. DTSC determined that the airborne asbestos levels obtained from the Initial Study warranted proceeding with resurfacing of the roadway rather than following-up with the full-scale study. DTSC's resurfacing approach involved a multi layer approach of compacted  $\frac{3}{4}$  aggregate, with a chipseal barrier and top surface of fine Lime Stone aggregate. DTSC completed resurfacing activities along Slodusty Road in early August 2003. Following the completion of resurfacing activities, the Volpe Center returned with DTSC to resample along the roadway under post remediated conditions. The objective of the follow-up sampling was to assess the effectiveness of the resurfacing activities in reducing airborne asbestos concentrations, near Slodusty Road.

Both sampling events involved monitoring levels of airborne asbestos in the vicinity of Slodusty Road Rd as they vary with distance and vehicular traffic (frequency and speed). Two traffic scenarios were tested using the same test vehicles (a 4x4 truck and a compact sedan). A 25 mph / 30 vph scenario was chosen to represent an extreme worst case traffic condition for Slodusty Road or a similar unpaved serpentine roadway. While a 10 mph / 10 vph scenario was chosen to represent actual speed and highest vehicle frequency conditions that may be encountered along Slodusty Road during peak commute. Local traffic frequencies observed during both sampling events confirmed the 10 mph / 10 vph scenario to be representative. Local traffic was observed at an average frequency of 5 vph during both the afternoon and morning scenarios, ranging in frequency from 0 to 9 vph. During both events meteorological data, particulate data, stationary air samples and personal air samples were collected. The samples were collected following the methodologies refined during the first three days of the Initial Study (Runs 00-01, 01-01, 01-02 and 02-01). The stationary air samples were located at similar distances on both (east and west) sides of the road, along a transect approximately perpendicular to the center-point of the test stretch of road. In order to directly compare sampling results collected during pre and post resurfacing activities the air samplers were positioned at the same locations as during the Initial Study, with two exceptions. An additional air station was established at a further distance (300 ft) on the west side of the road (a similar station was not established on the east side due to physical barriers). During the Post Resurfacing event another air station was established at a residence on Bayleaf Drive approximately 1½ miles from the site to serve as an ambient background sample.

All analysis were performed by laboratories that are fully accredited under the National Voluntary Laboratory Accreditation Program (NVLAP). All TEM asbestos results were provided on project specific electronic data deliverables (EDDs). The EDDs are a modified version of an EDD that was developed by EPA Region 8 as part of asbestos investigative and Superfund activities in Libby, MT. The purpose of the EDD is to electronically capture counting information in order to accommodate potential future changes in regulatory and health classification requirements, enabling samples to be re-evaluated in the future without needing the actual samples to be reanalyzed.

Results of the sampling and analysis activities are as follows:

- **Meteorological Sampling:** The Meteorological conditions were very similar during both the Initial Study and Post Resurfacing sampling events. Wind conditions were on average very consistent between the two studies, with little fluctuation during the day. The winds were predominantly out of the west and northwest direction and were generally less than 8 mph. The average morning temperature readings ranged from 82.4 to 88.6 degrees Fahrenheit, with temperatures increasing in the afternoon ranging from 89.8 to 90.0 degrees Fahrenheit. The afternoon relative humidity readings were also similar, ranging from 23.3 % to 26.0 %. However, the average morning relative humidity readings were higher during the Initial Study than they were during the Post Resurfacing sampling (49.5 % compared to 27.6 %).
- **Particulate Sampling:** Particulate measurements were collected by DTSC during both the 2002 (Initial Study) and 2003 (Post Resurfacing) sampling events. During the Initial Study spot measurements were taken west of the roadway at various distances (5, 10, 30, 50, 80, and 100 ft) during two different 25 mph traffic scenarios (10 vph and 30 vph). The measurements ranged from 0.02 to 3.48 mg/m<sup>3</sup> of particulate, depending on proximity to the roadway. Post Resurfacing measurements were taken only within immediate proximity to the road and ranged from 0.005 to 0.015 mg/m<sup>3</sup>, compared to Initial Study readings of 1.01 to 2.54 mg/m<sup>3</sup> at the same distance. Based on a limited set of particulate readings it appears the resurfacing activities reduced particulate emissions by more than 99 %, near Slodusty Road.
- **Stationary Air Sampling:** During the Initial Study a total of 120 stationary samples were collected. A data quality assessment of the 120 samples indicated the first 86 samples collected during Runs 00-01, 01-01, 01-02 and 02-01 should be used only for decisions regarding general data trends and refinements to the sampling methodology and not for health risk-based decisions. Also, sample P3-00007 should not be used due to documented equipment failure. The remaining 33 stationary samples representing both tested traffic scenarios were analyzed via the AHERA method. None of the

samples were overloaded and as such were analyzed following a direct sample preparation procedure. The AHERA results indicated the presence of asbestos at all distances sampled during the Initial Study. The asbestos concentrations of the 33 samples varied with proximity and traffic scenario, ranging from 0.0093 s/cc to 9.5 s/cc. In fact at 190 ft, which was the furthest sample collected during the Initial Study the average concentration was still 0.1870 s/cc for the 25 mph (30 vph) scenario. Plots of the average Initial Study results indicate that during both the 10 mph (10 vph) and 25 mph (30 vph) traffic scenarios the asbestos concentrations are significantly higher 5 ft from the roadway, with the concentration at 10 ft being 64 % to 70 % lower. As the distance increase from 10 ft away the concentration reduction is much more gradual, dropping by approximately 40 % every 20 ft. In addition to distance, traffic conditions appear to significantly affect the levels of asbestos that were emitted from the road. The concentrations for the Initial Study measured at each distance are approximately an order of magnitude higher for the 25 mph (30 vph) scenario when compared to the 10 mph (10 vph) scenario.

Following completion of the resurfacing activities the stationary sampling was repeated during the Summer of 2003 (Post Resurfacing) under similar conditions following the methodologies refined during the Initial Study. During the Post Resurfacing sampling 96 stationary samples were collected. A data quality assessment of the 96 samples indicated four samples should not be used to evaluate the effectiveness of the resurfacing activities. These four samples were documented to have been collected with flow rate fluctuations exceeding 20 %. Of the remaining 92 stationary samples 35 were selected in a phased approach and were determined to be representative of characterizing airborne asbestos levels at varying distances during both traffic scenarios. None of the samples were overloaded and as such were analyzed following a direct sample preparation procedure. The asbestos concentrations of the 35 samples varied with proximity and traffic scenario, ranging from <0.0043 s/cc to 0.0990 s/cc. The background sample results indicated a background concentration for the area of 0.0047 s/cc. Unlike during the Initial Study, there is no apparent trend to the asbestos concentrations in regards to distance for the 10 mph (10 vph) scenario. This is also true for the 25 mph (30 vph) scenario, with the exception being at 5 ft, which had an elevated average result compared to sampling stations further from the roadway. Also, there is no apparent trend to the average Post Resurfacing results in regards to traffic conditions other than that the average concentrations associated with simulated traffic tend to be slightly higher than the no simulation scenario and the background sample.

A review of the AHERA results associated with both sampling events indicates a fiber distribution of primarily complex chrysotile asbestos structures less than 5um in length, with a few free scattered chrysotile fibers. Approximately 90 % of the structures observed are less than (<) 5.0 um in

length and 10 % are greater than or equal to ( $\geq$ ) 5.0  $\mu\text{m}$  in length. Plots of the average Post Resurfacing results compared to the Initial Study results indicate an average 93 % reduction in asbestos concentrations during the 10 mph (10 vph) scenario and an average 98 % reduction during the 25 mph (30 vph) traffic scenario. The average Post Resurfacing results for the 10 mph (10 vph) scenario are approximately an order of magnitude lower than those of the Initial Study average results. While the average Post Resurfacing results for the 25 mph (30 vph) scenario are approximately two orders of magnitude lower than those of the Initial Study average results.

Based on a comparison of the Initial Study and Post results it appears the resurfacing activities reduced airborne asbestos concentrations by 93 % to 98 %, near Slodusty Road. It should be noted that these observed reductions are based on Post Resurfacing sampling that was performed 1 week following the completion of the road resurfacing activities and no known occurrences of precipitation.

- **Personal Air Sampling:** Field activities during both the Initial Study and Post Resurfacing events included collecting personal samples with the objective of monitoring worker exposure. Personal samples were collected each day with a representative sample for each day's task. Representative samples were collected from both Volpe and DTSC personnel for all days of each sampling event. A total of 19 personal samples were collected during both events (11 Initial and 8 Post). All personal samples were analyzed via both Phase Contrast Microscopy (PCM) and Transmission Electron Microscopy (TEM-AHERA analysis). A limitation of PCM technology is that it is unable to distinguish between asbestos fibers and non-asbestos fibers. Due to this known limitation the personal samples were also analyzed via TEM-AHERA for the purpose of supplementing the information provided by the PCM results. The PCM results are required by OSHA for use in monitoring worker exposures and making personnel safety decisions in the field.

The Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) is based on PCM analytical results. The OSHA 8 hr time-weighted-average TWA PEL is 0.1 f/cc. All of the Initial Study and Post Resurfacing PCM TWA concentrations are significantly below the OSHA PCM PEL. Based on these PCM results it was not necessary to upgrade or modify safety procedures during either event. Because the OSHA levels are based on occupational exposures assessed using the PCM technology, it is not appropriate to use TEM results for determining whether or not OSHA requirements have been met.

During the Initial Study as a result of logistical constraints regarding shipping and the additional time required to complete TEM analysis the first two TEM results weren't available in the field until prior to the last day of sampling.

Consequently TEM results were of limited supplemental use to PCM results while in the field. In the weeks following the completion of the Initial Study the laboratories reached consensus regarding the “scrolled lizardite” issue (see sections 4.3.1 and 5.2), which resulted in an increase in the TEM results initially reported. The updated TEM TWA results indicated that 7 of the 11 Initial Study personal samples exceeded 0.1 s/cc, ranging in concentration from 0.0413 s/cc to 1.6875 s/cc (initially reported at 0.0525 s/cc). A comparison of the Initial Study PCM and TEM results indicated no apparent trend between the results other than that the TEM results were higher in concentration as expected due to the increased sensitivity of TEM analysis to detect smaller, narrower structures than the PCM analysis.

All of the Post Resurfacing personal TWA concentrations were significantly below the OSHA PCM PEL of 0.1 f/cc (ranging from <0.0044 s/cc to 0.045 s/cc), with the exception of one sample that experienced equipment failure. The sample counter indicated 31 minutes of sampling prior to failure. If this counter reading was considered to be accurate the sample would be considered an excursion type sample and its TEM TWA of 0.3825 s/cc would be lower than OSHA PCM excursion regulations (1.0 f/cc). Unlike the Initial Study, a comparison of the Post Resurfacing PCM and TEM results indicated the TEM results being only slightly higher in concentration, with average TWAs of <0.0313 and <0.0101 respectively.

## SECTION 7. RECOMMENDATIONS

A review of the findings from the 2002 and 2003 sampling and analysis activities indicate the following recommendations should be considered regarding future investigative activities related to asbestos emissions along Slodusty Road or similar unpaved serpentine roadway.

- **Operational Improvements:** The majority of potential operational improvements were identified during the Initial Study and were incorporated into the Post Resurfacing Sampling activities. In addition those already incorporated, future similar sampling events should consider the following: (1) collecting replicate (co-located) stationary samples at a frequency of 10 %; (2) collecting excursion personal samples, not just full period samples, in order to obtain a better understanding of potential worker exposure; (3) collecting additional “background” samples, in order to obtain a better representation of actual background levels for the area; (4) collecting additional stationary samples at further distances from the roadside (>300 ft if possible); and (5) if possible selecting a test area with no vegetative or physical barriers, on either side of the road, that may alter meteorological conditions or limit stationary sample locations.
- **Follow-up Monitoring Along Slodusty Road:** Based on a comparison of the Initial Study and Post Resurfacing results it appears the resurfacing activities reduced airborne asbestos concentrations by 93 % to 98 %, near Slodusty Road. However, the long-term effectiveness of the resurfacing approach are unknown. The Post Resurfacing sampling activities should be repeated along Slodusty Road in the future to verify vehicular traffic has not over time sufficiently broken down the chipseal barrier to release the asbestos fibers present within the original roadbed.
- **Monitoring of Air-entrained Asbestos Near Additional Un-paved Serpentine Roads:** The Post Resurfacing sampling activities should be repeated along other similar unpaved roadways that have been sampled and identified to contain different levels of asbestos within the roadbed. The sampling should also be conducted on an unpaved serpentine roadway that was sampled and identified not to contain asbestos within the roadbed. Sampling along a roadway where asbestos is identified to be non-detect is important due to known limitations associated with sampling for and analyzing for asbestos within environmental media, such as a soil or gravel matrix. The results from these sampling events can supplement the data from the Initial Study to create a data matrix for assessing potential asbestos emission at other unpaved serpentine roadways based on the level of asbestos identified to be within the roadbed. A “screening” matrix of this type will enable decision makers to make initial conclusions based on bulk sampling of roadways which is a more economic and efficient approach

than conducting air sampling along each of the area's unpaved serpentine roadways, which isn't feasible. A screening matrix will also enable decision makers to prioritize among roadways determined to require further investigative and/or resurfacing activities.

- ***Investigations for Secondary Sources of Asbestos (resulting from asbestos emissions from unpaved serpentine roads)***  
The resurfacing activities have significantly reduced airborne asbestos concentrations associated with vehicular traffic along Slodusty Road. The Initial Study results have indicated that asbestos fibers have been transported from the roadway and potentially at distances greater than 300 ft. Asbestos fibers are environmentally persistent and as such, may still be present and a potential concern. Other secondary sources and exposure routes should be investigated. Potential secondary sources include, but aren't limited to residences, local cars, and soil adjacent to the roadway. Past studies have indicated that due to the persistent and physical characteristics of asbestos fibers they have the potential to accumulate over time within residences and vehicles.



## SECTION 8. REFERENCES

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## **Appendix A – Air Sampling Procedures**

## SECTION 1. GENERAL METHODOLOGY

The purpose of the sampling procedures described below is to evaluate asbestos emissions along an unpaved serpentine covered roadway by observing airborne asbestos concentration levels as they vary with distance and traffic frequency and speed. These procedures were developed from: (1) the CARB Standard Operating Procedure for Monitoring Asbestos in AIR (CARB 2001); (2) the ISO 10312 TEM method (ISO 10312); and (3) results and field observations from sampling activities conducted in July 2002 (Pilot Study) along Slodusty Road in Garden Valley, CA

Following the procedures described in the sections below, air samples are collected at various distances, along a transect that is approximately perpendicular to the center-point of a test stretch of road (on both sides of the road). The samples are collected in conjunction with conducting defined traffic simulations and collecting meteorological data (wind speed, wind direction, air temperature and humidity).

## SECTION 2. STATIONARY AIR SAMPLING

### 2.1 Air Sampler (Station) – Set-up & Associated Equipment

Each air sample station consists of a battery powered high volume diaphragm pump (Thomas Pump Model 007BDC19, or equivalent) that draws air thru a mounted system (via a support tripod) comprised of thick-wall vinyl tubing (LT-4-10V vinyl tubing, or equivalent), a rotameter (Dwyer Rotameter #RMA 22-TMV with a range of 2-25 l/min, or equivalent) for flow control and a three piece TEM filter cassette (see Figures 1 & 2 below). The TEM cassette should be mounted vertically facing down.

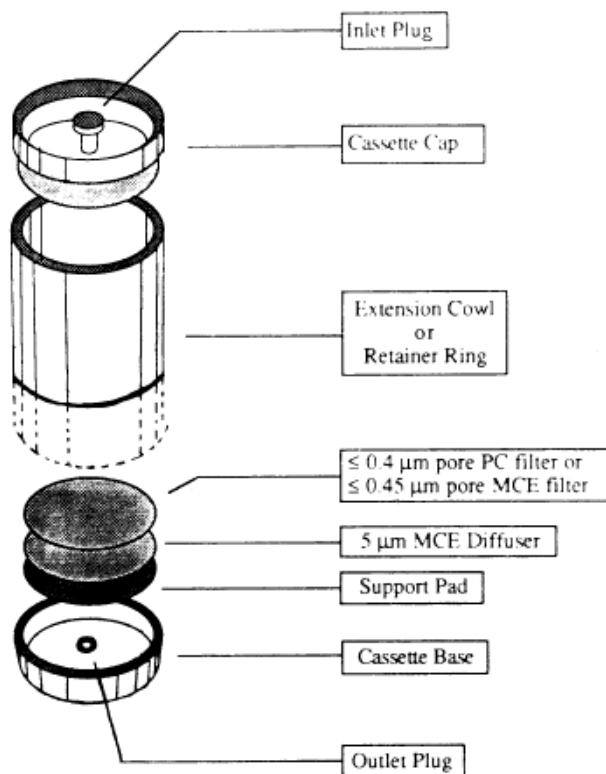
**Figures A-1 & A-2**

**Stationary Sampler Set-up**



A stationary air sample is collected by drawing a measured volume of air through a TEM filter cassette for a defined period of time. The TEM filter cassettes to be used should be mixed cellulose ester (MCE) membrane filters, 25 mm diameter in size and a pore size 0.45  $\mu\text{m}$ . Figure A-3 below provides a detailed breakdown of a sample TEM cassette.

**Figure A-3 TEM Sample Cassette Configuration**



The following sampling and associated equipment is needed in order to evaluate asbestos emissions along an unpaved serpentine covered roadway.

- 2 Fire Extinguishers (one to be placed on each side of the road)
- High Volume Pumps (Thomas Pump Model 007BDC19, or equivalent), include extra pumps in case of equipment failure
- Tripods for both Stationary and MET samplers (including extra in case of equipment failure)
- Meteorological Equipment (such as Gill UV sonic anemometers), including appropriate datalogger
- Thick-wall vinyl tubing (LT-4-10V vinyl tubing, or equivalent)
- Rotameters (Dwyer Rotameter #RMA 22-TMV with a range of 2-25 l/min, or equivalent)
- Tool Box
- Gel Cell Batteries

- Gel Cell Battery Chargers (including jumper cables, power strips)
- Universal Sampling Pump for Personal Samples (SKC Universal Sampling Pump Model #224-44XR, or equivalent)
- 12V Battery Chargers
- Primary Flow Meter with a range of approx. range 50 ml/min to 30 l/min (SKC DryCal Primary Flow Meter Model Cat.#717-05, or equivalent)
- Walky-talkies (Motorola walkabouts or equivalent)
- Measuring Tape
- Traffic Cones
- Landscape Flags
- Duct Tape
- Safety Vests
- Camcorder & digital Camera
- Field Logbooks, Chain of Custody Forms
- Boxes and tape for shipping samples
- Sample Custody Labels
- Sample Index Labels
- TEM Filter Cassettes
- Permanent Markers
- Zip Ties
- Zip Lock Bags (large & small)
- Personnel Protective Equipment (PPE)
- Test Vehicles

## **2.2 Air Sampler (Station) – Operating Procedures**

A summary of the air sampling operating procedures are provided below. Additional details of the sampling method are given in ISO 10312:1995(E).

### **Prior to Set-up**

- Fully charge sampler batteries.
- Determine sampler locations.
- Within the field logbook associate index IDs for each sample to be collected (that day).
- Based on traffic scenarios being tested and the sample locations associate target flowrates with each index ID (document the associated flowrates within the field logbook).
- Pre-label the sample index ID labels based on the field logbook information.

### **Set-up**

- Measure and mark-off test area of roadway with traffic cones.
- Set-up air sampler and MET stations.

- Test each sampling station for leaks. Plug inlet, turn on pump and check for flow indication at rotameter, should be zero or possibly maximum (rotameter ball has moved to the top of the column) due to high vacuum condition.
- Using a representative filter cassette (not the sample cassette) perform a pre-test calibration with a primary flowmeter. Adjust the flow rate with the rotameter according to the test plan (field logbook) and record the actual pretest flow rate and pump # in field Logbook.
- Attach the sample index ID label to TEM cassette and install the cassette with end cap assembly in place.

### **Sample Collection**

- At the start of the test run, sampling personnel shall apply power to all pumps as quickly as possible and record the start time.
- During the 2 hour test run (traffic scenario) the sampling personnel shall monitor the samplers for fluctuations in the flowrate by means of the rotameter and visible “kinks” in the tubing.
- At the conclusion of the traffic scenario the sampling personnel disconnect power to all pumps, record the time and replace the end caps on the filter cassettes.
- Once all sample pumps have been powered down each sample should be post calibrated. The end cap should be removed from the cassette and using the primary flowmeter perform an end of test calibration, record in the field logbook the end of test flow rate, and replace the end cap. The average of the pre and post flowrates should be used to calculate the sample volume.
- Remove the cassette from the sampler and attach a COC label over the capped ends (be sure not to cover the sample label) and place each cassette in individually sealed plastic bags.

### **Sample Analysis**

- Following collection all samples are to be shipped and maintained under chain-of-custody (COC) per sections 2.4 and 2.5 below.
- Stationary air samples are to be analyzed following approved TEM analytical methods, such as either ISO 10312 or AHERA

## **2.3 Air Sampler (Station) – Test Area and Location**

In order to best evaluate asbestos emissions along a selected unpaved serpentine covered roadway it is important to evaluate potential test areas against optimal site characteristics. The selected test area should (1) include a straight section (300-500 feet in length) of road, which will allow the test vehicles to reach and maintain the desired maximum test speed for at least 110 ft (33.5 m) on either side of the sampling transect (a total straight distance of 220 ft); and (2) the terrain should be level and lack any major obstructions (vegetation or

other physical obstacles) on either side of the road that would prevent sampling from being conducted (at least 200 ft to 300 ft from the road's edge).

Once a test area has been chosen the 220 ft test stretch should be measured and marked off with traffic cones. The MET Stations and sample stations are to be set-up at various distances along a transect that is approximately perpendicular to the center-point of the test stretch of road (on both sides of the road), see Figure A4 below. In addition, at least one "background" sampler should be located at a remote position from the roadway to attempt to characterize the background concentration for the area.

**Figure A-4. Air samplers, located at various distances from the road along a transect perpendicular to the road.**



It is important to collect the maximum volume of air possible within the 2 hour sampling period (during which a controlled traffic simulation takes place) without overloading the sample with particulate. The sample's particulate loading will vary with the sample flowrate, proximity to the roadway and traffic speed and frequency. Table A-1 below provides an overview of the target flowrates for each distance based on results and field observations from the sampling activities conducted in July 2002 (Pilot Study) along Slodusty Road in Garden Valley, CA. It is important to note that the flow rates indicated in Table A-1 were determined under fairly calm wind conditions (generally less than 8 mph). These flowrates are only a guide, site specific meteorological conditions and roadway

emissions may require the flow rate, total sampling time, and/or sampling volume to be modified. The decision to modify the flow rate, time, or volume will need to be made in the project manager or designate.

**Table A-1 Target Flow Rates (L/min) for Air Samplers**

<b>DISTANCE from Road (ft)</b>	<b>TRAFFIC SCENARIO Vehicle Speed (mph) / Vehicle Frequency (vph)</b>	
	<b>10 mph / 10 vph</b>	<b>25 mph / 30 vph</b>
<b>5</b>	8	4
<b>10</b>	10	6
<b>30</b>	12***	8
<b>50</b>	12***	10
<b>80</b>	12***	12***
<b>100</b>	12***	12***
<b>130</b>	12***	12***
<b>160</b>	12***	12***
<b>190</b>	12***	12***
<b>300</b>	12***	12***
<b>Background**</b>	12***	

Note: \*\* Background sample may be collected over 4 to 6 hours  
 \*\*\* Sampling pumps may have difficulty reaching and maintaining a flowrate of 12L/min. Based on Slodusty field activities the maximum flow rate that can consistently be obtained and maintained is 10L/min.

MET stations shall be set-up at a distance of 50 ft on each side of the road, in order to collect important meteorological parameters such as temperature, wind speed and direction.

## 2.4 Sample Documentation

Sampling activities are to be documented and maintained in a field logbook. The Field Team Leader is responsible for maintenance and document control of the field logbook.



Each air sample is to be identified with a unique code. The code is an index identification code. The index identification code is a sequential list of sample numbers that is to be used for all of the air samples collected. This coding system is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. To ensure that the laboratory does not receive certain specific information about a sample, only the index identification codes are to be used to label sample cassettes.

Each sample should include a two character prefix followed by a five digit sequential number. The first character of the prefix should represent the sampling event and the second character should represent the sampling day (or days).

Additionally, written documentation shall be supplemented with digital photographs and video footage during all traffic simulations.

## **2.5 Sample Custody, Packaging, & Shipping**

All air samples are to be classified and shipped as environmental samples. Chain-of-custody (COC) records are to be used as physical evidence of sample custody and control, and to provide the means to identify, track, and monitor each individual sample from the point of collection through shipment and final data reporting. The COC is also used to communicate analytical requirements to the laboratory, such as sample volume and method of analysis.

Shipping and COC procedures include:

- 1) Prior to sampling, a label is to be affixed to each air sample cassette. The label is indicate the sample index ID that corresponds to the index assigned to that particular sample in the field logbook
- 2) At the conclusion of the sampling period and end-flow measurement, the filter cassettes are to be removed from the sampler and tightly capped on both ends of the cassette.
- 3) A COC label is to be placed on each filter cassette such that it seals both ends of the cassette but does not obscure the sample label.
- 4) Each sealed filter cassette is to be individually stored in sealed plastic bags.
- 5) The individual filter cassette bags are then combined into a larger sealed plastic bag.
- 6) Bags of filter cassettes are to be packed carefully into a box such that the filter cassettes will not be jostled or damaged during shipping. No packing material that has the potential to contaminate the samples, such as vermiculite, should be used.
- 7) A signed copy of the COC record is to be placed inside the box with the samples and the box sealed.

- 8) Personnel samples need to be sent to the laboratory via overnight delivery for 24-hour turnaround; all other samples may be sent to the laboratory via standard shipping and turnaround analysis.

## 2.6 Quality Control Samples

Three types of field quality control (QC) samples are to be collected, lot blanks, field blanks, and sealed blanks. See Table A-2 below.

**Table A-2 Types of Field-Based Quality Control Samples**

Type of QC Sample	Description
<b>Field Blanks</b>	This is a filter cassette that is handled in the same manner as air samples, however no air is drawn through the cassette. Instead the caps are removed and the cassette is held open for approximately 30 seconds and then resealed. Field blanks are stored and shipped with sample cassettes.
<b>Lot Blanks</b>	This is an unused filter cassette taken from each new batch of cassettes. A batch may contain multiple boxes of cassettes (50 cassettes in one box). The filters are to be analyzed for asbestos fibers by the same method as will be used for the field samples.
<b>Sealed Blanks</b>	This is an unused filter cassette that is carried and shipped with each sample set. This representative cassette is NOT opened in the field. The filters are to be analyzed for asbestos fibers by the same method as will be used for the field samples.

The purpose of field blanks is to determine if any asbestos contamination has occurred during sample handling activities. For samples analyzed by AHERA the acceptable filter background level is 70 structures/mm<sup>2</sup>. The filter background level is the concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank. (TEM AHERA) The purpose of lot blanks is to verify that cassettes, as provided by the manufacturer, are “clean” of asbestos by determining the background asbestos structure concentration. The purpose of sealed blanks is determine if any asbestos contamination has occurred during sample storage and shipping.

AHERA requires for TEM analysis field blanks be collected at a frequency of two per I/O site (inside/outside) and sealed blanks at a frequency of one per I/O site.

### **SECTION 3. TEST PROCEDURES (TRAFFIC SIMULATIONS)**

Air samplers are to be installed on both sides of the road according to sections 2.1 and 2.3 above. The air samples are to be collected for 2 hour periods during defined traffic simulations (varying traffic frequency and speed). Ideally eight to nine field personnel are required to conduct each traffic simulation.

- Two, 2 person sampling teams (one team to set-up, operate, monitor, change out the samplers on each side of the road)
- 2 drivers (one for each test vehicle)
- 2 people to act as traffic controllers (one on each side of the test stretch of road to direct local traffic)
- 1 person to act as the traffic coordinator

Two different types of vehicles are to be utilized as test vehicles: a pick-up truck and a mid-size sedan. Each vehicle is to be alternately driven along the test stretch of road at regular intervals (based on the traffic scenario being tested) and needs to maintain the test speed for a distance of at least 110 ft on either side of the sampling transect (resulting in a total distance of 220 ft). A "traffic coordinator" will signal sampling personnel when to start the sampling pumps and then immediately begin controlling traffic flow via radio communication with the drivers. The desired test vehicle frequency is to be maintained as closely as possible. Local traffic is to be asked to either move very slowly (speed <10 mph) through the test site in the interim period between pass-bys of the test vehicles (local vehicle will not be counted) or to move through the test site at the test speed, thereby serving as a substitute for the test vehicle during that particular pass-by. Exact times of pass-bys, type of vehicle, target frequency and speed all need to be recorded within the field logbook, including information associated with local traffic (regardless whether the vehicle is counted or not). Public relations personnel or designates shall be positioned at both ends of the test stretch of road to serve as "traffic controllers". The traffic controllers shall stop and communicate with local drivers, informing them of the study, and to coordinate between local traffic and the traffic coordinator. At the conclusion of each traffic simulation the traffic coordinator will communicate with sampling personnel when to shut down the air samplers.

For the purposes of Slodusty Rd two traffic scenarios are to be tested, with vehicles traveling at 25 miles per hour (mph) at a frequency of 30 vehicles per hour (vph) and vehicles traveling at 10 mph at a frequency of 10 vph. The purpose of the 25mph/30vph scenario is to represent an extreme worst case traffic condition for the road. While the 10mph/10vph scenario is to represent actual speed and highest vehicle frequency conditions that may be encountered

along Slodusty Rd during peak commute. Note, the applicability of these traffic scenarios need to be re-evaluated for other roadways in regards to their intended purpose.

#### **SECTION 4. REFERENCES**

[1] CARB Standard Operating Procedure for Monitoring Asbestos in Ambient Air  
Air Monitoring North Section, March 1, 2001

[2] ISO 10312:1995(E) Ambient air-Determination of Asbestos fibres-Direct  
Transfer Transmission Electron Microscopy Method

[3] AHERA. 40 CFR – Chapter I – Part 763 – Appendix A to Subpart E of Part  
763 - Interim Transmission Electron Microscopy Analytical Methods – Mandatory  
and Nonmandatory – and Mandatory Section to Determine Completion of  
Response Actions (AHERA).

## **Appendix B – Sample Data**

# **SAMPLE DATA - INITIAL STUDY**

## **DTSC- Roadside Airborne Asbestos Monitoring Study, Initial Study**

NA= Not Applicable; nm= Not Measured

Index ID	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Date collected	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	Pump ID	Filter Diameter (mm)	Target Flow Rate (L/min)	Actual Start Flow Rate (L/min)	Actual Stop Flow Rate (L/min)	Total Volume collected (L air)	Estimated Actual Flow Rate (L/min)	Estimated Total Volume collected (L air)	Notes
PL-00001	LB	07/15/02	NA	NA	NA	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#9042032 (Environmental Express, 0.4u TEM Polycarbonate--this box had been opened earlier, and it belonged to DTSC); used only on Day 0
PL-00002	LB	07/15/02	NA	NA	NA	NA	NA	NA	47	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#H2HN15713 (Mini-Vol Filter so no cassette);used on Day 0
P0-00001	FB	07/15/02	NA	NA	00-B	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	field blank- Day 0 (0.4 u Polycarbonate filter)
P0-00016	P	07/15/02	NA	NA	NA	NA	NA		25	2	2	nm	350	NA	NA	Sedan Driver (DTSC personnel)
P0-00017	P	07/15/02	NA	NA	NA	NA	NA		25	2	2	nm	350	NA	NA	Traffic Controller (DTSC personnel)
P0-00002	F	07/15/02	E	5	00-01	30	25	V-05	25	6	nm	nm	nm	5.2	624	
P0-00003	F	07/15/02	E	10	00-01	30	25	V-01	25	8	nm	nm	nm	7.2	864	
P0-00004	F	07/15/02	E	30	00-01	30	25	V-02	25	10	nm	nm	nm	8.2	984	
P0-00005	F	07/15/02	E	30	00-01	30	25	Mini SN2664	47	5	nm	nm	nm	6	720	Mini-vol; used with an impactor; could not maintain flow rate (sample volume should not be considered valid)
P0-00006	F	07/15/02	E	50	00-01	30	25	V-14	25	15	nm	nm	nm	10.3	1,236	
P0-00007	F	07/15/02	E	80	00-01	30	25	V-06	25	15	nm	nm	nm	8.4	1,008	
P0-00008	F	07/15/02	E	100	00-01	30	25	DT-01	25	15	nm	nm	nm	11.5	1,380	
P0-00009	F	07/15/02	W	100	00-01	30	25	DT-02	25	15	nm	nm	nm	12	1,440	
P0-00010	F	07/15/02	W	80	00-01	30	25	V-12	25	15	nm	nm	nm	9.2	1,104	
P0-00011	F	07/15/02	W	30	00-01	30	25	V-08	25	10	nm	nm	nm	8.2	984	
P0-00012	F	07/15/02	W	30	00-01	30	25	Mini SN2888	47	5	nm	nm	nm	6	720	Mini-vol; used with an impactor
P0-00013	F	07/15/02	W	50	00-01	30	25	V-09	25	15	nm	nm	nm	10.1	1,212	
P0-00014	F	07/15/02	W	5	00-01	30	25	V-10	25	6	nm	nm	nm	5.4	648	
P0-00015	F	07/15/02	W	10	00-01	30	25	V-07	25	8	nm	nm	nm	7.5	900	
PL-00003	LB	07/16/02	NA	NA	NA	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#410FKA, Production Code 2135 (0.45 MCE Filters); used Day 1
PL-00004	LB	07/16/02	NA	NA	NA	NA	NA	NA	47	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#H2BN10114-3499 (Omega Cassettes); used for Day 1
P1-00001	FB	07/16/02	NA	NA	01-B	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	field blank- Day 1 (0.45u MCE filter)
P1-00060	P	07/16/02	NA	NA	NA	NA	NA		25	2	1.99	nm	746	NA	NA	In Pick-up Truck
P1-00061	P	07/16/02	NA	NA	NA	NA	NA		25	2	2.066	nm	775	NA	NA	Field Personnel (Volpe personnel)
P1-00062	P	07/16/02	NA	NA	NA	NA	NA		25	2	1.992	nm	745	NA	NA	Traffic Controller (DTSC personnel)
P1-00002	F	07/16/02	E	5	01-01	10	25	V-04	25	6	nm	nm	nm	5.7	684	
P1-00003	F	07/16/02	E	5	01-01	10	25	V-03	25	8	nm	nm	nm	7.4	888	
P1-00004	F	07/16/02	E	5	01-01	10	25	V-10	47	8	nm	nm	nm	6.8	816	
P1-00005	F	07/16/02	E	10	01-01	10	25	V-15	25	8	nm	nm	nm	7.5	900	
P1-00006	F	07/16/02	E	10	01-01	10	25	V-09	25	10	nm	nm	nm	7.9	948	
P1-00007	F	07/16/02	E	10	01-01	10	25	V-16	47	8	nm	nm	nm	7	840	
P1-00008	F	07/16/02	E	30	01-01	10	25	V-07	25	10	nm	nm	nm	8.9	1,068	
P1-00009	F	07/16/02	E	30	01-01	10	25	V-08	25	12	nm	nm	nm	8.9	1,068	
P1-00010	F	07/16/02	E	30	01-01	10	25	V-12	47	10	nm	nm	nm	8.4	1,008	
P1-00011	F	07/16/02	E	50	01-01	10	25	DT-08	25	15	nm	nm	nm	11.5	1,380	
P1-00012	F	07/16/02	E	80	01-01	10	25	DT-09	25	15	nm	nm	nm	10.9	1,308	
P1-00013	F	07/16/02	E	100	01-01	10	25	DT-10	25	15	nm	nm	nm	10.9	1,308	
P1-00014	F	07/16/02	W	5	01-01	10	25	V-01	25	6	nm	nm	nm	5.7	684	
P1-00015	F	07/16/02	W	5	01-01	10	25	V-14	25	8	nm	nm	nm	6.6	792	
P1-00016	F	07/16/02	W	5	01-01	10	25	V-02	47	8	nm	nm	nm	7	840	
P1-00017	F	07/16/02	W	10	01-01	10	25	DT-07	25	8	nm	nm	nm	7.3	876	
P1-00018	F	07/16/02	W	10	01-01	10	25	V-13	25	10	nm	nm	nm	8.2	984	
P1-00019	F	07/16/02	W	10	01-01	10	25	V-11	47	8	nm	nm	nm	6.9	828	
P1-00020	F	07/16/02	W	30	01-01	10	25	DT-04	25	10	nm	nm	nm	8.2	984	
P1-00021	F	07/16/02	W	30	01-01	10	25	DT-06	25	12	nm	nm	nm	9.5	1,140	
P1-00022	F	07/16/02	W	30	01-01	10	25	DT-05	47	10	nm	nm	nm	8.2	984	

