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REMEDIAL INVESTIGATION REPORT
SOUTH BAY ASBESTOS SITE
Volume I

ALVISO, CALIFORNIA

DECEMBER 1988

REM II

PERFORMANCE OF REMEDIAL RESPONSE ACTIVITIES AT UNCONTROLLED HAZARDOUS WASTE SITES

U.S. EPA CONTRACT NO. 68-01-6939

CDM Federal Programs Corporation
CAMP DRESSER & McKEE INC.
ROY F. WESTON INC.
WOODWARD-CLYDE CONSULTANTS
CLEMENT ASSOCIATES, INC.
ICF INCORPORATED
C.C. JOHNSON & MALHOTRA, P.C.

REMEDIAL INVESTIGATION REPORT
SOUTH BAY ASBESTOS SITE
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Prepared for:

U.S. Environmental Protection Agency
215 Fremont Street
San Francisco, CA 94105

Prepared by:

Camp Dresser & McKee Inc.
100 Spear Street, Suite 700
San Francisco, CA 94105

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SECTION 1.0

INTRODUCTION

1.0 INTRODUCTION

1.1 OBJECTIVES OF THE REMEDIAL INVESTIGATION

The U.S. Environmental Protection Agency (EPA) has been concerned with the potential health hazards associated with exposure to asbestos since the early 1970s. The concern is based on medical evidence that exposure to airborne asbestos may cause cancer as well as non-cancerous respiratory diseases (EPA, 1986a). More recently, the concern has expanded to include exposure outside the workplace because of the widespread use of asbestos-containing material.

In 1983, asbestos was discovered in the community of Alviso, located in San Jose, California. Past disposal of asbestos waste and construction of a flood control levee (the "ring levee") with asbestos-bearing fill were identified as contributing to the contamination problem at the site. The site was added to the National Priority List (NPL) in 1984, and in June 1986, EPA initiated a Remedial Investigation/Feasibility Study (RI/FS) for the South Bay Asbestos site. This document presents the results of the RI investigation.

The objectives of the RI/FS are to determine the nature and extent of hazardous substances contamination at the site and to determine the extent to which this contamination may pose a threat to human health and the environment. These were accomplished by the collection and analysis of samples from the site, interpretation of these data and of data collected in previous investigations, and the development of a risk assessment. All sampling and laboratory analyses were conducted using Region IX EPA-approved methods, in accordance with the EPA-approved Sampling and Analysis Plan and Quality Assurance Project Plan.

This RI Report covers the entire site investigation, although a portion of the site, the ring levee, has been investigated separately in another document, the Ring Levee Operable Unit Feasibility Study Report (EPA, 1988a). Both documents were prepared in accordance with the provisions of

the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) (42 U.S.C. 960.1, et seq) and the National Contingency Plan (NCP) (40 C.F.R.S300 et. seq). The U.S. EPA's document Draft Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERLA (EPA, 1988b) was also followed. Appendix A is a Glossary of Terms to assist the reader in interpreting this document.

1.2 SITE BACKGROUND

1.2.1 SITE LOCATION AND DESCRIPTION

The South Bay Asbestos Superfund site is located at the northern end of the Santa Clara Valley and at the southernmost extent of San Francisco Bay (Figure 1-1). The site, which includes the community of Alviso, is a mix of residential, commercial, light industrial, and agricultural land uses, comprising an area of about 550 acres (Figure 1-2) in the northernmost neighborhood or section of the City of San Jose. The older section of the community, located west of Gold Street and north of the Guadalupe River, is a designated National Register Historic District. About 1,700 residents live in Alviso, a quiet undeveloped suburb surrounded by Highway 237 to the south, rapidly-growing Santa Clara to the west and south, and expanding office development to the east and northeast.

As shown on Figure 1-2, the site boundary parallels the ring levee along the east and north sides of the community. The western boundary of the site is formed by the salt evaporation ponds on the edge of the San Francisco Bay. State Highway 237 is the southern boundary of the South Bay Asbestos site. The site boundaries were drawn to include all potential areas of asbestos contamination, based on the discovery and subsequent sampling events at the site, described in Section 2.0.

The community of Alviso is adjacent to some of the last remaining Bay wetlands. Over the last century, most of the tidal flats and marshlands which surrounded San Francisco Bay have been filled or altered, so that less than 20 percent of the original wetland area remains today (City of San Jose, 1984). Near Alviso, a fragment of the marshland survives as the

New Chicago Marsh, a National Wildlife Refuge about 300 acres in size. The Refuge has an active public education program through its Environmental Education Center, located about a mile northeast of the town and administered by the U.S. Fish and Wildlife Service.

The wetlands adjacent to Alviso are a significant wildlife habitat because they provide an interface between fresh and saltwater environments. The wetlands support several endangered or threatened species, including the Salt Marsh Harvest Mouse (Reithrodontomys raviventris), Golden Eagle (Aquila Chrysaetos), and Peregrine Falson (Falco Peregrinus). The burrowing owl is a protected species of special concern. Small mammals and a great number of birds and waterfowl species use the wetlands and surrounding "upland" habitats, i.e., land elevated a few feet above the marsh.

1.2.2 SITE HISTORY

Alviso is one of the oldest communities in the San Francisco Bay Area, having been settled about 200 years ago at the time that Mission Santa Clara was founded (City of San Jose, 1984). The town was located on an alluvial fan and natural levee, which provided slightly higher, well-drained building areas above the surrounding marsh (City of San Jose, 1984). As the Santa Clara Valley agricultural industry developed in the present century, the area around and including Alviso was used for fruit and vegetable farming. However, extensive withdrawal of groundwater for irrigation throughout the valley caused considerable ground subsidence. Alviso subsided about six feet (City of San Jose, 1984 and Aqua-Terra, 1986) between 1934 and 1967. The subsidence is important to the site history since it may have been the underlying impetus for site landfilling activities.

The Keasby & Mattison Company operated an asbestos pipe manufacturing plant about four miles south of the site at 2885 Lafayette Street, Santa Clara, from August 1953 through June 1962. CertainTeed Corporation purchased Keasby & Mattison Company on June 1, 1962 and manufactured asbestos/cement pressure and sewer pipe and fittings until June 9, 1982. Several types of

waste were produced at the CertainTeed plant, including broken asbestos/cement pipe, machining and processing waste, settling tank waste, and empty asbestos fiber bags. According to company employees, two to three truckloads of scrap pipe, machining waste, and material from settling basins were transported to local landfills daily. Several landfills were located within the site boundaries, including the Santos Landfill, Marshland Development Corporation Landfill (also known as the Hoxie Landfill, the Edgewater Landfill, the Leslie Salt Landfill, or the "fill dump"), and the Sainte Claire Corporation Landfill (Figure 1-2). All three landfills may have received asbestos-containing wastes. Waste from the asbestos pipe plant may also have been dumped within the community of Alviso, north of State street, at the location of an abandoned race track. Filling also occurred in many other locations in Alviso, based on interpretation of a series of historical aerial photographs provided by EPA. A number of truckyards are located along the northwest side of State Street, and are built on fill soils. Figures 1-3 through 1-6 show the progression of landfill and fill activity based on a series of historical aerial photographs.

In 1963, the Santos Landfill was disrupted during channelization of the Guadalupe River by the Santa Clara Valley Water District (SCVWD). The project was undertaken to provide flood control for the area. Material excavated from the new channel, or the landfill area (as shown in Figure 1-4), was desposited in the old river course or used to construct the levees (Aqua-Terra, 1986).

A large flood occurred in March 1983, which caused the evacuation of the town for sixteen days as flood waters overtopped Coyote Creek channels and existing levees. Immediately after the flood, the City of San Jose constructed a small levee along the San Francisco Bay side of the City of Alviso. The levee was called the "interim ring levee" or "ring levee" and was constructed in an attempt to divert possible future flood waters from the community. The ring levee is approximately two miles long, an average of six feet in height and eight to twelve feet in width (Figure 1-2). The levee materials was apparently imported from the Raische Quarry at 55 Hillsdale Avenue in San Jose with additional material imported from an

excavation on Homestead Road in Cupertino (Schwartz, personal communication). The fill is comprised largely of serpentine, an asbestos-containing rock.

Waste asbestos/cement pipe was discovered on August 8, 1983 in the levee of the Guadalupe River adjacent to Moffett Street in the City of Alviso. The discovery of asbestos pipe and waste occurred on property owned by the SCVWD and the City of San Jose during construction of a flood-control outfall structure. An industrial hygienist from the California Occupational Safety and Health Administration (CAL-OSHA) collected samples of excavated material which were found to contain concentrations of asbestos ranging from 20 to 40% by weight. Cal-OSHA referred the situation to California Department of Health Services (DHS). After confirming the presence of asbestos in the Guadalupe River levee, DHS ordered SCVWD to remove all the asbestos contaminated soil. As described in detail in Section 2.0, DHS collected additional samples of soil throughout Alviso. The sample results indicated that asbestos was randomly distributed throughout the community of Alviso including the ring levee. Based on the soil results, DHS completed a Hazardous Waste Site Ranking for the site, which received a score of 41.87 and was listed 17th on the proposed revised State Superfund list in December of 1983. In June of 1984, EPA completed its Hazard Ranking procedures and scored the site as 44.68. The site was added to the NPL in October 1984.

In June and August 1985, DHS conducted "worst case" scenario field experiments at the ring levee to determine if the asbestos present in the soils could pose a significant health risk. DHS forwarded the results to EPA which in turn forwarded the results to the Department of Health and Human Services, Agency for Toxic Substances Control and Disease Registry (ATSDR). ATSDR recommended that remedial measures be implemented to "stabilize those sites to prevent the asbestos from being suspended in the air where residents may inhale the fibers".

Based on the results generated by DHS field experiments, DHS initiated an Operable Unit Feasibility Study (OUFS) to select a permanent remedy for the levee. At the same time, EPA agreed to implement interim remedial measures

at the site which included paving of dirt lots and streets, and paving and cleanup at the Environmental Education Center. EPA sprayed the ring levee with a polymer dust suppressant to control asbestos dust in May 1986, and again in 1987; the City of San Jose continued the spraying in 1988.

DHS' OUFS was completed in April 1986 and recommended a clean soil cover for the levee. However, later that year, DHS referred the ring levee to EPA for further investigation and possible remediation. EPA had initiated the RI/FS for the entire site in 1986 under the direction of the Army Corps of Engineers (COE). However, because of unforeseen difficulties, EPA transferred the site from COE-lead to an EPA Remedial Contractor, Camp Dresser & McKee. This document is the remedial investigation report for the entire site, although the ring levee has been investigated as an "operable unit" in a separate report (EPA, 1988a).

The activities above have included community involvement and participation. State officials first communicated the asbestos problem to Alviso residents in 1983. Many community meetings have occurred since to assure that the residents are aware of the RI/FS process and to gather their input.

1.3 ASBESTOS IN THE ENVIRONMENT

Unlike some environmental substances that are discrete entities defined by a fixed chemical structure, asbestos comprises a group of materials with a broad range of chemical composition, crystalline structure, and physical properties, identified with diverse terminology.

Asbestos is a generic term used to describe a group of naturally occurring fibrous silicate minerals. These fibrous silicates fit into two mineralogical groups: serpentines and amphiboles. The most abundant type of asbestos is chrysotile, the fibrous form of the serpentine group. Chrysotile is composed of parallel curly fibers that can shear into small fibrils that take the shape of spirally wound tubes. Crocidolite and amosite are the two most common fibrous varieties of the amphibole group, although they are relatively uncommon compared with chrysotile. Unlike the rolled-up scroll configuration of chrysotile fibers, amphiboles crystallize

in straight double chains resulting in an elongated needle-like structure. Amphiboles are typically more brittle than chrysotile and tend to cleave longitudinally. Amphiboles, because of their dart-like characteristics, have greater tendency to become airborne than do the curly chrysotile fibers (NRC, 1984). These minerals may occur in nonfibrous form, in which case they are not classified as asbestos.

The high tensile strength, flexibility, heat and chemical resistance, and favorable frictional properties of asbestos fiber make it adaptable to a large number of uses. Depending on the length of fibers and other characteristics, asbestos can be spun or woven, used as structural reinforcement in materials such as cement, plastic and asphalt, or be pressed to form paper. There are an estimated 2000 current uses of asbestos, including textiles, electrical insulation, filtration media, brake linings, floor tile, and paints. As a consequence of man's utilization of asbestos, coupled with the natural occurrence of the minerals, asbestos fibers can be found virtually everywhere in the environment.

Asbestos is regulated in the work place by the Occupational Safety and Health Administration (OSHA), and EPA has issued legislation under the Toxic Substances Control Act (TSCA) to control asbestos in schools. Asbestos emissions are controlled at asbestos facilities under the Clean Water Act, Safe Drinking Water Act, and Clean Air Act and associated National Emission Standards for Hazardous Air Pollutants (NESHAPs). EPA is currently involved in developing regulations to control asbestos end products, and is investigating the role of naturally-occurring asbestos in ambient exposure. However, asbestos in the general environment, whether from naturally occurring deposits of asbestos minerals or from product manufacture or use, is not regulated and the risks from very low levels of ambient asbestos are not clearly defined. There are no established cleanup levels for environmental asbestos.

In California, serpentine is the designated state rock, and fibrous serpentine is widespread. Ambient levels in air tend to be higher than other parts of the country. In cases where the rock has been quarried and

transported to other areas for use as fill or road-building material, it is no longer considered naturally-occurring and becomes a potential concern for EPA and state health officials.

1.3.1 TOXICITY OF ASBESTOS

The adverse human health effects from exposure to asbestos are extremely serious. Asbestos is a known human carcinogen that also causes other lung diseases (EPA, 1986a). Asbestos has been thoroughly examined in numerous epidemiology studies. The diseases that have been repeatedly identified are asbestosis, lung cancer, and mesothelima. Some studies also associate asbestos exposure with cancers of the larynx, pharynx, gastrointestinal tract, kidney, and ovary, and with respiratory disease, such as pneumonia. The properties of asbestos that relate to exposure and health effects are fiber size and aspect ratio, durability, flexibility and tensile strength, surface area and charge, and chemical composition. Major health effects are summarized below, and are discussed further in Section 7.0.

Lung cancer is currently responsible for the largest number of deaths from exposure to asbestos. It has been associated with exposure to all the principal commercial asbestos fiber types. Excess lung cancer has been documented in groups involved with the mining and milling of asbestos and the manufacture and use of asbestos products. Studies (Nicholson et al., 1979; Seidman, 1984; Selikoff et al., 1979 as cited in Appendix K) in which the extent of exposure can be approximated provide evidence that lung cancer increases linearly with both level and duration of exposure. Cigarette smoking and asbestos exposure have a strong synergistic interaction in the development of lung cancer as shown in a study of 12,051 asbestos workers (Hammond et al., 1979 as cited in Appendix K). Consequently, when exposed to asbestos, the risk of lung cancer for smokers is much higher than that for nonsmokers exposed to asbestos.

Many human studies (Selikoff el al., 1979; Nicholson et al., 1982, as cited in Appendix K) have also shown that exposures to asbestos produce mesotheliomas, which are cancers that occur as thick diffuse masses in the serous membranes (mesothelia) that line body cavities. Mesotheliomas occur

in the pleura (the membrane that surrounds the lungs and lines the lung cavity) and the peritoneum (which surrounds the abdominal cavity). Epidemiology studies suggest that the incidence of mesothelioma is related to dose and time from first exposure. Association of mesothelioma with smoking is weak or nonexistent. Asbestos fibers appear, by far, to be the most common cause of mesotheliomas.

Asbestosis, which involves fibrosis of lung and pleural tissues, is another serious chronic asbestos disease, but it is associated with very high levels of exposure to asbestos. Asbestosis is diagnosed from findings which may include radiographic changes, breathlessness, and abnormal lung function. A history of occupational exposure to asbestos is often a key feature of its diagnosis. Asbestosis can appear and progress decades after exposure to asbestos fibers.

The conclusions from epidemiology studies concerning the health effects of asbestos are also supported by results of laboratory studies. Animals treated with asbestos have shown increased incidence of fibrosis, lung cancer, and mesotheliomas. All commercial forms and several other types of asbestos are implicated from a variety of modes of exposure.

Most occupational studies have been conducted on populations exposed to high airborne concentrations of asbestos for relatively long periods of time. However, short-term occupational exposures have also been shown to increase the risk of lung cancer and mesothelioma. In addition, there are many documented cases of mesothelioma linked to long-term exposure to low concentrations of asbestos.

Some evidence of adverse health effects from non-occupational asbestos exposure also exists. Persons who lived in the households of asbestos workers have developed pleural mesothelioma and asbestos-related radiographic changes. In an ongoing study, four cases of mesothelioma have been diagnosed among 626 family contacts of amosite workers. These figures are much higher than that expected to be found among the general population. In addition, 35.9 percent of the contacts showed chest x-ray abnormalities as compared with 4.6 percent of control subjects drawn from

the same community. A number of mesotheliomas have also been documented among populations whose only identified exposure was from living near asbestos mining areas, asbestos product factories, or shipyards where asbestos use had been very heavy. An estimated 1,600 cases of mesothelioma occur yearly in the U.S. among various population exposed to asbestos.

In addition to exposure to asbestos fibers in the air, the general population is also exposed through various oral sources, including drinking water containing asbestos. Because of the potential for oral exposure as well as the excess of gastrointestinal tract cancers that has frequently been found in occupational groups exposed to asbestos in the air, there has been much study of the possible health effects of ingestion of asbestos fibers. Despite those efforts, evidence showing health effects from ingestion is still ambiguous.

1.3.2 PROBLEMS WITH ASBESTOS MEASUREMENT

Although the role of asbestos as a cause of cancer is clear, the ways in which fibers cause disease are not well understood, and this has complicated efforts to measure asbestos successfully. Asbestos researchers have not agreed upon which attributes of asbestos are important to measure to assess risk, including size and shape of individual fibers, number of fibers, total mass of fibers, inclusion of asbestos bundles, clusters, and matrix debris in the fiber count, and asbestos mineralogical type. For example, there tends to be agreement that longer, thinner asbestos fibers (those longer than 5 microns in length with an aspect ratio greater than 3 to 1) are more carcinogenic, i.e., The "Stanton Hypothesis" (Stanton et al., 1981; OSHA, 1986; CARB, 1986). However, other researchers question this approach, suggesting that both long and short fibers may be biologically active (Schneiderman et al., 1981). In addition to fiber dimension, surface chemistry of the asbestos fibers may play a role in causing disease (Mossman, 1983 as cited in Appendix K). Further, there is disagreement whether mineral type is a factor in disease causation. Some would argue (Morgan and Seaton, 1984, as cited in Appendix K) that

chrysotile asbestos may partially dissolve in weakly acidic environments, facilitating fiber clearance from the lung. On the other hand, EPA (1986a) holds that all asbestos mineral types are equally carcinogenic.

To compound the problem, analysis of ambient samples for asbestos is much more difficult than occupational or work place samples, because the concentration of asbestos in the environment is much lower. Asbestos fibers found in ambient air are typically too short and thin to be detected by conventional microscopes, and may be agglomerated with other particulate matter so that they are masked or hidden. Further, although EPA has attempted to standardize asbestos analytical techniques, differences in sample handling, preparation, instrument capabilities, operator proficiency, and counting procedures make it extremely difficult to compare results from different laboratories. In short, accurate measurement of asbestos is impeded by many factors, greatly complicating any estimates of environmental risk. The advantages and disadvantages of each analytical technique used during the study are described briefly below. Analytical methods are included in Appendix B.

Polarized Light Microscopy (PLM)

The analytical technique employed for soil samples by previous investigators at the South Bay Asbestos Site is polarized light microscopy (PLM). Since the late 1970's, PLM has been the preferred method for detecting asbestos in bulk materials such as fire proofing and insulation. The advantages of PLM over other techniques include the ability to 1) identify all mineralogical asbestos types, 2) distinguish between asbestos minerals and their non-fibrous analogs, 3) identify most non-asbestos fibers, 4) identify most non-fibrous components of samples, and 5) be performed quickly and inexpensively.

As with most analytical techniques, limits exist to the information that can be derived from PLM. Some of the negative considerations when evaluating PLM include:

- Asbestos content determination is usually done by visual estimate, and is thus semi-quantitative; concentrations are reported as percent by area.

- Small fiber identification is difficult because the characteristics microscopic optical properties are hard to determine in small fibers.

- The absolute optical resolution is approximately 0.3 micrometers in diameter; fibers this small, though observable, cannot usually be identified as to type. Many smaller fibers are typically present in the sample which therefore are not detected.

When dealing with manufactured asbestos-containing materials, the above considerations do not usually pose a significant problem. This is because relatively large proportions of asbestos have been employed in the manufacture of asbestos products. The presence of large bundles of asbestos fiber in large amounts (greater than one percent) is easily identifiable by standard PLM methods.

In applying PLM methods to asbestos identification in soils, however, problems have been encountered. Because of the fact that soils are subjected to erosion and weathering, asbestos bundles become separated and broken into smaller, possibly suboptical, sizes much more quickly than fiber bundles in relatively undisturbed insulating materials. Asbestos fibers may also be dispersed by wind and by seasonal flooding. Therefore, a sizable fraction of the asbestos fibers in soil could be below optical resolution. Most asbestos experts agree that PLM analysis is not adequate to identify soil samples with less than one percent asbestos, even though soils with low asbestos fractions may still pose a health risk if the fibers are liberated into the air.

Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) is a much more powerful analytical method than PLM. It is the preferred instrumental technique for measuring asbestos in ambient atmosphere since it incorporates the most powerful combinations of identification methods (Chatfield, 1983). TEM can be used for air, water, or soil analysis. Although it is not limited in terms of

mineral identification, this method does have its drawbacks; it is extremely time consuming and expensive. In addition, methods of sample preparation are not standard among asbestos researchers.

TEM analysis uses electron microscopy at a magnification of 10,000 to 50,000 times to detect asbestos to the single fibril level. Fibers as small as 0.2 nanometers in diameters can be identified (Chatfield, 1983). Besides the transmission electron microscope, which allows the operator to locate very small fibers, this technique also utilized two mineral identification tools. These are Selected Area Electron Diffraction (SAED) and the Energy Dispensive X-ray Analyzer (EDXA). Using these, the operator can identify the mineral type from a single point on the specimen.

The previous soil samples taken from the South Bay Asbestos site for TEM analysis were analyzed using a preparation method developed by Dr. Steven Hayward of the California Department of Health Services (DHS). Samples collected for this RI were also prepared using Hayward's method. This method involves grinding soil to a fine powder, then suspending the powder in water in order to filter it. The filter is then analyzed by counting, like an air filter. This method does not allow the in-situ characterization of fiber sizes, but it does give results in total percent by weight.

Samples for air were analyzed by TEM by several laboratories, using two very different preparation techniques. The first, or direct technique, involves coating the air filter with a carbon film to trap the fibers, then placing the filter on a grid of known size and collapsing the filter onto the grid. The number of fibers are then counted. The second preparation method, or indirect technique, involves ashing the filter, then suspending the residue in water and filtering it through a second filter. The second filter is then prepared as for direct analysis. Based on the two sets of results (discussed in Section 5.0), the indirect method appears to give asbestos results higher than the direct method, because much of the obscuring matter included in the sample is dissolved during the resuspension. Although the indirect method may yield more accurate results, considerable controversy exists whether the indirectly-prepared

sample data are appropriate to use for risk estimation in ambient situations, since they cannot be compared directly to fiber counts obtained during medical studies using Phase Contrast Microscopy (discussed below).

The high resolving power of TEM allows estimates of not only total number of fibers but also fiber mass in nanograms, since the length, width, and diameter of each fiber can be observed (and multiplied by a given density). Although many previous studies, including asbestos medical data, are reported in mass concentration, controversy exists as to the relevance of mass measurements to risk estimation. A few large and thick fibers or clusters can have a disproportionate effect on the mass. For this reason, mass analysis of the air samples were not used in this investigation.

Phase Contrast Microscopy (PCM)

Phase contrast microscopy (PCM) is a method of optical microscopy that is commonly used to analyze air samples collected in the workplace. The method is better suited to analysis of workplace air than ambient air because in the work place one encounters a relatively large concentration of large bundles of asbestos fibers (NRC, 1984). Most of the available medical studies of asbestos diseases have measured asbestos using PCM. (Appendix K, p. 3-8).

The PCM technique has two major limitations concerning its use in the environment (Chatfield, 1983). The method cannot detect fibers with diameters of less than 0.2 micrometers. Many fibers in the environment are much smaller than this. Also, PCM does not distinguish between asbestos fibers and other types of fibers. Therefore, in the environment, the PCM fiber count may be completely unrelated to the asbestos fiber content. For these reasons, "it is widely accepted that the PCM method is totally unsuitable for measurement of asbestos fibers in ambient atmospheres" (Chatfield, 1983).

A major concern is how to convert between the TEM asbestos data collected during the RI and the PCM occupational data base upon which estimates of health risk are based. There is no consensus among asbestos researchers

and health experts as to the best way to equate an electron microscope measurement to the lower-resolution optical measurement of PCM. This issue is discussed further in Section 7.0, Risk Assessment.

Scanning Electron Microscope (SEM)

A few of the samples collected by previous workers at the South Bay Asbestos site were analyzed using scanning electron microscopy (SEM). Although this method has a resolution of as much as 5 nanometers, it is not generally suitable for environmental samples and was not employed during the RI.

According to Chatfield (1983), there are three problems with the SEM technique that render it inappropriate for environmental samples. First, the high resolution can only be obtained when analyzing ideal specimens, otherwise electrical noise interferes. Second, thin fibers and flat platy particles display poor contrast, and are therefore difficult to identify. Finally, SEM can not discriminate reliably between chrysotile and the elongated particles of other non-asbestos minerals.

1.4 REPORT ORGANIZATION

This report describes the Remedial Investigation that was performed for the South Bay Asbestos site. The investigation included the review and interpretation of work performed by previous investigators at the site; previous work is presented in Section 2.0 of this report. Section 3.0 describes the field work performed in this investigation. The data gathered for the remedial investigation are presented in Sections 4.0 and 5.0. Section 4.0 describes the physical characteristics of the site and Section 5.0 discusses the nature and extent of contamination at the site. Section 6.0 provides an interpretation of the fate and transport of contaminants at the site. The information discussed in Sections 5.0 and 6.0 is then used to develop the Risk Assessment in Section 7.0. Finally, Section 8.0 provides a summary and the conclusions of the Remedial Investigation.

SECTION 2.0

PREVIOUS INVESTIGATIONS

2.0 PREVIOUS INVESTIGATIONS AND RESPONSE HISTORY

2.1 SUMMARY OF PREVIOUS INVESTIGATIONS

The discovery of waste asbestos in Alviso on August 8, 1983 initiated a series of soil and air sampling programs in Alviso. This section provides a summary of the numerous (and in some cases previously undocumented) site sampling investigations in Alviso since the discovery of asbestos up to the Remedial Investigation performed by EPA in 1987. A chronological list of sampling events is provided in Table 2.1. The data obtained during these events were used to develop preliminary estimates of site contamination, and to determine the scope of the RI/FS investigation. In general, the previous soil asbestos data were used in this study as general indicators of site asbestos contamination. Previous air data were not used, because they were not long-term sampling events suitable for assessing inhalation risks. Much of the previous data were used specifically to guide interim emergency cleanup actions, described herein. The RI/FS effort focused on obtaining data to verify existing data, or to fill data gaps so that a risk assessment could be conducted.

The methods (where available) and results of each of the previous soil and air investigations are discussed below. The discussions are arranged chronologically by agency. All results are presented in Appendices C through E, summarized in Tables 2.1 and 2.2, and shown graphically in Figures 2-1 through 2-9.

2.2 PREVIOUS SOIL INVESTIGATIONS

2.2.1 CALIFORNIA OCCUPATIONAL SAFETY AND HEALTH ADMINISTRATION (Cal-OSHA)

Methods

Waste asbestos was discovered in the Guadalupe River levee during construction of a flood outfall structure. On August 8, 1983 during a

routine inspection, a safety engineer and industrial hygienist from the California Occupational Safety and Health Administration (Cal-OSHA) noticed white material in the excavation spoils and collected two samples to confirm its composition. Two days later, six soil samples were collected from the top and sides of the river levee and from the truck turnaround area in the adjoining field. The soil samples were collected using a hand trowel. "Sampling was diverted toward collecting what visually appeared to be fibrous material in surface soil," (Aqua-Terra, 1986). Sample locations are included on Figure 2-1.

The samples were analyzed by polarized light microscopy (PLM) by the Air Industrial Health Laboratory (AIHL) of the California Department of Health Services (DHS).

Results

The two samples of suspected asbestos that were collected on August 8, 1983 were found to contain 30 $\pm$ 10 percent asbestos by weight. The six soil samples contained from < 1 to 7.5 $\pm$ 2.5 percent asbestos by weight. Sample results are tabulated in Appendix C and shown in Figure 2-1. The sampling results prompted DHS and the Bay Area Air Quality Management District (BAAQMD) to require that the area be barricaded and that the excavation spoils be removed to a hazardous waste disposal facility. Removal of the contaminated soil took place from August to December 1983.

2.2.2 CALIFORNIA DEPARTMENT OF HEALTH SERVICES (DHS)

Initial Soil Sampling

Methods

On August 10, 18, 30, and September 1, 1983, DHS collected soil samples from the river levee, from fields adjacent to the river levee, and from the boundary of the Summerset Trailer Park. These were biased samples: if the sampling location had white material present, that was sampled preferentially. All but two of the samples were collected with a hand trowel. Two

samples were collected from depths of 1 1/2 feet and 4 feet using a hand auger. No duplicate samples were collected. The soil samples were analyzed by PLM by AIHL. According to Steve Hayward of AIHL, the PLM method he used has a detection limit of 0.01 area percent, rather than 1 percent typically reported by commercial laboratories.

Results

The soil samples were found to contain from < 0.01 to 30 ± 10 percent asbestos by weight. Sample results are tabulated in Appendix C and shown in Figure 2-1.

Random Grid

Methods

On September 12, 1983, DHS collected 20 samples from a "random grid" and collected another five samples from various locations around the community. The locations for these samples and the other soil samples collected by DHS are included on Figure 2-1.

A grid with squares numbered from 1 through 72 was prepared on a blank sheet of paper which was then overlain on a map of Alviso (the exact scale of the grid used is not available). The numbered squares were then reordered using a random numbers table. The samplers were instructed to go to the first square on the list to collect a sample; if sampling was not possible there, the samplers were to go to the second square on the list. They were to continue until 20 squares had been sampled. The samplers were to collect a sample from the same position within each square. The exact location was chosen to sample undisturbed, natural-looking soil. The sampled locations are distributed throughout the community.

The five additional samples were collected to identify unusual materials observed during the grid sampling. These included a "lavender powder" observed near the fire station, a "solid foam material" found in the marsh, and a "white pile" seen near the intersection of Catherine Street and State Street.

Results

All of these samples were initially analyzed by AIHL using PLM. Results were from < .01% to 3 $\pm$ 2 percent asbestos by weight. The asbestos values for each sample are tabulated in Appendix C and are shown in Figure 2-1.

One of the samples, which had been estimated to contain 0.1 percent asbestos by PLM was then examined using a Scanning Electron Microscope (SEM). This was to determine if the soil material that was too small to be resolved with PLM contained asbestos. With SEM, much of the background "soil" material was found to be fine asbestos fibers. TEM was then used to quantify the mass percentage of asbestos. This analysis showed that 10 to 20 percent of the mass of this soil sample was asbestos even though its microscopic appearance was no different than that of common soil.

After this discovery, AIHL developed a draft procedure for determining asbestos content in soil by TEM (Hayward and Lowe, no date). An additional six of the 20 grid samples were analyzed using this method. Results ranged from 0.074 to 26 percent asbestos by weight.

Simulations

Method

In June and September 1985, DHS performed three experiments to measure asbestos exposure under certain soil-disturbing conditions.

DHS performed two experiments on the ring levee at the intersection of Spreckles and Michigan Streets (Figure 2-1). The first, called the "worst case experiment", was performed on June 18, 1985. In this experiment, the

levee soil was disturbed with a shovel and a rake. An electric fan was used to blow the dust that was generated toward an air sampler. To calibrate the results of the air analysis to the asbestos content of the soil, DHS collected, and sent to AIHL, a sample of the disturbed soil and a sample of the dust that settled on top of the fan.

On September 17, 1985, the second experiment was performed at the same location. This experiment was similar to the first except that air samples were collected from the breathing zone. The levee soil was disturbed using a shovel and a toy dump truck. For calibration purposes, a sample of the levee soil was collected from the experiment site. On the same day, DHS also collected a sample of soil from a dusty path that crossed the levee several blocks north of the experiment site. The samples were analyzed by AIHL using TEM.

On September 25, 1985, DHS performed a traffic simulation. Air was sampled along the unpaved north end of Spreckles Street while a truck drove back and forth (described in Section 2.3.2 and Figure 2-7). Two soil samples were also collected. One was from the middle of the road, one was from the northeast edge of the road, which is downwind of the road. The samples were analyzed by AIHL using PLM.

Results

PLM analysis showed that the two soil samples collected during the worst case experiment contained <1 percent asbestos. However, when analyzed using TEM, one of the soil samples was found to contain 24 percent asbestos and the other was found to contain 36 percent asbestos by weight.

The two soil samples collected during the field experiment performed with a shovel and a toy truck in September, 1985, were found to contain 12.5 percent and 13.5 percent asbestos by TEM analysis.

PLM analysis indicated that the soil sample collected in the middle of the Spreckles Avenue contained 10 percent asbestos. The soil sample collected from the downwind side of the road contained 63 to 75 percent asbestos.

Results of the air analyses are discussed in Section 2.3. After the first of these experiments, the "worst case exposure" experiment, DHS briefed the EPA Emergency Response Section on the results. DHS asked EPA to consider emergency response action to limit asbestos emissions from the ring levee. DHS and EPA then jointly collected and analyzed samples taken from locations within the community and from the ring levee as described below.

2.2.3 U.S. ENVIRONMENTAL PROTECTION AGENCY EMERGENCY RESPONSE SECTION (EPA)

Joint Sampling with DHS

Methods

On August 27, 1985, soils at 13 locations within the community and along the levee were sampled by EPA. At one of the locations, EPA collected a duplicate for quality assurance purposes, and at 11 of the locations, DHS collected a duplicate sample. The two samples that DHS did not duplicate were rock and pipe, rather than soil, samples. EPA's set of soil samples was analyzed by EAL Laboratories (EAL) with PLM. The duplicate samples collected by DHS were sent to AIHL for analysis. AIHL used PLM to analyze six of the samples and used a combination of PLM and SEM to analyze the other five. AIHL also analyzed one of the samples using SEM and TEM. Sample locations are included on Figure 2-1.

Results

The analytical results (Appendix C) for the duplicate samples reveal slight variation between samples, between laboratories, and between analytical methods. The results are shown graphically in Figure 2-1.

Street and School Yard Sampling

Methods

In October 1985, the EPA Emergency Response Section sampled three unpaved areas in the community. On October 4, EPA collected 34 surface soil samples from the unpaved north end of Spreckles Avenue and 13 surface soil samples from the George Mayne Elementary School yard. On October 23, EPA collected 67 surface soil samples from streets within the O'Neill Tract. All of these samples were collected at predetermined intervals to avoid sampling bias. The sample locations are included on Figure 2-1. No duplicates were collected. The samples were analyzed using PLM.

Results

The samples collected from Spreckles Avenue and from the school yard contained from <1 to 3-5 percent asbestos. The O'Neill Tract samples contained less than 1 percent of asbestos, with the exception of one sample with 1 to 2 percent asbestos. The results for these sampling events are tabulated in Appendix C and shown in Figure 2-1. Based on these results, EPA paved the school yard and Spreckles Avenue on October 26 and 27, 1985. No action was taken at the O'Neill tract since asbestos results were so low.

Grid Sampling

Methods

From December 16 to 31, 1985, EPA sampled extensively throughout the community, along the ring levee, and at the Environmental Education Center. Within the community, 235 surface soil samples were collected at 250-foot intervals. Eleven of these were duplicate samples. The sample locations for the grid sampling included residential property, street dust, the levee of the Guadalupe River, the George Mayne Elementary School grounds, and the Summerset Trailer Park. One hundred-ten surface soil samples were collected along the ring levee at 100-foot intervals. Three of these were

duplicated. Twenty-four surface soil samples were collected from recent fill at the Environmental Education Center. One of these samples was a duplicate. The sample locations are shown on Figure 2-2. All of the samples collected during December 1985 were analyzed using PLM.

Results - Community and levee grid samples:

Results of the analyses are tabulated in Appendix C, and shown graphically in Figure 2-2. Asbestos was detected in approximately 65 percent of the grid samples, and more than 45 percent of these samples contained 1 percent or more asbestos. About 20 percent of the grid samples contained 5 percent or more asbestos, and 7 percent of the total contained at least 20 percent asbestos.

Within the community, asbestos levels were variable and did not reveal a pattern. In many cases, samples with no asbestos detected were near samples with over 5 percent asbestos. A "hot spot" identified is the two-block area bounded by Taylor Street, Hope Street, Elizabeth Street and El Dorado Street. Of the ten samples collected there, six contained 10 to 20 percent asbestos.

The grid sampling revealed several "hot spots" on the ring levee. Samples from the ring levee along Spreckles Avenue between Grand Boulevard and Wabash Street contained from 5 to 20 percent asbestos. Also, ring levee samples from El Dorado Street to Catherine Street contained from 5 to 40 percent asbestos. Samples from the part of the ring levee that parallels State Street also contained from 3 to 40 percent asbestos. Because of these results, EPA sprayed the ring levee with a polymer dust suppressant in May 1986.

Results - Environmental Education Center samples:

Of the 24 soil samples collected at the Environmental Education Center, asbestos was detected in 15. Six samples contained from 1 to 3 percent asbestos, three samples contained from 5 to 10 percent asbestos, and the remaining six contained more than 10 percent asbestos. The results are

tabulated in Appendix C and shown in Figure 2-2.. Based on these results EPA removed debris piles, paved some berms and walkways, and revegetated some areas at the Environmental Educational Center.

2.2.4 CITY OF SAN JOSE

Methods

During May, 1986, Woodward-Clyde Consultants (WCC), as the contractor for the City of San Jose, collected soil samples as part of their analysis of the ring levee. The ring levee investigation also included air sampling, which is discussed in Section 2.3. As a part of the air investigation, performed May 7, WCC collected one surface soil sample from the WSP truckyard, which was the location of one of the air samplers.

On May 15 and 16, WCC collected samples from 47 locations along the ring levee that were designated by the City of San Jose. The ring levee sample location are shown on Figure 2-3. From each location, with one exception, two samples were collected using a jeep-mounted auger. One was taken from a depth of 4 to 6 inches, the other from 12 to 18 inches. The soil samples were shipped to EMS Laboratories for PLM analysis.

Results

The topsoil collected from the WSP truckyard contained 2 percent chrysotile asbestos by PLM.

WCC found that approximately two-thirds of the samples collected from the ring levee contained at least 1 percent asbestos as shown in Figure 2-3. The areas of the levee that contained high concentrations of asbestos coincided approximately with sample results reported by EPA. These areas are as follows:

(1) Parallel to Spreckles Avenue between Grand Boulevard and State Street. Surface soil samples here contained up to 15 percent asbestos and the deeper samples contained up to 22 percent asbestos.

(2) Parallel to State Street north of the truckyards between Archer Street and Pacific Street. Surface soil samples contained up to 13 percent asbestos and the deeper samples contained up to 15 percent asbestos in this area. These samples contained lower asbestos concentrations than were found by EPA in this part of the levee (see page 2-11; compare Figures 2-2 and 2-3).

In addition, WCC found two other localized areas with high asbestos concentrations. One of these was the ring levee parallel to Grand Boulevard. Here, PLM analysis showed that surface samples contained 5 to 15 percent asbestos, and subsurface samples contained 4 to 15 percent asbestos. WCC also found high asbestos concentration along El Dorado Street, where two surface samples contained 8 and 10 percent asbestos, and the deeper samples contained 3 and 4 percent asbestos.

All of the WCC ring levee sample results are tabulated in Appendix C and shown in Figure 2-3.

2.2.5 ENVIRONMENTAL PROTECTION AGENCY FIELD INVESTIGATION TEAM (FIT)

Methods

In 1986, EPA directed its Field Investigation Team (FIT) contractor, Ecology and Environment Inc., to sample some unpaved truckyards in Alviso. The purpose of this investigation was to determine whether dust generated at these facilities contributed to asbestos exposure in the community and, if so, whether paving was necessary. EPA believed that the truckyards might be contaminated in two ways: redistribution of landfill material and direct dumping of industrial asbestos waste. Because the truckyards are unpaved and in some cases unvegetated, EPA was concerned that they may be significant sources of asbestos releases. In addition to the possibility that fill was transported from the Alviso landfills to the truckyards, asbestos waste may have been deposited directly at the location of current truckyards, near the old race track (Aqua-Terra, 1986). The racetrack, which was operational during the mid-1950s, was located north of State Street between Essex Street and Spreckles Avenue.

On July 23 and August 7, 1986, fifty-two surface soil samples were collected, including five duplicates, from ten truckyards. The sample locations within the truckyards were chosen using a predesignated grid pattern. Locations of the truckyards and of the soil samples are shown on Figure 2-4. In addition to the grid samples, one sample was collected of material that appeared to be asbestos.

The fifty-two samples were sent to Versar, Inc., for analysis by PLM. Later, Clayton Environmental Consultants Inc. reanalyzed five of the samples using PLM. In 1987, 21 of the samples were reanalyzed by EAL using TEM; EAL also analyzed 19 of the 21 samples using PLM.

Results

The truckyard samples contained very low concentrations of asbestos as shown in Figure 2-4 and Table 2.2. Versar found no asbestos in 19 of the samples, and only trace amounts in the other 33. Three of the Clayton analyses agreed with those of Versar; however, Clayton found that two samples contained more asbestos than Versar found. These samples contained 2 and 19 percent asbestos. The EAL analyses also revealed very low asbestos content. All 21 TEM analyses found less than 0.1 percent asbestos. The PLM results ranged from not detected to 2 percent.

2.3 AIR INVESTIGATIONS

2.3.1 BAY AREA AIR QUALITY MANAGEMENT DISTRICT (BAAQMD)

On August 22 and 23, 1983, BAAQMD collected air samples at the excavation site where asbestos was discovered (Aqua-Terra, 1986). Number and type of air samplers, sample times, and analytical methods and results are not known. Mr. Wayland Siu of the BAAQMD was contacted for information on the sampling effort. Although his records included information about later sampling efforts in Alviso, he had no record of BAAQMD's sampling during August 1983.

2.3.2 CALIFORNIA DEPARTMENT OF HEALTH SERVICES (DHS)

Ambient Air Sampling

Methods

During the period from October 7, 1983 through May 3, 1984, DHS collected 40 ambient air samples from nine stations. Samples were collected on 0.2 μm pore size nucleopore polycarbonate membrane filters using Sierra Instruments Monocot Samplers. Flow rates for ambient air samples averaged 15 liters per minute. For each sampling event, wind direction and velocity were noted. All but five of the samples were analyzed by AIHL using TEM. Twenty-five of the samples were split and analyzed by both AIHL and Med-Tox Associates. Five samples were analyzed by Med-Tox only.

The initial air samples, collected on discrete days by DHS from October 7, 1983 through May 3, 1984, were collected for two purposes: (1) to determine whether ambient asbestos levels upwind of Alviso were different than those measured in Alviso and (2) to determine whether indoor asbestos exposure differed from outdoor asbestos exposure. The sampling locations are shown on Figures 2-5 and 2-6. DHS used the William Smith yard, at the north end of Spreckles Avenue, as an upwind site and compared values there to values at the Fire Station and at the Summerset Trailer Park. DHS compared indoor and outdoor asbestos values at three sites: George Mayne Elementary School and the Martinez and Gordon residences.

To assist DHS in gathering further information on ambient asbestos levels in Alviso, BAAQMD conducted a sampling program from August 16, 1984 through November 1, 1984. Samples were collected from five sites in Alviso and from two reference sites outside of the community. Within the community, they sampled air at the Fire Station, George Mayne Elementary School, Sutter's Card Lounge, and Summerset Trailer Park. The upwind reference site is located at Moffett Field on Moffett Park Drive just west of Highway 101. The downwind reference site is the BAAQMD air monitoring station at Fourth Street and East San Carlos Street in San Jose.

Results

DHS did not prepare a formal report results of their air study; however, Aqua-Terra Technologies (ATT) presented these data in their 1986 Interim Report (Aqua-Terra, 1986). This section of the ATT report is included as Appendix D.

The general conclusion presented by ATT is that asbestos levels in the air at Alviso may be three to ten times higher than ambient levels at the Moffett Field site, and that the relative difference between Alviso and San Jose airborne asbestos concentrations is not clear. ATT also reported that the DHS study did not collect enough meteorologic data to be able to draw conclusions about which sources of asbestos were contributing to airborne asbestos levels. The conclusions presented in ATT's report should be read with caution. DHS's sampling program did not include enough samples collected at identical locations over time to draw statistically based conclusions concerning the magnitude of asbestos contractions within and outside of Alviso.

The data tables included in the ATT report in Appendix D show a large discrepancy between analyses performed by AIHL and those performed by Med-Tox. The fiber counts reported by Med-Tox are typically much lower than those reported by AIHL. The reason for this discrepancy has not been determined. The analytical method used by AIHL, which was developed by Dr. Steve Hayward, is included in Appendix B, and can be compared to the EPA draft method (Yamate et al., 1984), also in Appendix B, used by Med-Tox. Hayward's method is based on the Yamate method and uses similar fiber counting and sizing procedures. The fiber count differences may possibly be attributed to differences in operator procedures.

Exposure Simulations

Methods

DHS performed three experiments in 1985 that were designed to measure potential asbestos exposure to site residents under certain conditions.

On June 18, 1985, DHS performed the worst case experiment on the levee at the intersection of Spreckles Avenue and Michigan Street. This simulation was designed to measure asbestos concentrations in the dust cloud generated by disturbing the soil. In this experiment, a shovel and a rake were used to spill dirt in front of a fan while the fan created a wind of approximately 15 mph. An air sampler was positioned 10 feet downwind from the fan at a height of 3 feet. Two air samples were collected and analyzed by AIHL using TEM.

On September 17, 1985, DHS conducted a similar experiment at the same site. This experiment was designed to assess a person's exposure to airborne asbestos while gardening or playing. In the first part of the experiment, a shovel was used to turn the loose earth over while a sampler at a height of four feet collected samples from the breathing zone. In the second part of the experiment, the experimenter played with a toy dump truck, alternatively scooping loose dirt into it by hand and then dumping it out. The sampler was placed at a height of 1 foot to sample a child's breathing zone. The air samples were analyzed by AIHL using TEM. This experiment was also conducted at the Berkeley waterfront to obtain a control sample.

On September 25, 1985, DHS performed a traffic simulation to determine the airborne asbestos concentration in dust generated by vehicle traffic on the unpaved part of Spreckles Avenue. Five MSA personal monitoring pumps were used to collect air samples. One of these was attached to the driver's side rear-view mirror of a pickup truck. The other four were set up at a height of 4 feet, two on each side of the street, as shown on Figure 2-7. Filter cassettes were attached to the pumps to collect aerosols, which were later analyzed for asbestos fibers using PCLM (NIOSH Method 239). Pump rates were set at 2 liters per minute. After the pumps were turned on, the pickup truck was driven past the stationary pumps at 20 mph; after passing the end pumps, the truck turned around to drive by the pumps again. About sixty passes were completed along approximately 200 feet of Spreckles Avenue. During the experiment, wind speed was from 0 to 5 mph with gusts to 20 mph. Wind direction was generally from the west but varied from the west to the north. The road condition was dry.

Results

The air quality samples collected during the worst case experiment were found to contain 170 and 190 total fibers/cm^3 and 1.1 and 0.9 NIOSH-equivalent fibers/cm^3, by TEM. "NIOSH equivalent" fibers were defined by AIHL as those fibers with diameter greater than 0.25 microns, length greater than or equal to 5 microns, with an aspect ratio (length to width) of 3 or greater; it was assumed that fibers of such dimensions counted with a transmission electron microscope set at low power magnification (400 to 4000x) are equivalent to fibers detected by the PCM method. The sample collected during the shoveling simulation contained 0.35 fibers/cm^3, and the sample collected during the toy truck simulation contained 1.6 fibers/cm^3. (The shoveling and toy truck simulations did not have NIOSH-equivalent fiber counts reported).

On the Berkeley waterfront, the shoveling simulation produced 0.07 total fibers/cm^3 and the toy truck simulation produced 0.39 total fibers/cm^3. TEM examination of the Alviso and Berkeley samples showed that all fibers from Alviso were asbestos, whereas nearby all fibers from Berkeley were cellulose.

Figure 2-7 shows the results of the traffic simulation. Analyses of the downwind samples and the sample from the truck-mounted sampler revealed higher airborne asbestos concentrations than did those of the upwind samples.

The worst-case and toy truck experiment data were used in the ring levee Operable Unit Feasibility Study (OUFS) (EPA 1988a) to show the potential for asbestos exposure from soils at the levee. They were not used in the RI risk assessment because of lack of quality control information and possible inappropriate exposure assumptions. The traffic simulation data, however, were used in the risk assessment (Section 7.0) to estimate exposures to asbestos near vehicle traffic.

2.3.3 CITY OF SAN JOSE

During May 1986, Woodward-Clyde Consultants (WCC), as the contractor for the City of San Jose, conducted an air sampling program to learn whether the ring levee was a source of airborne asbestos (WCC, 1987). These data were used in the ring levee Operable Unit Feasibility Study (OUFS)(EPA, 1988a). The results are included in that document.

2.4 MARSHLAND LANDFILL INVESTIGATIONS

The South Bay Asbestos site boundaries include the Marshland landfill, a large inactive Class II landfill currently undergoing closure in accordance with State and Federal landfill regulations. Since it is part of the site, available data regarding the landfill were included for completeness.

Methods

In March and April 1985, Woodward-Clyde Consultants (WCC) conducted a study of the southwestern corner of the Marshland Landfill for the California Department of Transportation (Caltrans). The purpose of the study was to identify the presence and nature of hazardous wastes there because Caltrans was considering excavating part of the landfill in order to build a highway interchange.

WCC drilled three borings, from which they sampled soil, leachate, and soil gas. The boring locations are shown on Figure 2-8. Each boring was drilled to a 55-feet depth using a hollow-stem auger. Soil samples were collected at 5-foot intervals using a split-spoon sampler with brass tubes. Twenty-eight samples of soil and landfill debris were collected. After drilling each boring, a Teflon bailer was lowered into the hollow-stem auger to collect a leachate sample. Soil-gas samples were collected from each boring with the use of a down-hole sampling probe equipped with a low-volume vacuum pump. The pump drew gas across a charcoal tube that was later analyzed for organic vapors. To collect samples for hydrogen sulfide and sulfur dioxide gas analysis, the pump drew gas through liquid-filled glass impingers designed for these specific gases.

Soil samples were analyzed for asbestos using PLM by EAL Laboratories. SML Laboratories analyzed soil, leachate, and gases for Priority Pollutant metals. Stoner Laboratories analyzed soil and leachate samples for volatile organic compounds, pesticides, and PCBs. Stoner also analyzed the organic gas samples. In addition, Stoner analyzed field blanks for volatile organic compounds and quality control blanks for organic gases.

Results

In soils, copper and lead exceeded the State Total Threshold Limit Concentration (TTLC) in one boring. In two borings, soil samples contained TCE (trichloroethene) exceeding the TTLC. Asbestos was not detected in soils. Leachate collected from one boring exceeded the Soluble Threshold Limit Concentration (STLC) for lead. WCC's report states that no other leachate compounds exceeded standards. WCC compared the sample results to California regulatory standards. All of WCC's sample data are presented in Appendix E.

2.5 O'NEILL TRACT INVESTIGATION

As part of the overall site investigation of potential sources of both asbestos and hazardous wastes, EPA's Technical Assistance Team (TAT) contractor, Ecology and Environment (E&E), performed a preliminary inventory of drummed waste and asbestos waste stored at the O'Neill junkyard in the spring of 1988. This junkyard is located on the southeast side of Grand Boulevard between Essex Avenue and Pacific Avenue.

E&E visited the site on March 30 and April 18. During the first visit, the team counted 70 drums at the site. Most of these were empty. Twenty-two of the drums were numbered, inventoried, sampled, and classified. Several other drums were not sampled because they were not accessible.

On April 18, E&E returned to the site to sample and classify the previously inaccessible drums. At that time, the team also performed Seta flashpoint tests on the drums that previously tested positive for flammability. Also

during this visit, the team inventoried three piles of asbestos/cement pipe that were found on site.

Of the 37 drums that were sampled, four tested positive for flammability and three were characterized as potentially corrosive due to their high pH. A composite sample was collected from the three potentially corrosive drums; the sample was laboratory analyzed for total cyanide, total sulfide, and pH. Results showed that the liquid in these drums was below the RCRA definition of toxicity.

E&E found piles of asbestos pipe at three locations in the junkyard. These piles consisted of approximately twenty-two 3-foot long, 6-inch diameter asbestos pipe and eight pipes of 3.5-foot length and 14-inch diameter. No action was taken regarding the asbestos pipe.

As a result of the TAT investigation, a contractor for DHS removed the four drums of flammable liquid on May 11, 1988.

2.6 ALVISO OIL COMPANY

The Alviso Oil Company operates four tanks north of the intersection of State Street and Archer Street as part of its waste-oil recycling business. The four tanks at the facility contain the following:

#1	7,500 gallons oil and water, 50% each
#2	10,000 gallons oil
#3	9,000 gallons oil and water, 50% each
#4	20,000 gallons oil

The tanks are surrounded by a concrete/earthen berm approximately 18-inches high.

On September 13, 1985, Roy F. Weston Inc., as EPA's Technical Assistance Team (TAT) contractor, conducted a Spill Prevention Control and Countermeasure (SPCC) inspection of the facility. As a result of this inspection, EPA informed Alviso Oil Company that it was in violation of SPCC Regulations for two reasons: (1) the facility SPCC Plan was not made

available to the inspector because it had been lost in the 1983 flood and (2) the concrete/earthen berm around the tanks was worn down to 10 inches. The inspection team also noted evidence of oil spillage on the soil.

EPA's reason for concern about the berms was that oil spilled within the berm might flow into a drainage ditch that is approximately 100 feet north. After entering the drainage ditch, the oil could travel into the adjacent wetlands. The damaged berm was considered a violation of 40 CFR Part 112, U.S. EPA regulations pertaining to the prevention of oil spills from reaching waters of the United States.

The facility was reinspected by the TAT contractor during Fall 1986 and was found to be in compliance.

2.7 SUMMARY

A large body of existing data was available at the start of the RI/FS regarding asbestos and non-asbestos site contamination. These data were assembled and examined to determine how much was known about the site contamination levels and what additional data were required in the RI. In summary, the majority of the previous asbestos soil sample results were considered usable in the RI and were included in the risk assessment (Section 7.0). The soil data sets specifically used in the risk assessment are those collected by DHS (Section 2.2.2); EPA (Section 2.23); City of San Jose (Section 2.2.4) and the Field Investigation Team (Section 2.2.5). The Cal-OSHA soil samples were not used, since the soils at the discovery site have been removed. The previous air samples were not used in the risk assessment, with the exception of the DHS truck traffic simulation, since they were not collected over a long enough time period to allow long-term exposure estimates to be made. The DHS traffic simulation equipment, however, was used to estimate asbestos exposures to vehicular trraffic on unpaved areas. The following section describes the additional data collected during the RI investigation.

SECTION 3.0

RI/FS SITE INVESTIGATION

3.0 RI/FS SITE INVESTIGATION

EPA conducted the RI/FS site investigation at the South Bay Asbestos Site from May to October 1987, and May 1988. The investigation consisted of sampling air, surface soil, subsurface soil, groundwater, surface water, and river sediment. The field sampling programs have been described in detail in two field data reports: Field Report for the Soils and Water Sampling and Analysis Tasks (Doc. No. 288-RI1-RT-FLJY) and Field Report for Air Sampling and Analysis Tasks (Doc. No. 288-RI1-RT-FQND). The following discussion is a summary of those documents.

3.1 PHASE I SAMPLE COLLECTION

3.1.1 AIR SAMPLING

Previous investigations, as described in Section 2.0, yielded a great deal of data concerning asbestos concentrations in ambient air. However, the previous data base did not include an adequate number of samples collected at the same stations over time to draw statistically meaningful conclusions about long-term risk. EPA's air sampling program was designed to generate data that could be used to reach conclusions about long-term asbestos exposure in Alviso.

EPA collected air quality samples from a network of five monitoring stations during the period July 24 to October 31, 1987. Figure 3-1 shows the locations of the stations, which are different than previous air sample locations discussed in Section 2.0. The locations of stations numbered 2,3, and 4 are within the community of Alviso; they were chosen to provide information about the population's exposure and about the contributions of nearby sources. Stations numbered 1 and 5 are upwind and downwind reference stations. Site 1, at the Environmental Education Center, is generally upwind during prevailing wind flows, which are northwesterly. Site 5, which is approximately two miles southeast of Asbestos, is downwind of Alviso during prevailing wind conditions. During reverse wind conditions (southeasterly) Site 5 is upwind, and Site 1 is downwind.

Each of the five air sampling stations consisted of two flow-controlled and strip-chart monitored asbestos samplers, a high-volume sampler, (hi-vol), and an inhalable-particulate sampler (PM-10). The hi-vols and PM-10s were equipped with flow recorders. The equipment at each station was controlled by an automatic timer to ensure that each sampler ran for the same time interval. Asbestos samples were collected on 25mm mixed cellulose ester filters in preloaded conductive cowling filter cartridges. Particulate samples were collected on pre-weighed glass-fiber filters. At Site 4, an additional hi-vol and PM-10 were installed for the collection of quality-control duplicate samples. A 10-meter meteorological station was installed at Site 1 to provide information about weather conditions during sampling events. All instruments were calibrated prior to use, and quality assurance audits were conducted during the sampling period to ensure proper calibration.

The sampling events were of two types. Once every three days, 24-hour samples were collected. The purpose of these was to provide information that could be used to analyze the health effects of asbestos exposure. Thirty 24-hour sampling events were conducted. In addition to 24-hour sampling events, 18 episodic sampling events were conducted in order to document the sources of airborne asbestos. These sampling intervals, which were of 4 to 12 hour duration, were chosen to correspond to specific wind conditions. A total of 300 24-hour asbestos samples and 160 episodic asbestos samples were collected on 48 sampling days; approximately 586 particulate samples were collected on 50 sampling days.

The timer at each station and the flow recorders attached to each sampler provided information that allowed the calculation of total flow volume of air that passed through the filters. At all stations except Site 4, the two asbestos samplers were set at different flow rates. For 24-hour samples, rates of 2 liters per minute (lpm) and 3 lpm were used. Flow rates for episodic samples varied in order to draw an ample volume of air through the filter during short sampling intervals. At Site 4, the two asbestos samplers were set at identical flow rates in order to collect duplicate samples. The purpose of using different flowrates was to avoid

the problem of sample overloading, where excess particulate matter masks the asbestos fibers making direct analysis impossible. Typically the lower flow rate sample was submitted to the laboratory.

All of the particulate samples were submitted to the laboratory for analysis; however, due to analytical constraints, only a portion of the asbestos samples were analyzed. Selection of asbestos filters for analysis from 24-hour sampling days was based on the following criteria: First, only sampling events with data available from all samplers and from the meteorologic station were considered. Next, a representative number of samples was chosen from each of four predominant wind patterns. Asbestos filters were chosen from both weekday and weekend sampling events. Finally, filters were chosen that corresponded to a variety of known particulate concentrations. For the episodic sampling events, all asbestos filters were submitted for analysis. Appendix F includes a list of all samples submitted for analysis. A total of 127 asbestos samples and duplicates and 25 blank samples were analyzed. An additional 33 asbestos samples were submitted which could not be analyzed due to particulate overloading.

All of the asbestos filters not submitted for analysis are archived in the Region 9 REM II equipment repository. The archived samples are listed in Appendix F.

3.1.2 SURFACE SOIL SAMPLING

CDM collected surface soil samples from 40 locations throughout Alviso from June 1 to June 23, 1987. Sample locations are shown on Figure 3-2. The purpose of this sampling effort was to verify previous work and to collect data on the quantity of asbestos in the soil that is available to air transport. On September 30 and October 1, 1987, the same locations were sampled again and analyzed for moisture content and grain size in order to characterize soil erodibility. The latter sampling was correlated with meteorologic data collected by the weather station at air sampling Site 1.

To collect the surface soil samples, a 1-foot diameter steel ring was pressed into the soil. All of the soil to a depth of 1 inch was scooped out of the steel template and composited before it was placed in sample jars. At 18 of the locations, co-located samples were collected by placing the steel template directly adjacent to the first sampling spot. At four locations, split-sample duplicates were collected. In addition to all of these samples, which were submitted to the laboratory for analysis, a split-sample duplicate was taken at each location and archived for possible future analysis. The purpose of collecting and analyzing co-located samples and split-sample duplicates was to collect data regarding the source of variability in soil asbestos results. Sample collection information for surface soil samples is provided in Appendix G. A total of 62 samples including duplicates were collected.