**SAMPLE DATA - INITIAL STUDY**  
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P1-00023	F	07/16/02	W	50	01-01	10	25	V-05	25	15	nm	nm	nm	8.5	1,020	
P1-00024	F	07/16/02	W	80	01-01	10	25	DT-02	25	15	nm	nm	nm	12	1,440	
P1-00025	F	07/16/02	W	100	01-01	10	25	DT-01	25	15	nm	nm	nm	11.6	1,392	
P1-00026	F	07/16/02	E	5	01-02	30	25	V-04	25	6	nm	nm	nm	5.7	684	
P1-00027	F	07/16/02	E	5	01-02	30	25	V-03	25	8	nm	nm	nm	7.4	888	
P1-00028	F	07/16/02	E	5	01-02	30	25	V-10	47	8	nm	nm	nm	6.8	816	
P1-00029	F	07/16/02	E	10	01-02	30	25	V-15	25	8	nm	nm	nm	7.5	900	
P1-00030	F	07/16/02	E	10	01-02	30	25	V-09	25	10	nm	nm	nm	7.9	948	
P1-00031	F	07/16/02	E	10	01-02	30	25	V-16	47	8	nm	nm	nm	7	840	
P1-00032	F	07/16/02	E	30	01-02	30	25	V-07	25	10	nm	nm	nm	8.9	1,068	
P1-00033	F	07/16/02	E	30	01-02	30	25	V-08	25	12	nm	nm	nm	8.9	1,068	
P1-00034	F	07/16/02	E	30	01-02	30	25	V-12	47	10	nm	nm	nm	8.4	1,008	
P1-00035	F	07/16/02	E	50	01-02	30	25	DT-08	25	15	nm	nm	nm	11.5	1,380	
P1-00036	F	07/16/02	E	80	01-02	30	25	DT-09	25	15	nm	nm	nm	10.9	1,308	
P1-00037	F	07/16/02	E	100	01-02	30	25	DT-10	25	15	nm	nm	nm	10.9	1,308	
P1-00038	F	07/16/02	W	5	01-02	30	25	V-01	25	6	nm	nm	nm	5.7	684	
P1-00039	F	07/16/02	W	5	01-02	30	25	V-14	25	8	nm	nm	nm	6.6	792	
P1-00040	F	07/16/02	W	5	01-02	30	25	V-02	47	8	nm	nm	nm	7	840	
P1-00041	F	07/16/02	W	10	01-02	30	25	DT-07	25	8	nm	nm	nm	7.3	876	
P1-00042	F	07/16/02	W	10	01-02	30	25	V-13	25	10	nm	nm	nm	8.2	984	
P1-00043	F	07/16/02	W	10	01-02	30	25	V-11	47	8	nm	nm	nm	6.9	828	
P1-00044	F	07/16/02	W	30	01-02	30	25	DT-04	25	10	nm	nm	nm	8.2	984	
P1-00045	F	07/16/02	W	30	01-02	30	25	DT-06	25	12	nm	nm	nm	9.5	1,140	
P1-00046	F	07/16/02	W	30	01-02	30	25	DT-05	47	10	nm	nm	nm	8.2	984	
P1-00047	F	07/16/02	W	50	01-02	30	25	V-05	25	15	nm	nm	nm	8.5	1,020	
P1-00048	F	07/16/02	W	80	01-02	30	25	DT-02	25	15	nm	nm	nm	12	1,440	
P1-00049	F	07/16/02	W	100	01-02	30	25	DT-01	25	15	nm	nm	nm	11.6	1,392	
PL-00005	LB	07/17/02	NA	NA	NA	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#410FKA, Production Code 2135 (0.45 MCE Filters); used Day 2
PL-00006	LB	07/17/02	NA	NA	NA	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#410FKA, Production Code 2135 (0.45 MCE Filters); used Day 2
P2-00001	FB	07/17/02	NA	NA	02-B	NA	NA	NA	47	NA	NA	NA	NA	NA	NA	field blank- Day 2 (0.45u MCE filter)
P2-00002	P	07/17/02	NA	NA	NA	NA	NA		25	2	2.06	2.108	752	NA	NA	Field Personnel (DTSC personnel)
P2-00003	P	07/17/02	NA	NA	NA	NA	NA		25	2	1.995	2.045	728	NA	NA	Traffic Controller (DTSC personnel)
P2-00004	P	07/17/02	NA	NA	NA	NA	NA		25	2	2.013	2.249	721	NA	NA	Field Personnel (Volpe personnel)
P2-00005	F	07/17/02	E	5	02-01	30	25	V-10	25	2	1.1	1.25	141	NA	NA	
P2-00006	F	07/17/02	E	5	02-01	30	25	V-05	25	4	3.7	3.7	444	NA	NA	
P2-00007	F	07/17/02	E	10	02-01	30	25	V-14	25	4	4.2	3.95	489	NA	NA	
P2-00008	F	07/17/02	E	10	02-01	30	25	V-11	25	6	5.4	5.9	678	NA	NA	
P2-00009	F	07/17/02	E	30	02-01	30	25	V-01	25	6	5.7	6.2	714	NA	NA	
P2-00010	F	07/17/02	E	30	02-01	30	25	V-09	25	8	6.8	7	828	NA	NA	
P2-00011	F	07/17/02	E	50	02-01	30	25	DT-04	25	10	8.2	8.6	1008	NA	NA	
P2-00012	F	07/17/02	E	80	02-01	30	25	V-06	25	10	8	7.6	936	NA	NA	
P2-00013	F	07/17/02	E	100	02-01	30	25	DT-05	25	10	8.1	8.4	990	NA	NA	
P2-00014	F	07/17/02	E	130	02-01	30	25	DT-06	25	12	9.6	9.71	1159	NA	NA	
P2-00015	F	07/17/02	E	160	02-01	30	25	DT-09	25	12	9.6	9.5	1146	NA	NA	
P2-00016	F	07/17/02	E	190	02-01	30	25	DT-10	25	12	9.7	9.5	1152	NA	NA	
P2-00017	F	07/17/02	W	5	02-01	30	25	V-03	25	2	1.3	1.1	144	NA	NA	
P2-00018	F	07/17/02	W	5	02-01	30	25	V-13	25	4	4.1	4.1	492	NA	NA	
P2-00019	F	07/17/02	W	10	02-01	30	25	V-16	25	4	3.8	3.95	465	NA	NA	
P2-00020	F	07/17/02	W	10	02-01	30	25	V-04	25	6	5.7	5.7	684	NA	NA	
P2-00021	F	07/17/02	W	30	02-01	30	25	V-08	25	6	5.6	5.9	690	NA	NA	

# **SAMPLE DATA - INITIAL STUDY**

## **DTSC- Roadside Airborne Asbestos Monitoring Study, Initial Study**

NA= Not Applicable; nm= Not Measured

Index ID	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Date collected	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	Pump ID	Filter Diameter (mm)	Target Flow Rate (L/min)	Actual Start Flow Rate (L/min)	Actual Stop Flow Rate (L/min)	Total Volume collected (L air)	Estimated Actual Flow Rate (L/min)	Estimated Total Volume collected (L air)	Notes
P2-00022	F	07/17/02	W	30	02-01	30	25	V-15	25	8	7.2	7.7	894	NA	NA	
P2-00023	F	07/17/02	W	50	02-01	30	25	V-07	25	10	8.8	9.2	1080	NA	NA	
P2-00024	F	07/17/02	W	80	02-01	30	25	DT-07	25	10	8.8	8.8	1056	NA	NA	
P2-00025	F	07/17/02	W	100	02-01	30	25	DT-03	25	10	8.8	9.1	1074	NA	NA	
P2-00026	F	07/17/02	W	130	02-01	30	25	DT-02	25	12	10	9.4	1164	NA	NA	
P2-00027	F	07/17/02	W	160	02-01	30	25	DT-01	25	12	10.1	10.2	1218	NA	NA	
P2-00028	F	07/17/02	W	190	02-01	30	25	DT-08	25	12	10.1	9.3	1164	NA	NA	
P2-00029	F	07/17/02	E	5	02-02	30	25	V-10	25	2	1.94	1.8	224	NA	NA	
P2-00030	F	07/17/02	E	5	02-02	30	25	V-05	25	4	3.96	4.2	490	NA	NA	
P2-00031	F	07/17/02	E	10	02-02	30	25	V-14	25	4	3.9	4.5	504	NA	NA	
P2-00032	F	07/17/02	E	10	02-02	30	25	V-11	25	6	6.1	6.35	747	NA	NA	
P2-00033	F	07/17/02	E	30	02-02	30	25	V-01	25	6	5.97	6.06	722	NA	NA	
P2-00034	F	07/17/02	E	30	02-02	30	25	V-09	25	8	8.1	7.8	954	NA	NA	
P2-00035	F	07/17/02	E	50	02-02	30	25	DT-04	25	10	9.9	9.7	1176	NA	NA	
P2-00036	F	07/17/02	E	80	02-02	30	25	V-06	25	10	9.9	10.1	1200	NA	NA	
P2-00037	F	07/17/02	E	100	02-02	30	25	DT-05	25	10	9.9	9.1	1140	NA	NA	
P2-00038	F	07/17/02	E	130	02-02	30	25	DT-06	25	12	11	11	1320	NA	NA	
P2-00039	F	07/17/02	E	160	02-02	30	25	DT-09	25	12	11.3	11.7	1380	NA	NA	
P2-00040	F	07/17/02	E	190	02-02	30	25	DT-10	25	12	12	12.5	1470	NA	NA	
P2-00041	F	07/17/02	W	5	02-02	30	25	V-03	25	2	1.9	1.8	222	NA	NA	
P2-00042	F	07/17/02	W	5	02-02	30	25	V-13	25	4	4	4.2	492	NA	NA	
P2-00043	F	07/17/02	W	10	02-02	30	25	V-16	25	4	4	4.2	492	NA	NA	
P2-00044	F	07/17/02	W	10	02-02	30	25	V-04	25	6	6	5.8	708	NA	NA	
P2-00045	F	07/17/02	W	30	02-02	30	25	V-08	25	6	6	5.7	702	NA	NA	
P2-00046	F	07/17/02	W	30	02-02	30	25	V-15	25	8	8	7.6	936	NA	NA	
P2-00047	F	07/17/02	W	50	02-02	30	25	V-07	25	10	10.1	9.9	1200	NA	NA	
P2-00048	F	07/17/02	W	80	02-02	30	25	DT-07	25	10	10.1	10.8	1254	NA	NA	
P2-00049	F	07/17/02	W	100	02-02	30	25	DT-03	25	10	10	9.8	1188	NA	NA	
P2-00050	F	07/17/02	W	130	02-02	30	25	DT-02	25	12	11.9	12.2	1446	NA	NA	
P2-00051	F	07/17/02	W	160	02-02	30	25	DT-01	25	12	12	11.7	1422	NA	NA	
P2-00052	F	07/17/02	W	190	02-02	30	25	DT-08	25	12	10.8	10.7	1290	NA	NA	during sampling, pump had crimp in line
P3-00001	FB	07/18/02	NA	NA	03-B	NA	NA	NA	25	NA	NA	NA	NA	NA	NA	field blank- Day 3 (0.45u MCE filter); no asbestos detected during analysis
P3-00002	P	07/18/02	NA	NA	NA	NA	NA		25	2	2.005	2.133	299	NA	NA	Field Personnel (Volpe personnel)
P3-00003	P	07/18/02	NA	NA	NA	NA	NA		25	2	2.012	2.045	338	NA	NA	Field Personnel (DTSC personnel)
P3-00004	P	07/18/02	NA	NA	NA	NA	NA		25	2	2.027	2.091	314	NA	NA	Field Personnel (Volpe personnel)
P3-00005	F	07/18/02	E	5	03-01	10	10	DT-02	25	8	7.9	8.4	978	NA	NA	
P3-00006	F	07/18/02	E	10	03-01	10	10	DT-03	25	10	10	10.1	1206	NA	NA	
P3-00007	F	07/18/02	E	30	03-01	10	10	DT-10	25	12	10.5	2.6	786	NA	NA	during field sampling: tube collapsing under vacuum, need surgical tube -- thus produced low stop flow rate and low total volume
P3-00008	F	07/18/02	E	80	03-01	10	10	DT-08	25	12	12	11.6	1416	NA	NA	
P3-00009	F	07/18/02	E	130	03-01	10	10	DT-05	25	12	11.3	12.5	1428	NA	NA	
P3-00010	F	07/18/02	W	5	03-01	10	10	DT-04	25	8	7.9	7.5	924	NA	NA	
P3-00011	F	07/18/02	W	10	03-01	10	10	DT-10	25	10	10	9.7	1182	NA	NA	
P3-00012	F	07/18/02	W	30	03-01	10	10	DT-07	25	12	10.85	11.2	1323	NA	NA	
P3-00013	F	07/18/02	W	80	03-01	10	10	DT-09	25	12	9.9	12.3	1332	NA	NA	
P3-00014	F	07/18/02	W	130	03-01	10	10	DT-06	25	12	10.2	11	1272	NA	NA	

141 Total Samples Collected



# SAMPLE DATA - POST RESURFACING

## DTSC- Roadside Airborne Asbestos Monitoring Study, Post Resurfacing

NA= Not Applicable; NM= Not Measured

Index ID	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Date collected	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	Pump ID	Filter Diameter (mm)	Target Flow Rate (L/min)	Actual Start Flow Rate (L/min)	Actual Stop Flow Rate (L/min)	Estimated or Average Flow Rate (L/min)	Start Time	Stop Time	Duration (min), Personals based on Counter	Total Volume (L)	Notes
F1-00021	FB	08/18/03	NA	NA				NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	Sample Voided - Not Taken
F1-00022	FB	08/18/03	NA	NA				NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	Sample Voided - Not Taken
F1-00023	LB	08/18/03	NA	NA				NA	25	NA	NA	NA	NA	NA	NA	NA	NA	Lot Blank: Lot#H3EN2781 (0.45 MEC Filters); used for Day 1,2,3
F1-00024	P	08/18/03	NA	NA	NA	NA	NA		25	2	2	2	2.00	11:30:00 AM	3:01:00 PM	211	422	Task = Setup & Observer (DTSC personnel) - No Simulation Run
F1-00025	P	08/18/03	NA	NA	NA	NA	NA		25	2	2	2	2.00	11:32:00 AM	3:00:00 PM	207	414	Task = Setup & Observer (DTSC personnel) - No Simulation Run
F1-00001	F	08/18/03	E	5	NA	NA	NA	V-11	25	8	7.7	7.6	7.67	1:02:00 PM	3:00:00 PM	118	905	No Simulation Run
F1-00002	F	08/18/03	E	10	NA	NA	NA	V-06	25	9	7.5	7.4	7.47	1:01:30 PM	2:59:45 PM	118	881	No Simulation Run
F1-00003	F	08/18/03	E	30	NA	NA	NA	V-03	25	9	7.5	7.4	7.47	1:01:00 PM	2:59:30 PM	118	881	No Simulation Run
F1-00004	F	08/18/03	E	50	NA	NA	NA	V-16	25	12	8.0	7.9	7.97	1:00:30 PM	2:59:15 PM	118	940	No Simulation Run
F1-00005	F	08/18/03	E	80	NA	NA	NA	DT-08	25	12	9.7	9.6	9.67	1:00:00 PM	2:59:00 PM	119	1,151	No Simulation Run
F1-00006	F	08/18/03	E	100	NA	NA	NA	DT-05	25	12	12.2	12.1	12.17	12:59:30 PM	2:58:45 PM	119	1,448	No Simulation Run
F1-00007	F	08/18/03	E	130	NA	NA	NA	DT-03	25	12	8.4	8.3	8.37	12:59:00 PM	2:58:30 PM	119	996	No Simulation Run
F1-00008	F	08/18/03	E	160	NA	NA	NA	DT-09	25	12	9.6	9.5	9.57	12:58:30 PM	2:58:15 PM	119	1,139	No Simulation Run
F1-00009	F	08/18/03	E	190	NA	NA	NA	DT-20	25	12	9.3	9.2	9.27	12:58:00 PM	2:58:00 PM	120	1,112	No Simulation Run
F1-00010	F	08/18/03	W	5	NA	NA	NA	V-09	25	8	8.2	8.1	8.17	1:00:00 PM	3:00:00 PM	120	980	No Simulation Run
F1-00011	F	08/18/03	W	10	NA	NA	NA	V-08	25	9	7.5	7.4	7.47	12:59:51 PM	2:59:51 PM	120	896	No Simulation Run
F1-00012	F	08/18/03	W	30	NA	NA	NA	V-13	25	9	8.5	8.4	8.47	12:59:38 PM	2:59:38 PM	120	1,016	No Simulation Run
F1-00013	F	08/18/03	W	50	NA	NA	NA	V-10	25	12	7.6	7.5	7.57	12:59:25 PM	2:59:25 PM	120	908	No Simulation Run
F1-00014	F	08/18/03	W	80	NA	NA	NA	DT-10	25	12	9.1	9.0	9.07	12:59:05 PM	2:59:05 PM	120	1,088	No Simulation Run
F1-00015	F	08/18/03	W	100	NA	NA	NA	DT-04	25	12	10.2	10.1	10.17	12:58:52 PM	2:58:52 PM	120	1,220	No Simulation Run
F1-00016	F	08/18/03	W	130	NA	NA	NA	DT-06	25	12	9.6	9.5	9.57	12:58:39 PM	2:58:39 PM	120	1,148	No Simulation Run
F1-00017	F	08/18/03	W	160	NA	NA	NA	DT-07	25	12	10.1	10.0	10.07	12:58:26 PM	2:58:26 PM	120	1,208	No Simulation Run
F1-00018	F	08/18/03	W	190	NA	NA	NA	DT-30	25	12	11.0	10.9	10.97	12:58:13 PM	2:58:13 PM	120	1,316	No Simulation Run
F1-00019	F	08/18/03	W	300	NA	NA	NA	DT-01	25	12	9.7	9.6	9.67	12:58:00 PM	2:58:00 PM	120	1,160	300' - No Simulation Run
F1-00020	F	08/18/03	NA	NA	NA	NA	NA	V-05	25	12	7.3	7.2	7.27	1:36:00 PM	5:03:00 PM	207	1,505	Background sample taken at Residence (350 Bayleaf Drive, owner = Vance Fellows)
F2-00019	LB	08/19/03	NA	NA				NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	Sample Voided - Not Taken
F2-00020	FB	08/19/03	NA	NA				NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	Sample Voided - Not Taken
F2-00021	FB	08/19/03	NA	NA				NA	25	NA	NA	NA	NA	NA	NA	NA	NA	
F2-00042	P	08/19/03	NA	NA	NA	NA	NA		25	2	2	2	2.00			31	62	Task = Sampler (Volpe personnel), battery failure at 31min
F2-00043	P	08/19/03	NA	NA	NA	NA	NA		25	2	2	2	2.00			69	138	Task = Sedan Driver (DTSC personnel), battery failure at 69min
F2-00044	P	08/19/03	NA	NA	NA	NA	NA		25	2	2	2	2.00			300	600	Task = Setup & Observer (DTSC personnel)
F2-00001	F	08/19/03	E	5	2-01	10	10	V-09	25	8	7.6	7.3	7.45	10:53:00 AM	12:52:00 PM	119	887	
F2-00002	F	08/19/03	E	10	2-01	10	10	V-11	25	10	8.1	8.0	8.05	10:52:38 AM	12:51:45 PM	119	958	
F2-00003	F	08/19/03	E	30	2-01	10	10	V-08	25	12	8.3	7.6	7.95	10:52:15 AM	12:51:30 PM	119	946	
F2-00004	F	08/19/03	E	50	2-01	10	10	V-03	25	12	7.8	7.7	7.75	10:51:53 AM	12:51:15 PM	119	922	
F2-00005	F	08/19/03	E	80	2-01	10	10	DT-08	25	12	9.6	9.5	9.55	10:51:30 AM	12:51:00 PM	119	1,136	
F2-00006	F	08/19/03	E	100	2-01	10	10	DT-10	25	12	9.8	9.7	9.75	10:51:08 AM	12:50:45 PM	119	1,160	
F2-00007	F	08/19/03	E	130	2-01	10	10	DT-05	25	12	10.5	9.7	10.10	10:50:45 AM	12:50:30 PM	119	1,202	
F2-00008	F	08/19/03	E	160	2-01	10	10	DT-30	25	12	13.7	9.9	11.80	10:50:22 AM	12:50:15 PM	119	1,404	
F2-00009	F	08/19/03	E	190	2-01	10	10	DT-09	25	12	10.2	9.9	10.05	10:50:00 AM	12:50:00 PM	120	1,206	
F2-00010	F	08/19/03	W	5	2-01	10	10	V-13	25	8	8.2	7.9	8.05	10:53:00 AM	12:52:00 PM	119	958	
F2-00011	F	08/19/03	W	10	2-01	10	10	V-10	25	10	7.9	7.7	7.80	10:52:50 AM	12:51:44 PM	118	920	
F2-00012	F	08/19/03	W	30	2-01	10	10	V-06	25	12	8.1	7.8	7.95	10:52:35 AM	12:51:31 PM	118	938	
F2-00013	F	08/19/03	W	50	2-01	10	10	V-16	25	12	7.6	7.3	7.45	10:52:25 AM	12:51:18 PM	118	879	
F2-00014	F	08/19/03	W	80	2-01	10	10	DT-07	25	12	9.6	9.4	9.50	10:52:05 AM	12:51:05 PM	119	1,131	

**SAMPLE DATA - POST RESURFACING**

**DTSC- Roadside Airborne Asbestos Monitoring Study, Post Resurfacing**

NA= Not Applicable; NM= Not Measured

Index ID	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Date collected	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	Pump ID	Filter Diameter (mm)	Target Flow Rate (L/min)	Actual Start Flow Rate (L/min)	Actual Stop Flow Rate (L/min)	Estimated or Average Flow Rate (L/min)	Start Time	Stop Time	Duration (min), Personals based on Counter	Total Volume (L)	Notes
F2-00015	F	08/19/03	W	100	2-01	10	10	DT-03	25	12	9.0	9.4	9.20	10:51:50 AM	12:50:52 PM	119	1,095	
F2-00016	F	08/19/03	W	130	2-01	10	10	DT-04	25	12	9.4	21.3	15.35	10:51:25 AM	12:50:39 PM	119	1,827	
F2-00017	F	08/19/03	W	160	2-01	10	10	DT-20	25	12	9.5	8.6	9.05	10:51:05 AM	12:50:26 PM	119	1,077	
F2-00018	F	08/19/03	W	190	2-01	10	10	DT-01	25	12	9.5	9.3	9.40	10:50:50 AM	12:50:13 PM	119	1,119	
F2-00040	F	08/19/03	W	300	2-01	10	10	DT-06	25	12	9.5	9.3	9.40	10:50:00 AM	12:50:00 PM	120	1,128	
F2-00022	F	08/19/03	E	5	2-02	30	25	V-09	25	4	3.9	3.8	3.85	1:57:00 PM	3:57:00 PM	120	462	
F2-00023	F	08/19/03	E	10	2-02	30	25	V-11	25	6	6.1	6.4	6.25	1:56:45 PM	3:56:45 PM	120	750	
F2-00024	F	08/19/03	E	30	2-02	30	25	V-08	25	8	7.8	7.4	7.60	1:56:30 PM	3:56:30 PM	120	912	
F2-00025	F	08/19/03	E	50	2-02	30	25	V-03	25	10	7.5	7.5	7.50	1:56:15 PM	3:56:15 PM	120	900	
F2-00026	F	08/19/03	E	80	2-02	30	25	DT-08	25	12	9.8	9.7	9.75	1:56:00 PM	3:56:00 PM	120	1,170	
F2-00027	F	08/19/03	E	100	2-02	30	25	DT-10	25	12	9.6	9.7	9.65	1:55:45 PM	3:55:45 PM	120	1,158	
F2-00028	F	08/19/03	E	130	2-02	30	25	DT-05	25	12	9.7	9.4	9.55	1:55:30 PM	3:55:30 PM	120	1,146	
F2-00029	F	08/19/03	E	160	2-02	30	25	DT-30	25	12	9.7	9.5	9.60	1:55:15 PM	3:55:15 PM	120	1,152	
F2-00030	F	08/19/03	E	190	2-02	30	25	DT-09	25	12	9.6	9.2	9.40	1:55:00 PM	3:55:00 PM	120	1,128	
F2-00031	F	08/19/03	W	5	2-02	30	25	V-13	25 25	4	4.1	4.1	4.10	1:57:00 PM	3:57:00 PM	120	492	
F2-00032	F	08/19/03	W	10	2-02	30	25	V-10	25	6	6.2	9.8	8.00	1:56:44 PM	3:56:44 PM	120	960	
F2-00033	F	08/19/03	W	30	2-02	30	25	V-06	25	8	7.9	8.1	8.00	1:56:31 PM	3:56:31 PM	120	960	
F2-00034	F	08/19/03	W	50	2-02	30	25	V-16	25	10	7.7	7.7	7.70	1:56:18 PM	3:56:18 PM	120	924	
F2-00035	F	08/19/03	W	80	2-02	30	25	DT-07	25	12	9.0	20.8	14.90	1:56:05 PM	3:56:05 PM	120	1,788	FILTER RUPTURED COULDN'T BE PREPPED (ACCORDING TO LAB, RESI)
F2-00036	F	08/19/03	W	100	2-02	30	25	DT-03	25	12	9.6	9.5	9.55	1:55:52 PM	3:55:52 PM	120	1,146	
F2-00037	F	08/19/03	W	130	2-02	30	25	DT-04	25	12	9.0	8.9	8.95	1:55:39 PM	3:55:39 PM	120	1,074	
F2-00038	F	08/19/03	W	160	2-02	30	25	DT-20	25	12	9.9	10.0	9.95	1:55:26 PM	3:55:26 PM	120	1,194	
F2-00039	F	08/19/03	W	190	2-02	30	25	DT-01	25	12	9.3	9.3	9.30	1:55:13 PM	3:55:13 PM	120	1,116	
F2-00041	F	08/19/03	W	300	2-02	30	25	DT-06	25	12	9.6	9.7	9.65	1:55:00 PM	3:55:00 PM	120	1,158	
F3-00042	FB	08/20/03	NA	NA				NA	25	NA	NA	NA	NA	NA	NA	NA	NA	
F3-00043	FB	08/20/03	NA	NA				NA	25	NA	NA	NA	NA	NA	NA	NA	NA	
F3-00001	P	08/20/03	NA	NA				3716	25	2	2	2	2.00	8:50:00 AM	2:50:00 PM	360	720	Task = Setup & Observer (DTSC personnel)
F3-00002	P	08/20/03	NA	NA				3746	25	2	2	2	2.00	8:50:00 AM	2:55:00 PM	365	730	Task = Sampler (Volpe personnel)
F3-00003	P	08/20/03	NA	NA				3706	25	2	2	2	2.00	8:50:00 AM	2:45:00 PM	355	710	Task = Truck Driver (DTSC personnel)
F3-00004	F	08/20/03	E	5	3-01	10	10	V-10	25	8	7.8	7.9	7.85	10:26:45 AM	12:25:40 PM	118	926	
F3-00005	F	08/20/03	E	10	3-01	10	10	V-06	25	10	9.9	8.0	8.95	10:26:31 AM	12:25:35 PM	119	1,065	
F3-00006	F	08/20/03	E	30	3-01	10	10	V-08	25	12	8.9	8.3	8.60	10:26:18 AM	12:25:30 PM	119	1,023	
F3-00007	F	08/20/03	E	50	3-01	10	10	V-15	25	12	7.6	7.6	7.60	10:26:05 AM	12:25:25 PM	119	904	
F3-00008	F	08/20/03	E	80	3-01	10	10	DT-03	25	12	9.4	9.3	9.35	10:25:52 AM	12:25:20 PM	119	1,113	
F3-00009	F	08/20/03	E	100	3-01	10	10	DT-09	25	12	9.5	9.3	9.40	10:25:39 AM	12:25:15 PM	119	1,119	
F3-00010	F	08/20/03	E	130	3-01	10	10	DT-30	25	12	9.5	9.2	9.35	10:25:26 AM	12:25:10 PM	119	1,113	
F3-00011	F	08/20/03	E	160	3-01	10	10	DT-06	25	12	10.2	10.1	10.15	10:25:13 AM	12:25:05 PM	119	1,208	
F3-00012	F	08/20/03	E	190	3-01	10	10	DT-07	25	12	9.8	10.8	10.30	10:25:00 AM	12:25:00 PM	120	1,236	
F3-00013	F	08/20/03	W	5	3-01	10	10	V-09	25	8	8.0	6.7	7.35	10:26:45 AM	12:25:40 PM	118	867	
F3-00014	F	08/20/03	W	10	3-01	10	10	V-13	25	10	7.6	7.9	7.75	10:26:26 AM	12:25:36 PM	119	922	
F3-00015	F	08/20/03	W	30	3-01	10	10	V-16	25	12	7.3	7.4	7.35	10:26:14 AM	12:25:32 PM	119	875	
F3-00016	F	08/20/03	W	50	3-01	10	10	V-11	25	12	7.5	7.7	7.60	10:26:02 AM	12:25:28 PM	119	904	
F3-00017	F	08/20/03	W	80	3-01	10	10	DT-04	25	12	9.8	9.8	9.80	10:25:50 AM	12:25:24 PM	119	1,166	
F3-00018	F	08/20/03	W	100	3-01	10	10	DT-20	25	12	8.7	8.4	8.55	10:25:48 AM	12:25:20 PM	119	1,017	
F3-00019	F	08/20/03	W	130	3-01	10	10	DT-08	25	12	9.3	8.9	9.10	10:25:36 AM	12:25:16 PM	119	1,083	
F3-00020	F	08/20/03	W	160	3-01	10	10	DT-05	25	12	9.6	9.4	9.50	10:25:24 AM	12:25:12 PM	119	1,131	
F3-00021	F	08/20/03	W	190	3-01	10	10	DT-10	25	12	9.0	8.9	8.95	10:25:12 AM	12:25:04 PM	119	1,065	
F3-00022	F	08/20/03	W	300	3-01	10	10	DT-01	25	12	9.6	9.3	9.45	10:25:00 AM	12:25:00 PM	120	1,134	
F3-00023	F	08/20/03	E	5	3-02	30	25	V-10	25	4	4.1	4.1	4.10	1:41:54 PM	3:40:40 PM	118	484	

**SAMPLE DATA - POST RESURFACING**

**DTSC- Roadside Airborne Asbestos Monitoring Study, Post Resurfacing**

NA= Not Applicable; NM= Not Measured

Index ID	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Date collected	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	Pump ID	Filter Diameter (mm)	Target Flow Rate (L/min)	Actual Start Flow Rate (L/min)	Actual Stop Flow Rate (L/min)	Estimated or Average Flow Rate (L/min)	Start Time	Stop Time	Duration (min), Personals based on Counter	Total Volume (L)	Notes
F3-00024	F	08/20/03	E	10	3-02	30	25	V-06	25	6	5.9	5.9	5.90	1:41:38 PM	3:40:35 PM	118	696	
F3-00025	F	08/20/03	E	30	3-02	30	25	V-08	25	8	8.2	7.9	8.05	1:41:24 PM	3:40:30 PM	119	958	
F3-00026	F	08/20/03	E	50	3-02	30	25	V-15	25	10	7.2	7.3	7.25	1:41:10 PM	3:40:25 PM	119	863	
F3-00027	F	08/20/03	E	80	3-02	30	25	DT-03	25	12	9.2	9.1	9.15	1:40:56 PM	3:40:20 PM	119	1,089	
F3-00028	F	08/20/03	E	100	3-02	30	25	DT-09	25	12	9.4	9.5	9.45	1:40:42 PM	3:40:15 PM	119	1,125	
F3-00029	F	08/20/03	E	130	3-02	30	25	DT-30	25	12	10.0	10.3	10.15	1:40:28 PM	3:40:10 PM	119	1,208	
F3-00030	F	08/20/03	E	160	3-02	30	25	DT-06	25	12	9.2	9.2	9.20	1:40:14 PM	3:40:05 PM	119	1,095	
F3-00031	F	08/20/03	E	190	3-02	30	25	DT-07	25	12	10.8	9.5	10.15	1:40:00 PM	3:40:00 PM	120	1,218	
F3-00032	F	08/20/03	W	5	3-02	30	25	V-09	25	4	4.0	3.9	3.95	1:41:50 PM	3:41:00 PM	119	470	
F3-00033	F	08/20/03	W	10	3-02	30	25	V-13	25	6	6.0	6.6	6.30	1:41:40 PM	3:40:56 PM	119	750	
F3-00034	F	08/20/03	W	30	3-02	30	25	V-16	25	8	7.3	7.5	7.40	1:41:30 PM	3:40:49 PM	119	881	
F3-00035	F	08/20/03	W	50	3-02	30	25	V-11	25	10	6.9	7.5	7.20	1:41:25 PM	3:40:42 PM	119	857	
F3-00036	F	08/20/03	W	80	3-02	30	25	DT-04	25	12	8.7	9.6	9.15	1:41:10 PM	3:40:35 PM	119	1,089	
F3-00037	F	08/20/03	W	100	3-02	30	25	DT-20	25	12	8.8	8.8	8.80	1:41:00 PM	3:40:28 PM	119	1,047	
F3-00038	F	08/20/03	W	130	3-02	30	25	DT-08	25	12	9.7	9.4	9.55	1:40:50 PM	3:40:21 PM	119	1,136	
F3-00039	F	08/20/03	W	160	3-02	30	25	DT-05	25	12	8.7	8.9	8.80	1:40:40 PM	3:40:14 PM	119	1,047	
F3-00040	F	08/20/03	W	190	3-02	30	25	DT-10	25	12	8.6	8.6	8.60	1:40:35 PM	3:40:07 PM	119	1,023	
F3-00041	F	08/20/03	W	300	3-02	30	25	DT-01	25	12	9.8	11.4	10.60	1:40:00 PM	3:40:00 PM	120	1,272	

108 Total Samples Collected

**DTSC- Roadside Airborne Asbestos Monitoring Study, POST RESURFACING**  
**DTSC- Roadside Airborne Asbestos Monitoring Study, Post Resurfacing**

Index ID	AALBORG Start Flow Rate (L/min)	AALBORG Stop Flow Rate (L/min)	ESTIMATED Dry Cal DC Lite Start Flow Rate (L/min)	Dry CAL DC Lite Stop Flow Rate (L/min)	Estimated Average Flow Rate (L/min)
F1-00001	6.3	6.4	7.74	7.6	7.67
F1-00002	6.1	6.3	7.54	7.4	7.47
F1-00003	6.8	6.4	7.54	7.4	7.47
F1-00004	6.1	6.6	8.04	7.9	7.97
F1-00005	7.6	8.3	9.74	9.6	9.67
F1-00006	11.9	9.4	12.24	12.1	12.17
F1-00007	7.7	7.1	8.44	8.3	8.37
F1-00008	7.5	8.0	9.64	9.5	9.57
F1-00009	7.8	7.7	9.34	9.2	9.27
F1-00010	6.3	6.1	8.24	8.1	8.17
F1-00011	6.2	6.3	7.54	7.4	7.47
F1-00012	7.8	6.6	8.54	8.4	8.47
F1-00013	6.4	6.4	7.64	7.5	7.57
F1-00014	7.7	7.6	9.14	9.0	9.07
F1-00015	7.7	8.4	10.24	10.1	10.17
F1-00016	7.7	8.1	9.64	9.5	9.57
F1-00017	7.8	8.3	10.14	10.0	10.07
F1-00018	8.6	9.2	11.04	10.9	10.97
F1-00019	8.2	8.1	9.74	9.6	9.67
F1-00020	6.4	6.3	7.34	7.2	7.27

Index ID	Actual Start Flow Rate (L/min)	Actual Stop Flow Rate (L/min)	Difference (start-final)
F2-00001	7.6	7.3	0.3
F2-00002	8.1	8.0	0.1
F2-00003	8.3	7.6	0.7
F2-00004	7.8	7.7	0.1
F2-00005	9.6	9.5	0.1
F2-00006	9.8	9.7	0.1
F2-00007	10.5	9.7	0.8
F2-00008	13.7	9.9	3.8
F2-00009	10.2	9.9	0.3
F2-00010	8.2	7.9	0.3
F2-00011	7.9	7.7	0.2
F2-00012	8.1	7.8	0.3
F2-00013	7.6	7.3	0.3
F2-00014	9.6	9.4	0.2
F2-00015	9.0	9.4	-0.4
F2-00017	9.5	8.6	0.9
F2-00018	9.5	9.3	0.2
F2-00040	9.5	9.3	0.2
F2-00022	3.9	3.8	0.1
F2-00023	6.1	6.4	-0.3
F2-00024	7.8	7.4	0.4
F2-00025	7.5	7.5	0.0
F2-00026	9.8	9.7	0.1
F2-00027	9.6	9.7	-0.1
F2-00028	9.7	9.4	0.3
F2-00029	9.7	9.5	0.2
F2-00030	9.6	9.2	0.4
F2-00031	4.1	4.1	0.0
F2-00032	6.2	9.8	-3.6
F2-00033	7.9	8.1	-0.2
F2-00034	7.7	7.7	0.0
F2-00036	9.6	9.5	0.1
F2-00037	9.0	8.9	0.1
F2-00038	9.9	10.0	-0.1
F2-00039	9.3	9.3	0.0
F2-00041	9.5	9.3	0.2
F3-00004	9.6	9.7	-0.1
F3-00005	7.8	7.9	-0.1
F3-00006	9.9	8.0	1.9
F3-00007	8.9	8.3	0.6
F3-00008	7.6	7.6	0.0
F3-00009	9.4	9.3	0.1
F3-00010	9.5	9.3	0.2
F3-00011	9.5	9.2	0.3
F3-00012	10.2	10.1	0.1
F3-00013	9.8	10.8	-1.0
F3-00014	8.0	6.7	1.3
F3-00015	7.6	7.9	-0.3
F3-00016	7.3	7.4	-0.1
F3-00017	7.5	7.7	-0.2
F3-00018	9.8	9.8	0.0
F3-00019	8.7	8.4	0.3
F3-00020	9.3	8.9	0.4
F3-00021	9.6	9.4	0.2
F3-00022	9.0	8.9	0.1
F3-00023	9.6	9.3	0.3
F3-00024	4.1	4.1	0.0
F3-00025	5.9	5.9	0.0
F3-00026	8.2	7.9	0.3
F3-00027	7.2	7.3	-0.1
F3-00028	9.2	9.1	0.1
F3-00029	9.4	9.5	-0.1
F3-00030	10.0	10.3	-0.3
F3-00032	9.2	9.2	0.0
F3-00033	4.0	3.9	0.1
F3-00034	7.3	7.5	-0.2
F3-00037	8.8	8.8	0.0
F3-00038	9.7	9.4	0.3
F3-00039	8.7	8.9	-0.2
F3-00040	8.6	8.6	0.0