3.1.3 SUBSURFACE SOIL SAMPLING

CDM collected subsurface soil samples from fifteen locations in Alviso and from one background location. The locations in Alviso consisted of five groundwater-monitoring well borings installed by CDM, five groundwater-monitoring wells installed by Emcon Associates along the perimeter of the Marshland Landfill, and five soil borings drilled by CDM. These locations are shown on Figure 3-3.

The subsurface soil samples collected from the groundwater-monitoring well borings were analyzed for asbestos and one sample from each well was analyzed for compounds on EPA's Target Compound List (TCL). The five wells installed by CDM were located in areas that have been used as landfills and that may have received asbestos wastes. Samples from the five wells drilled by Emcon for their client, Leslie Salt, who is the owner of Marshland Landfill, were intended to provide information about hazardous compounds and asbestos in the landfill. Depths of the wells ranged from 14 to 36.5 feet.

The five soil borings were located in areas of fill within Alviso. Subsurface soil samples were collected to provide data on levels of asbestos contamination and on the depth of asbestos-contaminated fill. The samples were analyzed for asbestos content only. The borings were each approximately 10 feet deep.

All of the wells and soil borings in Alviso were drilled with a truck-mounted hollow-stem auger rig. Soil samples were collected at 5-foot intervals from the surface to the water table with a split-spoon sampler. For quality assurance purposes, duplicate samples were collected for approximately 10 percent of the samples.

The background sample location is approximately 2 miles southeast of Alviso at air quality Site 5. A subsurface soil sample was collected from a depth of 2 feet with a hand-driven auger. It was analyzed for asbestos and for compounds on the TCL.

Appendix G includes a table of soil descriptions for the wells and borings. A table listing sample collection information is also provided. A total of 51 samples were collected for asbestos analysis by PLM, 20 samples for asbestos analysis by TEM, and 13 samples for TCL analysis.

3.1.4 GROUNDWATER SAMPLING

After the groundwater-monitoring wells were installed and developed, CDM collected groundwater samples. The wells installed by CDM were sampled twice: June 24 to 26, 1987 and October 7 to 8, 1987. The five EMCON wells at Marshland Landfill were sampled only once, from November 16 to 18, 1987. The sampling procedure involved purging the well of three casing volumes before collecting a sample. Several wells produced very little water, and so were bailed to dryness then allowed to recover overnight. Instruments used to purge the wells were a Fultz pump, a bladder pump, and a Teflon bailer. Most samples were collected with a Teflon bailer, but three samples were collected with a bladder pump. The temperature, conductivity, pH, and salinity of each sample was measured in a separate container. This information is tabulated in Appendix G. For quality control purposes, a

rinsate blank was prepared for each day of sampling the CDM-installed wells. A field blank was submitted for each day of sampling the Emcon-installed wells. Three sets of duplicate samples were submitted for analysis. A total of 26 groundwater samples were collected, including duplicates and blanks.

Sample collection information is tabulated in Appendix G.

3.1.5 RIVER SEDIMENT AND SURFACE WATER SAMPLING

Samples of Guadalupe River sediments were collected to determine if asbestos or contaminants on the EPA's Target Compound List (TCL) have been transported from the site and deposited downstream, posing a potential threat to San Francisco Bay flora and fauna. Also, surface water as a potential human exposure pathway had not been investigated previously at the site. The Guadalupe River was channelized in 1963 by excavating the Santos Landfill. The course of the river and the sediment sampling locations are shown on Figure 3-3.

Sediment sampling was performed on May 21, 1987. Samples were collected from three locations downstream of Alviso and from one upstream location, as shown on Figure 3-3. The downstream samples are composite samples; each consists of sediment from three locations collected over a cross section of the river channel. To collect the downstream samples, field personnel lowered a ponar dredge over the side of a motor boat to the sediment/water interface then snapped the dredge shut to trap river-bottom sediment within the dredge. The dredge was then raised to the boat, where it was emptied into a large stainless steel mixing bowl. This procedure was followed at two additional locations across the river to obtain a composite sample over a cross section of the river channel. The material was composited with a stainless steel mixing spoon before filling the sample containers with the spoon. One member of the field crew paced off the distances between the sediment sample locations along the bank of the river and indicated the locations to the field personnel in the motor boat on the river. The person onshore then marked the locations on a map and in the field log book.

The upstream sediment sample was collected by lowering the ponar dredge twice over the center point of the north edge of the Highway 237 Bridge into sediment of the Guadalupe River. The material from the two dredges was then mixed together in a stainless steel mixing bowl with a stainless steel spoon before filling the sample containers.

Two samples of the water of the Guadalupe River were collected to determine whether contaminants are entering the river where it passes through the Santos Landfill and are being transported into the Bay. These samples were analyzed for asbestos and for compounds on the EPA's TCL. The sample locations, one upstream of the landfill and one downstream, are shown on Figure 3-3.

Surface water samples were collected by lowering a Teflon bailer on a clean rope over two bridges that cross the Guadalupe River. The sample locations are the midpoint of the north side of the Highway 237 Bridge and the midpoint of the east side of the Gold Street Bridge. Temperature, electrical conductivity, salinity, and pH were measured for each surface water sample in a separate container. These data are presented in Appendix G. Sample collection information is also provided in Appendix G.

3.2 PHASE II SAMPLE COLLECTION

Phase II sample collection and analysis was undertaken to better define the air exposure pathway, by directly measuring air during soil disturbance. In addition, a portion of the archived Phase I duplicate air samples were analyzed, for the reasons described below. Additional soil samples were also collected to better define a soil background.

3.2.1 FURTHER AIR SAMPLE ANALYSIS

Examination of the Phase I direct data revealed significant problems. Because of the high proportion of particulate matter in Alviso air, the sampling flow rates had been reduced to minimal values to prevent "overloading" of particulates on the air filter. The asbestos laboratory

procedure of counting a small area of the filter detected very few fibers because of the low air sample volumes. The number of fibers detected was so small due to the insufficient air volume that statistically the sample results could not be distinguished from blanks in 10% of the samples (see Data Quality discussion Section 5.3.1). Because of this limitation, nearly all results were considered as estimates only. A number of asbestos researchers, including Dr. William Nicholson of Mt. Sinai Hospital, Dr. Steven Hayward or DHS, Phil Cook of EPA, and Steve Bayard of EPA headquarters, examined the Phase I data during a Region IX Asbestos Workshop discussion and considered it unreliable for making site remedial decisions. For these reasons, it was determined to submit a subset of the duplicate air filters to a second laboratory for analysis to improve the quality of the data by counting a larger area of the filter.

Because the air samples had been collected in pairs, with one of the pair submitted for Phase I analysis, a nearly complete archive of sample duplicates existed from which to select a subset for Phase II analysis. Twenty-five of the sample duplicates were selected based on anticipated concentration, known meteorological conditions during sampling, and station location so that a similar number of samples from each station would be available with a range of concentrations. To avoid the analytical and data quality problems described above, EPA determined that an alternate analytical technique was required for the 25 sample duplicates. First, many of the samples from Phase I were reported overloaded by the laboratory, that is, particulates obscured the asbestos fibers so that fiber counting was impossible. At the Region IX Asbestos Workshop discussions in January, 1988, the general consensus of participants was that the indirect preparation method of analysis proposed by Yamate (Appendix B) was acceptable. In this method, particulate loading of the filter does not prevent analysis, since the field filter is ashed, and the residue suspended in water, then refiltered. EPA Region IX developed a preliminary indirect method for analysis of the 25 duplicates. Although it was recognized that indirect data from the duplicate analysis would be difficult to compare to the Phase I direct data, the practical considerations of the sample quality made the indirect method the only analytical option.

Samples were analyzed by EMS Laboratories in Pasadena, California.

3.2.2 EXPOSURE ASSESSMENT EXPERIMENTS

Additional field sampling was undertaken at the site to define the potential asbestos exposure to individuals during soil disturbance. Two experiments were located in Alviso, at locations where Phase I surface soil samples had detected asbestos. However, analytical results from these experiments were declared rejected by data validation staff. These results were not used in the RI.

3.2.3 ADDITIONAL BACKGROUND SOIL SAMPLES

On Friday, May 13, additional background soil samples were collected at the downwind air station (Site 5) near Agnews Hospital East. These were collected to confirm the presence of asbestos at this location. An additional sample was collected from the marsh area south of the Environmental Education Center to determine an off-site background soil asbestos level unaffected by filling.

3.3 ANALYTICAL METHODS

Air-Asbestos

Asbestos work place environments contain relatively high levels of large asbestos fibers, which can be detected by PCM, or Phase Contrast Microscopy. PCM is, therefore the analytical technique most commonly used in asbestos health studies. In this study, asbestos is present at much lower concentrations than in an asbestos factory or mill, so PCM was not appropriate. Transmission Electron Microscopy (TEM) can detect asbestos at much concentrations than PCM, therefore, TEM was used, with the intention that a factor would be applied to convert TEM data to equivalent PCM health risk data to determine human exposure.

The Phase I air samples were analyzed using direct preparation TEM, NIOSH method 7402 (issued 5/15/86) (Appendix B). Ten of the Phase I samples were also analyzed using PCM to assist in developing the appropriate conversion from TEM to PCM health risk data. The PCM method used was NIOSH method 7400 (Revision 1, 5/15/85) (Appendix B). Gravimetric analysis method used for the particulate samples is contained in the Quality Assurance Handbook for Air Pollution Measurement Systems (EPA-600/4-77-027A). Phase II air sample duplicates were analyzed by TEM, using indirect preparation and both high and low magnification scans (Yamate et al., 1984, adding Hayward's low magnification method, Appendix B).

The following is a brief explanation of the criteria used to identify asbestos using TEM for this study (from Yamate et al., 1984). At approximtely 20,000 times magnification, a fiber was identified as an asbestiform particle with an aspect ratio of 3 to 1 or greater (length at least 3 times greater than width) with parallel sides. A bundle was defined as several fibers in parallel arrangement, with fibers closer than the diameter of one fiber. A cluster is a random intermixed arrangement of many fibers. A matrix particle is a fiber or fibers with one end free, and the other end imbedded in or hidden by a particulate. Total asbestos structures is the sum of all the entities described above; total fibers is the number of single fibers. During analysis of Phase I air data, any structure shorter than 1 micron in length was not counted, and structures greater than 100 microns in length with a width greater than 3 microns were not included. Fibers or structures longer than 5 microns, wider than 0.25 microns, and with an aspect ratio greater than 3 were considered equivalent to PCM method structures and were called "PCM equivalents."

For Phase II, using the indirect preparation method, the counting procedures were modified slightly. In addition to the high magnification (20,000 times) scan, a low magnification scan at approximately 1,000 times was done as recommended by Steve Hayward (Appendix B) to detect PCM equivalent structures, or PCMes. This technique simulated the low resolution PCM method by identifying large asbestos structures at low magnification. The Region IX definition of PCMe structures using this method is considered equivalent to PCM fibers identified using the actual

PCM method. Data analysis in Section 5.0 was done for both high magnification total structures and PCMe structures; the risk assessment (Section 7.0) is based on PCMe structures.

Soil And Sediment - Asbestos

Asbestos analyses for soil and sediment included Polarizing Light Microscopy (PLM) and Transmission Electron Microscopy. Surface soils were also analyzed for particle size and moisture content.

The method for PLM was Interim Method for Determination of Asbestos in Bulk Insulation Samples (EPA-600/4-82-020) (Appendix B). The soil sample preparation for TEM is specified by Hayward and Lowe (no date) (Appendix B)in their procedure called Methodology for the Analysis of Asbestos in Soil by Transmission Electron Microscopy. For TEM analysis, the soil is ground in a mortar, then suspended in water. The water is then filtered, and the filter analyzed by TEM like an air filter. Particle size was determined using a standard sieve analysis according to ASTM-C136.

Water - Asbestos

Water samples were analyzed for asbestos using TEM, using the Interim Method for Determining Asbestos in Water (EPA-600/4-80-005) (Appendix B). Asbestos data is reported as millions of fibers per liter.

Target Compound List (TCL) Substances

Analyses for TCL substances were performed by EPA's Contract Laboratory Program as part of Routine Analytical Services (RAS). Analytical methods and quantification limits are contract specified. The following analyses were requested:

Volatiles
Semivolatiles
Pesticides/PCBs
Metals and cyanide

SECTION 4.0

PHYSICAL CHARACTERISTICS OF THE SITE

4.0 PHYSICAL CHARACTERISTICS OF THE SITE

4.1 INTRODUCTION

To understand the media influencing contaminant transport at the South Bay Asbestos site, the following summary of the physical characteristics of the site has been developed. An understanding of these characteristics will aid in evaluating potential remedial alternatives and in assessing risk to the public and to the environment. The primary media of asbestos transport is air, so the meteorologic characteristics of the site are critical to an understanding of contaminant transport. Meteorologic data were collected using an on-site weather station from August 1987 to October 1987. Site specific data collected during this time period plus regional data are presented in Section 4.2.

Airborne asbestos contamination may result from entrainment of fibers in surface soils into the air. Therefore, surface soils were studied to learn their grain size composition and moisture content. In addition, subsurface soils were described in order to understand the distribution of fill material, which may be contaminated. Section 4.3 presents the soil data collected.

The groundwater and surface water hydrologic systems were also investigated. Both groundwater and surface water may serve to transport contaminants within and away from the site. Because the site is on the edge of the San Francisco Bay, site hydrology is closely related to the Bay system. Section 4.4 describes site hydrology. In Section 4.5, the ecology of this unique Baylands site is discussed.

Finally, land use and demography are presented in Section 4.6 to aid in the assessment of risk to local populations and assist in the selection of remedial actions.

4.2 METEOROLOGY

Site-specific meteorological data was gathered from July 24 through October 31, 1987, at air monitoring site 1, located at the Environmental Education Center approximately 1 mile northeast of Alviso (Figure 3-1). The following parameters were recorded continuously during the field program:

1) Wind speed
2) Wind direction
3) Temperature
4) Precipitation
5) Sigma theta—the standard deviation of the horizontal wind direction

Data were recorded on analog strip charts. All wind and temperature data were sampled at 10 meters above ground level. The top of the precipitation gauge was approximately one meter above ground level.

Wind direction and wind speed information for the period are summarized in tables and figures provided in Appendix H. Total frequencies of occurrence for wind speed and direction are also provided in tabular form and graphical form in Appendix H.

The predominant wind direction during the sampling period was from the northwest (63.1% of the time) with secondary frequency maxima from the north-northeast (13.5%) and south-southeast (13.2%). Average wind speed was 3.3 meters per second (mps), or 7.4 mph. During the sampling period, 12% of observed wind speeds were above the threshold typically necessary for resuspension of surface soil particles (approximately 13 mph, Woodruff and Siddoway, 1965). A maximum hourly wind speed average of 9 mps (20 mph) was recorded several times during the monitoring period. On occasion, the average hourly wind speed was below the instrument threshold of 0.33 mps (0.74 mph).

Wind speed frequency distributions and wind direction by Pasquill-Gifford stability class are provided in tabular form in Appendix H. Atmospheric stability is a measure of the atmosphere's ability to disperse pollutants, and includes factors such as turbulence and vertical and horizontal mixing

of air. Appendix H also provides frequency distributions in graphical form for stability classes "A" (highly unstable, high mixing) through "F" (strongly stable, stratified). Stability classes were determined from measurements of the standard deviation of the horizontal wind direction (sigma theta), time of day, and wind speed. Frequencies of occurrence for classes "A" through "F" were 6.0, 2.0, 4.8, 37.7, 42.2 and 7.3 percent, respectively.

Daily frequency distributions of wind speed and direction for 4-hour periods during the sampling program are listed in the tables and figures provided in Appendix H. The strongest average wind speeds of 5.2 mps (11.6 mph) occurred during the period 1200 Pacific Standard Time (PST) to 2000 PST. This period showed a strong wind direction frequency maximum from the northwest (in excess of 96%). The late morning (0800-1200 PST) and nighttime (2000-2400 PST) periods also exhibited a strong northwesterly wind direction frequency maximum, although the average wind speeds were moderate at 2.5 and 2.8 meters per second, respectively. During the late night and early morning hours (0000-0800), wind speeds were light, averaging 1.9 meters per second (4.2 miles per hour). During the period 0000-0400 PST, winds were still predominately from the north-northwest (in excess of 44%) but a secondary wind direction frequency maximum from the southeast was also indicated (in excess of 23%). During the early morning hours (0400-0800 PST), the predominant wind direction became south-southeast (36.8%) with a secondary wind direction frequency maximum from the north-northwest (30.2%).

The average temperature for the sampling period was 17.5°C (63.4°F). The highest hourly average was 31°C (88°F), recorded during hour 14 (1300-1400 PST) on October 5. The minimum hourly average temperature of 11°C (52°F) occurred during hours 6 and 7 (0500-0700 PST) on October 19.

No precipitation was recorded from July 24 through October 22, 1987. Precipitation occurred on three days during the period October 23 through 28, with the highest daily total of 0.59 inches on October 27. The highest hourly amount of 0.20 inches was recorded during hour 20 (1900-2000 PST) on October 27.

Observed wind patterns from the on-site data were compared with large-scale weather (synoptic) events for the entire sampling period. Daily Weather Maps (U.S. Dept. of Commerce, National Weather Service) were reviewed to determine major weather events. Frontal passage through the area during this time occurred infrequently; however, a cold front passed just to the north on September 15 and again on September 26. According to National Weather Service observations at San Francisco, the wind direction was mainly from the northwest on both occasions. Data collected at the on-site meteorological station also indicated a predominantly northwest wind, particularly during daytime hours.

On several occasions, low pressure centers off the California coast at 500 millibars (mb) or surface lows in the Central Valley of California and over the Great Basin triggered widespread cloud cover and scattered precipitation throughout the San Francisco Bay region. Although Daily Weather Maps showed precipitation occurrences on October 23 and 24 as well as October 27 through 29, actual rainfall was only recorded on site on October 23, 27, and 28. For this sampling period, observed wind patterns and precipitation occurrences were generally consistent with National Weather Service observations.

Comparisons of the short-term, on-site meteorological data set were also made with climatological data records from the Moffett Field Naval Air Station (Moffett NAS) and the San Jose International Airport (SJ Airport). Climatological records compiled by the California Air Resources Board (CARB) listed the predominant annual wind directions for Moffett NAS and SJ Airport as north-northwest (36.2%) and northwest (35.9%). The secondary annual wind directions indicated by CARB for Moffett NAS and SJ Airport are listed as south-southeast (13.4%) and southeast (19.4%). Winds with a southeasterly component occurred most frequently during the winter quarter at Moffett NAS and SJ Airport. The predominant climatological wind direction for the summer and fall quarters was the same as the annual at Moffett NAS (north-northwest) and SJ Airport (northwest) but occurred at a higher frequency (51.6%, summer and 53.8%, fall). Secondary predominant wind directions for Moffett NAS for the summer and fall quarters were west

(9.2%) and south-southeast (13.0%). At the SJ Airport, the secondary predominant wind directions were north-northeast (9.8%) for the summer quarter and southeast (20.5%) for the winter quarter.

Average annual wind speeds at Moffett NAS and SJ Airport compiled by CARB were 5.1 mph and 7.2 mph, respectively. Average climatological summer and fall wind speeds at Moffett NAS and SJ Airport were 5.5 mph and 7.0 mph, respectively. The highest average quarterly wind speeds at Moffett NAS occurred during the summer quarter, while at the SJ Airport, the highest speeds occurred during the spring quarter.

Based on comparisons with National Weather Service observations and climatological data from Moffett NAS and SJ Airport, the on-site meteorological data collected from July 24 through October 31, 1987 appear representative for the Alviso area. The northwesterly wind direction pattern reflects the sea breeze and topographic effects which strongly influence winds on a day-to-day basis in the South Bay area. A weak southeasterly return flow occurs in the late night and early morning hours as a result of land breeze and topographic effects. Wind speed, wind direction, and precipitation data collected on-site appear to be typical of the region for this season and location.

4.3 SOILS

On September 30 and October 1, 1987, CDM collected 34 surface soil samples for grain size and moisture content analyses. Analytical results are presented in Appendix I and sample locations are provided in Figure 3-2. Of the 34 samples analyzed, 31 are composed of sand and gravel (grain size > 300 um) with minor fine sand (grain size from 75 to 300 um). Of these 31 samples, six are composed of more than 90 percent sand and gravel. Three of the 34 surface soil samples are composed of fine sand. These three samples were collected from the Environmental Education Center and the Marina.

Moisture content in the 34 samples ranges from 0.6 to 14.8%. Eighty percent of the samples contained less than 5% moisture; this reflects the

dry conditions typical of the site during early fall. The two highest moisture content values were for the two samples collected in the early morning from the Environmental Education Center levee. These values probably reflect the more moist conditions in the marsh area.

CDM collected subsurface soil samples from 10 soil borings within the community of Alviso and five groundwater monitoring well borings on the perimeter of the Marshland Landfill. Locations of all the wells and borings are shown on Figure 3-3. Descriptions of the soil samples are provided in Appendix G.

Within the community of Alviso, subsurface sediments consist of clay and silty clay, varying in color between dark brown, green, greenish black, black, and gray, typical of the fine-grained materials deposited in the Bay estuarine environment ("Bay Mud"). North of the Guadalupe River, the clay is overlain by fill material with a silty clay matrix. The thickness of fill ranged from 1 to 9 feet. The two wells drilled on the south side of the river are located on a levee contructed from material dredged from the Guadalupe River. Sediment in these well borings consists of silty clay and clay; no imported fill was observed.

In contrast to the wells and borings within the community of Alviso, the wells drilled on the perimeter of the Marshland Landfill encountered mainly fill material and little native clay. Fill material included concrete, wood, asphalt, metal, bricks, styrofoam, glass, and other types of refuse.

4.4 HYDROLOGY

The site is located adjacent to the Guadalupe River, which along with Coyote Creek, Los Gatos Creek, and Llagas Creek, forms a major drainage basin within the Santa Clara Valley. The Guadalupe River and Coyote Creek flow into San Francisco Bay immediately north of the site (Figure 4-1). Tidal effects near the Bay make the Guadalupe River water brackish and unsuitable for beneficial use except for non-contact recreation.

Intensive irrigation-based agricultural development began about 1900 in the Santa Clara Valley. This activity reached a peak around 1950 with the consumption of over 100,000 acre ft of groundwater per year. A water-level index well in the valley near San Jose showed a 220-foot drop in head between 1916 and 1966. As a result of this severe overdraft, land subsidence has occurred throughout much of the valley. Between 1934 and 1967, as much as eight feet of land subsidence was measured in parts of the San Jose area (Iwamura, 1980). In the years since 1966, the addition of imported water supplies and the implementation of recharge ponds has reversed this trend. Although water levels have risen, they no longer exhibit the artesian conditions which existed once in much of the Santa Clara Valley. Like surface water, groundwater near the Bay is unsuitable for use due to overpumpage and the intrusion of saline water.

Surface Water

The Guadalupe River enters San Francisco Bay just north of the South Bay Asbestos Site. The area surrounding the site is characterized by broad, flat marshes, salt evaporation ponds and some urban development. Miles of levees and dikes surround the salt evaporation ponds. The low topography and proximity of the site to the Bay make the area very susceptible to flooding. Flooding can occur from three sources: 1) Guadalupe River may overtop its levees, 2) Coyote Creek may escape its channel and flow toward Alviso, and 3) tide waters may overtop the levees around Alviso and become trapped in the community (City of San Jose, 1984). Tidal effects may also exacerbate the effects of high river flows. Channelizing of the Guadalupe River has reduced the flood risks associated with rain storms and runoff, but prevents natural drainage of site flood waters. It is estimated that during a 100-year flood, the community of Alviso would be inundated by seven to eight feet of water (U.S. Army Corps of Engineers, 1975), as observed during the flood of 1983.

Surface water quality at the site is affected by several factors. Rainfall in this area is seasonal, with 90 percent occurring in the late fall and winter months. Surface water quality during the wet season tends to be higher than during dry periods. Water quality in the Guadalupe River Basin

is typically at its lowest during late summer. In addition, tidal influence causes increased salinity in the river near the Bay, particularly during the dryer months when flows are less diluted by fresh water. Surface water samples collected for the RI contained high concentrations of dissolved constituents such as calcium, magnesium, and sodium; this reflects the marine influence on the river.

Groundwater and Hydrogeology

Aquifers of the Santa Clara Valley are composed of unconsolidated to semi-consolidated alluvial materials derived from the surrounding mountain ranges. Tidal and marine deposits are interbedded with these alluvial materials, becoming thicker in areas near San Francisco Bay. Confined and unconfined groundwater aquifers occur in the Santa Clara Valley. The maximum depth to bedrock in the valley is approximately 1,500 feet, with little usable water occurring below 800 feet. Depths to groundwater in the Santa Clara Valley range from less than 5 feet near the Bay to over 200 feet near some inland pumping centers. In general, groundwater flows from the elevated perimeters of the Valley toward the interior and the Bay. Large pumping depressions also influence the direction of groundwater flow. Vertical flow in the valley is predominantly downward, especially in areas where deep pumping occurs.

In the vicinity of the South Bay Asbestos site, the native sediments are predominantly fine-grained clays, silts, and sandy clays. These beds of fine-grained material are cut by ancient stream channels at varying depths below the surface. Buried stream channels are composed of coarser grained materials which constitute the local aquifers. Buried stream channels such as these are not continuous layers in the subsurface, but rather sinuous strands of porous material. These aquifers can range from a few inches to tens of feet in thickness. As discussed in Section 4.3, wells and borings drilled for the RI encountered only clay and silty clay in the upper 20 to 30 feet below the site.

The field investigation revealed that the groundwater table occurs between 5 and 10 feet below ground level in Alviso. As is discussed in Section

4.3, the wells and borings encountered mainly clay and silty clay in the subsurface. Several minor clay zones did contain sand grains. The low permeability of the sediments explains the very low flow rates during well sampling (Section 3.1.4). Several of the wells were pumped dry during sampling events.

Groundwater flow direction at the site is unclear due to the flatness of the topography, the river, salt evaporation ponds, and water mounding at the Marshland Landfill. A study performed by EMCON Associates showed that daily tides have little or no effect on groundwater levels in the Marshland landfill wells. (EMCON, personal communication).

Evacuation of groundwater from the Santa Clara Valley has resulted in increased salt water encroachment in areas near the Bay. Groundwater in the Alviso area is brackish and too saline for drinking or irrigation. This is reflected in the high concentrations of constituents such as calcium, sodium, magnesium, and potassium that were formed in groundwater samples collected during the RI (Appendix I). High electrical conductivities reflect the high salinities of these groundwater samples.

4.5 ECOLOGY

Introduction

Marshlands on the perimeter of San Francisco Bay collectively constitute the largest estuarine environment on the Pacific Coast. Once occupying over 300 square miles these Bay marshlands now cover approximately 75 square miles (BCDC, 1979). The South Bay Asbestos site and the community of Alviso lie in an area which was once dominated by salt marsh terrain. Much of the original marshland environment is now occupied by urban and industrial developments, including surrounding salt evaporation ponds, agricultural development, landfills, industry, and housing. However, undisturbed wetlands still exist near the site, some of which are part of the San Francisco Bay National Wildlife Refuge. The marsh environment or Baylands ecosystem is that habitat from sea level up to 10 feet above sea level, and can be characterized by three distinct zones: wetland, transition, and upland (Aqua-Terra, 1986).

The various habitats of the South Bay marshes support a diverse population of wildlife. At different times of the year, 200 species of birds and 40 species of mammals are found in South Bay marshes (Harvey, et al., 1977). Several endangered or rare bird species and endangered mammals are part of this diverse fauna. Also found in this population are many species of migratory shorebirds and wintering waterfowl. The wetlands and salt marshes of the South Bay support plants and algae which are essential to wildlife in and around the Bay. Construction of salt evaporation ponds and dredge spoil piles have caused alteration of native vegetation habitats.

Plant Communities

The wetland zone of the marsh environment is inhabited by a few plants which have adapted to the high salinities typical of this zone. These plants provide the habitat and breeding grounds for a wide variety of birds, most notably migratory shore birds and waterfowl. Vegetation diversifies as one moves inland and the water becomes less saline. The transition and upland zones of the marsh supports a wide variety of plants including agricultural crops.

In the wetland zone, dominant species include California cord grass and pickleweed (*Salicornia*). A variety of algae inhabit the saline tidal flats which are exposed at low tide in this area. These algae play an important role in the production of oxygen in tidal waters. Marsh plants in the outer portions of the marsh provide shelter and food for a diverse community of birds and insects.

Farther inland, where available water is less saline, the transition zone includes plants such as alkali heath, fat hen, salt grass, and rabbits-foot grass. In the upland zone, common on dikes and levees, gum plants, coyote bushes, and sweet fennel are found. In general, the plant community diversifies as fresher water becomes available.

The various plant communities in the Alviso area provide shelter and nesting grounds for many animals, especially birds. In addition, marsh

plants support large quantities of insects and marine invertebrates, which are primary food sources, especially for birds. Freshwater and marine fish are also supported directly and indirectly by plants of the marsh environment.

Animal Communities

Intense urban development has taken place in the South Bay in the last 50 years. However, a fairly diverse community of animals survives in the marshes and salt ponds around Alviso. This community includes mammals, migratory birds, shore birds, song birds, game birds, reptiles, and amphibians, as well as freshwater and marine invertebrates.

Mammals found in the marsh and salt pond habitats around Alviso include shrews, mice, raccoons, rabbits, and weasels. Included in this group is the endangered Salt Marsh Harvest Mouse (*Reithrodontomys raviventris*). In areas along the Guadalupe River, muskrats and raccoons are common mammals which feed on fish and invertebrates in the river. Along levees, dikes, and upland areas, jack rabbits, ground squirrels, and weasels are common, along with mice and rats.

Assorted reptiles, amphibians, and invertebrates inhabit the marshlands around Alviso. Reptiles and amphibians present in the site area include turtles, lizards, snakes, newts, salamanders, toads, and frogs. A wide variety of clams, shrimp, and other invertebrates also occupy various niches in the waters of the South Bay.

Rivers and streams in the site area once contained a variety of fresh water fish. Only a few non-game fish now remain. The elimination of many species is attributed to poor water quality and low summer stream flows. Most South Bay streams and rivers experienced runs of anadromous steelhead trout prior to the 1950's. However, pollution probably eliminated most steelhead and other anadromous species from this part of the Bay. In addition, channelization and the erection of barriers has made it impossible for some species of fish to breed. Eighteen or more species of fish exist in the wetland zone area of the South Bay. Shiner perch, top smelt, and staghorn culpin are the predominant marine species.

Birds constitute the largest and most diverse wildlife community in the South Bay estuary. Some 200 species of birds inhabit this area at various times of the year. The most noticeable group of birds are the large migratory shorebirds. Shorebirds such as herons and egrets occupy the Bay area marshlands year round. Other birds, including ducks, terns, gulls, rails, and blackbirds are common in the Alviso area. Endangered or rare species found near Alviso include the California clapper rail, California brown pelican, California least tern, the peregrine falcon, and the burrowing owl. The golden eagle has also been sighted using the marsh during winter months (Aqua-Terra, 1986).

4.6 LAND USE AND DEMOGRAPHY

Historically, Santa Clara Valley has been a major agricultural region. Due to the expansive growth of the semiconductor and computer industry, agricultural land is rapidly being replaced by urban development. Farm land in the valley is dotted with large and small light-industrial office complexes, which support the high-tech industry. Residential construction and service businesses are also rapidly expanding on former agricultural land.

The 550 acres which make up the Alviso community is zoned for a mixture of land uses. Alviso has land zoned for agriculture, light and heavy industry, multiple dwellings, and mobile-home parks. Manufacturing/industrial entities in Alviso include metal foundries and truckyards. Retail businesses in Alviso include a number of small markets and restaurants. Government and educational organizations in Alviso include a local post office, public library, and two grammar schools.

Alviso is located near several major transportation routes. Highway 237, less than a mile south of the site, runs southeast to northwest and connects U.S. Interstate Highways 101 and 880. US 101 is located to the west of Alviso and runs north and south along the western edge of the San Francisco Bay; US 880 runs north and south along the eastern edge of the San Francisco Bay.

The demographic composition of the site is summarized in Table 4.1 (taken from Aqua-Terra, 1986). In 1980, there were 528 households occupying 481 residences within Alviso. Approximately 54 percent of the homes in Alviso are owner-occupied. Of occupied housing units in 1980, 137 were built after 1969 when Alviso was incorporated into the City of San Jose, increasing the existing number of housing units in Alviso by nearly 50 percent. The population in Alviso appears to be quite mobile (Table 4.1). Only 40 percent of the site area residents were in the same house in 1980.

SECTION 5.0

NATURE AND EXTENT OF CONTAMINATION

5.0 NATURE AND EXTENT OF CONTAMINATION

The purpose of this section is to present and discuss a summary of the results of the RI sample collection and analysis, to determine the magnitude and extent of contamination in air, soil and water. As described in Section 3.0, air, surface soil, subsurface soil, groundwater, surface water, and sediment samples were collected to investigate the extent of both asbestos and other hazardous contaminants. This section describes the distribution and magnitude of contamination in each of the various sampled media. The data results are tabulated in Appendix I. The complete data analysis is presented in Appendix J; this section is a summary of the information presented there.

All data collected during this investigation were subjected to extensive Quality Assurance (QA) and Quality Control (QC) procedures. These procedures and the data quality results are presented in Appendix J. Sample data for each individual medium are presented, with a discussion of the magnitude and extent of contamination and important relationships which may influence contaminant transport. This information will be used in Section 6.0 to determine the exposure pathways of concern.

5.1 SOIL INVESTIGATION RESULTS

The discussion of soil results will be presented as follows. First, the surface soil asbestos results will be presented and discussed. The subsurface soil results will follow, for both asbestos and target compound list (TCL) compounds. Finally, the Guadalupe River sediment results will be presented for asbestos and TCL Compounds. In general, the soils data can be summarized by stating that asbestos fibers in surface soils is the contaminant of concern; hazardous constitutents (TCL) were not detected at significant levels in soils. The distribution of asbestos in surface soils does not follow any predictable pattern. In general, the RI soil results confirm the previous soil data in a gross sense; for example, the generally high concentrations in the ring levee soils were repeated. The results are discussed in more detail in each following section.

5.1.1 DATA QUALITY - SOIL

As presented in detail in Appendix J, precision and accuracy were evaluated for soil asbestos samples. A similar analysis was not performed for organic and inorganic TCL compounds, since these compounds were not detected frequently enough to allow a detailed error analysis. The duplicate analysis results are presented in Table 5.1. Standard results are presented in Table 5.2.

Table 5.1 indicates no clear difference in %RSD for any of the types of duplicates. This observation indicates that the laboratory variability of the TEM method (due to preparation, fiber counting, or other sources of error) dominates over other sources of variability. PLM duplicate results indicate that lab variability is less than for the TEM results. Thus the precision of the PLM analyses is greater than the precision of the TEM analyses. The standards results presented in Table 5.2 are inconclusive, so no general statements concerning accuracy or systematic errors can be made.

As a result of the poor performance of the laboratory on duplicate and standard samples, the Phase I TEM and PLM results should be considered as qualitative indicators of asbestos concentrations. Thus the data can be used to point out areas of high or low asbestos concentrations; however, the exact numerical concentration at any location is uncertain. The errors observed are such that a sample with a reported concentration of 0.5% could actually contain as low as 0.1% or as high as 2.0% asbestos. However, the actual concentration of this sample is very unlikely to be as large as 5.0%. This uncertainty must be considered when using the asbestos soil concentration data. The additional background samples collected in Phase II could not be evaluated because of the small number of samples.

The data quality for the TCL analyses can be summarized as follows. Semivolatile organic soil analyses were cited for poor precision, blank contamination, and poor spike recovery. Volatile organic analyses were cited for blank contamination and poor spike recovery. Inorganic samples were cited for exceeding mercury holding times, poor lead precision and

poor spike recoveries for mercury and antimony. The impact of these data validation citations on data quality are that all reported semivolatile concentrations should be considered as qualitatively but not quantitatively correct. Nearly all volatile organic concentrations are considered as estimates due to blank contamination. Metals concentrations are considered valid for all purposes except for lead, mercury, antimony, and zinc concentrations, which are qualitatively but not quantitatively identified.

5.1.2 SURFACE SOIL RESULTS

As described in Section 3.0, 62 samples of surface soil were collected at 40 locations in Alviso. The samples were collected from open areas with a high likelihood for human contact, from areas not sampled previously, or from previously indicated "hot spots" to confirm existing data. Figures 5-1 and 5-2 show the sampled locations and the results. All samples were analyzed by polarized light microscopy (PLM). About half of the samples were also analyzed by the more rigorous transmission electron microscopy method (TEM). Each sample location was also tested for soil grain size and moisture content. Sample results are included in tabular form in Appendix I.

The results for PLM and TEM have been summarized in Tables 5.3 and 5.4.

Extent of Asbestos Contamination in Surface Soil

The results indicate that asbestos is present in soils at or near known sources of serpentine rock, such as the ring levee and the small dike surrounding the Alviso Marina picnic area. High values near the intersection of Liberty and Catherine Streets probably indicate areas where levee building material was stockpiled during construction. When previous soil sampling data (Figures 2-1, 2-2, 2-3) are examined in conjunction with the RI data, the original discovery site where waste asbestos-cement pipe was dumped (at Liberty and Moffat Streets near the Guadalupe River levee) is also generally elevated in asbestos. The asbestos values in the remainder of the site, including most of the residential areas, are generally low or not detected.

There are no detectable differences between serpentine rock and waste asbestos sources, based on asbestos type, fiber size, or morphology. However, it should be noted that the specific concentration of asbestos can vary widely at any particular location, as shown by the co-located duplicate results (for example, PLM samples 037 and 038, TEM samples 040 and 041). This variability may be due to a combination of the natural variation of serpentine or waste asbestos in the fill soils in Alviso, and the variability of the asbestos soil analytical techniques. The data quality analysis (discussed in Section 5.1) appears to indicate that PLM results are less variable than TEM results.

Comparison of PLM and TEM Results

As described in Section 3.0, samples were analyzed by both the PLM and TEM methods to test the usefulness of both types of analysis for natural soils. The objective of performing both analyses was to investigate whether a site-specific relationship between TEM and PLM could be established, so that less-expensive PLM analyses could be substituted for the costlier TEM analyses. PLM is the method typically used for solids, but it has a relatively high level of detection (~1%) and estimates asbestos using a field-of-view counting method. TEM was anticipated to produce more accurate results, although grinding of the sample was required to achieve homogeneity during the water suspension step. The grinding may have broken up asbestos clusters or bundles in the natural soil, producing many smaller fibers which potentially could effect mass calculations.

Although the two methods are very different and produce results with different units, the sample pairs analyzed by TEM and PLM were plotted together to assess the possible site-specific comparability of the method results. Figure 5-3 is a scattergram showing TEM results versus PLM results for surface and subsurface soils (approximately 60 soil pairs). For the purpose of the figure, PLM non-detect values, or <1 percent, were set equal to zero. The plot shows that when PLM fails to detect asbestos, TEM values are an average of 0.105 percent by weight, and do not exceed 0.5

weight percent. This indicates that if PLM analyses are used to guide site remediation, it is likely that a non-detect PLM result contains less than 0.5 percent asbestos by weight.

5.1.3 SUBSURFACE SOIL RESULTS

As described in Section 3.0, 51 samples of subsurface soil were collected from 16 borings in Alviso. Five borings (Monitor Wells MW-001 through 0055) were drilled in areas of previous landfill activity, and groundwater monitor wells were installed in the borings. Five borings (Borings 007 through 011) were drilled at locations in Alviso suspected of having received soil fill. A background boring was drilled at air station 5 at Agnews Hospital. Finally, soil samples were also collected, in coordination with Emcon Associates, during installation of monitor wells around the perimeter of the Marshland Landfill. Table 5.5 summarizes the borings, sample depths, and analytical results. All samples were analyzed for asbestos by PLM, 20 samples were also analyzed for asbestos by TEM, and 13 samples were analyzed for Target Compound List (TCL) compounds to assess the potential for hazardous wastes. The samples results are tabulated in Appendix I and Table 5.5.

Extent of Asbestos Contamination in Subsurface Soil

Asbestos was detected in the upper 1.5 feet of boring MW-001 located at the original discovery location at the foot of Liberty Street adjacent to the Guadalupe River levee. Traces of asbestos were also detected in boring MW-002, across Gold Street at the St. Claire landfill. Boring MW-004, drilled in the Guadalupe River levee (Figure 3-3), had a value of 7% asbestos by PLM at a depth of approximately 10 feet (Table 5.5). This high value may be due to remnants of the Santos Landfill unearthed during levee construction in 1963. Borings MW-003 and 005 had no significant asbestos.

Of the soil borings (BOR 007-011), boring BOR-009 at Catherine and State streets had fairly high levels of asbestos (5% and 2% by PLM) at the surface and at 5 feet. Surface soil samples taken near boring 9 also showed detectable asbestos indicating that this area may have served as a

stockpile location of serpentine soil fill during ring levee construction. The other soil borings in Alviso showed no significant asbestos levels (Table 5.5).

The background soil boring at Agnews Hospital East was located at the edge of an agricultural field. The sample, taken from the upper 7 inches of soil, showed detectable asbestos (1% PLM and 0.08% by weight TEM). The background location was chosen based on its distance from the site (Figure 3-3) and the assumption that the soils are natural and in-place. The origin of the asbestos detected at this location is unknown. Additional surface samples were collected from the same location during Phase II; these results are discussed in Section 5.1.5.

Asbestos was detected in Boring G3 and G5 at the Marshland Landfill (Table 5.5). Boring G3 shows a TEM result of 0.3% (not detected by PLM) in the upper foot of soil, and Boring G5 shows a TEM result of 0.45% in the upper foot and 1.0% PLM at 16.5 feet. Minor amounts of asbestos were detected at depth by TEM in borings G6, G9, and G11 (Table 5.5) Although the borings are not located in the landfill itself, but around the perimeter, the field logs of the borings show that rubble and debris were encountered. The debris may be the source of the asbestos detected.

The asbestos present in the borings may also be due to asbestos-bearing fill rock or soils used as landfill cover or to construct the perimeter roads. The field logs of the borings note the presence of serpentine rock pebbles and fragments.

Extent of TCL Contamination in Subsurface Soil

Soil samples for Target Compound List (TCL) analysis were collected from each of the monitor well borings, located in or near former landfilling activities (Table 5.5). The results of the analyses from the borings in Alviso (Monitor wells 1-5) show no obvious evidence of hazardous waste disposal; the isolated occurrences of detectable compounds can be attributed to laboratory contamination. The borings around the Marshland Landfill show small amounts of common volatile compounds.

The following is a summary of the TCL compounds detected:

- Chloroform - One sample from 10 feet in Monitor well boring 3 showed 9 micrograms per kilogram (ppb) chloroform. This occurrence is assumed to be a laboratory contaminant, based on blank contamination (see Section 5.1.1, Data Quality).

- 4,4-DDD - A sample from 20 feet in Monitor well boring 4 showed 67.6 ppb of this pesticide compound. No other pesticide compound was detected in this boring, and Monitor well 5 from the same general area did not detect this compound. This occurrence could be attributed to either disposal in the Santos Landfill or pesticide use during previous agricultural activities in nearby fields, followed by disruption of the soils during river realignment.

- 2 - Butanone - The same sample in Monitor well boring 4 showed 21 ppb of 2-butanone. This is assumed to be a laboratory contaminant based on blank contamination results.

- 4,4-DDT and 4,4-DDE - The background boring showed concentrations of 129 ppb of 4,4-DDT and 126 ppb of 4,4-DDE. No other organic compounds were detected. The presence of these pesticides can be attributed to the use of the adjacent field for food crops.

- Various volatile compounds - Four of the Marshland Landfill borings showed detectable concentations of several volatile compounds. These are acetone, ethylbenzene, total xylenes, methylene chloride, 4-methyl-2-pentanone, and carbon disulfide. These are common laboratory contaminants, and may be due to blank contamination not indicative of site contamination.

- Inorganic compounds - Many inorganic compounds are present above detection limits. It should be noted, however, that the levels present are characteristic of the site soils since the levels are comparable to the background boring, and, in fact, are generally lower than average soil values compiled for the U.S. None of the inorganics, with the possible exception of antimony, are present at high enough levels to indicate a potential human health risk due to ingestion (EPA, 1987a).

5.1.4 RIVER SEDIMENTS RESULTS

As described in Section 3.0, samples of Guadalupe River sediments were collected to screen for the presence of asbestos or hazardous constituents, which may have entered the river from the former site landfilling activities. Three samples of sediment were collected at locations successively downstream from Alviso, as shown on Figure 3-3. A sample was also collected upstream of the site, from the Highway 237 bridge crossing the river. All samples were analyzed by PLM, and samples 001 and 002, downstream of Alviso, were also analyzed by TEM. All sample results are presented in Appendix I.

Extent of Contamination - Asbestos

The only sample in which asbestos was detected by PLM is the upstream or control sample, at 1 percent. The two TEM samples downstream detected very small quantities of asbestos. These results would tend to indicate that the river sediments are not an important "sink" or source of asbestos.

Extent of Contamination - TCL

No TCL organic contaminants were detected in the sediment samples. Inorganic results are characteristic of the soils and rock which are the source of the sediments. The sediment inorganic results are similar to the background boring and the subsurface soils discussed in the previous section.

5.1.5 PHASE II SOIL RESULTS

As described in Section 3.0, additional soil samples were collected during the Phase II investigation to better define "background" soil characteristics for asbestos. The soil samples were analyzed for PLM and TEM asbestos. Results are presented in Appendix I.

Additional Background Samples

Two co-located soil samples were collected at the background boring location (Air Station 5) shown in Figure 3-3. These Phase II samples were collected from the upper inch of soil, while the Phase I background boring was a composite of the upper 7 inches of soil. Each of these co-located samples showed no detectable asbestos.

An additional background sample collected in the marsh, between the Environmental Education Center and Alviso, contained no detectable asbestos.

5.2 WATER INVESTIGATION RESULTS

Waters from the Guadalupe River and site monitor wells were analyzed for asbestos and Target Compound List (TCL) compounds. The Guadalupe River at Alviso is strongly influenced by tidal action due to its proximity to San Francisco Bay; surface waters are saline and similar in composition to Bay water. The shallow groundwater aquifer has been intruded by Bay water and is also highly saline. One well, G11, at Marshland Landfill showed significant evidence of TCE contamination. Analytical results for waters are tabulated in Appendix I. The results are discussed in more detail below.

5.2.1 DATA QUALITY - WATER

As described in detail in Appendix J, the data validation findings for TCL water sample data indicate that it is highly unlikely that the analyses could miss actual organic or inorganic contamination, i.e., data quality is high enough that false negatives are unlikely. The analyses are therefore of sufficient quality to meet the primary data use.

Six groundwater and two surface water samples were analyzed for asbestos. QC samples submitted with these water samples were six rinsate or field blanks and three blind duplicate samples (Tables 5.6 and 5.7). Laboratory

QC samples consisted of three laboratory method blanks and four laboratory duplicates. No check samples of known concentration or spiked samples were analyzed.

In summary, the asbestos water samples passed data validation with few qualifications and, except for one sample, are considered valid for all uses.

5.2.2 SURFACE WATER RESULTS

As described in Section 3.0, samples of the Guadalupe River were collected both upstream and downstream of the former landfills to assess whether asbestos or other contaminants have impacted the river. One sample was collected slightly upstream from the site, at the Rt. 237 bridge, and one sample and a duplicate were collected from the Gold Street bridge (Figure 3-3), downstream from the former landfilling activities. The results of the analyses are summarized in Table 5.8, and the results are presented in tabular form in Appendix I.

Extent of Asbestos Contamination in Surface Water

Asbestos results in total fibers (millions of fibers per liter) show that the upstream sample contained slightly more total asbestos than the downstream sample indicating no impact to the river from the site. However, when only those fibers longer than 10 microns are examined, the downstream sample has 50 million fibers per liter, while the upstream sample contains no detectable fibers longer than 10 microns.

The Maximum Contaminant Level Goal (MCLG) proposed by EPA for asbestos in water is 7.1 million fibers per liter (mf/l) greater than 10 microns in length. If this criteria is applied, the value of 50 mf/l appears significant. However, the river is not used as a drinking water source, because of its salinity, and human contact through recreational use is very limited.

Extent of TCL Contamination in Surface Water

No organic contaminants were detected in surface water. The rinsate blank prepared at the site showed 16 ppb bis-(2-ethylhexyl)phthlate. The presence of this common plasticizer compound in the blank is assumed to be laboratory contamination. Inorganic results are consistent with saline Bay waters.

5.2.3 GROUNDWATER RESULTS

As described in Section 3.0, the five wells installed by EPA in the Santos and St. Claire landfill areas (Figure 3-3) were sampled twice, in June 1987 and October 1987. A total of 17 groundwater samples, including blanks, were collected from these wells. Five of the monitor wells installed by EMCON at Marshland Landfill were sampled in November 1987. One EMCON well, G9, produced so little water that sufficient volume was available for volatiles analysis only. The results are summarized in Table 5.8, and tabulated results are presented in Appendix I.

Extent of Asbestos Contamination in Groundwater

Asbestos was detected in all EPA Monitor wells, at levels similar to those reported in surface water. Fibers longer than 10 microns were only reported for EPA well 3. At the Marshland Landfill, asbestos was detected only in well G5 at concentrations comparable to the levels found in EPA well 3.

The site groundwater asbestos levels are significantly higher than those reported by Hayward (1984) for wells drilled in or near serpentine deposits. However, the groundwater not used for drinking water supply or agriculture because of high salinity.

Extent of Contamination in Groundwater

No TCL organic contaminants were found in the EPA monitor wells; one rinsate blank contained 18 ppb of 2-butanone, assumed to be laboratory contamination. One EMCON well at the Marshland Landfill, well G11, contained 320 ppb of trichloroethene. This contaminant was not found in any of the blanks prepared during the sampling period. TCL inorganic results are consistent with saline Bay water intrusion of the shallow aquifer system.

5.3 AIR INVESTIGATION

Air samples were collected during the period July 24 to October 31, 1987 from a network of five monitoring stations using collection equipment as discussed in Section 3.2.1. Both 24-hour time-integrated and shorter episodic samples were collected. A total of 127 samples and duplicates and 25 blank samples were submitted for Phase I TEM asbestos analyses. As described in Section 3.2.1, two asbestos samplers operated simultaneously at each monitoring station. One of the samples comprising the pair was analyzed while the other was archived. A portion of the archived samples was submitted to a second laboratory for Phase II TEM asbestos analyses. Phase I and II analyses differed in the method of sample preparation and the fiber counting procedures. Phase I samples were prepared via the direct method while Phase II samples were prepared via the indirect method, as described in Section 3.3. The air data base is quite large, and several analyses have been performed to interpret the data. To assist in understanding the data analyses performed, Figure 5-4 graphically shows the interrelationships of the various data sets. The complete data analysis is presented in Appendix J.

Asbestos air sampling results were examined by several methods in an attempt to determine the processes which contribute to the measured asbestos concentrations. The first step in the analysis was an investigation of the data quality and usability of the data. For Phase I asbestos results, the dependence of concentrations observed during episodic

samples were compared with wind speed, wind direction, and particulate concentrations. Similar comparisons were then performed for the 24-hour analyses. A statistical analysis of both the episopic and 24-hour samples was used to assess if asbestos concentrations within Alviso are significantly different from background concentrations. Similar analyses were repeated for the Phase II data. The section concludes with a summary of the major findings. Figures 5-5 and 5-6 clarify the interrelationship between the Phase I and Phase II data sets.

5.3.1 DATA QUALITY

Phase I and Phase II samples were analyzed by different laboratories following different sample preparation and analysis procedures, so a separate data quality evaluation is performed for each data set. Data quality results are presented in Appendix J; the following is a summary.

The data quality of the Phase I data was investigated by considering random and systematic errors. Duplicate sample results for total structures, total fibers, and PCM equivalents (terms defined in Section 3.3) are presented in Table 5.9. The average %RSD values calculated are much lower than the average %RSD obtained for asbestos soil (Section 5.1.1) analyses and similar to the average %RSD obtained for asbestos water analyses (Section 5.2.1).

Phase Contrast Microscopy equivalents measurements (PCMe) determined by TEM were compared with actual PCM measurements for eight non-blank split samples. The concentrations obtained for these samples via the two methods are presented in Table 5.10. In general PCM concentrations are larger than the corresponding PCMe. This result may be due to the tendency of the PCM method to include non-asbestos fibers as part of the total asbestos fiber count since the method cannot distinguish fiber type.

The likelihood of filter contamination was assessed through an examination of blank (unexposed filter media) samples. Trip (unopened filter canisters), field (filter media exposed briefly in the field), and lab (filter media kept in the lab) blank samples provided a total of 42 blank

analyses. Of these 42 samples, only 4 (or approximately 10%) contained any asbestos fibers. Extrapolating from these samples, it is possible to state that in 90% of the air samples analyzed, no errors will be introduced by blank contamination. In the remaining 10% of the samples, blank contamination is possible. Due to the observed blank contamination, nearly all of the Phase I data received a "UJ" flag during data validation (Appendix I). The "UJ" qualifier indicates that sufficient asbestos fibers were not observed in the sample to state with certainty that the reported concentrations are not influenced by blank contamination. However, because the data appeared to be internally consistent (based on precision), data analyses, as described in the following sections, was performed.

The Phase II indirect air data set is much smaller than the Phase I data set; correspondingly, the number of samples available to assess data quality is also much smaller. On the basis of one duplicate, the %RSD for total structure analyses is 31% and the %RSD for PCM equivalent structures is 25%. These %RSD values are very similar to the average %RSD values determined for the direct method.

The Phase II data validation results indicated no major deficiencies in the ambient air data set. In particular, no significant blank contamination was observed so none of the samples was qualified with a "UJ" flag. An important data validation finding was that thousands of small calcium sulfate particles were present in the samples prepared by the direct method. These particles may have obscured asbestos fibers and caused an underestimation of asbestos concentrations in all directly prepared samples. The conclusion of the ambient data validation report was that with the exception of one sample that was termed an estimate, the data are considered valid and usable for all purposes.

5.3.2 PHASE I SAMPLING RESULTS - PHYSICAL ANALYSIS

This section investigates the relationship between asbestos concentrations and physical variables such as wind speed. The purpose of this analysis is to explain site asbestos concentrations, and relate those concentrations to specific sources, if possible. Figure 5-5 graphically shows the types of analyses performed. Appendix J presents the detailed analysis.

Asbestos structure and PCM equivalents (PCMe) results for Phase I analyses are presented in Appendix I. A major feature of these results is that PCMe fibers were only infrequently detected. As a result, any analysis of PCMe will produce uncertain conclusions. For this reason, the data analysis stressed total asbestos structure rather than PCMe fiber results.

5.3.2.1 Analysis of Episodic Samples - Summary

Wind Direction - Samples were collected from each of the five air sampling stations during nine episodic sampling events. Under ideal conditions, this would yield 45 asbestos analyses; however, due to sample overloading by particulates, 42 analyses were obtained. During 8 of the episodes, wind directions ranged from the west to the north (azimuth 270 to 360). During the remaining episode, wind direction ranged from east-northeast to south (azimuth 70 to 180). The wind directions and asbestos structure concentrations observed during each of the episodes are presented in Table 5.11.

During the eight episodes with northwest winds, asbestos concentrations ranging from non-detect to more than 10,000 structures/m^3 were observed at Stations 2, 3, 4, and 5. The concentrations at Station 1 (upwind) ranged from non-detect to 4,200 structures/m^3. Based on these results, it appears that during constant northwest wind conditions, asbestos structure concentrations vary over a wide range at each of the five stations. Thus, northwest winds cannot be used to predict asbestos concentrations at any of the five stations.

The remaining episodic sample was collected during reverse flow conditions when the wind direction was approximately from the southeast. Under these conditions, Station 1, which is normally upwind, is downwind from Alviso. The largest episodic asbestos concentration at Station 1 was observed during these conditions; thus, reverse wind conditions may result in larger concentrations at Station 1. However, relatively high concentrations were observed at all five stations during this episode so other factors may be causing the observed relatively large asbestos concentrations. Based on this one episodic sample it is not possible to state conclusively that reverse conditions cause large asbestos concentrations at Station 1.

Wind Speed - The previous section demonstrates that wind direction does not, by itself, explain the asbestos concentrations observed. Information on wind speed, in conjunction with direction, may better explain the observed range of asbestos concentrations. During consistent northwest wind conditions, it is expected that as average wind speed increases, wind erosion and, hence, asbestos concentrations should also increase. To investigate this relationship, asbestos structure concentration was plotted against average wind speed for the eight northwesterly wind episodes (Figure 5-7).

No relationship is observed between wind speed and asbestos in Figure 5-7. Asbestos structure concentrations as large as 10,000 structures/m^3 occur at average wind speeds ranging from 2.5 to 15.5 mph. In addition, non-detectable asbestos concentrations occur at all wind speeds. Based on Figure 5-7, it is concluded that wind speed does not, by itself, explain the range of observed asbestos concentrations.

Particulate Concentrations - Particulate concentrations, measured in terms of total suspended particulates (TSP) and respirable particulates (PM-10), are plotted against asbestos concentrations in Figures 5-8 and 5-9. Since asbestos fibers are a portion of the total amount of particulates in the air, it was expected (assuming the same source for asbestos and particulate matter) that as particulate matter concentrations increase, asbestos concentrations will also increase. The concentrations plotted in Figures 5-8 and 5-9 do not follow this expected relationship. Large asbestos

concentrations occur over the entire range of particulate concentrations with the largest asbestos concentations corresponding to relatively low particulate concentrations. Particulate concentrations, therefore, cannot be used to predict asbestos concentrations, within this data set.

5.3.2.2 Analysis of 24 Hour Samples

Wind Speed - During the course of each 24-hour sample, wind direction shifts were large so no analysis of wind direction and asbestos concentration was performed. Wind speed and asbestos concentration were compared however. The plot of asbestos concentrations against wind speed averaged over the 24-hour sampling period (Figure 5-10) indicates poor correlation. Wind speed cannot account for the range of observed asbestos concentrations.

Particulate Concentrations - Plots of 24-hour asbestos concentrations against TSP and PM-10 do not show positive correlation (Figures 5-11 and 5-12). Based on these figures particulate concentrations clearly do not account for the range of observed asbestos concentrations. Importantly, the largest particulate concentrations tend to correspond to lower than average asbestos structure concentrations. A possible explanation of this relationship may be that as particulate concentrations increase, asbestos structures become obscured and more difficult to identify. Thus, particulates may be influencing the asbestos concentrations and masking relationships between asbestos and other variables such as wind, speed, wind direction and TSP concentration.

5.3.2.3 Statistical Analysis of Phase I Air Data

The concentrations of asbestos structures observed at each of the five air monitoring stations were analyzed via statistical methodologies to point out significant features of the data.

Qualitative Analysis of the Data - A qualitative examination of the air data presented in Appendix I indicates that there is very low correlation between asbestos structure or PCMe concentrations at the in-town stations.

Using total asbestos structure concentrations, correlation coefficients of 0.08 between Stations 2 and 3; 0.06 between Stations 2 and 4; and 0.67 between Stations 3 and 4 were calculated. A correlation coefficient of 1.0 indicates perfect linear correlation while a coefficient of 0 indicates no correlation. The measured correlation coefficients indicate that the structure concentrations observed at Station 2 are independent of the concentrations observed at Stations 3 and 4. However, there is some correlation between the concentrations observed at Stations 3 and 4. This result may indicate that the factors controlling the asbestos structure concentrations observed at Station 2 are different than the factors controlling the asbestos structure concentrations observed at Stations 3 and 4.

Basic Statistics - The statistics calculated on asbestos total structure concentrations measured at each station are the arithmetic average, the standard deviation, and the standard deviation of the sample mean. Similar statistics were not calculated for PCMe fibers since these fibers were detected very infrequently; thus, any statistics on PCMe fibers would be strongly influenced by whatever arbitary concentrations was assigned to detection limit samples. The arithmetic average provides an estimate of the average ambient asbestos structure concentration in the vicinity of the sampling stations over the three month sampling period. The total asbestos structures and PCMe asbestos statistics for each station are presented in Tables 5.12 and 5.13.

To determine if a significant quantitative difference in asbestos structure concentration exists between upwind and in town stations, a paired difference analysis was performed. A paired difference is defined as the difference between the asbestos concentrations observed at a pair of stations during the same sampling period. If the average asbestos concentration at each of the two stations comprising the pair are equal, the average paired difference will be zero. A paired difference test thus determines whether the average concentration difference between the two stations is significantly different from zero. The test assumes that the

individual differences are not correlated in time. This assumption is acceptable since no temporal correlation has been observed in asbestos concentrations.

As shown in Table 5.14, the z values for the average difference between asbestos concentrations at Station 1 and each of Stations 2, 3, and 4 are greater than 1.72; thus, the difference is significant at the 95% level. Thus, a significant difference in asbestos structure concentration does exist between Station 1 and stations within Alviso. The magnitude of the concentration differences are the averages presented in Table 5.14.

Analysis of Frequency Distributions - Figure 5-13 presents the range (difference between minimum and maximum concentration) of observed total asbestos structure concentrations at each station in relation to the average concentration. This figure illustrates that the maximum asbestos structure concentration at the in-town stations is five to seven times as large as the respective average concentrations. At the upwind and downwind stations, the maximum concentration is approximately three times as large as the average.