**Average = 0.14**

## **Appendix C – Analytical Data**

**QC Sample Results - Initial & Post Resurfacing QC Samples**

**DTSC- Roadside Airborne Asbestos Monitoring Study**

						TEM - ISO 10312		TEM - AHERA		TEM - AHERA		
Index ID	Date collected	Category (Field=F, Personal=P, Field Blank=FB Lot Blank=LB)	Filter Type	LOT #	Notes	Non QA Analysis		Non QA Analysis		QA Analysis		QC TYPE
						f/cc	LAB	S/cc	LAB	S/cc	LAB	
F1-00019	08/18/03	F	MCE (25mm, 0.45u)	Lot#H3EN2781	300' - No Simulation Run	NA	NA	0.0091	RESI	0.0091	RESI	VA
F2-00031	08/19/03	F	MCE (25mm, 0.45u)	Lot#H3EN2781		NA	NA	0.0920	RESI	0.0850	RESI	IL
F3-00015	08/20/03	F	MCE (25mm, 0.45u)	Lot#H3EN2781				4.40E-03	RESI	4.40E-03	RESI	VA
F3-00027	08/20/03	F	MCE (25mm, 0.45u)	Lot#H3EN2781		NA	NA	9.20E-03	RESI	9.20E-03	RESI	RD
F2-00021	08/19/03	FB	MCE (25mm, 0.45u)	Lot#H3EN2781		NA	NA	Non-Detect 10 GOs (9.1 s/mm2)	RESI	NA	NA	NA
F3-00042	08/20/03	FB	MCE (25mm, 0.45u)	Lot#H3EN2781		NA	NA	Non-Detect 10 GOs (9.1 s/mm2)	RESI	NA	NA	NA
F3-00043	08/20/03	FB	MCE (25mm, 0.45u)	Lot#H3EN2781		NA	NA	NA	NA	NA	NA	NA
P0-00001	07/15/02	FB	Poly (25mm, 0.45u)	Lot#9042032	field blank- Day 0 (0.4 u Polycarbonate filter)	NA	NA	NA	NA	NA	NA	NA
P1-00001	07/16/02	FB	MCE (25mm, 0.45u)	Lot#410FKA-2135	field blank- Day 1 (0.45u MCE filter)	Non-Detect 10 GOs (7.9 s/mm2)	EMSL	NA	NA	NA	NA	NA
P2-00001	07/17/02	FB	MCE (47mm, 0.45u)	Lot#H2BN10114-3499	field blank- Day 2 (0.45u MCE filter)	NA	NA	Non-Detect 10 GOs (8.6 s/mm2)	RESI	Non-Detect 10 GOs (8.6 s/mm2)	RESI	RS
P3-00001	07/18/02	FB	MCE (25mm, 0.45u)	Lot#410FKA-2135	field blank- Day 3 (0.45u MCE filter); no asbestos detected during analysis	NA	NA	Non-Detect 10 GOs (8.6 s/mm2)	RESI	NA	NA	NA
LB 91659	NA	LAB Blank	MCE (25mm, 0.45u)			NA	NA	NA	NA	Non-Detect 10 GOs (9.1 s/mm2)	RESI	LB
LB 96820	NA	LAB Blank	MCE (25mm, 0.45u)			NA	NA	NA	NA	Non-Detect 10 GOs (9.1 s/mm2)	RESI	LB
LB 96823	NA	LAB Blank	MCE (25mm, 0.45u)			NA	NA	NA	NA	Non-Detect 10 GOs (9.1 s/mm2)	RESI	LB
F1-00023	08/18/03	LB	MCE (25mm, 0.45u)	Lot#H3EN2781	Lot Blank: Lot#H3EN2781 (0.45 MEC Filters); used for Day 1,2,3	NA	NA	Non-Detect 10 GOs (9.1 s/mm2)	RESI	NA	NA	NPS
PL-00001	07/15/02	LB	Poly (25mm, 0.45u)	Lot#9042032	Lot Blank: Lot#9042032 (Environmental Express, 0.4u TEM Polycarbonate--this box had been opened earlier, and it belonged to DTSC); used only on Day 0	NA	NA	NA	NA	NA	NA	NA
PL-00002	07/15/02	LB	MCE (47 mm, 0.45u)	Lot#H2HN15713	Lot Blank: Lot#H2HN15713 (Mini-Vol Filter so no cassette);used on Day 0	NA	NA	NA	NA	NA	NA	NA
PL-00003	07/16/02	LB	MCE (25mm, 0.45u)	Lot#410FKA-2135	Lot Blank: Lot#410FKA, Production Code 2135 (0.45 MCE Filters); used Day 1	Non-Detect 10 GOs (7.9 s/mm2)	EMSL	NA	NA	NA	NA	NA
PL-00004	07/16/02	LB	MCE (47 mm, 0.45u)	Lot#H2BN10114-3499	Lot Blank: Lot#H2BN10114-3499 (Omega Cassettes); used for Day 1	Non-Detect 10 GOs (7.9 s/mm2)	EMSL	NA	NA	NA	NA	NA
PL-00005	07/17/02	LB	MCE (25mm, 0.45u)	Lot#410FKA-2135	Lot Blank: Lot#410FKA, Production Code 2135 (0.45 MCE Filters); used Day 2	NA	NA	NA	NA	NA	NA	NA
PL-00006	07/17/02	LB	MCE (25mm, 0.45u)	Lot#410FKA-2135	Lot Blank: Lot#410FKA, Production Code 2135 (0.45 MCE Filters); used Day 2	NA	NA	NA	NA	NA	NA	NA
P2-00004	07/17/02	P	MCE (25mm, 0.45u)	Lot#410FKA-2135	Field Personnel (Volpe personnel)	NA	NA	0.4100 0.5500	EMSL RESI	NA 0.2600	NA RESI	NA RPS

	NVLAP		RESI	
	%	# Required	# Analyzed	%
Lab Blanks (1 per 25)	4.0%	3	3	3.1%
Verifieds	1.0%	1	2	2.0%
Interlab	0.5%	0	1	1.0%
Recount Same	*	4	1	3.1%
Reprep	*		1	
Recount Different	*		1	

**INITIAL STUDY RESULTS - Comparison of Reservoir & EMSL Laboratory Results  
(Chrysotile Asbestos vs. Scrolled Lizardite)**

**DTSC - Roadside Airborne Asbestos Monitoring Study, Initial Study**

NA= Not Applicable; nm= Not Measured

Index ID	Category (Field=F, Personal=P, Blank=B)	Date collected	Run #	East (E) or West (W) of Road?	Vehicle frequency (vph)	Vehicle Speed (mph)	Distance from Road Edge (ft)	Notes	EMSL AHERA Results (structures counted as scrolled lizardite) <b>S/cc</b>	RESI AHERA Results (structures counted as chrysotile asbestos) <b>S/cc</b>
P2-00034	F	07/17/02	02-02	E	30	25	30		0.0320	0.8700
P2-00035	F	07/17/02	02-02	E	30	25	50		0.0510	1.6000
P2-00036	F	07/17/02	02-02	E	30	25	80		0.0510	1.2000
P2-00037	F	07/17/02	02-02	E	30	25	100		0.0044	0.7600
P2-00038	F	07/17/02	02-02	E	30	25	130		0.0091	0.9300
P2-00039	F	07/17/02	02-02	E	30	25	160		0.0220	0.5300
P2-00044	F	07/17/02	02-02	W	30	25	10		0.0240	2.2000
P2-00046	F	07/17/02	02-02	W	30	25	30		0.0320	2.3000
P3-00011	F	07/18/02	03-01	W	10	10	10		0.0085	0.1900
P1-00060	P	07/16/02	NA	NA	NA	NA	NA	In pick-up truck	0.3500	0.3300
P2-00002	P	07/17/02	NA	NA	NA	NA	NA	Field Personnel (DTSC personnel)	0.3400	0.4700
P2-00003	P	07/17/02	NA	NA	NA	NA	NA	Traffic Controller (DTSC personnel)	0.0610	0.0610
P2-00004	P	07/17/02	NA	NA	NA	NA	NA	Field Personnel (Volpe personnel)	0.4100	0.5500 & 0.2600 (QC-RPS)
P3-00002	P	07/18/02	NA	NA	NA	NA	NA	Field Personnel (Volpe personnel)	0.2100	0.4100
P3-00003	P	07/18/02	NA	NA	NA	NA	NA	Traffic Controller (DTSC personnel)	0.1400	4.5000
P3-00004	P	07/18/02	NA	NA	NA	NA	NA	Sedan Driver (Volpe personnel)	0.1500	0.1100

**16 Total Samples**

**NOTE: Above Personal Sample Results have NOT been converted to TWA concentrations**

**STATIONARY RESULTS - POST RESURFACING**  
**DTSC- Roadside Airborne Asbestos Monitoring Study**

										AHERA ASBESTOS RESULTS						
Index ID	Total Volume (L)	Category (Field=F, Personal=P, Blank=B)	Date collected	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Notes	# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)
F1-00020	1,505	F	08/18/03	NA	NA	NA	NA	NA	Background sample taken at Residence (350 Bayleaf Drive, owner = Vance Fellows)	0	2	0	2	10	0.0047	0.0023
<b>AVG. at Background</b>										<b>0</b>	<b>2</b>	<b>0</b>	<b>2</b>	<b>10</b>	<b>0.0047</b>	<b>0.0023</b>
F1-00001	905	F	08/18/03	NA	NA	NA	E	5	No Simulation Run	0	22	1	23	9	0.0990	0.0043
F1-00010	980	F	08/18/03	NA	NA	NA	W	5	No Simulation Run	1	4	0	4	8	0.0180	0.0045
<b>(No Simulation) - AVG. at 5 ft</b>										<b>1</b>	<b>13</b>	<b>1</b>	<b>14</b>	<b>9</b>	<b>0.0585</b>	<b>0.0044</b>
F2-00001	887	F	08/19/03	2-01	10	10	E	5		0	0	2	2	9	0.0088	0.0044
F3-00004	926	F	08/20/03	3-01	10	10	E	5		0	4	1	5	8	0.0240	0.0047
F2-00010	958	F	08/19/03	2-01	10	10	W	5		0	3	0	3	10	0.0110	0.0037
F3-00013	867	F	08/20/03	3-01	10	10	W	5		0	4	0	4	9	0.0180	0.0045
<b>(10mph / 10vph) - AVG. at 5 ft</b>										<b>0</b>	<b>3</b>	<b>1</b>	<b>4</b>	<b>9</b>	<b>0.0155</b>	<b>0.0043</b>
F2-00022	462	F	08/19/03	2-02	30	25	E	5		0	7	1	8	11	0.0550	0.0069
F3-00023	484	F	08/20/03	3-02	30	25	E	5		0	7	0	7	10	0.0510	0.0072
F2-00031 **	492	F	08/19/03	2-02	30	25	W	5		0	10	2	12	10	0.0885	0.0071
F3-00032	470	F	08/20/03	3-02	30	25	W	5		0	11	2	13	10	0.0885	0.0071
<b>(25mph / 30vph) - AVG. at 5 ft</b>										<b>0</b>	<b>8</b>	<b>1</b>	<b>9</b>	<b>10</b>	<b>0.0670</b>	<b>0.0074</b>
F1-00003	881	F	08/18/03	NA	NA	NA	E	30	No Simulation Run	0	2	0	2	9	0.0088	0.0044
F1-00012	1,016	F	08/18/03	NA	NA	NA	W	30	No Simulation Run	0	0	1	1	7	0.0049	0.0049
<b>(No Simulation) - AVG. at 30 ft</b>										<b>0</b>	<b>1</b>	<b>1</b>	<b>2</b>	<b>8</b>	<b>0.0069</b>	<b>0.0047</b>
F2-00003	946	F	08/19/03	2-01	10	10	E	30		0	0	0	0	12	< 0.0031	0.0031
F3-00006	1,023	F	08/20/03	3-01	10	10	E	30		0	4	2	6	7	0.0290	0.0049
F2-00012	938	F	08/19/03	2-01	10	10	W	30		0	4	1	5	10	0.0190	0.0037
F3-00015	875	F	08/20/03	3-01	10	10	W	30		0	1	0	1	9	0.0044	0.0044
<b>(10mph / 10vph) - AVG. at 30 ft</b>										<b>0</b>	<b>2</b>	<b>1</b>	<b>3</b>	<b>10</b>	<b>&lt; 0.0139</b>	<b>0.0040</b>
F2-00024	912	F	08/19/03	2-02	30	25	E	30		1	9	0	9	10	0.0350	0.0038
F3-00025	958	F	08/20/03	3-02	30	25	E	30		0	7	0	7	8	0.0320	0.0046
F2-00033	960	F	08/19/03	2-02	30	25	W	30		0	2	0	2	10	0.0073	0.0036
F3-00034	881	F	08/20/03	3-02	30	25	W	30		0	3	0	3	9	0.0130	0.0044
<b>(25mph / 30vph) - AVG. at 30 ft</b>										<b>0</b>	<b>5</b>	<b>0</b>	<b>5</b>	<b>9</b>	<b>0.0218</b>	<b>0.0041</b>
F1-00005	1,151	F	08/18/03	NA	NA	NA	E	80	No Simulation Run	0	0	0	0	7	< 0.0043	0.0043
F1-00014	1,088	F	08/18/03	NA	NA	NA	W	80	No Simulation Run	0	1	0	1	7	0.0046	0.0046
<b>(No Simulation) - AVG. at 80 ft</b>										<b>0</b>	<b>1</b>	<b>0</b>	<b>1</b>	<b>7</b>	<b>0.0046</b>	<b>0.0045</b>
F2-00005	1,136	F	08/19/03	2-01	10	10	E	80		0	1	0	1	10	0.0031	0.0031
F3-00008	1,113	F	08/20/03	3-01	10	10	E	80		0	9	1	10	7	0.0450	0.0045
F2-00014	1,131	F	08/19/03	2-01	10	10	W	80		0	8	0	8	10	0.0250	0.0031
F3-00017	1,166	F	08/20/03	3-01	10	10	W	80		0	6	0	6	9	0.0270	0.0044
<b>(10mph / 10vph) - AVG. at 80 ft</b>										<b>0</b>	<b>6</b>	<b>0</b>	<b>6</b>	<b>9</b>	<b>0.0250</b>	<b>0.0038</b>
F2-00026	1,170	F	08/19/03	2-02	30	25	E	80		0	2	0	2	10	0.0060	0.0030
F3-00027	1,089	F	08/20/03	3-02	30	25	E	80		0	2	0	2	7	0.0092	0.0046
<b>(25mph / 30vph) - AVG. at 80 ft</b>										<b>0</b>	<b>3</b>	<b>0</b>	<b>3</b>	<b>9</b>	<b>0.0076</b>	<b>0.0038</b>
F2-00036	1,146	F	08/19/03	2-02	30	25	W	100		0	3	0	3	7	0.0130	0.0044
<b>(25mph / 30vph) - AVG. at 100 ft</b>										<b>0</b>	<b>3</b>	<b>0</b>	<b>3</b>	<b>7</b>	<b>0.0130</b>	<b>0.0044</b>
F3-00039	1,047	F	08/20/03	3-02	30	25	W	160		0	0	0	0	7	< 0.0046	0.0046
<b>(25mph / 30vph) - AVG. at 160 ft</b>										<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>7</b>	<b>&lt; 0.0046</b>	<b>0.0046</b>
F2-00039	1,116	F	08/19/03	2-02	30	25	W	190		0	2	0	2	7	0.0090	0.0045
<b>(25mph / 30vph) - AVG. at 190 ft</b>										<b>0</b>	<b>2</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>0.0090</b>	<b>0.0045</b>
F1-00019	1,160	F	08/18/03	NA	NA	NA	W	300	300' - No Simulation Run	0	1	2	3	10	0.0091	0.0030
<b>(No Simulation) - AVG. at 300 ft</b>										<b>0</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>10</b>	<b>0.0091</b>	<b>0.0030</b>
F2-00040	1,128	F	08/19/03	2-01	10	10	W	300		0	7	0	7	6	0.0360	0.0052
<b>(10mph / 10vph) - AVG. at 300 ft</b>										<b>0</b>	<b>7</b>	<b>0</b>	<b>7</b>	<b>6</b>	<b>0.0360</b>	<b>0.0052</b>
F2-00041	1,158	F	08/19/03	2-02	30	25	W	300		0	0	0	0	7	< 0.0043	0.0043
<b>(25mph / 30vph) - AVG. at 300 ft</b>										<b>0</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>7</b>	<b>&lt; 0.0043</b>	<b>0.0043</b>

NOTE: For Sample F2-00031 an average concentration of the Non-QC and QC (inter-lab) analytical results was used. The IL result was slightly less than the non-QC result (0.085s/cc compared to 0.092 s/cc). In all other cases the QC analysis of the stationary samples was equal to the non-QC.

35 Total Stationary Samples Analyzed



**POST RESURFACING - AVG. RESULTS (Stationary Samples)**

		AHERA ASBESTOS RESULTS						
LOCATION / SCENARIO		# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)
Scenario	Distance (ft)							
Background sample taken at Residence (350 Bayleaf Drive, owner = Vance Fellows)	Background	0	2	0	2	10	0.0047	0.0023
No Simulation	5	1	13	1	14	9	0.0585	0.0044
(10 mph / 10 vph)	5	0	3	1	4	9	0.0155	0.0043
(25 mph / 30 vph)	5	0	8	1	9	10	0.0654	0.0071
No Simulation	30	0	1	1	2	8	0.0069	0.0047
(10 mph / 10 vph)	30	0	2	1	3	10	< 0.0139	0.0040
(25 mph / 30 vph)	30	0	5	0	5	9	0.0218	0.0041
No Simulation	80	0	1	0	1	7	0.0046	0.0045
(10 mph / 10 vph)	80	0	6	0	6	9	0.0250	0.0038
(25 mph / 30 vph)	80	0	3	0	3	9	0.0076	0.0038
(25 mph / 30 vph)	100	0	3	0	3	7	0.0130	0.0044
(25 mph / 30 vph)	160	0	0	0	0	7	< 0.0046	0.0046
(25 mph / 30 vph)	190	0	2	0	2	7	0.0090	0.0045
No Simulation	300	0	1	2	3	10	0.0091	0.0030
(10 mph / 10 vph)	300	0	7	0	7	6	0.0360	0.0052
(25 mph / 30 vph)	300	0	0	0	0	7	< 0.0043	0.0043

90%

9%

**PERSONAL RESULTS - POST RESURFACING**  
**DTSC- Roadside Airborne Asbestos Monitoring Study**

						AHERA ASBESTOS RESULTS										PCM ASBESTOS RESULTS	
Index ID	Total Volume (L)	Category (Field=F, Personal=P, Blank=B)	Date collected	Notes	Estimated Exposure Period (hr)	# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)	TEM TWA (S/cc)	LAB	Fibers / cc	PCM TWA (f/cc)	
F1-00024	422	P	08/18/03	Task = Setup & Observer (DTSC personnel) - No Simulation Run	4.0	0	4	1	5	10	0.0410	0.0083	0.0205	RESI	0.012	0.0060	
F1-00025	414	P	08/18/03	Task = Setup & Observer (DTSC personnel) - No Simulation Run	3.0	0	13	1	14	10	0.1200	0.0085	0.0450	RESI	0.012	0.0045	
F2-00042	62	P	08/19/03	Task = Sampler (Volpe personnel), battery failure at 31min	6.0	0	9	0	9	10	0.5100	0.0560	0.3825	RESI	0.079	0.0593	
F2-00043	138	P	08/19/03	Task = Sedan Driver (DTSC personnel), battery failure at 69min	5.0	0	1	0	1	10	0.0250	0.0250	0.0156	RESI	< 0.020	< 0.0125	
F2-00044	600	P	08/19/03	Task = Setup & Observer (DTSC personnel)	6.0	0	0	0	0	10	< 0.0058	0.0058	< 0.0044	RESI	0.018	0.0135	
F3-00001	720	P	08/20/03	Task = Setup & Observer (DTSC personnel)	6.0	0	18	1	19	11	0.0840	0.0044	0.0630	RESI	0.021	0.0158	
F3-00002	730	P	08/20/03	Task = Sampler (Volpe personnel)	6.0	0	7	0	7	10	0.0340	0.0048	0.0255	RESI	0.016	0.0120	
F3-00003	710	P	08/20/03	Task = Truck Driver (DTSC personnel)	6.0	0	5	3	8	10	0.0390	0.0049	0.0293	RESI	0.012	0.0090	
8 Total Personal Samples						0	7 90%	1 10%	8	10	< 0.1074	0.0147	< 0.0313		< 0.0152	< 0.0101	

**STATIONARY RESULTS - INITIAL STUDY (RUNS 2-02 & 3-01)**  
**DTSC - Roadside Airborne Asbestos Monitoring Study**

										AHERA ASBESTOS RESULTS							
Index IDs	Total Volume (L)	Category (Field=F, Personal=P, Blank=B)	Date collected	Run #	Vehicle frequency (vph)	Vehicle Speed (mph)	East (E) or West (W) of Road?	Distance from Road Edge (ft)	Notes	# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)	
P2-00001	na	B	07/17/02	02-B	na	na	na	na	field blank- Day 2 (0.45u MCE filter)	0	0	0	0	10	Blank	Blank	
P3-00001	na	B	07/18/02	03-B	na	na	na	na	field blank- Day 3 (0.45u MCE filter); no asbestos detected during analysis	0	0	0	0	10	Blank	Blank	
P3-00005	978	F	07/18/02	03-01	10	10	E	5		4	46	1	47	3	0.5300	0.0110	
P3-00010	924	F	07/18/02	03-01	10	10	W	5		11	52	3	55	2	0.9800	0.0180	
									(10mph / 10vph) - AVG. at 5 ft	8	49	2	51	3	0.7550	0.0145	
P2-00029	224.4	F	07/17/02	02-02	30	25	E	5		8	61	3	64	1	9.5000	0.1500	
P2-00030	489.6	F	07/17/02	02-02	30	25	E	5		3	84	10	94	1	3.7000	0.0390	
P2-00042	492	F	07/17/02	02-02	30	25	W	5		4	42	7	49	1	3.3000	0.0670	
P2-00041	222	F	07/17/02	02-02	30	25	W	5		12	53	5	58	1	8.7000	0.1500	
									(25mph / 30vph) - AVG. at 5 ft	7	60	6	66	1	6.3000	0.1015	
P3-00006	1206	F	07/18/02	03-01	10	10	E	10		5	36	12	48	5	0.2600	0.0055	
P3-00011	1182	F	07/18/02	03-01	10	10	W	10		3	37	4	41	6	0.1900	0.0047	
									(10mph / 10vph) - AVG. at 10 ft	4	37	8	45	6	0.2250	0.0051	
P2-00032	747	F	07/17/02	02-02	30	25	E	10		37	36	4	40	1	1.8000	0.0440	
P2-00031	504	F	07/17/02	02-02	30	25	E	10		2	44	11	55	1	3.6000	0.0660	
P2-00044	708	F	07/17/02	02-02	30	25	W	10		19	43	4	47	1	2.2000	0.0470	
P2-00043	492	F	07/17/02	02-02	30	25	W	10		10	42	1	43	2	1.5000	0.0340	
									(25mph / 30vph) - AVG. at 10 ft	17	41	5	46	1	2.2750	0.0478	
P3-00007**	786	F	07/18/02	03-01	10	10	E	30	during field sampling: tube collapsing under vacuum, need surgical tube -- thus produced low stop flow rate and low total volume	0	3	0	3	10	0.0130	0.0042	
P3-00012	1323	F	07/18/02	03-01	10	10	W	30		12	46	7	53	4	0.3300	0.0063	
									(10mph / 10vph) - AVG. at 30 ft	12	46	7	53	4	0.3300	0.0063	
P2-00034	954	F	07/17/02	02-02	30	25	E	30		24	21	4	25	1	0.8700	0.0350	
P2-00033	721.8	F	07/17/02	02-02	30	25	E	30		7	38	6	44	1	2.0000	0.0460	
P2-00046	936	F	07/17/02	02-02	30	25	W	30		11	60	4	64	1	2.3000	0.0350	
P2-00045	702	F	07/17/02	02-02	30	25	W	30		9	37	4	41	2	0.9700	0.0240	
									(25mph / 30vph) - AVG. at 30 ft	13	39	5	44	1	1.5350	0.0350	
P2-00035	1176	F	07/17/02	02-02	30	25	E	50		3	87	9	96	1	1.6000	0.0160	
P2-00047	1200	F	07/17/02	02-02	30	25	W	50		9	39	1	40	5	0.2200	0.0055	
									(25mph / 30vph) - AVG. at 50 ft	6	63	5	68	3	0.9100	0.0108	
P3-00008	1416	F	07/18/02	03-01	10	10	E	80		1	8	1	9	4	0.0530	0.0059	
P3-00013	1332	F	07/18/02	03-01	10	10	W	80		9	42	3	45	3	0.3700	0.0083	
									(10mph / 10vph) - AVG. at 80 ft	5	25	2	27	4	0.2115	0.0071	
P2-00036	1200	F	07/17/02	02-02	30	25	E	80		21	42	1	43	1	1.2000	0.0280	
P2-00048	1254	F	07/17/02	02-02	30	25	W	80		2	41	8	49	6	0.2200	0.0044	
									(25mph / 30vph) - AVG. at 80 ft	12	42	5	46	4	0.7100	0.0162	
P2-00037	1140	F	07/17/02	02-02	30	25	E	100		9	26	0	26	1	0.7600	0.0290	
P2-00049	1188	F	07/17/02	02-02	30	25	W	100		2	15	5	20	6	0.0930	0.0047	
									(25mph / 30vph) - AVG. at 100 ft	6	21	3	23	4	0.4265	0.0169	
P3-00009	1428	F	07/18/02	03-01	10	10	E	130		0	2	0	2	5	0.0093	0.0046	
P3-00014	1272	F	07/18/02	03-01	10	10	W	130		10	32	1	33	10	0.0860	0.0026	
									(10mph / 10vph) - AVG. at 130 ft	5	17	1	18	8	0.0477	0.0036	
P2-00038	1320	F	07/17/02	02-02	30	25	E	130		4	35	2	37	1	0.9300	0.0250	
P2-00050	1446	F	07/17/02	02-02	30	25	W	130		5	14	0	14	4	0.0800	0.0057	
									(25mph / 30vph) - AVG. at 130 ft	5	25	1	26	3	0.5050	0.0154	
P2-00039	1380	F	07/17/02	02-02	30	25	E	160		17	43	1	44	2	0.5300	0.0120	
P2-00051	1422	F	07/17/02	02-02	30	25	W	160		6	26	3	29	4	0.1700	0.0058	
									(25mph / 30vph) - AVG. at 160 ft	12	35	2	37	3	0.3500	0.0089	
P2-00040	1470	F	07/17/02	02-02	30	25	E	190		7	33	14	47	4	0.2800	0.0060	
P2-00052	1290	F	07/17/02	02-02	30	25	W	190	during sampling, pump had crimp in line	8	22	0	22	6	0.0940	0.0043	
									(25mph / 30vph) - AVG. at 190 ft	8	28	7	35	5	0.1870	0.0052	

\*\* NOTE: The result for sample P3-00007 was not included in the calculated average, since a sampling tube collapsed under vacuum, producing a low stop flow rate and low/inaccurate total volume

34 Total Stationary Samples Analyzed  
2 Total Field Blanks Analyzed

# INITIAL STUDY - AVG. RESULTS (Stationary Samples)

		AHERA ASBESTOS RESULTS						
LOCATION / SCENARIO		# Non- AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)
Scenario	Distance (ft)							
(10 mph / 10 vph)	5	8	49	2	51	3	0.7550	0.0145
(25 mph / 30 vph)	5	7	60	6	66	1	6.3000	0.1015
(10 mph / 10 vph)	10	4	37	8	45	6	0.2250	0.0051
(25 mph / 30 vph)	10	17	41	5	46	1	2.2750	0.0478
(10 mph / 10 vph)	30	12	46	7	53	4	0.3300	0.0063
(25 mph / 30 vph)	30	13	39	5	44	1	1.5350	0.0350
(25 mph / 30 vph)	50	6	63	5	68	3	0.9100	0.0108
(10 mph / 10 vph)	80	5	25	2	27	4	0.2115	0.0071
(25 mph / 30 vph)	80	12	42	5	46	4	0.7100	0.0162
(25 mph / 30 vph)	100	6	21	3	23	4	0.4265	0.0169
(10 mph / 10 vph)	130	5	17	1	18	8	0.0477	0.0036
(25 mph / 30 vph)	130	5	25	1	26	3	0.5050	0.0154
(25 mph / 30 vph)	160	12	35	2	37	3	0.3500	0.0089
(25 mph / 30 vph)	190	8	28	7	35	5	0.1870	0.0052

90%

10%

**PERSONAL RESULTS - INITIAL STUDY**  
**DTSC - Roadside Airborne Asbestos Monitoring Study**

						AHERA ASBESTOS RESULTS								PCM ASBESTOS RESULTS				
Index IDs	Total Volume (L)	Category (Field=F, Personal=P, Blank=B)	Date collected	Notes	Estimated Exposure Period (hr)	# Non-AHERA Asbestos (excluded structures)	# AHERA Structures (< 5um)	# AHERA Structures (>= 5um)	# AHERA Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)	TEM TWA (S/cc)	LAB	Fibers / cc	PCM TWA (f/cc)	LAB	
P0-00016	350	P	07/15/02	Sedan Driver (DTSC personnel)	3.0	0	2	3	5	29	0.0150	0.0030	0.0056	EMSL++	could not analyze, filter type is PC		EMSL	
P0-00017	350	P	07/15/02	Traffic Controller (DTSC personnel)	3.0	0	47	10	57	8	0.6200	0.0110	0.2325	EMSL++				
P1-00060	746	P	07/16/02	In pick-up truck	6.5	0	40	12	52	6	0.3500	0.0068	0.2844	EMSL++	0.016	0.0130	EMSL	
						18	44	5	49	2	0.3300	0.0130	0.2681	RESI				
P1-00061	775	P	07/16/02	Field Personnel (Volpe personnel)	6.5	0	32	18	50	7	0.2800	0.0056	0.2275	EMSL++	0.006	0.0049	EMSL	
P1-00062	745	P	07/16/02	Traffic Controller (DTSC personnel)	6.5	0	2	1	3	9	0.0140	0.0046	0.0114	EMSL++	< 0.004	< 0.0033	EMSL	
P2-00002	752	P	07/17/02	Field Personnel (DTSC personnel)	6.5	19	29	4	33	4	0.3400	0.0100	0.2763	EMSL++	0.006	0.0049	EMSL	
						8	32	5	37	2	0.4700	0.0130	0.3819	RESI				
P2-00003	728	P	07/17/02	Traffic Controller (DTSC personnel)	6.5	7	12	1	13	9	0.0610	0.0047	0.0496	EMSL++	< 0.004	< 0.0033	EMSL	
						0	8	0	8	6	0.0610	0.0076	0.0496	RESI				
P2-00004	721	P	07/17/02	Field Personnel (Volpe personnel)	6.5	10	40	8	48	5	0.4100	0.0085	0.3331	EMSL++	< 0.004	< 0.0033	EMSL	
						0	24	0	24	2	0.5500	0.0230	0.4469	RESI				
						1	21	1	22	2	0.2600	0.0230	0.2113	RESI QC-RPs				
P3-00002	299	P	07/18/02	Field Personnel (Volpe personnel)	3.0	1	11	0	11	3	0.4100	0.0370	0.1538	RESI	< 0.009	< 0.0034	EMSL	
						0	10	11	21	10	0.2100	0.0100	0.0788	EMSL++				
P3-00003	338	P	07/18/02	Field Personnel (DTSC personnel)	3.0	6	45	1	46	1	4.5000	0.0980	1.6875	RESI	0.012	0.0045	EMSL	
						10	11	5	16	10	0.1400	0.0090	0.0525	EMSL++				
P3-00004	314	P	07/18/02	Sedan Driver (Volpe personnel)	3.0	1	9	0	9	9	0.1100	0.0120	0.0413	RESI	< 0.009	< 0.0034	EMSL	
						3	13	2	15	10	0.1500	0.0097	0.0563	EMSL++				
11 Total Personal Samples						AVERAGE		4	23	5	27	7	0.4885	0.0163	0.2552	< 0.0078	< 0.0049	
								83%	17%									

**NOTE:** "++" EMSL concentrations excluded structures that appeared as "scrolled lizardite", concentrations were determined by EMSL prior to reaching a consensus among laboratories that structures that appear as "scrolled lizardite"

INITIAL STATIONARY - INITIAL STUDY RESULTS - Except for Runs 02-02 & 03-01

DTSC- Roadside Airborne Asbestos Monitoring Study, Initial Study

NA= Not Applicable; nm= Not Measured										TEM RESULTS (AHERA & ISO)									PCM ASBESTOS RESULTS	
Index ID	Category (Field=F, Personal=P, Blank=B)	Date collected	Run #	East (E) or West (W) of Road?	Vehicle frequency (vph)	Vehicle Speed (mph)	Distance from Road Edge (ft)	Filter Diameter (mm)	Notes	# Non-Method Asbestos (excluded structures)	# Structures (< 5um)	# Structures (>= 5um)	# Structures (total)	#GOs	[Asbestos] (S / cc)	Analytical Sensitivity (S/cc)	METHOD	LAB	Fibers / cc	LAB
P1-00041	F	07/16/02	01-02	W	30	25	10	25		9	84	10	94	9	3.64E-01	3.54E-03	ISO	EMSL	NA	
P1-00043	F	07/16/02	01-02	W	30	25	10	47		4	35	6	41	10	5.35E-01	1.19E-02	ISO	EMSL	NA	

## **Appendix D – Comparison of Analytical Results**

## Comparison of Average Results

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**10 mph / 10 vph**

Distance	Initial	Post	% Diff (Pre & Post)	No Simulation	Background
5	0.7550	0.0155	98%	0.0585	0.0047
10	0.2250	NA		NA	0.0047
30	0.3300	< 0.0139	96%	0.0069	0.0047
50	nm	NA		NA	0.0047
80	0.2115	0.0250	88%	0.0046	0.0047
130	0.0477	NA		NA	0.0047
160	nm	NA		NA	0.0047
190	nm	NA		NA	0.0047
300	nm	0.0360		0.0091	0.0047

**AVG**

**94%**

NA

Not Analyzed

nm

Not Measured

**25 mph / 30 vph**

Distance	Initial	Post	% Diff (Pre & Post)	No Simulation	Background
5	6.3000	0.0654	99%	0.0585	0.0047
10	2.2750	NA		NA	0.0047
30	1.5350	0.0218	99%	0.0069	0.0047
50	0.9100	NA		NA	0.0047
80	0.7100	0.0076	99%	0.0046	0.0047
100	0.4265	0.0130	97%	NA	0.0047
130	0.5050	NA		NA	0.0047
160	0.3500	< 0.0046	99%	NA	0.0047
190	0.1870	0.0090	95%	NA	0.0047
300	nm	< 0.0043		0.0091	0.0047

**AVG**

**98%**

NA

Not Analyzed

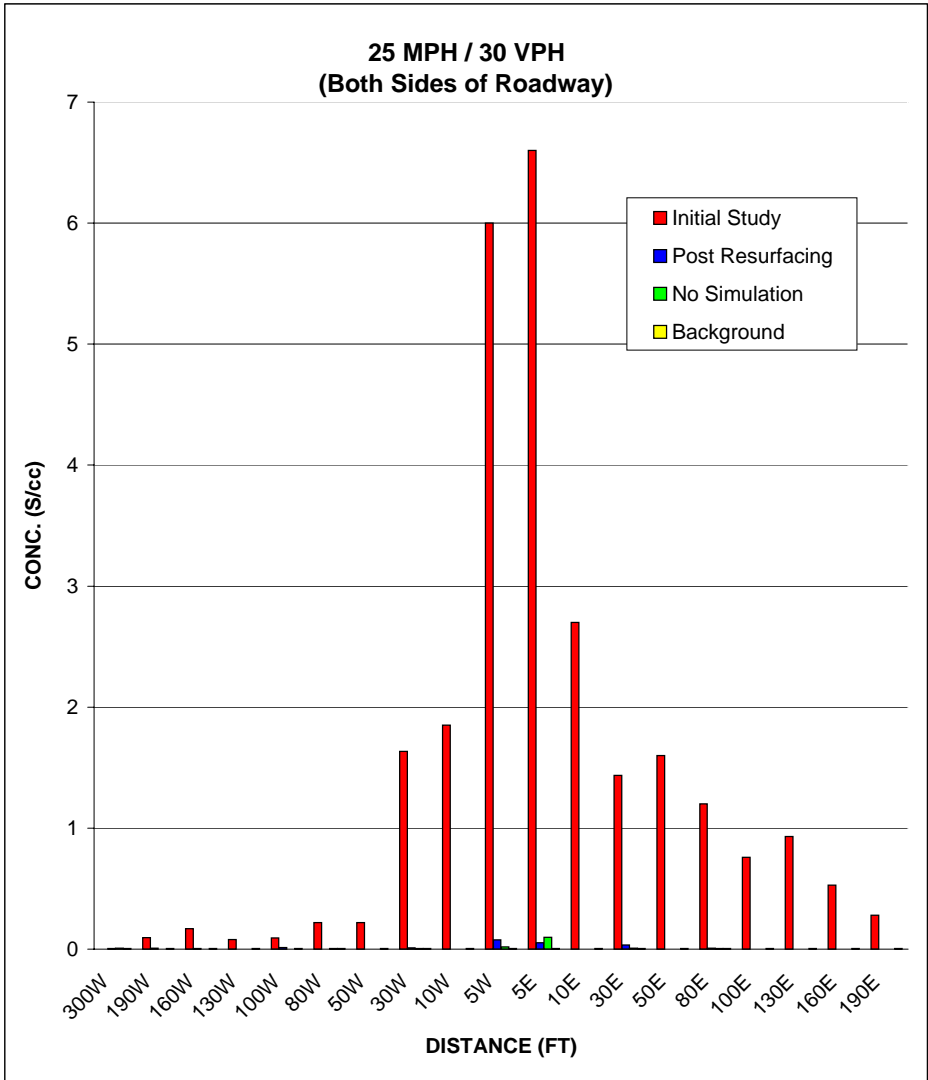
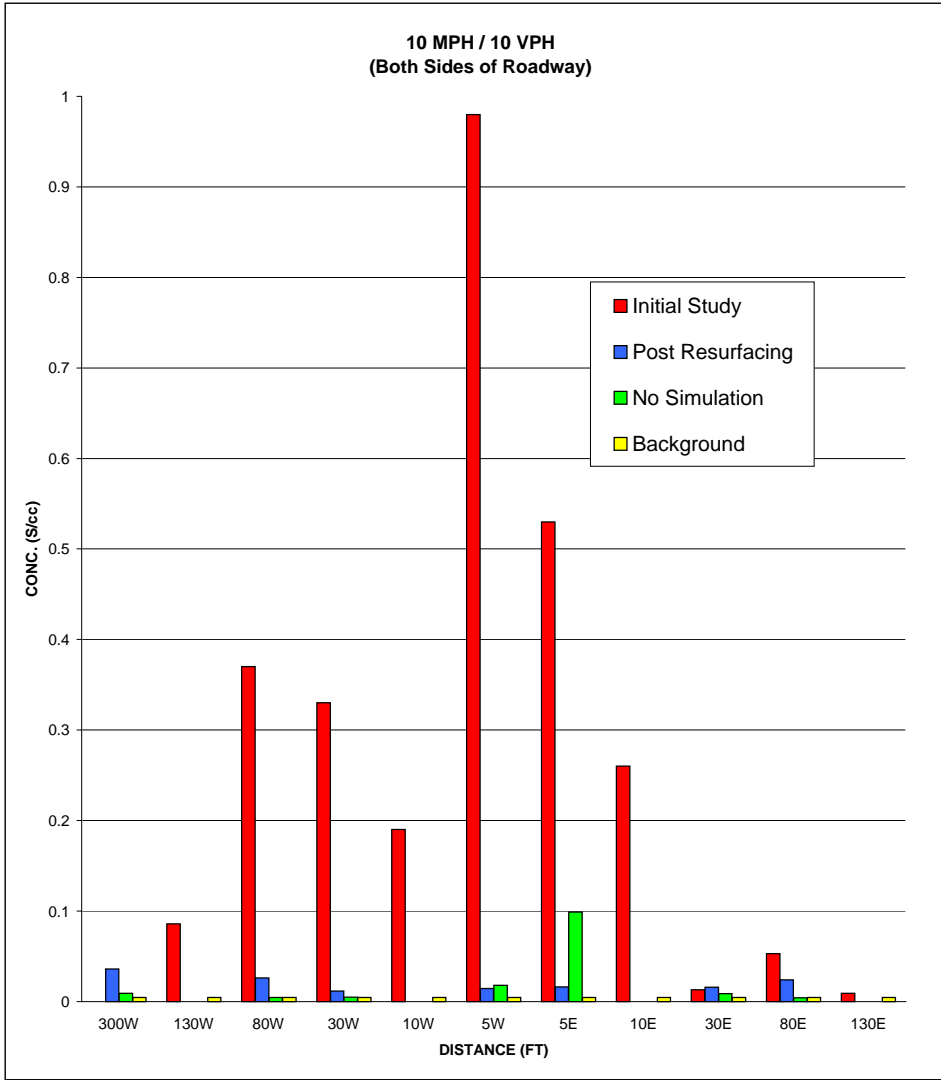
nm

Not Measured



10 mph / 10 vph				
DISTANCE	Initial Conc.	Post Conc. (Average)	No Simulation	Background
300W	nm	0.0360	0.0091	0.0047
130W	0.0860	NA	NA	0.0047
80W	0.3700	0.0260	0.0046	0.0047
30W	0.3300	0.0117	0.0049	0.0047
10W	0.1900	NA	NA	0.0047
5W	0.9800	0.0145	0.0180	0.0047
5E	0.5300	0.0164	0.0990	0.0047
10E	0.2600	NA	NA	0.0047
30E	0.0130	0.0161	0.0088	0.0047
80E	0.0530	0.0241	0.0043	0.0047
130E	0.0093	NA	NA	0.0047
NA Not Analyzed				
nm Not Measured				

25 mph / 30 vph				
DISTANCE	Initial Conc. (Average)	Post Conc. (Average)	No Simulation	Background
300W	nm	0.0043	0.0091	0.0047
190W	0.0940	0.0090	NA	0.0047
160W	0.1700	0.0046	NA	0.0047
130W	0.0800	NA	NA	0.0047
100W	0.0930	0.0130	NA	0.0047
80W	0.2200	NA	0.0046	0.0047
50W	0.2200	NA	NA	0.0047
30W	1.6350	0.0102	0.0049	0.0047
10W	1.8500	NA	NA	0.0047
5W	6.0000	0.0778	0.0180	0.0047
5E	6.6000	0.0530	0.0990	0.0047
10E	2.7000	NA	NA	0.0047
30E	1.4350	0.0335	0.0088	0.0047
50E	1.6000	NA	NA	0.0047
80E	1.2000	0.0076	0.0043	0.0047
100E	0.7600	NA	NA	0.0047
130E	0.9300	NA	NA	0.0047
160E	0.5300	NA	NA	0.0047
190E	0.2800	NA	NA	0.0047
NA Not Analyzed				
nm Not Measured				

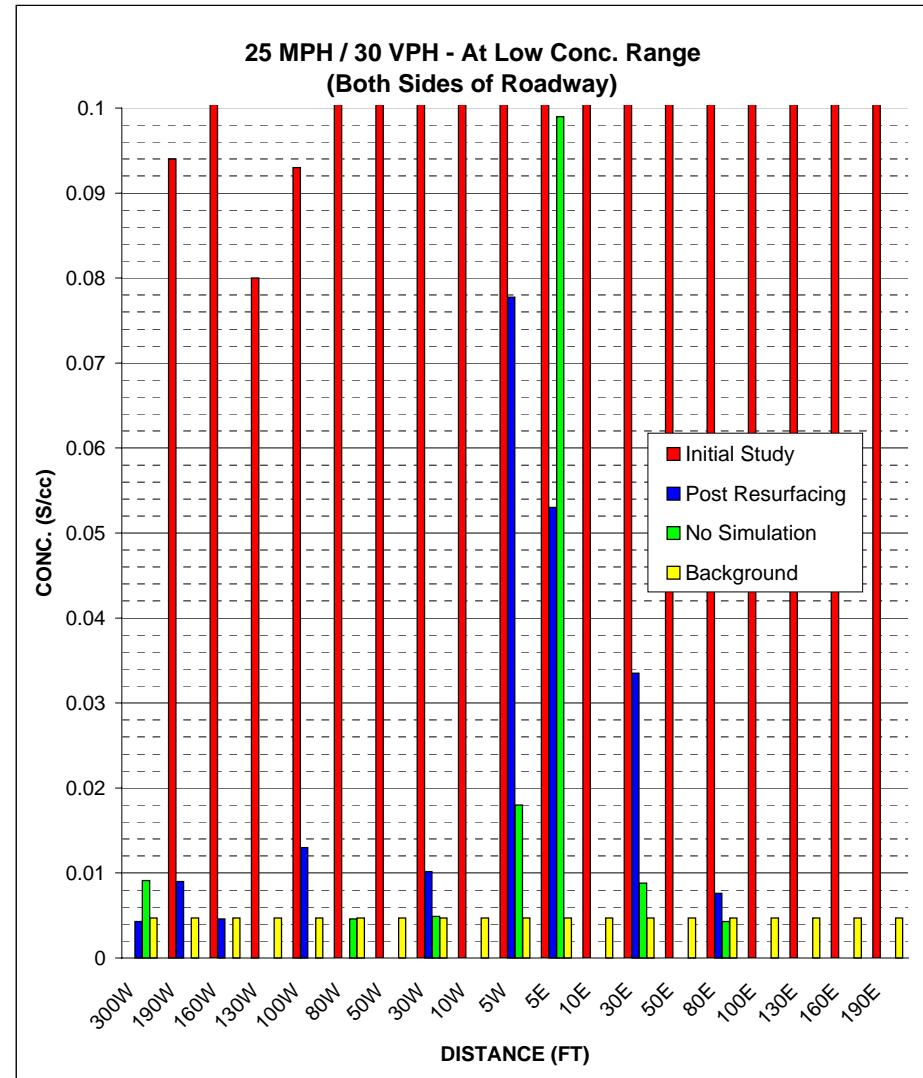
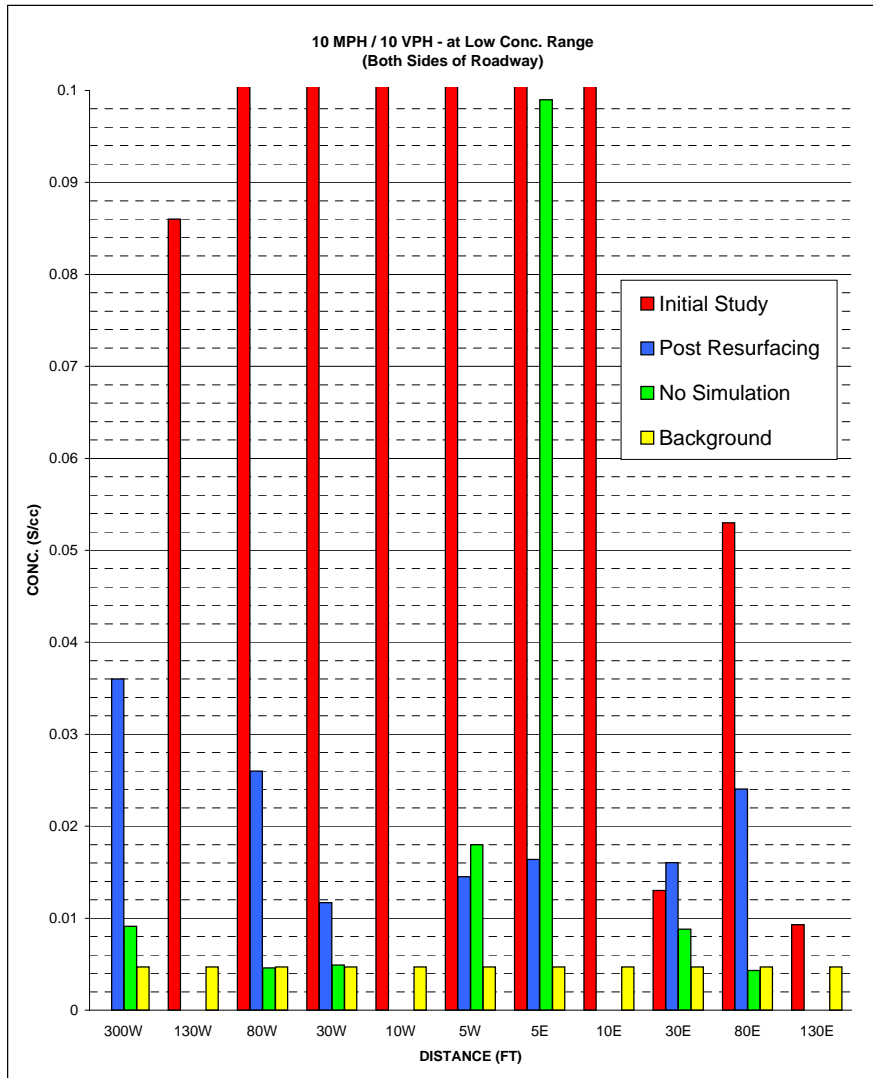


10 mph / 10 vph				
DISTANCE	Initial Conc.	Post Conc. (Average)	No Simulation	Background
300W	nm	0.0360	0.0091	0.0047
130W	0.0860	NA	NA	0.0047
80W	0.3700	0.0260	0.0046	0.0047
30W	0.3300	0.0117	0.0049	0.0047
10W	0.1900	NA	NA	0.0047
5W	0.9800	0.0145	0.0180	0.0047
5E	0.5300	0.0164	0.0990	0.0047
10E	0.2600	NA	NA	0.0047
30E	0.0130	0.0161	0.0088	0.0047
80E	0.0530	0.0241	0.0043	0.0047
130E	0.0093	NA	NA	0.0047

NA Not Analyzed  
nm Not Measured

25 mph / 30 vph				
DISTANCE	Initial Conc.	Post Conc. (Average)	No Simulation	Background
300W	nm	0.0043	0.0091	0.0047
190W	0.0940	0.0090	NA	0.0047
160W	0.1700	0.0046	NA	0.0047
130W	0.0800	NA	NA	0.0047
100W	0.0930	0.0130	NA	0.0047
80W	0.2200	NA	0.0046	0.0047
50W	0.2200	NA	NA	0.0047
30W	1.6350	0.0102	0.0049	0.0047
10W	1.8500	NA	NA	0.0047
5W	6.0000	0.0778	0.0180	0.0047
5E	6.6000	0.0530	0.0990	0.0047
10E	2.7000	NA	NA	0.0047
30E	1.4350	0.0335	0.0088	0.0047
50E	1.6000	NA	NA	0.0047
80E	1.2000	0.0076	0.0043	0.0047
100E	0.7600	NA	NA	0.0047
130E	0.9300	NA	NA	0.0047
160E	0.5300	NA	NA	0.0047
190E	0.2800	NA	NA	0.0047

NA Not Analyzed  
nm Not Measured



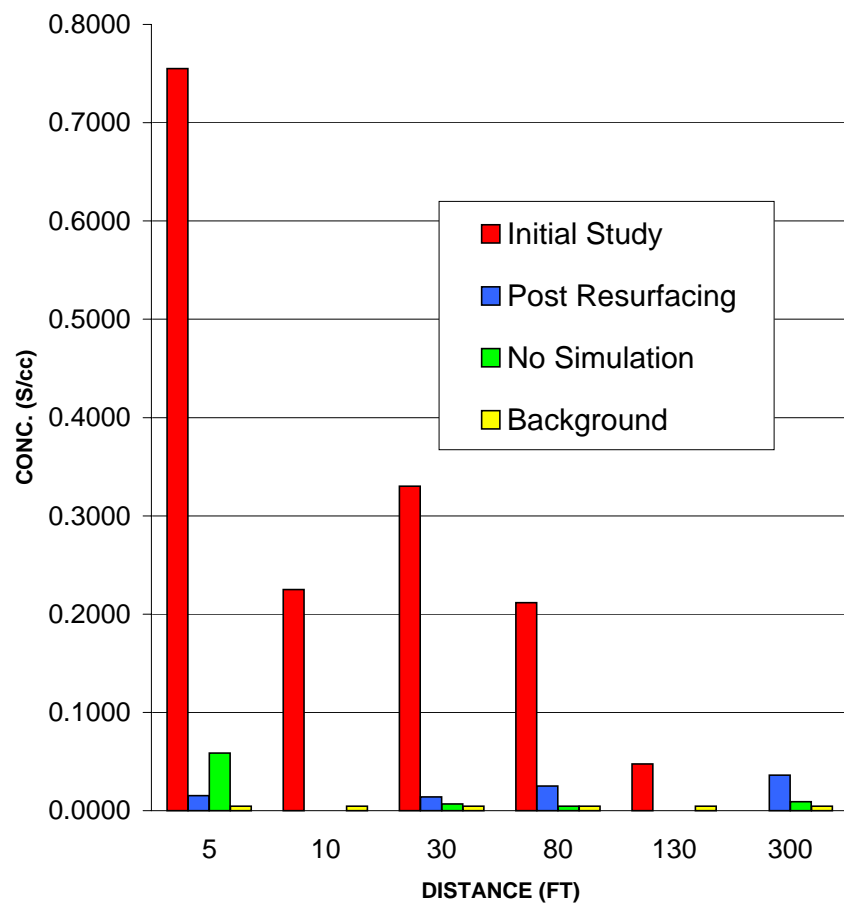
10 mph / 10 vph				
Distance	Initial	Post	No Simulation	Background
5	0.7550	0.0155	0.0585	0.0047
10	0.2250	NA	NA	0.0047
30	0.3300	< 0.0139	0.0069	0.0047
80	0.2115	0.0250	0.0046	0.0047
130	0.0477	NA	NA	0.0047
300	nm	0.0360	0.0091	0.0047

NA Not Analyzed  
nm Not Measured

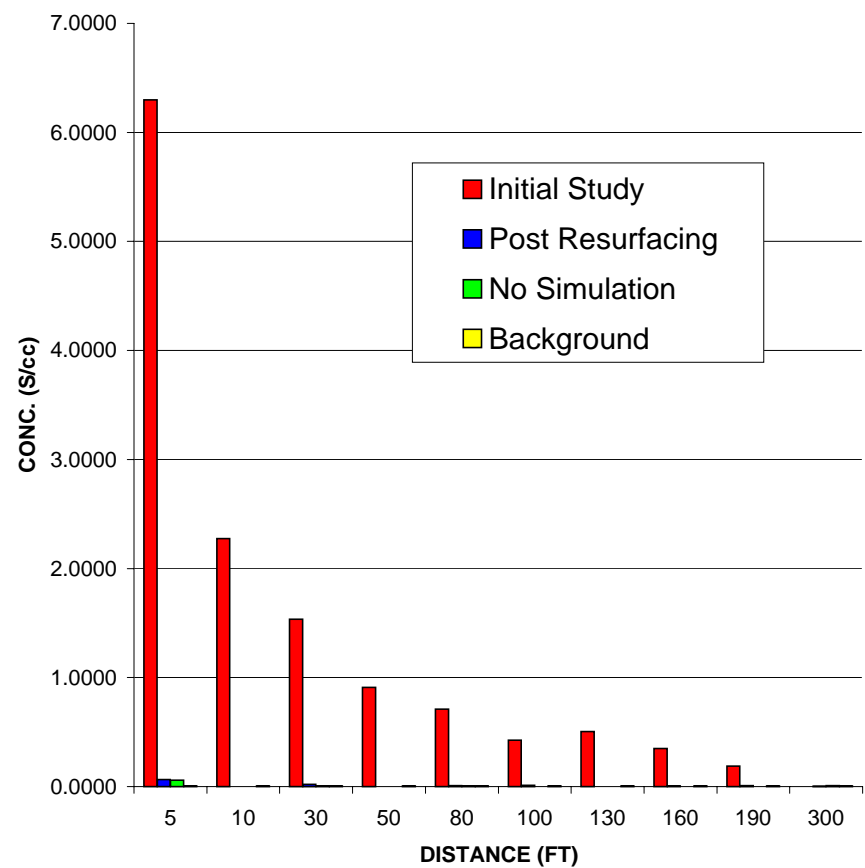
25 mph / 30 vph				
Distance	Initial	Post	No Simulation	Background
5	6.3000	0.0654	0.0585	0.0047
10	2.2750	NA	NA	0.0047
30	1.5350	0.0218	0.0069	0.0047
50	0.9100	NA	NA	0.0047
80	0.7100	0.0076	0.0046	0.0047
100	0.4265	0.0130	NA	0.0047
130	0.5050	NA	NA	0.0047
160	0.3500	< 0.0046	NA	0.0047
190	0.1870	0.0090	NA	0.0047
300	nm	< 0.0043	0.0091	0.0047

NA Not Analyzed  
nm Not Measured

**10 MPH / 10 VPH**  
(AVG of Both Sides of Roadway)



**25 MPH / 30 VPH**  
(AVG of Both Sides of Roadway)



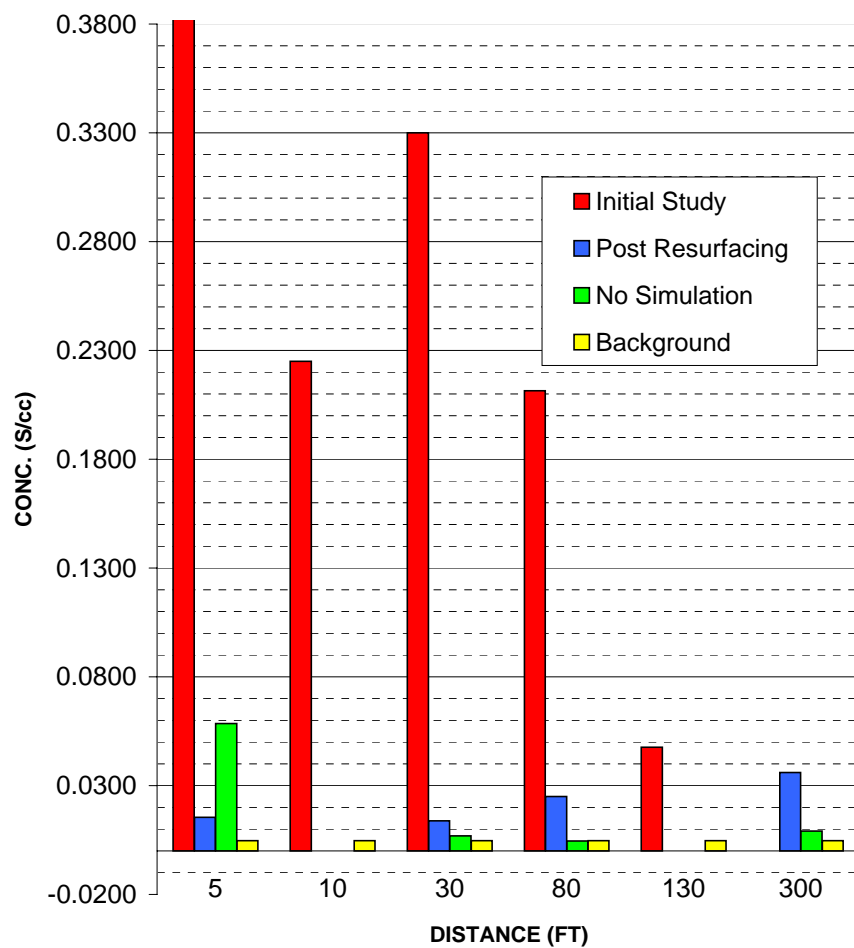
10 mph / 10 vph				
Distance	Initial	Post	No Simulation	Background
5	0.7550	0.0155	0.0585	0.0047
10	0.2250	NA	NA	0.0047
30	0.3300	< 0.0139	0.0069	0.0047
80	0.2115	0.0250	0.0046	0.0047
130	0.0477	NA	NA	0.0047
300	nm	0.0360	0.0091	0.0047

NA Not Analyzed  
nm Not Measured

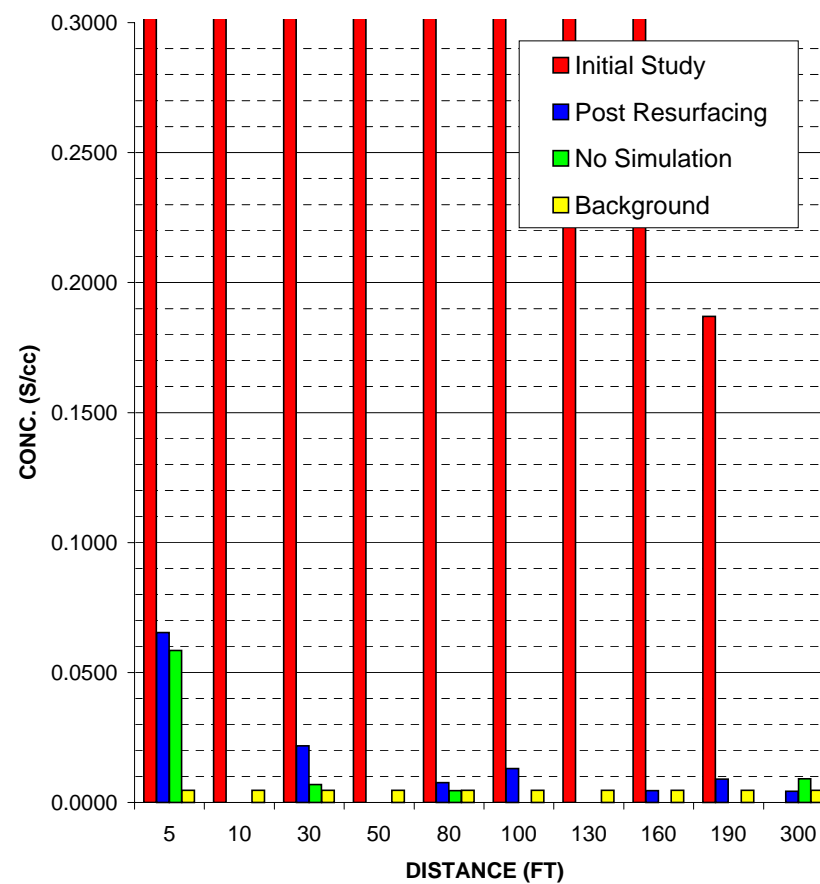
25 mph / 30 vph				
Distance	Initial	Post	No Simulation	Background
5	6.3000	0.0654	0.0585	0.0047
10	2.2750	NA	NA	0.0047
30	1.5350	0.0218	0.0069	0.0047
50	0.9100	NA	NA	0.0047
80	0.7100	0.0076	0.0046	0.0047
100	0.4265	0.0130	NA	0.0047
130	0.5050	NA	NA	0.0047
160	0.3500	< 0.0046	NA	0.0047
190	0.1870	0.0090	NA	0.0047
300	nm	< 0.0043	0.0091	0.0047

NA Not Analyzed  
nm Not Measured

**10 MPH / 10 VPH - At Low Conc. Range  
(AVG of Both Sides of Roadway)**



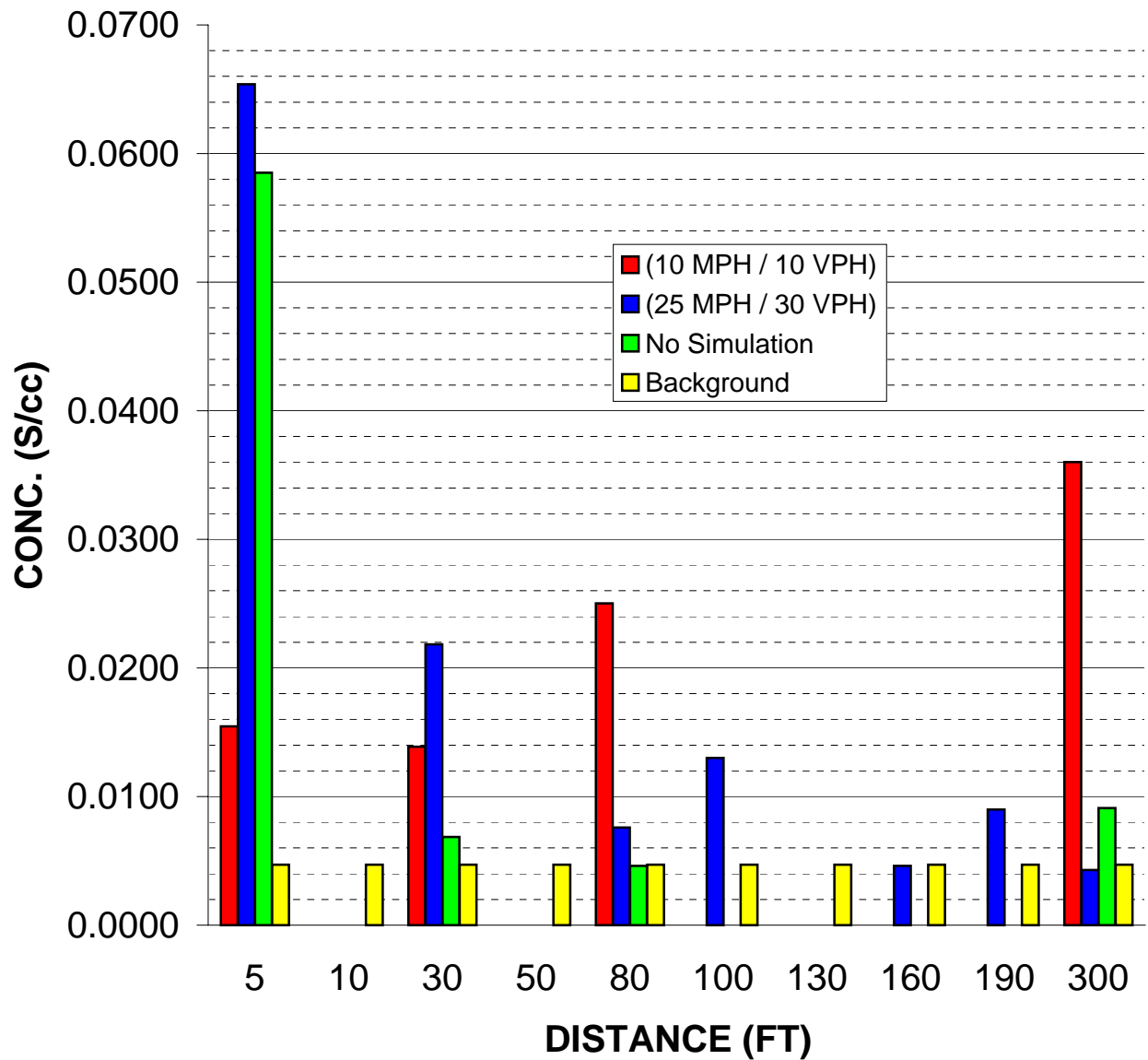
**25 MPH / 30 VPH - At Low Conc. Range  
(AVG of Both Sides of Roadway)**



POST REMEDIATION				
Distance	Post (10mph/10vph)	Post (25mph/30vph)	No Simulation	Background
5	0.0155	0.0654	0.0585	0.0047
10	NA	NA	NA	0.0047
30	< 0.0139	0.0218	0.0069	0.0047
50	NA	NA	NA	0.0047
80	0.0250	0.0076	0.0046	0.0047
100	NA	0.0130	NA	0.0047
130	NA	NA	NA	0.0047
160	NA	< 0.0046	NA	0.0047
190	NA	0.0090	NA	0.0047
300	0.0360	< 0.0043	0.0091	0.0047

NA Not Analyzed  
nm Not Measured

**Post Remediation  
(AVG of Both Sides of Roadway)**



## **Appendix E – Traffic Data**

## INITIAL STUDY - 7/15/02 Traffic Data

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**Scenario 00-01 = 30 vph & 25 mph**  
(vehicle every 2 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)
7/15/2002	00-01	1	12:56	12:58	
7/15/2002	00-01	2	13:00	13:02	
7/15/2002	00-01	3	13:04	13:06	
7/15/2002	00-01	4	13:08	13:10	
7/15/2002	00-01	5	13:12	13:14	
7/15/2002	00-01	6	13:16	13:18	
7/15/2002	00-01	7	13:20	13:22	
7/15/2002	00-01	8	13:24	13:26	
7/15/2002	00-01	9	13:28	13:30	
7/15/2002	00-01	10	13:32	13:34	
7/15/2002	00-01	11	13:36	13:38	
7/15/2002	00-01	12	13:40	13:42	
7/15/2002	00-01	13	13:44	13:46	
7/15/2002	00-01	14	13:48	13:50	
7/15/2002	00-01	15	13:52	13:54	
7/15/2002	00-01	16	13:56	13:58	
7/15/2002	00-01	17	14:00	14:02	
7/15/2002	00-01	18	14:04	14:06	
7/15/2002	00-01	19	14:08	14:10	
7/15/2002	00-01	20	14:12	14:14	
7/15/2002	00-01	21	14:16	14:18	
7/15/2002	00-01	22	14:20	14:22	
7/15/2002	00-01	23	14:24	14:26	
7/15/2002	00-01	24	14:28	14:30	
7/15/2002	00-01	25	14:32	14:34	
7/15/2002	00-01	26	14:36	14:38	
7/15/2002	00-01	27	14:40	14:42	
7/15/2002	00-01	28	14:44	14:46	
7/15/2002	00-01	29	14:48	14:50	
7/15/2002	00-01	30	14:52	14:54	

60 Total vehicles recorded over 2 hours (30 passenger & 30 pick-ups)

## INITIAL STUDY - 7/16/02 Traffic Data

### Scenario 01-01 = 10 vph & 25 mph (vehicle every 6 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)**	
7/16/2002	01-01	1	10:11	10:05	10:25	Local Car, Slow
7/16/2002	01-01	2	10:27	10:17	10:29	Counted - Local Truck, Fast
7/16/2002	01-01	3	10:35	10:41	10:31	Local Truck, Slow
7/16/2002	01-01	4	10:47	10:53	10:32	Local Car, Slow
7/16/2002	01-01	5	10:59	11:05	10:40	Local Car, Slow
7/16/2002	01-01	6	11:11	11:17	11:01	Local Truck, Slow
7/16/2002	01-01	7	11:23	11:30		
7/16/2002	01-01	8	11:35	11:41		
7/16/2002	01-01	9	11:47	11:53		
7/16/2002	01-01	10	11:59			

23 Total vehicles recorded over 2 hours (13 passenger & 11 pick-ups)

\*\*Local cars were driven slow (<10 mph) creating minimal dust. Local 10:29 driven fast creating dust.

### Scenario 01-02 = 30 vph & 25 mph (vehicle every 2 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)**	
7/16/2002	01-02	1	13:18	13:16	13:23	Local Truck, Slow
7/16/2002	01-02	2	13:22	13:20	13:25	Local Truck, Slow
7/16/2002	01-02	3	13:26	13:24	13:40	Local Car, Slow
7/16/2002	01-02	4	13:30	13:28	14:27	Taurus*
7/16/2002	01-02	5	13:34	13:32	14:48	Local Car, Slow
7/16/2002	01-02	6	13:38	13:36		
7/16/2002	01-02	7	13:42	13:40		
7/16/2002	01-02	8	13:47	13:44		
7/16/2002	01-02	9	13:50	13:48		
7/16/2002	01-02	10	13:54	13:52		
7/16/2002	01-02	11	13:58	13:56		
7/16/2002	01-02	12	14:02	14:00		
7/16/2002	01-02	13	14:06	14:04		
7/16/2002	01-02	14	14:10	14:08		
7/16/2002	01-02	15	14:14	14:12		
7/16/2002	01-02	16	14:18	14:16		
7/16/2002	01-02	17	14:22	14:20		
7/16/2002	01-02	18	14:26	14:24		
7/16/2002	01-02	19	14:30	14:28		
7/16/2002	01-02	20	14:34	14:32		
7/16/2002	01-02	21	14:38	14:36		
7/16/2002	01-02	22	14:42	14:40		
7/16/2002	01-02	23	14:46	14:44		
7/16/2002	01-02	24	14:50	14:49		
7/16/2002	01-02	25	14:54	14:52		
7/16/2002	01-02	26	14:58	14:56		
7/16/2002	01-02	27	15:02	15:00		
7/16/2002	01-02	28	15:06	15:04		
7/16/2002	01-02	29	15:10	15:08		
7/16/2002	01-02	30	15:14	15:12		

65 Total vehicles recorded over 2 hours (33 passenger & 32 pick-ups)

\*Staff car driven slowly to avoid creating dust.

\*\*Local cars were driven slow (<10 mph) creating minimal dust.



## INITIAL STUDY - 7/17/02 Traffic Data

### Scenario 02-01 = 30 vph & 25\* mph (vehicle every 2 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)**
7/17/2002	02-01	1	12:06	12:07	12:29 Local Car, Slow
7/17/2002	02-01	2	12:09	12:11	
7/17/2002	02-01	3	12:13	12:15	
7/17/2002	02-01	4	12:17	12:19	
7/17/2002	02-01	5	12:21	12:23	
7/17/2002	02-01	6	12:25	12:27	
7/17/2002	02-01	7	12:29	12:31	
7/17/2002	02-01	8	12:33	12:35	
7/17/2002	02-01	9	12:37	12:39	
7/17/2002	02-01	10	12:41	12:43	
7/17/2002	02-01	11	12:45	12:47	
7/17/2002	02-01	12	12:49	12:51	
7/17/2002	02-01	13	12:53	12:55	
7/17/2002	02-01	14	12:57	12:59	
7/17/2002	02-01	15	13:01	13:03	
7/17/2002	02-01	16	13:05	13:07	
7/17/2002	02-01	17	13:09	13:11	
7/17/2002	02-01	18	13:13	13:15	
7/17/2002	02-01	19	13:17	13:19	
7/17/2002	02-01	20	13:21	13:23	
7/17/2002	02-01	21	13:25	13:27	
7/17/2002	02-01	22	13:29	13:31	
7/17/2002	02-01	23	13:33	13:35	
7/17/2002	02-01	24	13:37	13:39	
7/17/2002	02-01	25	13:41	13:43	
7/17/2002	02-01	26	13:45	13:47	
7/17/2002	02-01	27	13:49	13:51	
7/17/2002	02-01	28	13:53	13:55	
7/17/2002	02-01	29	13:57	13:59	
7/17/2002	02-01	30	14:01	14:03	

61 Total vehicles recorded over 2 hours (31 passenger & 30 pick-ups)

\*Radar indicated vehicle speed=22-23 mph

\*\*Local cars were driven slow (<10 mph) creating minimal dust. Local 10:29 driven fast creating dust.

### Scenario 02-02 = 30 vph & 25 mph (vehicle every 2 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)**
7/17/2002	02-02	1	15:11	15:09	15:20 Local Car, Slow
7/17/2002	02-02	2	15:15	15:13	15:47 Local Truck, Slow
7/17/2002	02-02	3	15:19	15:17	16:16 Local Car, Slow
7/17/2002	02-02	4	15:23	15:21	16:34 Local Car, Slow
7/17/2002	02-02	5	15:27	15:25	17:00 Local Car, Slow
7/17/2002	02-02	6	15:31	15:29	
7/17/2002	02-02	7	15:35	15:33	
7/17/2002	02-02	8	15:39	15:37	
7/17/2002	02-02	9	15:43	15:41	
7/17/2002	02-02	10	15:47	15:45	
7/17/2002	02-02	11	15:51	15:49	
7/17/2002	02-02	12	15:55	15:53	
7/17/2002	02-02	13	15:59	15:57	
7/17/2002	02-02	14	16:03	16:01	
7/17/2002	02-02	15	16:07	16:05	
7/17/2002	02-02	16	16:11	16:09	
7/17/2002	02-02	17	16:15	16:14	
7/17/2002	02-02	18	16:19	16:17	
7/17/2002	02-02	19	16:23	16:21	
7/17/2002	02-02	20	16:27	16:25	
7/17/2002	02-02	21	16:31	16:28	
7/17/2002	02-02	22	16:35	16:33	
7/17/2002	02-02	23	16:39	16:37	
7/17/2002	02-02	24	16:43	16:41	
7/17/2002	02-02	25	16:47	16:45	
7/17/2002	02-02	26	16:51	16:49	
7/17/2002	02-02	27	16:55	16:53	
7/17/2002	02-02	28	16:59	16:57	
7/17/2002	02-02	29	17:03	17:01	
7/17/2002	02-02	30	17:07	17:05	

65 Total vehicles recorded over 2 hours (34 passenger & 31 pick-ups)

## INITIAL STUDY - 7/18/02 Traffic Data

---

**Scenario 03-01 = 10 vph & 10 mph**  
(vehicle every 6 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)
7/18/2002	03-01	1	8:25	8:31	8:45 Local Car, Slow
7/18/2002	03-01	2	8:36	8:42	8:50 Local Truck, Slow
7/18/2002	03-01	3	8:48	8:54	9:11 Local Car, Slow
7/18/2002	03-01	4	9:00	9:06	9:12 Counted - Local Car, 10 mph
7/18/2002	03-01	5	9:24	9:30	9:13 Local Car, Slow
7/18/2002	03-01	6	9:36	9:42	9:14 Local Motorcycle, Slow
7/18/2002	03-01	7	9:48	9:54	9:15 Local Truck, Slow
7/18/2002	03-01	8	10:00	10:06	
7/18/2002	03-01	9	10:12	10:18	

25 Total vehicles recorded over 2 hours (13 passenger, 11 pick-ups, & 1 motorcycle)

Note: 9:18 run skipped because of high vehicle frequency in past 5 minutes

\*\*Local cars were driven slow (<10 mph) creating minimal dust. Local 10:29 driven fast creating dust.