Histograms of asbestos structures are presented for each station in figures 5-14 and 5-15. The percentiles of the asbestos structure concentrations shown in the figures are presented in Table 5.12. The median (50th percentile) structure concentration is similar across all 5 stations. At the 75th percentile, the in-town and downwind stations show higher concentrations than the upwind station. At the 90th percentile this trend becomes clear as the concentrations at the in-town and downwind stations are at least twice as large as the upwind concentration. The highest observed concentration in-town (28,000 structures/m^3) is over five times as large as the highest upwind concentration (5200 structures/m^3) and nearly three times as large as the largest down-wind concentration (10,500 structures/m^3).

The interpretation of these results is that on 50% of the sampling days there was no clear difference in concentrations between any of the stations. On 25% of the sampling days there was a slight difference

between the upwind station and the remaining stations and on 10% of the days the concentrations at the in-town stations are clearly different from the upwind station. Finally, examining the maximum concentrations, larger concentrations are observed at the stations within Alviso than either up-wind or down-wind of Alviso. Thus, although the mechanism of asbestos entrainment into ambient air is not well-understood, the data indicate that under some conditions, higher concentrations of asbestos are observed within Alviso than either upwind or downwind of Alviso.

5.3.3 PHASE II AMBIENT AIR DATA - DUPLICATE RESULTS

As discussed previously (Sections 3.2.1 and 5.3.1), the reported concentrations of ambient asbestos in the Phase I data set may not be fully representative of the actual concentrations. To ensure that actual ambient asbestos concentrations have been measured, a portion of the asbestos air data was reanalyzed at another laboratory using a modified sample preparation technique and improved fiber counting procedures.

The steps taken in the analysis of Phase II data are shown in Figure 5.6. The complete data analysis is presented in Appendix J.

5.3.3.1 Comparison Between Indirect (Phase II) and Direct (Phase I) Air Analyses

A subset of 24 duplicate samples, analyzed in Phase I by the direct method, were analyzed in Phase II by the indirect method to determine total asbestos structures and PCMe fibers (Section 3.2.1). The samples selected for reanalysis were distributed among the five air sampling stations and over the range of measured concentrations to obtain a representative sample.

The exact samples reanalyzed and the direct and indirect analysis results for these samples are presented in Table 5.15. The total asbestos structure information contained in this table is presented graphically in Figure 5-16 which compares the directly and indirectly obtained asbestos

concentrations for each sample. An important feature of this figure is that the indirect analyses measured significantly higher quantities of asbestos than the direct analyses.

Figure 5-16 indicates that no numerical relationship can be developed which relates the Phase I direct and the reanalyzed Phase II indirect asbestos structure results. Some of the largest indirect concentrations (above 1 million structures per cubic meter) correspond to very low or non-detectable direct concentrations. Conversely, the highest direct concentrations do not correspnd to high indirect concentrations. Thus, it is impossible to correct the Phase I direct asbestos data base to obtain equivalent indirect concentrations.

Table 5.15 indicates that asbestos PCM equivalent structures (PCMe, or those structures longer than 5 microns which would have been detected as fibers using the PCM method) are also significantly higher when measured via the indirect method. Of the 24 samples, only three showed detectable PCMe structures in Phase I (direct method), while 23 of these samples contained detectable PCMe structures when analyzed by the indirect method in Phase II. Clearly, the indirect method is much more sensitive than the direct method for detecting PCMe structures.

5.3.3.2 Comparison Between Phase I and Phase II Direct Analyses

Three samples were reanalyzed via the direct method. This reanalysis was performed to determine if the original direct analyses are reproducible and to examine interlaboratory variability. The reanalyzed asbestos structure concentrations are graphically compared to the original concentrations in Figure 5.17. Although only three samples were reanalyzed, there is reasonable correlation between the original and reanalyzed direct asbestos structure concentrations. It is reasonable to conclude that the Phase I direct analyses did not grossly over- or underestimate the asbestos structure concentration that can be determined by the direct method. As discussed in Section 5.3.1, however, the direct analyses were hindered by the presence of thousands of small calcium sulfate particles which may have

obscured asbestos fibers. Thus, although these two sets of direct measurements are roughly comparable, direct analyses may not provide a representative measure of ambient asbestos concentration at this site.

The original or Phase I direct PCMe results for the three samples indicated that two of the three samples contained no detectable PCMe fibers (termed structures in Phase II) while the third sample contained 8,000 structures/m^3 (Table 5.15). The Phase II direct PCMe results for these three samples show structures in all samples. The Phase II direct PCMe results appear more sensitive and precise than the original results since low PCMe concentrations (10 structures/m^3) are reported in the duplicate samples whereas the original analyses did not quantify concentrations less than 1000 structures/m^3. Based on this information, the reanalyzed direct PCMe analyses are believed to be superior to the original PCMe analyses.

5.3.3.3 Qualitative Assessment of Indirect Analyses

PCMe and total asbestos structure concentrations for the 24 Phase II indirect preparation samples can be compared on a scatterplot (Figure 5-18). This scatterplot indicates that PCMe and total structure concentrations are positively correlated. This relationship, which was not observed in the Phase I data, indicates that the Phase II data are internally consistent and are more likely to provide representative determinations of asbestos PCMe concentrations than the Phase I analyses.

5.3.3.4 Comparison of Indirect Data with Wind Speed and Direction

To investigate the effects of wind direction and velocity on asbestos concentration, plots of these variables against Phase II asbestos concentrations were examined.

Wind Speed - Average wind speed over the sampling period was plotted against total asbestos structure concentration in Figure 5-19. This plot indicates two potentially distinct asbestos populations. At concentrations below 600,000 structures/m^3 an apparent linear relationship between wind speed and asbestos concentration is shown. At asbestos concentrations

exceeding 600,000 structures/m^3, no relationship between wind speed and concentration is apparent, indicating that large asbestos concentrations can occur at any wind speed. An interpretation of this relationship between wind speed and asbestos structure concentration is that wind is only one of several possible variables which influence the asbestos concentrations.

Asbestos PCMe concentrations (Figure 5-20) do not exhibit any strong relationship with wind speed. Thus wind speed does not appear to be useful for predicting PCMe concentrations.

Wind Direction - To analyze the impact of wind direction on concentration, four approximately equal percentage wind classes were developed based on predominant wind frequencies. The four classes defined and the frequency at which wind directions fall within these classes are follows:

CLASS DEFINITION

Class	Compass Azimuth	Direction	Frequency
1	304-326	NW	30%
2	236-304	WNW - SW	20%
3	326-34	NNW - NNE	31%
4	34-236	NE - SSW	19%

During the collection of each asbestos sample, wind direction was continuously recorded. Based on this continuous record the average hourly wind direction was determined. The hourly wind directions were then used to calculate the percentage of time that wind direction occurred within one of the four direction classes. For example, if the wind direction was from the northwest for 6 hours during a 24 hour sampling period then the frequency of Class 1 winds is 25% (6/24). The frequency of winds for other classes was similarly defined.

Application of the multiple regression method showed that wind direction cannot be used as a predictor of asbestos concentration. Strong positive correlations ($r > .7$) between wind direction class frequency and asbestos

structure or PCM equivalant concentrations do not occur at any of the stations. Furthermore, the predictive power of the multiple regression equations, as measured by the multiple coefficient of determination is poor ($r^2 < .4$) at all three stations. Asbestos soil sampling results indicate discrete asbestos sources exist; however, the wind direction data do not confirm that wind transport from the sources toward the air monitoring stations is a major factor in describing observed asbestos concentrations.

5.3.3.5 Comparison of Indirect Data and Particulate Concentrations

The Phase II asbestos concentrations measured at each location were compared to the particulate concentration measured at the same location over an identical sampling period. Investigation of the relationship between asbestos and particulate was undertaken to indicate if particulate concentration measurements can serve as a screening tool to predict asbestos concentrations. Also, the strength of the correlation between particulate and asbestos concentrations indicates whether asbestos and particulates are transported by identical or different combinations of wind speed and direction, and whether distinct sources of asbestos and particulates exist.

Basic Statistics - Basic statistics of particulate concentrations corresponding to the indirect samples are provided in Table 5.16. Total suspended particulates (TSP) concentrations are largest at Stations 2 and 3 located within Alviso. Average TSP concentrations at those stations are up to twice as large as at upwind Station 1. Station 4 (located within Alviso) and Station 5 (the downwind station) have average concentrations intermediate to the average concentrations observed at Stations 1, 2, and 3.

PM-10 (or particulates less than 10 microns in aerodynamic diameter) average concentrations behave similarly to TSP concentrations in that the average concentrations at Stations 2 and 3 are larger than at Station 1. However, the magnitude of the difference is not as large. Average PM-10 concentrations at Station 4 and Station 1 are very similar while the PM-10 concentration at Station 5 is lower than the average at Station 1.

These statistics show that the distribution of average particulate concentrations among the stations is generally similar to the distribution of asbestos concentration among the stations. This relationship concerning average concentrations is an indication that particulate concentration may act as a predictor of asbestos.

Asbestos-Particulate Scatterplots - Four different scatterplots of asbestos (both PCMe and total structures) and particulates (both TSP and PM-10) were developed (Figures 5-21 through 5-24). Each of the plots presents an asbestos concentration and a particulate concentration for each re-analyzed sample. Summary correlation statistics for each plot are provided in Table 5.17.

The scatterplot between TSP and total asbestos structures (Figure 5-21) indicates that mild linear correlation exists between these two variables. The correlation (r=.72) is not sufficiently strong to allow accurate prediction of asbestos from particulate concentration; however, the scatterplot indicates that TSP is a qualitative predictor of total asbestos structure concentration. For instance, when TSP exceed 100 $\mu g/m^3$, asbestos structures always exceed 600,000 structures/m^3 and often exceed 1,000,000 structures/m^3.

An important aspect of Figure 5-21 is that the largest particulate and asbestos concentrations are observed at Stations 2, 3, and 4. Relatively high (>600,000 structures/m^3) asbestos levels are observed at Stations 1 (upwind) and 5 (downwind) on only two occasions. These two relatively high asbestos concentrations are also accompanied by relatively high particulate concentrations indicating that the relationship between particulates and asbestos structures is not strongly dependent on location.

The plot of PM-10 against total asbestos structures (Figure 5-22) is similar to the previous plot for TSP. Asbestos and particulates are again related; however, the correlation between PM-10 and asbestos structures (r = .56) is less than the correlation between TSP and asbestos structures (r = .72).

The plot of TSP against asbestos PCMe structures (Figure 5-23) indicates that TSP and PCMes are generally related. PCMe concentrations of three particular samples are much greater than would be expected based solely on TSP concentration. The mechanism which causes the higher PCMe in these three samples is unknown; however, all three of these samples were from stations within Alviso. Thus the mechanism causing high PCMe may be related to the proximity of asbestos source material plus an unpredictable soil disturbance mechanism.

The plot of PM-10 against asbestos PCMe (Figure 5-24) is nearly identical to Figure 5-23. The great majority of the samples contain less than 1500 PCMe structures/m^3 and these samples show a linear relationship with PM-10. Again, however, there are three samples which do not fit the observed linear relationship.

Based on the scatterplots presented, TSP shows a stronger relationship with both asbestos structures and PCMe than does PM-10. Although the relationship between asbestos and TSP concentrations exists at all stations, the correlation is not sufficiently strong to allow accurate prediction of asbestos based on a measurement of particulate concentration. The relationship can, however, be used to qualitatively predict asbestos concentration. That is, if TSP concentration exceeds 100 $\mu g/m^3$, asbestos concentration will very likely exceed 500,000 structures/m^3 and will most likely be in the ranges of 1,000,000 to 1,500,000 structures/m^3. Thus, at this level of accuracy particulate concentrations could potentially be used as an asbestos screening tool.

5.3.3.6 Statistical Analysis of Phase II Data

Due to the large number of variables which might influence asbestos concentrations, the indirect data set is too small to reach any firm conclusions concerning the statistical significance of any concentration differences observed between stations. Thus the statistical analysis was restricted to a presentation of the concentrations observed and basic summary statistics. Confidence interval determination was not attempted

since the assumptions implicit in defining these intervals are not valid and the number of data is too small to allow modification of the basic assumptions (i.e., normality and independence).

The total asbestos structures and PCM equivalent structures observed at each of the five stations are presented in Figures 5-25 and 5-26. Table 5.18 presents numerical calculations of statistics summarizing the information presented in these figures. Asbestos structure concentrations are largest, in average, at Station 3 and smallest at Station 1 (upwind station). The average concentration at all three in-town stations (Stations 2, 3, and 4) are greater than the average concentrations at either Station 1 or Station 5 (downwind station). By combining the concentrations observed at Stations 2, 3, and 4, a single average concentration within town is obtained. This average concentration (71,100 structures/cubic meter) is 3.5 times larger than the average upwind concentration (21,300 structures/cubic meter).

The average asbestos concentrations at each station are influenced by atypically large concentrations which may be due to abnormal weather or soil disturbance conditions. The occurrence of these atypical values causes high variability in the data. This variability is expressed in the large spread between the smallest and largest concentrations observed at each station (Figure 5-25).

The average PCMe concentration at the stations within Alviso (1580 structures/m^3) is six times greater than the average concentration at the upwind station (260 structures/m^3). In addition, the average concentrations at in-town Stations 2 and 3 (2140 and 2040 structures/m^3) are nearly an order of magnitude greater than the average upwind concentration. The distribution of PCMe concentrations (Figure 5-26) indicates that concentrations less than 1500 structures/m^3 are common at all five stations. At the in-town stations, however, concentrations up to 8,500 structures/m^3 occasionally occur.

5.3.3.7 Phase II Activity Experiment Air Data

Activity-specific exposure experiments were conducted at three locations as described in Section 3.2.2. The air data collected during these experiments was rejected during data validation and, as a result, cannot be used to relate soil asbestos to air asbestos concentrations during actual activities.

5.3.4 SUMMARY OF AIR INVESTIGATION RESULTS

The major findings of the Phase I air data analysis are as follows:

- PCM equivalent asbestos structures were infrequently detected.
- Episodic asbestos concentrations were not strongly correlated with wind direction, wind speed, or particulate concentrations.
- Asbestos 24 hour concentrations were not correlated with wind speed or particulate concentrations.
- At large particulate concentrations, asbestos concentrations tend to decrease indicating that particulate matter may obscure asbestos fibers causing an underestimation of asbestos concentrations.
- Asbestos concentrations were not strongly spatially or temporally correlated.
- Statistically significant differences in average total asbestos structure concentration exist between Station 1 and the Stations 2, 3, and 4.
- The average asbestos concentrations observed in air were less than concentrations observed during previous site studies.
- Asbestos structure histograms demonstrate that large asbestos concentrations are more common at the stations within Alviso than either upwind or downwind of Alviso.

The major finding from the analysis of the Phase II air data are as follows:

- No numerical relationship between direct and indirect asbestos structure concentrations can be developed.

- A numerical relationship between direct and indirect asbestos PCMe structures can be developed; however, this relationship is suspect since it is based on a small data set containing only three samples.

- Asbestos structure concentrations measured by the indirect method are up to 3 orders of magnitude greater than concentrations measured by the direct method.

- Asbestos PCMe structure concentrations are quantified much more frequently via the indirect method than the direct method.

- No strong relationship between total asbestos structures or PCMe structures and wind speed or direction is observed.

- Total suspended particulates (TSP) and asbestos structure concentrations are positively correlated. TSP and PCMe concentrations are weakly positively correlated. TSP concentrations can be used to qualitatively predict asbestos concentration.

- PM-10 particulate concentations are weakly correlated with asbestos concentrations.

- The indirect Phase II analyses are apparently not impacted by particulate obscuration of asbestos fibers which probably caused the direct Phase I analyses to underestimate asbestos concentrations.

- Total asbestos structures and PCMe structures are, on average, 3 to 6 times larger within Alviso than at the background stations. The absolute difference in structure concentration between the background stations and the stations within Alviso range from 340,000 to 590,000 structures/m^3 while the difference in PCMe structures ranges from 640 to 1900 structures/m^3.

SECTION 6.0

CONTAMINANT FATE AND TRANSPORT

6.0 CONTAMINANT FATE AND TRANSPORT

As discussed in the previous section, numerous air, soil, and water samples have been analyzed for asbestos and Target Compound List contaminants. In this section, the sample results obtained from the various media will be considered in conjunction with potential routes of migration, contaminant persistence, and contaminant migration to assist in determining important contaminant exposure pathways.

6.1 POTENTIAL ROUTES OF MIGRATION

Potential routes available for asbestos migration at this site are soil to air, soil to surface water, and soil to groundwater. Asbestos was detected in air at levels higher than local background levels (Section 5.0), potentially posing a health threat. Therefore soil to air will be considered an important migration route. Although the surface and ground water sample results also show the presence of asbestos, the salinity of the water prevents its use by humans. Therefore neither surface nor groundwater are viable migration routes for human exposure. The environmental risk assessment (Section 7.0) will examine the impact of asbestos in surface water on the site environment.

Analyses of groundwater, surface water, soil and sediment failed to detect significant concentrations of Target Compound List (TCL) contaminants, with the exception of one well at the Marshland Landfill. Again, since the groundwater is not usuable due to high salinity, groundwater is not an important migration route for TCL compounds and is not considered further.

6.2 CONTAMINANT PERSISTENCE

Asbestos fibers of all types are extremely persistent. They exhibit very limited chemical reactivity but are susceptible to physical breakup into smaller and/or thinner fibers. Although the fibers are not soluble in water, cations may be leached under acidic conditions leaving the fibrous silica structure behind (Choi and Smith 1972). The effect that leaching of cations has on the structural integrity of the crystal is unclear (USEPA

1979). Acid leaching alters the surface properties of chrysotile, but not of amphibole asbestos fibers (Seshan 1983). Complete dissolution or breakup has not been quantified. Lauth and Schurr (1983) noted that, although there are no serpentine formations in the Great Lakes basin, 80% of fibers in Lake Michigan are chrysotile asbestos. Chrysotile, which accounts for 95% of asbestos produced for commercial use, presumably enters the Great Lakes as a result of human activity in the area.

Amphibole fiber concentrations in Lake Superior resulting from mining activity in the western end of the lake are reduced by more than 30% during transport to the eastern end of the lake (USEPA 1979). The long distance transport of intact chrysotile fibers has been noted in California water supplies (McGuire et al. 1983).

Both amphibole and chrysotile fibers can bioaccumulate (Batterman and Cook 1981; Belanger 1986). For example, chrysotile fibers are taken up by the gills of molluscs and move throughout the body to other tissues. Mussels do not slough off fibers from the intestinal lining (Halsband 1974). Except for leaching in body fluid and stomach acid, biotransformation of asbestos fibers has not been observed. In analyzing transport mechanisms, asbestos fibers will be assumed to be non-reactive with soil or other particles entrained in the air.

6.3 CONTAMINANT TRANSPORT

The physical processes by which mass (solid particles) is transferred from soil or other surfaces to the atmosphere and then transferred back to the surface are known as suspension (resuspension) and deposition, respectively. While both mass transfer mechanisms have been the topic of extensive research, site-specific transfer rates are quite difficult to predict accurately, since they depend upon an array of surface and atmospheric variables. Nevertheless, some understanding of resuspension and deposition theory and practical applications is necessary to evaluate potential air to soil and soil to air transport of asbestos in and near the South Bay site. Each of these mechanisms are discussed in some detail in the following sections.

6.3.1 SOIL TO AIR TRANSPORT (PARTICLE RESUSPENSION)

Resuspension of particles from surface soil or other surfaces (e.g. roadways, vegetated areas, etc. occurs both as a result of shearing forces caused by wind flow over the surface, and by mechanical disturbance of the surface. The importance of each varies greatly with the physical characteristics of the site, local meteorological factors, and associated human activities such as vehicular traffic, excavation, walking and bicycling. The potential for resuspension due to both wind forces and mechanical disturbances are discussed below.

6.3.1.1 Wind Erosion

Many investigations (including Sehmel, 1984; Chepil, 1951, and others) have shown that resuspension of particles due to wind erosion is a complex process dependent upon many physical variables. While mathematical expressions have been derived for resuspension of respirable, non-respirable and bulk soil loss media, it is clear that the rates of resuspension are generally dependent upon the following variables or soil characteristics:

- The individual sizes and size distribution (material texture) of surface soil or contaminant particles;
- Surface soil moisture levels which contribute to cementation or cohesion of individual particles or surface crusting;
- The existence of non-erodible elements and surface roughness, and
- The frequency of mechanical disturbances which result in the exposure of new, erodible materials.

The speed of the wind across the soil surface and the resultant shearing forces on the soil particles also plays an important part in wind erosion potential. Various investigations have shown that wind erosion potential varies with the cube of the wind speed above the speed threshold necessary to initiate resuspension. This threshold speed has been shown to vary from 4-8 meters per second (9-18 mph) depending upon particle size distributions

and surface moisture content. Others have assumed that over climatological time frames, erosion generally occurs at or above 12 mph (Woodruff and Siddoway, 1965).

In an effort to develop a predictive tool for estimating emissions from various surfaces under varying conditions, EPA and others have performed extensive field tests to develop comprehensive "emission factors". In these tests and analyses, empirical sampling data are compared to theoretical predictions with the intent to improve predictive capabilities. EPA (1985a, 1987b) has recently sponsored efforts to develop improved methods specific to assessing erosion potential from surface contamination sites.

6.3.1.2 Mechanical Disturbances

Extensive research and field studies have also been performed in efforts to develop "emission factors" for dust generation from human activities on varying surfaces (EPA, 1985b, EPA 1978). As examples, emission factors have been developed for light duty and heavy vehicles for both paved and unpaved road surfaces, for equipment-specific aspects of mining and excavation activities such as bulldozing, grading and blasting, and for operations associated with agricultural tilling.

In all cases, however, assumptions are made as to the aerodynamic diameters of the particles, assuming they are all spherical in shape. Resuspension potential, transport and deposition characteristics are each dependent upon the size, density and shape of the particle. Unfortunately for the specific case of asbestos fibers, little has been learned about how these characteristics vary for particles that are needle-like and obviously non-spherical in nature (Sehmel, 1984). This lack of basic understanding about the resuspension, transport and deposition characteristics of asbestiform fibers and fiber bundles makes more difficult the analysis of asbestos concentrations, meteorological factors, and resuspension potential on the South Bay site.

6.3.2 AIR TO SOIL (PARTICLE DEPOSITION)

Like the process of resuspension, extensive experiments have been performed in attempts to characterize the physics of particle deposition. However, according to Sehmel (1984), "...the results from these experiments have not been generalized, because of experimental uncertainties and limited data." What is known in general about deposition phenomena is that total deposition rates depend upon particle size and airborne concentration, the aerodynamic roughness length over the depositing surface and the terrain over which deposition occurs. In general, large particles tend to be deposited nearer to their source locations than do smaller particles, with smaller particles therefore having longer residence times in the atmosphere.

Additional variables which affect deposition rates are particle density and shape, wind speed, and the type of depositing surface (e.g., vegetation will likely intercept and retain greater numbers of particles than flat surfaces). Deposition of asbestos within Alviso may be an important phenomena in that it is plausible to believe that asbestos suspended from suspected primary asbestos sources may have been deposited in somewhat random fashion throughout the area. Additional resuspension of asbestos from these deposits may then act as secondary sources of asbestos and locally mask the effects of what were once the primary source regions.

6.3.3 EFFECTS OF RESUSPENSION AND DEPOSITION ON AIRBORNE CONCENTRATION AND EXPOSURE

In Section 6.2.1 it has been established that the rate of resuspension of soil particles should increase with the cube of the wind speed above the wind threshold at which resuspension is initiated. It is also known from simple mass concentration arguments that given a consistant rate of emission of a contaminant, atmospheric concentrations are inversely proportional to wind speed, since concentrations decrease with increasing dilution. The end results of these two competing relationships are that:

- If the sole contributor to ambient asbestos concentrations is wind erosion, one would expect marked increases (roughly proportional to the square of the speed) in ambient asbestos concentrations at speeds above the erosion threshold, as long as the amount of erodible material (e.g. the emission rate) remains constant, and

- At wind speeds below the threshold, wind erosion contributes nothing to the ambient concentration. If ambient loadings are therefore the result of mechanical resuspension or other unspecified sources (e.g. automotive brake linings), local concentrations should decrease with increasing wind speed up to the threshold wind speed, because of dilution.

The analysis of the site ambient asbestos data indicates that ambient asbestos fiber and structure data are not well correlated with wind speed. Some of the highest concentrations occurred with very low wind speeds while other high concentrations were associated with moderate (>15 mph) wind speed events. This result suggests, but does not prove, that within the wind speed ranges observed, wind erosion is not the most significant contributor to airborne asbestos loadings in Alviso. This result also suggests that if local airborne asbestos loadings are indeed a result of site sources, mechanical disturbances and human activities may play more significant roles in contributing to resuspension and local concentrations than wind.

Analysis of total suspended particulate and PM-10 particulate data at Alviso also showed a lack of strong dependence on wind speed. This result further suggests that factors other than wind erosion may be the most significant contributors to airborne loadings of surface materials and associated contaminants.

6.4 SUMMARY

It appears that the occurrence of asbestos in ambient air in is not regularly resuspended and redeposited solely by the action of the wind. As explained in the preceding discussion, such a mechanism would be expected to lead to airborne asbestos concentrations which increase rapidly with increasing wind speed; such a dependence on wind speed was not observed.

As discussed in Section 5.0, on the majority of sampling days asbestos concentrations at the in-town stations were similar to the upwind station. However, on 10 percent of the sampling days, the in-town concentrations are clearly higher than upwind. No pattern in the high concentrations was observed, related to weather or time of sampling. The results suggest that under some conditions, higher concentrations are observed within Alviso than upwind of Alviso. The suggestive evidence that particulates are roughly proportional to asbestos (Section 5.0) implies that short-term soil disturbances within the community are responsible for elevated asbestos in air.

While limitations in data quality, the variability in analysis methods, and the absence of data from high wind speeds make firm conclusions impossible, the results suggest that:

- Wind erosion of contaminated soil is not a major contributor to high airborne concentrations;
- Sources of asbestos are variable and dispersed through the town of Alviso, rather than concentrated in a few highly localized areas;
- Asbestos concentrations occur throughout the area and are roughly correlated with levels of general particulate pollution;
- Occasionally, much higher asbestos concentrations occur within Alviso; the cause or location of these sources cannot be defined from the available air monitoring data. Because of their localized and short-term character, it is plausible that these concentrations are related to local human activities, vehicle traffic, mechanical soil disturbance, or similar activities.

SECTION 7.0

RISK ASSESSMENT

7.0 RISK ASSESSMENT

7.1 INTRODUCTION

This Risk Assessment addresses the potential human health and environmental impacts associated with the site under the no-action alternative, that is, in the absence of remedial (corrective) action. Evaluation of the no-action alternative is required under Section 300.68(f)(v) of the National Contingency Plan (NCP).

The widespread presence of asbestos at the South Bay Asbestos site may be of concern to human health and the environment. The purpose of this Risk Assessment is, therefore, to evaluate the potential for adverse effects to humans and the environment at the South Bay Asbestos site. The reader should, however, be aware that the exposures to asbestos treated in this report are of both a continuous and an episodic nature. For example, asbestos may become entrained in air when some soil-disturbing activities are undertaken (e.g., a child playing) and may result in intermittent episodic inhalation exposures. During these intermittent activities, direct soil contact may also occur resulting in incidental ingestion of asbestos-contaminated soil. However, the overall probability of episodic exposure is rather low compared to daily inhalation exposure to ambient levels of asbestos in air.

The risk assessment is organized as follows: Section 7.2 summarizes the RI sampling results by environmental media to identify site-related chemicals that will be evaluated in this analysis. In Section 7.3, Exposure Assessment, the potential human exposure pathways of greatest concern will be identified for detailed analysis. The exposure point concentrations for these pathways will also be presented. Section 7.4 presents the Hazard Identification in which the potential health effects associated with exposure to asbestos are discussed, along with the quantitative health criteria used in the risk assessment. In Section 7.5, Risk Characterization, concentrations of chemicals in sampled environmental media will be compared with Applicable or Relevant and Appropriate Requirements (ARARs). In addition, the potential exposures through the

identified pathways will be quantified where possible, and the estimated exposure levels will be combined with the health criteria presented in Section 7.4 to estimate potential risks. Section 7.6 will present an ecological risk assessment for the South Bay Asbestos site. Section 7.7 will discuss the uncertainties associated with the risk assessment. Conclusions to the risk assessment are presented in Section 7.8. Two Appendices are also associated with this risk assessment. Appendix K is a detailed discussion of the human health and environmental impacts of exposure to environmental levels of asbestos.

7.2 IDENTIFICATION OF SITE RELATED CHEMICALS

Earlier sections of this report discussed site background information and the results of the investigations in detail. A summary of all site sampling programs is provided in Section 2.0. The results of sampling efforts performed at the South Bay Asbestos site, primarily those for the RI but also some previous analyses (e.g., DHS and WCC soil results and DHS air results), are summarized again in this section to identify chemicals to be evaluated in detail in this risk assessment. Sampling results are presented below by environmental medium (air, soil, surface water/sediment, and groundwater).

Data are summarized for each medium by presenting frequencies of detection, concentration ranges or geometric means and maximum detected values of each chemical. The following guidelines were used in evaluating data:

- To calculate the geometric mean for a medium in which a chemical was positively detected in one or more samples, non-detects were included in the mean by using one-half of each sample-specific detection limit. Where sample-specific detection limits were unavailable, one-half of the EPA's Contract-Required

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[2] Geometric means rather than arithmetic means were calculated because collections of environmental concentrations tend to be log-normally distributed (Dean 1981, Ott 1988). When data follow a long-normal distribution, the geometric mean is a more robust estimate of central tendency than the arithmetic mean.

Quantification Limit (CRQL) was used. This arbitrarily-selected value (one-half) is commonly assigned to non-detects when averaging data for risk assessment purposes, since the actual value can be between zero and a value just below the detection limit (Vollmerhausen and Turnham 1988).

- Sample concentrations of certain inorganic chemicals were compared with those levels considered to be naturally occurring in order to determine if the detected levels were elevated above background. In comparing inorganic results with naturally occurring levels, a straight comparison of site soil results with available regional soil background values was made. In addition, a comparison of site surface water results with available regional surface water qualit was made. Sample levels which were within these background ranges were considered to be present at naturally occurring levels and were not further evaluated. Available background data for inorganic chemicals in soil and surface water are presented in Table 7-1.

- Concentrations reported for duplicate samples at a given sampling point were first averaged by calculating a geometric mean of the sample and its associated duplicate. If this geometric mean was below the sample detection limit, the sample was treated as a non-detect.

- Soil samples collected by Cal/OSHA, WCC, EPA and E&E were analyzed using PLM methods for asbestos (Section 3.3); the detection limit for this method is one area%. Soil samples collected by DHS and CDM were analyzed by PLM using a different sample preparation method developed by Steve Hayward of DHS. This method has a detection limit of 0.01 area% asbestos; however, in the CDM data, no results less than one area% were reported. Therefore, for all data except the DHS data, the value used for non-detected samples was set at 0.5 area%.

- The majority of the data collected prior to the RI was not analyzed using EPA's Contract Laboratory Program and therefore, was not subject to EPA's data validation process. For this reason soil samples from Cal/OSHA, EPA, and WCC, which are used in this assessment, should be considered preliminary.

7.2.1 AIR

Previous Investigations

Numerous air sampling programs have been conducted at the site. Preliminary investigations conducted between 1983 to 1987 documented the presence of asbestos at the site. The results of these investigations are discussed in Section 2.3 and are presented in Appendix D. As part of the

Phase I RI, ambient air samples were collected at five sampling stations from July 27 to October 31, 1987. For the Phase I investigation, a total of 150 24-hour and 70 episodic samples were submitted for Transmission Electron Microscope (TEM) asbestos analysis using the direct method of preparation. The Phase I sampling and analysis methodologies are presented in Sections 3.1 and 3.3. The results of the Phase I data analysis are presented in Section 5.0. Results of the Phase I investigation further confirmed the presence of asbestos at the site. However, the analytical data provided by the Phase I investigation were not of sufficient quality to perform a quantitative risk assessment. It would be inappropriate to estimate exposure point concentrations using the Phase I data for the following reasons: asbestos was detected too infrequently, the potential for blank contamination in some air samples, the possible underestimation of asbestos levels due to the presence of calcium sulfate particles on the filters, and overall uncertainty in data associated with the laboratory analysis.

Phase II Investigation

The Phase II investigation involved the reanalysis of a portion of the samples archived from Phase I. The samples were submitted to a different laboratory for TEM analysis, using the indirect method of prueporation. Both 24-hour average and episodic (4- to 12-hour average) samples were collected. These samples were collected at five sampling stations between July 27 and October 31, 1987. Sampling station 1 was located northeast of the site at the Environmental Education Center, generally upwind of Alviso due to prevailing northwesterly winds. Stations 2-4 were located in Alviso on-site. It should be noted that station 2 was located near two known or suspected sources of asbestos, the truck yards and State Street. Station 5 was located about two miles southeast of Alviso, generally downwind of the site. The location of the sampling stations and the sampling technique are fully described in Section 3.1.1. A summary of the results of the indirect analysis is shown in Table 7-2 in Phase Contract Microscope equivalent (PCMe) structures/m3 concentrations. A PCMe structure is an asbestos structure (a contiguous grouping of asbestos fibers) with an aspect ratio greater than or equal to 3:1, longer than 5 um, and which has a mean diameter between 0.2 um and 3.0 um.

The Phase II indirect data are considered to be most appropriate for use in this risk assessment for the following reasons:

- The counting rules for PCMe structures for the Phase II reanalysis were representative of the concentration of PCM fibers that would have been measured in the original epidemiological studies. The asbestos counting rules used in the original occupational epidemiology studies with PCM were to count all fibers (an elongate particle which has parallel or stepped sides) longer than 5 um in length with an aspect ratio of 3:1 or greater. The resolution of PCM also limits structures counted to those thicker than approximately 0.3 um in diameter);
- The indirect methodology used for determining PCMe structures in the Phase II reanalysis was more precise and sensitive than the direct methodology used in the Phase I analysis; and
- The results and validation procedures of the Phase II indirect data were of sufficient quality to perform a quantitative risk assessment.

Comparison of On- and Off-Site Concentrations

A limitation of these air monitoring results stems from the inability to clearly and consistently differentiate between background ambient asbestos levels and site-related levels of asbestos. The concentrations used to estimate exposures in a site-specific risk assessment should in theory reflect site-specific conditions, not background conditions. Nicholson (1987) has stated, however, that asbestos of the chrysotile variety are ubiquitous in ambient air in relatively uncontaminated areas (exact locations unspecified).

A wide variety of potential sources of asbestos, particularly in certain manufactured products, can yield airborne concentrations of asbestos fibers. For instance, urban areas with heavy traffic may have increased levels of asbestos in ambient air due to asbestos present in the brake linings of vehicles. In addition, serpentine rock, which contains asbestos fibers, is naturally occurring in California and natural degradation of this rock may contribute to ubiquitous airborne levels of asbestos fibers.

Background concentrations of asbestos fibers in ambient air will vary depending on whether measurements are taken in areas with naturally occurring and/or anthropogenic sources. Therefore, the background values used for study of hazardous waste sites containing asbestos must be close enough to the site to have similar geological and urban/rural development characteristics, but must also be at a sufficient distance so that airborne asbestos levels are not influenced by potential site emissions. Data collected at the Environmental Education Center (Station 1) and at various locations in California were used to represent local and state background levels, respectively.

Comparison with Local Background Levels (Station 1) - In accordance with an approach developed by EPA Region IX specifically for this site, a statistical comparison was made between upwind (off-site, station 1) and on-site stations (stations 2, 3, and 4). If such a difference was observed, a quantitative estimate of ambient risk would be made for both on- site and upwind and downwind off-site stations. This comparison was performed using the Phase I data, as presented in section 5.3.

Based on a qualitative analysis of the Phase I data conducted in Section 5.3, it was concluded that the reported total structure concentrations at the in- town stations (Stations 2-4) are somewhat greater than concentrations at the upwind station (Station 1). Statistical evaluation of PCMe f/cm3 was not possible due to the low frequency of detection of asbestos. The results of a statistical paired difference analysis of asbestos structure concentrations (between Stations 1 and 2, 1 and 3, and 1 and 4) in Section 5.3.2.3 suggest a significant difference between the in-town and upwind stations. The conclusion that there is a significant difference between on- and off-site concentrations, however, is uncertain because of the many uncertainties affecting the Phase I data and the limitations of the statistical analyses performed to differentiate between possible on-site and off-site concentrations.

Comparison of Phase II indirect data collected at station 1 (off-site and upwind of the site) and on-site stations 2-4, as presented in Table 7-2, reveal that on-site concentrations of asbestos are much higher than

off-site upwind concentrations. The geometric mean concentration of asbestos on-site (combination of stations 2-4) was over 6 times larger than the geometric mean concentration measured upwind at station 1. Also, the maximum concentration of 8,500 PCMe structures/m3 detected at station 2 is nearly ten times larger than the maximum concentration measured up-wind (it should be noted, however, that station 2 is near known sources of asbestos contamination).

Comparisons with California Background Levels - Table 7-3 summarizes and compares direct preparation measured asbestos levels at the South Bay Asbestos site (Phase I direct TEM analysis) and ambient air samples collected by Science Applications Inc (also using direct TEM analysis) (SAI 1981). The Phase II reanalysis data were based on the indirect method of preparation and thus are not comparable to the direct preparation results shown in Table 7-3.

Science Applications Inc. (SAI 1981) collected a series of ambient air samples at several locations in california. According to the California Air Resource Board and the DHS, the SAI data is the best available data for assessing asbestos contamination levels in the State of California. The ambient air samples were collected in accordance with EPA asbestos sampling and data validation procedures. These results are discussed in detail in a California Air Resources Board report (CARB, 1986). Four 4-hour samples were collected at each location on a single day. Each location was characterized by the type of potential asbestos source, including urban, non-urban, industrial, and natural deposits. Urban areas were characterized as those with large populations and high concentrations of particulate matter. Non-urban areas were located away from urban centers and away from natural deposit areas. Industrial areas were characterized as those with asbestos users in industry clusters. As shown in Table 7-3, the TEM total fiber concentrations measured at these California locations were generally greater than those measured at the site, although the maximum concentrations measured were within the lower range of values observed at the other California sites. The reader should keep in mind, however, the uncertainties inherent in the Phase I direct data (see Sections 5 and 7.2.1 as described previously). Although the Phase I direct

data are not considered adequate for use in a quantitative risk assessment, they can be compared qualitatively with other similar types of measurements.

7.2.2 SOIL

As noted previously, both surface and subsurface soil samples were collected for the RI/FS within the South Bay Asbestos site area. Previous data were collected by Cal/OSHA, DHS, EPA, the City of San Jose and their respective contractors. Details concerning these soil sampling efforts and the subsequent results are found in Sections 2.0, 3.0 and 5.1 of this report and in Appendices B through I, soil sample results. All soil samples were analyzed by PLM; the standard asbestos analytical protocol has a detection limit of one area percent (1 area %). The DHS samples used a different preparation method than the standard protocol and had a reported PLM detection limit of 0.01 area %. Surface soil samples were analyzed only for asbestos; subsurface samples collected from former landfill areas were analyzed for volatile and semivolatile organics, pesticides, metals and cyanides as well as asbestos by PLM and TEM. Asbestos is considered to be the most toxic and prevalent contaminant present in soils at the site. Therefore, although results of non-asbestos chemical analyses will be discussed, this risk assessment will focus on the potential health impacts of asbestos on exposed individuals.

Table 7-4 presents the surface soil samples asbestos analysis results. These results are summaries of all RI/FS data and previous data combined by area, with the exception of the ring levee. The data collected at the ring levee were not included in this risk assessment because the levee is considered a separate operable unit (EPA, 1988a) and is being remediated by the City of San Jose. DHS and EPA performed area wide sampling in an effort to characterize the site. E&E data exists only for the truckyards. The Cal/OSHA samples were collected from the discovery area which was subsequently excavated then backfilled and, therefore, these data will not be used in this risk assessment. Descriptions of the areas sampled are provided below.

Residential Areas

Two major residential areas have been designated for this risk assessment, the town of Alviso and the Summerset Trailer Park, located to the south of the Guadalupe River (see Table 7-4 for results). Sample data for Alviso were obtained from yards and vacant lots in the area bounded by Taylor Street and the ring levee (at George Mayne School), Spreckles Avenue, State Street and Gold Street. Sample data for the trailer park were obtained from curbside areas not covered by structures or vegetation.

George Mayne School

A total of 20 surface soil samples were collected by EPA in December 1985 at the George Mayne School (see Table 7-4 for results). No other samples have been collected because the school yard was paved by EPA as a result of the concentrations of asbestos detected. The potential for direct contact is considered to be negligible in this area, because the unpaved areas have been covered.

Environmental Education Center

A total of 23 samples were collected at the Environmental Education Center located northeast of the levee and the site (see Table 7-4 for results). The peak concentration of 40 area% was detected in a debris pile which has since been removed by EPA during emergency response. Concentrations representative of soil remaining following the removal are provided by the three RI samples, all of which were nondetects.

Vacant Lots

A total of 52 samples were collected from vacant lots outside the predominantly residential portion of the community (See Table 7-4 for results). Most of the surface soil samples collected in the area of local landfills have been incorporated into this vacant lot category or other relevant categories covered in this soil section.

Truckyards

A total of 39 surface soil samples were collected from local truckyards or immediately adjacent locations located primarily along the north side of State Street (see Table 7-4 for results). There has been considerable reanalysis of the E&E samples from the truckyards, as discussed in Section 2.0. The original analysis revealed only trace amounts of asbestos in 29 of the 52 original samples and no asbestos in the remaining samples. A second round of analysis for five of the samples revealed levels of asbestos ranging from non-detectable to 19 area%. A final reanalysis of 19 of the E&E samples yielded results ranging from non-detectable to 2 area%. The data from the final two analyses rather than the first analysis were conservatively included for use in this risk assessment.

Streets

Unpaved streets at the site were sampled in previous site investigations (see Table 7-4 for results). As a result of the initial EPA sample results from 1985, the unpaved portion of Spreckles Avenue was paved in 1986. All street dust samples were collected from accumulated dirt in gutter areas. All soil samples collected at locations on or adjacent to roads will be used for purposes of this assessment.

Subsurface Soils

As mentioned above, subsurface samples were gathered during the RI. These subsurface samples were collected at the two former landfills and the Marshland Landfill to characterize subsurface conditions. A total of 42 subsurface samples were collected with concentrations of asbestos ranging from non-detectable to 7 area% at a depth of 9-10.5 feet. Twelve subsurface landfill samples were also analyzed for volatile and semivolatile organics, pesticides and PCBs, metals and cyanides (summarized in Table 5-5). Metals detected in subsurface samples include aluminum, arsenic, beryllium, copper, chromium, nickel and zinc. All detected levels of inorganic compounds are lower than the background levels presented in Table 7-1 and are thus not considered to be site-related. Vinyl chloride

(9 ug/kg), 2-butanone (21 ug/kg), 4,4'-DDD (67.6 ug/kg), 4,4'-DDE (126 ug/kg), 4,4'-DDT (129 ug/kg), chloroform (9 ug/kg) and 1,1,1-trichloroethane (4 ug/kg) were each detected only one time in the 12 subsurface soil samples. Because these contaminants were detected very infrequently, it is not clear whether they may or may not be site-related; however, among all the chemicals analyzed for and detected in soil, asbestos is considered to be both the most prevalent and of greatest potential concern to human health.

7.2.3 SURFACE WATER AND SEDIMENTS

During the RI, surface water and sediment samples were collected from the Guadalupe River upstream and downstream of where it passes through the former Santos Landfill. These samples were analyzed for asbestos and volatile and semivolatile organics, pesticides and PCBs, metals and cyanide. Two surface water samples were collected; in addition to asbestos, aluminum, vanadium, potassium, sodium and magnesium were detected. Downstream water concentrations of all inorganic contaminants including asbestos were less than upstream concentrations for all contaminants except aluminum. Upstream and downstream water concentrations of total asbestos fibers were 3,266 million fibers/liter (MF/L) and 2,600 MF/L, respectively. The downstream sample had 50 MF/L of fibers greater than 10 um in length. The aluminum concentration of 73 ug/L is greater than regional background of 10 ug/l and therefore, may be site-related; however, insufficient data exist to evaluate this further. Bis(2-ethylhexyl)phthalate was detected in the downstream surface water sample at a concentration of 16 ug/L; it was not detected in the upstream samples. Bis(2-ethylhexyl)phthalate is a common laboratory contaminant; therefore, this value may be an artifact of the analysis rather than indicative of on-site conditions. In the South Bay area, asbestos is considered to be of greater potential concern to human health than these chemicals, and thus they will not be considered further.

One upstream and three downstream sediment samples were collected. For sediment samples, the downstream concentrations of inorganics did not exceed twice the upstream concentrations and therefore, these contaminants

are not considered to be site-related. No organic contaminants were detected in sediment samples. Asbestos was not detected in upstream sediments and only trace amounts (i.e., <1 area%) were detected in downstream sediments.

7.2.4 GROUNDWATER

As part of the RI, five groundwater monitoring wells were installed in locations near the former landfills. Samples were collected from both these wells and five existing wells at the Marshland Landfill. Two rounds of sampling were performed at the RI wells and one round at the Marshland wells. Samples were analyzed for asbestos as well as the entire target compound list (TCL) chemicals. Groundwater sample results are summarized in Table 7-5.

Chemicals found in groundwater include asbestos, naturally occurring inorganic compounds, as well as a few organic compounds. Most of the organic compounds detected in groundwater samples were also found in rinsate blanks or field blanks and therefore they may be laboratory contaminants rather than indicative of groundwater conditions.

Groundwater in the site area is saline due to intrusion by the Bay. There are no known uses of groundwater in the South Bay Asbestos site area. Based on this information as well as the low concentrations of chemicals detected, CDM, under Phase II of the RI, abandoned the monitoring wells at the request of EPA Region IX. Abandonment was conducted in accordance with Santa Clara County and California Department of Natural Resources sealing requirements.

7.3 EXPOSURE ASSESSMENT

In this section an exposure assessment will be conducted for the site. The complexity of exposure at the South Bay site to asbestos, the only chemical consistently detected across all sampled environmental media and selected for detailed evaluation, requires a series of simplifying steps. In this report, we follow a general approach to exposure assessment which has been

developed by specialists in this field and has been codified in general terms by the EPA (EPA 1986b,c,d,e). In this approach, exposure assessment is conducted in two general steps. The first step is a pathway analysis in which the exposure pathways most likely to be significant are extracted from a list of all possible pathways. This screening process is necessary to focus the assessment on the most significant exposure pathways and is justified by experience, which shows that in most circumstances exposure via a few exposure pathways dominates estimates of potential risks.

The second step is a detailed assessment of the exposures determined to be of potential concern. For each of these exposures, an exposure scenario is developed based on assumptions about the environmental behavior and transport of asbestos, and the extent, frequency, and duration of exposures. These factors are used to predict potential intakes of asbestos for both an average and a maximum plausible exposure case.

7.3.1 POTENTIALLY EXPOSED POPULATIONS

Based on 1980 census information, approximately 1,870 individuals reside in Alviso (Section 4.6 and Aqua-Terra, 1986). The Alviso population is highly mobile; among those residing in Alviso in 1980, only 38% were living in Alviso in 1975. In 1980, approximately 10% of the population were under five years of age and 4.5% were over 65 years of age. Thirty-two percent of the population in 1980 was between five and 20 years of age (Western Economic Research Co. 1980 as cited in Aqua-Terra 1986).

There are several groups of persons who may potentially be exposed to contaminants originating at the South Bay Asbestos site. Since asbestos has been found throughout the community, Alviso residents as well as children who attend the George Mayne School are potentially exposed populations. Residents, especially children, may frequent the non-residential portions of Alviso (e.g., vacant lots). Workers employed at the fifteen unpaved truckyards in the site area may be exposed occupationally, but exposure to workers will not be estimated since the protection of worker health is the responsibility of the Occupational Safety and Health Administration (OSHA), not EPA.

7.3.2 IDENTIFICATION OF EXPOSURE PATHWAYS

The purpose of this section is to identify the most significant potential pathways through which humans may be exposed to asbestos at the site. An exposure pathway is composed of the following four elements (EPA 1986c):

(1) A source and mechanism of chemical release to the environment;

(2) An environmental transport medium (e.g., air) for the released chemical, and/or a mechanism of transfer of the chemical from one medium to another (e.g., deposition of particles onto soil);

(3) A point of potential contact of humans or biota with the contaminated medium (the exposure point); and

(4) An exposure route (e.g., inhalation) at the exposure point.

All four elements must be present for a pathway to be considered complete.

In the risk assessment, exposure pathways are identified for the no-action (existing condition) alternative, that is, assuming no site remediation occurs. This means that no limits on site access or use, such as fences, paving, polymer applications, or soil cover, would be instituted. Under these conditions, it is possible that individuals could repeatedly contact potentially contaminated areas and engage in activities resulting in exposures to asbestos.

There are two general routes through which individuals may be exposed to asbestos at South Bay Asbestos site: inhalation and ingestion. Experimental and epidemiological studies indicate that inhalation exposures to asbestos are of greatest potential concern to human health (see Asbestos Toxicity Profile, Appendix K). While not of primary importance, ingestion exposures to asbestos may also be associated with an increased risk of cancer (see Appendix K). These ingestion exposures include direct ingestion of soil contaminated with asbestos and indirect ingestion of asbestos which has been inhaled. Dermal contact and subsequent absorption is not an exposure route of concern at South Bay Asbestos site since asbestos is not likely to be absorbed through the skin.

Inhalation Pathways

Individuals may inhale asbestos fibers which are present in ambient air and asbestos fibers present due to specific activities which stir up fibers. These pathways are discussed further below.

Inhalation of Asbestos in Ambient Air. Asbestos present in soil may act as a source of asbestos in ambient air as a result of mechanical disturbances (e.g., vehicle traffic on unpaved surfaces, biking on levee, etc.) and/or as a result of wind erosion. Individuals residing in Alviso and nearby areas may thus be exposed to ambient airborne asbestos. As described in Sections 5.3 and 7.2.1, ambient concentrations of asbestos were detected in both on-site and off-site areas by the air monitoring conducted during the RI and by DHS in 1983-1984. Thus, the exposure pathway consisting of inhalation of ambient airborne asbestos is complete and will be evaluated in this risk assessment.

Of the available RI ambient air results, the Phase II indirect data are considered to be most appropriate for use in this risk assessment because the data were analyzed using a more representative and sensitive counting procedure and the results were of sufficient quality (as discussed in Sections 5.3 and 7.2.1). Table 7-2 summarized the PCMe structures/m3 concentrations derived by indirect preparation.

Averaging Time for Air Concentrations. To evaluate risks associated with chronic inhalation of asbestos in ambient air, long-term annual average concentrations should ideally be used as exposure point concentrations. The longest sampling periods for Phase II ambient air were 24-hour averages. Because of the few numbers of Phase II indirect sample results available, however, both 24-hour average and episodic (4- to 12-hour averages) samples were used in this risk assessment to estimate exposure point concentrations. Although there is uncertainty in these estimates, estimated exposure point concentrations represent conservative upper-bound estimates of the actual long-term annual average concentrations because of the inherent bias in the sampling data (i.e., the episodic samples were not

randomly collected, but rather correspond to specific wind conditions; and some samples were collected near known or suspected sources of asbestos contamination [i.e., station 2]).

Conversion of Phase II Indirect Air Concentration Units. The asbestos dose-response relationship available for use in risk assessment is derived for exposures expressed in Phase Contrast Microscopy equivalent (PCMe) fibers/m3 as determined by the direct TEM method. For the Phase II indirect data shown in Table 7-2 to be used in risk assessment, the indirect concentrations must be converted to equivalent direct concentrations.

Such an indirect to direct conversion has been difficult to develop, due to the lack of a consistent relationship between direct and indirect asbestos analyses. The method of sample preparation differs significantly, as well as detection limits and sensitivities. As described in Section 5.3, for example, the Phase I direct asbestos structure concentrations did not correlate with the Phase II indirect structure concentrations. The potential correlation between PCMe fiber concentrations analyzed using Phase I direct and Phase II indirect methodologies could not be analyzed due to the low frequency of PCMe detection in the Phase I direct analysis. A comparison between the three samples analyzed in Phase II using the direct method (a larger area of each filter was examined compared to Phase I) with the three matching indirectly analyzed samples did, however, yield a good correlation for PCMe structure concentrations (r2 = 0.99). This correlation was, however, based on only three data points; for the same three pairs of samples, no correlation was observed for the other reported measures of asbestos concentrations (e.g., PCMe f/cm3) in Phase II.

In conclusion, the available site-specific data that were anticipated to provide a conversion from indirect asbestos concentrations to direct concentrations were considered inadequate for use in this Risk Assessment. Instead, for the purposes of this analysis a range of potential conversion factors were used based on EPA's Environmental Service Assistance Team's (ESAT) preliminary review of information from the scientific literature (ESAT, 1988). ESAT (1988) has suggested the use of a conversion factor

range from 2 to 30 (with both factors being equally possible) based on the limited number of available studies reviewed to date and discussions with several experts in the field (Sebastien, 1986, Chatfield, 1986, Chatfield, 1983, Cook and Markland, 1980, Chatfield, 1988, Baxter, 1988, Chesson, 1988). ESAT (1988) stresses that this conversion factor range is preliminary and may change pending completion of a more comprehensive data review. However, the order of magnitude uncertainty in these factors is unlikely to be higher than the uncertainty associated with other data also used to estimate potential risks. Also, conservative estimates were used to derive this conversion factor range in order to ensure that exposure point concentrations would not be underestimated.

Thus the PCMe structure concentrations from the Phase II indirect reanalysis can be divided by the conversion factors to produce an estimate of the equivalent PCMe direct structure concentrations. These PCMe direct structure concentrations can then be used in the risk assessment based on the assumption that they are most likely to be representative of actual PCM f/m3 (units used in epidemiological studies).

Table 7-6 presents the ambient Phase II indirect and equivalent direct PCMe asbestos structure concentrations using the 2-30 conversion factor range. These average and maximum equivalent direct concentrations will be used as exposure point concentrations for this risk assessment.

Inhalation of Asbestos During Soil-Disturbing Activities

Exposure to ambient asbestos can occur via inhalation of asbestos during soil-disturbing activities in which asbestos is resuspended from soil into the air. Several activities that may be undertaken frequently in the South Bay Asbestos Area include children playing in soil, children and teenagers biking on soil, vehicle traffic in unpaved areas, and adults gardening and landscaping.

Evaluation of this pathway is of importance because concentrations of asbestos generated during some activities can exceed ambient air concentrations by as much as several orders of magnitude. Thus, although

activity-related asbestos exposures are of short duration in comparison to long-term inhalation of contaminated ambient air, activity-related exposures could potentially result in cumulative asbestos exposures of concern to human health. Potential activity-related exposure scenarios for the South Bay Asbestos site are presented below.

Activities in Residential Areas. Some residential yards in the South Bay Asbestos site are partially unvegetated, and children play and ride bicycles in these areas. Children playing in their yards may inhale asbestos which becomes airborne during play activities. Adults in this area may engage in gardening and landscaping activities and may inhale asbestos fibers while doing these activities.

Activities in Unpaved Areas. Other activities that can resuspend significant quantities of asbestos from soil into air involve vehicle traffic on unpaved areas such as truckyards, bike riding on vacant lots and construction activities at sites where asbestos is present in soil. Although there have been unpaved roads on site in the past, all roads are currently paved (with the exception of the O'Neill Tract roads which were found to have no asbestos). The principal unpaved areas with vehicle traffic in the South Bay Asbestos site are the truckyards, which are of particular concern since much onging traffic activity takes place at these locations. Truck traffic may pose a greater risk than automobile traffic due to the heavier weight of trucks and therefore, increased ability to generate dust. The asbestos fibers stirred up by truck traffic may be inhaled by truckyard personnel and by persons living downwind from the truckyard.

Unpaved non-residential areas of the South Bay Asbestos site include truckyards and vacant lots. Soil concentrations of asbestos range from a geometric mean concentration of less than 1 area% in the truckyards and vacant lots to a maximum concentration of 19 area% for truckyards and 10 area% for vacant lots.

Department of Health Services Experiments. In 1985, DHS performed activity-related experiments for four scenarios: a worst-case scenario

where asbestos-contaminated soil was thrown in front of a fan, a vehicle scenario, a playing scenario and a gardening scenario. Few data were collected as part of these experiments and furthermore these data were not validated. As a result, data from the DHS experiments is subject to considerable uncertainty. The DHS results for specific exposure scenarios were not considered for use in this assessment because of the lack of quality assurance. However, the available data from the DHS vehicle experiment will be used in this assessment because of somewhat more rigorous experimental controls. Workers at truckyards and residents living adjacent to the truckyards on State Street and other areas may be exposed to asbestos generated as a result of semi-truck traffic on these yards. The exposure to workers was not quantified, however, because workers are already protected under OSHA.

In the DHS vehicle study, a pick-up truck was driven along an unpaved 200 foot stretch of Spreckles Avenue (now paved) at a speed of approximately 20 miles per hour (Aqua-Terra 1986). Air samples were collected at upwind and downwind locations with samplers placed four feet above ground surface; a sampler was also attached to the rear view mirror. No samples were collected beyond the edge of the road. Soil samples collected during the actual DHS experiment were reported to contain 3-5 area% asbestos using PLM. Based on the limited soil data, the value of 3 area% will be conservatively used to generate the scaling factor of air concentration to soil concentration (ECa/ECs) for vehicle activities. In order to determine the air concentration to be used in deriving this scaling factor, the upwind concentration was subtracted from the downwind concentration prior to calculating the factor. Then this factor was doubled to adjust for the significantly greater weight of the semi-trailer trucks used in Alviso in comparison to the weight of the pickup truck used in the DHS experiment. The resulting ECa/ECs scaling factor of 0.062 was used to calculate air concentrations from asbestos-containing soil during heavy truck traffic.

The vehicle exposure point concentrations are 1.18, 0.62, and 0.19 PCM f/cm3 for residents exposed to asbestos via inhalation originating from the truckyards, roadways, and street dusts, respectively. The DHS air concentration measurements were based on PCM direct analysis and are

presented in units directly comparable to the asbestos dose-response information (i.e., no conversions are necessary). The reader should keep in mind that the exposure point concentrations are probably overestimates of the true exposure concentrations. A conservative approach is used to ensure that the associated risks are not underestimated.

7.3.3 INGESTION PATHWAYS

In addition to inhaling asbestos fibers generated during various activities in yards, children and adults may directly contact and subsequently ingest asbestos-contaminated soil adhering to hands, toys, gardening tools, etc. Inadvertent ingestion of asbestos in soil is most likely to occur in young children, although exposures could possibly occur among adults who engage in activities involving soil contact such as farming or gardening. Children are considered more likely to engage in activities resulting in soil contact than adults. Activities potentially resulting in exposures to on-site asbestos in soil by children include playing in residential and non-residential (vacant lots) and walking to and from school. Since asbestos has been detected in soil throughout the community, in the yards of local residences and trucking firms, adults may also be exposed to asbestos. Therefore, in this risk assessment, asbestos exposures for two populations via incidental soil ingestion will be evaluated: 1) children who play in non-residential portions of the community (i.e., vacant lots), and 2) residents of Alviso and the Summerset Trailer Park.

Areas where soil exposures are most likely to occur under present site use conditions include contaminated soil areas within Alviso, local truckyards and the Summerset Trailer Park. In the future, exposure to potentially contaminated areas on site could increase, if current asbestos containment measures were removed (i.e., road pavement). Use of potentially contaminated areas could enhance erosion, and potentially increase human exposure to asbestos in soil. Thus in the future with no site remediation, both the magnitude of potential exposures and the size of the potentially exposed population via incidental soil ingestion could increase.

The exposure point concentrations to be used to evaluate exposures via soil ingestion were based on surface soil measurements taken in both on-site and off-site areas where exposures may potentially occur (see Table 7-4). The surface soil concentrations used to evaluate this pathway have been reported in PLM percent by area units. For the purposes of providing an indication of potential risks associated with ingestion of this chemical from soil, it was assumed that the percent asbestos soil concentrations by area equaled their percent concentrations on a mass basis. The RI data show that PLM and TEM soil percentages differ somewhat but are comparable (see Section 5.1).

Surface water ingestion is not considered to be a complete pathway because individuals are not known to engage in recreational or play activities in the Guadalupe River.

7.3.4 SUMMARY OF EXPOSURES TO BE QUANTITATIVELY EVALUATED

Based on the discussion above, the following exposure pathways will be evaluated in detail in this risk assessment. These pathways were selected based on their likelihood and frequency of occurrence, and on their potential for generating asbestos levels in air that may be of concern to human health.

- Lifetime exposure of residents by inhalation of ambient air;
- Lifetime exposure of residents by inhalation of airborne asbestos generated by trucks in unpaved truckyards;
- Exposure of children by direct contact with and subsequent incidental ingestion of asbestos-contaminated soil in non-residential areas (i.e., vacant lots); and
- Lifetime exposure of residents by incidental ingestion of asbestos-contaminated soil during outdoor activities.

7.4 HAZARD IDENTIFICATION

In this section of the South Bay risk assessment, the potential human health effects of asbestos (the only chemical selected for evaluation based

on review of chemical concentrations in various media [Section 7.2] and on evaluation of exposure pathways [Section 7.3]) will be reviewed, and the available toxicity criteria that can be used in a quantitative risk assessment will be presented. The section begins with a general discussion of how chemicals are classified for purposes of a risk assessment.

7.4.1 CLASSIFICATION OF HUMAN HEALTH EFFECTS

For hazard identification purposes in a risk assessment, individual chemicals are separated into two categories of toxicity depending on whether they exhibit noncarcinogenic or carcinogenic effects. For chemicals that exhibit carcinogenic effects, most authorities recognize that one or more molecular events can evoke changes in a single cell or a small number of cells that can lead to tumor formation. This is the non-threshold theory of carcinogenesis which purports that any level of exposure to a carcinogen can result in some finite possibility of generating the disease. The hypothesis that no threshold exists for carcinogens is not yet proven; however, regulatory agencies generally assume the no-threshold hypothesis for carcinogens unless sufficient data on the chemical's mechanism of action are available.

EPA's Carcinogen Assessment Group (CAG) has developed cancer potency factors for estimating the upper bound excess lifetime cancer risks associated with various levels of lifetime exposure to potential human carcinogens. Upper bound is a term EPA uses to indicate that risks estimated using these cancer potency factors, which are very conservative, are unlikely to be underestimated but may very well be overestimated. The cancer potency factor is usually expressed in units of mg chemical per kg body weight per day or $(mg/kg/day)^{-1}$. For asbestos, unit risk factors expressed as liters of water/fiber or ml of air/fiber may also be used. In practice, cancer potency factors are derived from the results of human epidemiological studies or chronic animal bioassays. Dose-response data derived from human epidemiological studies are fitted to dose-time-response curves on an individual basis. The data from animal studies are fitted to the linearized multistage model and a dose-response curve is obtained. The slope of the dose-response curve is subjected to various adjustments and an

interspecies scaling factor is applied to derive the cancer potency factor for humans likely to be exposed at lower doses than the animals. These models provide rough, but plausible, estimates of the upper limits on lifetime risk. By using very conservative assumptions in deriving these cancer potency factors, the actual risk is unlikely to be higher than the estimated risk but could be considerably lower.

In addition, there are varying degrees of confidence in the weight of evidence for carcinogenicity of a given chemical. EPA (1986b) has proposed a system for characterizing the overall weight of evidence for a chemical's carcinogenicity based on the availability of animal, human, and other supportive data. The weight-of-evidence classification is an attempt to determine the likelihood that an agent is a human carcinogen and thus affects the estimation of potential health risks. Three major steps are followed in characterizing the overall weight of evidence of carcinogenicity: (1) the quality of the evidence from human studies and (2) the quality of evidence from animal studies which are combined into a characterization of the overall weight of evidence of human carcinogenicity, and then (3) other supportive information which is assessed to determine whether the overall weight of evidence should be modified. EPA's classification of the overall weight of evidence has five categories:

Group A—Human Carcinogen

> This category indicates that there is sufficient evidence from epidemiological studies to support a causal association between an agent and cancer.

Group B—Probable Human Carcinogen

> This category generally indicates that there is at least limited evidence from epidemiological studies of carcinogenicity to humans (Group B1) or that, in the absence of adequate data on humans, there is sufficient evidence of carcinogenicity in animals (Group B2).

Group C—Possible Human Carcinogen

> This category indicates that there is limited evidence of carcinogenicity in animals in the absence of data on humans.

Group D--Not Classified

This category indicates that the evidence of carcinogenicity in animals in inadequate.

Group E--No Evidence of Carcinogenicity to Humans

This category indicates that there is no evidence for carcinogenicity in at least two adequate animal tests in different species or in both epidemiological and animal studies.

7.4.2 RANGE OF POTENTIAL HEALTH EFFECTS FOR ASBESTOS

The available health criteria for asbestos (cancer potency factors) are included in the summary of potential health effects associated with exposure to asbestos presented below. See Appendix K for an indepth discussion of the health effects for asbestos.

Deposition and absorption of asbestos fibers in the body can be influenced by fiber characteristics such as fiber length, fiber diameter, aspect ratio (ratio of length to diameter), fiber number, stability of fibers in the body, surface chemistry of the fiber, interactions between fibers and other surfaces, fiber translocation and migration, overall fiber dose, and fiber type (Schneiderman et al. 1981). Specific data relating individual asbestos type and physical characteristics of the fiber with biological activity via ingestion are lacking. Following inhalation, there is some evidence to suggest a relationship between asbestos fiber dimension and carcinogenic potential. This is known as the Stanton Hypothesis and is based on correlations between pleural sarcomas in rats and dimensions of fibers in addition to human epidemiologic data (Stanton et al. 1981). Long, thin fibers (> 5 microns in length, aspect ratio >3) appear to elicit the greatest biological response. However, a critical fiber length below which there would be no carcinogenic activity has not been demonstrated. Fibers less than 5 microns in length appear to be capable of producing mesothelioma (OSHA 1986, EPA 1986a), and the results of one analysis show that carcinogenicity appears to be a continuously increasing function of the aspect ratio (Bertrand and Pezerat 1980). A reanalysis of Stanton's original data (Wylie et al. 1988) concludes that factors other than size

and shape may play a role in asbestos carcinogenicity. Therefore, for purposes of this risk assessment, all asbestos fibers will be considered to be carcinogenic, although direct preparation total TEM fiber counts (where available) will be converted to PCM equivalents for purposes of using human health data derived from epidemiologic studies in which exposure was measured by PCM.

The primary noncarcinogenic health effect of asbestos is asbestosis, a chronic lung disease associated with functional disabilities and early mortality; however, development of asbestosis is usually associated only with high-level occupational exposure (EPA 1986a). Recent work by Morton Lippmann (1988) correlated asbestos to total surface area of retained asbestos fibers with a length of 2 um and a diameter of 0.15 um. Asbestosis was not considered in this risk assessment, because of the low-level environmental exposure at this site.

The carcinogenicity of asbestos following ingestion has not been conclusively demonstrated by direct studies. In an NTP (1984) bioassay in male rats, a significant increase in benign epithelial neoplasms in the large intestine was interpreted as limited evidence that orally ingested chrysotile fibers may be carcinogenic (EPA 1985c). Available data from occupational studies also suggest a link between inhalation and subsequent ingestion of asbestos and gastrointestinal cancer (EPA 1986a). Inhalation exposure in humans and experimental animals can result in both lung cancer and mesothelioma (EPA 1986a). Both lung cancer and mesothelioma in humans were correlated to the number of retained asbestos fibers with a length of 10 um and 5 um and a diameter of 0.15 um and 0.1 um, respectively (Lippman 1988). According to EPA (1986e), asbestos is classified as a human carcinogen: Group A. There is sufficient evidence from epidemiological studies to support a causal association between asbestos exposure and cancer in humans, according to the EPA (1986e).

The calculation of risk for inhalation exposure is primarily based on the methodology set forth in EPA's Airborne Asbestos Health Effects Update (EPA 1986a) and described in detail in Appendix K. This calculation relies on the use of risk tables which give maximum likelihood estimates for

mesothelioma and lung cancer as a function of sex, age at onset of exposure, years of exposure, and ambient atmospheric concentrations. Situations not exactly described by the risk tables are evaluated by linear interpolation among values on the tables. Since the tabulated function is non-linear, for situations when concentrations are substantially higher than those shown on the tables, the resulting risk presented will not exceed the maximum risk shown in the health risk table ($3x10^{-3}$), but will rather be listed as "$>3x10^{-3}$". For situations below the range of the tables (e.g. exposure periods of less than one year, concentrations substantially lower than 10^{-4} to 10^{-6} fiber/cm^3), the linearized unit risk of 2.3186×10^{-1} (fibers/cm^3)$^{-1}$ developed by EPA (1987b) for a lifetime exposure will be used which is also based on an extrapolation of the risk tables in the airborne asbestos Health Effects Update. The risk calculated by this method will be adjusted to compensate for less than lifetime exposure.

EPA (1985c) developed an oral unit risk factor of $1.4x10^{-13}$ (fiber/liter)$^{-1}$ based on the NTP (1984) bioassay in which benign neoplasms were observed in male rats exposed to asbestos (>10 microns in length) in drinking water; this cancer potency factor was used by EPA as the basis for the drinking water maximum contaminant level goal. There are a number of uncertainties associated with this approach including the absence of adequate dose-response data from human populations exposed via ingestion, the induction of benign tumors only, and that the criterion is limited to fibers greater than 10 microns in length. No oral cancer potency factor has been derived for any environmental medium other than drinking water in which asbestos concentration may be reported as fiber mass per unit volume (ug/m^3) rather than fibers per unit volume. The health effects of asbestos are further detailed in the toxicity profile in Appendix K.

7.5 RISK CHARACTERIZATION

In this section, concentrations of asbestos in environmental media will be compared to available asbestos standards or regulations promulgated by State or Federal agencies, known as Applicable and Relevant and Appropriate Requirements (ARARs). In addition, because ARARs are not available for

asbestos in all of the sampled environmental media, a quantitative risk characterization will be conducted. In this quantitative evaluation, estimates of potential exposures through each pathway identified for evaluation will be combined with the toxicity criteria presented in the Hazard Identification section to predict potential risks associated with the South Bay site. For each pathway, an exposure scenario will be developed based on assumptions about the environmental behavior and transport of asbestos, and the extent, frequency, and duration of exposures. These factors are used to predict potential exposures to asbestos for both an average and a maximum plausible exposure case. This risk assessment follows a general approach to estimating exposures and risks which has been developed by specialists in this field and has been codified in general terms by the U.S. Environmental Protection Agency (EPA 1986b,c,d,e).

7.5.1 COMPARISON TO APPLICABLE OR RELEVANT AND APPROPRIATE REQUIREMENTS

Asbestos-specific ARARs were evaluated relative to the South Bay Asbestos site. The EPA's interim guidance on ARARs (EPA 1987d) defines ARARs as follows: Applicable requirements means those cleanup standards, standards of control, or other environmental protection requirements, criteria, or limitations which are promulgated under Federal or State law and specifically address a hazardous substance or other circumstance at a CERCLA site. On the other hand, relevant and appropriate requirements address situations which are sufficiently similar to a CERCLA site that their use is well suited to the particular site or situation.

Federal regulatory action on asbestos has taken on a variety of forms. Regulations have been promulgated by numerous agencies including EPA, the Consumer Product Safety Commission (CPSC), Department of Transportation (DOT), Food and Drug Administration (FDA), Mine Safety and Health Administration (MSHA), and the Occupational Safety and Health Administration (OSHA). Although none of the promulgated regulations may be applicable to the South Bay site, they may be relevant and appropriate.

In 1980, the National Institute for Occupational Safety and Health (NIOSH) recommended to OSHA a maximum level for asbestos in the workplace in addition to several measures which would act to minimize exposure. Recently, OSHA issued a rule implementing many of these regulations and lowering the old workplace standard of 2 f/cm^3 to 0.2 f/cm^3 of air as an eight-hour time weighted average (51 CFR 22612, 1986). The MSHA has prescribed exposure levels for asbestos dust for workers in domestic mines of 2 f/cm^3 (43 CFR 54064, 1978).

The CPSC has banned some materials from household use on the basis of findings that they contained respirable asbestos (16 CFR Parts 1304 and 1305). Additionally, CPSC imposed labeling requirements on household products which contain asbestos and which are likely to release fibers under any reasonably forseeable conditions of handling (51 CFR 33911, 1986).

EPA has undertaken regulatory action on asbestos under the Clean Air Act, Clean Water Act, Safe Drinking Water Act, and the Toxic Substances Control Act. Asbestos was first designated as a hazardous air pollutant under the Clean Air Act in 1971. Since its initial promulgation in 1973, the National Emissions Standards for Hazardous Air Pollutants (NESHAP) for asbestos has been revised several times. As they currently read, the regulations call for no visible emissions from milling, manufacturing, and asbestos waste disposal activities (43 CFR 26372, 1977) and require that asbestos-containing waste be kept thoroughly wet with water during handling. The standard of no visible emissions may be relevant and appropriate to the asbestos soils and levee at South Bay.

Asbestos was first determined to be a hazardous water pollutant in 1973. Effluent limitation guidelines for asbestos manufacturing have been promulgated (40 CFR 1874, 1975). Also under the Clean Water Act, EPA published an Ambient Water Quality Criteria document for asbestos (EPA 1980). The document noted that the database was inadequate to issue criteria for the protection of aquatic life. For protection of human health, the estimated levels of asbestos in water which would result in increased lifetime cancer risks of 10^{-5}, 10^{-6}, and 10^{-7} are 300,000 total

fibers/liter, 30,000 total fibers/liter, and 3,000 total fibers/liter respectively. Estimates for risk associated with consumption of aquatic organisms only could not be made. EPA has also proposed an MCLG for asbestos in drinking water under the Safe Drinking Water Act which equated an excess cancer risk of 10^{-6} to an asbestos concentration of 7.1×10^{6} fibers greater than 10 um in length per liter (EPA 1985c).

EPA has promulgated several rules under the Toxic Substances Control Act (TSCA) to regulate asbestos in the environment. The most significant of these is the Asbestos Hazard Emergency Response Act (AHERA) which enacted Title II of TSCA. Under this act, EPA has published regulations related to inspection and management of friable asbestos in schools (52 CFR 42826, 1987). The monitoring procedures specified in this regulation may be relevant to hazardous waste sites. In the rule, local agencies must consider an area to contain asbestos if asbestos fibers are found in any sample at greater than one percent as analyzed by PLM. Since one percent is generally accepted as the detection limit for asbestos in soil, this could represent a standard for non-detectable asbestos, i.e., that any detectable asbestos in soil warrants action.

Of this group of rules, the NESHAPs "no visible emissions" criterion appears to be relevant to the South Bay sites. Visible emissions have been observed under present conditions in the area of the truckyards and along paved streets due to dust in gutters. Remedial activities resulting in site disturbances are also likely to result in some visible emissions; however, stringent dust controls would be implemented to control the resultant emissions.

The asbestos concentrations measured in the Guadalupe River upstream and downstream of the site exceed both the Ambient Water Quality Criterion for protection of human health and the proposed MCLG, as do the asbestos concentrations measured in groundwater at the site. However, neither the groundwater nor surface water areas are currently or expected to be used as a drinking water source.

7.5.2 QUANTITATIVE RISK ASSESSMENT

Inhalation of Ambient Levels of Airborne Asbestos

As discussed in Section 7.3, the risks associated with lifetime daily inhalation of ambient airborne asbestos will be evaluated for the South Bay Asbestos site area. The exposure point concentrations for this pathway, presented in Table 7-6, are equivalent direct concentrations in PCMe structures/m^3 based on the Phase II indirect reanalysis PCMe structures/m^3. The exposure point concentrations range from 3 PCMe structures/m^3 to 1,700 PCMe structures/m^3, and are considered to be within the general range of air concentrations covered in the EPA cancer risk table (Appendix K). Risks were thus estimated by scaling from the risk table concentration of 100 f/m^3 to the exposure point concentrations of asbestos.

Risks are estimated separately for Station 1 (upwind/off-site), Stations 2-4 (on site), and Station 5 (downwind/off-site). For each exposure case (average or maximum), a range of risks is presented to correspond to the possible range of the conversion factor (2-30) used to develop equivalent direct concentrations from indirect concentrations. The resulting mesothelioma and lung cancer risk estimates are presented for males and females in Table 7-7. As shown in this table, the excess lifetime lung cancer risks are roughly an order of magnitude higher for on-site locations than for off-site locations. Excess lifetime mesothelioma risks are slightly less than an order of magnitude higher at on-site versus off-site locations. The highest predicted risks are associated with use of the lowest conversion factor (two) in conjunction with the maximum detected air concentrations. For on-site locations, the cancer risks are in the 10^{-6} to 10^{-4} range. The cancer risks for Station 1 (off-site) are in the 10^{-7} to 10^{-5} range and those for Station 5 (off-site) in the 10^{-7} to 10^{-4} range.

There are, however, many significant uncertainties associated with these risk estimates. Because of these uncertainties, conservative techniques were implemented to assess the risk from asbestos exposure. This conservative approach ensures that the actual risks are not underestimated. A full discussion of the uncertainties associated with these risk estimates is found in Section 7.7.

Comparison with California Background Risks

The asbestos air measurements conducted by SAI (1983) for various California locations as described in Section 7.3.2 can also be used to predict potential lifetime inhalation risks. These risk estimates may provide a context for interpretation of the on- and off-site risk estimates. The measured TEM f/m^3 concentrations for the SAI (1983) data were presented in Table 7-3. Exposure point concentrations were derived from these data by multiplying the reported TEM direct fiber concentrations (f/m^3) by a conversion factor (NRC 1983) of 0.017 PCM f/m^3 per 1 TEM f/m^3 (see Appendix K). The estimated direct PCM f/m^3 exposure point concentrations, as shown in Table 7-8, range from 88 to 1,768 PCM f/m^3, within the general range of air concentrations covered in the EPA cancer risk table (Appendix K). As with the RI site data, risks were estimated by scaling from the risk table concentrations.

Table 7-9 presents the estimated excess lifetime mesothelioma and lung cancer risks associated with lifetime exposure to airborne asbestos at various locations in California. The excess lifetime mesothelioma risks are in the 10^{-5} risk range for the average case and in the 10^{-5} to 10^{-4} risk range for the maximum case. These risks are lower than on-site risks which are estimated to be in the range of 10^{-6} to 10^{-4} for the average case and 10^{-5} to 10^{-3} for the maximum case, as presented in Table 7-7. The excess lifetime lung cancer risks for locations in California are in the 10^{-6} to 10^{-5} range for the average case, and the 10^{-5} to 10^{-4} range for the maximum case. These risks are similar to the on-site risks for lung cancer presented in Table 7-7. There are important uncertainties associated with the background risk estimates using the SAI data as well and thus they too should be interpreted with caution. For example, the conversion factor from TEM f/m^3 to PCMe f/m^3 is uncertain as is the use of only four sample measurements per location (all collected on the same day) to characterize average and maximum case long-term exposure point concentrations.

Inhalation of Airborne Asbestos Generated During Various Activities

As discussed in Section 7.3.2, exposure via inhalation can occur both through ambient exposure to airborne asbestos and exposure through the generation of airborne asbestos via soil-disturbing activities. Soil disturbing activities which include children playing and adults gardening will be discussed qualitatively because of the lack of data needed to assess the actual risk from exposure to asbestos. Residential exposures to asbestos generated as a result of truck traffic on unpaved roads and truckyards of the community will be discussed quantitatively.

Exposure to Activity Generated Dust. Areas where children are expected to play include residential yards and non-residential areas (vacant lots and truckyards). Residents in Alviso and the Summerset Trailer Park may also be exposed to asbestos generated during gardening. For individuals who may repeatedly engage in these activities, cumulative asbestos exposures of concern to human health could potentially result. However, data of sufficient quality is unavailable to actually quantify these risks.

Exposure to Vehicle Generated Dust. Residents living adjacent to the truckyards on State Street and other areas may be exposed to asbestos generated as a result of semitruck traffic on these yards. Data from the DHS experiments were used to calculate exposures for these scenarios. Data from this experiment were collected along the road next to where the truck was driving; no samples were collected further from the road in order to determine dilution in air. Therefore, the use of concentrations generated by this experiment for nearby resident may overestimate exposures.

The residential exposure parameters were developed based on information available in the literature or based on site-specific information; these exposure assumptions are presented in Table 7-10. The frequency and duration of exposure estimates were assumed values designed to approximately reflect the average and plausible maximum exposure scenarios for this site.

Risks for the maximum case were calculated using the asbestos risk table; no average case risks were calculated. All calculated risks exceeded the maximum risk value of $3x10^{-3}$ presented in the Asbestos Health Risk table (Appendix K). Therefore, the risks estimates would be outside the linear range represented by the table and are reported in Table 7-11 as ">$3x10^{-3}$."

Ingestion of Soil

As mentioned earlier, two types of general soil exposure scenarios will be evaluated in this risk assessment. Lifetime exposures to local residents and intermittent exposures to children and trespassers, using the South Bay Asbestos Area will be considered. Chronic daily intakes (CDIs) as a result of incidental soil ingestion for these exposure groups can be estimated using the equation:

$$CDI = \frac{(C_s)(I_s)(F)(YR)}{(BW)(100)(25,550)}$$

where

CDI	=	Chronic daily intake (mg/kg/day),
C_s	=	Asbestos soil concentration (weight %),
I_s	=	Soil ingestion rate (mg/day),
F	=	Frequency of exposure (days/year),
YR	=	Years of exposure (years),
BW	=	Average body weight over period of exposure (kg),
100	=	Conversion factor (from percent concentration to mg/mg), and
25,550	=	Number of days in a 70-year lifetime.

Table 7-12 lists the values used in this equation for several scenarios: 1) intermittent exposures to children and playing on non-residential areas (i.e., vacant lots), and 2) local lifetime residents exposed in yards (e.g., a person potentially exposed to asbestos as a child and then as an adult).

The parameter values listed in this table were based on information available in the literature or based on site-specific information, where possible. For all exposure scenarios except the worker scenario,

time-weighted averages for the amount of soil ingested per exposure event (I^s) and an individual's body weight (BW) were calculated and used to quantitatively evaluate intakes over the exposure period. Incidental soil ingestion rates are based on a review of the literature of soil ingestion in children as presented in Lagoy (1987). Body weight estimates were based on age-specific data presented in EPA (1985d).

The frequency (F) and duration of exposure (YR) estimates were assumed values designed to approximately reflect average and plausible maximum exposure scenarios for this site. The asbestos soil concentrations used were those presented in Table 7-4 by location. For trespassers, exposure to soil in non-residential areas (i.e., vacant lots) is assumed to occur from ages 5 to 11 for the average case and from ages 5 to 16 for the plausible maximum case. For local residents, exposures are assumed to occur over a lifetime (e.g., for asbestos potentially present in yards). The CDIs for asbestos are averaged over a 70-year period (25,550 days) according to EPA (1986b) guidelines for carcinogenic risk assessment. Table 7-13 presents the estimated CDIs for the four evaluated exposure scenarios.

Estimating cancer risks for incidental ingestion of asbestos present in soil is complicated because the EPA (1985c) has developed a unit risk factor for exposure to asbestos in surface water [1.4×10^{-13} $(\text{fibers/liter})^{-1}$] only (fibers greater than 10 um in length) and not for exposure to asbestos from other environmental media where concentrations may be reported on a mass (not fiber) basis. This unit risk factor was used as the basis of the Maximum Contaminant Level Goal (MCLG). There are several important uncertainties associated with even this unit risk factor, as discussed in Section 7.4. In order to quantify risks associated with incidental ingestion of asbestos in soil, the EPA unit risk factor was converted into a mass-based potency factor (mg/kg/day)-1 using the following equation:

$$\text{Mass-based cancer potency factor} = \text{unit risk factor}_{kg} \times \frac{\text{day}}{\text{2 liter}} \times \frac{0.129 \times 10^{9} \text{ fiber}}{\text{1 mg}} \times 70$$

$$= 1.4 \times 10^{-13} \times 0.129 \times 10^{9} \times 70 / 2$$
$$= 6.3 \times 10^{-4} \text{ (mg/kg/day)}^{-1}$$

This conversion was done only for the purposes of providing a rough indication of the potential excess lifetime cancer risks associated with direct contact with asbestos in soil and subsequent incidental ingestion at the site. The conversion factor from fibers to mass of asbestos was taken to be 0.129×10^{9} fiber per mg asbestos based on TEM drinking water measurements performed at the Illinois Institute of Technology Research (NTP 1984) in conjunction with development of the MCLG. There is a significant level of uncertainty associated with this conversion as the average fiber count per mass for the site is likely to differ from the average fiber count per mass observed in the bioassay used to derive the MCLG.

The converted asbestos potency factor can be multiplied by the CDI to derive an approximate estimate of the excess lifetime cancer risks associated with each exposure scenario. Table 7-14 presents the cancer risks estimated for each soil exposure scenario evaluated in this risk assessment. Area-specific risks will not be summed. For the maximum case, the risks for individuals contacting soil in non-residential areas (i.e., vacant lots) was 3×10^{-5}. For residents assumed to incidentally ingest soil from a backyard over a lifetime, the excess lifetime cancer risks ranged from 5×10^{-5} (Alviso) to 6×10^{-5} (Summerset Trailer Park) for the maximum case estimates; no average case risks were calculated since the geometric means were less than 1 area%.

Because of the uncertainties inherent in these risk estimates, particularly converting from a fiber-based unit risk factor to a mass-based potency factor and accurate estimation of exposure point concentrations, the uncertainty associated with risks related to exposure to asbestos through this pathway may exceed an order of magnitude. Additional uncertainty is added by the fact that only benign tumors were noted in the bioassay which

is the basis of the potency factor. Uncertainties were, however, compensated for by adopting conservative assumptions in the risk estimates. Consideration of these uncertainties suggests that the soil ingestion risks predicted for residents and trespassers are not likely to be underestimated but also do not vary significantly among exposed individuals within the maximum case scenarios across the specific exposure locations examined in this risk assessment.

7.6 ENVIRONMENTAL ASSESSMENT

The environmental assessment section for the South Bay Asbestos site consists of hazard characterization and risk characterization. The potential receptors were identified and discussed in Section 4.0.

7.6.1 ENVIRONMENTAL HAZARD CHARACTERIZATION

This section consists of an assessment of contaminant exposure pathways and a description of the ecotoxicity of asbestos. Section 6.0 discussed the environmental fate of asbestos in the ecosystem.

Exposure of Wildlife

There are a number of direct pathways by which wildlife can be exposed to asbestos at the South Bay Asbestos site. Direct pathways include contact with or ingestion of contaminated media such as soil, sediment, water, or air. For example, fish and other aquatic organisms found in the Guadalupe River near the site would be exposed to asbestos via direct contact and ingestion of contaminated water and sediments (asbestos was detected in surface water samples of Guadalupe River at concentrations of 3,266 million fibers/liter [MF/L]). Birds such as the belted kingfisher which build their nest in river banks and feed on fish in the river, may be exposed to asbestos directly via contact and ingestion of water, soil, and sediments. Birds which engage in dust bathing activities also increase direct exposure to asbestos via exposure to contaminated soil and inhalation of dust. Mammals such as rabbits and raccoons groom frequently and are likely to ingest contaminated soil while grooming. Mammals and birds may also be

exposed to asbestos via inhalation and ingestion of surface water from the Guadalupe River. Unfortunately, data are not adequate to assess exposure or impacts to terrestrial animals via any of these pathways. Only the exposure and impact of direct contact with contaminated surface water to fish and aquatic organisms can be assessed.

Wildlife can also be exposed to asbestos via indirect pathways. Indirect pathways are those in which an animal consumes other previously contaminated organisms. Aquatic invertebrates have been shown to bioconcentrate asbestos in the field and laboratory exposure studies. Therefore, birds such as ducks, belted kingfishers, and herons and mammals such as racoons and muskrat which prey on aquatic invertebrates may be exposed to significant levels of asbestos. However, the available data are not sufficient to estimate uptake and accumulation of asbestos in the food chain.

Exposure of Plants

Plants may be exposed to asbestos through surface deposition of airborne fibers and through uptake of fibers from contaminated soil and water. Surface water runoff from contaminated areas may percolate into plant root zones, exposing plants to asbestos via root uptake. However, because asbestos phytotoxicity data are limited, and plant uptake values vary greatly from species to species, exposure via this pathway cannot be quantified.

Ecotoxicity

EPA (1980) has concluded that available asbestos toxicity data are insufficient to establish acute and chronic ambient water quality criteria for the protection of aquatic life. However, there are other toxicity parameters available for assessing the acute and chronic toxic effects of asbestos on fish and aquatic life, as described below (see Appendix K, Section 8.0 for a more detailed discussion).

Asbestos is acutely toxic to aquatic organisms only at very high concentrations. Larval Asiatic clams (Corbicula sp.) had significantly higher mortality rates than controls when exposed to chrysotile fiber concentrations between 10^2-10^8 f/liter. Fathead minnows exposed to 10^{12} f/liter for 96 hours or 10^8 f/liter for 30 days did not exhibit increased mortality over controls (Belanger 1986). Japanese medaka (pisces, Oryzias latipes) exposed to 10^{10} f/liter for 60 days experienced 100% morta lity. Stewart and Schurr (1980) reported that maximum mortality of Artemia occurred between 10^7-10^8 f/liter.

Asbestos exposure can result in deleterious impacts on growth, reproduction, physiological equilibrium, and behavioral traits in algae. Exposure to chrysotile fibers at 1-1.5x10^6 f/liter for 48 hours resulted in severe clumping of cells of the algae Cryptamonas erosa (Lauth and Schurr 1983). It was postulated that this would result in loss of mobility and death due to settling out.

Adult and juvenile Asiatic clams exhibited reduced siphoning rate and shell growth when exposed to 10^5 f/liter for 30 days (Belanger 1986). Juvenile clams also had reduced weight gain at 10^4 f/liter in summer temperatures, and at 10^5 f/liter in winter temperatures. Weight gain of juvenile flathead minnows was reduced after 30 days exposure to between 10^6 and 10^8 f/liter. Length was not affected, however (Belanger 1986). Larval medaka showed reduced growth after 14 days exposure to the same levels.

Asbestos exposure also results in tumor formation in fish and clams. Coho salmon developed tumorous swellings in the gills following exposure to 3x10^6 f/liter of chrysotile (Belanger et al. 1986), in addition to neoplastic cell abnormalities in epidermal tissue. Medaka developed epidermal tumors following exposure to 10^{10} f/liter. Mollies (Poecilia formosa) also developed epidermal hypertrophy after 6 months exposure to chrysotile (Woodhead et al. 1983).

No information was found concerning the toxicity of asbestos to terrestrial wildlife species. However, the carcinogenicity of asbestos fibers in mammals is well established through laboratory animal experiments and human epidemiological studies.

No data were found regarding the toxicity of asbestos to terrestrial plant species.

7.6.2 ENVIRONMENTAL RISK CHARACTERIZATION

Characterization of the risks to plant and animal species associated with exposure to asbestos at the South Bay Asbestos site is complicated due to the limited availability of monitoring data and the lack of toxicity threshold data for terrestrial plants and wildlife. The monitoring data that are available must be evaluated with respect to the analytical problems associated with measuring the type and amount of asbestos fibers in environmental media, and the degree to which the concentrations are representative of the temporal variation in levels in the various media.

There is opportunity for exposure of aquatic and terrestrial plant and animal species to asbestos at the site and in the adjacent marshland areas through contact with and ingestion of contaminated environmental media and food items. With the limited data available, aquatic organisms and borrowing animals appear to be at greatest risk at the South Bay Asbestos site and in the adjacent marshland areas.

Aquatic Organisms

The monitored concentration of approximately $3x10^9$ f/liters in surface waster samples from the Guadalupe River exceeds acute and chronic toxicity threshold levels for algae, invertebrates, and juvenile fish in the published literature (see Ecotoxicity section). If the limited monitoring data available are representative of long-term exposure conditions in surface waters at the site, there is a concern for the acute and chronic toxicity of asbestos to aquatic organisms coming into contact with these contaminated waters.

Terrestrial Organisms

The significance of the limited soil monitoring data available cannot be

evaluated due to a lack of toxicity threshold data for terrestrial plants and animals. In addition, exposure data are insufficient for comparison with the available laboratory acute and chronic toxicity data for rats and mice. Plants growing on or near the levee appear to have the highest potential for exposure to asbestos through root uptake from contaminated soils or surface runoff and foliar deposition of airborne fibers from contaminated surface soil. Burrowing animals appear to have the highest exposure potential through both inhalation and ingestion of fibers from soil. The significance of these potential exposures in terms of the acute and chronic toxicity and reproductive success of the exposed organisms is unknown.

Endangered Species

The significance of potential asbestos exposure to the endangered species indigenous to the refuge area adjacent to the site (i.e., salt marsh harvest mouse and clapper rail) is also unknown. These organisms may be adversely affected directly (e.g., through ingestion of asbestos in surface water, contact with and ingestion of soil when preening, grooming, or foraging for food) and through adverse effects on habitats (e.g., pickleweed) and food sources (e.g., reduced populations of prey, ingestion of contaminated prey).

7.7 DISCUSSION OF UNCERTAINTIES

All risk assessments involve the use of assumptions, judgement, and imperfect data to varying degrees, which results in uncertainty in the final estimates of risk. General sources of uncertainty in a risk assessment include:

- Environmental chemistry sampling and analysis;
- Misidentification or failure to be all-inclusive in hazard identification;
- Choice of models and input parameters in exposure assessment and fate and transport modeling;

- Choice of models or evaluation of toxicological data in dose-response quantification; and
- Assumptions concerning exposure scenarios and population distributions.

Uncertainty may be magnified in the assessment through a combination of these sources. In the South Bay risk assessment, there are additional sources of uncertainty associated with asbestos measurement and risk quantification.

In risk assessments in which considerable uncertainty is anticipated, a technique commonly employed to compensate for uncertainty is to bias the assessment in the direction of overestimation of risk. This is often termed a "worst-case" or "conservative" analysis. The net effect of combining numerous conservative assumptions is that the final estimates of risk may be greatly overestimated, however, they will not lead to decisions which will be non- protective of human health and the environment.

Environmental chemistry sampling and analysis error can stem from the error inherent in the procedures, from a failure to take an adequte number of samples to arrive at sufficient areal resolution, from mistakes on the part of the sampler, or from the heterogeneity of the matrix being sampled. One of the most effective ways of minimizing procedural or systematic error is to subject the data to a strict quality control review. Even with all data rigorously quality assured, however, there is still error inherent in all analytical procedures, and it is still not possible to definitively determine if the sample is truly representative of site conditions. As discussed in Sections 2 and 4, accurate measurements of asbestos concentrations in different environmental media are difficult to achieve and as a result, sampling and analysis errors are particularly important sources of potential uncertainty in this risk assessment. There is no routinely accepted standard procedure for measurement of asbestos in soil which could result in considerable uncertainty. The fact that TEM and PLM measurements are generally comparable, however, indicates that this uncertainty is not likely to exceed an order of magnitude.

In order to quantify risks for this risk assessment, asbestos concentrations were converted from one set of units to another. For example, for the ambient inhalation pathway, PCMe structure concentrations reported for the Phase II reanalysis, analyzed using the indirect method, were converted to equivalent direct PCMe structure concentrations using a conversion factor range. The conversion factors are based on a limited set of information (ESAT 1988) and are considered to be order of magnitude estimates. The lower conversion factor (i.e., the one resulting in the higher risks) should not, however, underestimate risks by more than a factor of two. Asbestos air measurements collected in various California locations were also converted from TEM f/m^3 to PCM f/m^3 using a conversion factor. This conversion factor is also characterized by uncertainty. For the activity-related (i.e., soil- disturbing) exposure pathways and the incidental soil ingestion pathway, the units for the soil concentrations were PLM area%, which were assumed to be equal to PLM weight%. All these conversions add considerable uncertainty to the risk estimates.

The absence of environmental parameter measurement also contributes to uncertainty. Lack of site-specific measurements requires that estimates must be based on literature values, regression equations, extrapolations, and/or best professional judgement. Modeling errors can stem from a lack of validation or verification of the models. Typically an order of magnitude result is considered to be satisfactory for most complex modeling scenarios.

In almost all risk assessments, the largest source of uncertainty is in critical toxicity values (RfDs and cancer potency factors), and these uncertainties may significantly affect the magnitude of the risk estimates presented in a risk assessment. Health criteria for evaluating long-term exposures such as RfDs or cancer potency factors are based on concepts and assumptions which bias an evaluation in the direction of overestimation of health risk. EPA noted in its Guidelines for Carcinogenic Risk Assessment (EPA 1986b):

> There are major uncertainties in extrapolating both from animals to humans and from high to low doses. There are important species differences in uptake, metabolism, and

organ distribution of carcinogens, as well as species and strain differences in target site susceptibility. Human populations are variable with respect to geometric constitution, diet, occupational and home environment, activity patterns, and other cultural factors.

These uncertainties are compensated for by using upper bounds for cancer potency factors for carcinogens and safety factors for reference doses for noncarcinogens. At best, the assumptions used in a risk assessment provide a rough but plausible estimate of the upper limit of risk, i.e., it is not likely that the true risk would be much more than the estimated risk, but it could very well be considerably lower, even approaching zero.

In addition, there are varying degrees of confidence in the weight of evidence for carcinogenicity of a given chemical. EPA's (1986b) weight-of-evidence classification provides information which can indicate the level of confidence or uncertainty in the data obtained from studies inhumans or experimental animals. Some of the uncertainties in the hazard evaluation are further compensated for by assuming that animal carcinogens behave as human carcinogens. The summation of the risks associated with all potential carcinogens tends to overestimate risk by including probable human carcinogens (Group B) with demonstrated human carcinogens (Group A), although this was not done in this risk assessment.

For asbestos, in particular, there are important uncertainties associated with the toxicity criteria used to quantify human health risks. For the purposes of this risk assessment, the unit risk factor developed by the EPA (from which the MCLG was derived) was converted from a fiber-based number to a mass-based cancer potency factor. A conversion factor representing the fiber count per mass observed in the bioassay from which the unit risk factor was derived was used. This conversion factor, however, may not reflect the actual fiber count per mass observed in samples collected from soil at the South Bay Asbestos site. Additionally, the toxicologic basis for evaluation was drawn from a wide variety of epidemiologic studies which encompassed different forms of asbestos in addition to widely differing exposure conditions. The closeness of fit of these studies to the South Bay Asbestos site is not presently known.

One major source of error in asbestos risk assessment is evaluation of risks associated with ingestion. It should be kept in mind that only benign tumors were associated with ingestion of asbestos by the experimental animals in the bioassay which was the basis of the cancer potency factor. EPA guidance (EPA 1986b) for carcinogenic risk assessment does allow for inclusion of benign neoplasms, however, this could significantly overestimate risk.

There are also inherent uncertainties in determining the exposure parameters that are combined with toxicological information to estimate risk. For example, there are uncertainties regarding assumptions in estimating the likelihood than an individual would come into contact with contaminants originating at the site (e.g., the frequency of exposure estimates used for the soil ingestion pathways), the concentration of chemicals in the environmental media of concern (e.g., soil and air), and the period of time over which such exposures would occur (e.g., assuming children would repeatedly play in asbestos-containing soil). In general, conservative assumptions were made in estimating exposure point concentrations and estimating chemical intakes.

All of these individual sources of error may be propagated into larger errors by mathematical combination in the risk assessment. For purposes of evaluating remedial alternatives under Superfund, however, risk assessment provides a method to compare risks associated with various exposure routes, and this information can then be used to determine if and how remedial actions should be taken. There is a general perception that asbestos risk assessment is inherently less certain than risk assessments performed for other chemicals in the context of regulatory toxicology. This perception is not supported by a close examination of uncertainties associated with risk assessments in general. For example, nickel associated with copper mining tailings is assumed to be carcinogenic by inhalation without any evidentiary basis; lead in soil is often assumed to be 100% bioavailable by ingestion; the range of uncertainty between the upper and lower bounds on the cancer potency of 2,3,7,8-tetrachlorodibenzodioxin by the linearized multistage model is five orders of magnitude. In this context, it can be readily seen that asbestos risk assessments are no less uncertain than many

other risk assessments. After consideration of all the sources of uncertainty in the larger context of regulatory decision making, there appears to be no reason why public health and environmental protection decisions cannot be made on the basis of asbestos risk assessments.

Again, it should be noted that as with other risk assessments, the risks presented in this risk assessment are upper-bound estimates of the actual risk posed by the site. Conservative techniques were used to estimate all the risks presented in this document because of the uncertainty surrounding various analytical techniques in order that the actual risks would not be underestimated. This ensures that risk management decisions based on the results presented in this document would result in the protection of human health.

7.8 CONCLUSIONS AND SUMMARY

The results of sampling efforts performed at the South Bay Asbestos site, primarily those for the RI but also some previous analyses were summarized to identify chemicals to be evaluated in detail in this risk assessment. The primary chemical of concern with respect to potential effects on human health and the environment at the South Bay site is asbestos, which was detected in all sampled environmental media.

The following exposure pathways were selected for detailed evaluation in this risk assessment:

- Lifetime exposure of residents by inhalation of ambient air;
- Lifetime exposure of residents by inhalation of airborne asbestos generated by trucks in unpaved truckyards;
- Exposure of children by direct contact with and subsequent incidental ingestion of asbestos-contaminated soil in non-residential areas (i.e., vacant lots); and
- Lifetime exposure of residents by incidental ingestion of asbestos-contaminated soil during outdoor activities.

These pathways were selected based on their likelihood and frequency of occurrence, and on their potential for generating asbestos levels in air that may be of concern to human health.

The potentially exposed population includes the approximately 1,870 individuals who reside in Alviso and workers who may be employed in the town. The Alviso population is highly mobile; among those residing in Alviso in 1980, only 38% were living in Alviso in 1975. Approximately 10% of the Alviso population is under five years of age (187) and 4.5% over 65 years of age (84). Thirty-two percent of the population in 1980 was between five and 20 years of age (600).

Risks from the pathways listed above were characterized by first comparing information on the presence and concentrations of asbestos in the sampled environmental media to Applicable or Relevant and Appropriate Requirements (ARARs) identified for the South Bay site. Because asbestos ARARs were not available for all of the sampled environmental media, a quantitative risk characterization was also conducted. In this evaluation, estimates of potential asbestos exposures through each pathway identified for evaluation were combined with asbestos-specific toxicity values to predict potential risks associated with the site. For each pathway, an exposure scenario was developed based on assumptions about the environmental behavior and transport of asbestos, and the extent, frequency, and duration of exposures. These factors were used to predict potential exposures to asbestos for both average and a maximum plausible exposure case.

For chemical carcinogens such as asbestos, the excess upper bound lifetime cancer risk was estimated for each pathway; this risk is expressed as a probability. A risk of 1×10^{-6}, for example, represents the probability that an individual will develop cancer as a result of exposure to a carcinogenic chemical over a 70-year lifetime.

Based on a comparison to ARARs, the NESHAPs "no visible emissions" criterion appears to be relevant to the South Bay site. Visible emissions have been observed under present conditions in the area of the truckyards and along paved streets due to dust in gutters. Remedial activities resulting in site disturbances are also likely to result in some visible emissions.

The asbestos concentrations measured in the Guadalupe River upstream and downstream of the site exceed both the Ambient Water Quality Criterion for protection of human health and the proposed MCLG, as do the asbestos concentrations measured in groundwater at the site. However, neither the groundwater nor surface water areas are currently or expected to be used as a drinking water source.

Table 7-15 summarizes the results of the quantitative risk assessment for each exposure pathway and receptor group. For the inhalation of ambient air by Alviso and other nearby residents, excess lifetime cancer risks were estimated based on the Phase II reanalysis air data. The risks for the on-site sampling stations were slightly higher than those at either the upwind (Station 1) or downwind (Station 5) sampling locations. There are, however, many significant uncertainties associated with these risk estimates. The use of indirect PCMe structures/m^3 as a basis for deriving exposure point concentrations may be associated with roughly an order of magnitude uncertainty but is likely to provide conservative estimates of exposure point concentrations. The indirect-to-direct conversion factor alone is likely to have a minimum range of uncertainty of an order of magnitude, but the lower conversion factor of two (i.e., the one producing the higher risk) is unlikely to be underestimated by more than a factor of two. For the maximum case scenarios, the use of a single maximum air measurement to calculate lifetime inhalation risks is extremely conservative and thus the maximum case risks are more representative of upper bound risks rather than maximum plausible risks. Another major uncertainty in the ambient inhalation risk estimates is associated with uncertainties in the asbestos dose-response data. Therefore, considering the potential impact of the combination of these and other uncertainties, these risk results should be interpreted as order of magnitude estimates.

Lifetime inhalation risks associated with exposure to airborne asbestos at various locations in California were also estimated (SAI 1988). The excess lifetime cancer risks were predicted to be in the 10^{-6} to 10^{-4} range. There are important uncertainties associated with these risk estimates as well and thus they too should be interpreted with caution. For example, the conversion factor from TEM f/m^3 to PCM f/m^3 is uncertain as is the use

of only four sample measurements per location (all collected on the same day) to characterize average and maximum case long-term exposure point concentrations.

The DHS activity experiment data were used to evaluate risks associated with inhalation of vehicle-generated dusts. Only a few experimental sample data values were available and these were unvalidated. Thus there are significant uncertainties associated with these pathways of exposure. Based on the DHS data, the excess lifetime cancer risks for maximum case scenarios of exposure were all estimated to exceed 3×10^{-3}. This risk level is the highest value presented in EPA's Health Risk Table (Appendix K) from which the vehicle-generated inhalation risks were estimated.

Ingestion risks were predicted for children playing in soil and residents contacting soil in yards. The ingestion risk was 3×10^{-3} for children, and in the 10^{-5} range for residents.

It should be recognized that this summary risk table does not capture all aspects of uncertainty associated with this risk assessment. A discussion of these uncertainties was presented in Section 7.7. The risks presented in this assessment are not precise, and are presented solely to guide the decision of whether to take remedial action at the South Bay Asbestos site. As discussed in Section 7.7, the exposure assumptions and models used to estimate risks are generally conservative; therefore, the risk estimates shown in Table 7-15 are expected to be near to or higher than the upper end of the actual range of risks.

SECTION 8.0

SUMMARY AND CONCLUSIONS

8.0 SUMMARY AND CONCLUSIONS

EPA has conducted a remedial investigation at the South Bay Asbestos site because of concern that the asbestos in site soil and air is a health risk. Although there are still many unanswered questions, the consensus in the medical community is that asbestos fibers pose a cancer risk when inhaled. This report has presented the results of the environmental sampling, and the estimate of health risk at the site.

The sample results reveal that asbestos is present in surface soils in varying amounts throughout the community, wherever dumping has occurred or fill soils have been imported. There is no general pattern of soil contamination, and exact concentrations cannot be defined because of analytical uncertainties. The mechanisms which entrain or transport the asbestos from soil into the air have also not been clearly defined. It appears that soil-disturbing activities such as vehicle traffic or digging are more important than wind in releasing the fibers from the soil.

The risk assessment has shown that the ambient air in Alviso presents an excess lifetime lung cancer risk roughly an order of magnitude higher than the off-site locations measured. Exposure is also possible during soil-disturbing activities, with excess cancer risk estimates ranging up to 2 orders of magnitude higher than ambient risk, depending on soil asbestos concentration and type of activity. The highest risks were estimated for vehicle traffic in unpaved areas.

The limitations of asbestos measurement and analytical techniques, with the uncertainties of risk assessment estimates, make the precise definition of site risks difficult. It is clear that asbestos is present and may pose a risk higher than off-site locations.

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TABLES

Table 2.1

PREVIOUS INVESTIGATIONS AT SOUTH BAY ASBESTOS SITE

Sampling Date	No. of Samples	Location	Analytical Method	Agency
SURFACE SOIL SAMPLING				
8/8/83	2	Outfall structure	PLM	Cal-OSHA
8/10/83	6	Vicinity of outfall control structure	PLM	Cal-OSHA
8/10/83	7	Vicinity of outfall Control structure	PLM	DHS
8/18/83	42	Area of Liberty/ Moffat and also river levee	PLM	DHS
8/30/83 and 9/1/83	16	Community around Gold/ Moffat	PLM	DHS
9/12/83	25	Random grid (20) and around community	PLM (25), TEM (7)	DHS
6/18/85	2	Levee at Michigan	PLM & TEM	DHS worst case experiment
8/27/85	14	Community and ring levee	PLM (14), PLM & SEM (5) SEM & TEM (1)	EPA and DHS
9/17/85	2	Levee at Michigan and at State	TEM	DHS field experiment
9/25/85	2	Spreckles Avenue	PLM	DHS traffic experiment
10/4/85	47	N. end Spreckles (34) and Mayne School (13)	PLM	EPA
10/23/85	67	O'Neill Tract Streets	PLM	EPA
12/16/85 to 12/31/85	369	250' grid in community and every 100' on levee; recent fill at Environmental Ed. Center	PLM	EPA

TABLE 2.1, continued

Sampling Date	No. of Samples	Location	Analytical Method	Agency
5/7/86	1	Topsoil at WSP truck-yard	PLM	City of SJ (WCC)
5/15/86 and 5/16/86	*	Ring Levee	PLM	City of SJ (WCC)
7/23/86 and 8/7/86	52	Truckyards	PLM and TEM	EPA (E&E)

*93 samples at 47 locations

AIR SAMPLING

8/22/83 and 8/23/83	?	Outfall structure	?	BAAQMD
10/7/83	2	Fire station and N. end of Spreckles	TEM	DHS
10/12/83	2	Trailer Park and N. end of Spreckles	TEM	DHS
12/15/83 to 3/2/84	6	Mayne School, indoors and outdoors	TEM	DHS
4/11/84 and 5/3/84	4	2 residences, indoors and outdoors	TEM	DHS
8/16/84 to 11/1/84	26	7 sites, 5 in Alviso, also Moffett and San Jose	TEM	BAAQMD
6/18/85	2	Worst case experiment Spreckles and Michigan	TEM	DHS

Table 2.1, continued

Sampling Date	No. of Samples	Location	Analytical Method	Agency
9/17/85	2	Field experiment Spreckles and Michigan	TEM	DHS
9/25/85	5	Traffic simulation, N. end of Spreckles	PCM	DHS
5/7/86 to 5/13/86	30	4 sites near levee	PCM (12) and TEM (18)	City of SJ (WCC)
MARSHLAND LANDFILL INVESTIGATIONS				
3/11/85 to 3/25/85	3	SW corner of landfill	Priority Pollutants and Asbestos (PLM)	CalTrans/WCC

Note: Sample locations are shown on Figures 2-1 through 2-9.

Table 2.2
Truckyard Sampling Results

Sampling Date	Sample Number	Location	% Asbestos Versar PLM	Clayton PLM	EAL PLM	EAL TEM	Agency
08/07/86	A-RHM-01	Reinholm Crane and Rigging, at north end of Spreckles	<1 ND		-	0.0594	EPA (E & E)
	A-RHM-02	Reinholm Crane and Rigging, at north end of Spreckles	<1 ND				
	A-RHM-03	Reinholm Crane and Rigging, at north end of Spreckles	<1 TR	<1			
	A-RHM-04	Duplicate of A-RHM-03	<1 TR				
	A-RHM-05	Reinholm Crane and Rigging, at north end of Spreckles	<1 TR				
	A-RHM-06	Reinholm Crane and Rigging, at north end of Spreckles	<1 TR		-	0.0227	
	A-RHM-07	Duplicate of A-RHM-06	<1 ND		<1 ND		
07/23/87	A-WSP-07	WSP Trucking at intersection of Spreckles and State	<1 TR		<1 ND	0.0024	
	A-WSP-08	WSP Trucking at intersection of Spreckles and State	<1 TR				
	A-WSP-09	WSP Trucking at intersection of Spreckles and State	<1 TR		1	0.0018	
	A-WSP-10	WSP Trucking at intersection of Spreckles and State	<1 TR				
	A-WSP-11	WSP Trucking at intersection of Spreckles and State	<1 TR				
	A-JMM-12	J.M. Mattos Trucking, on State St.	<1 TR				
	A-JMM-13	J.M. Mattos Trucking, on State St.	<1 TR		2	0.0079	
	A-JMM-14	J.M. Mattos Trucking, on State St.	<1 TR				
	A-WSC-15	W.S. Bennett Construction, 1432 State St.	<1 TR				
	A-WSC-16	W.S. Bennett Construction, 1432 State St.	<1 ND				
	A-WSC-17	W.S. Bennett Construction, 1432 State St.	<1 ND		2	0.0024	
	A-WSC-18	W.S. Bennett Construction, 1432 State St.	<1 TR	19			
	A-WSC-19	W.S. Bennett Construction, 1432 State St.	<1 TR		1	0.0091	
	A-REB-20	Rebar Spacer Company, 1400 State St.	<1 ND		1	0.0021	
	A-REB-21	Rebar Spacer Company, 1400 State St.	<1 ND				
	A-REB-22	Rebar Spacer Company, 1400 State St.	<1 ND				
	A-REB-23	Rebar Spacer Company, 1400 State St.	<1 ND		<1 ND	0.0029	
	A-REB-24	Rebar Spacer Company, 1400 State St.	<1 ND				
	A-UAS-25	Truckyard at end of Archer near State St.	<1 ND	ND			

Continued on next page

Table 2.2
Truckyard Sampling Results

Sampling Date	Sample Number	Location	% Asbestos Versar PLM	Clayton PLM	EAL PLM	EAL TEM	Agency
07/23/87	A-UAS-26	Duplicate of A-UAS-25	<1 ND				EPA (E & E)
	A-UAS-27	Truckyard at end of Archer near State St.	<1 ND		<1 ND	0.0277	
	A-UAS-28	Truckyard at end of Archer near State St.	<1 TR		<1 ND	0.0194	
	A-BIA-30	WSP Trucking at intersection Spreckles and State	<1 TR		1	0.0001	
	A-PAL-41	Truckyard with pallets on Grand Blvd.	<1 ND				
	A-PAL-42	Duplicate of A-PAL-41	<1 ND		1	0.0198	
	A-PAL-43	Truckyard with pallets on Grand Blvd.	<1 ND	ND			
	A-PAL-44	Truckyard with pallets on Grand Blvd.	<1 ND		1	0.0264	
	A-PAL-45	Escalante Truckyard at intersection of Pacific and Grand	<1 TR		<1 ND	0.0038	
	A-PAL-46	Escalante Truckyard at intersection of Pacific and Grand	<1 TR		<1 ND	0.0020	
	A-ESC-47	Escalante Truckyard at intersection of Pacific and Grand	<1 TR				
	A-ESC-48	Duplicate of A-ESC-47	<1 TR				
	A-JMM-49	Truckyard at intersection of El Dorado and Moffet	<1 TR		1	0.0070	
	A-JMM-50	Duplicate of A-JMM-49	<1 TR				
	A-JMM-51	Truckyard at intersection of El Dorado and Moffet	<1 TR				
	A-JMM-52	Truckyard at intersection of El Dorado and Moffet	<1 TR				
	A-BRE-53	BRE Truckyard, 956 Catherine St.	<1 TR		2	0.0027	
	A-BRE-54	BRE Truckyard, 956 Catherine St.	<1 TR	2			
	A-BRE-55	BRE Truckyard, 956 Catherine St.	<1 ND		<1 ND	0.0004	
	A-BRE-56	Duplicate of A-BRE-55	<1 ND				
08/07/86	A-MAC-57	Maciel Truckyard on State St.	<1 TR		<1 ND	0.0007	
	A-MAC-58	Maciel Truckyard on State St.	<1 TR				
	A-MAC-59	Maciel Truckyard on State St.	<1 TR				
	A-MAC-60	Maciel Truckyard on State St.	<1 TR		2	0.0318	
	A-MAC-61	Maciel Truckyard on State St.	<1 TR				
	A-MAC-62	Maciel Truckyard on State St.	<1 TR				

TABLE 4.1. DEMOGRAPHIC COMPOSITION OF ALVISO

Description	Percent[a] Alviso	San Jose
SEX AND AGE COMPOSITION		
Total Population	100	100
Male	49.5	49.5
Female	50.5	50.5
Under 5 years of age	10.0	NA
5 to 20 years of age	31.7	NA
20 to 64 years of age	53.5	NA
65 years of age and older	4.5	6.2
Median age (in years)	22.6	27.4
ETHNIC COMPOSITION		
Total Population	100	100
White	97.0	73.6
White - Non Hispanic	33.2	51.3
White - Hispanic	63.8	22.3
Others	3.0	26.4
Black	0	4.6
American Indian	3.0	0.8
Other	0	8.3
MOBILITY		
Lived in same house 5 or more years	40	NA
Lived in different house in San Jose area 5 years ago	20	NA

[a] Reference: Aqua-Terra, 1986. Based on a population for Alviso of 1,877 persons, and population for San Jose of 629,000 persons.

NA - data not available from above source.