## POST RESURFACING - 8/19/03 Traffic Data

### Scenario 2-01 = 10 vph & 10 mph (vehicle every 6 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)
8/19/2003	2-01	1	10:54 AM	11:06 AM	11:00 AM Counted - Local Ford Tempo
8/19/2003	2-01	2	11:12 AM	11:24 AM	11:13 AM Counted Local Pick-up (@ ~15mph)
8/19/2003	2-01	3	11:30 AM	11:36 AM	11:38 AM Counted Local Pick-up (@ ~15mph)
8/19/2003	2-01	4	11:48 AM	11:54 AM	12:26 PM Counted Local Car
8/19/2003	2-01	5	12:00 PM	12:06 PM	
8/19/2003	2-01	6	12:12 PM	12:18 PM	
8/19/2003	2-01	7	12:24 PM	12:36 PM	
8/19/2003	2-01	8	12:42 PM	12:48 PM	

20 Total vehicles recorded over 2 hours (10 passenger & 10 pick-ups)

Note: Speeds of local traffic is approximate and was not measured.

### Scenario 2-02 = 30 vph & 25 mph (vehicle every 2 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)
8/19/2003	2-02	1	1:58 PM	2:00 PM	2:12 PM Counted Local - Car (@ 10 mph)
8/19/2003	2-02	2	2:02 PM	2:04 PM	2:36 PM Counted Local Car (@ 10 mph)
8/19/2003	2-02	3	2:06 PM	2:08 PM	2:41 PM Counted Local Car (@ 15 mph)
8/19/2003	2-02	4	2:10 PM	2:14 PM	2:54 PM Counted Local Car (@ 15 mph)
8/19/2003	2-02	5	2:16 PM	2:18 PM	3:07 PM Counted Local Car (@ 15 mph)
8/19/2003	2-02	6	2:20 PM	2:22 PM	3:23 PM Counted Local Car (@ 15 mph)
8/19/2003	2-02	7	2:24 PM	2:26 PM	3:43 PM Counted Local Car (@ 10 mph)
8/19/2003	2-02	8	2:28 PM	2:30 PM	3:49 PM Counted Local Car (@ 10 mph)
8/19/2003	2-02	9	2:32 PM	2:34 PM	3:54 PM Counted Local Car (@ 10 mph)
8/19/2003	2-02	10	2:38 PM	2:40 PM	
8/19/2003	2-02	11	2:44 PM	2:46 PM	
8/19/2003	2-02	12	2:48 PM	2:50 PM	
8/19/2003	2-02	13	2:52 PM	2:56 PM	
8/19/2003	2-02	14	2:58 PM	3:00 PM	
8/19/2003	2-02	15	3:02 PM	3:04 PM	
8/19/2003	2-02	16	3:06 PM	3:10 PM	
8/19/2003	2-02	17	3:12 PM	3:14 PM	
8/19/2003	2-02	18	3:16 PM	3:18 PM	
8/19/2003	2-02	19	3:20 PM	3:22 PM	
8/19/2003	2-02	20	3:26 PM	3:28 PM	
8/19/2003	2-02	21	3:30 PM	3:32 PM	
8/19/2003	2-02	22	3:34 PM	3:36 PM	
8/19/2003	2-02	23	3:38 PM	3:40 PM	
8/19/2003	2-02	24	3:42 PM	3:46 PM	
8/19/2003	2-02	25	3:48 PM	3:51 PM	
8/19/2003	2-02	26	3:53 PM		

60 Total vehicles recorded over 2 hours (35 passenger & 25 pick-ups)

Note: Speeds of local traffic is approximate and was not measured.

## POST RESURFACING - 8/20/03 Traffic Data

### Scenario 3-01 = 10 vph & 10 mph (vehicle every 6 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)
8/20/2003	3-01	1	10:29 AM	10:31 AM	10:44 AM Counted Local Motorcycle (@ 10 mph)
8/20/2003	3-01	2	10:37 AM	10:43 AM	10:53 AM Counted Local Car (@ 10 mph)
8/20/2003	3-01	3	11:01 AM	11:06 AM	11:10 AM Counted Local Car (@ 10 mph)
8/20/2003	3-01	4	11:18 AM	11:25 AM	11:18 AM Counted Local Car (@ 15 mph)
8/20/2003	3-01	5	11:31 PM	11:37 PM	
8/20/2003	3-01	6	11:43 PM	11:49 PM	
8/20/2003	3-01	7	11:55 PM	12:01 PM	
8/20/2003	3-01	8	12:07 PM	12:13 PM	

20 Total vehicles recorded over 2 hours (12 passenger & 8 pick-ups)

Note: Speeds of local traffic is approximate and was not measured.

### Scenario 3-02 = 30 vph & 25 mph (vehicle every 2 min)

Date	Scenario	Pass	Car	Truck	Misc - Vehicles (local etc.)
8/20/2003	3-02	1	1:42 PM	1:44 PM	2:20 PM Counted local Car (@ 15 mph)
8/20/2003	3-02	2	1:46 PM	1:48 PM	2:42 PM Counted Local Car (@ 15 mph)
8/20/2003	3-02	3	1:50 PM	1:52 PM	2:42 PM Counted Local Car (@ 10 mph)
8/20/2003	3-02	4	1:54 PM	1:56 PM	2:42 PM Counted Local Car (@ 10 mph)
8/20/2003	3-02	5	1:58 PM	2:00 PM	2:48 PM Counted Local Car (@ 10 mph)
8/20/2003	3-02	6	2:02 PM	2:04 PM	3:02 PM Counted Local Truck (@ 25 mph)
8/20/2003	3-02	7	2:06 PM	2:08 PM	3:05 PM Counted Local Car (@ 15 mph)
8/20/2003	3-02	8	2:10 PM	2:12 PM	3:27 PM Counted Local Car (@ 10 mph)
8/20/2003	3-02	9	2:14 PM	2:16 PM	3:28 PM Counted local Car (@ 20 mph)
8/20/2003	3-02	10	2:18 PM	2:22 PM	
8/20/2003	3-02	11	2:24 PM	2:26 PM	
8/20/2003	3-02	12	2:28 PM	2:30 PM	
8/20/2003	3-02	13	2:32 PM	2:34 PM	
8/20/2003	3-02	14	2:36 PM	2:38 PM	
8/20/2003	3-02	15	2:40 PM	2:46 PM	
8/20/2003	3-02	16	2:50 PM	2:52 PM	
8/20/2003	3-02	17	2:54 PM	2:56 PM	
8/20/2003	3-02	18	2:58 PM	3:00 PM	
8/20/2003	3-02	19	3:04 PM	3:06 PM	
8/20/2003	3-02	20	3:08 PM	3:10 PM	
8/20/2003	3-02	21	3:12 PM	3:14 PM	
8/20/2003	3-02	22	3:16 PM	3:18 PM	
8/20/2003	3-02	23	3:20 PM	3:22 PM	
8/20/2003	3-02	24	3:24 PM	3:26 PM	
8/20/2003	3-02	25	3:28 PM	3:34 PM	
8/20/2003	3-02	26	3:38 PM		

60 Total vehicles recorded over 2 hours (34 passenger & 26 pick-ups)

Note: Speeds of local traffic is approximate and was not measured.

## **Appendix F – Meteorological (MET) Data**

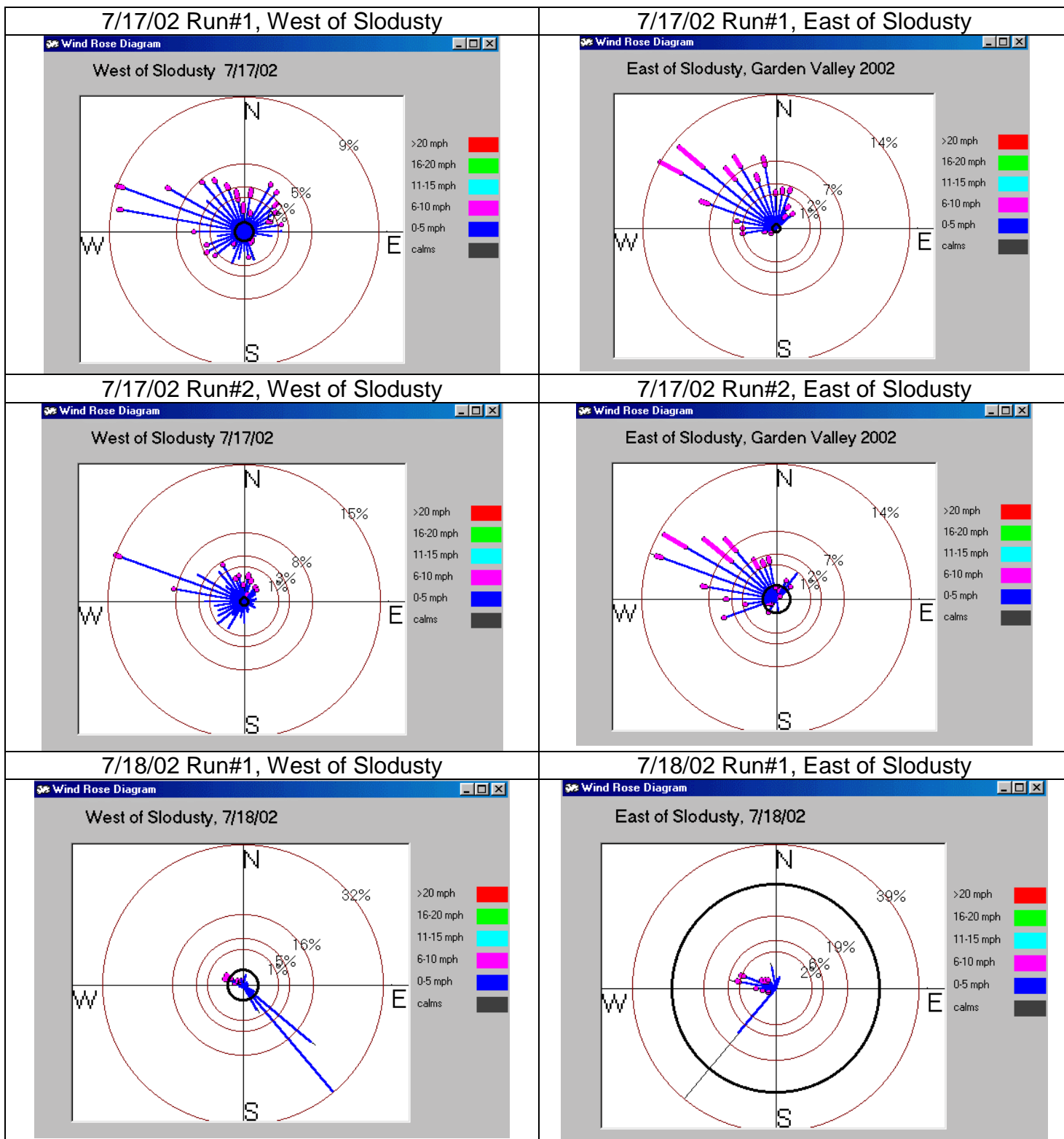
## Average Wind Speed and Direction Results for Initial Study and Post Resurfacing

<i>Study</i>	<b>Initial Study</b>					
<i>Date</i>	7/17/02 Run#1		7/17/02 Run#2		7/18/02 Run#1	
<i>Traffic Scenario</i>	25 mph / 30 vph		25 mph / 30 vph		10 mph / 10 vph	
<i>Time</i>	12:06p to 2:05p		3:09p to 5:09p		8:25a to 10:25a	
<i>Met. Station location</i>	West	East	West	East	West	East
<i>Avg. wind speed (mph)</i>	2.9	3.5	2.6	3.3	2.2	2.6
<i>Avg. wind direction (degrees)</i>	335	321	313	309	80	268
<i>Temperature (Fahrenheit)</i>	87.3	89.1	91.0	88.3	80.6	84.2
<i>Relative Humidity (%)</i>	27.5	28.6	26.4	28.0	27.0	29.0

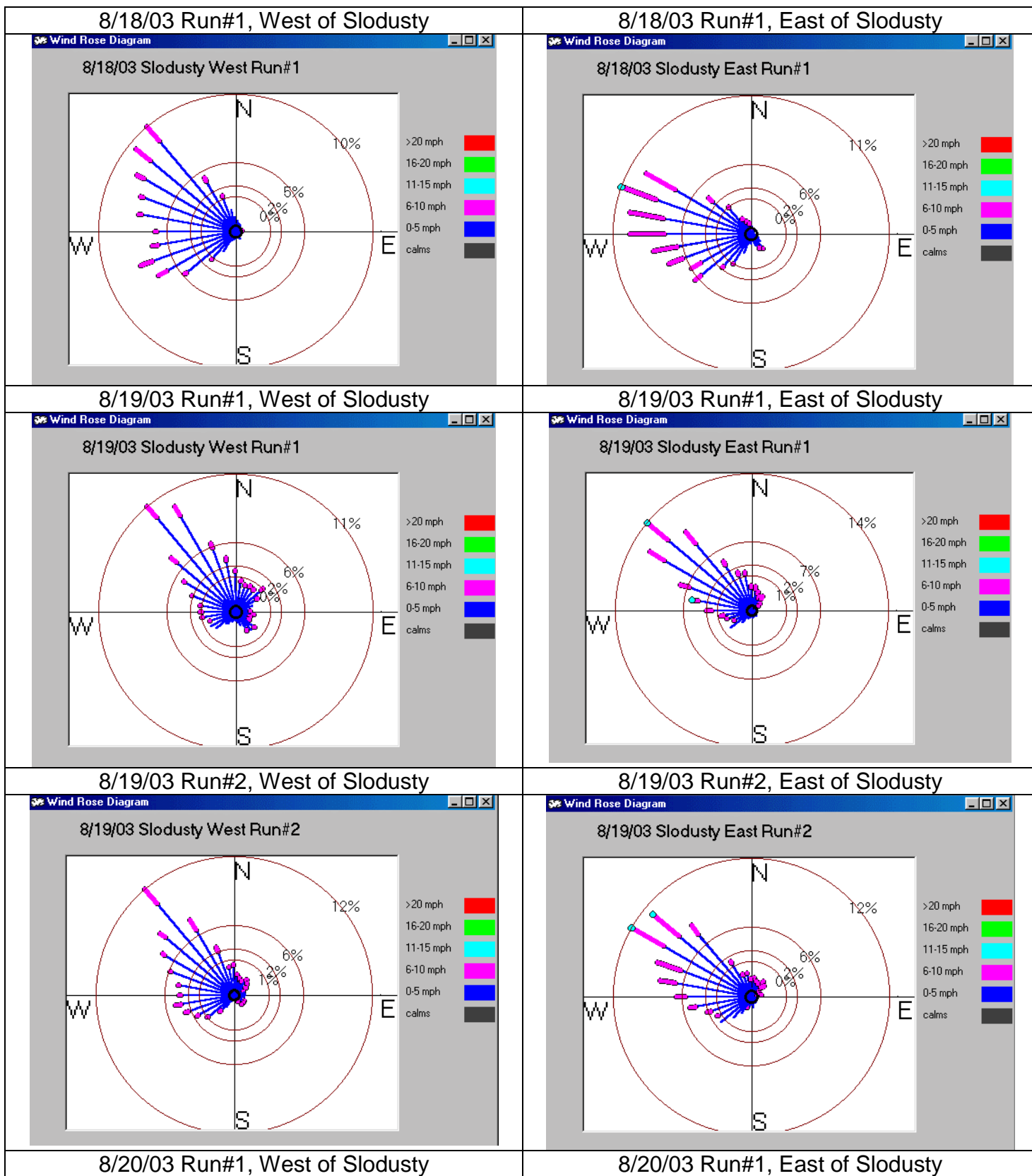
<i>Study</i>	<b>Post Resurfacing</b>					
<i>Date</i>	8/18/03 Run#1		8/19/03 Run#1		8/19/03 Run#2	
<i>Traffic Scenario</i>	No Simulation		10 mph / 10 vph		25 mph / 30 vph	
<i>Time</i>	1:00p to 3:00p		10:50a to 12:50p		1:55 to 3:55p	
<i>Met. Station location</i>	West	East	West	East	West	East
<i>Avg. wind speed (mph)</i>	2.9	3.3	2.6	3.1	2.6	3.1
<i>Avg. wind direction (degrees)</i>	293	289	341	313	316	305
<i>Temperature (Fahrenheit)</i>	Not Measured		90.0		87.0	
<i>Relative Humidity (%)</i>	Not Measured		24.0		19.0	

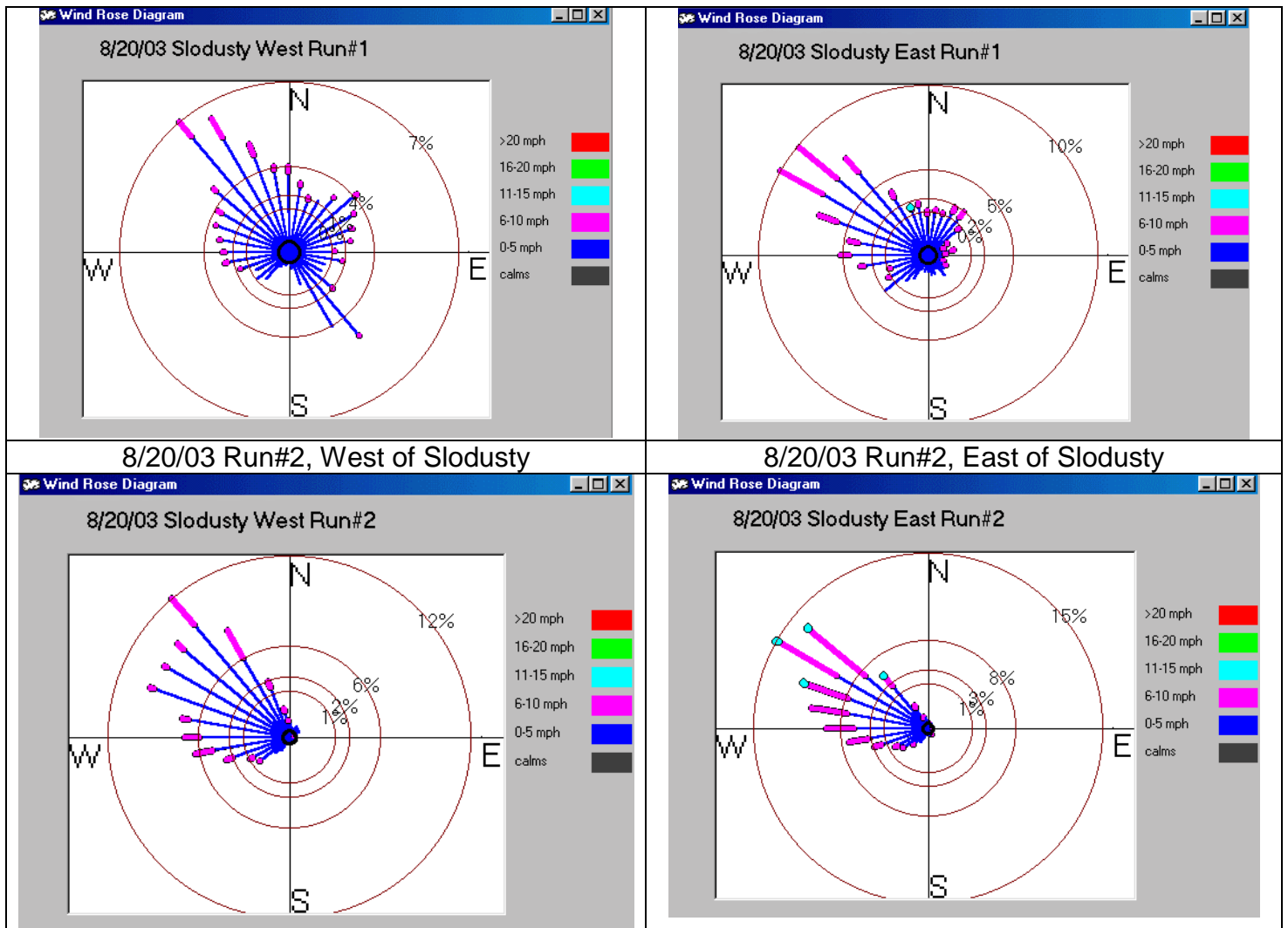
<i>Study</i>	<b>Post Resurfacing</b>			
<i>Date</i>	8/20/03 Run#1		8/20/03 Run#2	
<i>Traffic Scenario</i>	10 mph / 10 vph		25 mph / 30 vph	
<i>Time</i>	10:25a to 12:25p		1:40p to 3:40p	
<i>Met. Station location</i>	West	East	West	East
<i>Avg. wind speed (mph)</i>	2.4	2.9	3.1	3.9
<i>Avg. wind direction (degrees)</i>	2	327	300	290
<i>Temperature (Fahrenheit)</i>	90.0		91.0	
<i>Relative Humidity (%)</i>	27.0		24.0	

## Wind Roses for Initial study and Post Resurfacing

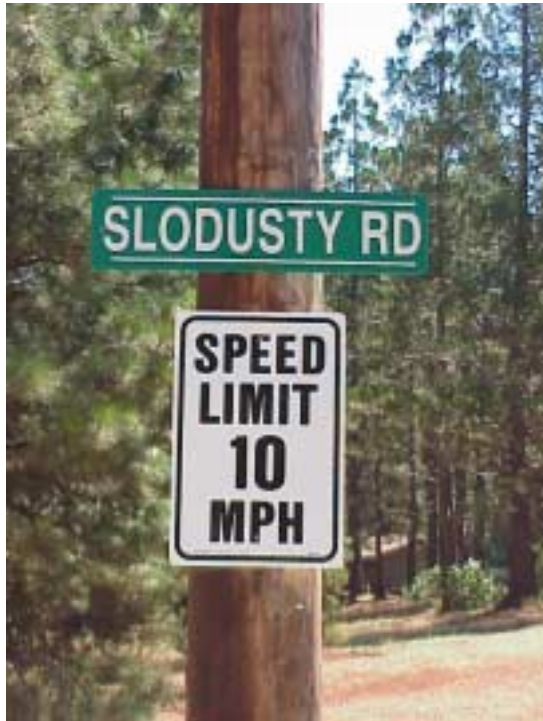








## **Appendix G – Field Pictures**



**Slodusty Road Test Area  
(Initial Study, July 2002)**





**Slodusty Rd. – Road bed in July 2002  
(Pre-Resurfacing)**



**Slodusty Rd. – Road bed in August 2003  
(following Resurfacing)**



**Volpe High Volume Air Sampling  
Pump & Battery**



**DTSC High Volume Air Sampling  
Pump & Battery**



**Stationary Air Sampler Set-up**





**Rotometer (Flow Control)  
Stationary Air Sampler Set-up**



**DTSC Particulate Monitor  
(Initial Study, July 2002)**



**Transportable Automated Meteorological  
Station (TAMS)  
(Initial Study, July 2002)**

**Gill WindObserver II Ultra Sonic  
Anemometer  
(Post Resurfacing, August 2003)**





**Stationary Air Sampler – West Side of Slodusty Rd  
(Initial Study, July 2002)**



**Stationary Air Sampler – West Side of Slodusty Rd  
(Post Resurfacing, August 2003)**





**25 mph / 30 vph Traffic Scenario  
(Initial Study, July 2002)**





**25 mph / 30 vph Traffic Scenario  
(Initial Study, July 2002)**







**25 mph / 30 vph Traffic Scenario  
(Post Resurfacing, August 2003)**



## **Appendix H – Laboratory Record of Modification**



**Record /  
Request for Modification  
To  
Laboratory Activities  
L-000001**

**Instructions to Requester: Fax to contacts at bottom of form for review and approval.**

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, ASTM D5755, EPA/540/2-90/005a, Other: \_\_\_\_\_

Requester: Jeanne Orr Title: President  
Company: Reservoirs Environmental, Inc. Date: June 17, 2003

**Description of Modification:**

Modification and clarification to the Transmission Electron Microscopy analysis by the AHERA protocol of air samples from the DTSC Project. The purpose of the attached is to document permanent historic modifications & clarifications.

**Reason for Modification:**

The samples from this project were heavily loaded with asbestos structures and debris. Counting these samples using the AHERA stopping rules would require excessive microscope and analyst time. To further clarify the data recorded by the analysts.

**Potential Implications of this Modification:**

Modifications reflect changes necessary to clarify AHERA requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are documentation of procedures, increased efficiency and enabling completion of analysis in a reasonable time.

Laboratory Applicability (circle one): All Individual(s) Reservoirs Environmental, Inc.

**Duration of Deviation (circle one):**

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

Permanent (complete Proposed Modification Section) Effective Date: HISTORIC

**Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):**

Please see the attached for the description of the TEM-AHERA clarifications/modifications

Technical Review: Jeanne Orr Date: 7/7/03  
(Laboratory Manager or designate)

Project Review and Approval: Mark [Signature] Date: 7/7/03  
(Volpe: Project Technical Lead or designate)

## AIR ANALYSES, AHERA

- 1. Modification:** AHERA stopping rules require a minimum of 4 grid openings or 50 structures counted. In the case of abundant fibers/structures on these samples, less than 4 grid openings were counted on some of the samples. In all cases the analyst examined two sample grids to assure even loading between the grids.
- 2 Clarification:** Record the following in the “structure type” column on the Electronic Data Delivery (EDD) Data Entry2 spreadsheet.

Chrysotile Fiber	CF
Chrysotile Bundle	CB
Chrysotile Cluster	CC
Chrysotile Matrix	CM
Chrysotile Bundle in Matrix	CBM
Chrysotile Fiber in Matrix	CFM
Chrysotile Cluster in Matrix	CCM
Chrysotile Bundle in a Cluster	CBC
Chrysotile Bundle/Cluster/Matrix	CBCM
Chrysotile Bundle/Cluster/Matrix , Crossing Grid Bar	CBCMX
Chrysotile Bundle in Matrix, Crossing Grid Bar	CBMX

If the analyst determines that additional information is needed to describe a structure, comments pertaining to the structure in addition to a sketch will be recorded in the comments column.

- 3. Clarification:** The AHERA method requires the analyst to “record the length category and structure type classification on the count sheet after the field number and fiber number”. As a clarification to this the actual length and width of individual fibers, bundles, compact clusters and compact matrices will be recorded. For disperse clusters and matrices the length of the longest protruding structure will be recorded (it will not be necessary to record all matrix structures as in ISO 10312). Structure measurements may be recorded in screen units or in microns.
- 4. Modification:** AHERA refers to two recording definitions when no fibers are detected in the grid opening, NSD in the text and NFD on the example count sheet. For this project, ND (none detected) will be used.
- 5. Clarification:** One of the illustrations in the AHERA rule for bundles is unclear and looks like three fibers. The AHERA written definition for bundles will be used.
- 6. Modification:** Grid rejection criteria of >25% obscured area will be used.
- 7. Modification:** Cassettes with a 0.8 µm pore size and no 5.0 µm diffuser filter (PCM cassettes) may be used for AHERA sample collection.
- 8. Modifications/Clarifications:** Structure counting and recording:
  - Non-asbestos structures are not being counted or recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.

- b. Structures that are non-countable (e.g. for reasons such as insufficient aspect ratio or no exposed termination) are being recorded for informational purposes only, but not included in structure density or structure concentration calculations. A zero will be recorded in the “Total” column to indicate the structure should be excluded from the concentration calculations.
- c. A maximum of ten grid openings are analyzed for an AHERA analysis, regardless of sample volume. In some instances the specified analytical sensitivity of 0.005 s/cc may not be obtained.

## **Appendix I – Example TEM Electronic Data Deliverable (EDD)**



# INSTRUCTIONS

This spreadsheet is designed to record the raw fiber counts for air and dust samples analyzed by TEM25

This is version TEM26c

## Raw Data Recording

Raw data are to be recorded by the analyst in hard copy using Lab sheet 1 and as many Lab Continuation pages as needed

## Data Entry

Data on the hard copy lab sheets are to be entered electronically on data entry sheets 1 and 2

Areas for data entry are highlighted in **YELLOW**



OR are indicated by a **PULL-DOWN MENU**



Cells that are shaded gray do not require any data input

Cells that are shaded red either require data input or contain an apparent data inconsistency

**Do not enter data in any other location!**

Avoid the drag and drop method for copying.

Enter all values individually

## File Save

After entering all data on Data Entry 1 (or any time thereafter), SAVE THE FILE by clicking on the macro button located on data Entry 1 or Data Entry 2.

The file name is generated automatically by concatenating information provided in Data Entry 1, including

a) EPA Index ID, b) Lab Sample Number, c) Counting Rule, d) Analysis Date, e) Prep method, and f) QA type.

For example, if the EPA number is **2-012345** and the lab ID number is **abc-1234**, the method is **AHERA**, the analysis date is **03-04-01**, the prep method is **Direct**, and the QA type is **Recount Same**

the file name will be: "**2-012345\_abc-1234\_AHERA\_03-04-01\_DRS.XLS**"

opened:

TEM25.

If you open TEM25 from Explorer, then the file will be saved in your default directory for Excel (usually this is C:\Documents and Settings\My Documents)

**TEM - General Counting Rules**

Follow established project-specific counting and stopping rules.  
Do not count non-asbestos material (NAM) structures.

**ISO 10312 Counting Rules:**

Enter all asbestos structures into Data Entry 2 regardless of dimensions.

If a structure is non-countable (e.g.: crosses the grid bar), it should be identified with a "0" in the Total column.

All other countable structures should be identified with a non-zero sequential number in the Total column.

*NOTE: Data Entry 2, column R is designed to flag any potential data entry errors associated with countable/non-countable assignments.*

**AHERA/ASTM Counting Rules:**

Valid Structure Types are Fiber (F), Matrix (M), Cluster (C), and Bundle (B).

Enter Fibers (F) only if the Aspect Ratio is greater than or equal to 5:1.

The lab analyst is responsible for identifying if a complex structure (M, C, B) is countable under AHERA.

If a non-countable structure is entered into Data Entry 2, it should be identified with a "0" in the Total column.

All other countable structures should be identified with a non-zero sequential number in the Total column.

*NOTE: Data Entry 2, column R is designed to flag any potential data entry errors associated with countable/non-countable assignments.*

**DATA ENTRY INSTRUCTIONS**

Data Item	Description	Notes
<b>DATA ENTRY 1</b>		
Laboratory Name	Name of lab performing analysis	Use a standard name for all sheets.
Instrument	Instrument used for analysis	
Voltage (KV)	Voltage used for analysis	
Magnification	Magnification used for analysis	
Grid opening area	Size of grid opening	Enter as a value in units of mm <sup>2</sup> . This field should not contain any text.
Scale: 1L =	Scale adjustment factor for length	Enter 1.0 if dimensions are expressed as um. Otherwise, enter um per unit screen length.
Scale: 1D =	Scale adjustment factor for width	Enter 1.0 if dimensions are expressed as um. Otherwise, enter um per unit screen width.
Primary filter area	Size of primary effective filter area (EFA)	Enter as a value in units of mm <sup>2</sup> . This field should not contain any text.
Secondary filter area	Size of secondary effective filter area (EFA)	Enter as a value in units of mm <sup>2</sup> . This field should not contain any text.
Category	Sample type	Select the appropriate category (Field, Replicate, Duplicate, Blank) from the pull-down list.
Filter Status	Status of the sample filter	Select the appropriate category (Analyzed, Overloaded, Damaged, Missing, Cancelled) from the pull-down list.
EPA Index ID	Unique sample identifier	Enter the EPA Index ID exactly as it appears on the sample (ie: 2-00013). This field should not contain any spaces or a lab QA type suffix.
Sample Type	Sample media	Select the appropriate category (Air, Dust) from the pull-down list.
Air volume (L) or dust area (cm2)	Air volume (L) or dust area (cm2)	If the sample media is air, enter the air volume in units of L. If the sample media is dust, enter the dust sample area in units of cm <sup>2</sup> . This field should not contain any text.
Date Received	Date sample was received by lab	Enter as a valid date with the format MM/DD/YY.
Lab Job Number	Job number assigned to analysis by lab	
Lab Sample Number	Sample identifier as designated by lab	If the sample is a lab QA Type, DO NOT add the QA suffix to the Lab Sample ID.
Number of grids prepared	Number of grids prepared by lab	
Prepared by	Name of lab preparation personnel	
Preparation date	Date sample was prepared	Enter as a valid date with the format MM/DD/YY.
EPA COC Number	EPA chain of custody number	
Analyzed by	Name of analyst	
Date Analyzed	Date sample was analyzed by lab	Enter as a valid date with the format MM/DD/YY.
Prep	Analysis Prep Type	Select the appropriate Prep type (Direct, Indirect, Indirect - Ashed) from the pull-down list. Note: "Indirect - Ashed" is not a valid prep type for air samples.
Counting Rules	Counting Rules utilized to analyze sample	Select the appropriate Counting Rule from the pull-down list. If the sample media is dust, select either ASTM-AHERA or ISO. If the sample media is air, select either AHERA or ISO.
Grid Storage Location	Grid storage location identifier	
F-factor Calculation:		For Direct prep, f-factor is set to equal 1 and specific f-factor calculations are not required.
<i>For Air:</i>		
Fraction of primary filter used	Fraction of primary filter used	Enter the fraction as a value. This field should not contain any text.
Total resuspension volume (mL)	Total resuspension volume (mL)	Enter as a value in units of mL. This field should not contain any text.
Volume filtered for secondary prep (mL)	Volume filtered for secondary prep (mL)	Enter as a value in units of mL. This field should not contain any text.
<i>For Dust (not ashed):</i>		
Total resuspension volume (mL)	Total resuspension volume (mL)	Enter as a value in units of mL. This field should not contain any text.
Volume applied to secondary filter (mL)	Volume applied to secondary filter (mL)	Enter as a value in units of mL. This field should not contain any text.
<i>For Dust (ashed):</i>		
Total resuspension volume, pre-ashing (mL)	Total resuspension volume, pre-ashing (mL)	Enter as a value in units of mL. This field should not contain any text.
Volume applied to filter for ashing (mL)	Volume applied to filter for ashing (mL)	Enter as a value in units of mL. This field should not contain any text.
Fraction of filter that was ashed	Fraction of filter that was ashed	Enter the fraction as a value. This field should not contain any text.
Volume used to resuspend ashed residue (mL)	Volume used to resuspend ashed residue (mL)	Enter as a value in units of mL. This field should not contain any text.
Volume applied to secondary filter (mL)	Volume applied to secondary filter (mL)	Enter as a value in units of mL. This field should not contain any text.
QA Type	Lab quality control code	Select the appropriate laboratory QA type (Not QA, Re-count Same, Re-count Different, Repreparation, Verified Analysis, Reconciliation, Lab Blank, Interlab) from the pull-down list.
Comments	Sample/Analysis comments	
<b>DATA ENTRY 2</b>		
Data Entry by	Name of data entry personnel	
Date Entry Date	Date results were entered	Enter as a valid date with the format MM/DD/YY.
QA by	Name of QA personnel	
QA Date	Date results were QA'ed	Enter as a valid date with the format MM/DD/YY.
Grid	Grid identifier	Enter the appropriate grid in the first row only. If there are multiple rows associated with the grid, it is not necessary to repeat the grid in every row.
Grid Opening	Grid opening location identifier	Enter the appropriate grid opening in the first row only. If there are multiple rows associated with the grid opening, it is not necessary to repeat the grid opening in every row. Indicate grid openings that DO NOT count crysotile with an "*" following the grid opening name (eg: A-4*).
Structure Type	Structure Type code	Enter the structure type code. For ISO, see the analytical method for valid structure type codes. For AHERA, valid structure types are Fiber (F), Matrix (M), Cluster (C), and Bundle (B).
Primary	Primary Structure identifier	For ISO, enter a non-zero sequential number for any unique primary structure in the first row only. If there are multiple rows associated with the primary structure, it is not necessary to repeat the primary number in every row. This field is not used for AHERA or ASTM.
Total	Total Structure identifier	Assign a "0" to any non-countable or excluded structure and a non-zero number to any countable structure. **See specific instructions above for details on populating this field for ISO and AHERA/ASTM.
Length	Structure length	Enter dimensions either in absolute units (um) or in screen units.
Width	Structure width	Enter dimensions either in absolute units (um) or in screen units.
Identification	Identification	
Mineral Class	Description of the structure mineral class type	Enter a "1" in the appropriate column; choices include "LA" (Libby amphibole), "OA" (other amphibole), "C" (chrysotile). You may only select one mineral class type for each structure. NOTE: Do not enter "NAM" (non-asbestos material) structures.
Comments	Structure comments	
Sketch	Sketch	Enter a "1" in this column if yes, otherwise leave this field blank.
Photo	Photo	Enter a "1" in this column if yes, otherwise leave this field blank.
EDS	EDS	Enter a "1" in this column if yes, otherwise leave this field blank.