TABLE 5.1
DUPLICATE ANALYSIS RESULTS
ASBESTOS IN SOIL AND SEDIMENT
(ANALYSES BY TEM & PLM)

Co-located samples				Field Duplicates				Lab Duplicates			
Samples	Concentrations		%RSD	Samples	Concentrations		%RSD	Samples	Concentrations		%RSD
TEM Analyses:											
SL 5, 6	6.42	2.89	53.6	SL 10	0.013	0.010	18.4	SL 39	0.334	0.242	22
SL 12, 13	0.009	0.012	20.2	SL 20	0.135	0.004	133	SS 06	0.08	0.08	0
SL 21, 22	0.37	0.279	19.8	SL 40	0.044	0.387	113	SL 50	0.03	0.067	
SL 25, 26	0.346	0.83	58.2					SS 02	0.011	0.011	
SL 31, 32	0.056	0.066	11.6					SS 03	0.0016	0.0063	84.1
SL 33, 34	0.017	0.009	43.5								
SL 35, 36	0.325	0.144	54.6								
SL 40, 40, 41	0.044	0.387 0.22	79.0								
SL 42, 43	0.838	1.4	35.5								
SL 46, 47	0.103	0.25	58.9								
SL 57, 58	0.032	0.024	20.2								
PLM Analyses:											
SL 5, 6	6	8	20.2	SL 10	3	8	64.3	SL 36	ND	ND	
SL 8, 9	ND	ND	-	SL 20	2	2	0	SS 09	5	5	0
SL 12, 13	ND	ND	-	SL 30	ND	ND	-	SL 38	2	2	0
SL 16, 17	ND	ND	-	SL 40	1	ND	-	SL 42	3	3	0
SL 21, 22	ND	1	-					SD 04	ND	ND	
SL 23, 24	ND	ND	-								
SL 25, 26	1	2	47.1								
SL 27, 28	ND	ND	-								
SL 29, 30, 30	ND	ND 1	-								
SL 31, 32	ND	ND	-								
SL 33, 34	ND	ND	-								
SL 35, 36	1	ND	-								
SL 37, 38	ND	2	-								
SL 40, 40, 41	2 1	ND	-								
SL 42, 43	3	8	64.3								
SL 46, 47	2	2	0								
SL 46, 47	ND	ND	-								
SL 57, 58	1	ND	-								
Station 5 (additional background sample)	ND	ND									

Table 5.2
Soil Asbestos Standards Analyses

Analyses By TEM

Sample	Reported Concentration (R)	Actual Concentration (A)	% Bias	% RSD
DOHS STD-004	0.342	1.0	-65.0	69.4
DOHS STD-005	0.347	1.0	-65.3	68.6
Lab Dup.	0.321	1.0	-67.9	72.7
DOHS STD-002	0.25	.10	150	60.6
DOHS STD-003	21.4	5.0	328	87.9
DOHS STD-001	0.046	0.01	360	90.9

Analyses By PLM

Reported Concentration	Actual Concentration	% Bias	% RSD
9%	3%	200	70.7
50%	50%	0	0

R = Reported Concentration

A = Actual Concentration

%Bias = (R-A)/A

%RSD = 200 |R-A|/(1.414 (R+A))

TABLE 5.3
SUMMARY OF PHASE I SURFACE SOIL PLM ASBESTOS RESULTS

SAMPLE LOCATION	DUPLICATE SAMPLE	CO-LOCATED SAMPLE	ANALYSIS REQUESTED	ASBESTOS RESULT	COMMENTS
SL-014-001			PLM	26.0	Ring levee
SL-015-001			PLM	14.0	Ring levee
SL-043-001		CO-LOCATED WITH 042	PLM	8.0 J	Liberty/Catherine St.
SL-006-001		CO-LOCATED WITH 005	PLM	8.0 J	Alviso Marina
SL-005-001			PLM	6.0 J	Alviso Marina
SL-055-001			PLM	4.0 J	Field east of ring levee
SL-053-001			PLM	4.0 J	Field east of ring levee
SL-042-001			PLM	3.0 J	Liberty/Catherine St.
SL-056-001			PLM	2.0 J	Field east of ring levee
SL-045-001		CO-LOCATED WITH 044	PLM	2.0 J	Catherine St.
SL-044-001			PLM	2.0 J	Catherine St.
SL-040-001			PLM	2.0 J	
SL-038-001		CO-LOCATED WITH 037	PLM	2.0 J	
SL-026-001		CO-LOCATED WITH 025	PLM	2.0 J	
SL-020-002	SL-020-001		PLM	2.0 J	Street dust
SL-004-001			PLM	2.0 J	Alviso Marina
SL-057-001		CO-LOCATED WITH 058	PLM	1.0 J	
SL-050-001			PLM	1.0 J	
SL-041-001		CO-LOCATED WITH 040	PLM	1.0 J	
SL-035-001			PLM	1.0 J	
SL-030-002	SL-030-001		PLM	1.0 J	
SL-025-001			PLM	1.0 J	
SL-022-001		CO-LOCATED WITH 021	PLM	1.0 J	
SL-020-001			PLM	1.0 J	Street dust
SL-019-001			PLM	1.0 J	Street dust
SL-002-001			PLM	1.0 J	
SL-058-001			PLM	<1 ND	
SL-054-001			PLM	<1 ND	
SL-052-001			PLM	<1 ND	
SL-051-001			PLM	<1 ND	
SL-049-001			PLM	<1 ND	
SL-048-001			PLM	<1 ND	
SL-047-001		CO-LOCATED WITH 046	PLM	<1 ND	
SL-046-001			PLM	<1 ND	
SL-040-002	SL-040-001		PLM	<1 ND	
SL-039-001			PLM	<1 ND	
SL-037-001			PLM	<1 ND	
SL-036-001		CO-LOCATED WITH 035	PLM	<1 ND	
SL-034-001		CO-LOCATED WITH 033	PLM	<1 ND	
SL-033-001			PLM	<1 ND	
SL-032-001		CO-LOCATED WITH 031	PLM	<1 ND	
SL-031-001			PLM	<1 ND	
SL-030-001		CO-LOCATED WITH 029	PLM	<1 ND	
SL-029-001			PLM	<1 ND	
SL-028-001		CO-LOCATED WITH 027	PLM	<1 ND	

TABLE 5.3

SUMMARY OF PHASE I SURFACE SOIL PLM ASBESTOS RESULTS (con'd)

SAMPLE LOCATION	DUPLICATE SAMPLE	CO-LOCATED SAMPLE	ANALYSIS REQUESTED	ASBESTOS RESULT	COMMENTS
SL-027-001			PLM	<1 ND	
SL-024-001		CO-LOCATED WITH 023	PLM	<1 ND	
SL-023-001			PLM	<1 ND	
SL-021-001			PLM	<1 ND	
SL-018-001			PLM	<1 ND	
SL-017-001		CO-LOCATED WITH 016	PLM	<1 ND	
SL-016-001			PLM	<1 ND	
SL-013-001		CO-LOCATED WITH 012	PLM	<1 ND	
SL-012-001			PLM	<1 ND	
SL-011-001			PLM	<1 ND	
SL-010-002	SL-010-001		PLM	<1 ND	
SL-010-001			PLM	<1 ND	
SL-009-001		CO-LOCATED WITH 008	PLM	<1 ND	
SL-008-001			PLM	<1 ND	
SL-007-001			PLM	<1 ND	
SL-003-001			PLM	<1 ND	
SL-001-001			PLM	<1 ND	
STD-A			PLM	9.0	EPA R.T.I STANDARD -3.0%
STD-B			PLM	50.0	EPA R.T.I STANDARD -50.0%

TOTALS:

62 SAMPLES @ 40 LOCATIONS — 4 FIELD DUPLICATES — 18 CO-LOCATED SAMPLES

TABLE 5.4

SUMMARY OF PHASE I SURFACE SOIL TEM ASBESTOS RESULTS

SAMPLE LOCATION	DUPLICATE SAMPLE	CO-LOCATED SAMPLE	ANALYSIS REQUESTED	ASBESTOS RESULT	COMMENTS
SL-014-001			TEM	102.000 J	Ring Levee
SL-005-001			TEM	6.420 J	Alviso Marina
SL-006-001		CO-LOCATED WITH 005	TEM	2.890 J	Alviso Marina
SL-043-001		CO-LOCATED WITH 042	TEM	1.400 J	Liberty/Catherine St.
SL-042-001			TEM	0.840 J	
SL-026-001		CO-LOCATED WITH 025	TEM	0.830 J	
SL-040-002	SL-040-001		TEM	0.390 J	
SL-021-001			TEM	0.370 J	
SL-025-001			TEM	0.350 J	
SL-035-001			TEM	0.320 J	
SL-022-001		CO-LOCATED WITH 021	TEM	0.280 J	
SL-047-001		CO-LOCATED WITH 046	TEM	0.250 J	
SL-039-001			TEM	0.242 J	Ring Levee
SL-041-001		CO-LOCATED WITH 040	TEM	0.220 J	
SL-036-001		CO-LOCATED WITH 035	TEM	0.144 J	
SL-019-001			TEM	0.140 J	
SL-020-001			TEM	0.135 J	
SL-046-001			TEM	0.103 J	
SL-032-001		CO-LOCATED WITH 031	TEM	0.070 J	
SL-001-001			TEM	0.066 J	
SL-053-001			TEM	0.064 J	
SL-031-001			TEM	0.056 J	
SL-040-001			TEM	0.044 J	
SL-057-001		CO-LOCATED WITH 058	TEM	0.032 J	
SL-050-001			TEM	0.030 J	
SL-058-001			TEM	0.024 J	
SL-033-001			TEM	0.017 J	
SL-010-001			TEM	0.013 J	
SL-013-001		CO-LOCATED WITH 012	TEM	0.012 J	
SL-002-001			TEM	0.012 J	
SL-010-002	SL-010-001		TEM	0.010 J	
SL-034-001		CO-LOCATED WITH 033	TEM	0.009 J	
SL-012-001			TEM	0.009 J	
SL-020-002	SL-020-001		TEM	0.004 J	
SL-048-001			TEM	0.003 J	
STD-001			TEM	0.046 J	CA DOHS STANDARD- 0.01%
STD-002			TEM	0.250 J	CA DOHS STANDARD- 0.1%
STD-003			TEM	21.400 J	CA DOHS STANDARD- 5.0%
STD-004			TEM	0.340	CA DOHS STANDARD- 1.0%
STD-005	STD-004		TEM	0.350	CA DOHS STANDARD- 1.0%
LAB. DUPE.	STD-004		TEM	0.321	LABORATORY DUPLICATE-1.0%

TOTALS:

35 SAMPLES BY TEM — 3 FIELD DUPLICATES — 11 CO-LOCATED DUPLICATES

TABLE 5.5
SUMMARY OF SUBSURFACE SOIL SAMPLE RESULTS
ASBESTOS AND TARGET COMPOUND LIST (TCL) DATA

SUBSURFACE BORING NUMBER	SAMPLE NUMBER	SAMPLE DEPTH	QUALITY CONTROL SAMPLE INFO	PLM ASBESTOS RESULT	TEM ASBESTOS RESULT	TCL CONTAMINANTS RESULT
MONITOR WELL 1	SS-001-001	0 - 1.5		2.0 J	0.147 J	NA
(SS-MW-001)	SS-001-002	3.5 - 5.0		<1.0 ND	NA	NA
	SS-001-003	8.0 - 9.5		<1.0 ND	NA	NO SIGNIF. CONCEN.
	SS-001-004	8.0 - 9.5	DUPLICATE	<1.0 ND	NA	NO SIGNIF. CONCEN.
MONITOR WELL 2	SS-002-001	0 - 1.5		1.0 J	NA	NA
(SS-MW-002)	SS-002-002	0 - 1.5	DUPLICATE	<1.0 ND	0.042 J	NA
	SS-002-003	4.0 - 5.5		<1.0 ND	0.011 J	NA
	SS-002-004	9.0 - 10.5		<1.0 ND	NA	NO SIGNIF. CONCEN.
MONITOR WELL 3	SS-003-001	2.5 - 4.0		<1.0 ND	0.003 J	NA
(SS-MW-003)	SS-003-002	3.5 - 5.0		<1.0 ND	NA	NA
	SS-003-003	8.5 - 10.0		<1.0 ND	NA	NO SIGNIF. CONCEN.
	SS-003-004	2.5 - 4.0	DUPLICATE	<1.0 ND	0.0016 J	NA
MONITOR WELL 4	SS-004-001	0 - 1.5		<1.0 ND	NA	NA
(SS-MW-004)	SS-004-002	4.0 - 5.5		<1.0 ND	0.024 J	NA
	SS-004-003	9.0 - 10.5		7.0	NA	NA
	SS-004-004	14.0 - 15.5		<1.0 ND	NA	NA
	SS-004-005	19.0 - 20.5		<1.0 ND	NA	NO SIGNIF. CONCEN.
	SS-004-006	24.0 - 25.5		<1.0 ND	NA	NA
MONITOR WELL 5	SS-005-001	0 - 1.5		<1.0 ND	NA	NA
(SS-MW-005)	SS-005-002	4.0 - 5.5		<1.0 ND	0.004 J	NA
	SS-005-003	9.0 - 10.5		<1.0 ND	NA	NO SIGNIF. CONCEN.
BACKGROUND BORING	SS-006-001	0 - 0.6	BACKGROUND	1.0 J	0.08 J	NO SIGNIF. CONCEN.
(SS-MW-006)						
SOIL BORING 7	SS-007-001	0 - 1.5		<1.0 ND	NA	NA
(SS-BOR-007)	SS-007-002	4.0 - 5.5		<1.0 ND	0.013	NA
	SS-007-003	9.0 - 10.5		<1.0 ND	NA	NA
SOIL BORING 8	SS-008-001	0 - 1.5		<1.0 ND	NA	NA
(SS-BOR-008)	SS-008-002	3.5 - 5.0		<1.0 ND	0.036 J	NA
	SS-008-003	8.5 - 10.0		<1.0 ND	NA	NA
SOIL BORING 9	SS-009-001	0 - 1.5		5.0 J	NA	NA
(SS-BOR-009)	SS-009-002	4.0 - 5.5		2.0 J	0.474 J	NA
	SS-009-003	9.0 - 10.5		<1.0 ND	NA	NA

TABLE 5.5 (con'd)
SUMMARY OF SUBSURFACE SOIL SAMPLE RESULTS
ASBESTOS AND TARGET COMPOUND LIST (TCL) DATA

SUBSURFACE BORING NUMBER	SAMPLE NUMBER	SAMPLE DEPTH	QUALITY CONTROL SAMPLE INFO	PLM ASBESTOS RESULT	TEM ASBESTOS RESULT	TCL CONTAMINANTS RESULT
SOIL BORING 10	SS-010-001	1.0 - 2.5		<1.0 ND	NA	NA
(SS-BOR-010)	SS-010-002	3.5 - 5.0		<1.0 ND	0.018 J	NA
	SS-010-003	8.5 - 10.0		<1.0 ND	NA	NA
	SS-010-004	3.5 - 5.0	DUPLICATE	<1.0 ND	0.010 J	NA
SOIL BORING 11	SS-011-001	0 - 1.5		<1.0 ND	NA	NA
(SS-BOR-011)	SS-011-002	4.0 - 5.5		<1.0 ND	0.003 J	NA
	SS-011-003	9.0 - 10.5		<1.0 ND	NA	NA
	SS-011-004	0 - 1.5	DUPLICATE	<1.0 ND	NA	NA
MARSHLAND BORING G3	SS-G03-001	0 - 1.0		<1.0 ND	0.300	NA
(SS-G3)	SS-G03-002	8.5 - 11.5		<1.0 ND	NA	NO SIGNIF. CONCEN.
MARSHLAND BORING G4	SS-G04-001	0 - 3.0		<1.0 ND	NA	NA
(SS-G4)						
MARSHLAND BORING G5	SS-G05-001	0 - 1.5		<1.0 ND	0.450	NA
(SS-G5)	SS-G05-002	16.4 - 16.5		1.0	NA	NA
	SS-G05-003	16.5 - 18.0		NA	NA	NO SIGNIF. CONCEN.
MARSHLAND BORING G6	SS-G06-001	0 - 1.5		<1.0 ND	NA	NA
(SS-G6)	SS-G06-002	4.5 - 5.5		<1.0 ND	0.010	NO SIGNIF. CONCEN.
	SS-G06-003	4.5 - 5.5	DUPLICATE	<1.0 ND	0.050	NO SIGNIF. CONCEN.
MARSHLAND BORING G9	SS-G09-001	0 - 1.5		<1.0 ND	NA	NA
(SS-G9)	SS-G09-002	3.3 - 5.3		<1.0 ND	0.170	NO SIGNIF. CONCEN.
MARSHLAND BORING G11	SS-G11-001	0 - 1.5		<1.0 ND	NA	NA
(SS-G11)	SS-G11-002	6.0 - 8.0		<1.0 ND	0.012	NO SIGNIF. CONCEN.

NA = NOT ANALYZED
ND = NOT DETECTED
NO SIGNIF. CONCEN. = NP SIGNIFICANT CONCENTRATION

Table 5.6
Asbestos Duplicate Analyses
Ground and Surface Water

Sample	Type	Duplicate Pair Asbestos Concentrations (mf/L)	%RSD
SW-02	F	2160,3077	25
SW-02	L	3077,2575	13
GW-04	L	1867,2345	16
GW-05	F	209,318	29
GW13*	L	1.7,2.0	11
GWG5	F	70,320	91
		Average % RSD =	31%

Explanation:

Type - F = Field Duplicate
L = Lab Duplicate

GW13* - This sample is a Field Blank

%RSD - Percent Relative Standard Deviation

$$\%RSD = 200\ (|C_1 - C_2| / ((C_1 + C_2)\ \sqrt{2}))$$

C_1, C_2 = Concentration of Duplicates

mf/L - million fibers per liter

Table 5.7
Asbestos Blank Analyses
Ground and Surface Water

Blank Type	Total Asbestos (mf/L)
F	2.0
F	2.0
L	ND
F	19
F	21
F	7
L	.0025
F	1.13
L	.013

Blank Type - F = field blank
L = Laboratory Method Blank
mf/L = million fibers/liters

TABLE 5.8
SUMMARY OF WATER SAMPLE RESULTS
ASBESTOS AND TOXIC CONTAMINANT LIST (TCL) DATA

SAMPLE LOCATION	SAMPLE NUMBER	DATE COLLECTED	ASBESTOS (TOTAL FIBERS)	ASBESTOS (FIBERS >10 MICRONS)	TCL RESULT	COMMENTS
GUADALUPE RIVER (UPSTREAM)	SW-001-001	5/21/87	3266 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	Water asbestos results in millions of fibers per liter.
GUADALUPE RIVER (DOWNSTREAM)	SW-002-001	5/21/87	2160 MF/L	50 MF/L	NO SIGNIFICANT LEVELS DETECTED	
GUADALUPE RIVER (DOWNSTREAM DUPLICATE)	SW-002-002	5/21/87	3077 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
RINSATE BLANK	SW-003-001	5/21/87	1.13 MF/L	ND	BNA	16 ppb bis(2-ethylhexyl)phthlate detected. Assumed to be laboratory contamination.
MONITOR WELL 1	GW-001-001	6/25/87	4774 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 1 (second round)	GW-001-002	10/7/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 2	GW-002-001	6/25/87	2471 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 2 (second round)	GW-002-002	10/7/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 2 (second round duplicate)	GW-002-003	10/7/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 5	GW-003-001	6/26/87	2211 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 3 (second round)	GW-003-002	10/8/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 4	GW-004-001	6/26/87	1867 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 5 (second round)	GW-004-002	10/8/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	

TABLE 5.8
SUMMARY OF WATER SAMPLE RESULTS
ASBESTOS AND TOXIC CONTAMINANT LIST (TCL) DATA

SAMPLE LOCATION	SAMPLE NUMBER	DATE COLLECTED	ASBESTOS (TOTAL FIBERS)	ASBESTOS (FIBERS >10 MICRONS)	TCL RESULT	COMMENTS
MONITOR WELL 3	GW-005-001	6/24/87	209 MF/L	8.4 MF/L	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 3 (first round duplicate)	GW-005-002	6/24/87	318 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MONITOR WELL 4 (second round)	GW-005-003	10/6/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
RINSATE BLANK	GW-006-001	6/24/87	19 MF/L	ND	VOLATILE	18 ppb 2-Butanone detected; assumed to be laboratory contamination.
RINSATE BLANK	GW-006-002	10/07/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
FIELD BLANK	GW-006-003	11/16/87	2 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
RINSATE BLANK	GW-013-001	6/25/87	21 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
RINSATE BLANK	GW-013-002	10/08/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
FIELD BLANK	GW-013-003	11/17/87	1.7 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
RINSATE BLANK	GW-014-001	6/26/87	7 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MARSHLAND LANDFILL WELL G3	GW-G3-001	11/16/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	
MARSHLAND LANDFILL WELL G5	GW-G5-001	11/17/87	320 MF/L	ND	NO SIGNIFICANT LEVELS DETECTED	
MARSHLAND LANDFILL WELL G5 (duplicate)	GW-G5-002	11/17/87	70 MF/L	10 MF/L	NO SIGNIFICANT LEVELS DETECTED	
MARSHLAND LANDFILL WELL G6	GW-G6-001	11/17/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	Very low yield;sample for full organics only.
MARSHLAND LANDFILL WELL G6	GW-G6-002	11/18/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	Very low yield; sample for metals only.
MARSHLAND LANDFILL WELL G9	GW-G9-001	11/18/87	NA	NA	NO SIGNIFICANT LEVELS DETECTED	Very low yield; sampled for volatiles only.
MARSHLAND LANDFILL WELL G11	GW-G11-001	11/16/87	NA	NA	volatile	320 ppb trichloroethene detected.

NA = NOT ANALYZED
ND = NOT DETECTED

TABLE 5.9
AIR DUPLICATE DATA

Sample ID	Duplicate Type	Structures per Cubic Meter		%RSD	PCME per Cubic Meter		%RSD
1-004	Field	2800	2400	10.9	0	0	
2-004	Field	7800	6600	11.8	0	0	
2-038E	Field	1000	0		1000	0	
3-004	Field	7500	6900	5.9	0	600	
4-003	Field	1020	4800	50.9	0	0	
4-004	Field	3200	8000	60.6	0	1400	
4-010E	Field	0	0		0	0	
4-029E	Field	1500	5600	81.7	0	800	
4-030E	Field	0	1600		0	800	
4-036E	Field	3000	2000	28.3	0	0	
4-038E	Field	1000	0		0	0	
4-041E	Field	4400	5900	20.6	0	0	
4-042E	Field	0	1500		0	0	
4-045E	Field	1600	5500	69.1	1000	0	
4-049E	Field	0	0		0	0	
5-004	Field	0	3300		0	0	
5-036E	Field	0	8000		0	0	
5-041E	Field	0	3000		0	0	
5-042E	Field	14600	5900	60.0	0	0	
5-045E	Field	0	1000		0	0	
5-049E	Field	0	0		0	0	
3-041E	Lab	6200	6200	0.0	3100	0	
5-049E	Lab	0	0		0	0	
1-021	Lab	0	0		0	0	
3-025	Lab	0	0		0	0	
1-035	Lab	0	0		0	0	
3-047	Lab	0	0		0	0	
4-030E	Lab	0	0		0	0	
4-045E	Lab	1500	1600	4.6	1000	3000	70.7
2-023E	Lab	0	0		0	0	
5G-022	Lab	0	0		0	0	
1G-010E	Lab	0	0		0	0	
1A-003	Lab	3700	1900	45.5	900	0	
Average %RSD Values				34.6			70.7

Table 5.10
Comparison of PCM and PCME Analyses

Sample	PCM (Fibers/m^3)	PCME (Fibers/m^3)
AQ-2A-004	1,200	ND
AQ-3A-004	2,100	ND
AQ-3B-004	2,700	600
AQ-4A-004	2,900	ND
AQ-4B-004	2,100	1,400
AQ-5B-004	1,000	ND
AQ-2A-010E	1,300	2,300
AQ-2A-010E	1,600	2,300

PMC - Asbestos PCM fiber concentrations determined by Phase Contrast Microscopy.

PCME - PCM equivalent fibers determined by Transmission Electron Microscopy. Samples were prepared for analysis by the direct method.

Table 5.11
Episodic Sample Results

Episode Date	Wind Direction (Azimuth)	Asbestos Concentrations (structures/m^3) Station 1	Station 2	Station 3	Station 4	Station 5
08/19/87	300-315	ND	8100	1500	ND	700
09/24/87	315-355	ND	2400	800	3100	800
09/25/87	320-360	1600	NA	ND	1000	NA
10/07/87	335-340	ND	9000	1800	2400	7,500
10/09/87	295-355	ND	1700	9720	1000	2,800
10/15/87	70-180	4600	4400	6200	5151	3,000
10/16/87	290-315	1500	16,500	ND	950	5,900
10/22/87	270-335	4200	NA	28,000	11,000	10,400
10/31/87	280-330	1500	ND	1500	600	ND

NA - Sample not analyzed due to overloading
ND - No structures observed
Duplicate concentrations are averaged

Table 5.12

Phase I

Basic Statistics on Asbestos Structures by Station

	Asbestos Concentrations (structures/m^3)					
	Station 1	Station 2	Station 3	Station 4	Station 5	Stations 2, 3, 4 Combined
Count	22	22	22	22	22	66
Average	1550	3110	5024	2624	2931	3586
Standard Deviation	1574	3961	7698	2885	3430	5410
Std. Dev. of Mean	336	844	1641	615	731	666
Upper 95% Conf. Limit	2248	4867	8438	3903	4452	4891
Lower 95% Conf. Limit	852	1354	1610	1345	1410	2280
Median	950	1700	1500	1100	1780	
75th Percentile	2100	3400	6200	3400	2800	
90th Percentile	4200	8100	10300	7500	10000	
Maximum	5200	16500	28000	16000	10500	
Frequency Table						
<1000	11	10	10	10	10	
1000 to 2000	3	2	3	3	5	
2000 to 3000	4	2	1	2	2	
3000 to 4000	1	2	1	2	0	
4000 to 5000	2	1	0	0	0	
5000 to 6000	1	1	0	2	1	
6000 to 7000	0	0	1	0	0	
>7000	0	4	6	3	4	

Table 5.13

Phase I
PCME Summmary Statistics by Station

	Station Number				
	1	2	3	4	5
Number of Samples	23	24	24	33	23
Number Detected	2	8	6	5	3
% Detected	8.7	33	25	15	13
Average (PCME/m^3)	2,350	1,725	3,070	920	970

Number of Samples = # of Samples Analyzed

Number Detected = # of Samples containing detectable PCME Fibers

Percentage Detected = Percentage of Samples containing detectable PCME Fibers

Average = Average of detectable concentrations

Table 5.14

Paired Difference Analysis

	Pairs		
	Stations 1 and 2	Stations 1 and 3	Stations 1 and 4
Count	21	22	22
Average	1890	3490	1090
Standard Deviation	4220	7390	2454
Standard Error	926	1570	523
Standard Variable (z)	2.05	2.21	2.08

Concentrations in Asbestos Structures/m^3

Count	=	Number of pairs (n)
Average	=	Average difference (x)
Standard Deviation	=	Standard deviation of the differences (s)
Standard Error	=	$s/\sqrt{n} = S_x$
z	=	x/S_x

TABLE 5.15

Comparison of Phase I and Phase II Asbestos Analysis

Indirect Analyses (Phase II)			Direct Analyses (Phase I)		
Sample ID	Total	PCME	Sample ID	Total	PCME
AQ-01B-009	140,000	310	AQ-01A-009	2,100	0
AQ-01B-015	43,000	90	AQ-01A-015	2,100	0
AQ-01B-025	850,000	860	AQ-01A-025	0	0
AQ-01B-026	8,100	0	AQ-01-026	0	0
AQ-01B-030	190,000	210	AQ-01-030E	1,600	0
AQ-01B-041	47,000	80	AQ-01A-041E	4,600	0
AQ-02B-010	540,000	430	AQ-02A-010E	9,000	2,300
AQ-02B-015	820,000	980	AQ-02A-015	3,400	0
AQ-02B-021	1,100,000	8,500	AQ-02A-021	0	0
AQ-02B-025	1,300,000	1,200	AQ-02A-025	900	0
AQ-02B-026	69,000	650	AQ-02A-026	0	0
AQ-02B-047	1,000,000	1,100	AQ-02A-047	2,600	0
AQ-03A-036	1,500,000	4,500	AQ-03B-036E	2,000	0
AQ-03B-035	830,000	1,300	AQ-03A-035	26,000	8,000
AQ-03B-040	180,000	310	AQ-03A-040	0	0
AQ-03B-043	1,500,000	7,600	AQ-03A-093	NA	NA
AQ-04A-015	990,000	600	AQ-04B-015	1,000	0
AQ-04A-021	420,000	710	AQ-04B-021	0	0
AQ-04A-025	230,000	510	AQ-04B-025	1,000	0
AQ-04B-009	330,000	390	AQ-04A-009	7,600	0
AQ-04B-026	47,000	140	AQ-04A-026	0	0
AQ-04B-035	1,800,000	3,300	AQ-04A-035	2,800	0
AQ-04B-047	220,000	630	AQ-04A-047	3,400	0
AQ-05B-009	690,000	1,100	AQ-05A-009	10,000	1,000
AQ-05B-015	65,000	40	AQ-05A-015	2,000	0
EMS Direct Analyses			Clayton Direct Analyses		
AQ-01B-009	2,300	10	AQ-01A-009	2,100	0
AQ-04B-009	2,300	30	AQ-04A-009	7,600	0
AQ-05B-009	16,000	190	AQ-05A-009	10,000	8,000

NOTES: 1) Clayton Environmental performed the Phase I analyses.

EMS performed the Phase II reanalysis.

2) A zero indicates no observable fibers.

3) Concentrations in asbestos structures/m^3

4) NA = Not analyzed due to overloading.

Table 5.16

Phase II

Statistics on Particulate Data

Total Suspended Particulates

Station	Count	Average	Standard Deviation
1	4	57.3	20.4
2	5	132.7	44.2
3	3	102.2	34.9
4	7	73.4	25.6
5	2	90.7	39.0

PM-10

Station	Count	Average	Standard Deviation
1	4	47.4	30.3
2	5	59.9	20.0
3	3	66.9	22.7
4	7	48.1	20.3
5	2	35.6	8.2

Table 5.17

Correlation Coefficients For
Asbestos and Particulate Scatterplots

Correlation Matrix		
	TSP	PM 10
Asbestos Structures	.72	.56
PCM Equivalents	.54	.31

Values presented in the correlation matrix are correlation coefficients (r).

Example: The correlation coefficient between PCM and PC-10 concentrations is .31.

See Figures J-21 through J-24 for associated plots.

Table 5.18

Basic Statistics on Phase II Asbestos Concentrations

Total Asbestos Structures

	Count	Average	Standard Deviation
Station 1	6	213,000	292,000
Stations 2,3,4	16	711,000	522,000
Station 5	2	378,000	313,000
Station 2	6	805,000	405,000
Station 3	3	837,000	539,000
Station 4	7	577,000	571,000

PCME Structures

	Count	Average	Standard Deviation
Station 1	6	260	290
Stations 2,3,4	16	1,580	2,110
Station 5	2	570	530
Station 2	6	2,140	2,850
Station 3	3	2,040	1,790
Station 4	7	900	1,000

TABLE 7-1

BACKGROUND CONCENTRATIONS OF INORGANIC CHEMICALS PRESENT IN SOILS AND SURFACE WATER IN THE REGION OF THE SOUTH BAY ASBESTOS AREA SITE AND IN WESTERN UNITED STATES

Chemical	Surficial Soils (mg/kg)[a]	B Horizon Soils, Western U.S. (mg/kg)[b]	Surface Water (ug/liter)[c]
Aluminum	60,000 - 85,000	5,000 - >100,000	ND - 10
Antimony	<1	NT	2 - 3
Arsenic	5.3 - 8.3	<0.2 - 97	NT
Barium	600 - 850	70 - 5,000	NT
Beryllium	<1	<10 - 300	NT
Cadmium	NT	<1 - 10	ND - <1
Calcium	15,000 - 35,000	100 - 400,000	22,000 - 48,000
Chromium	85 - >2000	3 - 1,500	10
Cobalt	12.5 - >70	<3 - 50	NT
Copper	25 - 40	2 - 300	2
Iron	25,000 - 40,000	1,500 - >100,000	<10 - 60
Lead	25 - >700	<7 - 700	2 - 5
Magnesium	12,500 - 10,000	5,000 - 100,000	16,000 - 38,000
Manganese	400 - 600	30 - 5,000	ND - 20
Mercury	0.17 - 0.26	0.01 - 4.6	ND
Nickel	25 - >700	<3 - 700	ND
Potassium	13,500 - 18,000	1,900 - 63,000	NT
Silver	NT	<0.5 - 5	NT
Sodium	15,000 - >100,000	500 - 100,000	9,100 - 23,000
Vanadium	125 - >500	7 - 500	NT
Zinc	97 - >3,500	<10 - 2,000	50 - 90

NT = Not tested. Chemical was not an analyte for surface water or soil samples.
ND = Not detected.

[a]Shacklette and Boerngen (1984). California surficial soil concentrations in the region of the South Bay Asbestos Area.
[b]Connor and Shacklette (1975).
[c]USGS (1982). Guadelupe River at San Jose.

TABLE 7-2

SUMMARY OF PHASE II INDIRECT ASBESTOS REANALYSIS (a)

Date Sampled (Sample No.)	Concentration (PCMe structures/m3)					
	Station 1	Station 2	Station 3	Station 4	Stations 2-4	Station 5
8/18/87 (009)	310	---	---	390	---	1,100
8/19/87 (010)E	---	430	---	---	---	---
8/30/87 (015)	90	980	---	600	---	40
9/11/87 (021)	---	8,500	---	710	---	---
9/17/87 (025)	860	1,200	---	510	---	---
9/20/87 (026)	50 U	650	---	140	---	---
9/25/87 (030)E	210	---	---	---	---	---
10/5/87 (035)E	---	---	1,300	3,300	---	---
10/7/87 (036)	---	---	4,500	---	---	---
10/14/87 (040)E	---	---	310	---	---	---
10/15/87 (041)E	80	---	---	---	---	---
10/17/87 (043)E	---	---	7,600	---	---	---
10/26/87 (047)	---	1,100	---	630	---	---
Summary Information						
No. of Samples Analyzed	6	6	4	7	17	2
Frequency of Detection	5/6	6/6	4/4	7/7	17/17	2/2
Geometric Mean Concentration (b)	147	1,206	1,927	589	1,003	210
Maximum Concentration	860	8,500	7,600	3,300	8,500	1,100

(a) Samples are from CDM's Phase II reanalysis of Phase I ambient air samples. Station 1 is located upwind, Stations 2-4 on site, and Station 5 off-site downwind.
(b) Geometric means were calculated using one-half of the reported detection limit for undetected samples.

U = Not detected. The listed value is the detection limit.
E = Episodic (4- to 12-hour average) sample. All others are 24-hour average samples.

Source: U.S. Environmental Protection Agency (USEPA). 1988. Memorandum from K. Kitchingham, Environmental Services Branch, to G. Baker, Remedial Branch TWPD. EPA REgion IX. Subject: Review of Analytical Data for the South Bay Asbestos Site.

TABLE 7-3

COMPARISON OF STATE BACKGROUND LEVELS (SAI 1981)
AND PHASE I DIRECT DATA (1987)

Sample Location	Asbestos Source Type	Asbestos Concentration (TEM f/m3)		Asbestos Concentration (TEM structures/m3)	
		Range	Geometric Mean (b)	Range	Geometric Mean (b)
SAI (1981) Study (a):					
Sonora	Natural Deposits	4,800 - 14,400	5,200	NR	NR
San Jose	Industrial	11,800 - 23,900	19,300	NR	NR
San Diego	Urban	2,900 - 27,500	9,600	NR	NR
Napa	Non-Urban	4,700 - 11,800	7,000	NR	NR
Oildale	Non-Urban	3,100 - 104,000	9,400	NR	NR
Sherman Oaks	Urban	16,000 - 28,000	9,900	NR	NR
Phase I RI 1987 SBAA Air Data (c):					
Station 1 (off-site/upwind)		<500 - 2,800	NC	<350 - 2,800	NC
Stations 2-4 (on-site)		<350 - 10,300	NC	<350 - 28,000	1,000
Station 5 (off-site/downwind)		<450 - 8,700	NC	<450 - 10,500	960

(a) SAI collected four four-hour samples at each sample location on a single day. Samples were analyzed using transmission electron microscopy (TEM). Only fibers with a length:diameter ratio > 3 were counted. Nearly all fibers observed were less than 5 um in length, and most samples had mean diameters less than 1.5 um (SAI 1981).

(b) Geometric means were calculated using one-half the detection limit for non-detected samples.

(c) Phase II indirect data cannot be compared with SAI or Phase I data because the Phase II data are measured in PCMe structures/m3.

NR = Not reported.

NC = Not calculated because of the infrequent detections of asbestos, except for the USEPA Roads Study data for which insufficient information was available to calculate a mean.

TABLE 7-4

SUMMARY OF ASBESTOS CONCENTRATIONS IN SURFACE SOIL
IN THE SOUTH BAY ASBESTOS SITE AREA[a]

Location	Frequency of Detection	Concentration (%)[b]	
		Geometric Mean[c]	Maximum[d]
Residential Areas			
- Town of Alviso[e]	19/72	<1[f]	8
- Summerset Trailer Park[g]	13/26	<1	10
George Mayne School[h]	12/20	1.7	20
Environmental Education Center[i]	11/23	1.8	40
	0/3 (RI samples only)	<1	<1
Vacant Lots[j]	8/43	<1	10
Truckyards[k]	25/39	<1	19
Roads			
- Street Dust	7/13	<1	3
- Streets[m]	69/196	<1	10

[a] Based on surface soil samples collected by Cal/OSHA (1983), DHS (1983), USEPA (1985), Ecology and Environment (1986), Woodward-Clyde Consultants (1986) and during the RI.
[b] Concentrations are reported in PLM area%.
[c] Geometric means were calculated using one half of the detection limit for non-detected samples. Detection limit for all samples except DHS samples is 1%; DHS detection limit is 0.01%.
[d] Maximum may no longer be indicative of on-site surface soil conditions because of emergency response actions.
[e] Includes samples collected from yards and vacant lots in the area bounded by Taylor Street and the Levee to Spreckles Avenue, and State Street to Gold Street. Geometric mean obtained using 12 samples from DHS, 50 samples from USEPA, and 10 samples from the RI.
[f] All values less than one percent will be reported as <1%.
[g] 15 samples from USEPA and 11 samples from DHS.
[h] 20 samples from USEPA; samples were collected prior to paving of schoolyard.
[i] 20 samples from USEPA and 3 samples from the RI.
[j] Includes all vacant lots in non-residential areas; 7 samples from DHS, 36 samples from USEPA, and 9 samples from the RI.
[k] Samples collected primarily from truckyards along State Street; 24 samples from E&E (TMA reanalysis), 11 samples from DHS, and 4 samples from the RI.
[l] 5 samples from the RI, and 8 samples from USEPA.
[m] 176 samples from USEPA, and 20 samples from DHS.

TABLE 7-5

CONCENTRATIONS OF CHEMICALS FOUND IN GROUNDWATER IN THE SOUTH BAY ASBESTOS SITE AREA (a)

Chemical	Frequency of Detection	Concentration (ug/L) (b) Geometric Mean (c)	Maximum
Asbestos	7/7	1,102 mf/L	4,774 mf/L
Soluble Inorganic Compounds			
Aluminum	8/13	102 ug/L	220 ug/L
Cadmium	5/13	<4 (d)	33.1
Arsenic	2/12	<10	14.2
Antimony	3/13	<60	183
Calcium	13/13	75,400	534,000
Barium	12/13	219	2,380
Chromium (totaO)	3/13	<10	33
Copper	9/12	<11	42.4
Cobalt	11/13	<30	174
Cyanide	3/13	<10	60
Iron	13/13	<1,050	57,700
Lead	8/13	9.3	63
Magnesium	13/13	303,000	924,000
Manganese	13/13	1,590	6,850
Mercury	10/13	1.9	18
Nickel	9/13	57	989
Potassium	12/13	59,100	986,000
Silver	3/13	<4	11
Sodium	13/13	3,830,000	11,600,000
Tin		NC	40
Vanadium	6/13	<36	33
Zinc	13/13	65	417
Organic Compounds			
Acetone	2/14	<10	12
2-Butanone	1/14	NC	3
Chloroform	1/14	NC	1
Naphthalene	3/14	<10	11
Bis(2-ethylhexyl)phthalate	1/14	NC	10
Benzyl Alcohol	2/14	<10	2
Endrin	1/13	NC	0.08
Endosulfan Sulfate	1/13	NC	0.09
4-Methylphenol	2/14	<10	3
Pentachlorophenol	1/14	NC	3
Toluene	1/14	NC	1
Trichloroethene	1/14	NC	320

(a) Based on groundwater samples collected by CDM during the RI. Includes data designated by the USEPA data validators as estimated.

(b) Results for all chemicals except asbestos are reported in ug/liter; asbestos results are reported as million fibers/liter (mf/L).

(c) Geometric means were calculated using one half of the detection limit for non-detected samples.

(d) Geometric mean value is lower than the detection limit for this compound. The detection limit is given as an upper bound for the geometric mean.

NC = Not calculated because the analyte was detected in less than 10% of the samples.

TABLE 7-6

REPORTED INDIRECT PHASE II RENANALYSIS STRUCTURE CONCENTRATIONS
AND EQUIVALENT DIRECT CONCENTRATIONS

Sampling Station	Measured Concentration From Phase II Indirect Analysis (PCMe structures/m3)		Equivalent Direct Analysis Concentration: Conversion Factor Range of 2 to 30 (PCMe structures/m3) (a)			
	Geometric Mean	Maximum	Average Case Conv.=1/30	Average Case Conv.=1/2	Maximum Case Conv.=1/30	Maximum Case Conv.=1/2
1: Upwind/Off-Site	147	860	5	73	29	430
2-4: On-Site	1,003	8,500	33	502	283	4,250
5: Downwind/Off Site	210	1,100	7	105	37	550

(a) Concentrations reported from Phase II as PCMe structures/m3 are considered most representative of concentrations relevant for risk assessment. Indirect concentrations are converted to equivalent direct concentrations by dividing by a range of possible conversion factors, 2-30 (ESAT 1988).

TABLE 7-7

EXCESS LIFETIME INHALATION CANCER RISKS FOR ASBESTOS IN AMBIENT AIR

(Based on Phase II Reanalysis Data)*

Cancer Risk Estimate	Sampling Location: Station 1 upwind/ off-site	Stations 2-4 on-site	Station 5 downwind/ off-site
Excess Lifetime Mesothelioma Risk: Females			
Average Case	$1x10^{-6}$ - $2x10^{-5}$	$1x10^{-5}$ - $1x10^{-4}$	$2x10^{-6}$ - $3x10^{-5}$
Maximum Case	$8x10^{-6}$ - $1x10^{-4}$	$8x10^{-5}$ - $1x10^{-3}$	$1x10^{-5}$ - $2x10^{-4}$
Excess Lifetime Mesothelioma Risk: Males			
Average Case	$1x10^{-6}$ - $1x10^{-5}$	$6x10^{-6}$ - $1x10^{-4}$	$1x10^{-6}$ - $2x10^{-5}$
Maximum Case	$6x10^{-6}$ - $8x10^{-5}$	$5x10^{-5}$ - $8x10^{-4}$	$7x10^{-6}$ - $1x10^{-4}$
Excess Lifetime Lung Cancer Risk: Females			
Average Case	$3x10^{-7}$ - $4x10^{-6}$	$2x10^{-6}$ - $3x10^{-5}$	$4x10^{-7}$ - $5x10^{-6}$
Maximum Case	$1x10^{-6}$ - $2x10^{-5}$	$1x10^{-5}$ - $2x10^{-4}$	$2x10^{-6}$ - $3x10^{-5}$
Excess Lifetime Lung Cancer Risk: Males			
Average Case	$9x10^{-7}$ - $1x10^{-5}$	$6x10^{-6}$ - $9x10^{-5}$	$1x10^{-6}$ - $2x10^{-5}$
Maximum Case	$5x10^{-6}$ - $7x10^{-5}$	$5x10^{-5}$ - $7x10^{-4}$	$6x10^{-6}$ - $9x10^{-5}$

*Calculated based on Phase II reanalysis air data and health risk Table 6-1 in Appendix J. For each case (average and maximum), a range of risks is presented to correspond with the range in conversion factors between indirect PCMe structures and equivalent direct PCMe structures (2-30).

TABLE 7-8

ESTIMATION OF EXPOSURE POINT CONCENTRATIONS
FOR INHALATION OF ASBESTOS AT VARIOUS
LOCATIONS IN CALIFORNIA (a)

Sample Location	Asbestos Source Type	Asbestos Air Concentration (TEM f/m3)		Equivalent Asbestos PCM Air Concentration (PCM f/m3)	
		Geometric Mean	Maximum	Average Case	Maximum Case
Sonora	Natural deposits	5,200	14,400	88	245
San Jose	Industrial	19,300	23,900	328	406
San Diego	Urban	9,600	27,500	163	468
Napa	Non-urban	7,000	11,800	119	201
Oildale	Non-urban	9,400	104,000	160	1,768
Sherman Oaks	Urban	9,900	28,000	168	476

(a) Source of ambient air data: SAI (1983).
(b) Direct TEM f/m3 concentrations were converted to equivalent direct PCM f/m3 by multiplying by a factor of 0.017 (based on NRC 1983).

TABLE 7-9

EXCESS LIFETIME INHALATION CANCER RISKS
AT VARIOUS LOCATIONS IN CALIFORNIA[a]

Sample Location	Excess Lifetime Mesothelioma Risk		Excess Lifetime Lung Cancer Risk	
	Females	Males	Females	Males
Sonora				
Average Case	$2x10^{-5}$	$2x10^{-5}$	$4x10^{-6}$	$1x10^{-5}$
Maximum Case	$7x10^{-5}$	$5x10^{-5}$	$1x10^{-5}$	$4x10^{-5}$
San Jose				
Average Case	$9x10^{-5}$	$6x10^{-5}$	$2x10^{-5}$	$6x10^{-5}$
Maximum Case	$1x10^{-4}$	$8x10^{-5}$	$2x10^{-5}$	$7x10^{-5}$
San Diego				
Average Case	$5x10^{-5}$	$3x10^{-5}$	$8x10^{-6}$	$3x10^{-5}$
Maximum Case	$1x10^{-4}$	$9x10^{-5}$	$2x10^{-5}$	$8x10^{-5}$
Napa				
Average Case	$3x10^{-5}$	$2x10^{-5}$	$6x10^{-6}$	$2x10^{-5}$
Maximum Case	$6x10^{-5}$	$4x10^{-5}$	$1x10^{-5}$	$3x10^{-5}$
Oildale				
Average Case	$4x10^{-5}$	$3x10^{-5}$	$8x10^{-6}$	$3x10^{-5}$
Maximum Case	$5x10^{-4}$	$3x10^{-4}$	$9x10^{-5}$	$3x10^{-4}$
Sherman Oaks				
Average Case	$5x10^{-5}$	$3x10^{-5}$	$8x10^{-6}$	$3x10^{-5}$
Maximum Case	$1x10^{-4}$	$9x10^{-5}$	$2x10^{-5}$	$8x10^{-5}$

[a] Excess lifetime cancer risks were calculated from Table 6-1 in Appendix J (USEPA 1986a) by scaling to differences in air concentrations for lifetime risk estimates. Source of ambient air data was SAI (1983).

TABLE 7-10

EXPOSURE PARAMETER VALUES USED TO ESTIMATE INHALATION EXPOSURES TO RESIDENTS NEAR UNPAVED ROADS

Exposure Parameter	Residents Living Near Unpaved Truckyards	
	Average Case	Maximum Case
Age Period of Exposure (years)	Lifetime	Lifetime
Total Years Exposed (years)	70	70
Frequency of Exposure (events/yr)	100	250 (a)
Duration of Event (hours/event)	2	8

(a) Assumes exposure occurs 2 times/week for 50 weeks per year for the average case (100 events/year) and 5 times/week for the maximum case (250 events/year).

TABLE 7-11

EXCESS LIFETIME INHALATION CANCER RISKS ASSOCIATED WITH RESIDENTS LIVING NEAR UNPAVED ROADS

Location	Gender[a]	Endpoint	Individual Excess Lifetime Cancer Risk	
			Average Case	Maximum Case
Residents				
- Truckyards	Female	Mesothelioma	NC	$>3x10^{-3}$[b]
	Female	Lung Cancer	NC	$>3x10^{-3}$
	Male	Mesothelioma	NC	$>3x10^{-3}$
	Male	Lung Cancer	NC	$>3x10^{-3}$
- Roadways	Female	Mesothelioma	NC	$>3x10^{-3}$
	Female	Lung Cancer	NC	$>3x10^{-3}$
	Male	Mesothelioma	NC	$>3x10^{-3}$
	Male	Lung Cancer	NC	$>3x10^{-3}$
- Street Dust	Female	Mesothelioma	NC	$>3x10^{-3}$
	Female	Lung Cancer	NC	$>3x10^{-3}$
	Male	Mesothelioma	NC	$>3x10^{-3}$
	Male	Lung Cancer	NC	$>3x10^{-3}$

NC = Risk could not be calculated.

[a] For exposure scenarios with continuous exposures greater than a year, risks were estimated from the health risk table in Appendix J for males and females and mesothelioma and lung cancer. For continuous exposure durations less than one year, risks were estimated using the unit risk factor and are representative of both males and females.

[b] Risk table is not quantified for risks greater than $3x10^{-3}$.

TABLE 7-12

EXPOSURE PARAMETER VALUES USED TO ESTIMATE EXPOSURES VIA DIRECT CONTACT WITH SOIL AND SUBSEQUENT INCIDENTAL INGESTION

Exposure Parameter	Scenario 1 Children in Non-Residential Areas		Scenario 2 Lifetime Exposure to Residential Soil	
	Average Case	Maximum Case	Average Case	Maximum Case
Age Period of Exposure (years)	5-11	5-16	Lifetime	Lifetime
Total Years Exposed (years)	7	12	70	70
Average Body Weight (kg) (a)	29	39	62	62
Frequency of Exposure (events/yr)	104	156 (c)	104	156 (c)
Incidental Soil Ingestion Rate (mg/event) (b)	64	241	54	145

(a) Average body weight for period of exposure based on a weighted average of age-specific body weights presented in USEPA (1985).

(b) Incidental soil ingestion rate is a weighted average over total exposure period based on data in LaGoy (1987).

(c) Assumes exposure occurs 2 times/week every week of the year for the average case (104 events/year) and 3 times/week every week of the year for the maximum case (156 events/year).

TABLE 7-13

ESTIMATED CHRONIC DAILY INTAKES ASSOCIATED WITH INCIDENTAL INGESTION OF ASBESTOS FROM SURFACE SOIL BY INDIVIDUALS IN THE SOUTH BAY ASBESTOS SITE AREA

RECEPTOR AND EXPOSURE LOCATION	EXPOSURE POINT CONCENTRATION (PLM area%)		ESTIMATED CHRONIC DAILY INTAKE (a) (mg/kg/day)	
	Geometric Mean	Maximum	Average Case	Plausible Maximum Case
Children playing in:				
Non-residential Areas (i.e., vacant lots)	<1	10	NC	4.53E-02
Residents:				
Town of Alviso	<1	8	NC	8.00E-02
Summerset Trailer Park	<1	10	NC	1.00E-01

The notation "1E-06" is equivalent to 1x10-6 or 0.000001

<1 = Calculated geometric mean is less than the detection limit.
NC = Not calculated
(a) Exposure assumptions differ for school children, trespassers, workers and residents. See text for description of exposure assumptions.

TABLE 7-14

EXCESS LIFETIME CANCER RISKS FOR INDIVIDUALS EXPOSED TO ASBESTOS VIA INCIDENTAL INGESTION OF SOIL IN THE SOUTH BAY ASBESTOS SITE AREA

RECEPTOR AND EXPOSURE LOCATION	ESTIMATED CHRONIC DAILY INTAKE (mg/kg/day)		CANCER POTENCY FACTOR (mg/kg/day)-1	EXCESS UPPER BOUND LIFETIME CANCER RISK	
	Average Case	Plausible Maximum Case		Average Case	Maximum Plausible Case
Children:					
Non-Residential Areas (i.e., vacant lots)	NC	4.53E-02	6.3E-04	NC	3E-05
Residents:					
Town of Alviso	NC	8.00E-02	6.3E-04	NC	5E-05
Summerset Trailer Park	NC	1.00E-01	6.3E-04	NC	6E-05

The notation "1E-06" is equivalent to 1x10-6 or 0.000001.
NC = Not calculated.
(a) Listed potency factor for asbestos was derived from the unit risk factor of 1.4E-13 liter/fiber for ingestion of drinking water. EPA has not developed a cancer potency factor or unit risk factor for ingestion of asbestos from other media such as soil. This conversion was done only for the purposes of this risk assessment to allow quantitative evaluation of this pathway.

TABLE 7-15

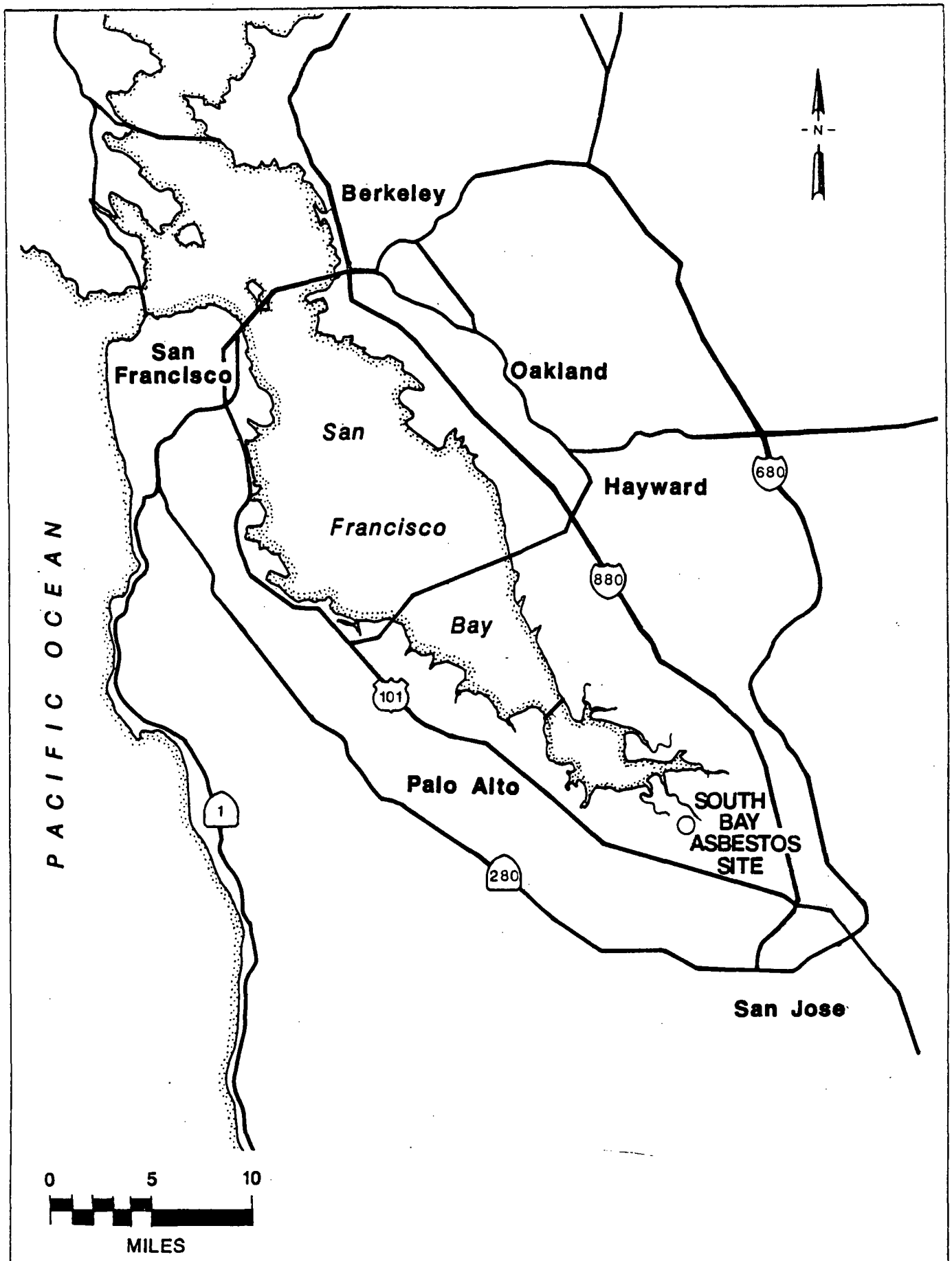
SUMMARY OF EXCESS INDIVIDUAL LIFETIME CANCER RISKS FOR EXPOSURE TO ASBESTOS AT THE SOUTH BAY SITE

Exposure Pathway*	Average Case	Maximum Case
Inhalation - Ambient Air		
Station 1 (off-site/upwind)	$1 \times 10^{-6} - 3 \times 10^{-5}$	$7 \times 10^{-6} - 2 \times 10^{-4}$
Stations 2-4 (on-site)	$8 \times 10^{-6} - 2 \times 10^{-4}$	$6 \times 10^{-5} - 2 \times 10^{-3}$
Station 5 (off-site/downwind)	$1 \times 10^{-6} - 5 \times 10^{-5}$	$9 \times 10^{-6} - 3 \times 10^{-4}$
Inhalation - Activity Generated Airborne Asbestos		
Truck Traffic on Unpaved Surfaces		
Residents Inhaling:		
Dust from Truckyards	NC	$>3 \times 10^{-3}$
Dust from Unpaved Roadways	NC	$>3 \times 10^{-3}$
Street Dust from Paved Roadways	NC	$>3 \times 10^{-3}$
Ingestion of Soil		
Children Playing on Non-Residential Areas (i.e., vacant lots)	NC	3×10^{-5}
Lifetime Exposure from:		
Alviso Yards	NC	5×10^{-5}
Trailer Park Yards	NC	6×10^{-5}

NC = Not calculated because geometric mean soil level was below the detection limit.

* The potential size of the populations exposed are estimated to be: <560-1,870 for inhalation of asbestos in ambient air and ingestion exposure to asbestos in soil; <150-600 for children/teenager exposure pathways; <100-1,000 for adult gardening scenarios; <560-1,870 for inhalation of vehicle-generated dusts by residents. The potential size of the worker population is not known.

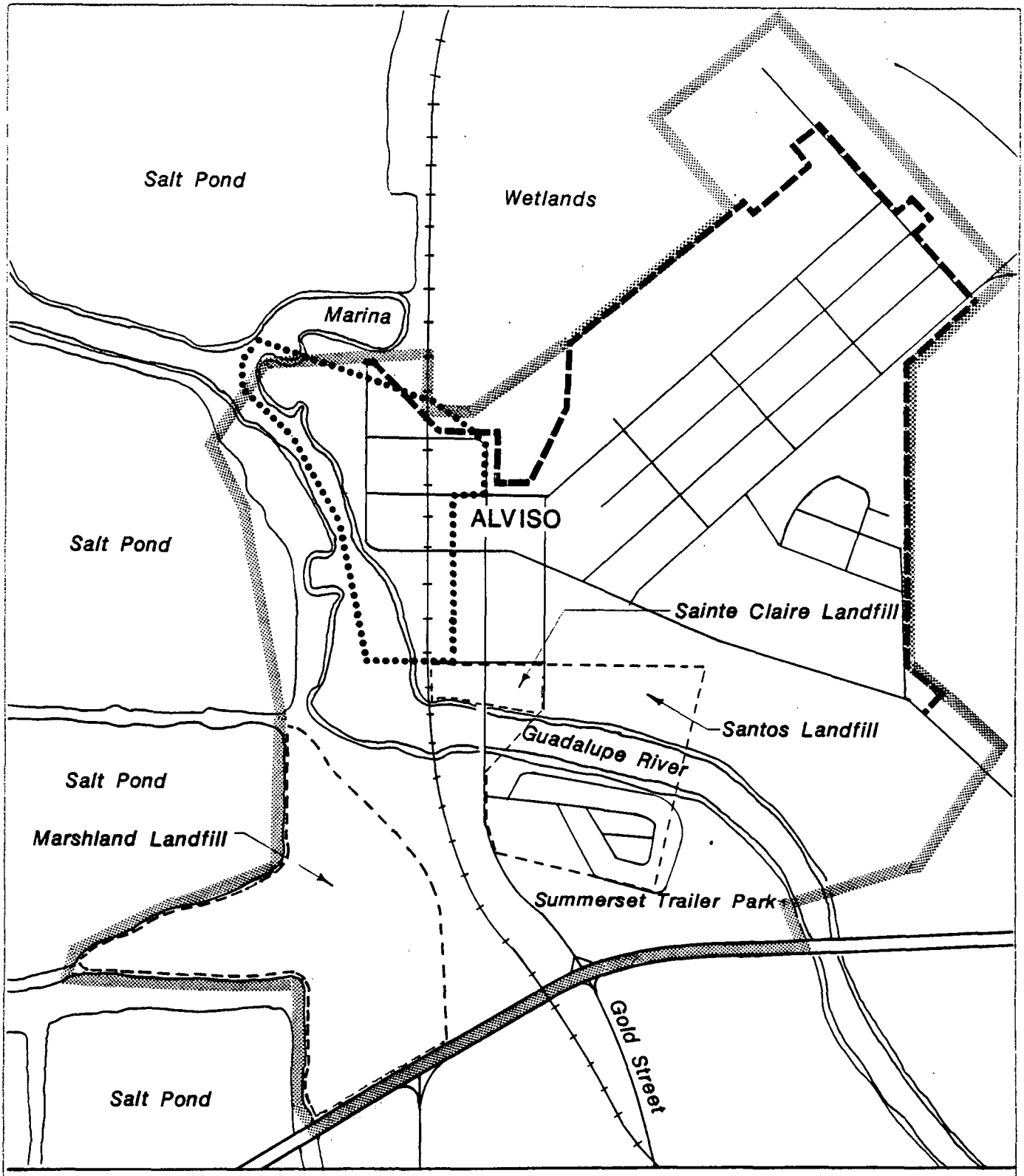
FIGURES



Project No. 288-RI1	South Bay Asbestos Site Camp Dresser & McKee Inc.	REGIONAL LOCATION MAP	Figure 1-1

N84939

DATAPRINT



LEGEND

- Historic District Boundary
- Site Boundary
- Landfill Boundary
- Existing Ring Levee

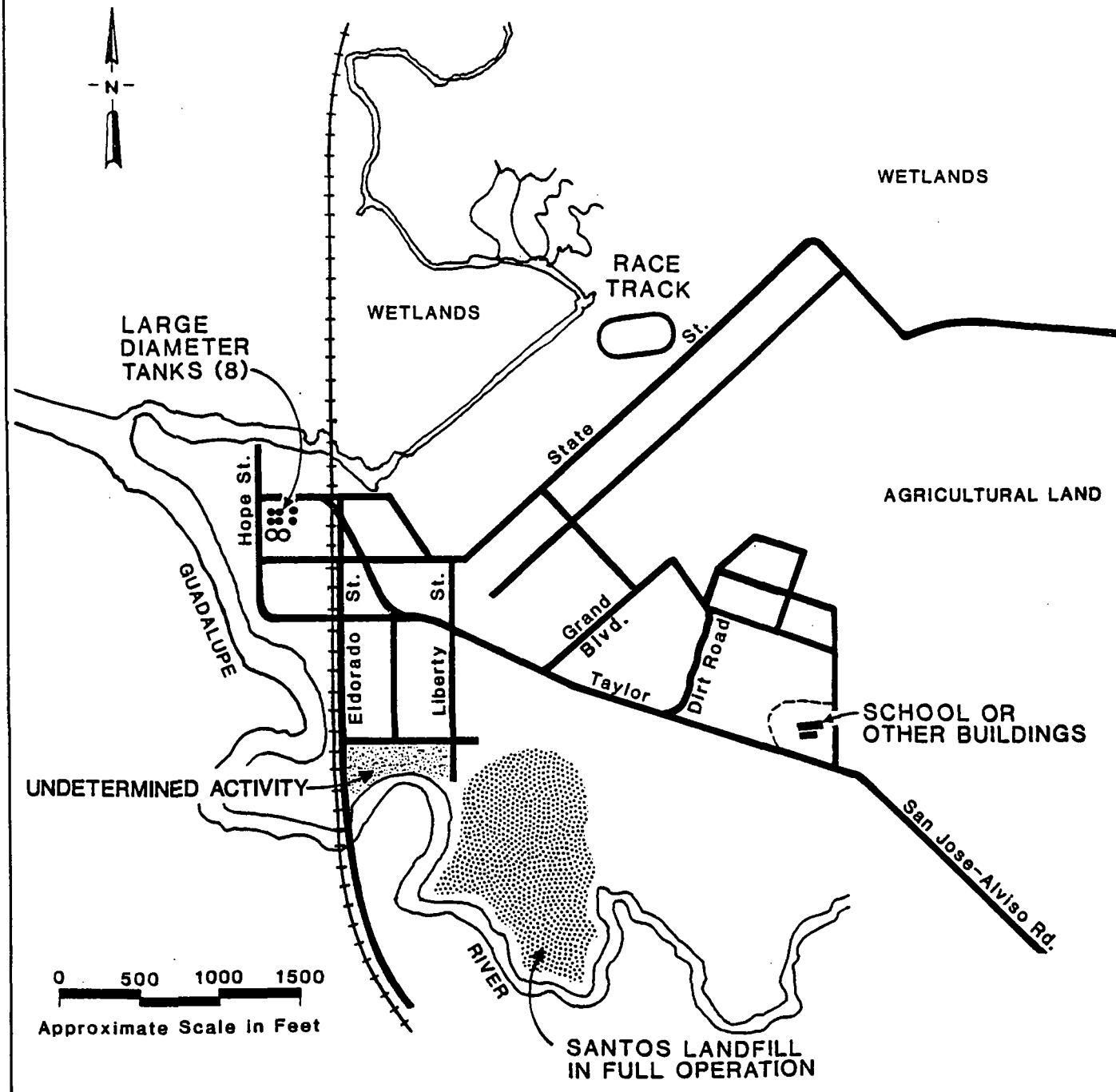
0 500 1000 2000

FEET

Project No.	South Bay Asbestos Site	SITE LOCATION MAP	Figure
288-RI1	Camp Dresser & McKee Inc.		1-2

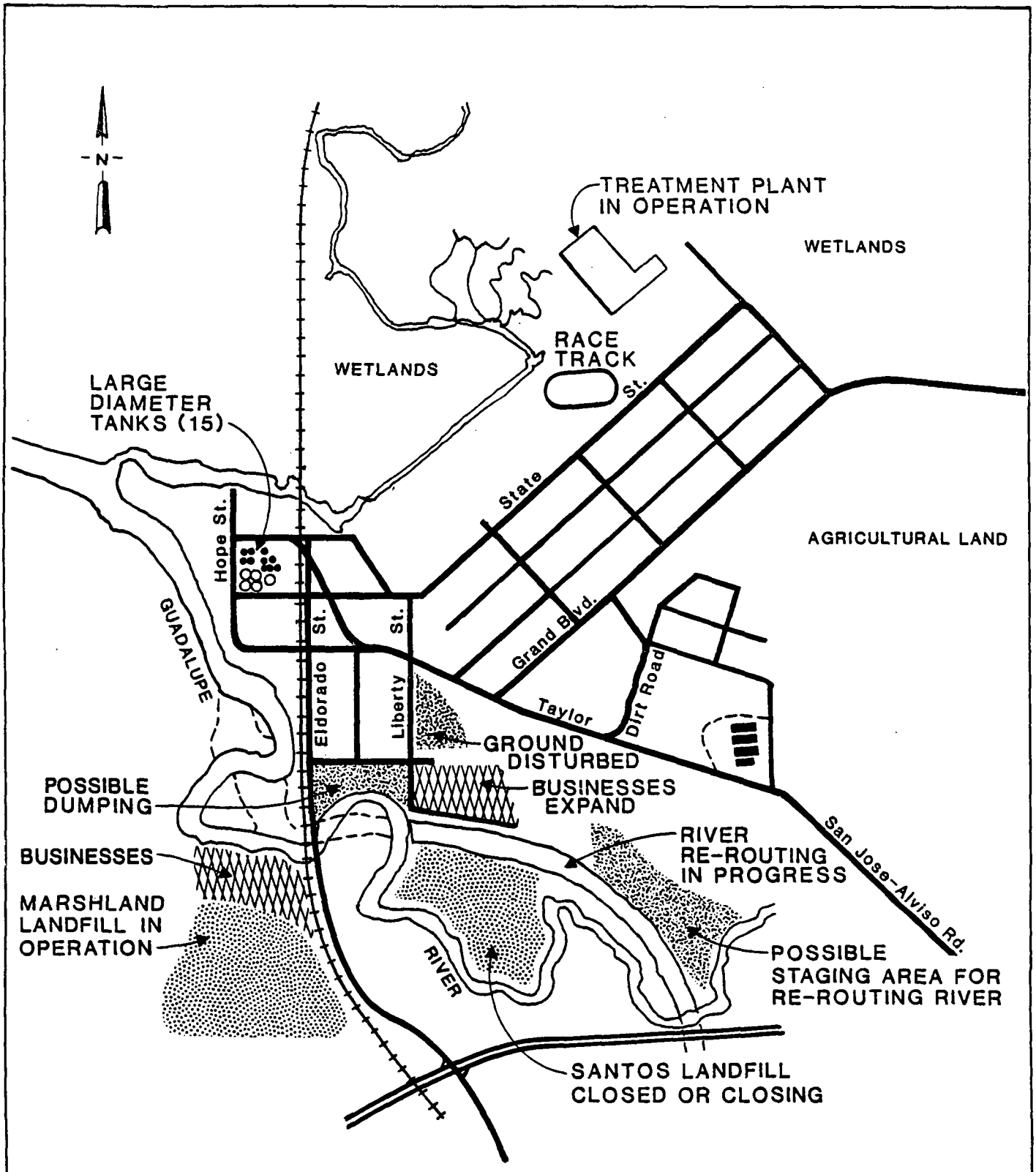
N02280

DATAPRINT



NOTE: This Historical Record is Based on Aerial Photo Interpretation. Information Presented on this Figure is an Approximation only. Photos provided by EPA.