FILE NAME: F3-00023\_96817-808540\_AHERA\_10-18-03\_D.xls

**DTSC**  
**TEM Asbestos Structure Count**

Laboratory name:	Reservoirs
Instrument	JEOL 100 C
Voltage (KV)	100KV
Magnification	20,000
Grid opening area (mm2)	0.0110
Scale: 1L =	0.280
Scale: 1D =	0.056
Primary filter area (mm2)	385.0
Secondary Filter Area (mm2)	201.0
Category	Field ▼
Filter Status	Analyzed ▼

EPA Sample Number:	F3-00023
Sample Type	Air ▼
Air volume (L) or dust area (cm2)	484
Date received by lab	8/26/2003
Lab Job Number:	96817
Lab Sample Number:	96817-808540
Number of grids prepared	3
Prepared by	LK
Preparation date	10/9/2003
EPA COC Number	F-09

Analyzed by	J. Orr
Analysis date	10/18/2003
Prep	Direct ▼
Counting rules	AHERA (Air) ▼
Grid storage location	96817
Enter the appropriate data in the cells to the right to calculate the F-factor >>>>>	
F- factor	1.000
QA Type	Not QA ▼

**COMMENTS**

ERROR CHECK

OK - No errors found

QA by:	J. Orr
QA date:	11/3/2003

**TEM Asbestos Structure Count -- AHERA****SAMPLE ID**

Status Analyzed  
 EPA Sample Number F3-00023  
 QA Sample Type Not QA  
 Lab Sample Number 96817-808540  
 Sample Type Air  
 Category Field  
 Prep Direct  
 Counting rules AHERA

**PARAMETERS**

Number of Grid Openings (amphibole) 10  
 Number of Grid Openings (chrysotile) 10  
 Area of grid opening (mm2) 0.011  
 F Factor 1.00E+00  
 Effective primary filter area (mm2) 385.0  
 Effective secondary filter area (mm2) 201.0  
 Volume (L) or Sample Area (cm2) 484  
 Area counted (mm2) for LA/OA 0.110  
 Area counted (mm2) for C 0.110

**TOTAL COUNTS**

Total Structures	7
Total Asbestos structures	7
Total NAM	0
Total AHERA structures	7
Total Non-AHERA asbestos structures	0

**COUNTS (based on countable AHERA structures only)**

	LA	OA	C	Total
AHERA Fibers (< 5 um)	0	0	7	7
AHERA Fibers (>= 5 um)	0	0	0	0

**AIR CONCENTRATION (s/cc)**

	LA	OA	C	Total	
Loading on primary filter (s/mm2)	<9.1E+00	<9.1E+00	6.4E+01	6.4E+01	(if Total = ND, DL is based on LA)
Air Conc (s/cc)	<7.2E-03	<7.2E-03	5.1E-02	5.1E-02	(if Total = ND, DL is based on LA)

**DETECTION LIMITS**

	LA/OA	C
Loading on primary filter (s/mm2)	9.1E+00	9.1E+00
Sensitivity (s/cc)	7.2E-03	7.2E-03

## **Appendix J – TEM, AHERA**

### **(analytical method)**



**THIS DATA CURRENT AS OF THE FEDERAL REGISTER DATED MARCH 26, 2002**

## **40 CFR - CHAPTER I - PART 763**

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### **Appendix A to Subpart E -- Interim Transmission Electron Microscopy Analytical Methods -- Mandatory and Nonmandatory -- and Mandatory Section to Determine Completion of Response Actions**

#### **I. Introduction**

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for asbestos by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis. EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

#### **II. Mandatory Transmission Electron Microscopy Method**

##### **A. Definitions of Terms**

1. *Analytical sensitivity* -- Airborne asbestos concentration represented by each fiber counted under the

electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm<sup>3</sup>.

2. *Asbestiform* -- A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio* -- A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle* -- A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area* -- A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm<sup>2</sup> for any single preparation for that same area.

6. *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 must have more than two intersections.

7. *ED* -- Electron diffraction.

8. *EDXA* -- Energy dispersive X-ray analysis.

9. *Fiber* -- A structure greater than or equal to 0.5  $\mu$ m in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. *Grid* -- An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. *Intersection* -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. *Laboratory sample coordinator* -- That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. *Filter background level* -- The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm<sup>2</sup>.



14. *Matrix* -- Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. *NSD* -- No structure detected.

16. *Operator* -- A person responsible for the TEM instrumental analysis of the sample.

17. *PCM* -- Phase contrast microscopy.

18. *SAED* -- Selected area electron diffraction.

19. *SEM* -- Scanning electron microscope.

20. *STEM* -- Scanning transmission electron microscope.

21. *Structure* -- a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. *S/cm<sup>3</sup>* -- Structures per cubic centimeter.

23. *S/mm<sup>2</sup>* -- Structures per square millimeter.

24. *TEM* -- Transmission electron microscope. B. Sampling

1. The sampling agency must have written quality control procedures and documents which verify compliance.

2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3, and 5 of Unit II.J.).

3. Sampling for airborne asbestos following an abatement action must use commercially available cassettes.

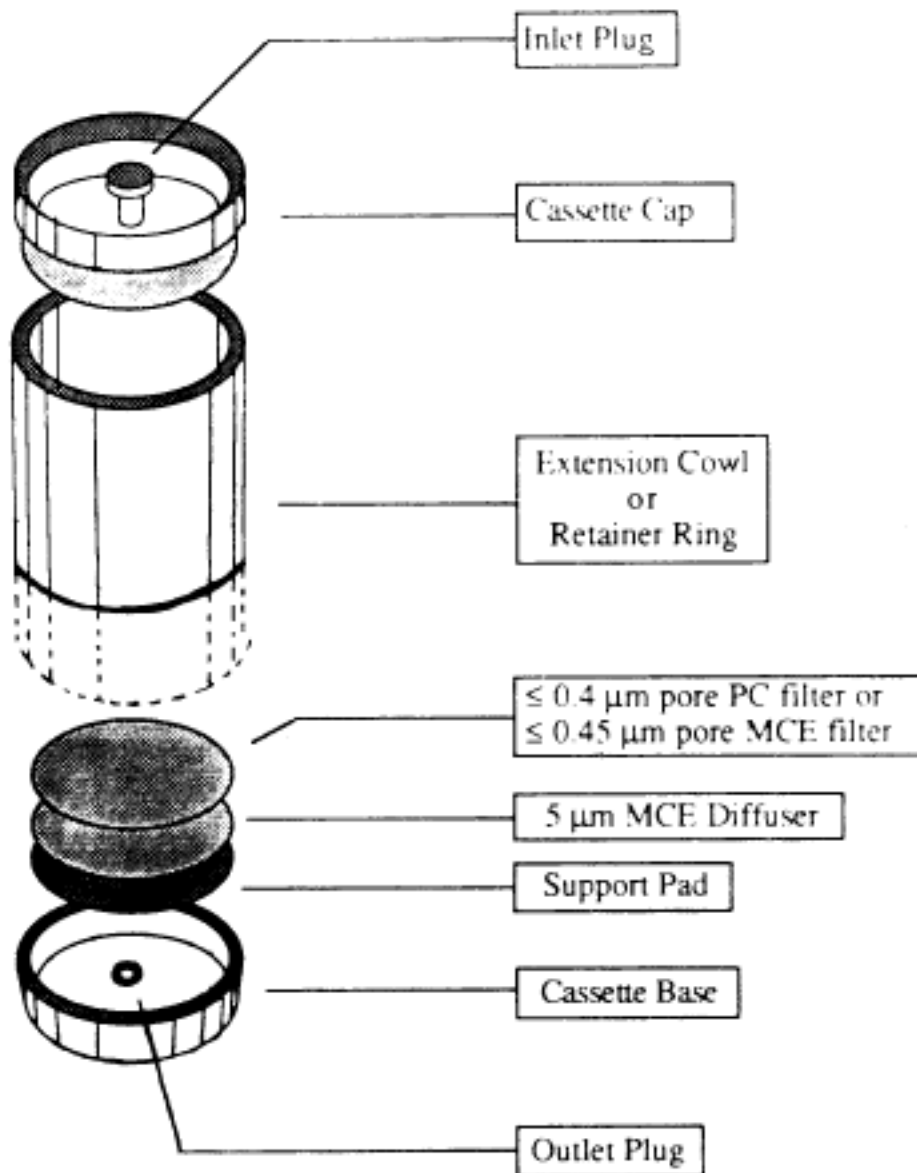
4. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm<sup>2</sup> for that same area is acceptable for this method.

5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to 0.4 •m or mixed cellulose ester having a pore size less than or equal to 0.45 •m.

6. Place these filters in series with a 5.0 •m backup filter (to serve as a diffuser) and a support pad. See

the following Figure 1:

FIGURE I--SAMPLING CASSETTE CONFIGURATION



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7. Reloading of used cassettes is not permitted.
8. Orient the cassette downward at approximately 45 degrees from the horizontal.
9. Maintain a log of all pertinent sampling information.

10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.
11. Record all calibration information.
12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.
14. The final plastic barrier around the abatement area remains in place for the sampling period.
15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.B.7.d.)
16. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.
17. A minimum of 13 samples are to be collected for each testing site consisting of the following:
  - a. A minimum of five samples per abatement area.
  - b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.
  - c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:
    - i. Near the entrance to each abatement area.
    - ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)
  - d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.
18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

19. The following Table I specifies volume ranges to be used:

TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS  
(0.0057 mm<sup>2</sup>) THAT NEED TO BE ANALYZED TO  
MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC  
BASED ON VOLUME AND EFFECTIVE FILTER AREA

Effective Filter Area 385 sq mm		Effective Filter Area 855 sq mm	
Volume (liters)	# of grid openings	Volume (liters)	# of grid openings
560	24	1,250	24
600	23	1,300	23
700	19	1,400	21
800	17	1,600	19
900	15	1,800	17
1,000	14	2,000	15
1,100	12	2,200	14
1,200	11	2,400	13
1,300	10	2,600	12
1,400	10	2,800	11
1,500	9	3,000	10
1,600	8	3,200	9
1,700	8	3,400	9
1,800	8	3,600	8
1,900	7	3,800	8
2,000	7	4,000	8
2,100	6	4,200	7
2,200	6	4,400	7
2,300	6	4,600	7
2,400	6	4,800	6
2,500	5	5,000	6
2,600	5	5,200	6
2,700	5	5,400	6
2,800	5	5,600	5
2,900	5	5,800	5
3,000	5	6,000	5
3,100	4	6,200	5
3,200	4	6,400	5
3,300	4	6,600	5
3,400	4	6,800	4
3,500	4	7,000	4
3,600	4	7,200	4
3,700	4	7,400	4
3,800	4	7,600	4

Note minimum volumes required:  
25 mm : 560 liters  
37 mm : 1250 liters

Filter diameter of 25 mm = effective area of 385 sq mm  
Filter diameter of 37 mm = effective area of 855 sq mm

20. Ensure that the sampler is turned upright before interrupting the pump flow.

21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

22. Ensure that the samples are stored in a secure and representative location.

23. Do not change containers if portions of these filters are taken for other purposes.

24. A summary of Sample Data Quality Objectives is shown in the following Table II:

TABLE II--SUMMARY OF SAMPLING AGENCY DATA QUALITY OBJECTIVES

This table summarizes the data quality objectives from the performance of this method in terms of precision, accuracy, completeness, representativeness, and comparability. These objectives are assured by the periodic control checks and reference checks listed here and described in the text of the method.

Unit Operation	QC Check	Frequency	Conformance Expectation
Sampling materials	Sealed blank	1 per I/O site	95%
Sample procedures	Field blanks	2 per I/O site	95%
	Pump calibration	Before and after each field series	90%
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample shipment	Review of sending report	Each sample	95% complete

### C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples. D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected. E. Sample Preparation

1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.

2. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them into the clean room facility.

3. Perform sample preparation in a well-equipped clean facility.

**>Note:** The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18 s/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm<sup>2</sup> for that same area.

4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.

5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.

a. TEM Grid Opening Area measurement must be done as follows:

- i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.
  - ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field diameters. From the data, calculate the field area for an average grid opening.
  - iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.
- b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G. or other equivalent methods may be used.
- c. TEM specimen preparation from mixed cellulose ester (MCE) filters.
- i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)
  - ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. 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 conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate asher and operating conditions will then be set such that a 1-2  $\mu\text{m}$  (10 percent) layer of collapsed surface will be removed.
  - iii. Procedures as described in Unit III. or other equivalent methods may be used to prepare samples. F.

#### TEM Method

1. An 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.

2. *Determination of Camera Constant and ED Pattern Analysis.* The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved 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. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.

3. *Magnification Calibration.* The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate a eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.

5. Microscope settings: 80-120 kV, grid assessment 250-1,000X, then 15,000-20,000X screen magnification for analysis.

6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

7. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.

8. Reject the grid if:

a. Less than 50 percent of the grid openings covered by the replica are intact.

b. The replica is doubled or folded.

c. The replica is too dark because of incomplete dissolution of the filter.

## 9. Recording Rules.

a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu\text{m}$  is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. See the following Figure 2:

FIGURE 2--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES

Count as 1 fiber; 1 Structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.



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Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTURES:



Fiber protrusion  
<5:1 Aspect Ratio



No fiber protrusion



Fiber protrusion  
<0.5 micrometer

— <0.5 micrometer in length  
— <5:1 Aspect Ratio

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i. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *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 must have more than two intersections.

- iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- b. Separate categories will be maintained for fibers less than 5  $\mu\text{m}$  and for fibers equal to or greater than 5  $\mu\text{m}$  in length.
- c. Record NSD when no structures are detected in the field.
- d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)
- e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.
- f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm<sup>2</sup> concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)
- g. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration, the fact that they are not asbestos must be confirmed by EDXA or measurement of a zone axis diffraction pattern.
- h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70 s/mm<sup>2</sup> have been exceeded for a particular sample.
- i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70 s/mm<sup>2</sup> have been exceeded for a particular sample.)
- j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.
- k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.
- l. If an EDXA spectrum was attempted but not observed, record N on the count sheet.
- m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.

## 10. Classification Rules.

- a. *Fiber*. A structure having a minimum length greater than or equal to 0.5 •m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- b. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- c. *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 must have more than two intersections.
- d. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

### G. Sample Analytical Sequence

1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.
  2. Carry out visual inspection of work site prior to air monitoring.
  3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.
  4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix.
- ### H. Reporting
1. The following information must be reported to the client for each sample analyzed:
    - a. Concentration in structures per square millimeter and structures per cubic centimeter.
    - b. Analytical sensitivity used for the analysis.
    - c. Number of asbestos structures.
    - d. Area analyzed.

- e. Volume of air sampled (which must be initially supplied to lab by client).
- f. Copy of the count sheet must be included with the report.
- g. Signature of laboratory official to indicate that the laboratory met specifications of the method.
- h. Report form must contain official laboratory identification (e.g., letterhead).
- i. Type of asbestos. I. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE III--SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or reclean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma etch blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
2. Check all laboratory reagents and supplies for acceptable asbestos background levels.
3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.
4. Prepare multiple grids of each sample.
5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm<sup>2</sup> per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.
6. Perform a system check on the transmission electron microscope daily.
7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.I.

8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.I.
  9. Validate all data entries.
  10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.I.
  11. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns.
  12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures.
- J. References

For additional background information on this method, the following references should be consulted.

1. "Guidance for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.
2. "Measuring Airborne Asbestos Following an Abatement Action," USEPA, Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.
7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters," WERL SOP 87-1, March 5, 1987.

9. NIOSH Method 7402 for Asbestos Fibers, 12-11-86 Draft.

10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," Draft report, USEPA Contract 68-02-3266, July 1984.

11. "Guidance to the Preparation of Quality Assurance Project Plans," USEPA, Office of Pollution Prevention and Toxics, 1984.

### **III. Nonmandatory Transmission Electron Microscopy Method**

#### **A. Definitions of Terms**

1. *Analytical sensitivity* -- Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 s/cm<sup>3</sup>.

2. *Asbestiform* -- A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio* -- A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle* -- A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area* -- A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm<sup>2</sup> for no more than one single preparation for that same area.

6. *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 must have more than two intersections.

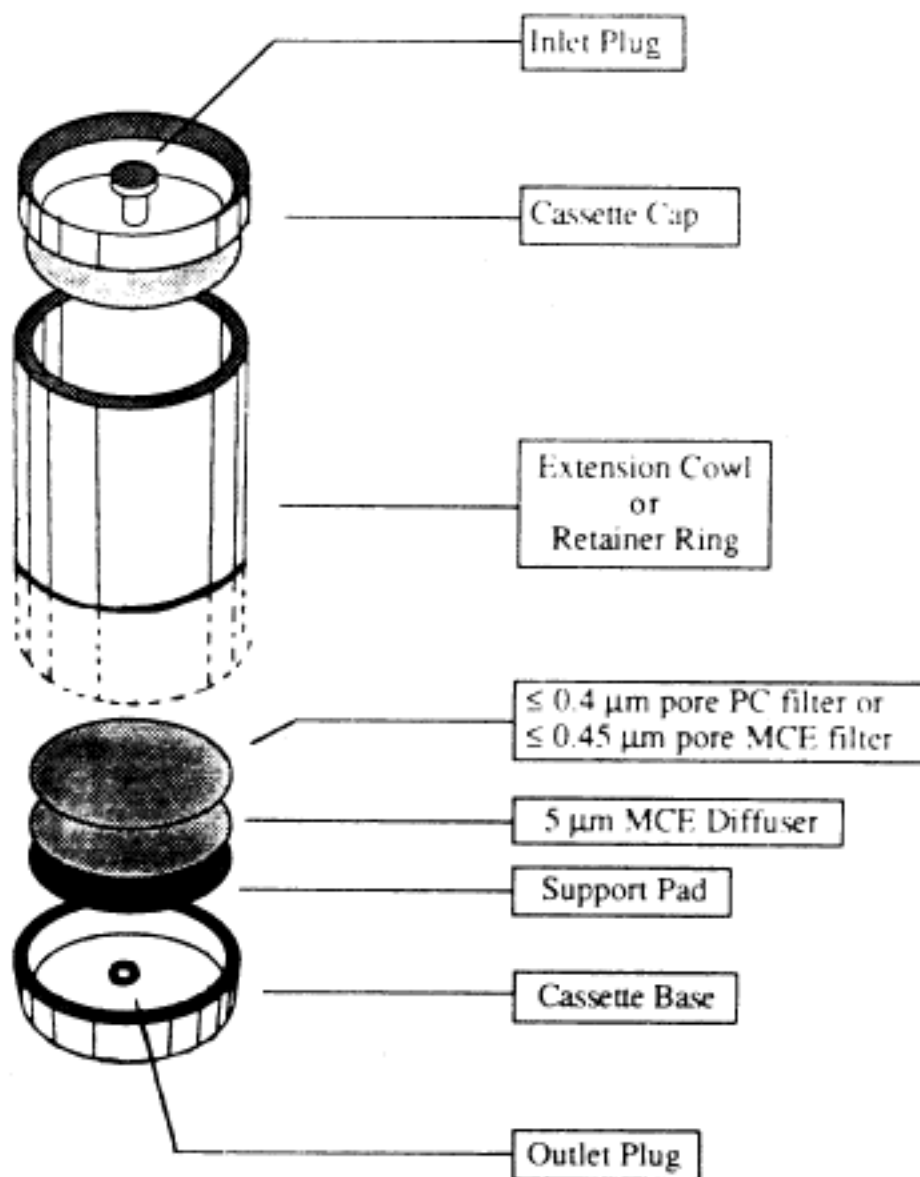
7. *ED* -- Electron diffraction.

8. *EDXA* -- Energy dispersive X-ray analysis.
9. *Fiber* -- A structure greater than or equal to 0.5  $\mu$ m in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.
10. *Grid* -- An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.
11. *Intersection* -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.
12. *Laboratory sample coordinator* -- That person responsible for the conduct of sample handling and the certification of the testing procedures.
13. *Filter background level* -- The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on blanks (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm<sup>2</sup>.
14. *Matrix* -- Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
15. *NSD* -- No structure detected.
16. *Operator* -- A person responsible for the TEM instrumental analysis of the sample.
17. *PCM* -- Phase contrast microscopy.
18. *SAED* -- Selected area electron diffraction.
19. *SEM* -- Scanning electron microscope.
20. *STEM* -- Scanning transmission electron microscope.
21. *Structure* -- a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.
22. *S/cm<sup>3</sup>* -- Structures per cubic centimeter.
23. *S/mm<sup>2</sup>* -- Structures per square millimeter.
24. *TEM* -- Transmission electron microscope. B. Sampling



1. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (See References 1, 2, and 5 of Unit III.L.) Special precautions should be taken to avoid contamination of the sample. For example, materials that have not been prescreened for their asbestos background content should not be used; also, sample handling procedures which do not take cross contamination possibilities into account should not be used.
2. Material and supply checks for asbestos contamination should be made on all critical supplies, reagents, and procedures before their use in a monitoring study.
3. Quality control and quality assurance steps are needed to identify problem areas and isolate the cause of the contamination (see Reference 5 of Unit III.L.). Control checks shall be permanently recorded to document the quality of the information produced. The sampling firm must have written quality control procedures and documents which verify compliance. Independent audits by a qualified consultant or firm should be performed once a year. All documentation of compliance should be retained indefinitely to provide a guarantee of quality. A summary of Sample Data Quality Objectives is shown in Table II of Unit II.B.
4. Sampling materials.
  - a. Sample for airborne asbestos following an abatement action using commercially available cassettes.
  - b. Use either a cowl or a filter-retaining middle piece. Conductive material may reduce the potential for particulates to adhere to the walls of the cowl.
  - c. Cassettes must be verified as "clean" prior to use in the field. If packaged filters are used for loading or preloaded cassettes are purchased from the manufacturer or a distributor, the manufacturer's name and lot number should be entered on all field data sheets provided to the laboratory, and are required to be listed on all reports from the laboratory.
  - d. Assemble the cassettes in a clean facility (See definition of clean area under Unit III.A.).
  - e. Reloading of used cassettes is not permitted.
  - f. Use sample collection filters which are either polycarbonate having a pore size of less than or equal to 0.4  $\mu$ m or mixed cellulose ester having a pore size of less than or equal to 0.45  $\mu$ m.
  - g. Place these filters in series with a backup filter with a pore size of 5.0  $\mu$ m (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE I--SAMPLING CASSETTE CONFIGURATION



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- h. When polycarbonate filters are used, position the highly reflective face such that the incoming particulate is received on this surface.
- i. Seal the cassettes to prevent leakage around the filter edges or between cassette part joints. A mechanical press may be useful to achieve a reproducible leak-free seal. Shrink fit gel-bands may be used for this purpose and are available from filter manufacturers and their authorized distributors.

j. Use wrinkle-free loaded cassettes in the sampling operation.

## 5. Pump setup.

a. Calibrate the sampling pump over the range of flow rates and loads anticipated for the monitoring period with this flow measuring device in series. Perform this calibration using guidance from EPA Method 2A each time the unit is sent to the field (See Reference 6 of Unit III.L.).

b. Configure the sampling system to preclude pump vibrations from being transmitted to the cassette by using a sampling stand separate from the pump station and making connections with flexible tubing.

c. Maintain continuous smooth flow conditions by damping out any pump action fluctuations if necessary.

d. Check the sampling system for leaks with the end cap still in place and the pump operating before initiating sample collection. Trace and stop the source of any flow indicated by the flowmeter under these conditions.

e. Select an appropriate flow rate equal to or greater than 1 L/min or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

f. Orient the cassette downward at approximately 45 degrees from the horizontal.

g. Maintain a log of all pertinent sampling information, such as pump identification number, calibration data, sample location, date, sample identification number, flow rates at the beginning, middle, and end, start and stop times, and other useful information or comments. Use of a sampling log form is recommended. See the following Figure 2:

FIGURE 2--SAMPLING LOG FORM

Sample Number	Location of Sample	Pump I.D.	Start Time	Middle Time	End Time	Flow Rate

Inspector: \_\_\_\_\_ Date: \_\_\_\_\_

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- h. Initiate a chain of custody procedure at the start of each sampling, if this is requested by the client.
- i. Maintain a close check of all aspects of the sampling operation on a regular basis.
- j. Continue sampling until at least the minimum volume is collected, as specified in the following Table I:

TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS  
(0.0057 MM<sup>2</sup>) THAT NEED TO BE ANALYZED TO  
MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC  
BASED ON VOLUME AND EFFECTIVE FILTER AREA

Effective Filter Area 365 sq mm		Effective Filter Area 855 sq mm	
Volume (liters)	# of grid openings	Volume (liters)	# of grid openings
560	24	1,250	24
600	23	1,300	23
700	19	1,400	21
800	17	1,600	19
900	15	1,800	17
1,000	14	2,000	15
1,100	12	2,200	14
1,200	11	2,400	13
1,300	10	2,600	12
1,400	10	2,800	11
1,500	9	3,000	10
1,600	8	3,200	9
1,700	8	3,400	9
1,800	8	3,600	8
1,900	7	3,800	8
2,000	7	4,000	8
2,100	6	4,200	7
2,200	6	4,400	7
2,300	6	4,600	7
2,400	6	4,800	6
2,500	5	5,000	6
2,600	5	5,200	6
2,700	5	5,400	6
2,800	5	5,600	5
2,900	5	5,800	5
3,000	5	6,000	5
3,100	4	6,200	5
3,200	4	6,400	5
3,300	4	6,600	5
3,400	4	6,800	4
3,500	4	7,000	4
3,600	4	7,200	4
3,700	4	7,400	4
3,800	4	7,600	4

Note minimum volumes required:  
25 mm : 560 liters  
37 mm : 1250 liters

Filter diameter of 25 mm = effective area of 365 sq mm  
Filter diameter of 37 mm = effective area of 855 sq mm

k. At the conclusion of sampling, turn the cassette upward before stopping the flow to minimize possible particle loss. If the sampling is resumed, restart the flow before reorienting the cassette downward. Note the condition of the filter at the conclusion of sampling.

l. Double check to see that all information has been recorded on the data collection forms and that the cassette is securely closed and appropriately identified using a waterproof label. Protect cassettes in individual clean resealed polyethylene bags. Bags are to be used for storing cassette caps when they are removed for sampling purposes. Caps and plugs should only be removed or replaced using clean hands or clean disposable plastic gloves.

m. Do not change containers if portions of these filters are taken for other purposes.

6. Minimum sample number per site. A minimum of 13 samples are to be collected for each testing consisting of the following:

a. A minimum of five samples per abatement area.

- b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.
- c. Two field blanks are to be taken by removing the cap for not more than 30 sec and replacing it at the time of sampling before sampling is initiated at the following places:
  - i. Near the entrance to each ambient area.
  - ii. At one of the ambient sites.

**(Note:** Do not leave the blank open during the sampling period.)

- d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

## 7. Abatement area sampling.

- a. Conduct final clearance sampling only after the primary containment barriers have been removed; the abatement area has been thoroughly dried; and, it has passed visual inspection tests by qualified personnel. (See Reference 1 of Unit III.L.)
- b. Containment barriers over windows, doors, and air passageways must remain in place until the TEM clearance sampling and analysis is completed and results meet clearance test criteria. The final plastic barrier remains in place for the sampling period.
- c. Select sampling sites in the abatement area on a random basis to provide unbiased and representative samples.
- d. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust.
  - i. Equipment used in aggressive sampling such as a leaf blower and/or fan should be properly cleaned and decontaminated before use.
  - ii. Air filtration units shall remain on during the air monitoring period.
  - iii. Prior to air monitoring, floors, ceiling and walls shall be swept with the exhaust of a minimum one (1) horsepower leaf blower.
  - iv. Stationary fans are placed in locations which will not interfere with air monitoring equipment. Fan air

is directed toward the ceiling. One fan shall be used for each 10,000 ft<sup>3</sup> of worksite.

v. Monitoring of an abatement work area with high-volume pumps and the use of circulating fans will require electrical power. Electrical outlets in the abatement area may be used if available. If no such outlets are available, the equipment must be supplied with electricity by the use of extension cords and strip plug units. All electrical power supply equipment of this type must be approved Underwriter Laboratory equipment that has not been modified. All wiring must be grounded. Ground fault interrupters should be used. Extreme care must be taken to clean up any residual water and ensure that electrical equipment does not become wet while operational.

vi. Low volume pumps may be carefully wrapped in 6-mil polyethylene to insulate the pump from the air. High volume pumps cannot be sealed in this manner since the heat of the motor may melt the plastic. The pump exhausts should be kept free.

vii. If recleaning is necessary, removal of this equipment from the work area must be handled with care. It is not possible to completely decontaminate the pump motor and parts since these areas cannot be wetted. To minimize any problems in this area, all equipment such as fans and pumps should be carefully wet wiped prior to removal from the abatement area. Wrapping and sealing low volume pumps in 6-mil polyethylene will provide easier decontamination of this equipment. Use of clean water and disposable wipes should be available for this purpose.

e. Pump flow rate equal to or greater than 1 L/min or less than 10 L/min may be used for 25 mm cassettes. The larger cassette diameters may have comparably increased flow.

f. Sample a volume of air sufficient to ensure the minimum quantitation limits. (See Table I of Unit III.B.5.j.)

## 8. Ambient sampling.

a. Position ambient samplers at locations representative of the air entering the abatement site. If makeup air entering the abatement site is drawn from another area of the building which is outside of the abatement area, place the pumps in the building, pumps should be placed out of doors located near the building and away from any obstructions that may influence wind patterns. If construction is in progress immediately outside the enclosure, it may be necessary to select another ambient site. Samples should be representative of any air entering the work site.

b. Locate the ambient samplers at least 3 ft apart and protect them from adverse weather conditions.

c. Sample same volume of air as samples taken inside the abatement site. C. Sample Shipment

1. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

2. Select a rigid shipping container and pack the cassettes upright in a noncontaminating nonfibrous medium such as a bubble pack. The use of resealable polyethylene bags may help to prevent jostling of individual cassettes.
  3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.
  4. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.
  5. Hand-carry samples to the laboratory in an upright position if possible; otherwise choose that mode of transportation least likely to jar the samples in transit.
  6. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain of custody and sample tracking procedures. This will also help the laboratory schedule timely analysis for the samples when they are received.
- D. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined, and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the text below.

1. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a maximum of 53 s/mm<sup>2</sup> for that same area for any single preparation is acceptable for this method.
2. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter -- not the filter which will be used in sampling -- before and after the sampling operation.



3. Record all calibration information with the data to be used on a standard sampling form.
  4. Ensure that the samples are stored in a secure and representative location.
  5. Ensure that mechanical calibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
  6. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by installing a damping chamber if necessary.
  7. Open a loaded cassette momentarily at one of the indoor sampling sites when sampling is initiated. This sample will serve as an indoor field blank.
  8. Open a loaded cassette momentarily at one of the outdoor sampling sites when sampling is initiated. This sample will serve as an outdoor field blank.
  9. Carry a sealed blank into the field with each sample series. Do not open this cassette in the field.
  10. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.
  11. Ensure that the sampler is turned upright before interrupting the pump flow.
  12. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.
- E. Sample Receiving
1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.
  2. Adhere to the following procedures to ensure both the continued chain-of-custody and the accountability of all samples passing through the laboratory:
    - a. Note the condition of the shipping package and data written on it upon receipt.
    - b. Retain all bills of lading or shipping slips to document the shipper and delivery time.
    - c. Examine the chain-of-custody seal, if any, and the package for its integrity.
    - d. If there has been a break in the seal or substantive damage to the package, the sample coordinator shall

immediately notify the shipper and a responsible laboratory manager before any action is taken to unpack the shipment.

e. Packages with significant damage shall be accepted only by the responsible laboratory manager after discussions with the client.

3. Unwrap the shipment in a clean, uncluttered facility. The sample coordinator or his or her designee will record the contents, including a description of each item and all identifying numbers or marks. A Sample Receiving Form to document this information is attached for use when necessary. (See the following Figure 3.)

FIGURE 3--SAMPLE RECEIVING FORM

Date of package delivery \_\_\_\_\_ Package shipped from \_\_\_\_\_  
 Carrier \_\_\_\_\_ Shipping bill retained \_\_\_\_\_  
 \*Condition of package on receipt \_\_\_\_\_  
 \*Condition of custody seal \_\_\_\_\_  
 Number of samples received \_\_\_\_\_ Shipping manifest attached \_\_\_\_\_  
 Purchase Order No. \_\_\_\_\_ Project ID: \_\_\_\_\_  
 Comments \_\_\_\_\_

No.	Description	Sampling Medium		Sampled Volume Liters	Receiving ID #	Assigned #
		PC	MCE			
1	_____	_____	_____	_____	_____	_____
2	_____	_____	_____	_____	_____	_____
3	_____	_____	_____	_____	_____	_____
4	_____	_____	_____	_____	_____	_____
5	_____	_____	_____	_____	_____	_____
6	_____	_____	_____	_____	_____	_____
7	_____	_____	_____	_____	_____	_____
8	_____	_____	_____	_____	_____	_____
9	_____	_____	_____	_____	_____	_____
10	_____	_____	_____	_____	_____	_____
11	_____	_____	_____	_____	_____	_____
12	_____	_____	_____	_____	_____	_____
13	_____	_____	_____	_____	_____	_____

(Use as many additional sheets as needed.)

Comments \_\_\_\_\_

Date of acceptance into sample bank \_\_\_\_\_

Signature of chain-of-custody recipient \_\_\_\_\_

Disposition of samples \_\_\_\_\_

**\*Note:** If the package has sustained substantial damage or the custody seal is broken, stop and contact the project manager and the shipper.

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**Note:** The person breaking the chain-of-custody seal and itemizing the contents assumes responsibility for the shipment and signs documents accordingly.

4. Assign a laboratory number and schedule an analysis sequence.

5. Manage all chain-of-custody samples within the laboratory such that their integrity can be ensured and documented.

#### F. Sample Preparation

1. Personnel not affiliated with the Abatement Contractor shall be used to prepare samples and conduct TEM analysis. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them to the clean sample preparation facility.

2. Perform sample preparation in a well-equipped clean facility.

**Note:** The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA filtered. The cumulative analytical blank concentration must average less than 18 s/mm<sup>2</sup> in an area of 0.057 s/mm<sup>2</sup> (nominally 10 200-mesh grid openings) with no more than one single preparation to exceed 53 s/mm<sup>2</sup> for that same area.

3. Preparation areas for air samples must be separated from preparation areas for bulk samples. Personnel must not prepare air samples if they have previously been preparing bulk samples without performing appropriate personal hygiene procedures, i.e., clothing change, showering, etc.

4. *Preparation.* Direct preparation techniques are required. The objective is to produce an intact carbon film containing the particulates from the filter surface which is sufficiently clear for TEM analysis. Currently recommended direct preparation procedures for polycarbonate (PC) and mixed cellulose ester (MCE) filters are described in Unit III.F.7. and 8. Sample preparation is a subject requiring additional research. Variation on those steps which do not substantively change the procedure, which improve filter clearing or which reduce contamination problems in a laboratory are permitted.

a. Use only TEM grids that have had grid opening areas measured according to directions in Unit III.J.

b. Remove the inlet and outlet plugs prior to opening the cassette to minimize any pressure differential that may be present.

c. Examples of techniques used to prepare polycarbonate filters are described in Unit III.F.7.

d. Examples of techniques used to prepare mixed cellulose ester filters are described in Unit III.F.8.

- e. Prepare multiple grids for each sample.
- f. Store the three grids to be measured in appropriately labeled grid holders or polyethylene capsules.

## 5. Equipment.

- a. Clean area.
- b. Tweezers. Fine-point tweezers for handling of filters and TEM grids.
- c. Scalpel Holder and Curved No. 10 Surgical Blades.
- d. Microscope slides.
- e. Double-coated adhesive tape.
- f. Gummed page reinforcements.
- g. Micro-pipet with disposal tips 10 to 100  $\mu$ L variable volume.
- h. Vacuum coating unit with facilities for evaporation of carbon. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surface by oil from the pumping system. The vacuum-coating unit can also be used for deposition of a thin film of gold.
- i. *Carbon rod electrodes*. Spectrochemically pure carbon rods are required for use in the vacuum evaporator for carbon coating of filters.
- j. *Carbon rod sharpener*. This is used to sharpen carbon rods to a neck. The use of necked carbon rods (or equivalent) allows the carbon to be applied to the filters with a minimum of heating.
- k. *Low-temperature plasma asher*. This is used to etch the surface of collapsed mixed cellulose ester (MCE) filters. The asher should be supplied with oxygen, and should be modified as necessary to provide a throttle or bleed valve to control the speed of the vacuum to minimize disturbance of the filter. Some early models of ashers admit air too rapidly, which may disturb particulates on the surface of the filter during the etching step.
- l. *Glass petri dishes, 10 cm in diameter, 1 cm high*. For prevention of excessive evaporation of solvent when these are in use, a good seal must be provided between the base and the lid. The seal can be improved by grinding the base and lid together with an abrasive grinding material.
- m. Stainless steel mesh.

- n. Lens tissue.
- o. Copper 200-mesh TEM grids, 3 mm in diameter, or equivalent.
- p. Gold 200-mesh TEM grids, 3 mm in diameter, or equivalent.
- q. Condensation washer.
- r. Carbon-coated, 200-mesh TEM grids, or equivalent.
- s. Analytical balance, 0.1 mg sensitivity.
- t. Filter paper, 9 cm in diameter.
- u. Oven or slide warmer. Must be capable of maintaining a temperature of 65-70 °C.
- v. Polyurethane foam, 6 mm thickness.
- w. Gold wire for evaporation.

#### 6. Reagents.

- a. *General.* A supply of ultra-clean, fiber-free water must be available for washing of all components used in the analysis. Water that has been distilled in glass or filtered or deionized water is satisfactory for this purpose. Reagents must be fiber-free.
- b. Polycarbonate preparation method -- chloroform.
- c. Mixed Cellulose Ester (MCE) preparation method -- acetone or the Burdette procedure (Ref. 7 of Unit III.L.).

#### 7. TEM specimen preparation from polycarbonate filters.

- a. *Specimen preparation laboratory.* It is most important to ensure that contamination of TEM specimens by extraneous asbestos fibers is minimized during preparation.
- b. Cleaning of sample cassettes. Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces.

c. Preparation of the carbon evaporator. If the polycarbonate filter has already been carbon-coated prior to receipt, the carbon coating step will be omitted, unless the analyst believes the carbon film is too thin. If there is a need to apply more carbon, the filter will be treated in the same way as an uncoated filter. Carbon coating must be performed with a high-vacuum coating unit. 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 must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 to 12 cm from the surface of a microscope slide held in the rotating and tilting device.

d. Selection of filter area for carbon coating. Before preparation of the filters, a 75 mm x 50 mm microscope slide is washed and dried. This slide is used to support strips of filter during the carbon evaporation. Two parallel strips of double-sided adhesive tape are applied along the length of the slide. Polycarbonate filters are easily stretched during handling, and cutting of areas for further preparation must be performed with great care. The filter and the MCE backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. The filter can be cut with a curved scalpel blade by rocking the blade from the point placed in contact with the filter. The process can be repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of polycarbonate filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide. The filter strip can be held with fine-point tweezers and supported underneath by the scalpel blade during placement on the microscope slide. The analyst can place several such strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and tweezers before handling a new sample. The filter strips should be identified by etching the glass slide or marking the slide using a marker insoluble in water and solvents. After the filter strip has been cut from each filter, the residual parts of the filter must be returned to the cassette and held in position by reassembly of the cassette. The cassette will then be archived for a period of 30 days or returned to the client upon request.

e. Carbon coating of filter strips. The glass slide holding the filter strips is placed on the rotation-tilting device, and the evaporator chamber is evacuated. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation is too rapid, the strips of polycarbonate filter will begin to curl, which will lead to cross-linking of the surface material and make it relatively insoluble in chloroform. An experienced analyst can judge the thickness of carbon film to be applied, and some test should be made first on unused filters. If the 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. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film will also lead to a TEM image that is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film should be as thin as possible and remain intact on most of the grid openings of the TEM specimen intact.

f. 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. A washer consisting of a simple stainless steel bridge is recommended. Several pieces of lens tissue approximately 1.0 cm x 0.5 cm are placed on the stainless

steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. See References 8 and 10 of Unit III.L.

g. Placing of specimens into the Jaffe washer. The TEM grids are first placed on a piece of lens tissue so that individual grids can be picked up with tweezers. Using a curved scalpel blade, the analyst excises three 3 mm square pieces of the carbon-coated polycarbonate filter from the filter strip. The three squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. The piece of filter is placed on a TEM specimen grid with the shiny side of the TEM grid facing upwards, and the whole assembly is placed boldly onto the saturated lens tissue in the Jaffe washer. If carbon-coated grids are used, the filter should be placed carbon-coated side down. The three excised squares of filters are placed on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight.

h. *Condensation washing.* It has been found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if the surface of the filter was overheated during the carbon evaporation. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuration of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a low result. Undissolved filter medium also compromises the ability to obtain ED patterns. Before they are counted, TEM grids must be examined critically to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids after the initial Jaffe washer treatment, with chloroform as the solvent, clears all residual filter medium in a period of approximately 1 hour. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of the condensation washer, and the washer is operated for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washer step may be unnecessary.

## 8. TEM specimen preparation from MCE filters.

a. This method of preparing TEM specimens from MCE filters is similar to that specified in NIOSH Method 7402. See References 7, 8, and 9 of Unit III.L.

b. Upon receipt at the analytical laboratory, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces before entering the clean sample preparation area.

c. Remove a section from any quadrant of the sample and blank filters.

d. Place the section on a clean microscope slide. Affix the filter section to the slide with a gummed paged reinforcement or other suitable means. Label the slide with a water and solvent-proof marking pen.

- e. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 minutes for the sample filter to fuse and clear.
- f. Plasma etching of the collapsed filter is required.
- i. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. 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 conditions that should be used. This is one area of the method that requires further evaluation. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for a particular asher and operating conditions will then be set such that a 1-2  $\mu$ m (10 percent) layer of collapsed surface will be removed.
- ii. Place the slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.
- g. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 mm x 5 mm section of graphite rod onto the cleared filter. Remove the slide to a clean, dry, covered petri dish.
- h. Prepare a second petri dish as a Jaffe washer with the wicking substrate prepared from filter or lens paper placed on top of a 6 mm thick disk of clean spongy polyurethane foam. Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent. The wicking substrate should be thin enough to fit into the petri dish without touching the lid.
- i. Place carbon-coated TEM grids face up on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish lid and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated. The level of acetone should be just high enough to saturate the filter paper without creating puddles.
- j. Remove about a quarter section of the carbon-coated filter samples from the glass slides using a surgical knife and tweezers. Carefully place the section of the filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation.

G. TEM Method

### 1. *Instrumentation.*

- a. Use an 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed



with calibrated gradations. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely (see Unit III.J.) for magnification and camera constant.

b. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory. This must be an Energy Dispersive X-ray Detector mounted on TEM column and associated hardware/software to collect, save, and read out spectral information. Calibration of Multi-Channel Analyzer shall be checked regularly for Al at 1.48 KeV and Cu at 8.04 KeV, as well as the manufacturer's procedures.

i. Standard replica grating may be used to determine magnification (e.g., 2160 lines/mm).

ii. Gold standard may be used to determine camera constant.

c. Use a specimen holder with single tilt and/or double tilt capabilities.

## 2. Procedure.

a. Start a new Count Sheet for each sample to be analyzed. Record on count sheet: analyst's initials and date; lab sample number; client sample number microscope identification; magnification for analysis; number of predetermined grid openings to be analyzed; and grid identification. See the following Figure 4:

FIGURE 4--COUNT SHEET

Lab Sample No. \_\_\_\_\_ Filter Type \_\_\_\_\_ Operator \_\_\_\_\_  
 Client Sample No. \_\_\_\_\_ Filter Area \_\_\_\_\_ Date \_\_\_\_\_  
 Instrument I.D. \_\_\_\_\_ Grid I.D. \_\_\_\_\_ Comments \_\_\_\_\_  
 Magnification \_\_\_\_\_ Grid Opening (GO) Area \_\_\_\_\_  
 Acc. Voltage \_\_\_\_\_ No. GO to be Analyzed \_\_\_\_\_

[illegible][illegible]

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

NFD = No fibers detected  
N = No diffraction obtained

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- b. Check that the microscope is properly aligned and calibrated according to the manufacturer's specifications and instructions.
- c. Microscope settings: 80-120 kV, grid assessment 250-1000X, then 15,000-20,000X screen magnification for analysis.
- d. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.
- e. Determine the suitability of the grid.

i. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading shall not be analyzed.

ii. Examine the grid at low magnification (<1000X) to determine its suitability for detailed study at higher magnifications.

iii. Reject the grid if:

(1) Less than 50 percent of the grid openings covered by the replica are intact.

(2) It is doubled or folded.

(3) It is too dark because of incomplete dissolution of the filter.

iv. If the grid is rejected, load the next sample grid.

v. If the grid is acceptable, continue on to Step 6 if mapping is to be used; otherwise proceed to Step 7.

f. Grid Map (Optional).

i. Set the TEM to the low magnification mode.

ii. Use flat edge or finder grids for mapping.

iii. Index the grid openings (fields) to be counted by marking the acceptable fields for one-half (0.5) of the area needed for analysis on each of the two grids to be analyzed. These may be marked just before examining each grid opening (field), if desired.

iv. Draw in any details which will allow the grid to be properly oriented if it is reloaded into the microscope and a particular field is to be reliably identified.

g. Scan the grid.

i. Select a field to start the examination.

ii. Choose the appropriate magnification (15,000 to 20,000X screen magnification).

iii. Scan the grid as follows.

(1) At the selected magnification, make a series of parallel traverses across the field. On reaching the end

of one traverse, move the image one window and reverse the traverse.

**Note:** A slight overlap should be used so as not to miss any part of the grid opening (field).

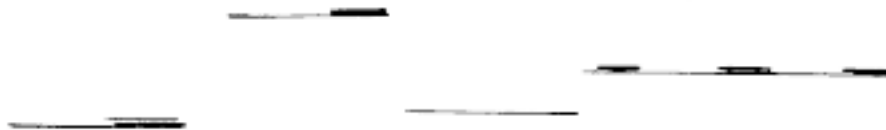
(2) Make parallel traverses until the entire grid opening (field) has been scanned.

h. Identify each structure for appearance and size.

i. Appearance and size: Any continuous grouping of particles in which an asbestos fiber within aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu$ m is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. See the following Figure 5:

FIGURE 5--COUNTING GUIDELINES USED IN  
DETERMINING ASBESTOS STRUCTURES

Count as 1 fiber; 1 Structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.





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Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTURES:



Fiber protrusion  
<5:1 Aspect Ratio



No fiber protrusion



Fiber protrusion  
<0.5 micrometer

— <0.5 micrometer in length  
— <5:1 Aspect Ratio

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An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. Combinations such as a matrix and cluster, matrix and bundle, or bundle and cluster are categorized by the dominant fiber quality -- cluster, bundle, and matrix, respectively. Separate categories will be maintained for fibers less than 5  $\mu\text{m}$  and for fibers greater than or equal to 5  $\mu\text{m}$  in length. Not required, but useful, may be to record the fiber length in 1  $\mu\text{m}$  intervals. (Identify each structure morphologically and analyze it as it enters the "window".)

- (1) *Fiber*. A structure having a minimum length greater than 0.5  $\mu$ m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed, no intersections.
- (2) *Bundle*. A structure composed of 3 or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- (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 must have more than 2 intersections.
- (4) *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- (5) *NSD*. Record NSD when no structures are detected in the field.
- (6) *Intersection*. Non-parallel touching or crossing of fibers, with the projection having an aspect ratio 5:1 or greater.

ii. Structure Measurement.

- (1) Recognize the structure that is to be sized.
  - (2) Memorize its location in the "window" relative to the sides, inscribed square and to other particulates in the field so this exact location can be found again when scanning is resumed.
  - (3) Measure the structure using the scale on the screen.
  - (4) Record the length category and structure type classification on the count sheet after the field number and fiber number.
  - (5) Return the fiber to its original location in the window and scan the rest of the field for other fibers; if the direction of travel is not remembered, return to the right side of the field and begin the traverse again.
- i. Visual identification of Electron Diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)
- i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)

ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.

(1) Chrysotile: The chrysotile asbestos pattern has characteristic streaks on the layer lines other than the central line and some streaking also on the central line. There will be spots of normal sharpness on the central layer line and on alternate lines (2nd, 4th, etc.). The repeat distance between layer lines is 0.53 nm and the center doublet is at 0.73 nm. The pattern should display (002), (110), (130) diffraction maxima; distances and geometry should match a chrysotile pattern and be measured semiquantitatively.

(2) Amphibole Group [includes grunerite (amosite), crocidolite, anthophyllite, tremolite, and actinolite]: Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and the repeat distance between layer lines is also about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology.

iii. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns. In the event that examination of the pattern by the qualified individual indicates that the pattern had been misidentified visually, the client shall be contacted. If the pattern is a suspected chrysotile, take a photograph of the diffraction pattern at 0 degrees tilt. If the structure is suspected to be amphibole, the sample may have to be tilted to obtain a simple geometric array of spots.

#### j. Energy Dispersive X-Ray Analysis (EDXA).

i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm<sup>2</sup> concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

ii. Can be used alone to confirm chrysotile after the 70 s/mm<sup>2</sup> concentration has been exceeded.

iii. Can be used alone to confirm all nonasbestos.

iv. Compare spectrum profiles with profiles obtained from asbestos standards. The closest match identifies and categorizes the structure.

v. If the EDXA is used for confirmation, record the properly labeled spectrum on a computer disk, or if a hard copy, file with analysis data.

vi. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration, their identities must be confirmed by EDXA or measurement of a zone axis diffraction pattern to establish that the particles are nonasbestos.

k. Stopping Rules.

- i. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.
- ii. After having counted 50 asbestiform structures in a minimum of 4 grid openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.
- iii. For blank samples, the analysis is always continued until 10 grid openings have been analyzed.
- iv. In all other samples the analysis shall be continued until an analytical sensitivity of 0.005 s/cm<sup>3</sup> is reached.

l. Recording Rules. The count sheet should contain the following information:

- i. Field (grid opening): List field number.
- ii. Record "NSD" if no structures are detected.
- iii. Structure information.

(1) If fibers, bundles, clusters, and/or matrices are found, list them in consecutive numerical order, starting over with each field.

(2) Length. Record length category of asbestos fibers examined. Indicate if less than 5 •m or greater than or equal to 5 •m.

(3) Structure Type. Positive identification of asbestos fibers is required by the method. At least one diffraction pattern of each fiber type from every five samples must be recorded and compared with a standard diffraction pattern. For each asbestos fiber reported, both a morphological descriptor and an identification descriptor shall be specified on the count sheet.

(4) Fibers classified as chrysotile must be identified by diffraction and/or X-ray analysis and recorded on the count sheet. X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.

(5) Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and



recorded on the count sheet. (X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.)

(6) If a diffraction pattern was recorded on film, the micrograph number must be indicated on the count sheet.

(7) If an electron diffraction was attempted and an appropriate spectra is not observed, N should be recorded on the count sheet.

(8) If an X-ray analysis is attempted but not observed, N should be recorded on the count sheet.

(9) If an X-ray analysis spectrum is stored, the file and disk number must be recorded on the count sheet.

m. Classification Rules.

i. *Fiber*. A structure having a minimum length greater than or equal to 0.5 •m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *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 must have more than two intersections.

iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

v. *NSD*. Record NSD when no structures are detected in the field.

n. After all necessary analyses of a particle structure have been completed, return the goniometer stage to 0 degrees, and return the structure to its original location by recall of the original location.

o. Continue scanning until all the structures are identified, classified and sized in the field.

p. Select additional fields (grid openings) at low magnification; scan at a chosen magnification (15,000 to 20,000X screen magnification); and analyze until the stopping rule becomes applicable.

q. Carefully record all data as they are being collected, and check for accuracy.

r. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid hold.

Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request. H.

### Sample Analytical Sequence

1. Carry out visual inspection of work site prior to air monitoring.
2. Collect a minimum of five air samples inside the work site and five samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.
3. Analyze the abatement area samples according to this protocol. The analysis must meet the 0.005 s/cm<sup>3</sup> analytical sensitivity.
4. Remaining steps in the analytical sequence are contained in Unit IV. of this Appendix. I. Reporting

The following information must be reported to the client. See the following Table II:

TABLE II--EXAMPLE LABORATORY LETTERHEAD

Laboratory I.D.	Client I.D.	FILTER MEDIA DATA				Analyzed Area, mm <sup>2</sup>	Sample Volume, cc
		Type	Diameter, mm	Effective Area, mm <sup>2</sup>	Pore Size, $\mu$ m		

INDIVIDUAL ANALYTICAL RESULTS

Laboratory I.D.	Client I.D.	# Asbestos Structures	Analytical Sensitivity, slv	CONCENTRATION	
				Structures/mm <sup>2</sup>	Structures/cc

The analysis was carried out to the approved TEM method. This laboratory is in compliance with the quality specified by the method.

\_\_\_\_\_  
Authorized Signature

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1. Concentration in structures per square millimeter and structures per cubic centimeter.
2. Analytical sensitivity used for the analysis.
3. Number of asbestos structures.
4. Area analyzed.
5. Volume of air samples (which was initially provided by client).

6. Average grid size opening.
7. Number of grids analyzed.
8. Copy of the count sheet must be included with the report.
9. Signature of laboratory official to indicate that the laboratory met specifications of the AHERA method.
10. Report form must contain official laboratory identification (e.g., letterhead).
11. Type of asbestos. J. Calibration Methodology

**Note:** Appropriate implementation of the method requires a person knowledgeable in electron diffraction and mineral identification by ED and EDXA. Those inexperienced laboratories wishing to develop capabilities may acquire necessary knowledge through analysis of appropriate standards and by following detailed methods as described in References 8 and 10 of Unit III.L.

1. *Equipment Calibration.* In this method, calibration is required for the air-sampling equipment and the transmission electron microscope (TEM).

a. *TEM Magnification.* The magnification at the fluorescent screen of the TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica. A logbook must be maintained, and the dates of calibration depend on the past history of the particular microscope; no frequency is specified. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

b. Determination of the TEM magnification on the fluorescent screen.

i. Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

ii. Insert a diffraction grating replica (for example a grating containing 2,160 lines/mm) into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 degrees.

iii. Adjust microscope magnification to 10,000X or 20,000X. Measure the distance (mm) between two widely separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).

**Note:** The more spaces included in the measurement, the more accurate the final calculation. On most microscopes, however, the magnification is substantially constant only within the central 8-10 cm diameter region of the fluorescent screen.

iv. Calculate the true magnification (M) on the fluorescent screen:

$$M=XG/Y$$

where:

X=total distance (mm) between the designated grating lines;

G=calibration constant of the grating replica (lines/mm):

Y=number of grating replica spaces counted along X.

c. Calibration of the EDXA System. Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of asbestos can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for asbestos abatement work, and in this procedure the calibration is specified in terms of a "fingerprint" technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.

d. Calibration of Grid Openings.

i. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass slide and examining it under the PCM. Use a calibrated graticule to measure the average field diameter and use this number to calculate the field area for an average grid opening. Grids are to be randomly selected from batches up to 1,000.

**Note:** A grid opening is considered as one field.

ii. The mean grid opening area must be measured for the type of specimen grids in use. This can be accomplished on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

e. Determination of Camera Constant and ED Pattern Analysis.

i. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved 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.

ii. 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. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulates. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter,  $D$ , of the rings times the interplanar spacing,  $d$ , of the ring being measured.

K. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE III--SUMMARY OF LABORATORY  
DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/tot. of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or re-clean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma each blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
2. Check all laboratory reagents and supplies for acceptable asbestos background levels.
3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks and special testing after cleaning or servicing the room.
4. Prepare multiple grids of each sample.
5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If this average is greater than 53 f/mm 2 per 10 200-mesh grid openings, check the system for possible sources of contamination.
6. Check for recovery of asbestos from cellulose ester filters submitted to plasma asher.
7. Check for asbestos carryover in the plasma asher by including a blank alongside the positive control sample.

8. Perform a systems check on the transmission electron microscope daily.
9. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III of Unit III.K.
10. Ensure qualified operator performance by evaluation of replicate counting, duplicate analysis, and standard sample comparisons as set forth in Table III of Unit III.K.
11. Validate all data entries.
12. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III.
13. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The outline of quality control procedures presented above is viewed as the minimum required to assure that quality data is produced for clearance testing of an asbestos abated area. Additional information may be gained by other control tests. Specifics on those control procedures and options available for environmental testing can be obtained by consulting References 6, 7, and 11 of Unit III.L. L. References

For additional background information on this method the following references should be consulted.

1. "Guidelines for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.
2. "Measuring Airborne Asbestos Following an Abatement Action," USEP/Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
6. Method 2A: Direct Measurement of Gas Volume Through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.



7. Burdette, G.J. Health & Safety Exec., Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."
8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc. "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters." WERL SOP 87-1, March 5, 1987.
9. NIOSH. Method 7402 for Asbestos Fibers, December 11, 1986 Draft.
10. Yamate, G., S.C. Agarwall, R.D. Gibbons, IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." Draft report, USEPA Contract 68-02-3266, July 1984.
11. Guidance to the Preparation of Quality Assurance Project Plans. USEPA, Office of Pollution Prevention and Toxics, 1984.

#### **IV. Mandatory Interpretation of Transmission Electron Microscopy Results to Determine Completion of Response Actions**

##### **A. Introduction**

A response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne asbestos concentration inside the abatement area is no higher than concentrations at locations outside the abatement area. "Outside" means outside the abatement area, but not necessarily outside the building. EPA reasons that an asbestos removal contractor cannot be expected to clean an abatement area to an airborne asbestos concentration that is lower than the concentration of air entering the abatement area from outdoors or from other parts of the building. After the abatement area has passed a thorough visual inspection, and before the outer containment barrier is removed, a minimum of five air samples inside the abatement area and a minimum of five air samples outside the abatement area must be collected. Hence, the response action is determined to be completed when the average airborne asbestos concentration measured inside the abatement area is not statistically different from the average airborne asbestos concentration measured outside the abatement area.

The inside and outside concentrations are compared by the Z-test, a statistical test that takes into account the variability in the measurement process. A minimum of five samples inside the abatement area and five samples outside the abatement area are required to control the false negative error rate, i.e., the probability of declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

When volumes greater than or equal to 1,199 L for a 25 mm filter and 2,799 L for a 37 mm filter have

been collected and the average number of asbestos structures on samples inside the abatement area is no greater than 70 s/mm<sup>2</sup> of filter, the response action may be considered complete without comparing the inside samples to the outside samples. EPA is permitting this initial screening test to save analysis costs in situations where the airborne asbestos concentration is sufficiently low so that it cannot be distinguished from the filter contamination/background level (fibers deposited on the filter that are unrelated to the air being sampled). The screening test cannot be used when volumes of less than 1,199 L for 25 mm filter or 2,799 L for a 37 mm filter are collected because the ability to distinguish levels significantly different from filter background is reduced at low volumes.

The initial screening test is expressed in structures per square millimeter of filter because filter background levels come from sources other than the air being sampled and cannot be meaningfully expressed as a concentration per cubic centimeter of air. The value of 70 s/mm<sup>2</sup> is based on the experience of the panel of microscopists who consider one structure in 10 grid openings (each grid opening with an area of 0.0057 mm<sup>2</sup>) to be comparable with contamination/background levels of blank filters. The decision is based, in part, on Poisson statistics which indicate that four structures must be counted on a filter before the fiber count is statistically distinguishable from the count for one structure. As more information on the performance of the method is collected, this criterion may be modified. Since different combinations of the number and size of grid openings are permitted under the TEM protocol, the criterion is expressed in structures per square millimeter of filter to be consistent across all combinations. Four structures per 10 grid openings corresponds to approximately 70 s/mm<sup>2</sup>. B. Sample Collection and Analysis

1. A minimum of 13 samples is required: five samples collected inside the abatement area, five samples collected outside the abatement area, two field blanks, and one sealed blank.

2. Sampling and TEM analysis must be done according to either the mandatory or nonmandatory protocols in Appendix A. At least 0.057 mm<sup>2</sup> of filter must be examined on blank filters. C. Interpretation of Results

1. The response action shall be considered complete if either:

- a. Each sample collected inside the abatement area consists of at least 1,199 L of air for a 25 mm filter, or 2,799 L of air for a 37 mm filter, and the arithmetic mean of their asbestos structure concentrations per square millimeter of filter is less than or equal to 70 s/mm<sup>2</sup>; or

- b. The three blank samples have an arithmetic mean of the asbestos structure concentration on the blank filters that is less than or equal to 70 s/mm<sup>2</sup> and the average airborne asbestos concentration measured inside the abatement area is not statistically higher than the average airborne asbestos concentration measured outside the abatement area as determined by the Z-test. The Z-test is carried out by calculating

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

where  $\bar{Y}_I$  is the average of the natural logarithms of the inside samples and  $\bar{Y}_O$  is the average of the natural logarithms of the outside samples,  $n_I$  is the number of inside samples and  $n_O$  is the number of outside samples. The response action is considered complete if  $Z$  is less than or equal to 1.65.

**Note:** When no fibers are counted, the calculated detection limit for that analysis is inserted for the concentration.

2. If the abatement site does not satisfy either (1) or (2) of this Section C, the site must be recleaned and a new set of samples collected. D. Sequence for Analyzing Samples

It is possible to determine completion of the response action without analyzing all samples. Also, at any point in the process, a decision may be made to terminate the analysis of existing samples, reclean the abatement site, and collect a new set of samples. The following sequence is outlined to minimize the number of analyses needed to reach a decision.

1. Analyze the inside samples.
2. If at least 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for each inside sample and the arithmetic mean concentration of structures per square millimeter of filter is less than or equal to 70 s/mm<sup>2</sup>, the response action is complete and no further analysis is needed.
3. If less than 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for any of the inside samples, or the arithmetic mean concentration of structures per square millimeter of filter is greater than 70 s/mm<sup>2</sup>, analyze the three blanks.
4. If the arithmetic mean concentration of structures per square millimeter on the blank filters is greater than 70 s/mm<sup>2</sup>, terminate the analysis, identify and correct the source of blank contamination, and collect a new set of samples.
5. If the arithmetic mean concentration of structures per square millimeter on the blank filters is less than or equal to 70 s/mm<sup>2</sup>, analyze the outside samples and perform the Z-test.
6. If the Z-statistic is less than or equal to 1.65, the response action is complete. If the Z-statistic is greater than 1.65, reclean the abatement site and collect a new set of samples.

[52 FR 41857, Oct. 30, 1987]



## **Appendix K – TEM, ISO 10312**

### **(analytical method)**

INTERNATIONAL  
STANDARD

**ISO**  
**10312**

First edition  
1995-05-01

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**Ambient air — Determination of asbestos  
fibres — Direct-transfer transmission  
electron microscopy method**

*Air ambient — Détermination des fibres d'amiante — Méthode de  
microscopie électronique à transmission directe*



Reference number  
ISO 10312:1995(E)

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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<sup>2</sup> to 7 000 structures/mm<sup>2</sup> 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<sup>3</sup>, 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<sup>2</sup> 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<sup>2</sup> 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.

## 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*.

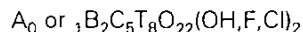
ISO Standard Handbook No. 3:1989, *Statistical Methods*.

## 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:



where

A = K, Na

B = Fe<sup>2+</sup>, Mn, Mg, Ca, Na

C = Al, Cr, Ti, Fe<sup>3+</sup>, Mg, Fe<sup>2+</sup>

T = Si, Al, Cr, Fe<sup>3+</sup>, 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), grunerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

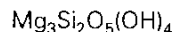
**3.7 asbestos structure:** A term applied to any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles.

**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



Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al<sup>3+</sup> may occur. Minor substitution of magnesium by Al<sup>3+</sup>, Fe<sup>2+</sup>, Fe<sup>3+</sup>, Ni<sup>2+</sup>, Mn<sup>2+</sup> and Co<sup>2+</sup> 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 li-

tre, 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.

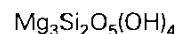
**3.30 PCM equivalent structure:** A fibrous structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

**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



**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 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<sup>-6</sup> gram)

µm = micrometre (10<sup>-6</sup> metre)

nm = nanometre (10<sup>-9</sup> 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  $\pm 10\%$  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 area 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 type 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  $\times 300$  to  $\times 100\,000$  shall be used. The ability to obtain a direct screen magnification of about  $\times 100\,000$  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.

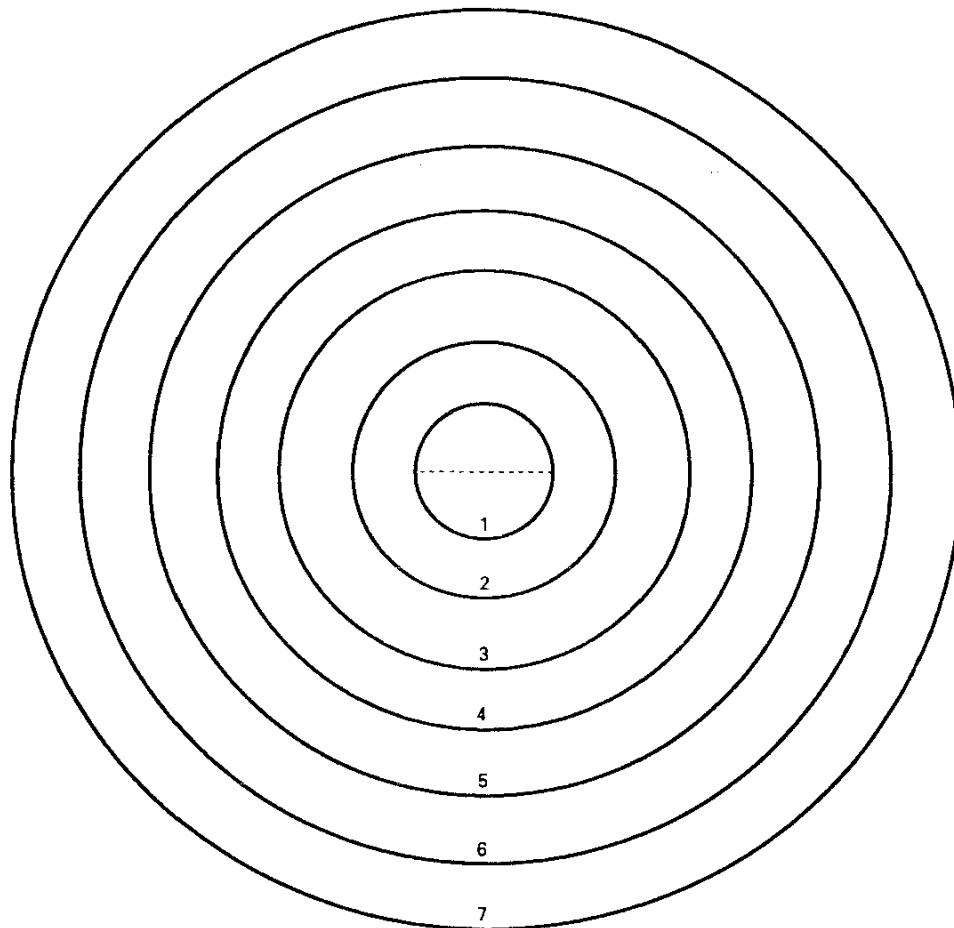


Figure 1 — Example of calibration markings on TEM viewing screen

For Bragg angles less than 0,01 rad, the TEM shall be capable of performing ED from an area of  $0,6 \mu\text{m}^2$  or less, selected from an in-focus image at a screen magnification of  $\times 20\,000$ . 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

$$A = 0,785\,4 \times \left( \frac{D}{M} + 2\,000C_s\theta^3 \right)^2$$

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<sub>s</sub>* 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  $360^\circ$ , combined with tilting through at least  $+30^\circ$  to  $-30^\circ$  about an axis in the plane of the specimen;
- b) tilted through at least  $+30^\circ$  to  $-30^\circ$  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  $\text{MnK}\alpha$ . 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  $\text{NaK}\alpha$  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.

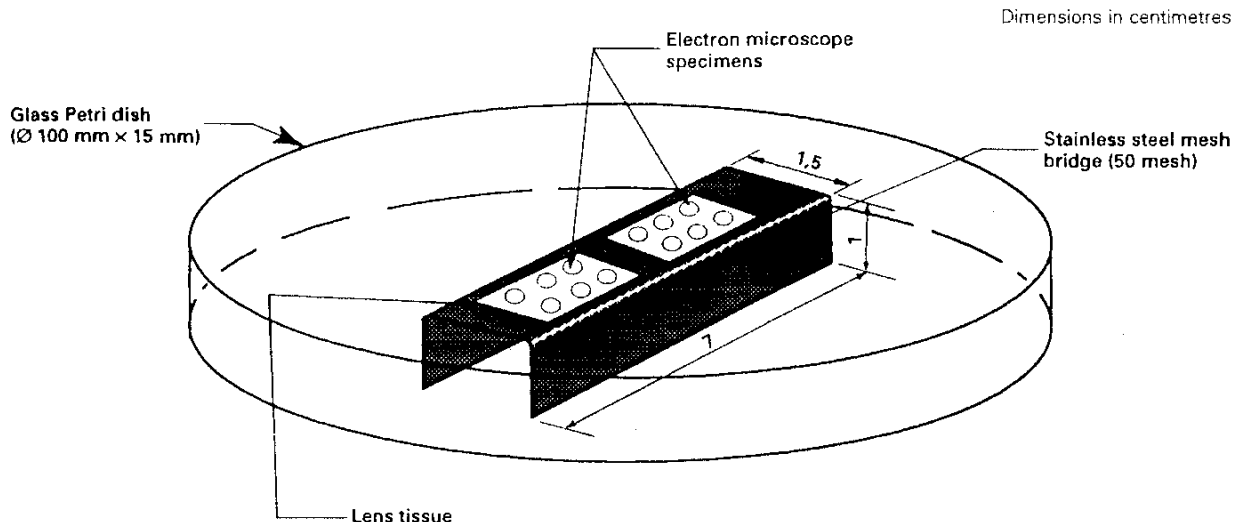
### 7.3.7 Solvent washer (Jaffe washer)

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.



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)

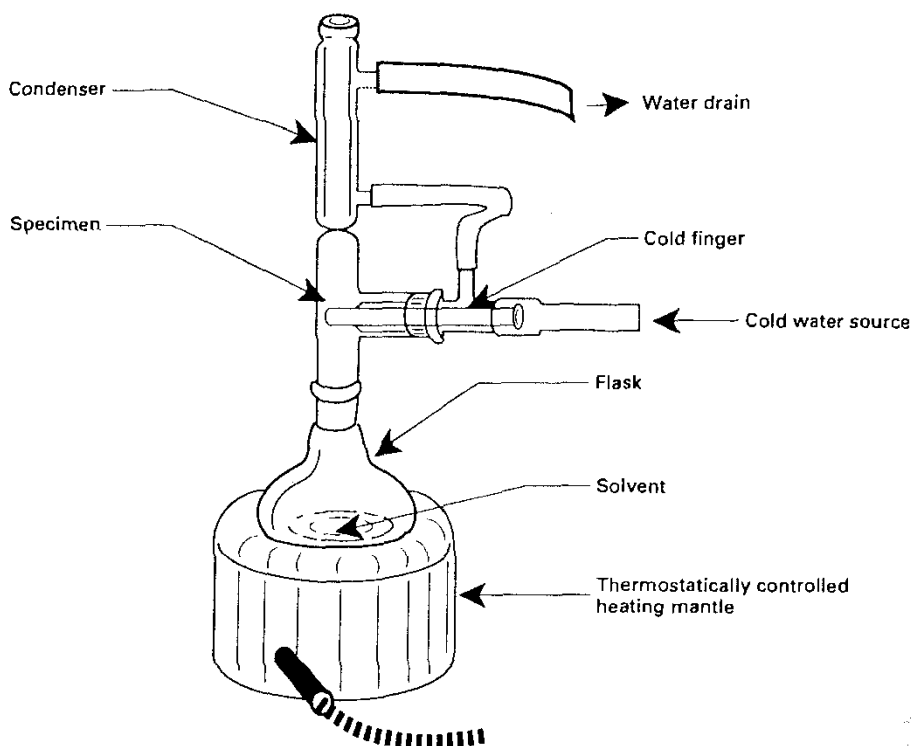


Figure 3 — Example of design of condensation washer

#### 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<sup>2</sup> is assumed, and the TEM grid openings are assumed to be 85 µm<sup>2</sup> 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:

$$S = \frac{A_i}{kA_gV}$$

where

- $A_i$  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 adhere to the exterior surfaces of air sampling cassettes, and these fibres can be inad-

vertently 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 from 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  $15 \mu\text{l}/\text{cm}^2$  to  $25 \mu\text{l}/\text{cm}^2$  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  $20^\circ$  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  $65^\circ\text{C}$  to  $70^\circ\text{C}$ , 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  $0.8 \mu\text{m}$  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 (6.6) 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 ( $\times 300$  to  $\times 1\,000$ ) 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 analysed 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<sup>2</sup>; 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  $\times 20\,000$ , 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:

$$k = \frac{A_f}{A_0 VS}$$

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  $\times 20\,000$ ). 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 $\mu\text{m}$

Consider improving the statistical validity for measurement of asbestos fibres and bundles longer than 5  $\mu\text{m}$  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  $\mu\text{m}$  in accordance with the procedures specified in annex E. Use a magnification of approximately  $\times 10\,000$  for counting all asbestos fibres and bundles longer than 5  $\mu\text{m}$ , or approximately  $\times 5\,000$  if only fibres and bundles within the diameter range 0,2  $\mu\text{m}$  to 3,0  $\mu\text{m}$  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: .....

Air volume: ..... litres

Sample number: .....

File name: .....

Sample filter area: ..... mm<sup>2</sup>

Sample description: .....

Magnification: .....

Preparation date: ..... By: .....

Grid opening dimension: .....  $\mu\text{m}$

Analysis date: ..... By: .....

Computer entry date: ..... By: .....

Level of analysis (C): .....

(A): .....