Project No. 288-RI1	South Bay Asbestos	Historical Record	Figure
	Camp Dresser & McKee	JUNE 9, 1956	1-3

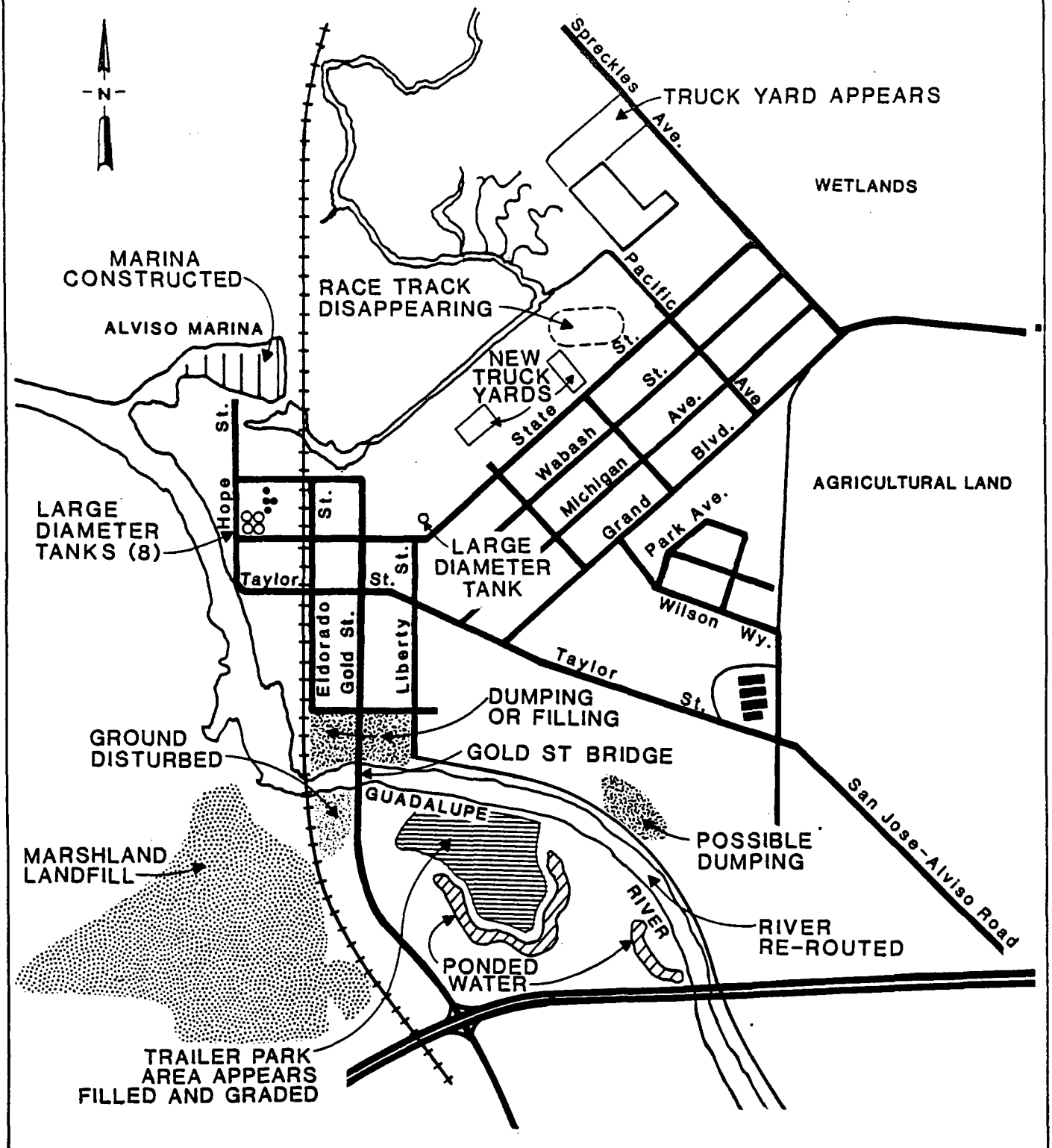


0 500 1000 1500

Approximate Scale in Feet

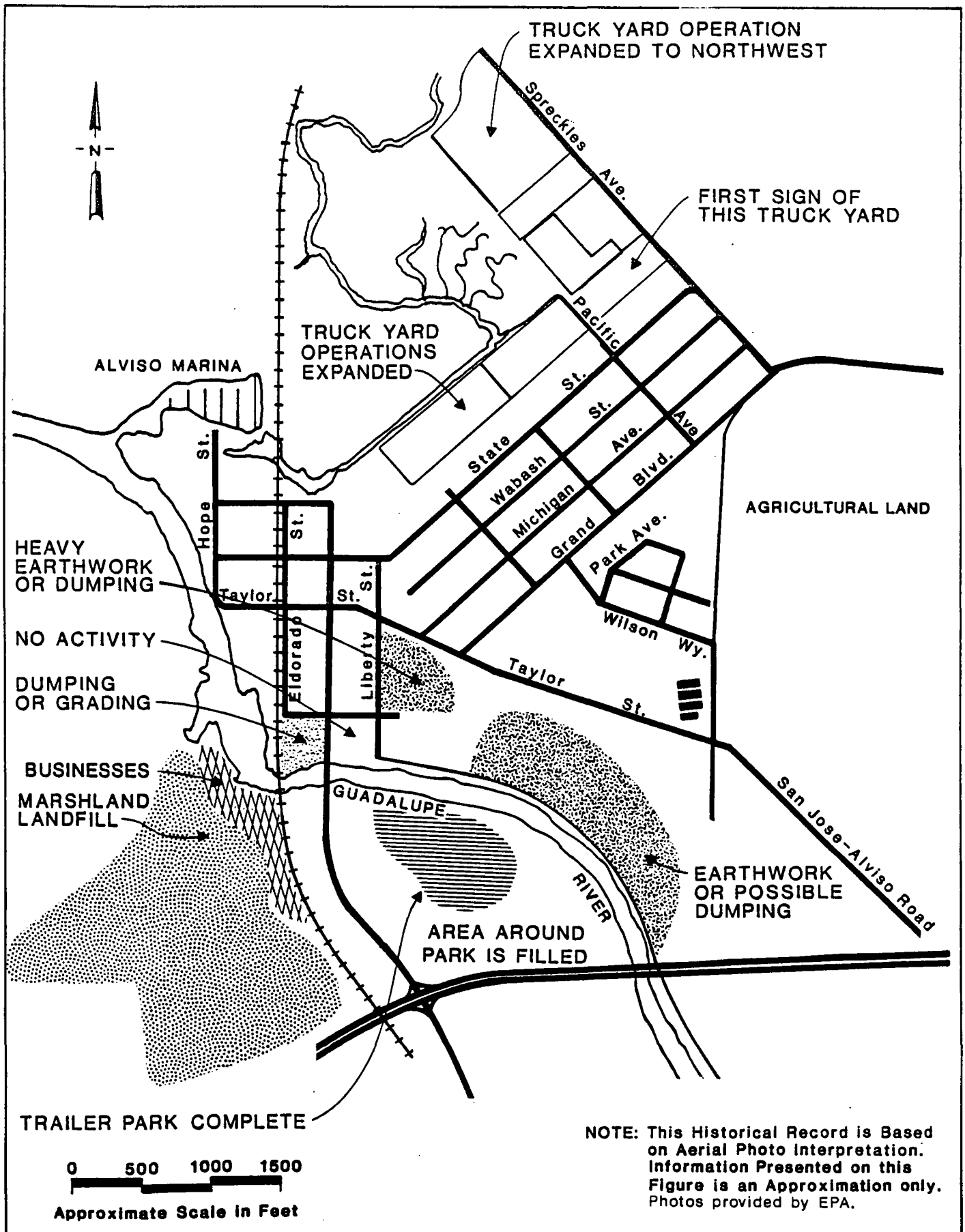
NOTE: This Historical Record is Based on Aerial Photo Interpretation. Information Presented on this Figure is an Approximation only. Photos provided by EPA.

Project No.	South Bay Asbestos	Historical Record	Figure
288-RI1	Camp Dresser & McKee	OCTOBER 13, 1963	1-4



NOTE: This Historical Record is Based on Aerial Photo Interpretation. Information Presented on this Figure is an Approximation only. Photos provided by EPA.

Project No.	South Bay Asbestos	Historical Record	Figure
288-RI1	Camp Dresser & McKee	JUNE 13, 1968	1-5



NOTE: This Historical Record is Based on Aerial Photo Interpretation. Information Presented on this Figure is an Approximation only. Photos provided by EPA.

Project No. 288-RI1	South Bay Asbestos	Historical Record	Figure
	Camp Dresser & McKee	MAY 28, 1982	1-6

PARTIALLY SCANNED
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See document # 2215569
for partially scanned image(s).
FIGURE C-1
FIGURE 2-1 TO 2-2
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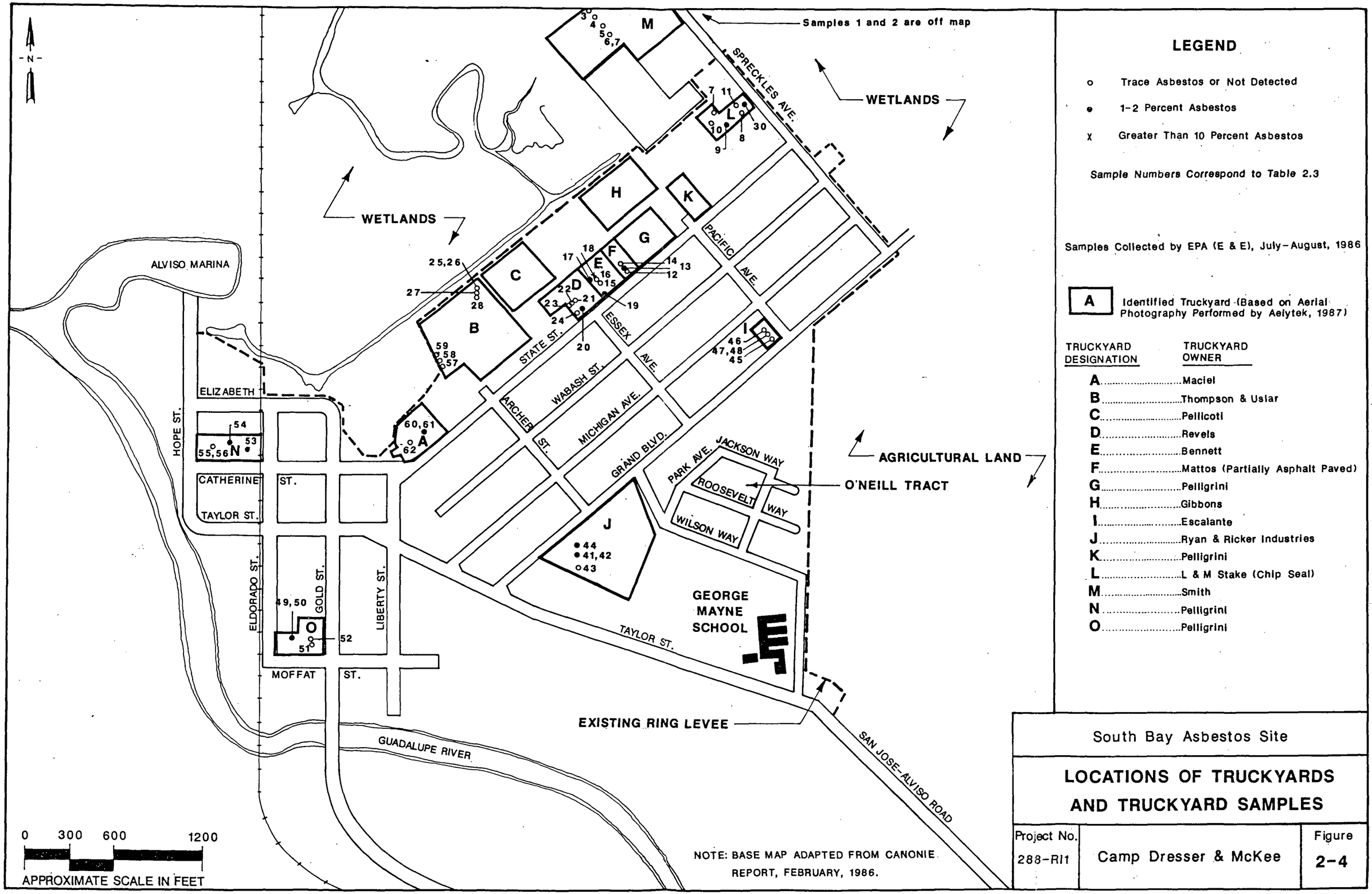


LEGEND

- o Trace Asbestos or Not Detected
- • 1-2 Percent Asbestos
- X Greater Than 10 Percent Asbestos

Sample Numbers Correspond to Table 2.3

Samples Collected by EPA (E & E), July-August, 1986

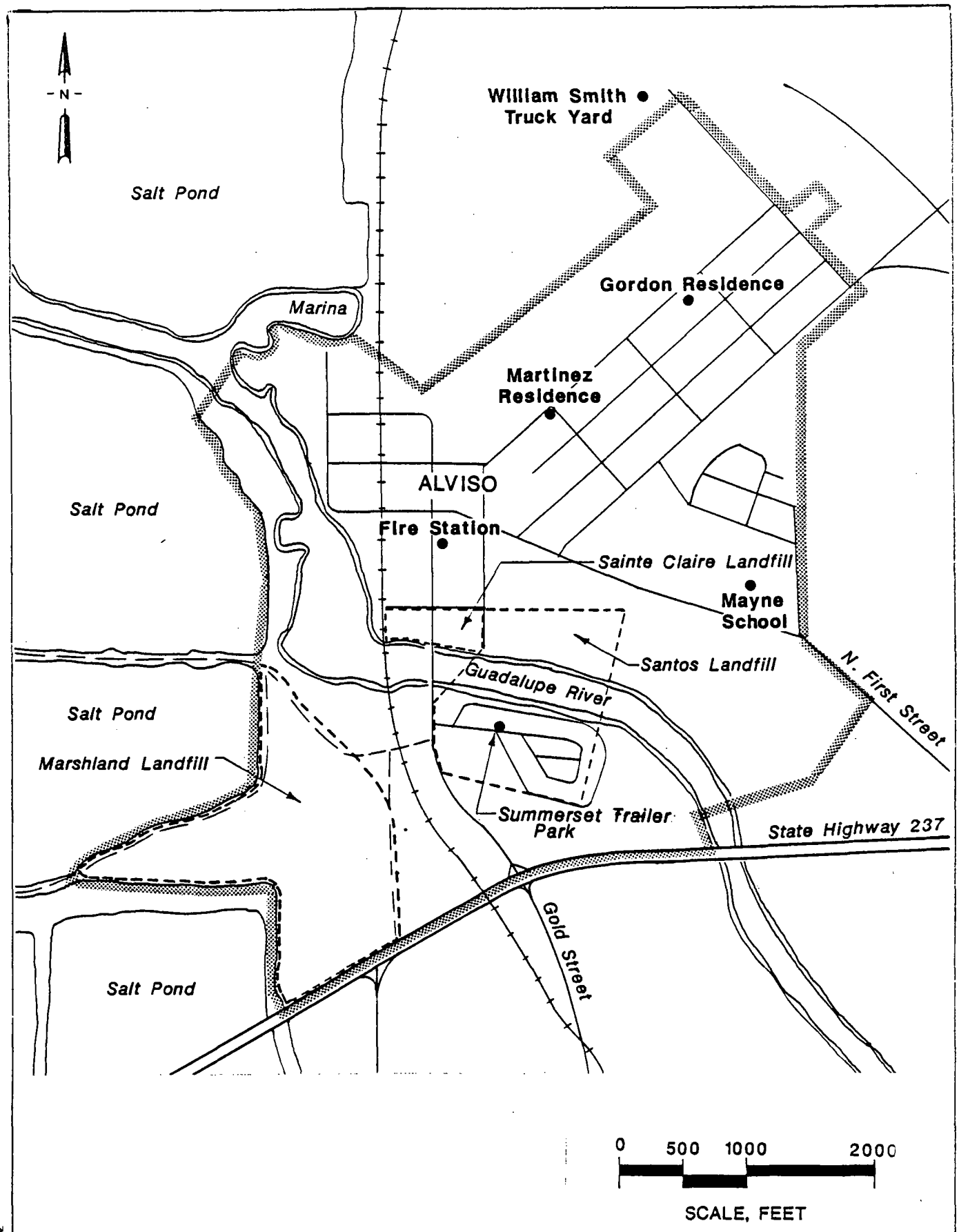
A Identified Truckyard (Based on Aerial Photography Performed by Aelytek, 1987)

TRUCKYARD DESIGNATION	TRUCKYARD OWNER
A	Maciel
B	Thompson & Uslar
C	Pellicoti
D	Revels
E	Bennett
F	Mattos (Partially Asphalt Paved)
G	Pelligrini
H	Gibbons
I	Escalante
J	Ryan & Ricker Industries
K	Pelligrini
L	L & M Stake (Chip Seal)
M	Smith
N	Pelligrini
O	Pelligrini

South Bay Asbestos Site

LOCATIONS OF TRUCKYARDS AND TRUCKYARD SAMPLES

Project No.		Figure
288-RI1	Camp Dresser & McKee	2-4

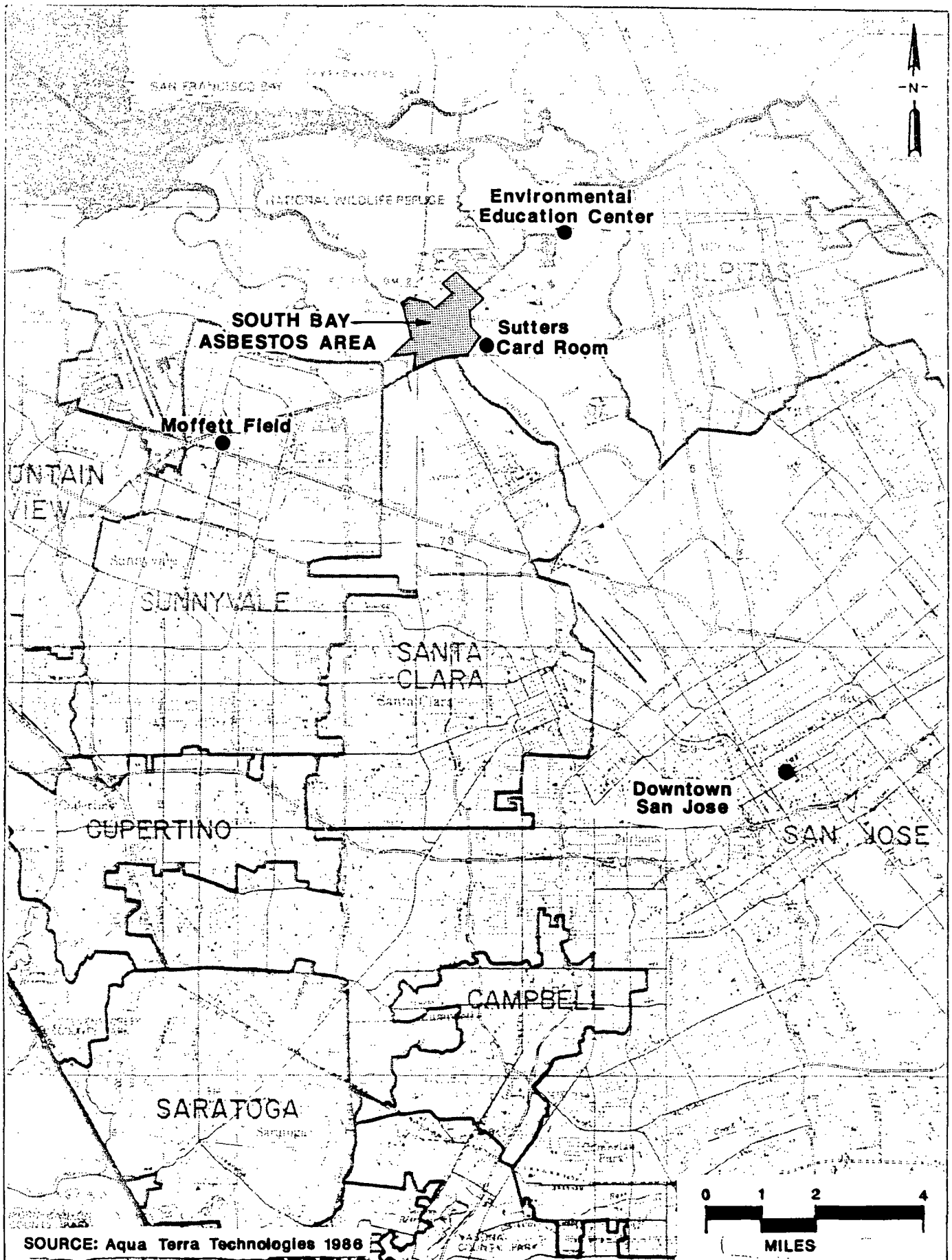


SOURCE: Aqua Terra Technologies, 1986

Project No. 288-RI1	South Bay Asbestos Site	DHS AIR SAMPLING LOCATIONS WITHIN ALVISO	Figure 2-5
	Camp Dresser & McKee Inc.		

N0228Q

DATAPRINT

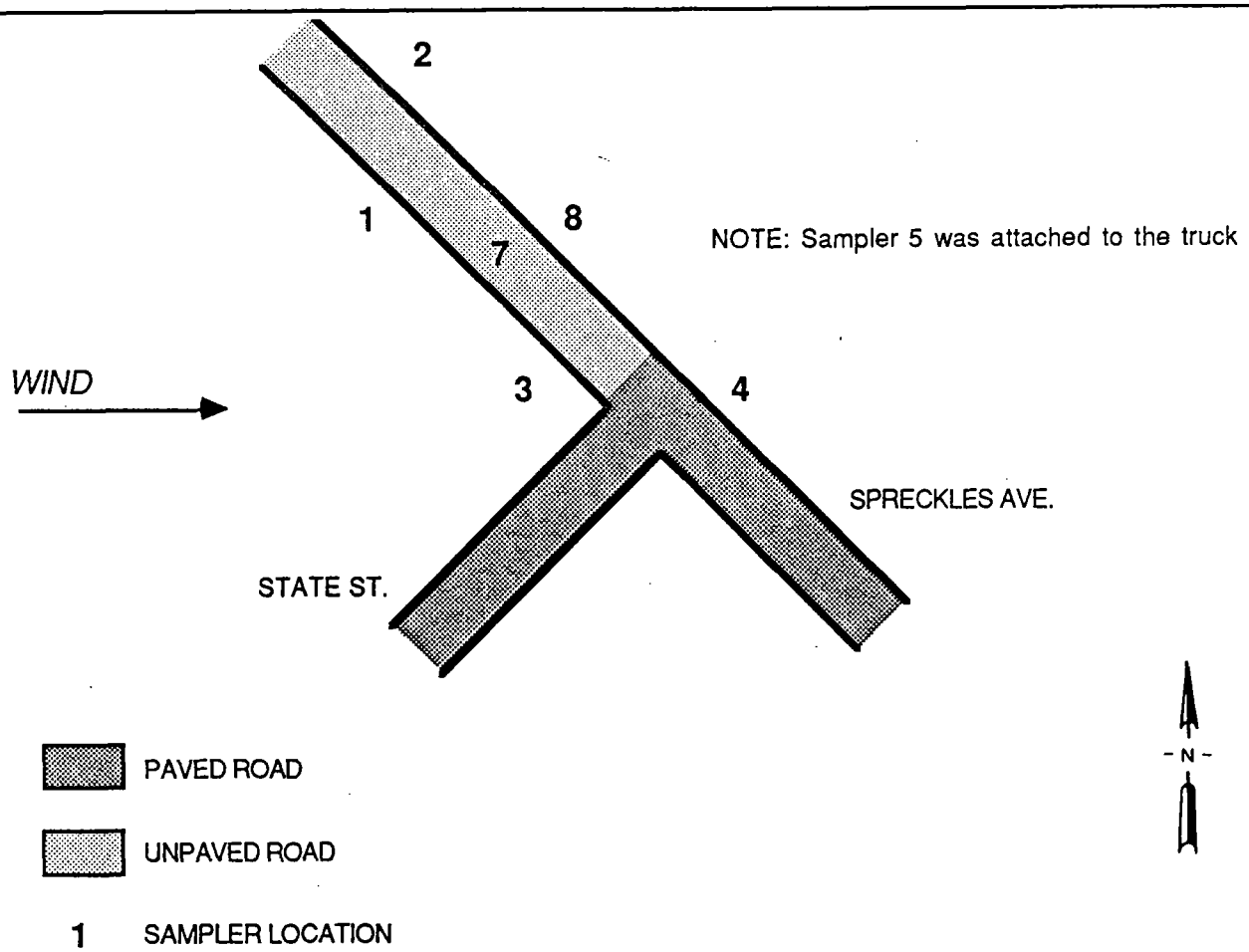


SOURCE: Aqua Terra Technologies 1986

Project No.	South Bay Asbestos Site	DHS AIR SAMPLING LOCATIONS OUTSIDE OF ALVISO	Figure
288-RI1	Camp Dresser & McKee Inc.		2-6

N02280

DATAPRINT



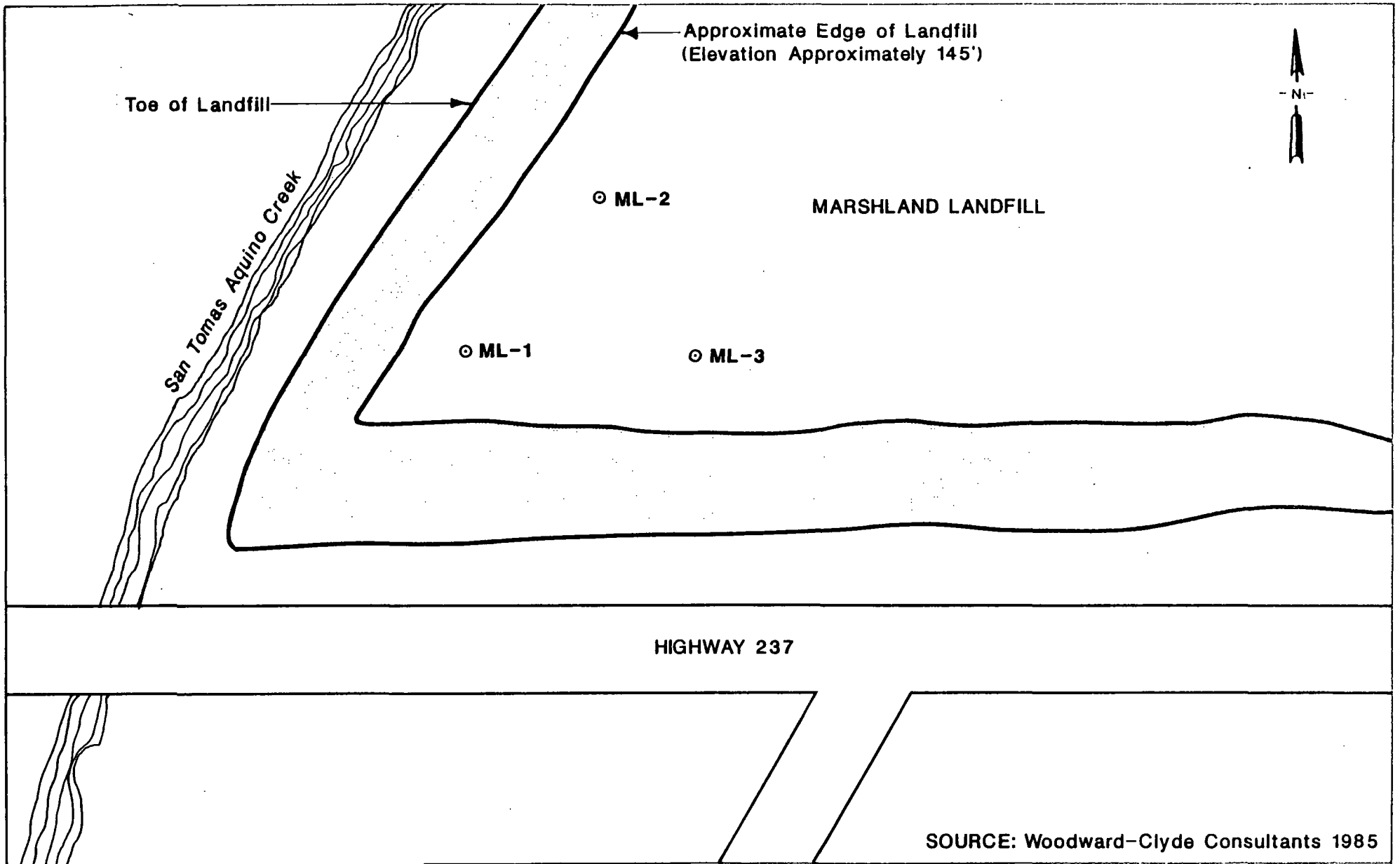
AIR SAMPLE RESULTS

Sample No.	Total Dust (mg/M³)	Fibers/cc
1	0.34	0
2	4.02	0.223
3	0.53	0.018
4	1.38	0.055
5	2.37	0.107

SOIL SAMPLE RESULTS

Sample No.	Asbestos Percent / Type	Other components Percent / Type
7	10% Serpentine	5-10% Iron Oxide 20-30% Clay 40-50% Quartz
8	3-5% Chrysotile 60-70% Serpentine	20-25% Quartz

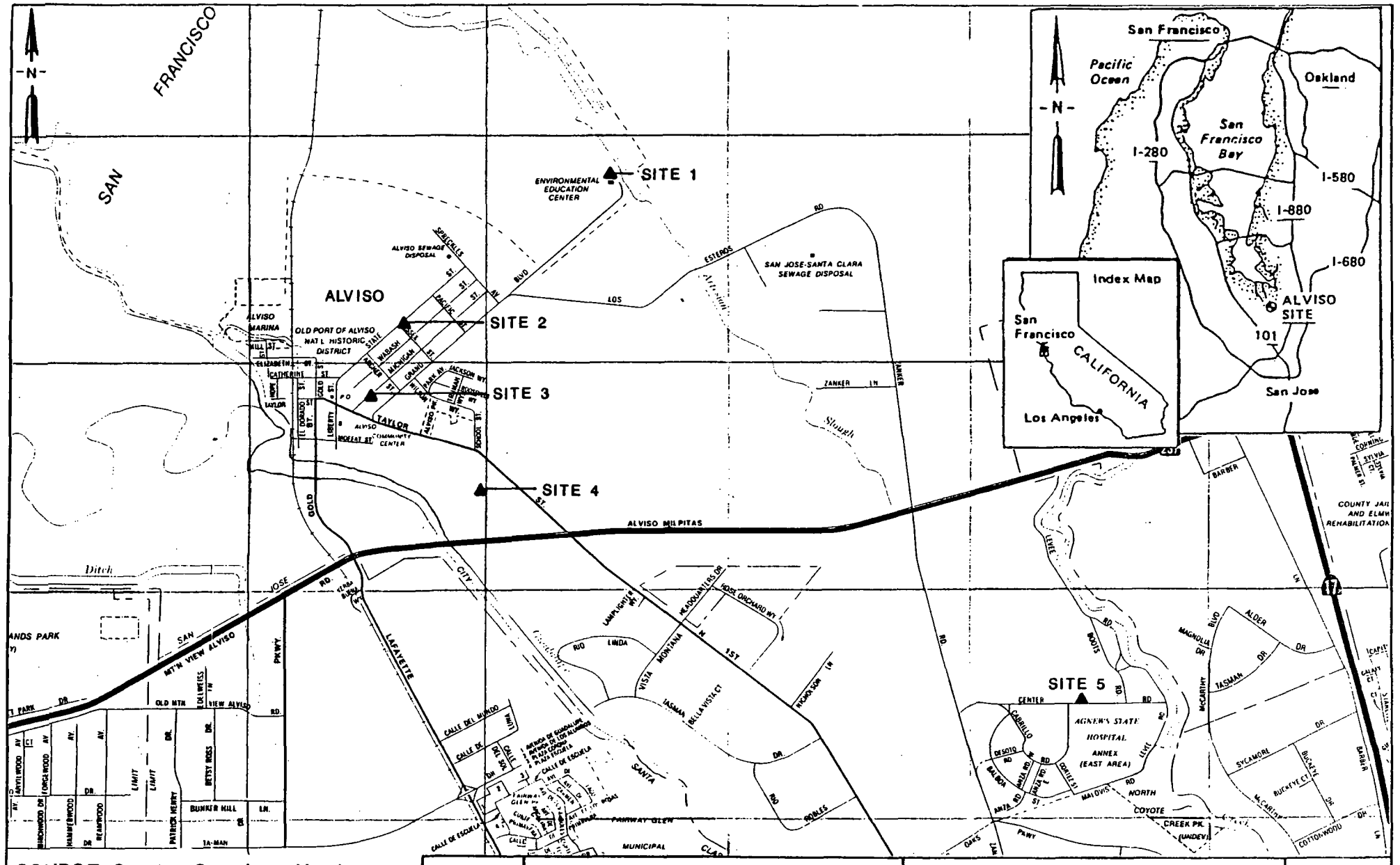
Project No. 288-RI1	South Bay Asbestos Site	DHS TRAFFIC SIMULATION RESULTS	Figure 2-7
	Camp Dresser & McKee		



SOURCE: Woodward-Clyde Consultants 1985

Project No.	South Bay Asbestos Site	SITE PLAN OF BORING LOCATIONS MARSHLAND LANDFILL	Figure
288-RI1	Camp Dresser & McKee Inc.		2-8

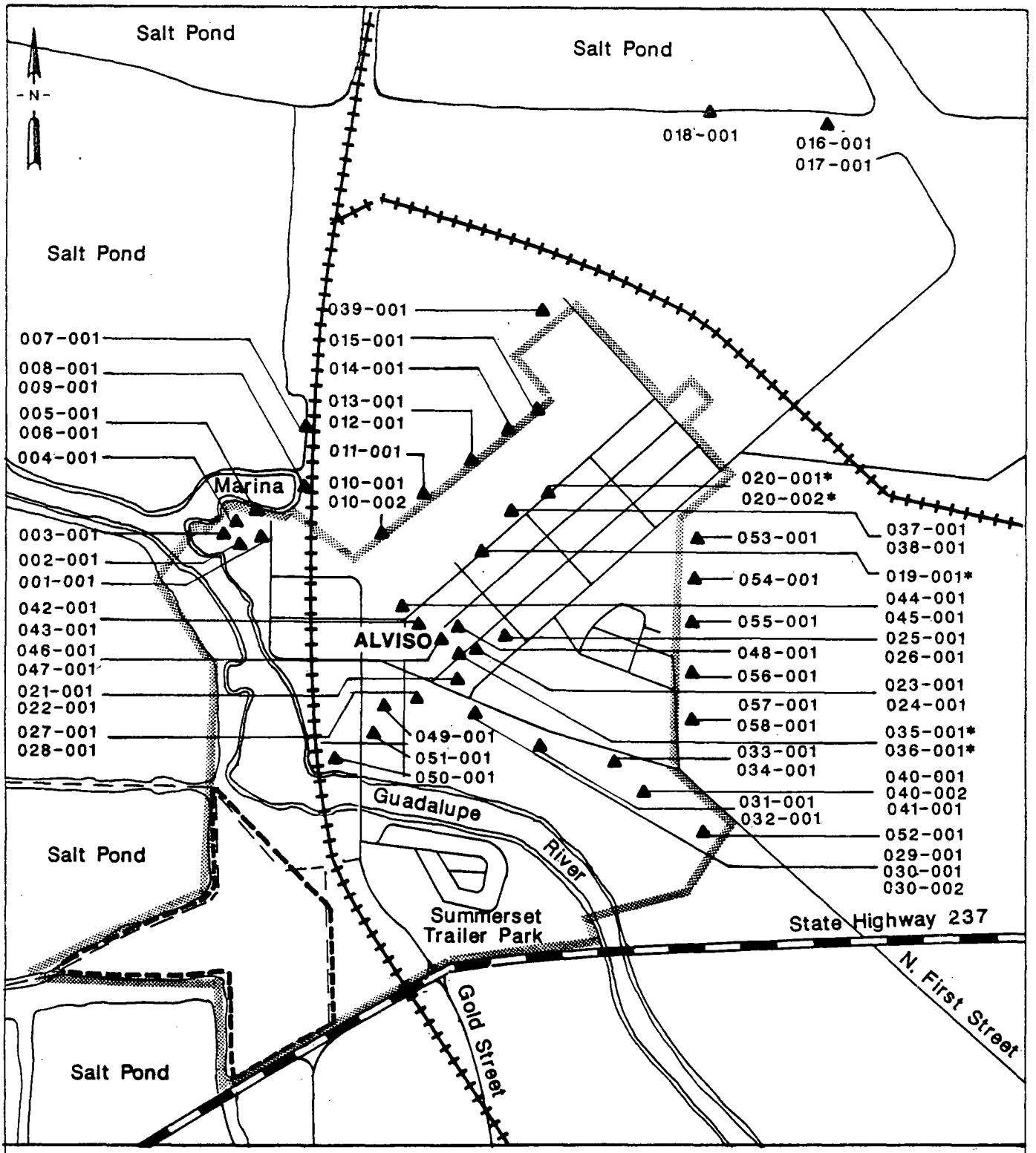
DATAPRINT N02280



SOURCE: Greater San Jose Northern Area AAA Map

FEET 0 1000 2000 4000

Project No.	South Bay Asbestos Site	AIR SAMPLING LOCATIONS	Figure
288-RI1	Camp Dresser & McKee Inc.		3-1



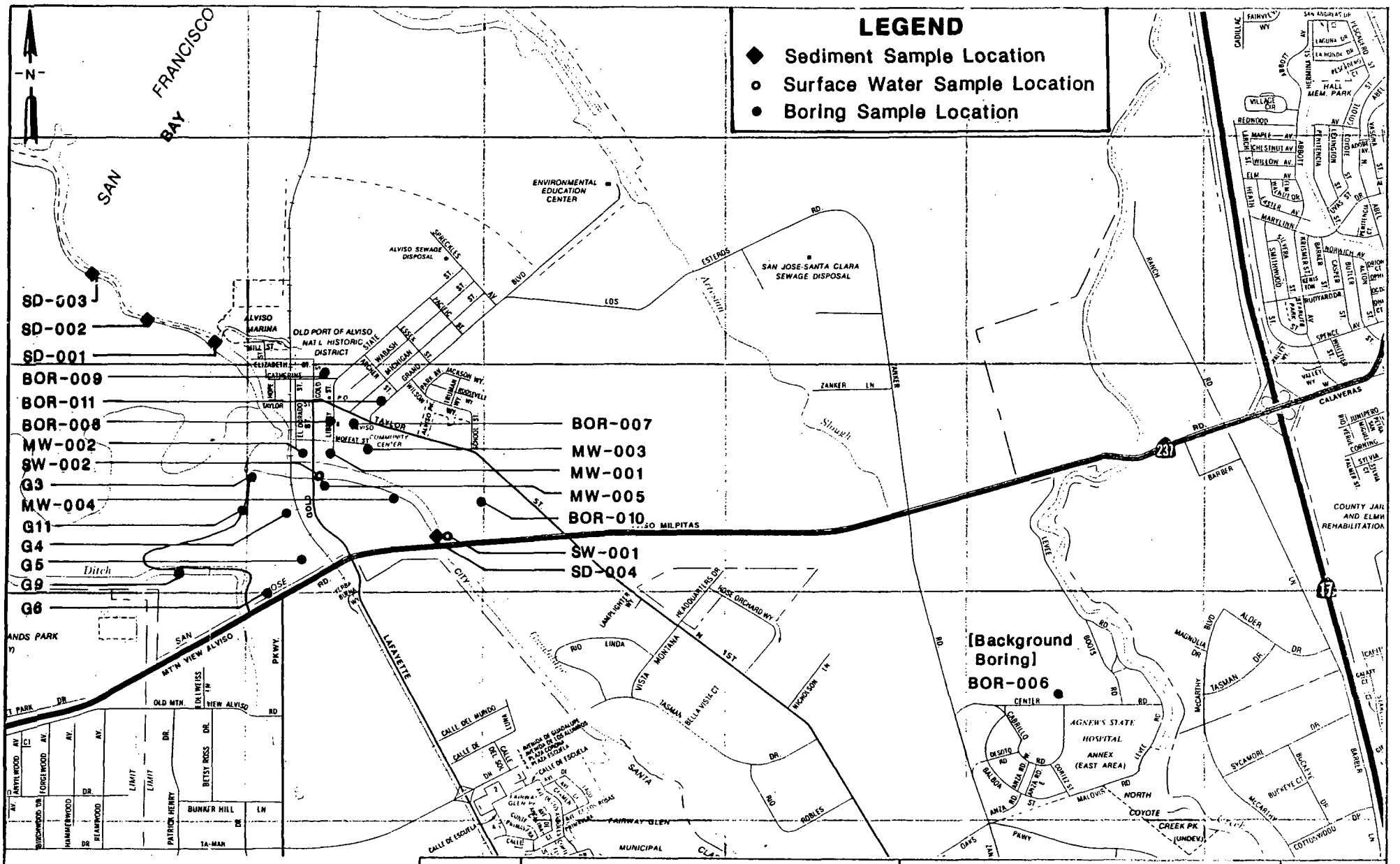
LEGEND

- Site Boundary
- Access Roads
- Marshland Landfill Boundary
- * Street Dust
- ▲ Surface Soil Samples

0 5 10 20
HUNDREDS OF FEET

Project No.	South Bay Asbestos Site	SURFACE SOIL SAMPLE LOCATIONS	Figure
288-RI1	Camp Dresser & McKee Inc.		3-2

N84939
DATAPRINT

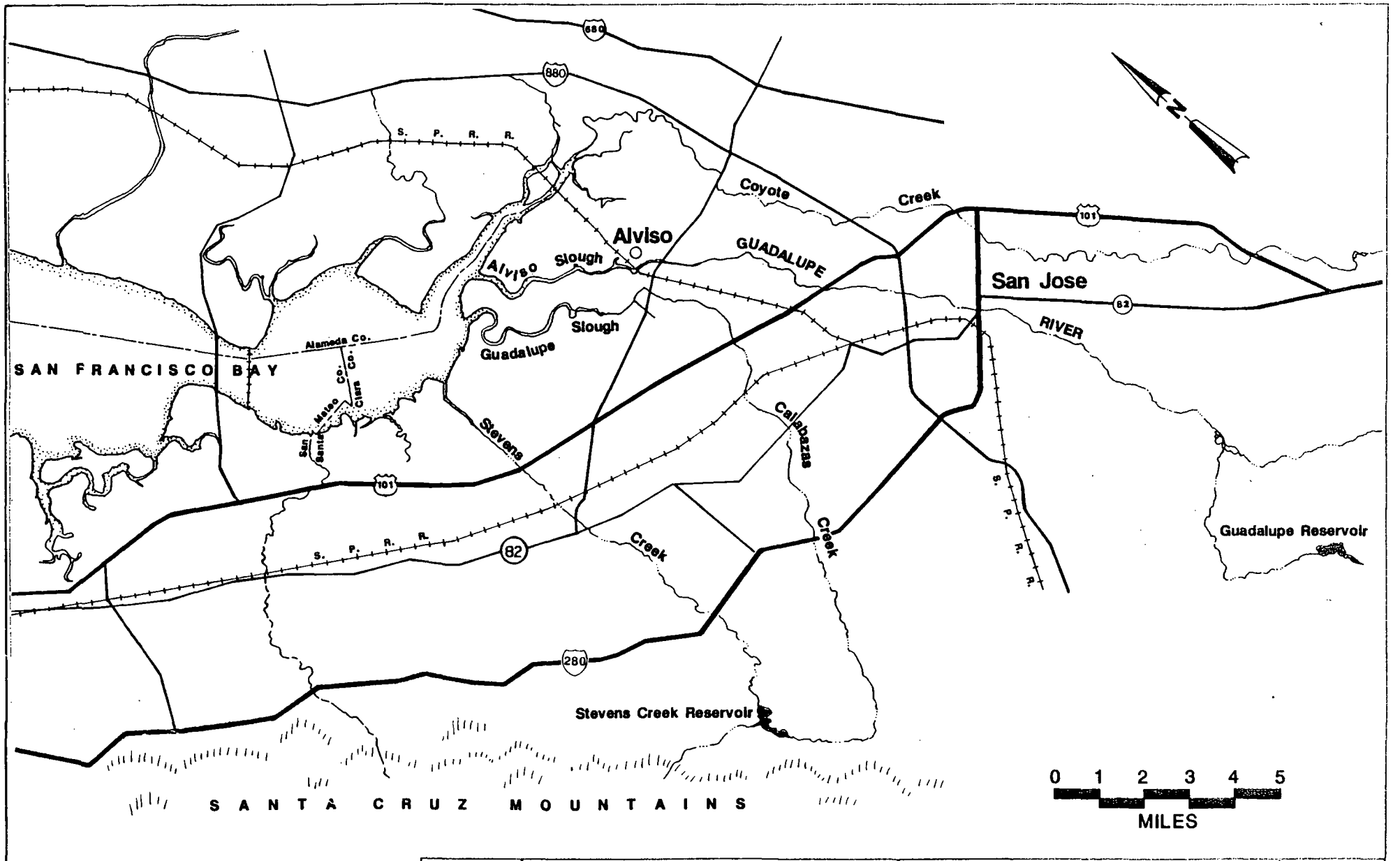


SOURCE: Greater San Jose Northern Area AAA Map

FEET 0 1000 2000 4000

Project No.	South Bay Asbestos Site	SAMPLE LOCATION MAP FOR SURFACE WATER, SEDIMENT, AND BORINGS	Figure
288-RI1	Camp Dresser & McKee Inc.		3-3

DATAPRINT N84939



Project No.	South Bay Asbestos Site	REGIONAL SURFACE WATER HYDROLOGY	Figure
288-RI1	Camp Dresser & McKee Inc.		4-1

DATAPRINT N02280

PARTIALLY SCANNED OVERSIZE ITEM(S)

See document # 221556 9
for partially scanned image(s).
FIGURE 2-3
FIGURE 5-1 to 5-2
For complete hardcopy version of the oversize document
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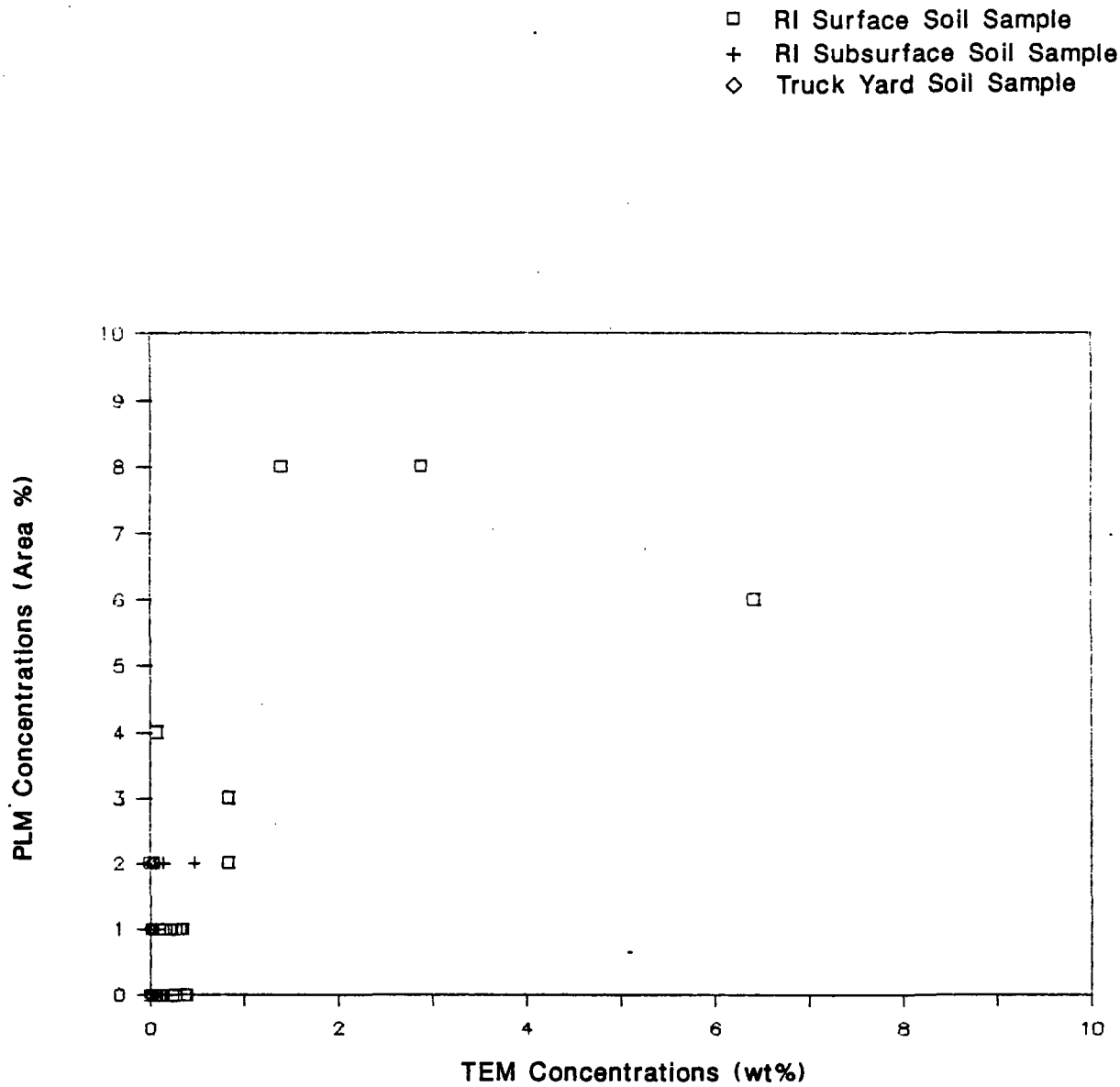
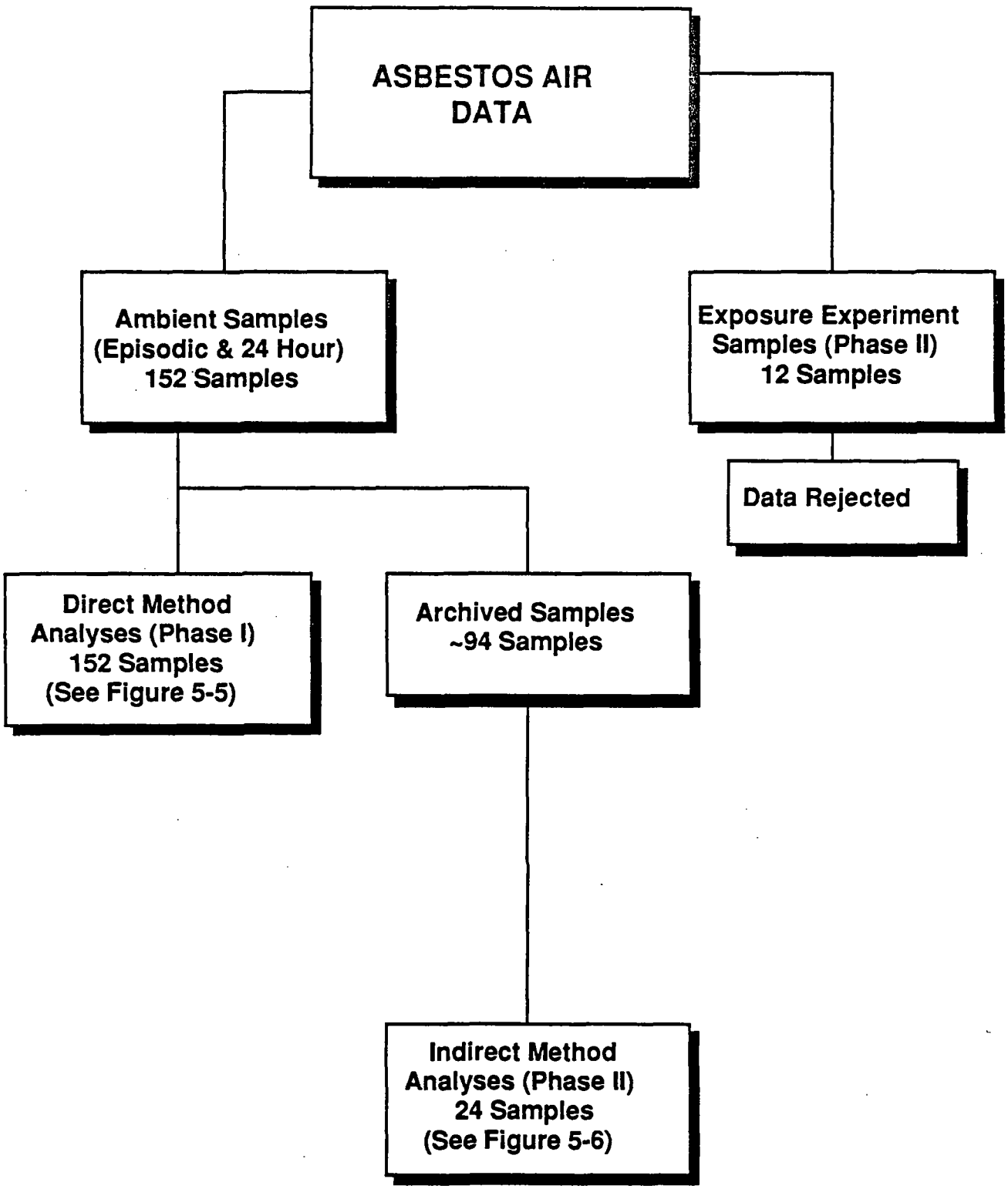
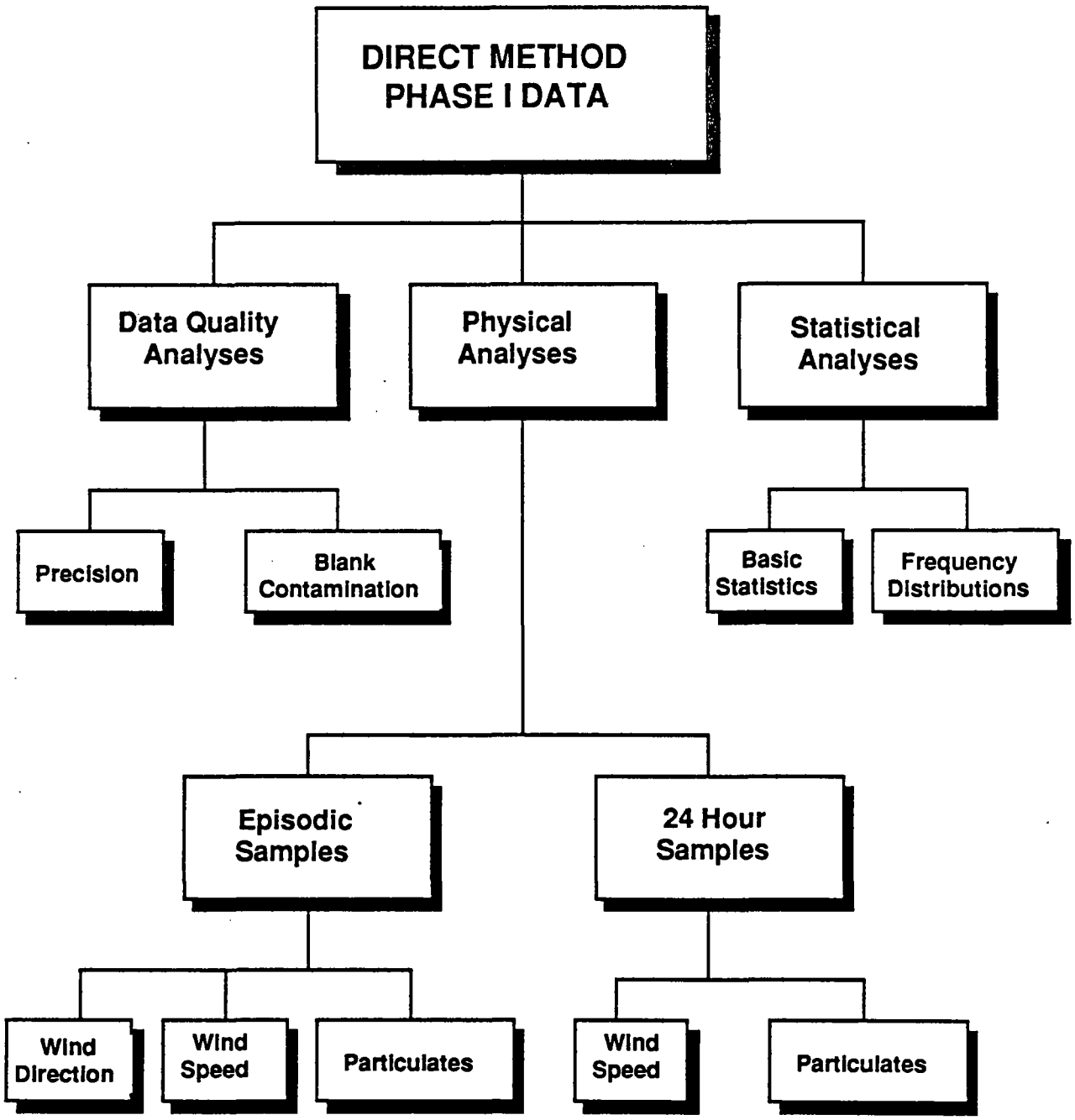