[illegible]

**Figure 4 — Example of structure counting form**

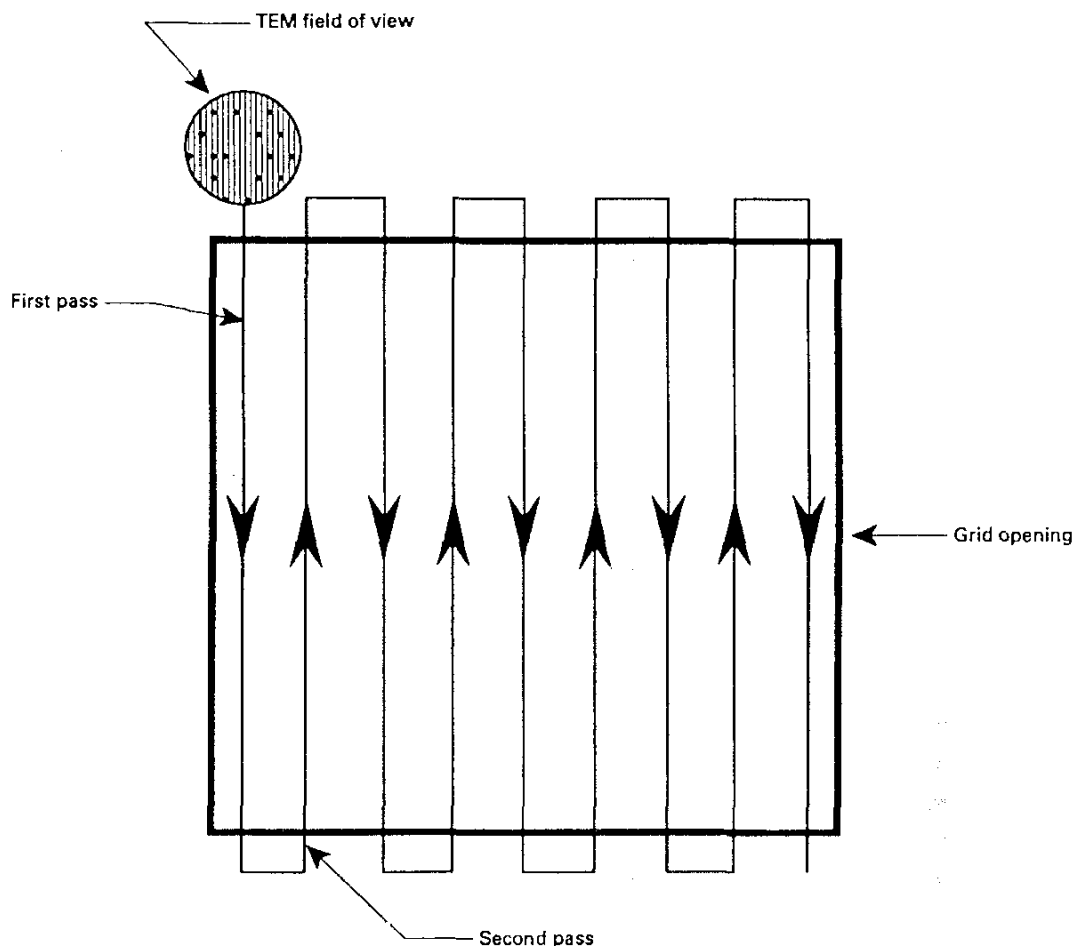


Figure 5 — Example of scanning procedure for TEM specimen examination

### 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/ $\text{mm}^2$ , or if the mean fibre count for asbestos fibres and bundles longer than  $5\text{ }\mu\text{m}$  is more than 0,1 fibre/ $\text{mm}^2$ , 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, par-

ticularly 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) distortion 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  $\pm 25$  % 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<sup>2</sup> 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  $\mu\text{m}$ ;

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
<b>Sample:</b> 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 <sup>2</sup>
Level of analysis (chrysotile):		CD or CMQ
Level of analysis (amphibole):		ADQ
Magnification used for fibre counting:		$\times 20\ 500$
Aspect ratio for fibre definition:		5/1
Mean dimension of grid openings:		95,4 $\mu\text{m}$
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 $\mu\text{m}$ :		7
Number of asbestos fibres and bundles > 5 $\mu\text{m}$ :		10
Number of PCM equivalent asbestos structures:		3
Number of PCM equivalent asbestos fibres:		5

**Figure 6 — 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		Identification <sup>1)</sup>	Structure type	Length	Width	Comments
		primary	total			µm	µm	
A	F4-4	1	1	CD	F	1,7	0,045	Crocidolite
		2	2	CMQ	B	2,6	0,09	
		3	3	ADQ	F	4,0	0,15	
	E3-6	4	4	CD	MC+0	3,5	1,3	
	E5-1	5	5	CD	MD43	7,5	5,0	
			6	CD	MB	7,7	0,30	
			7	CMQ	MF	5,6	0,045	
			8	CD	MB	5,1	0,30	
B	F4-1	6	9	CD	CD+0	6,5	3,0	
			10	CD	CB	3,5	0,15	
			11	CMQ	CR+0	2,6	1,9	
			12	CD	CD31	6,1	3,2	
			13	CMQ	CB	5,6	0,3	
	G5-1	7	14	CMQ	CF	4,0	0,045	
			15	CMQ	CB	3,2	0,090	
			16	CD	B	1,5	0,23	
	E4-4	8	17	AD	F	8,7	0,15	
C	G4-4	10	18	CMQ	CD42	25	5,6	
			19	CMQ	CB	15	0,15	
			20	CMQ	CF	9,4	0,045	
			21	ADQ	CF	3,6	0,30	
			22	CM	CF	4,2	0,045	
	E4-4	11	23	No fibres				Tremolite
			24	ADQ	CD+3	9,4	2,5	
			25	ADQ	CF	7,1	0,30	
	E5-6	11	26	ADQ	CF	6,2	0,10	Amosite Crocidolite
			27	CM	CB	5,1	0,2	
			28	CM	CR+0	3,3	1,8	
			29	CMQ	MC10	3,7	2,1	
	F4-1	12	30	CD	CC+0	7,4	0,5	
		13	31					

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 is maximized. Measure the time required for complete oxidation of the filter. Determine operating parameters which result in complete oxidation 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,  $\lambda L$ , 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

- $\lambda$  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

$$\lambda L = 0,117\ 74D \text{ mm}\cdot\text{nm (smallest ring)}$$

$$\lambda L = 0,101\ 97D \text{ mm}\cdot\text{nm (second ring)}$$

#### 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  $\times 20\,000$ , 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.

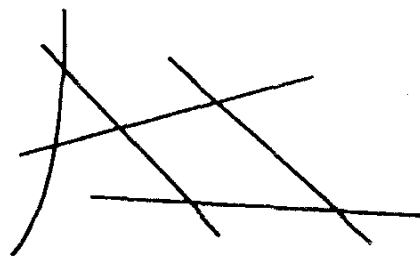
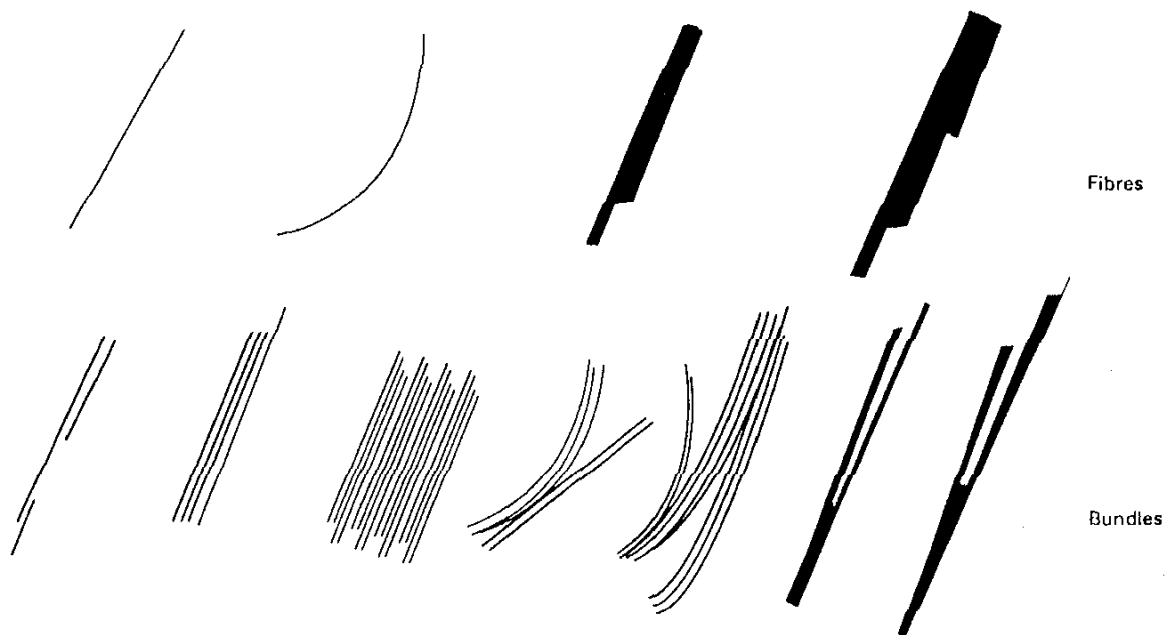
##### C.2.3 Cluster

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.

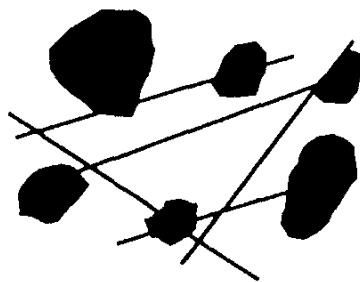
In practice, clusters can occur in which the characteristics of both types of cluster occur in the same structure. Where this occurs, the structure should be defined as a disperse cluster, and then a logical procedure should be followed by recording structure components according to the counting criteria. The procedure for treatment of clusters is illustrated by examples in figure C.2.



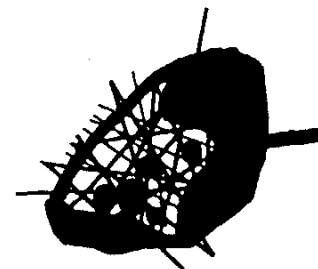
a) Disperse cluster (type D)



b) Compact cluster (type C)

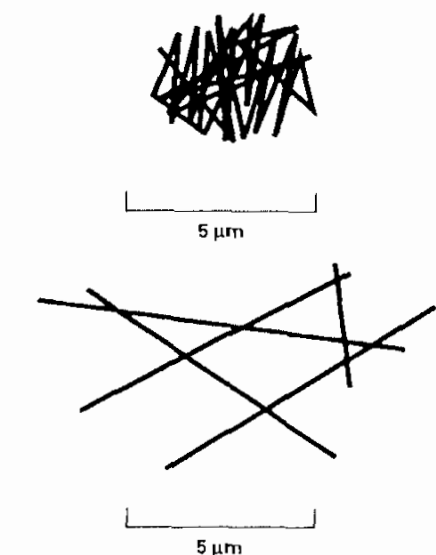


c) Disperse matrix (type D)



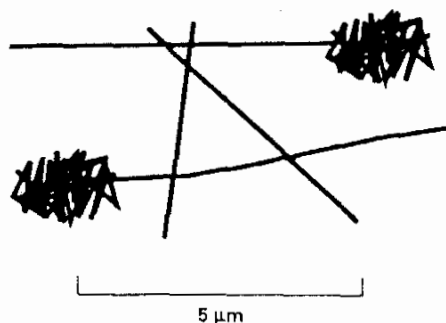
d) Compact matrix (type C)

Figure C.1 — Fundamental morphological structure types



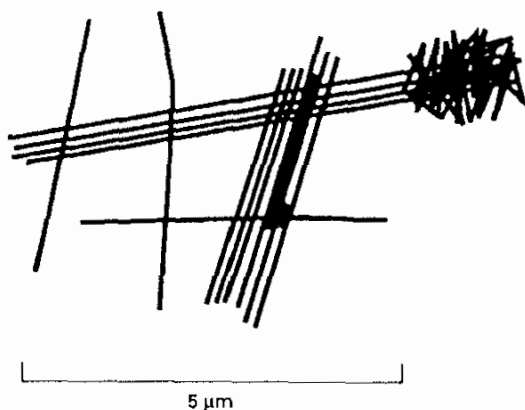
Count as 1 compact cluster containing more than 9 fibres (all fibres shorter than 5  $\mu\text{m}$ )

Record as CC+0



Count as 1 disperse cluster consisting of 5 fibres, 4 of which are longer than 5  $\mu\text{m}$ , 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  $\mu\text{m}$ , 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.

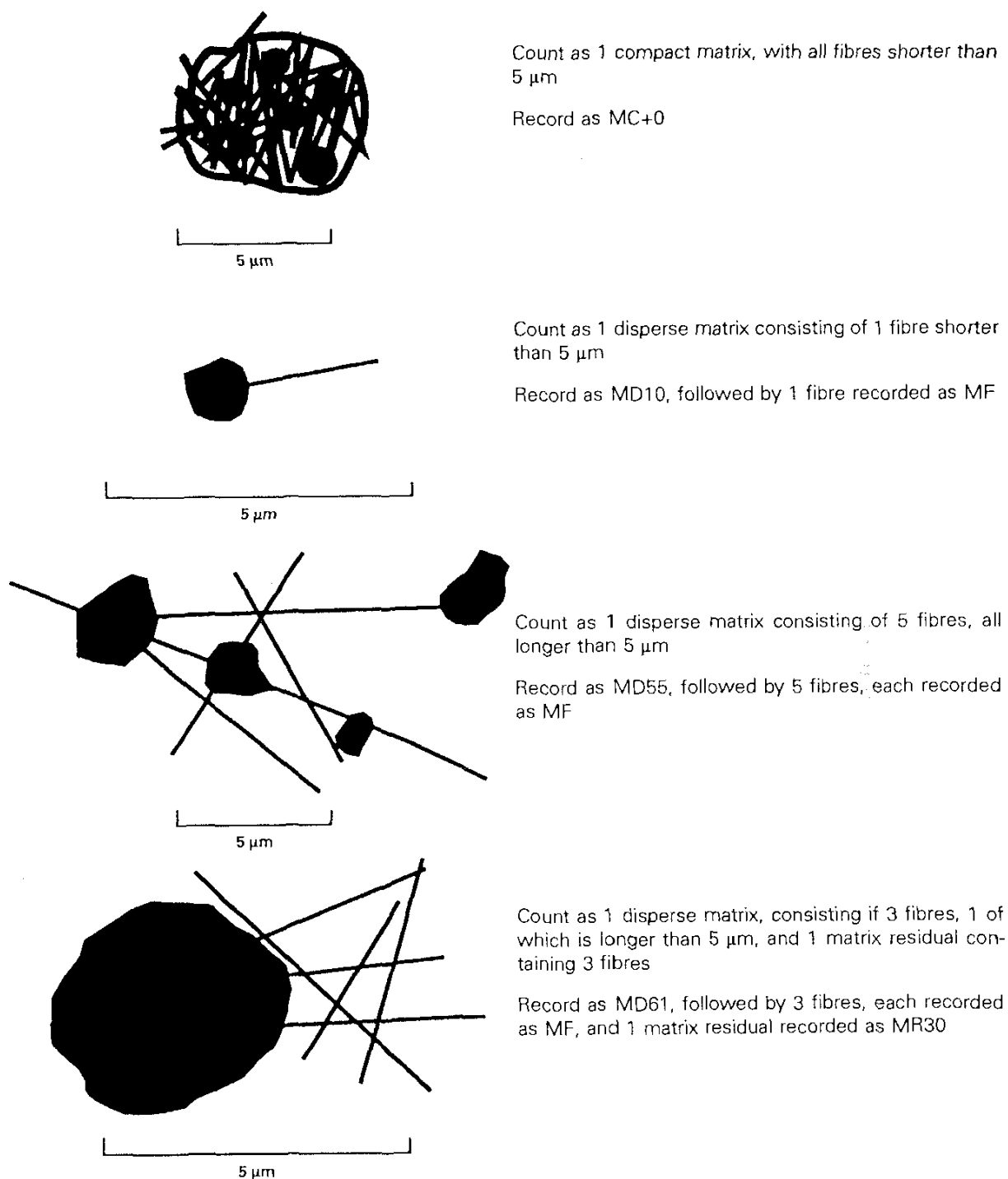


Figure C.3 — Examples of recording of complex asbestos matrices

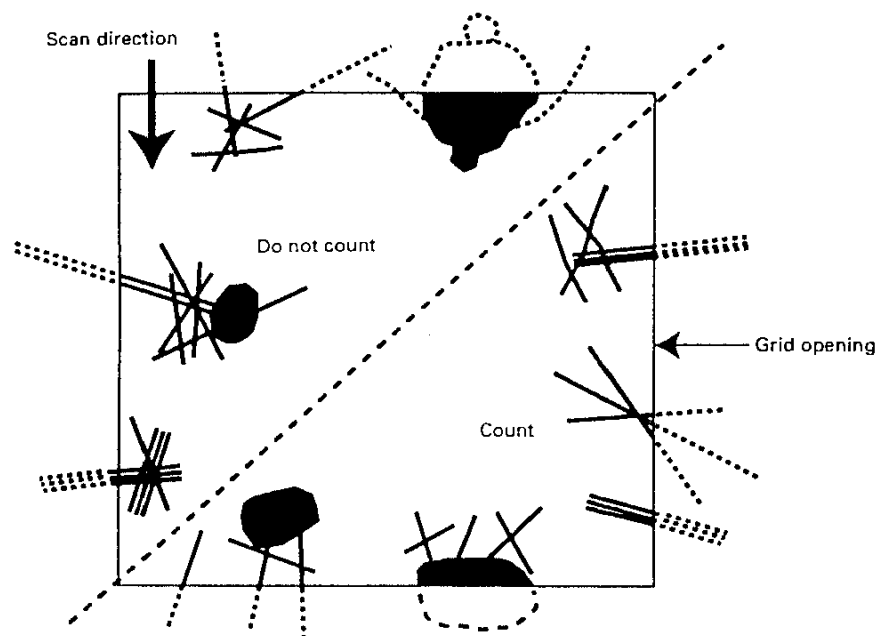


Figure C.4 — Example of counting of structures which intersect grid bars

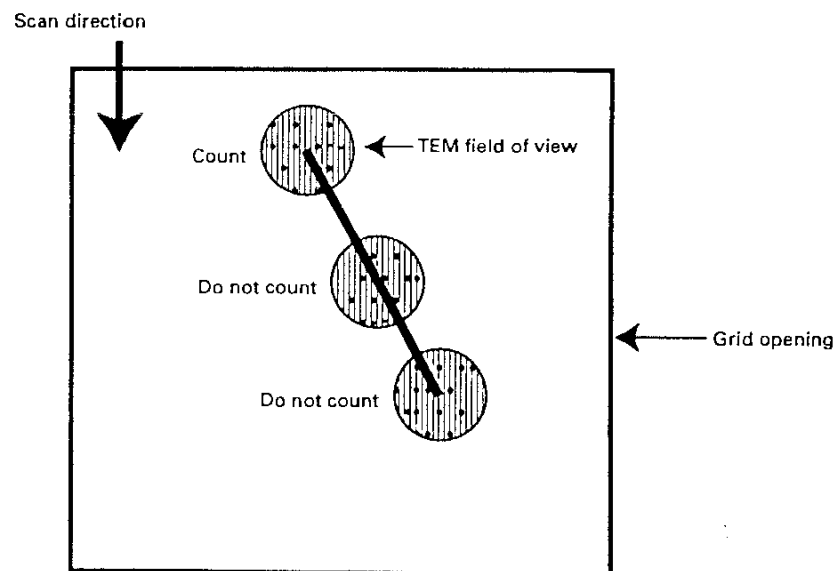


Figure C.5 — Example of counting of fibres which extend outside the field of view

## 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 constituent 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 constituent 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  $\mu\text{m}$  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  $\mu\text{m}$  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 it 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

$$\lambda L = 0,117\ 74D \text{ mm}\cdot\text{nm (first ring)}$$

$$\lambda L = 0,101\ 97D \text{ mm}\cdot\text{nm (second ring)}$$

### 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.

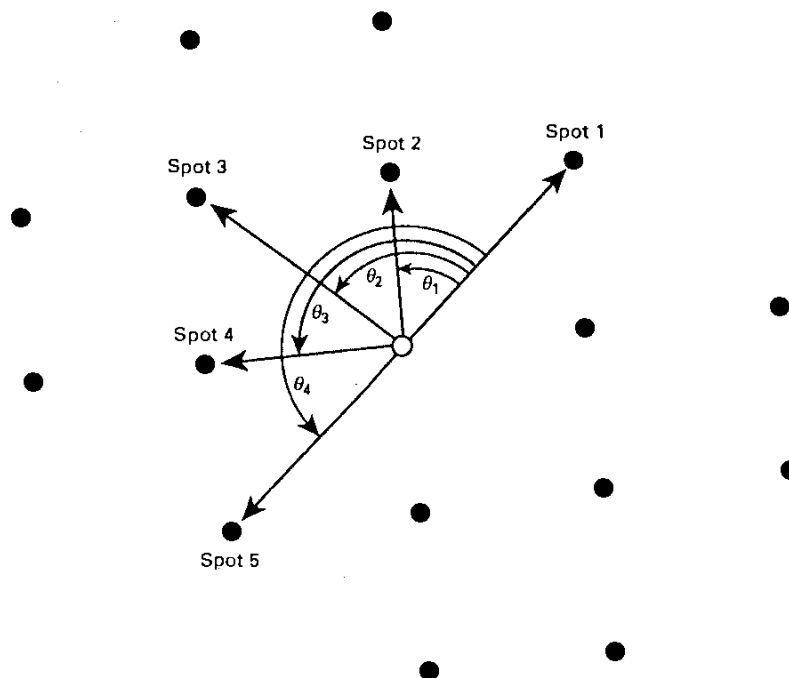


Figure D.1 — Example of measurement of zone-axis SAED patterns

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 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 instrument. 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 other 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 an 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  $\bar{c}$ -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).

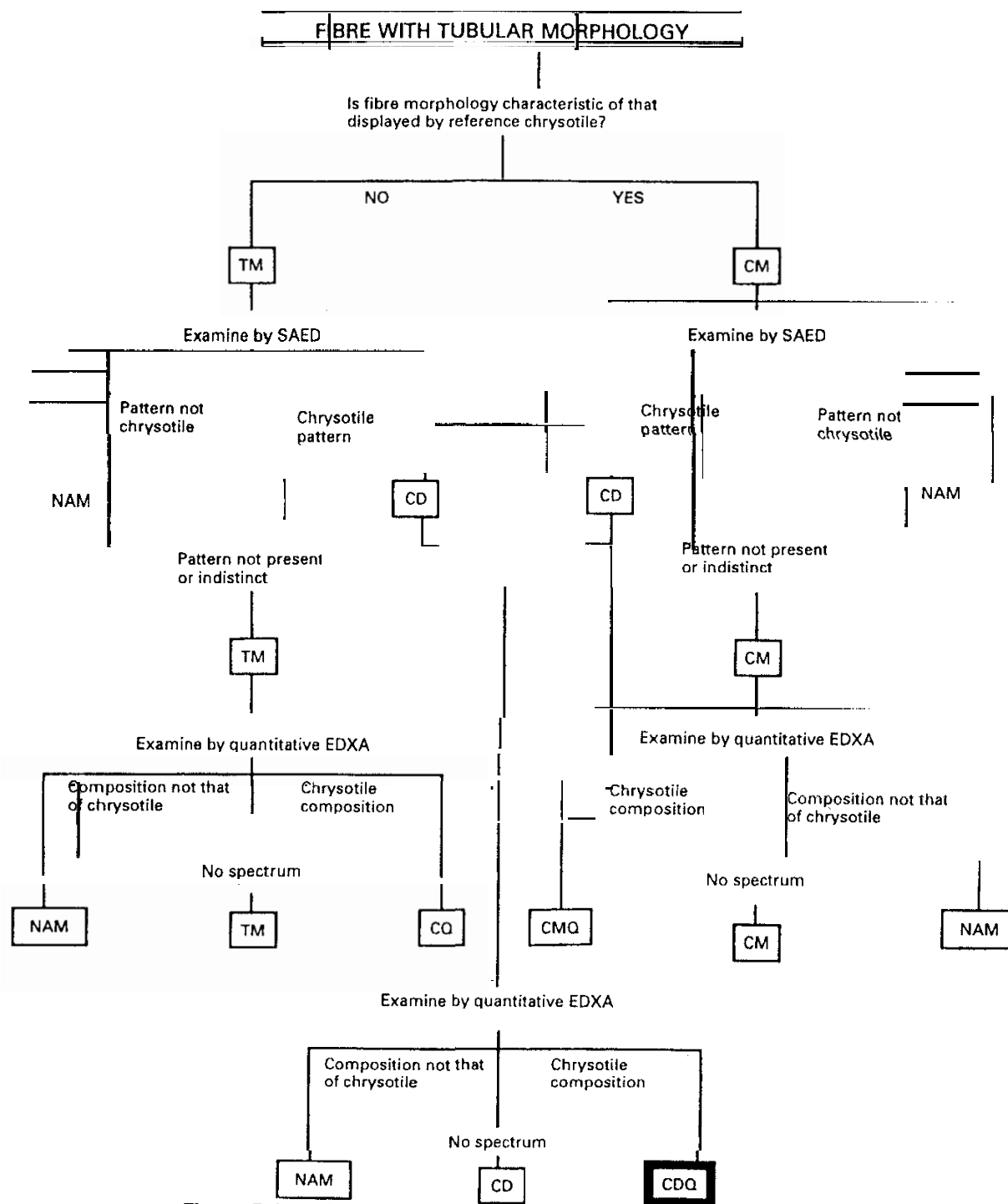


Figure D.2 — Classification chart for fibre with tubular morphology

For classification as CM, the morphological characteristics required are the following:

- the individual fibrils should have high aspect ratios exceeding 5/1, and be about 30 nm to 40 nm in diameter;
- 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;
- 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:

- the (002) reflections should be examined to determine that they correspond closely to a spacing of 0,73 nm;
- the layer line repeat distance should correspond to 0,53 nm;
- there should be "streaking" of the (110) and (130) reflections.

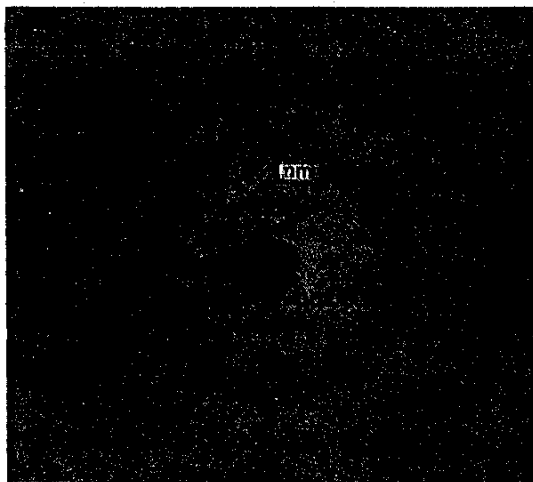


Figure D.3 — Chrysotile SAED pattern

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 CO, 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-

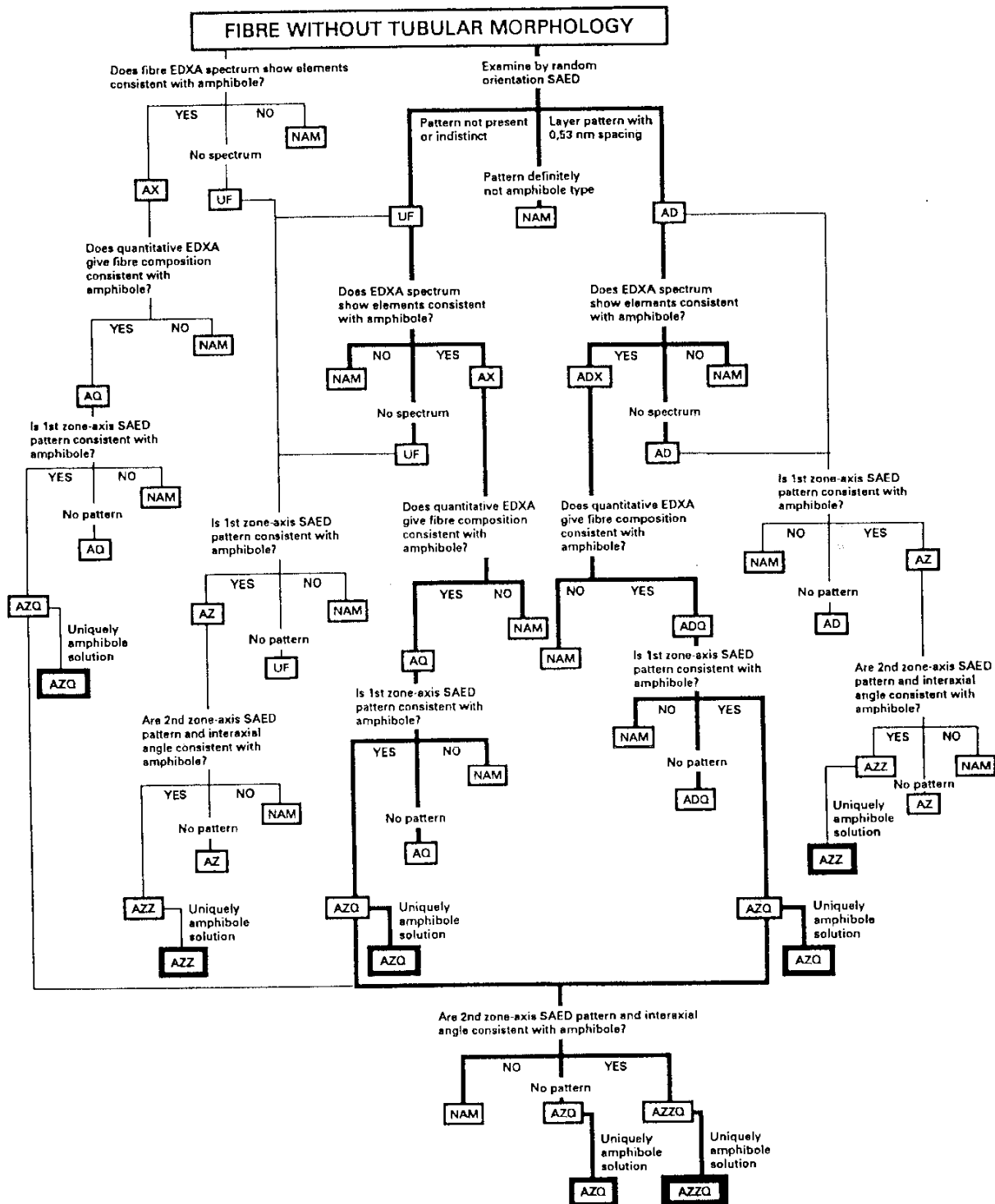


bres leads to contributions to the layers in the patterns by several individual parallel crystals of different axial orientations. This apparently random positioning of the spots along the layer lines, if also associated with a high fibre aspect ratio, is a characteristic of amphibole asbestos, and thus 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 should 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 $\mu\text{m}$ , 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  $\mu\text{m}$ , 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  $\mu\text{m}$ ". For this examination, use a magnification of approximately  $\times 10\,000$ , 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  $\mu\text{m}$ . Record cluster and matrix components only if their lengths exceed 5  $\mu\text{m}$ .

It may also be decided to provide increased statistical precision and improved analytical sensitivity for fibrous structures longer than 5  $\mu\text{m}$ , with diameters between 0,2  $\mu\text{m}$  and 3,0  $\mu\text{m}$ , which have historically been the basis of risk estimation in the occupational environment (PCM equivalent asbestos fibres). Use a magnification of approximately  $\times 5\,000$  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  $\mu\text{m}$ .

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<sup>2</sup>, 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  $\mu\text{m}$ , with diameters between 0,2  $\mu\text{m}$  and 3,0  $\mu\text{m}$  be counted. Use a magnification of  $\times 5\,000$  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 check 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 grid 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

$$\chi^2 = \sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

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:

$$S = \frac{A_f}{kA_gV}$$

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  $\cong$  2,99 times the analytical sensitivity
- 1 structure  $\cong$  4,74 times the analytical sensitivity
- 2 structures  $\cong$  6,30 times the analytical sensitivity
- 3 structures  $\cong$  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:

$$s^2 = \frac{\sum_{i=1}^{k} (n_i - np_i)^2}{(k-1)}$$

where

- $n_i$  is the number of structures on the  $i$ th 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  $i$ th 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

$$L_1 = \frac{n}{k} - \frac{ts}{\sqrt{k}}$$

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  $\cong$  4,74 times the analytical sensitivity
- 2 structures  $\cong$  6,30 times the analytical sensitivity
- 3 structures  $\cong$  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  $\mu\text{m}$  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  $k$ th class;
- $n_i$  is the number of structures in the  $i$ th 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 <sup>1)</sup>	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	53,315	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 mini-

mum 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  $\mu\text{m}$  to 6  $\mu\text{m}$  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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**ICS 13.040.20**

**Descriptors:** air, quality, air pollution, tests, determination, particle density (concentration), asbestos, microscopic analysis.

Price based on 51 pages

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**Appendix L – PCM, NIOSH 7400**  
**(analytical method)**

Various

MW: Various

CAS: Various

RTECS: Various

METHOD: 7400, Issue 2

EVALUATION: FULL

Issue 1: Rev. 3 on 15 May 1989

Issue 2: 15 August 1994

**OSHA :** 0.1 asbestos fiber (> 5 µm long)/cc;  
1 f/cc/30 min excursion; carcinogen

**MSHA:** 2 asbestos fibers/cc

**NIOSH:** 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen

**ACGIH:** 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other  
asbestos, fibers/cc; carcinogen

**PROPERTIES:** solid, fibrous, crystalline, anisotropic

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

SAMPLING		MEASUREMENT	
<b>SAMPLER:</b>	FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm; conductive cowl on cassette)	<b>TECHNIQUE:</b>	LIGHT MICROSCOPY, PHASE CONTRAST
<b>FLOW RATE*:</b>	0.5 to 16 L/min	<b>ANALYTE:</b>	fibers (manual count)
<b>VOL-MIN*:</b>	400 L @ 0.1 fiber/cc	<b>SAMPLE PREPARATION:</b>	acetone - collapse/triacetin - immersion method [2]
<b>-MAX*:</b>	(step 4, sampling) *Adjust to give 100 to 1300 fiber/mm <sup>2</sup>	<b>COUNTING RULES:</b>	described in previous version of this method as "A" rules [1,3]
<b>SHIPMENT:</b>	routine (pack to reduce shock)	<b>EQUIPMENT:</b>	1. positive phase-contrast microscope 2. Walton-Beckett graticule (100-µm field of view) Type G-22 3. phase-shift test slide (HSE/NPL)
<b>SAMPLE STABILITY:</b>	stable	<b>CALIBRATION:</b>	HSE/NPL test slide
<b>BLANKS:</b>	2 to 10 field blanks per set	<b>RANGE:</b>	100 to 1300 fibers/mm <sup>2</sup> filter area
<b>ACCURACY</b>		<b>ESTIMATED LOD:</b>	7 fibers/mm <sup>2</sup> filter area
<b>RANGE STUDIED:</b>	80 to 100 fibers counted	<b>PRECISION (<math>\hat{S}_r</math>):</b>	0.10 to 0.12 [1]; see EVALUATION OF METHOD
<b>BIAS:</b>	see EVALUATION OF METHOD		
<b>OVERALL PRECISION (<math>\hat{S}_{rT}</math>):</b>	0.115 to 0.13 [1]		
<b>ACCURACY:</b>	see EVALUATION OF METHOD		

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

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

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

## REAGENTS:

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

\* See SPECIAL PRECAUTIONS.

## EQUIPMENT:

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



## EQUIPMENT:

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

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

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## SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.  
NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E, of 100 to 1300 fibers/mm<sup>2</sup> ( $3.85 \cdot 10^4$  to  $5 \cdot 10^5$  fibers per 25-mm filter with effective collection area  $A_c = 385$  mm<sup>2</sup>) for optimum accuracy. These variables are related

to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

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

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

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

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

## SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤1.46. This method collapses the filter for easier focusing and produces permanent (1 - 10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7,8,9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9,10,11].

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

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [2].  
NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
  - a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.  
NOTE: Static electricity will usually keep the wedge on the slide.

- b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250  $\mu$ L acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their ports.

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

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

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

- d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.
- e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.

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

## CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturers instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.
  - a. Each time a sample is examined, do the following:
    - (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.
    - (2) Focus on the particulate material to be examined.
    - (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.
  - b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:
    - (1) Center the HSE/NPL phase-contrast test slide under the phase objective.
    - (2) Bring the blocks of grooved lines into focus in the graticule area.
 

NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.
    - (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.
11. Document the laboratory's precision for each counter for replicate fiber counts.
  - a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field

and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

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

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

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.  
NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

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

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm<sup>2</sup>) exceeds  $2.77 (X)S_r'$ , where  $X$  = average of the square roots of the two fiber counts (in

fiber/mm<sup>2</sup>) and  $S_r' = \frac{S_r}{2}$ , where  $S_r$  is the intracounter relative standard deviation for the

appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

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

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

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

## MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.
17. Adjust the microscope (Step 10).  
NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25  $\mu$ m) [4].
18. Counting rules: (same as P&CAM 239 rules [1,10,11]: see examples in APPENDIX B).
  - a. Count any fiber longer than 5  $\mu$ m which lies entirely within the graticule area.
    - (1) Count only fibers longer than 5  $\mu$ m. Measure length of curved fibers along the curve.
    - (2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.
  - b. For fibers which cross the boundary of the graticule field:

- (1) Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.
  - (2) Do not count any fiber which crosses the graticule boundary more than once.
  - (3) Reject and do not count all other fibers.
  - c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
  - d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.
19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.
- NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.
- NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 µm), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].
- NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.
- NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].
- NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/mm<sup>2</sup>) is defined as the average count (fibers/field) divided by the field (graticule) area (mm<sup>2</sup>/field).

## CALCULATIONS AND REPORTING OF RESULTS

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

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

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

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

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

NOTE: Periodically check and adjust the value of A<sub>c</sub>, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.

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

#### EVALUATION OF METHOD:

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

1. Sampling:

The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m<sup>3</sup> full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].

2. Sample Preparation Technique:

The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.

3. Measurement:

a. The Walton-Beckett graticule standardizes the area observed [14,18,19].

b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].

c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm<sup>2</sup> filter area (a total of 78.5 fibers counted in 100 fields, each with field area = .00785 mm<sup>2</sup>.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S<sub>r</sub> in the range of 0.10 to 0.17 [21,22,23].

- B. Interlaboratory comparability:

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

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

\* Under AIA rules, only fibers having a diameter less than 3  $\mu\text{m}$  are counted and fibers attached to particles larger than 3  $\mu\text{m}$  are not counted. NIOSH A Rules are otherwise similar to the AIA rules.

\*\* See Appendix C.

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

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

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

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

Thus  $S_r$  is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual  $S_r$  found in a number of studies is greater than these theoretical numbers [17,19,20,21].

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

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

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

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

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

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

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter,  $0.00785 \text{ mm}^2$  counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

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

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

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

Figure 1. Interlaboratory Precision of Fiber Counts



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

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

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

where  $S_r$  = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory  $S_r$  when approximately 100 fibers are counted.

$X$  = total fibers counted on sample

LCL = lower 95% confidence limit.

UCL = upper 95% confidence limit.

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

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#### APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE:

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

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

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

Example: If  $L_o = 112 \mu\text{m}$ ,  $L_a = 4.5 \text{ mm}$  and  $D = 100 \mu\text{m}$ , then  $d_c = 4.02 \text{ mm}$ .

8. Check the field diameter,  $D$  (acceptable range  $100 \mu\text{m} \pm 2 \mu\text{m}$ ) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range  $0.00754 \text{ mm}^2$  to  $0.00817 \text{ mm}^2$ ).

## **APPENDIX B: COMPARISON OF COUNTING RULES:**

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

Figure 2. Walton-Beckett graticule with fibers.

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

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

**APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS**

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

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

**"B" COUNTING RULES:**

1. Count only ends of fibers. Each fiber must be longer than 5  $\mu\text{m}$  and less than 3  $\mu\text{m}$  diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3  $\mu\text{m}$  in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

**APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION**

<u>fiber density on filter*</u>		<u>fiber concentration in air, f/cc</u>	
<u>fibers</u>		<u>400-L air</u>	<u>1000-L air</u>
<u>per 100 fields</u>	<u>fibers/mm<sup>2</sup></u>	<u>sample</u>	<u>sample</u>
200	255	0.25	0.10
100	127	0.125	0.05
LOQ.....80.....	102.....	0.10.....	0.04
50	64	0.0625	0.025
25	32	0.03	0.0125
20	25	0.025	0.010
10	12.7	0.0125	0.005
8	10.2	0.010	0.004
LOD.....5.5.....	7.....	0.00675.....	0.0027

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