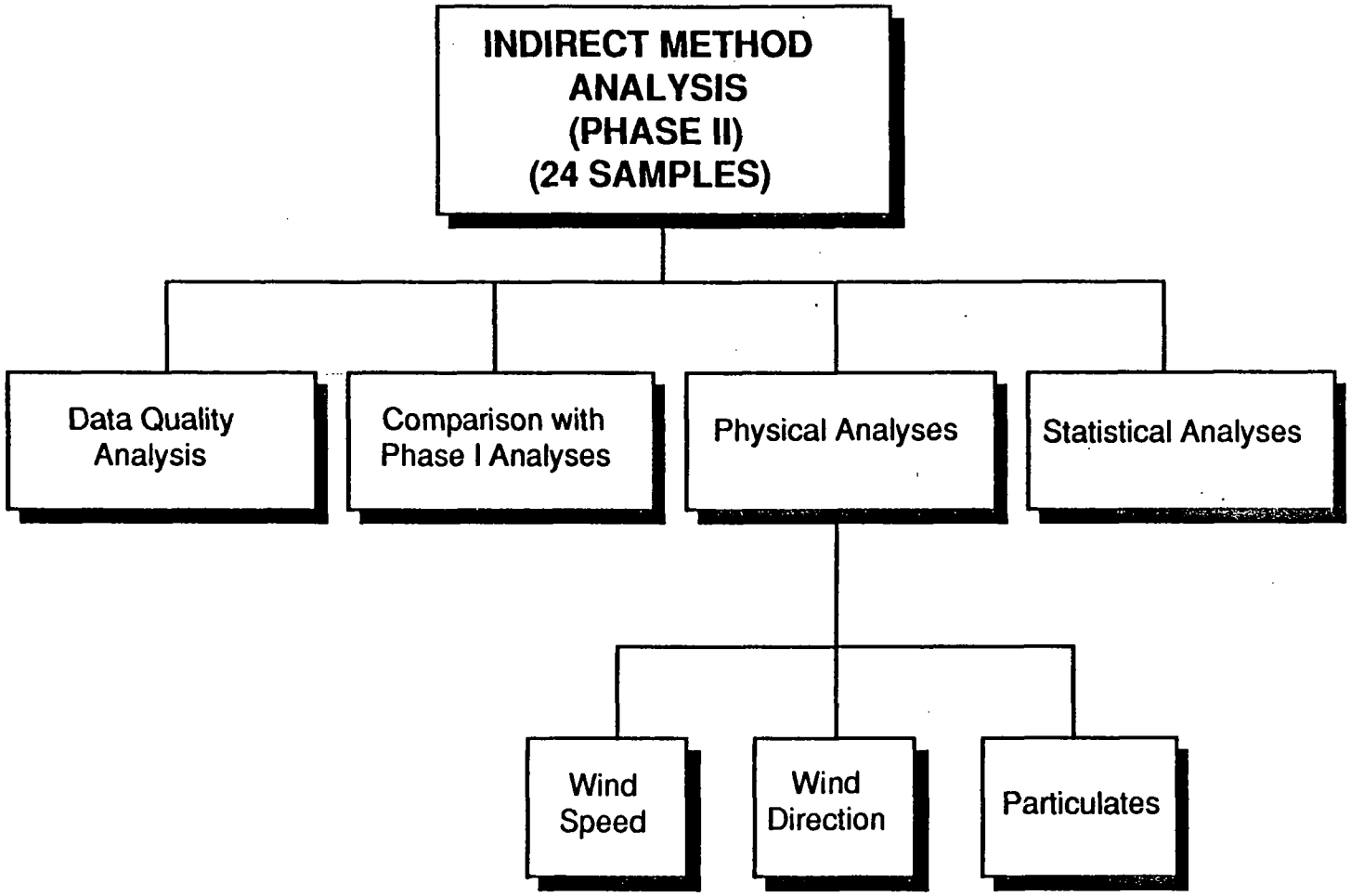
Project No. 288-RI1	South Bay Asbestos	PLM vs TEM SOIL RESULTS	Figure 5-3
	Camp Dresser & McKee		



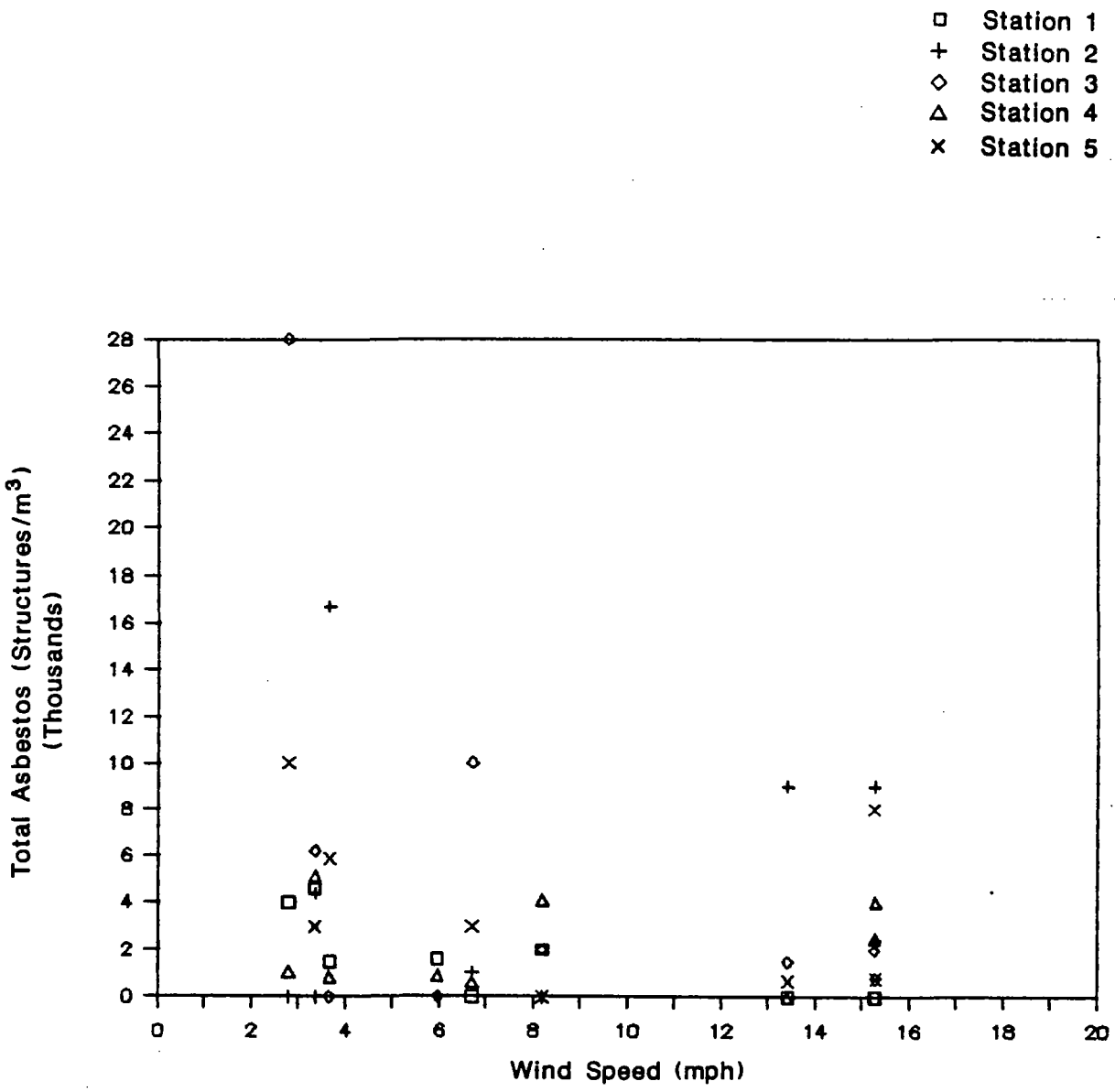
Project No.	South Bay Asbestos	Relationship of Air Data Analysis	Figure
288-RI1	Camp Dresser & McKee		5-4



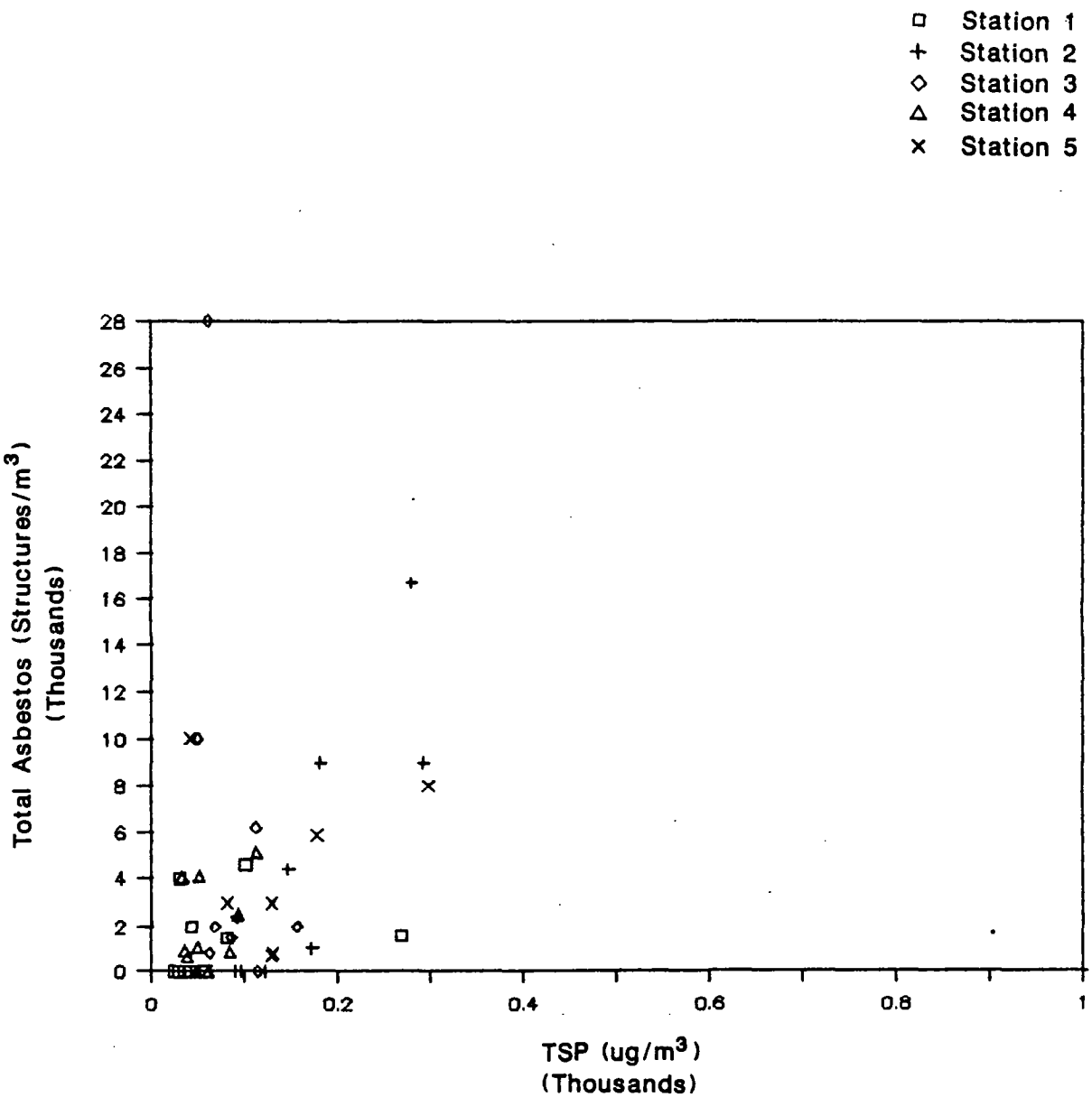
Project No.	South Bay Asbestos	Analyses Performed on Phase I Air Data	Figure
288-RI1	Camp Dresser & McKee		5-5



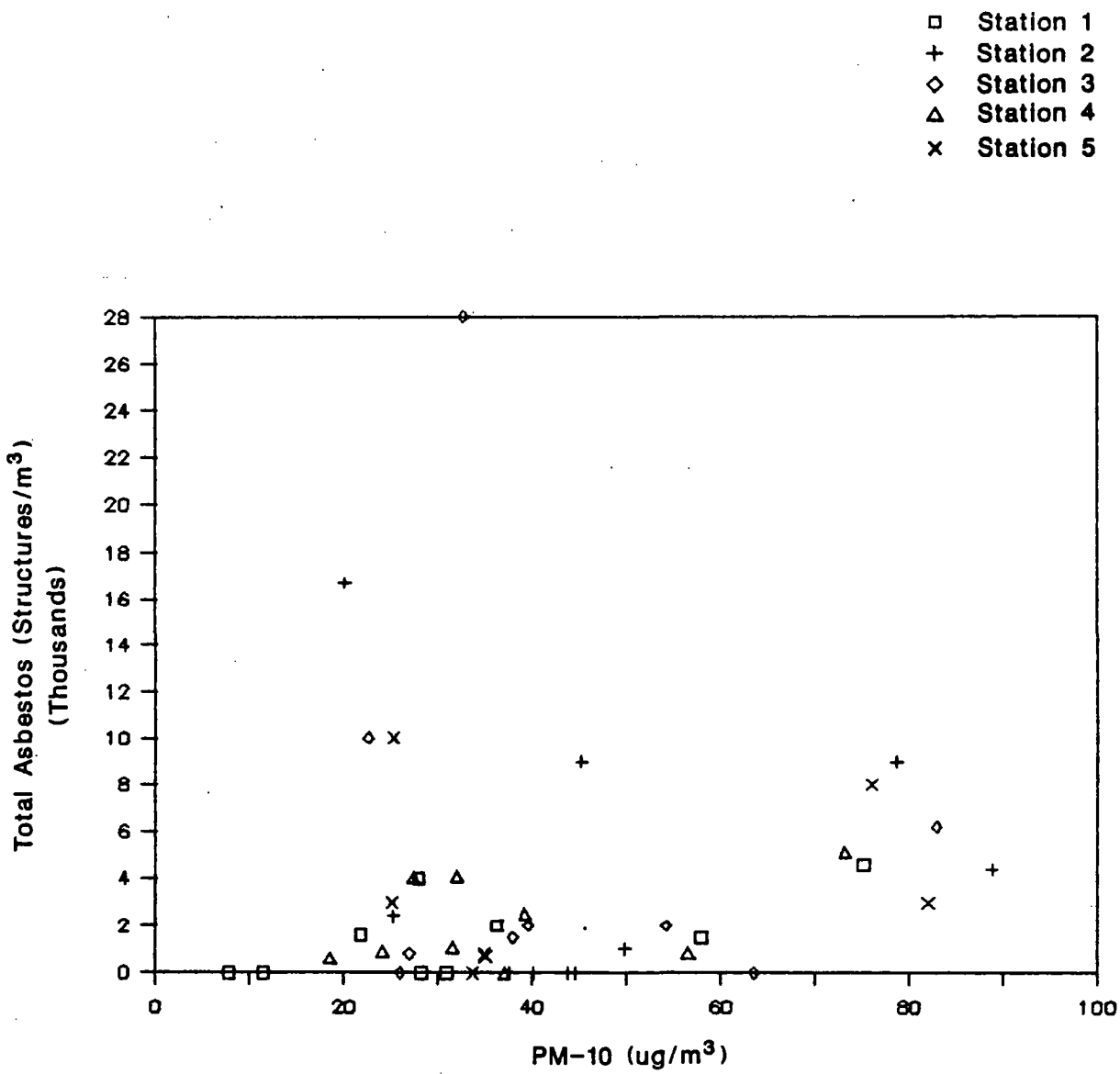
Project No.	South Bay Asbestos	Analyses Performed on Phase II Air Data	Figure
288-RI1	Camp Dresser & McKee		5-6



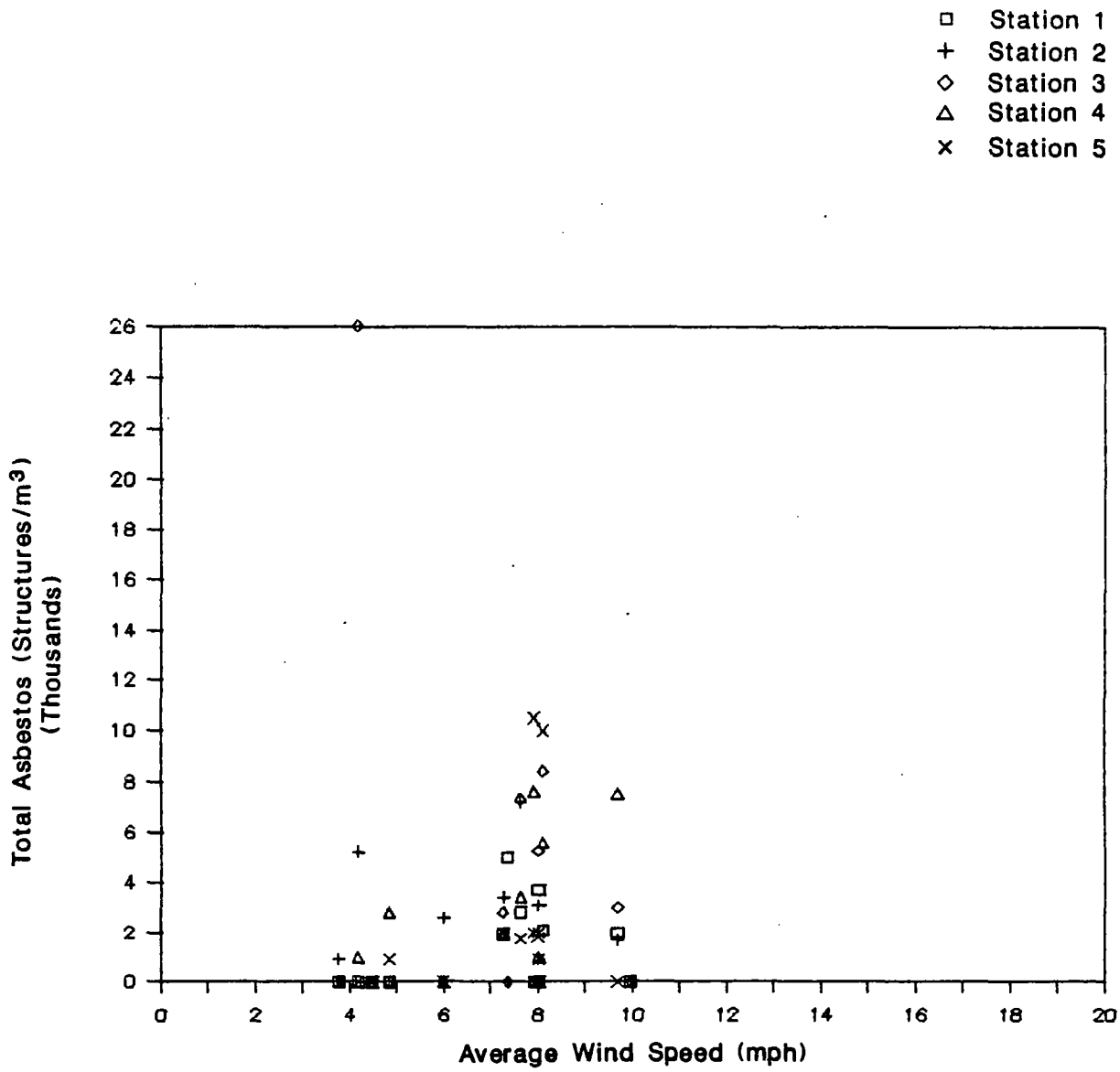
Project No.	South Bay Asbestos	EPISODIC ASBESTOS STRUCTURES vs WIND SPEED	Figure
288-RI1	Camp Dresser & McKee		5-7



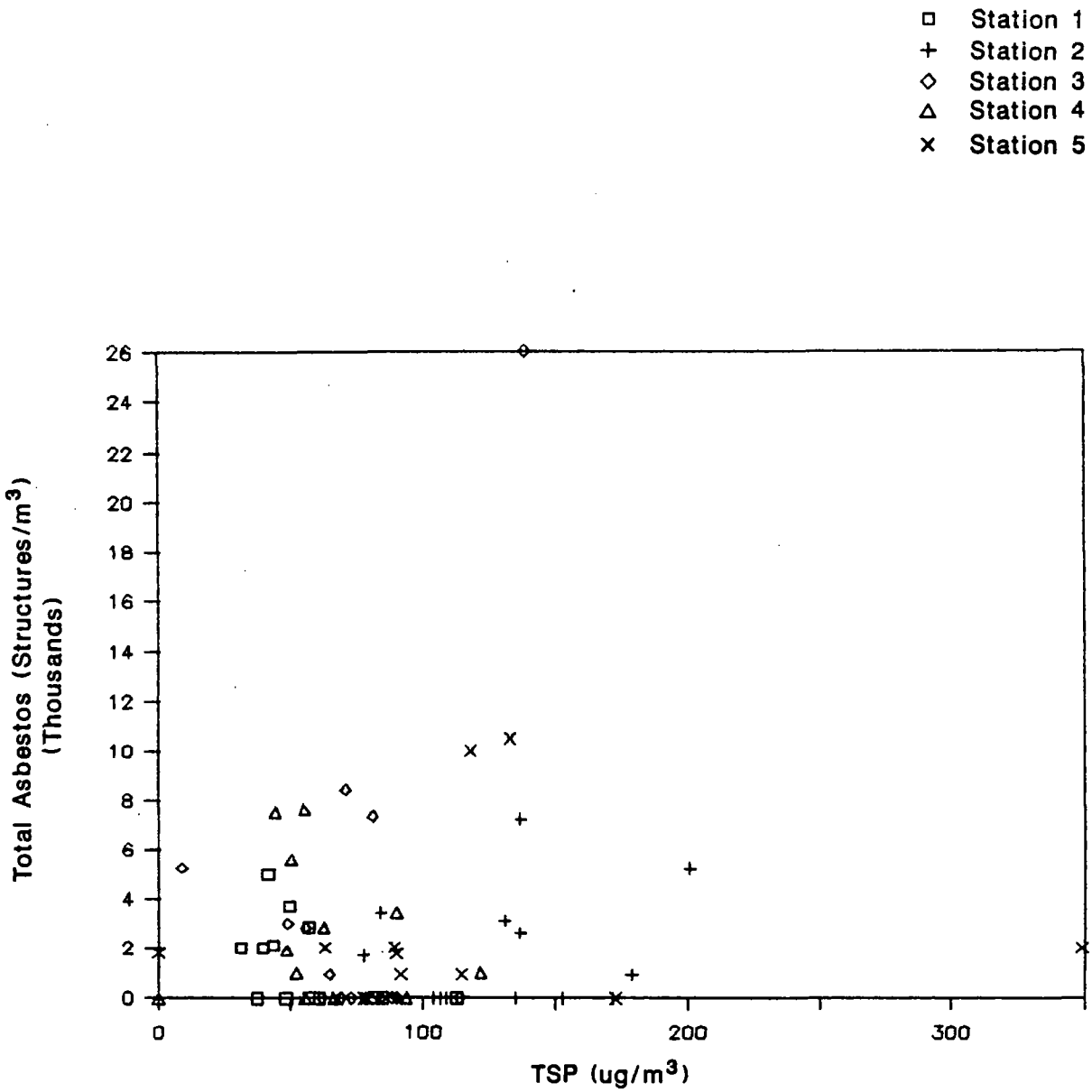
Project No. 288-R11	South Bay Asbestos	EPISODIC ASBESTOS STRUCTURES vs TSP	Figure 5-8
	Camp Dresser & McKee		



Project No. 288-RI1	South Bay Asbestos	EPISODIC ASBESTOS STRUCTURES vs PM-10	Figure 5-9
	Camp Dresser & McKee		



Project No.	South Bay Asbestos	24 HOUR ASBESTOS STRUCTURES vs WIND SPEED	Figure
288-RI1	Camp Dresser & McKee		5-10



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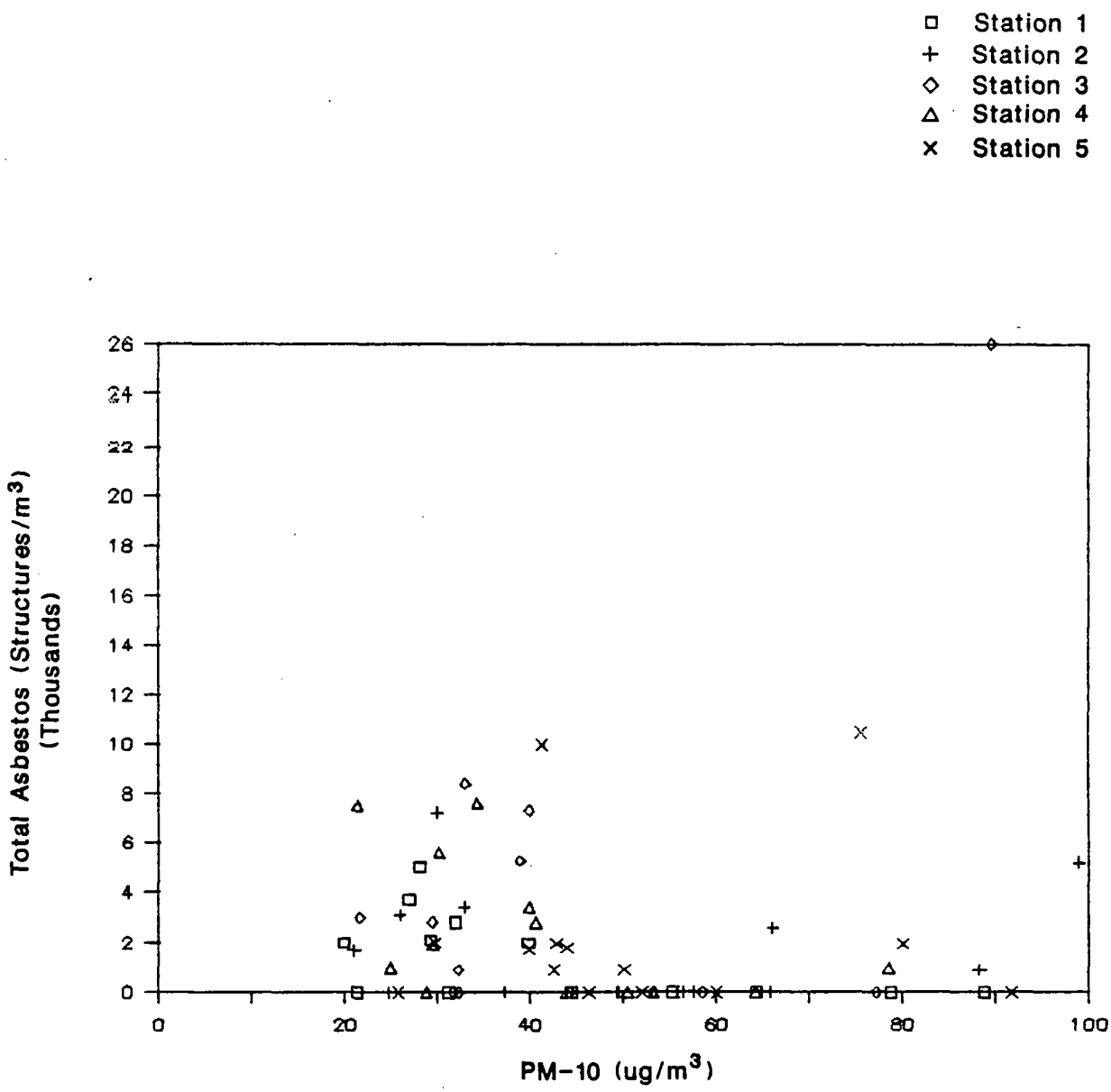
288-RI1	Camp Dresser & McKee		5-11



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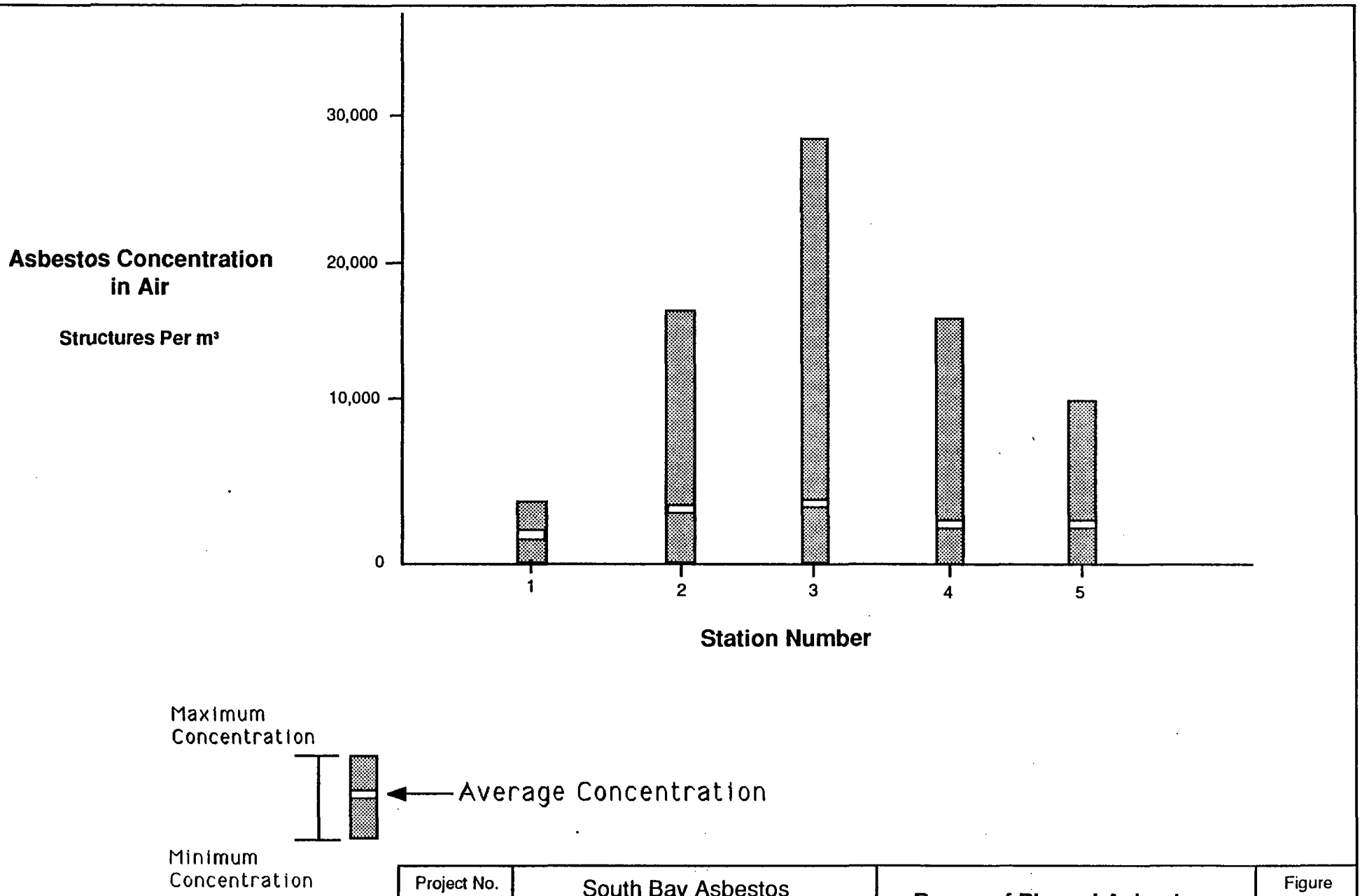
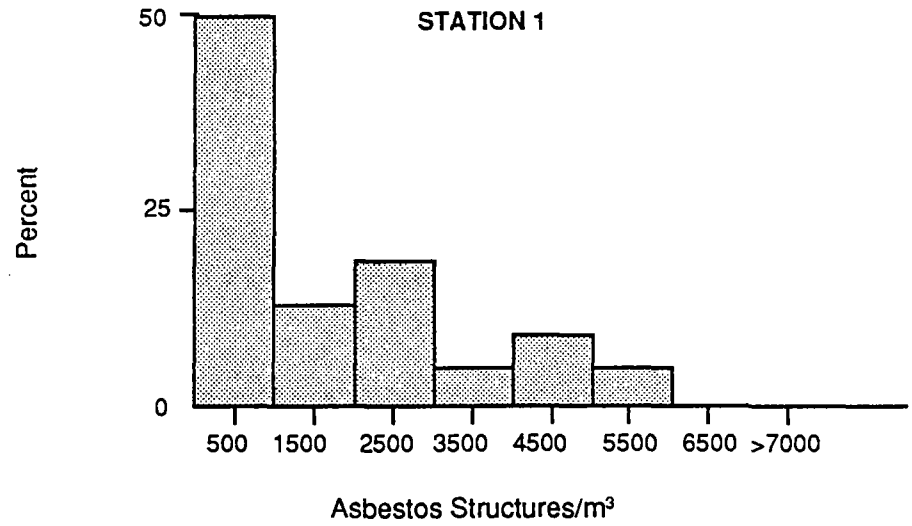
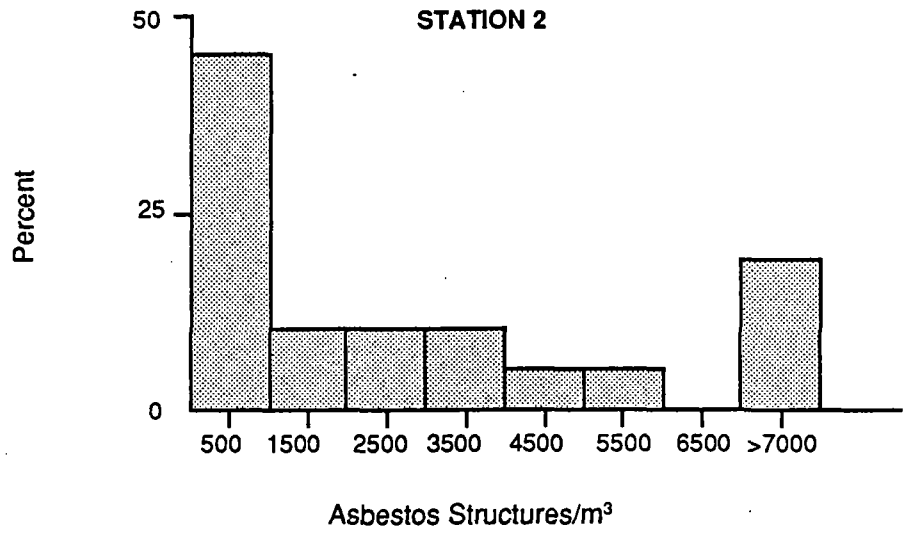
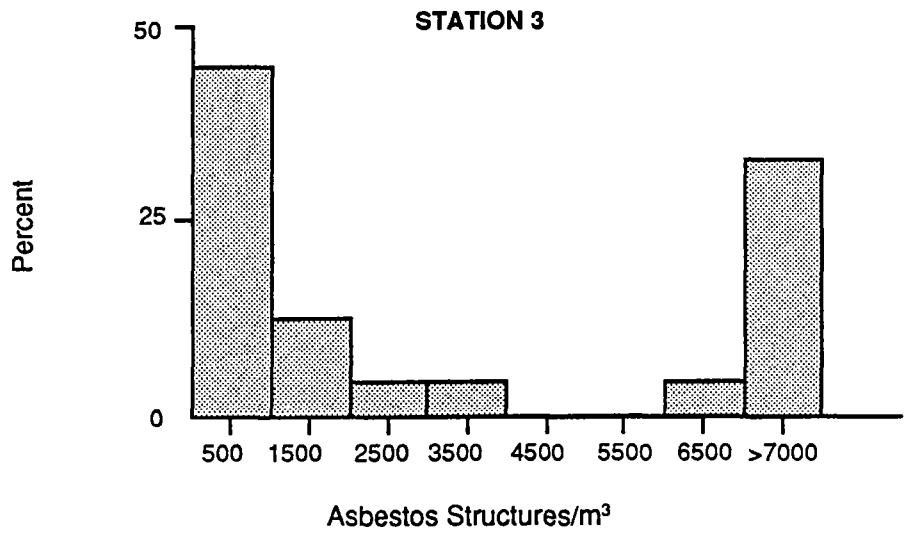
288-RI1	Camp Dresser & McKee		5-12



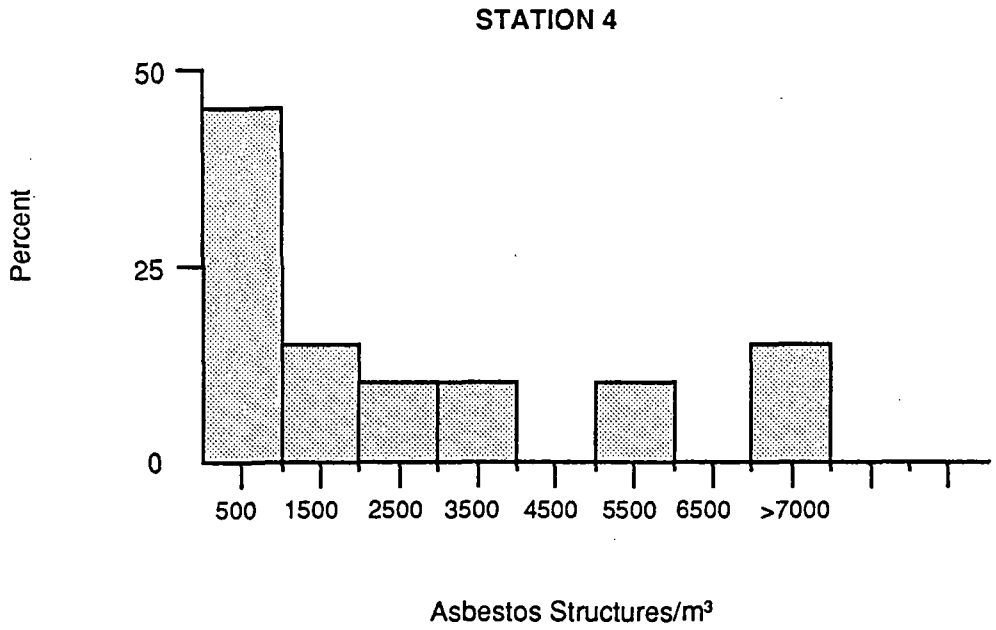
Project No.	South Bay Asbestos	Range of Phase I Asbestos Structure Concentrations	Figure
288-RI1	Camp Dresser & McKee		5-13

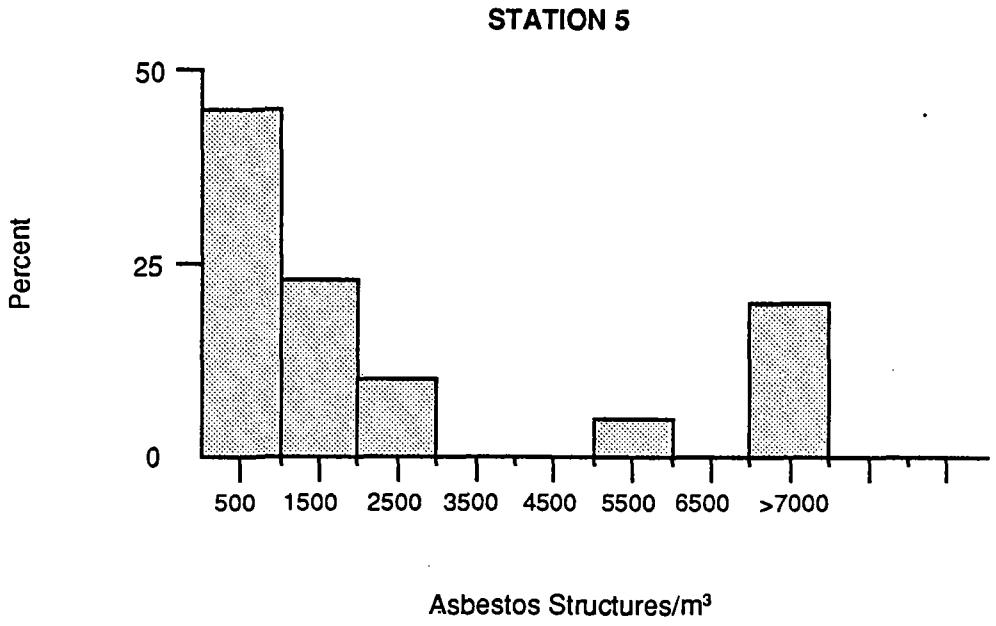




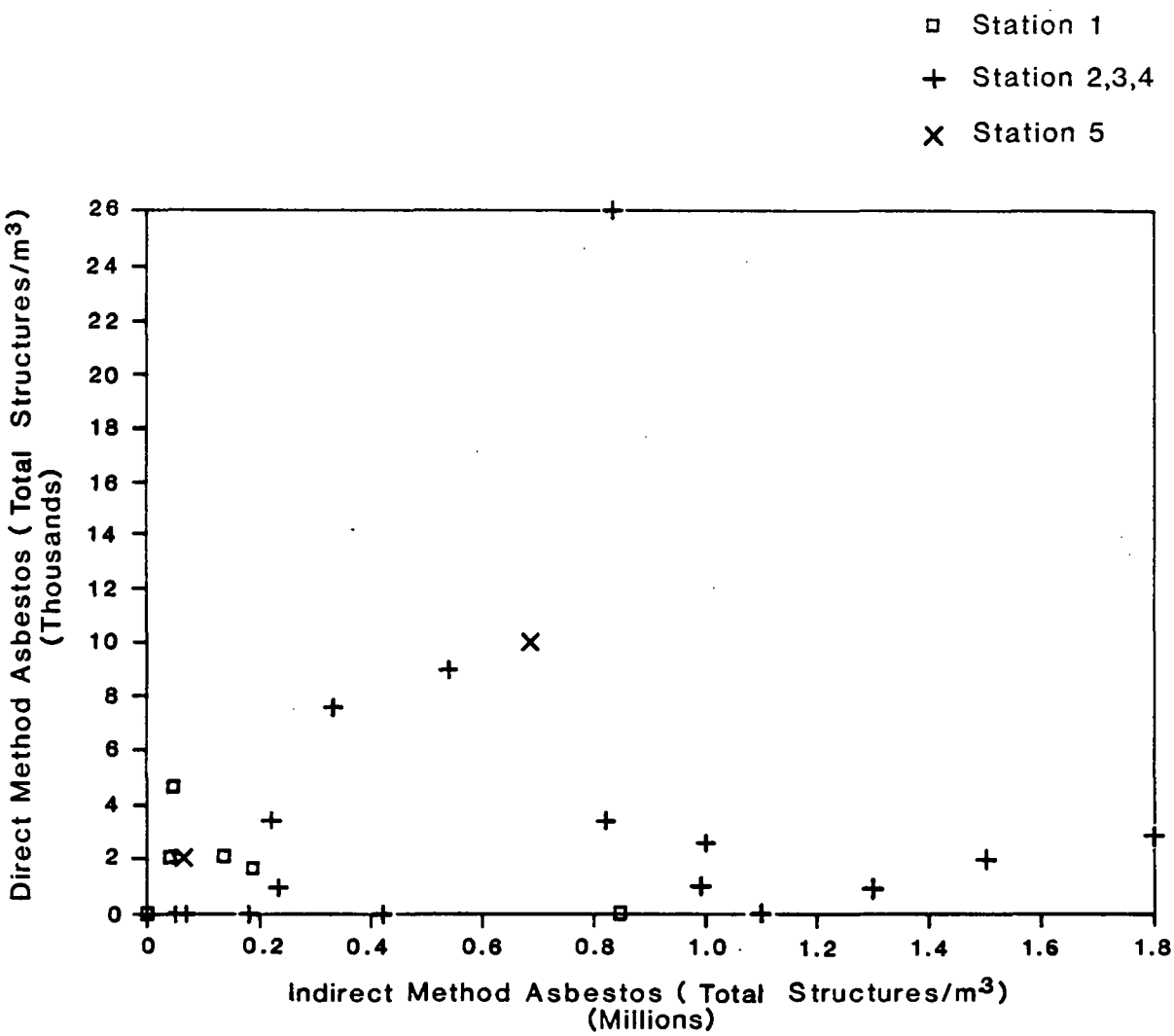


Project No.	South Bay Asbestos	HISTOGRAMS OF ASBESTOS STRUCTURE CONCENTRATIONS	Figure
288-RI1	Camp Dresser & McKee		5-14

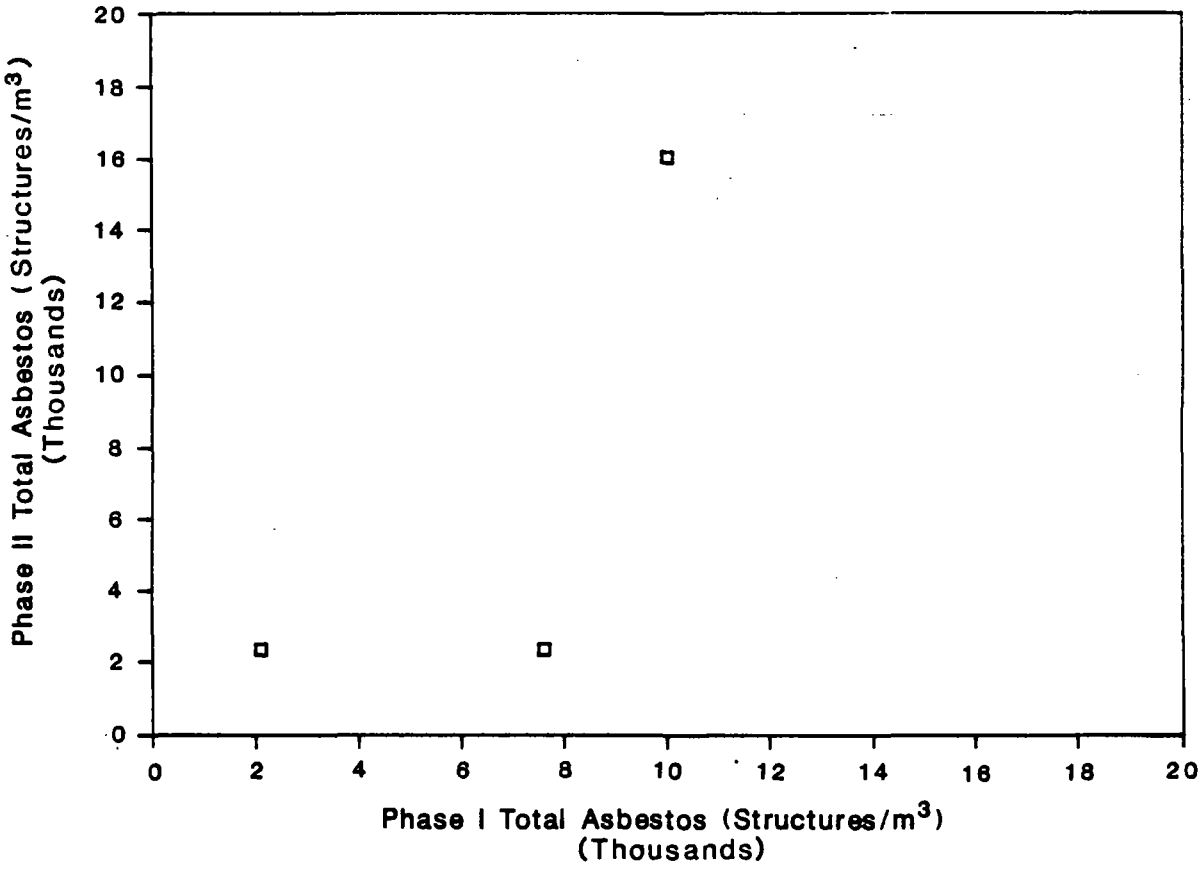




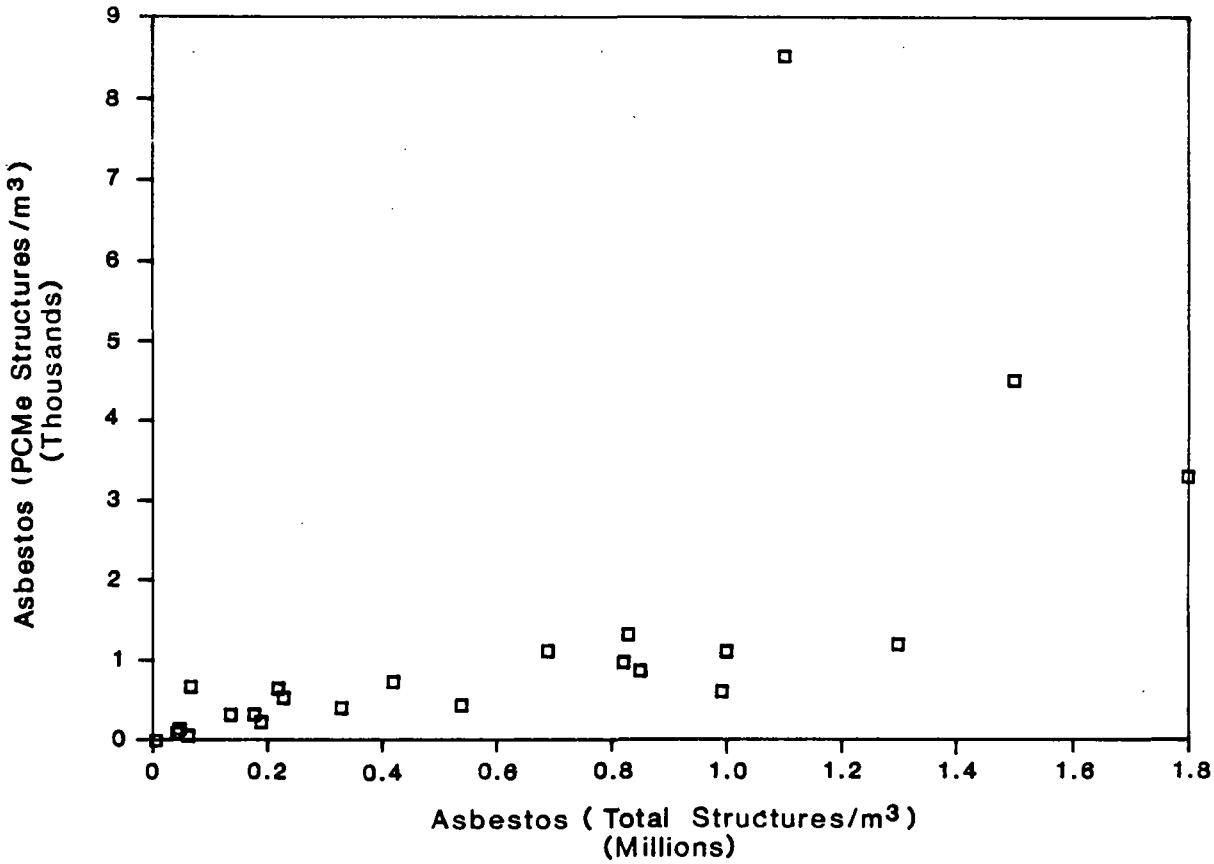
Project No.	South Bay Asbestos	HISTOGRAMS OF ASBESTOS STRUCTURE CONCENTRATIONS	Figure
288-RI1	Camp Dresser & McKee		5-15



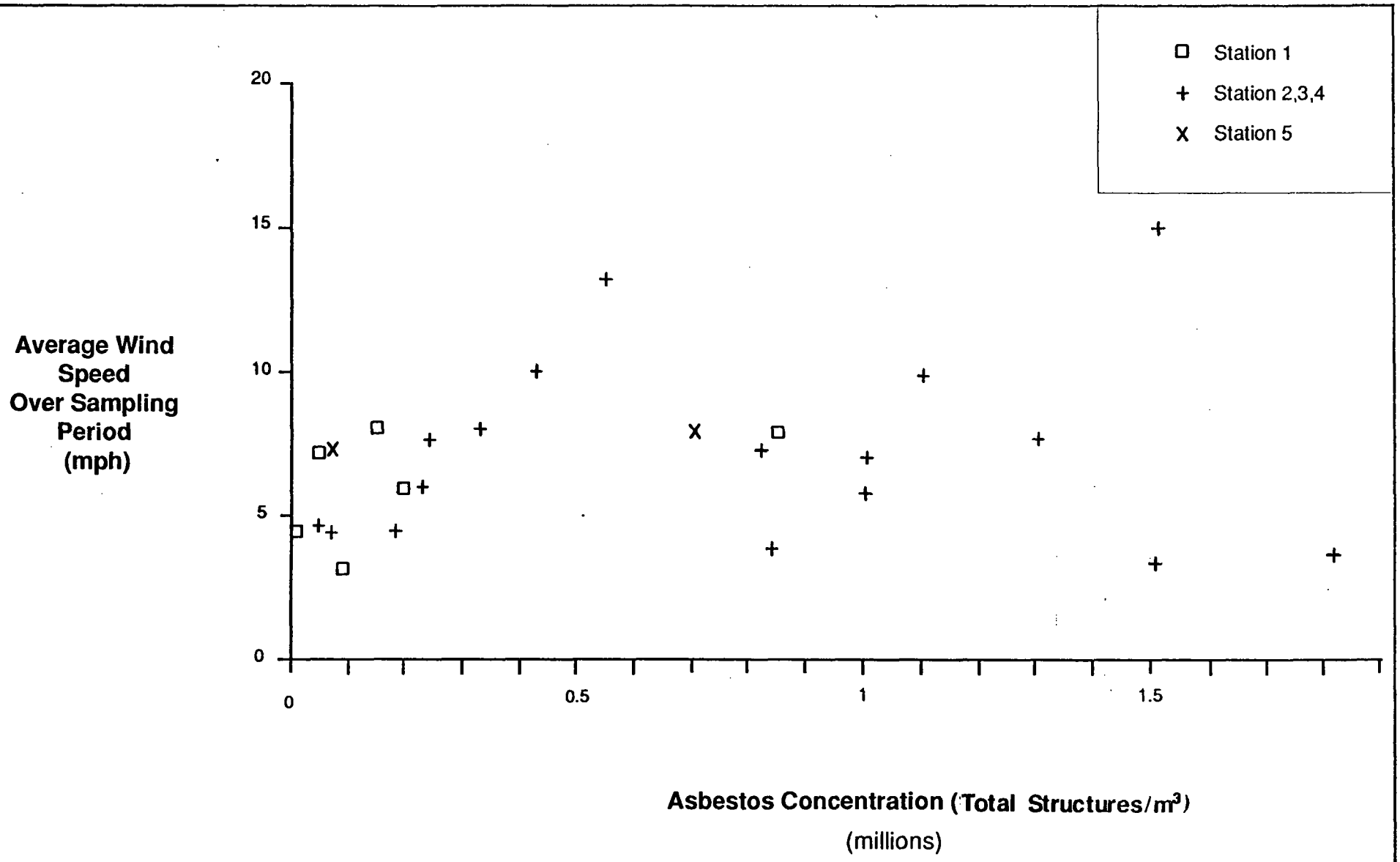
Project No.	South Bay Asbestos	DIRECT AND INDIRECT ANALYSIS COMPARISON	Figure
288-RI1	Camp Dresser & McKee		5-16



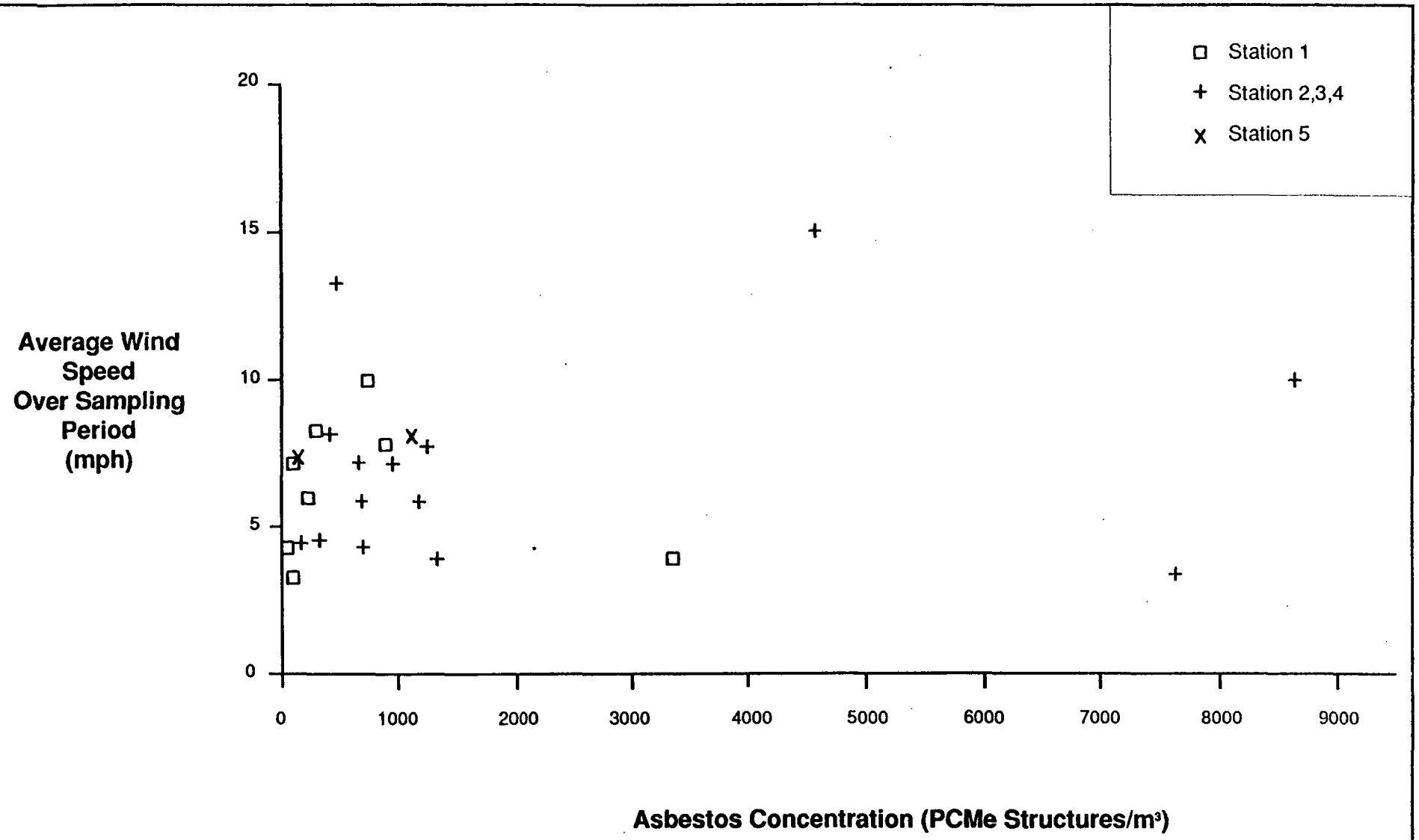
Project No. 288-RI1	South Bay Asbestos	COMPARISON BETWEEN DIRECT ANALYSES	Figure 5-17
	Camp Dresser & McKee		



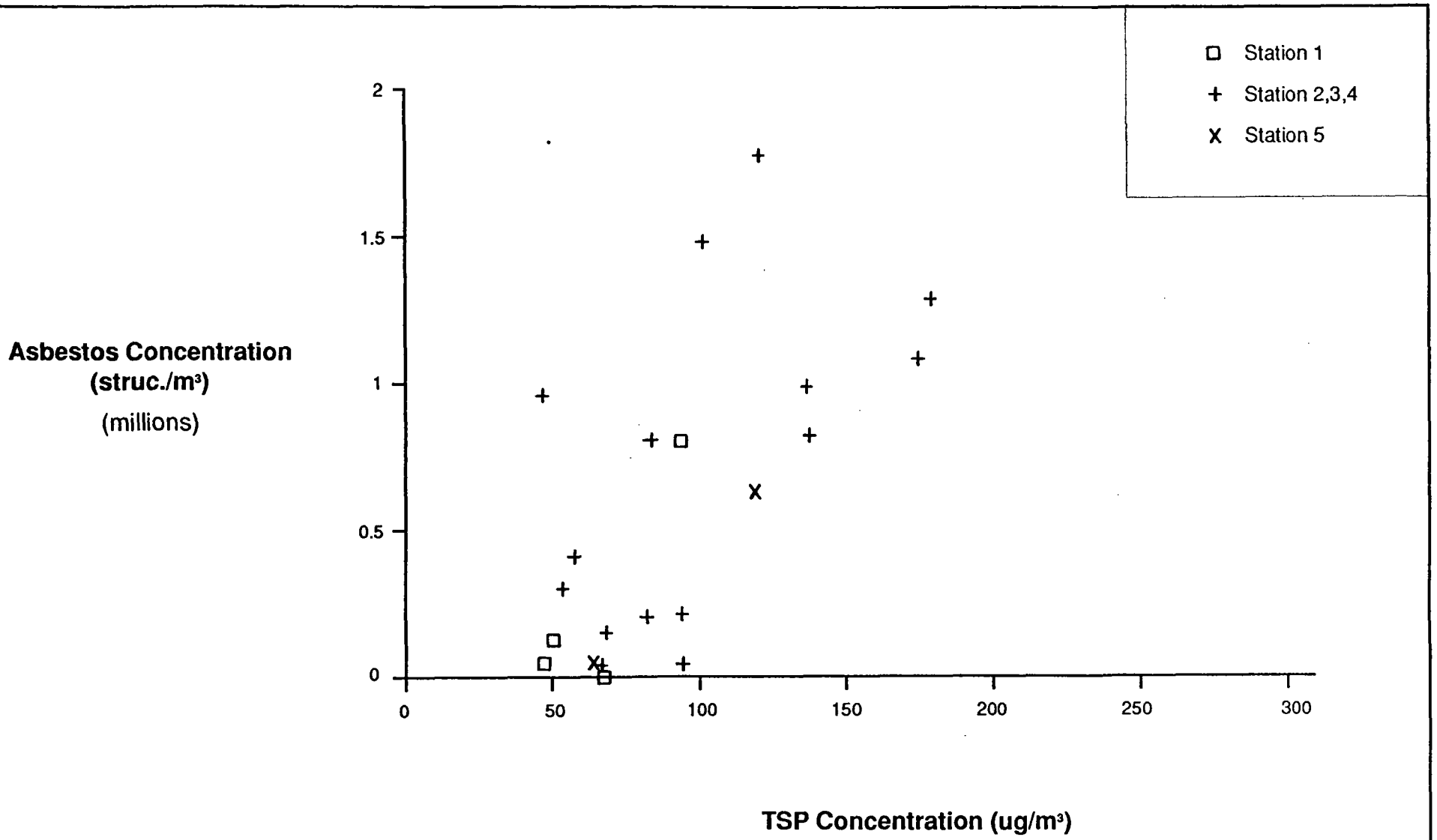
Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	**PCM EQUIVALENTS vs TOTAL STRUCTURES**	5-18



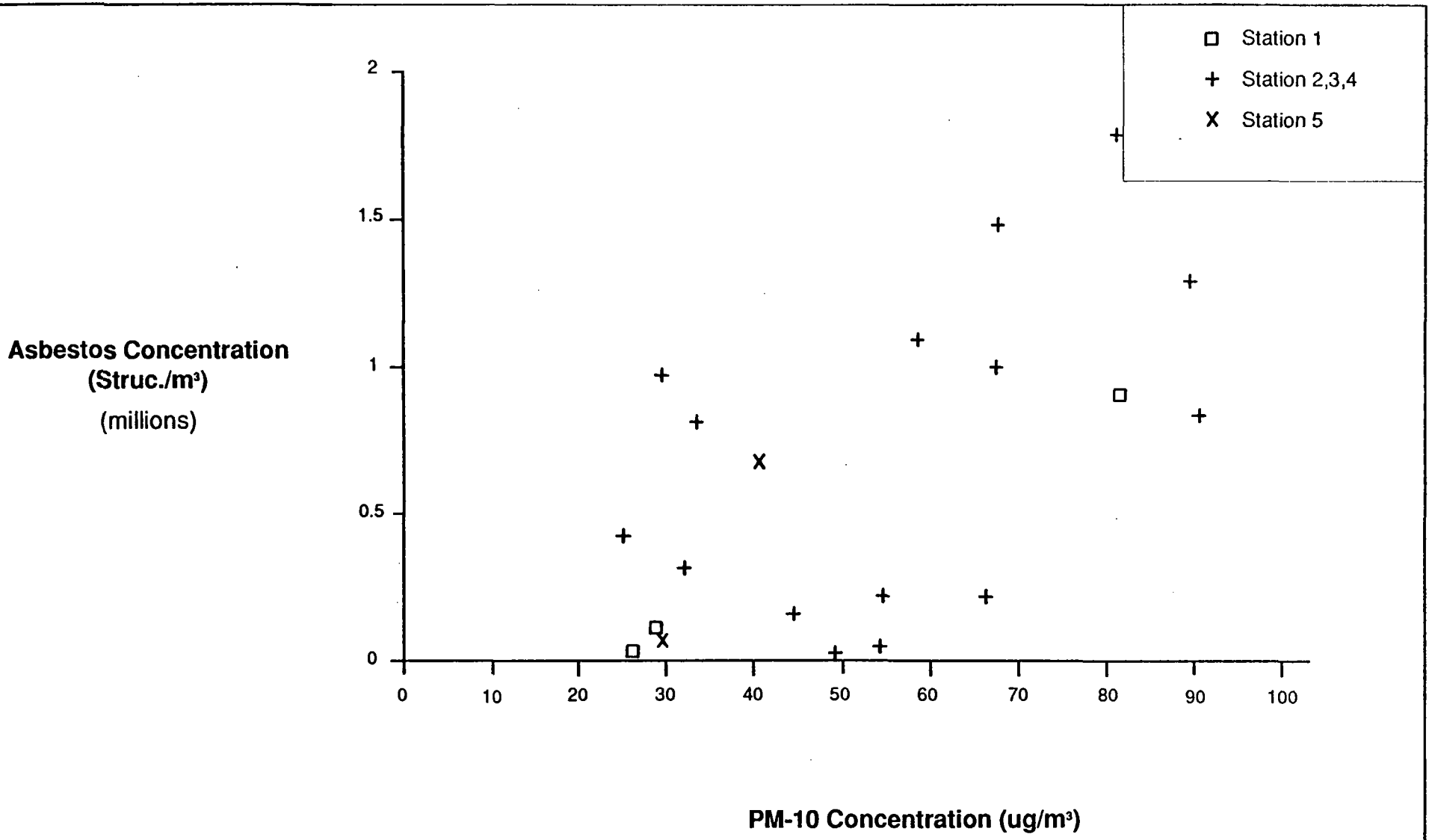
Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	AVERAGE WIND SPEED - TOTAL ASBESTOS SCATTERPLOT	5-19



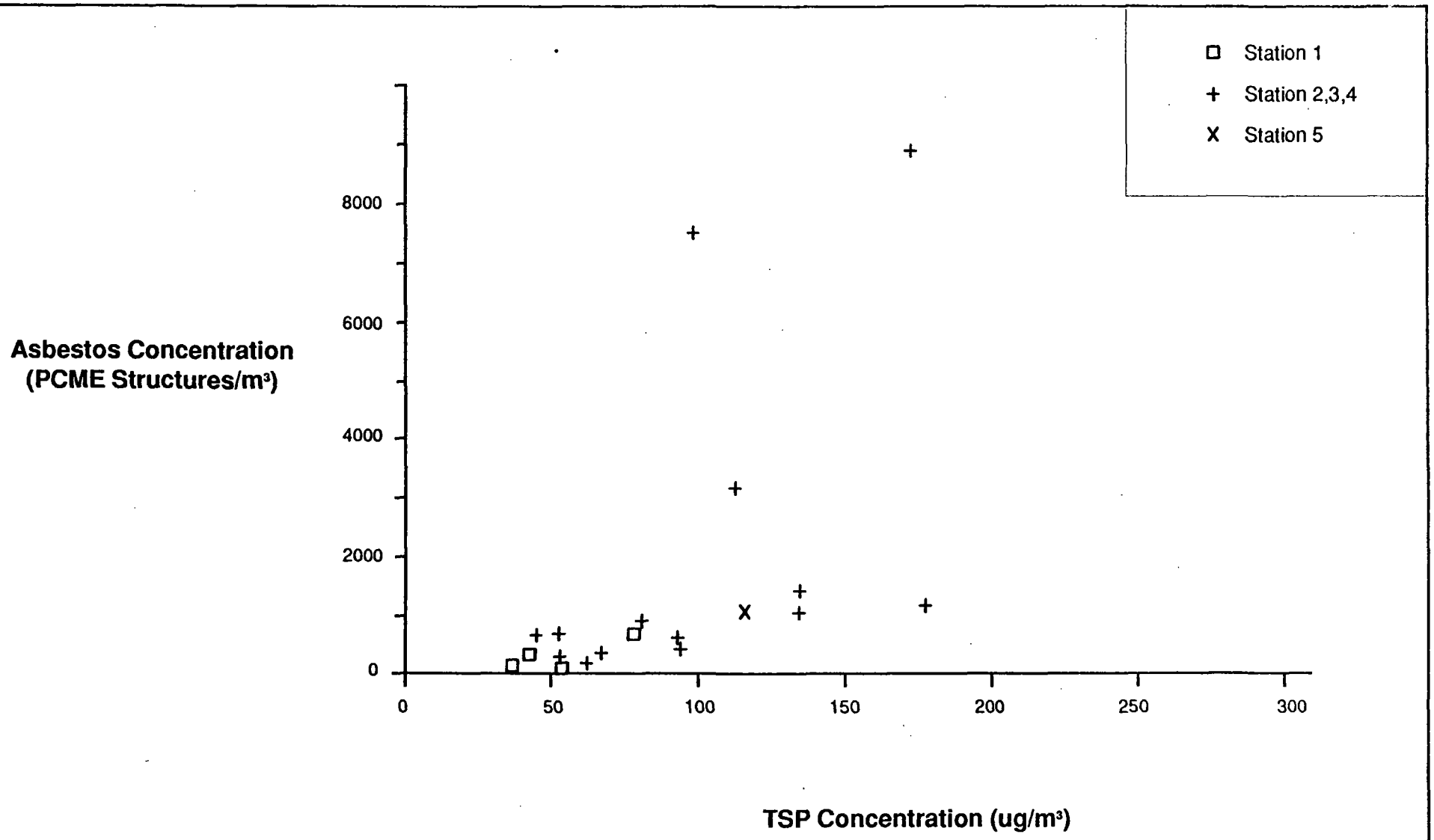
Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	AVERAGE WIND SPEED - PCME SCATTERPLOT	5-20



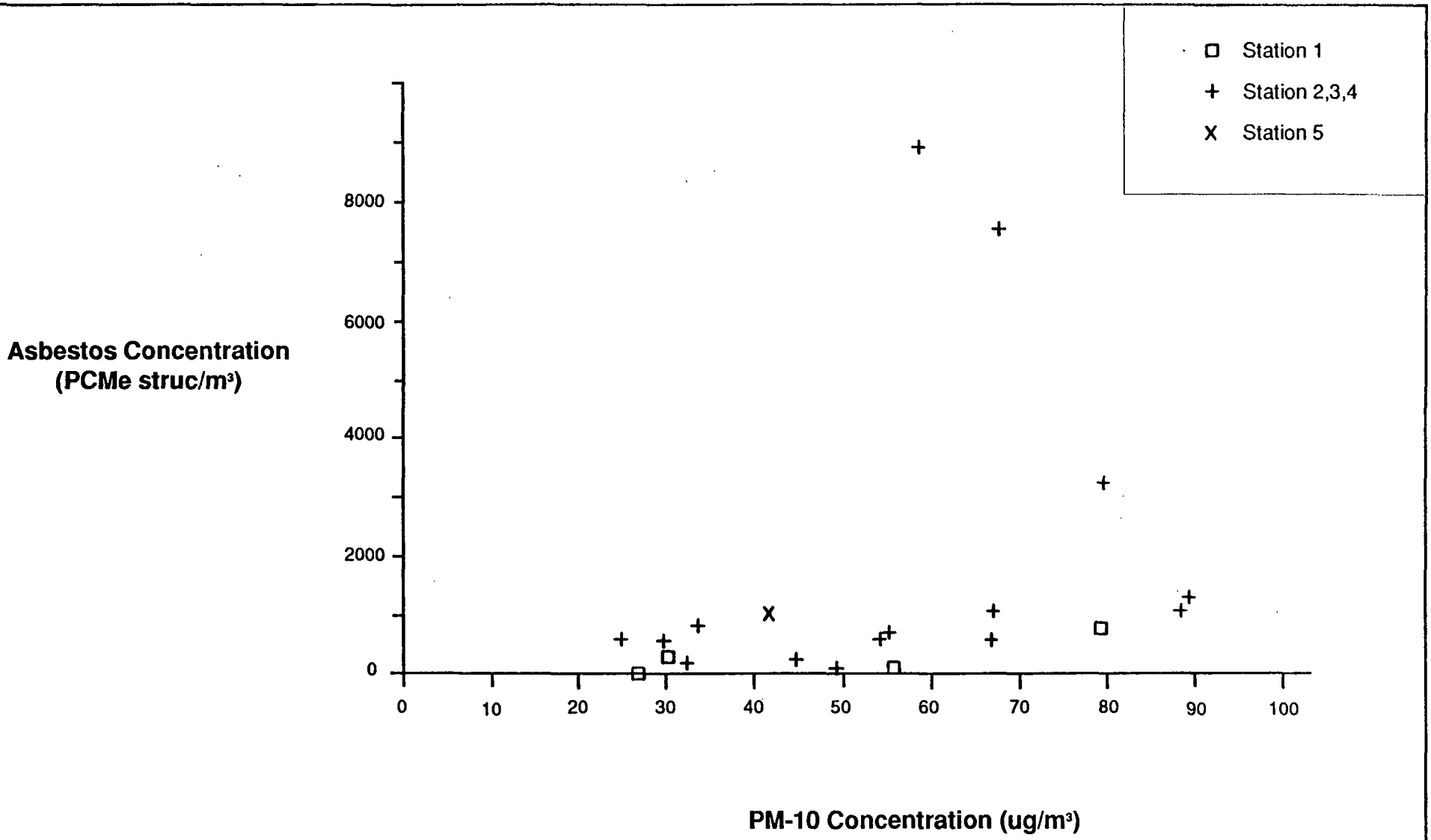
Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	TOTAL ASBESTOS — TSP SCATTERPLOT	5-21



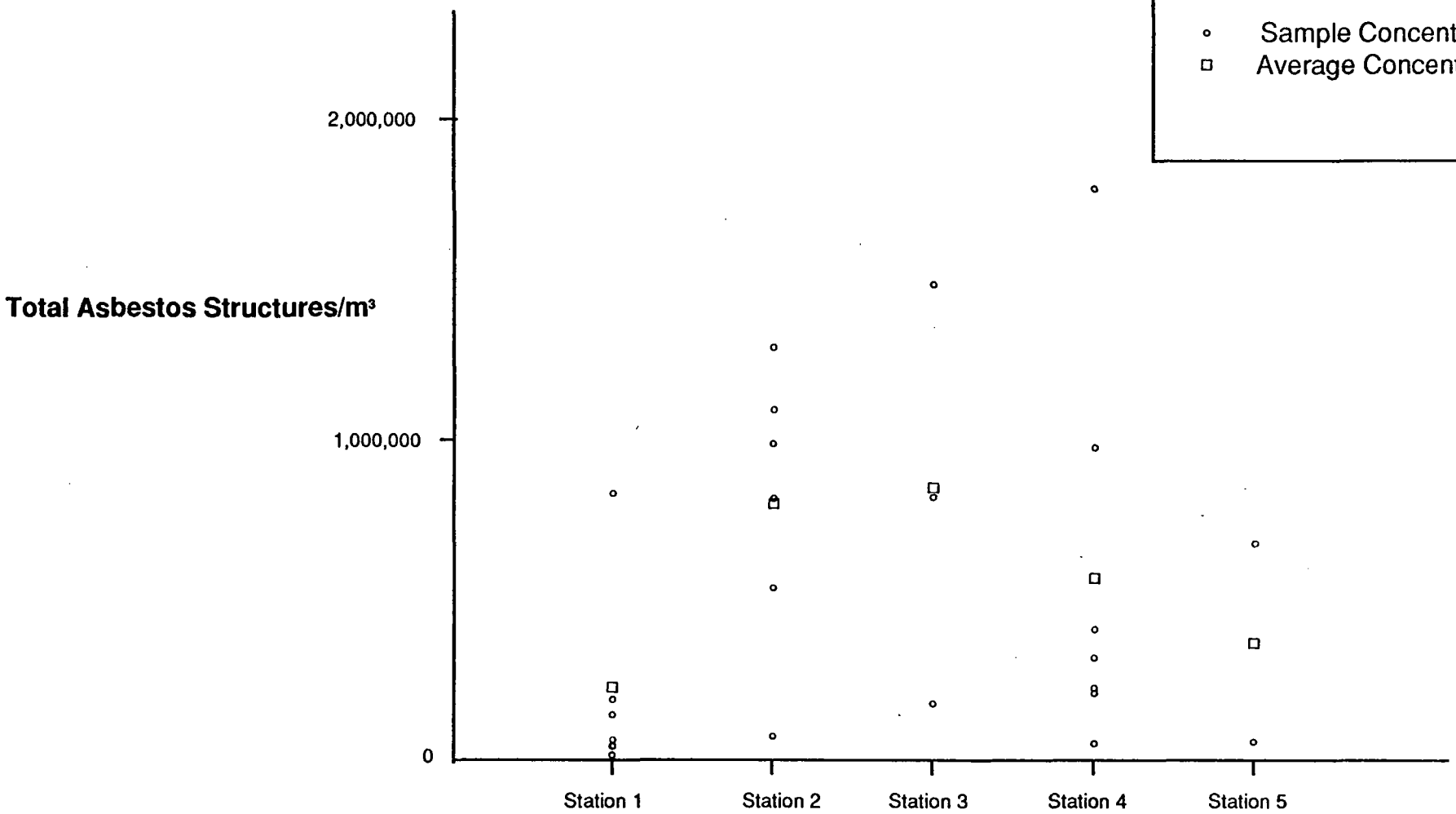
Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	TOTAL ASBESTOS — PM-10 SCATTERPLOT	5-22



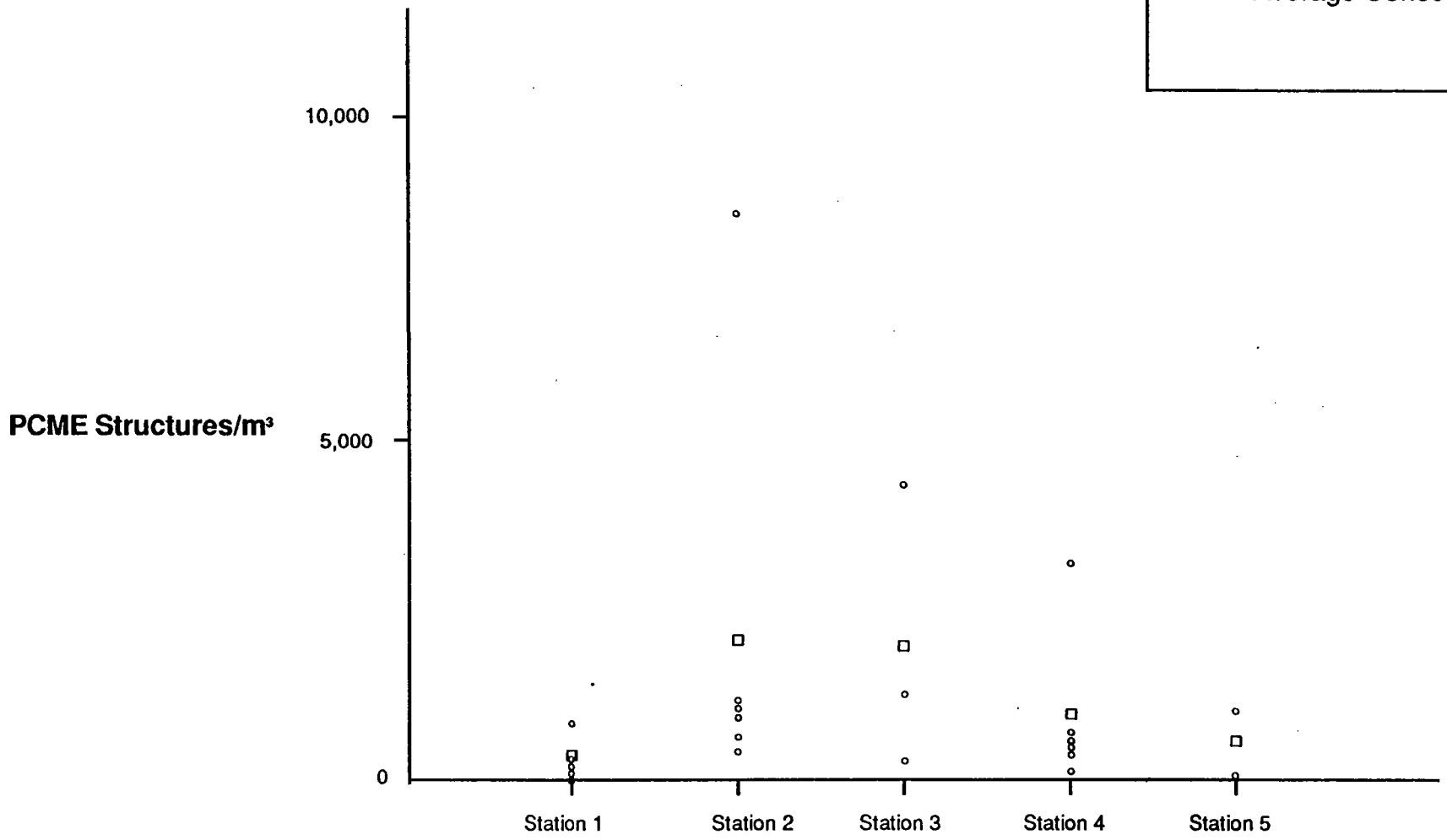
Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	PCME-TSP SCATTERPLOT	5-23



Project No.	South Bay Asbestos	Phase II	Figure
288-RI1	Camp Dresser & McKee	PCME-PM-10 SCATTERPLOT	5-24



Project No.	South Bay Asbestos	DISTRIBUTION OF PHASE II TOTAL ASBESTOS STRUCTURE CONCENTRATIONS	Figure
288-RI1	Camp Dresser & McKee		5-25



Project No.	South Bay Asbestos	Distribution of Phase II Asbestos PCM Equivalent Concentrations	Figure
288-RI1	Camp Dresser & McKee		5-26

APPENDICES

APPENDIX A

GLOSSARY OF TERMS

GLOSSARY

ARAR - Applicable or Relevant and Appropriate Requirements. Existing laws, regulations or guidelines which are pertinent to a Superfund site.

AHERA - Asbestos Hazard Emergency Response Act, enacted to control asbestos in public schools.

AIHL - Air and Industrial Hygiene Laboratory of the California Department of Health Services.

ASTM-C136 - American Society of Testing Materials, Test C136, for determining soil grain size.

ATSDR - Agency for Toxic Substances and Disease Registry: A branch of the Federal Center for Disease Control that is responsible for preparing health assessments at sites.

ATT - Aqua-Terra Technologies, consultant to the COE during an earlier phase of the site investigation.

Asbestos - Mineral fibers which can be separated and used for a variety of industrial applications.

Asbestos structure - see Structure.

BAAQMD - Bay Area Air Quality Management District.

BCDC - Bay Conservation and Development Commission.

Bench Scale - Treatability tests performed on a small scale, usually in a laboratory, to better define parameters of a treatment technology.

CAA - Clean Air Act.

Cal-Osha - California Occupational Health and Safety Administration.

CDM - Camp Dresser & McKee Inc.

CEQA - California Environmental Quality Act.

CERCLA - Comprehensive Environmental Response, Compensation, and Liability Act of 1980, also known as Superfund: Amended in 1986 by the Superfund Amendments and Reauthorization Act (SARA).

CFR - Code of Federal Regulations.

CLP - Contract Laboratory Program.

Corps - United States Army Corps of Engineers (COE).

COE - United States Army Corps of Engineers.

CWA - Clean Water Act.

Chrysotile - The fibrous variety of the mineral serpentine with a chemical composition of $(M_3Fe)_3\ Si_2O_5(OH)_4$.

DHS - California Department of Health and Services.

DQO - Data Quality Objectives: Statements that specify the data needed to support decisions regarding remedial response activities.

EAL - EAL Laboratories.

EECA - Engineering Evaluation and Cost Analysis: Prepared by EPA to justify cost of Expedited Remedial Actions (ERAs).

EIS - Environmental Impact Statement.

EMCON - Consultants to Leslie Salt Company, owners of the Marshland Landfill.

EPA - Environmental Protection Agency.

ERA - Expedited Response Action.

Excess lifetime cancer risk - The potential for carcinogenic effects from exposure to one or more chemicals.

FIT - Field Investigtion Team.

FS - Feasibility Study.

RI/FS - Remedial Investigation/Feasibility Study.

FSP - Field Sampling Plan: Defines in detail the sampling and data gathering activities to be used at a site (see SAP).

General response action - General types of actions, such as containment, that may be taken to achieve exposure limits specified by remedial action objectives.

HLA - Harding Lawson Associates.

Health assessment - Assessment of existing risk to human health posed by NPL sites, prepared by the ATSDR or contractors to EPA.

hi-Vol - High volume sampler.

HEP - Habitat Evaluation Procedure - a habitat-based evaluation methodology used to determine project impacts on wetlands and identify mitigation goals.

ISV - In-situ vitrification, a high-temperature treatment technology for asbestos.

Innovative technologies - Technologies that are fully developed but lack sufficient cost or performance data for routine use at CERCLA sites.

Lead agency - The agency, either the EPA, Federal agency, or appropriate State agency having primary responsibility and authority for planning and executing the remediation at a site.

MCL - Maximum Contaminant Level: Established under the Safe Drinking Water Act.

MCLG - Maximum Contaminant Level Goal.

NAAQS - National Ambient Air Quality Standards.

NCP - National Oil and Hazardous Substances Contingency Plan.

NEPA - National Environmental Policy Act.

NESHAPS - National Emission Standards for Hazardous Air Pollutants, as promulgated under Clean Air Act.

NIOSH - National Institute for Occupational Safety and Health.

NPDES - National Pollutant Discharge Elimination System.

NPL - National Priorities List: A list of sites identified for remediation under CERCLA.

O&M - Operation and maintenance.

OSHA - Occupational Safety and Health Administration, responsible for work place safety.

OSWER - Office of Solid Waste and Emergency Response, a branch of EPA.

Operable Unit - A discrete part of a Superfund site that can be remediated independently as a unit to prevent or minimize a release or threat of release.

OUFS - Operable Unit Feasibility Study, an accelerated FS prepared for an operable unit.

PCM - Phase Contrast Light Microscopy, an optical microscopic technique used to analyze air samples.

PCMe - Phase Contrast Microscopy fiber equivalent, or an asbestos fiber identified by TEM that is the same size, or equivalent to, a fiber that would be identified by PCM.

PEL - Permissible Exposure Limit, established by OSHA.

PLM - Polarized Light Microscopy, an optical microscopic technique, typically used to analyze bulk asbestos material or soil.

PM-10 - Particulate matter having a diameter less than or equal to 10 microns, assumed to be respirable.

PRP - Potentially Responsible Party.

Pilot Scale - Treatability tests performed on a large scale to simulate the physical, as well as chemical, parameters of a process.

Present Worth Analysis - A summary of costs to be incurred over a period of time, discounted to the present.

q_1^* - Cancer Potency Factor: The lifetime cancer risk for each additional mg/kg of body weight per day of exposure.

QA/QC - Quality assurance/quality control.

RCRA - Resource Conservation and Recovery Act.

RI/FS - Remedial Investigation/Feasibility Study.

RMCL - Recommended Maximum Contaminant Level: Developed under Safe Drinking Water Act.

ROD - Record of Decision, prepared to document the selection of Superfund-financed remedy.

Reference Dose (RfD) - For noncarcinogenic effects, the amount of a chemical that can be taken into the body each day over a lifetime without causing adverse effects.

Remedial Action Alternative - A potential approach to preventing or mitigating site-specific contamination problems, defined in terms of a remedial action technology option or combination of options and the volumes or areas of media to which the option or options will be applied.

Remedial Action Objective - A description of remedial goals for each medium of concern at a site; expressed in terms of the contamination of concern exposure route(s) and receptor(s), and maximum acceptable exposure level(s).

Remedial Action Technology Type (or technology type) - A general category encompassing a number of remedial action technology options that address a similar problem (e.g., capping, containment barriers, chemical treatment).

Remedial Action Technology Process Option (or process option) - A specific process, system, or action that may be used to clean up or mitigate contaminant problems (e.g., clay cap, slurry wall, neutralization).

SARA - Superfund Amendments and Reauthorization Act of 1986 (see CERCLA).

SEM - Scanning Electron Microscopy.

SCVWD - Santa Clara Valley Water District.

SDWA - Safe Drinking Water Act.

SITE - Superfund Innovative Technology Evaluation.

SPCC - Spill Prevention Control and Counter Measure.

STLC - Soluble Threshold Limit Concentration.

SWDA - Solid Waste Disposal Act.

Sensitivity Analysis - A test to determine the overall changes that will result from any small change in one or more procedural elements.

Serpentine - A group of hydrous magnesium-rich phyllosilicate minerals typically found in low grade metamorphic environments; can be fibrous and non-fibrous.

Serpentinite - A rock composition dominated by serpentine.

Structure - Any identified asbestos occurrence in a microscopic sample; includes individual fibers, clusters of fibers, bundles, and fibers attached to particulate matrix debris.

Support Agency - The agency, either the Federal EPA or the State agency, responsible for review and concurrence in developing and selecting a remedy at a CERCLA site.

TAT - Technical Investigation Team.

TCL - Target Compound List.

TEM - Transmission Electron Microscopy, a microscopic technique that can achieve very high (100,000 times) magnification.

TSCA - Toxic Substances Control Act.

TTLC - Total Threshold Limit Concentration, defined by State of California regulations.

Technology Process Option - See remedial action technology process.

Technology Type - See remedial action technology type.

Treatability Test - Studies, usually performed after the FS Phase II, to better define the physical and chemical parameters of technology process options being evaluated for use at CERCLA sites.

USC - United States Code.

USFWS - United States Fish and Wildlife Service.

WCC - Woodward-Clyde Consultants.

APPENDIX B

ASBESTOS ANALYTICAL METHODS

AIR SAMPLE
TEM ANALYSIS METHOD USED IN RI
NIOSH 7402

(Similar to Yamate et. al)

FORMULA: various

M.W.: various

ASBESTOS FIBERS

METHOD: 7402

ISSUED: 5/15/86

OSHA: 2.0 asbestos fibers (>5 µm long/mL)
NIOSH: 0.1 asbestos f/mL [1]
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, f/mL

PROPERTIES: solid, fibrous

SYNONYMS: actinolite asbestos [CAS #13768-00-8], grunerite asbestos (amosite) [CAS #12172-73-5], anthophyllite asbestos [CAS #17068-78-9], chrysotile asbestos [CAS #12001-29-5], crocidolite asbestos [CAS #12001-28-4], tremolite asbestos[CAS #14567-73-8].

SAMPLING

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

FLOW RATE*: 0.5 to 16 L/min (see step 5)

VOL-MIN*: 400 L @ 0.1 fiber/mL
-MAX*: 2000 L
*Adjust to give 100 to 1300 fibers/mm²

SHIPMENT: in antistatic packing material

SAMPLE STABILITY: stable

FIELD BLANKS: 10% (≥2) of samples

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: not determined

OVERALL PRECISION (s_r): see EVALUATION OF METHOD

MEASUREMENT

TECHNIQUE: MICROSCOPY, TRANSMISSION ELECTRON (TEM)

ANALYTE: asbestos fibers

SAMPLE PREPARATION: modified Jaffe wick

EQUIPMENT: transmission electron microscope; energy dispersive X-ray detector and analyzer

CALIBRATION: qualitative SAED/EDX; calibration of TEM and EDS system magnification

RANGE: 100 to 1300 fibers/mm² filter area [6]

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

PRECISION: 0.20

APPLICABILITY: The working range is 0.02 fiber/mL (2000-L air sample) to 1.25 fibers/mL (400-L air sample). The method measures the asbestos fiber concentration in air samples taken on mixed cellulose ester filters. The method measures effectively asbestos fibers of the smallest diameter (<0.05 µm) but allows comparison of fiber counts to be made with phase contrast light microscopic data when fiber diameters are rigidly defined.

INTERFERENCES: Non-asbestiform varieties of the amphiboles may interfere in the TEM analysis if the individual particles have aspect ratios greater than 3:1. These interferences can only be eliminated by quantitative zone axis electron diffraction analysis. High concentrations of background dust interfere with fiber identification.

OTHER METHODS: NIOSH Method 7400 is the phase-contrast microscopic method designed for use with this method.

REAGENTS:

1. Acetone.*

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

3 piece cassette with 50 mm electrically-conductive

1. Sampler: field monitor, 25-mm, [redacted] extension cowl and cellulose ester membrane filter, 0.8- to 1.2-µm pore size, [redacted] pad
   NOTE 1: Analyze representative filters for fiber background before use and discard the filter lot if more than 5 fibers/100 fields are found.
   NOTE 2: An electrically conductive extension cowl is recommended to reduce electrostatic effects on fiber sampling and during sample shipment.
2. Personal sampling pump, ≥0.5 L/min (see step 4 for flow rate), with flexible connecting tubing.
3. Microscope, transmission electron, with electron diffraction and energy-dispersive X-ray capabilities.
4. Diffraction grating replica with known number of lines/mm.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Knife, #10 surgical steel, curved blade.
7. Tweezers.
8. Grids, 200-mesh TEM copper, carbon-coated.
9. Petri dishes, 15-mm depth. The top and bottom of the petri dish must fit together. To assure a tight fit, grind the top and bottom pieces together with an abrasive such as carborundum to produce a ground-glass contact surface.
10. Foam, polyurethane, spongy, 12-mm thick.
11. Low-temperature plasma asher.
12. Filters, Whatman No. 1 qualitative paper or equivalent, or lens paper.
13. Vacuum evaporator.
14. Cork borer, No. 5 (8-mm).
15. Pen, waterproof, marking.
16. Reinforcement, page, gummed.

SPECIAL PRECAUTIONS: Acetone is an extremely flammable liquid (flash point = 0 °F) and precautions must be taken not to ignite it. Heating of >1 mL acetone must be done in a fume hood using a flameless, spark-free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line [3].
2. Remove the top cover from the end of the cowl extension (open face) and orient sampler face down. Wrap the joint between the extender and monitor body with shrink tape to prevent air leaks. Where possible, especially at >80%RH, attach the sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove the caps from the field blank cassettes and store the caps and cassettes in a clean area (bag or box) during the sampling period. Replace the caps in the cassettes when sampling is completed.

4. Sample at 0.5 L/min or greater [8]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E (fibers/mm²), of 100 to 1300 fibers/mm² [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area (A_c= 385 mm²)] for optimum counting precision. Do not exceed 0.5 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/mL), of the fibrous aerosol being sampled by:

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

NOTE: A sampling flow rate of 1 to 4 L/min for 8 hrs is appropriate in non-dusty atmospheres containing ca. 0.1 fiber/mL. Dusty atmospheres require smaller sample volumes; ambient, cleaner atmospheres require larger air samples for optimum counting precision. Flow rates up to 16 L/min are permitted [8].

5. Remove the field monitor at the end of sampling. Replace the plastic top cover and small end caps.
6. Ship the samples upright with conductive cowl attached in a rigid container with sufficient packing material to prevent jostling or damage.
   NOTE: Use only special antistatic polystyrene foam in the shipping container to prevent fiber loss from the sampler filter due to electrostatic forces.

SAMPLE PREPARATION:

7. Remove a circular section from any quadrant of the sample filter using a cork borer.
8. Place the excised section on a clean microscope slide. Affix the filter circle to the slide with a gummed page reinforcement. Label the slide with a waterproof marking pen.
   NOTE: Up to eight filter sections may be attached to the same slide.
9. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 min for the sample filter(s) to fuse and clear.
   NOTE: The "hot block" clearing technique of Method 7400 may be used instead of step 9.

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10. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 x 8-mm section of a graphite rod onto the cleared filter(s). Remove the slide to a clean, dry, covered petri dish [17].

10 11. Place the slide containing the collapsed filters into a low-temperature plasma asher. Etch the filter at 100 °C for ca. 2 min at an O_2 pressure of 130 Pa (1 mm Hg).
   NOTE: Plasma ashers may vary. Determine optimum etching time on blank filters before etching samples.

12. Prepare a second petri dish as a Jaffe wick washer with the wicking substrate prepared from filter or lens paper placed on top of a 12-mm thick disk of 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.
   NOTE: The wicking substrate should be small enough to fit into the petri dish without touching the lid.
13. 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. Fill the dish with acetone until the wicking substrate is saturated.
   NOTE: The level of acetone should be just high enough to saturate the filter paper without creating puddles.

14. Remove about a quarter section of the carbon-coated filter samples using a surgical knife and tweezers. Carefully place the excised filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to 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.

CALIBRATION AND QUALITY CONTROL:

15. Determine the TEM magnification on the fluorescent screen:
    a. Define a field of view on the fluorescent screen either by markings or physical boundaries.
       NOTE: The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric) [14].
    b. Insert a diffraction grating replica into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 °C.
    c. Adjust microscope magnification to 10,000X. 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.
    d. Calculate the true magnification (M) on the fluorescent screen:

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

       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 distance "X."
    e. Once calibration has been completed, note the apparent sizes of 0.25 and 3.0 µm on the fluorescent screen. (These dimensions are essentially the diameter boundary limits for counting asbestos fibers by phase contrast microscopy.) Do not count fibers above 3.0 µm diameter. Count fibers below 0.25 µm diameter but do not include this fraction in calculations when correlating TEM data to PCM counts. Do not count fibers <1 µm long [15].
16. Measure 20 grid openings at random on a 200-mesh copper grid by placing a grid on a glass slide and examining it under the PCM. Use the Walton-Beckett graticule to measure the grid opening diameters. Calculate an average field diameter from the data and use this number to calculate the field area for an average grid opening.
    NOTE: A grid opening is considered as one field.
17. Obtain reference selected area electron diffraction (SAED) patterns from standard asbestos materials prepared for TEM analysis.
    NOTE: This is a visual reference technique. No quantitative SAED analysis is required [14].
    a. Ensure that the specimen holder is oriented at 0 °C tilt for SAED pattern inspection.
    b. Center a fiber, focus, and center the smallest field-limiting aperature on the fiber. Use a 20-cm camera length and 10X binocular head. Obtain an SAED pattern. Photograph each distinctive pattern and keep the photo for comparison to unknowns.
       NOTE: Not all fibers will present SAED patterns; however, the objective lens current may need to be adjusted to give optimum pattern visibility.

18. Acquire energy-dispersive X-ray (EDX) spectra on approximately 5 fibers having diameters between 0.25 and 0.5 µm of each asbestos variety obtained from standard reference materials [14].
    a. Prepare TEM grids of all asbestos varieties.
    b. Use acquisition times (up to 100 sec) sufficient to show a silicon peak at least 75% of the monitor screen height at a vertical scale of ≥500 counts per channel.
    c. Estimate the elemental peak heights visually as follows:
        (1) Normalize all peaks to silicon (assigned an arbitrary value of 10).
        (2) Visually interpret all other peaks present and assign values relative to the silicon peak.
        (3) Determine an elemental profile for the fiber using the elements Na, Mg, Si, Ca, and Fe. Example: 0-4-10-3-<1 [14].
        (4) Determine a typical range of profiles for each asbestos variety and record the profiles for comparison to unknowns.

MEASUREMENT:

19. Perform an SAED pattern inspection on all fibers counted under the TEM, using the procedures given in step 16. Inspect the SAED pattern and assign it to one of the following:
    a. chrysotile;
    b. amphibole;
    c. ambiguous;
    d. none.
20. Obtain EDX spectra in either the TEM or STEM modes from fibers on field samples using the procedure of step 17. Using the SAED pattern and EDX spectrum, classify the fiber:
    a. For a chrysotile structure, obtain EDX spectra on the first five fibers; then one out of ten thereafter. Label profiles of 0-5-10-0-0- to 0-10-10-0-0 as "chrysotile."
    b. For an amphibole structure, obtain EDX spectra on the first 10 fibers; then one out of ten thereafter. Label profiles 0-2-10-0-7 as "possible amosite"; profiles 1-1-10-0-6 as "possible crocidolite"; profiles 0-4-10-3-<1 as "possible tremolite"; and profiles 0-3-10-0-1 as "possible anthophyllite."
    c. For an ambiguous structure, obtain EDX spectra on all fibers. Label profiles similar to the chrysotile profile as "possible chrysotile." Label profiles similar to the various amphiboles as "possible amphiboles." Label all others as "unknown" or "non-asbestos."
    NOTE: Fibers smaller than 0.2 µm in diameter may not produce sufficient peak heights to allow an elemental profile to be determined. These fibers should be identified as "probable" asbestos fibers, if SAED patterns are not determinable.
21. Counting and Sizing:
    a. Insert the sample into the specimen grid holder and scan the grid at 0 °C tilt at low magnification (ca. 300 to 500X). Ensure that the carbon film is intact and unbroken over approximately 75% of the grid openings.
    b. Estimate the number of fibers per grid opening during the low magnification scan (ca. 1000X). Use the following rules when picking grid openings to count [14]:
        (1) Light (<5 fibers per opening): count every grid opening in which the carbon film is intact.
        (2) Moderate (5 to 25 fibers per opening): count every fifth grid opening. If the carbon film is damaged, proceed to the next available opening in which the film is intact.
        (3) Heavy (>25 fibers per opening): count every tenth grid opening. If the carbon film is damaged, proceed to the next available opening in which the carbon film is intact.

c. Increase magnification to 10,000X. Begin counting at one end of the specimen grid and systematically traverse the grid by rows, reversing direction at row ends.

d. Size all asbestos fibers using a scale inscribed or overlaid on the fluorescent screen.

NOTE: Data can be recorded directly off the screen in mm and later converted to µm by computer. Count and record asbestos fibers and structures >1 µ long of all dimensions [15]. However, when comparing TEM data to the PCM counts, only those fibers greater than 5 µm long and between 0.25 and 3.0 µm diameter which have aspect ratios of 3:1 or greater should be included. These size-adjusted data are necessary to test the comparability of counts between the two methods. If the size-adjusted counts between the two methods show reasonable equivalency, then further analysis using the small-fiber data will show what fraction of airborne asbestos fibers is being missed by the PCM method.

e. Count fibers and asbestos structures using the following rules:

(1) Count all particles less than 3 µm diameter which meet the definition of a fiber (aspect ratio ≥3:1, with parallel sides).

NOTE: Particles which are of questionable morphology should be analyzed by SAED and EDX to aid in identification.

(2) Use the same counting rules as were used for the PCM method for counting single fibers and small bundles (A or B rules). Record separately asbestos fibers between 1 and 5 µm long [14].

(3) Record other asbestos structures according to the morphological definitions:

NOTE: Combinations of these structures are labeled according to the dominant quality (i.e., fiber, bundle, cluster, matrix).

(a) Bundle (B) - a compact arrangement of parallel fibers in which separate fibers or fibrils may only be visible at the ends or edges of the bundle. Asbestos bundles having aspect ratios of 3:1 or greater and less than 3 µm in diameter are counted as fibers.

(b) Cluster (C) - a network of randomly-oriented interlocking fibers arranged so that no fiber is isolated from the group. Dimensions of clusters can only be roughly estimated and clusters are defined arbitrarily to consist of more than four individual fibers.

(c) Matrix (M) - one or more fibers attached to or imbedded in a non-asbestos particle.

(4) Count fibers which are partially obscured by the grid.

(5) If a fiber is partially obscured by the grid bar at the edge of the field of view, count it as a fiber greater than 5 µm only if more than 2.5 µm of fiber is visible. Otherwise, log the fiber as a "short" fiber, measuring only that portion which is visible.

(6) Size each fiber as it is counted and log the diameter and length (in mm). Sizing of fibers is accomplished by moving the fiber to the center of the screen and reading directly the length of the fiber from the scale inscribed on the screen. The scale is most efficient if it consists of a series of scale inscribed on the screen or partial circles every 2 cm distant from the center. For fibers which extend beyond the field of view, the fiber must be moved and superimposed upon the concentric circles until its entire length has been measured. When a fiber has been sized, return to the starting point and continue the traverse to the next fiber.

EVALUATION OF METHOD:

The TEM method has been shown to have an overall precision of 20.1% (s_p) in an evaluation of mixed amosite and wollastonite fibers. The estimate of the asbestos fraction, however, had a precision of 11.0% (s_p) [12].

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METHOD WRITTEN BY: James W. Carter; Paul A. Baron, Ph.D.; and David G. Taylor, Ph.D.; NIOSH/DPSE.

[illegible] LABORATORY
1781 South 300 West
P.O. Box 15200
Salt Lake City, Utah 84115-0200

27 November 1985

Dr. James R. Millette
McCrone Environmental Services, Inc.
200 Oakbrook Parkway
Norcross, GA 30093

Dear James,

I am glad to hear of your success with my method. We have never published it even though we have been using it for about six years. You are certainly welcome to quote from the writeup as you require. I thank you for any referrals and recognition pertaining to it. Should you require anything else feel free to call or write.

Sincerely,

Daniel T. Crane
Supervisor,
Microscopy Branch
USDOL-SLCAL
1781 S. 300 W.
Salt Lake City, Utah 84115-0200
(801)-524-4270

AIR SAMPLE
PCM ANALYSIS METHOD USED IN RI
NIOSH 7400

FORMULA: various

M.W.: various

FIBERS

METHOD: 7400
ISSUED: 2/15/84
REVISION #1: 5/15/85

OSHA: 0.5 asbestos fibers (> 5 µm long)/mL
NIOSH: 0.1 asbestos f/mL [1]; 3 glass fibers (>10 µm x <3.5 µm)/mL [2]
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, f/mL

PROPERTIES: solid, fibrous

SYNONYMS: asbestos (actinolite [CAS #13768-00-8], grunerite (amosite) [CAS #12172-73-5], anthophyllite [CAS #17068-78-9], chrysotile [CAS #12001-29-5], crocidolite [CAS #12001-28-4], tremolite [CAS #14567-73-8]); fibrous glass.

SAMPLING

SAMPLER: FILTER
(0.8 to 1.2 µm cellulose ester membrane, 25-mm diameter; foil-wrapped cassette)

FLOW RATE*: ≥ 0.5 L/min (see Step 5)

VOL-MIN*: 400 L @ 0.1 fiber/mL
-MAX*: 2000 L
*Adjust for 100 to 1300 fibers/mm² (step 5)

SHIPMENT: routine

SAMPLE STABILITY: stable

FIELD BLANKS: 10% (≥2) of samples

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: EVALUATION OF METHOD

OVERALL PRECISION (s_r): 0.115 to 0.13 (A Rules) [3]

MEASUREMENT

TECHNIQUE: MICROSCOPY, POSITIVE PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone/triacetin method

COUNTING RULES: Set A (P&CAM 239 [3,4]) or Set B (modified CRS [5])

EQUIPMENT:
1. positive phase-contrast microscope
2. Walton-Beckett graticule (100 µm field diameter): A Rules use G-22; B Rules use Type G-24
3. phase-shift test slide (HSE/NPL)

CALIBRATION: phase-shift detection limit about 3 degrees [7]

RANGE: 100 to 1300 fibers/mm² filter area [6]

ESTIMATED LOD: 7 fibers/mm² filter area

PRECISION: 0.10 to 0.12 (A Rules) [3]

APPLICABILITY: The working range is 0.02 fiber/mL (1920-L air sample) to 1.25 fibers/mL (400-L air sample). The method gives an index of airborne asbestos fibers but may be used for other materials such as fibrous glass by inserting suitable parameters into the counting rules. The method does not differentiate between asbestos and other fibers. Asbestos fibers less than ca. 0.25 µm diameter will not be detected by this method [7].

INTERFERENCES: 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 method introduces changes for improved sensitivity and reproducibility. It also replaces P&CAM 239 [3,4] and Method 7400 (dated 2/15/84).

REAGENTS:

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

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with 50-mm extension cowl with cellulose ester filter, 0.8- to 1.2-µm pore size and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use and discard the filter lot if more than 5 fibers/100 fields are found.
   NOTE 2: An electrically conductive extension cowl is recommended to reduce electrostatic effects on fiber sampling.
   NOTE 3: Extension cowl not required for area or ambient air sampling.
2. Personal sampling pump, ≥0.5 L/min (see step 5 for flow rate), with flexible connecting tubing.
3. Microscope, positive phase contrast, with green or blue filter, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
4. Slides, glass, single-frosted, pre-cleaned, 25 x 75 mm.
5. Cover slips, 22 x 22 mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
6. Lacquer or nail polish.
7. Knife, #10 surgical steel, curved blade.
8. Tweezers.
9. Flask, Guth-type, insulated neck, 250- to 500-mL (with single-holed rubber stopper and elbow-jointed glass tubing, 16 to 22 cm long).
10. Hotplate, spark-free, stirring type; heating mantle; or infrared lamp and magnetic stirrer.
11. Micropipets, 5-µL.
12. Graticule, Walton-Beckett type with 100-µm diameter circular field at the specimen plane (area = 0.00785 mm^2) (Type G-22 for A Rules; Type G-24 for B Rules). Available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 (Telephone [617] 891-6000).
    NOTE: The graticule is custom-made for each microscope. Specify disc diameter needed to fit exactly the ocular of the microscope and the diameter (mm) of the circular counting area (see step 12).
13. HSE/NPL phase contrast test slide, Mark II. Available from PTR Optics Ltd.
14. Telescope, ocular phase-ring centering.
15. Stage micrometer (0.01-mm divisions).
16. Aluminum foil.

SPECIAL PRECAUTIONS: Acetone is an extremely flammable liquid and precautions must be taken not to ignite it. Heating of acetone must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line [3].
2. Fasten the sampler to the worker's lapel as close as possible to the worker's mouth. Remove the top cover from the end of the cowl extension (open face) and orient face down. Wrap the joint between the extender and monitor body with shrink tape to prevent air leaks.
3. Wrap the cassette tightly in aluminum foil being sure not to obstruct airflow into the open end.
   NOTE: This step is not necessary if an electrically conductive cowl is used.
4. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove the caps from the field blank cassettes and store the caps and cassettes in a clean area (bag or box) during the sampling period. Replace the caps in the cassettes when sampling is completed.
5. Sample at 0.5 L/min or greater [8]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E (fibers/mm^2), of 100 to 1300 fibers/mm^2 [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area (A_c= 385 mm^2)] for optimum counting precision (step 22). Do not exceed 1 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/mL), of the fibrous aerosol being sampled by:

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

   NOTE: A sampling flow rate of 1 to 4 L/min for 8 hrs is appropriate in non-dusty atmospheres containing ca. 0.1 fiber/mL. Dusty atmospheres require smaller sample volumes; ambient. clean atmospheres require larger air samples for optimum counting precision.
6. Remove the field monitor at the end of sampling, replace the plastic top cover and small end caps, and store the monitor.
7. Ship the samples, still wrapped in aluminum foil, in a rigid container with sufficient packing material to prevent jostling or damage.
   NOTE: Do not use polystyrene foam in the shipping container because electrostatic forces may cause fiber loss from the sampler filter.

SAMPLE PREPARATION:

NOTE: The object is to produce samples with a smooth (non-grainy) background in a medium with a refractive index equal to or less than 1.46. The method below collapses the filter for easier focusing and produces permanent mounts which are useful for quality control and interlaboratory comparison. Other mounting techniques meeting the above criteria may also be used (e.g., the non-permanent field mounting technique used in P&CAM 239 [1,3,4]).

8. Ensure that the glass slides and cover slips are free of dust and fibers.
9. Place 40 to 60 mL of acetone into a Guth-type flask. Stopper the flask with a single-hole rubber stopper through which a glass tube extends 10 to 12 cm into the flask. The portion of the glass tube which exits the top of the stopper (8 to 10 cm) is bent downward in an elbow which makes an angle of 20 to 30° with the horizontal.
10. Place the flask on a stirring hotplate or wrap in a heating mantle. Heat the acetone gradually to its boiling temperature (ca. 58 °C).
    CAUTION: The acetone vapor must be generated in a ventilated fume hood away from all open flames and spark sources. Alternate heating methods can be used, providing no open flame or sparks are present.
11. 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 steel surgical knife using a rocking motion to prevent tearing.

b. Place the filter or wedge, dust side up, on the slide. Static electricity will usually keep the filter on the slide until it is cleared.
c. Hold the glass slide supporting the filter ca. 1 cm from the glass tube port where the acetone vapor is escaping from the heated flask. The acetone vapor stream should cause a condensation spot on the glass slide ca. 2 to 3 cm in diameter. Move the glass slide gently in the vapor stream. The filter should clear in 2 to 5 sec. If the filter curls, distorts, or is otherwise rendered unusable, the vapor stream is probably not strong enough. Periodically wipe the outlet port with tissue to prevent liquid acetone dripping onto the filter.
d. Using the 5-µL micropipet, place 3.0 to 3.5 µL triacetin on the filter. Gently lower a clean cover slip down onto the filter at a slight angle to reduce the possibility of forming bubbles.
   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 in contact with the edge of the filter under the cover slip, fiber migration may occur at the edges.
e. Glue the edges of the cover slip to the glass slide using a lacquer or nail polish [9].
   NOTE: If clearing is slow, the slide may be warmed on a hotplate (surface temperature 50 °C) for 15 min to hasten clearing. Counting may proceed immediately after clearing and mounting are completed.

CALIBRATION AND QUALITY CONTROL:

12. Calibration of the Walton-Beckett graticule to obtain a counting area (D) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.
    a. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
    b. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
    c. Install the 40 to 45X phase objective.
    d. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
    e. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
    f. 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.
    g. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

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

    Example: If L_o = 108 µm, L_a = 2.93 mm and D = 100 µm, then d_c = 2.71 mm.
    h. Check the field diameter, D (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (mm^2).
13. Microscope adjustments. Follow the manufacturer's instructions and also the following:
    a. Adjust the light source for even illumination across the field of view at the condenser iris.
       NOTE: Köhler illumination is preferred, where available.
    b. Focus on the particulate material to be examined.
    c. Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

d. Use the telescope ocular supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

14. Check the phase-shift detection limit of the microscope periodically.
    a. Remove the HSE/NPL phase-contrast test slide from its shipping container and center it under the phase objective.
    b. Bring the blocks of grooved lines into focus.
       NOTE: The slide consists of seven sets of grooves (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7. The requirements for asbestos counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 and 7 must be invisible. Sets 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 either too low or too high a resolution to be used for asbestos counting.
    c. If the image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.
15. Quality control of fiber counts.
    a. Prepare and count field blanks along with the field samples. Report the counts on each blank. Calculate the mean of the field blank counts and subtract this value from each sample count before reporting the results.
       NOTE 1: The identity of the blank filters should be unknown to the counter until all counts have been completed.
       NOTE 2: If a field blank yields fiber counts greater than 7 fibers/100 fields, report possible contamination of the samples.
    b. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter).
16. Use the following test to determine whether a pair of counts on the same filter should be rejected because of possible bias. This statistic estimates the counting repeatability at the 95% confidence level. Discard the sample if the difference between the two counts exceeds 2.77 $(\bar{F})s_r$, where $\bar{F}$ = average of the two fiber counts and s_r = relative standard deviation, which should be derived by each laboratory based on historical in-house data.
    NOTE: If a pair of counts is rejected as a result of this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts.
17. Enroll each new counter in a training course which compares performance of counters on a variety of samples using this procedure.
    NOTE: To ensure good reproducibility, all laboratories engaged in asbestos counting should participate in an asbestos proficiency testing program such as the NIOSH Proficiency Analytical Testing (PAT) Program and routinely participate with other asbestos fiber counting laboratories in the exchange of field samples to compare performance of counters.

MEASUREMENT:

18. Place the slide on the mechanical stage of the calibrated microscope with the center of the filter under the objective lens. Focus the microscope on the plane of the filter.
19. Regularly check phase-ring alignment and Köhler illumination [7].

20. Select one of the following sets of counting rules:

NOTE: The two sets of rules have been demonstrated to produce equivalent mean counts on a variety of asbestos sample types [5] and must be strictly followed in order to obtain valid results. No hybridizing of the two sets of rules is permitted. The calibration of the microscope with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm).

a. A Rules (same as P&CAM 239 rules [1,3,4]).
   1. Count only fibers longer than 5 µm. Measure the length of curved fibers along the curve.
   2. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
   3. For fibers which cross the boundary of the graticule field, do the following:
      a. Count any fiber longer than 5 µm which lies entirely within the graticule area.
      b. Count as 1/2 fiber any fiber with only one end lying within the graticule area.
      c. Do not count any fiber which crosses the graticule boundary more than once.
      d. Reject and do not count all other fibers.
   4. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
   5. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 fields regardless of fiber count.

b. B Rules

NOTE: The B Rules are preferred analytically because of their demonstrated ability to improve the reproducibility of fiber counts [5].

   1. Count only ends of fibers. Each fiber must be longer than 5 µm and less than 3 µm diameter.
   2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
   3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules b.1 and b.2.
   4. Count visibly free ends which meet rules b.1 and b.2 when the fiber appears to be attached to another particle, regardless of the size of the other particle.
   5. Count the 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 b.1 and b.2.
   6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 fields. Stop at 100 fields, regardless of the fiber count.
   7. Divide the total end count by 2 to yield fiber count.

      NOTE: Split fibers will normally be counted as more than two ends if the free ends meet rules b.1. and b.2.

21. Start counting from one end of the filter and progress along a radial line to the other end, shift either up or down on the filter, and continue in the reverse direction [10]. Select fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. When an agglomerate covers ca. 1/6 or more of the field of view, reject the field and select another. Do not report rejected fields in the number of total fields counted.

NOTE: When counting a 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.

CALCULATIONS:

22. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the total fiber count, F, minus the mean field blank count, B, by the number of fields, n, and the field area, A_f (0.00785 mm^2 for a properly calibrated Walton-Beckett graticule):

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

23. Calculate the concentration, C (fibers/mL), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (385 mm² for a 25-mm filter):

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

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

EVALUATION OF METHOD:

This method is a revision of NIOSH Method P&CAM 239 [1,3,4]. A summary of the revisions is as follows:

A. Sampling:

The change from a 37-mm to a 25-mm filter size was incorporated to improve sensitivity and reduce problems associated with non-uniform fiber loading reported on the 37-mm filters [10]. The change in flow rates allows for 2 m³ 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 affected by changes in flow rate in the range 0.5 to 16 L/min [8].

B. Sample Preparation Technique:

The acetone vapor-triacetin preparation technique has been incorporated in the method as a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [1,3,4,11].

C. Measurement:

1. The inclusion of the Walton-Beckett graticule in the method was made to standardize the field area observed through the eyepiece [6,11].
2. The introduction of the HSE/NPL test slide was made to standardize microscope optics for sensitivity to fiber diameter [7,11].
3. An international collaborative study involved 16 laboratories using prepared slides from the asbestos, cement, milling, mining, textile, and friction material industries [5]. The relative levels of count by different counting rules were:

Sample Type	Number of Samples	Aspect Ratio > 3:1 AIA	Aspect Ratio > 3:1 Mod. CRS*	Aspect Ratio > 5:1 AIA	Aspect Ratio > 5:1 Mod. CRS*
Mining	10	100	127	74	92
Milling	10	100	112	84	95
Asbestos Cement	14	100	146	90	137
Textile Chrysotile	10	100	109	89	99
Friction Material	10	100	130	87	116
Others (Insulation, Amosite)	6	100	127	92	118
TOTAL:	60	MEAN: 100	125	86	110

*Arithmetic means of counts made by different laboratories relative to the AIA counts.

The modified CRS (NIOSH B) Rules were found to be more precise than the AIA (NIOSH A)* Rules. The ranges of relative standard deviations (s_r) which varied with sample type and laboratory were:

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

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

The B Rules have also been favorably received by analysts as less ambiguous and simpler to use; these rules also showed the least bias relative to AIA rules in the collaborative study. An independent NIOSH laboratory study using amosite fibers reported a relative standard deviation, including within- and between-sample variability, of 0.157 for the B Rules [12]. Adding an estimated sampling pump error of 0.05 [13] to the within-sample variability in this study results in an estimate of overall precision, s_T, of 0.102 for the B Rules.

4. Because of past inaccuracies associated with low fiber counts, the minimum loading has been increased to 100 fibers/mm^2 filter area (60 fibers total count). This level yields an overall s_T = 0.13, as indicated in Figure 3 (revised) of P&CAM 239 [3,4] which corresponds to a measurement s_T = 0.12 after removal of pump error [13]. Similarly, at the maximum count of 100 fibers, overall s_T = 0.15 and measurement s_T = 0.10 are obtained.

D. Evaluation of the method using the A and B counting rules will proceed on a continuing basis through the NIOSH Proficiency Analytical Testing (PAT) Program. The new PAT reporting form allows for reporting of results by either set of rules as of January, 1984.

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METHOD REVISED BY: James W. Carter, David G. Taylor, Ph.D., CIH, and Paul A. Baron, Ph.D., NIOSH/DPSE; based on the revised Method P&CAM 239 [1,3,4].

AIR SAMPLE
TEM ANALYSIS REFERENCE METHOD

Yamate, Agarwal, and Gibbons
EPA Draft Method

DRAFT

EPA Report No.

July 1984

This document is a preliminary draft. It has not been formally released by EPA and should not at this stage be construed to represent Agency policy. It is being circulated for comment on its technical accuracy and policy implications.

METHODOLOGY FOR THE MEASUREMENT OF AIRBORNE ASBESTOS BY ELECTRON MICROSCOPY

by

George Yamate
Satish C. Agarwal
Robert D. Gibbons

IIT Research Institute
Chicago, Illinois 60616

Contract No. 68-02-3266

Project Officer

Michael E. Beard
Quality Assurance Division
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

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PREFACE

ABSTRACT

The provisional electron microscope methodology for measuring the concentration of airborne asbestos fibers was refined. The methodology is divided into separate protocols. The step-by-step procedures for each protocol are nearly identical, so that cumulative data can be obtained and uncertainties, especially in asbestos identification, can be clarified. The operational steps encompass (1) type of sample, (2) collection and transport, (3) sample preparation, (4) examination under the transmission electron microscope (TEM) and data collection, (5) data reduction and reporting of results, and (6) quality control-quality assurance.

The TEM analytical protocol is subdivided into three levels of analysis: Level I, for screening many samples; Level II, for regulatory action; and Level III, for confirmatory analysis of controversial samples. Because identification of asbestos structures is critical, the level of analysis is directly related to the information sought:

Level I—morphology and visual selected area electron diffraction (SAED) pattern recognition.

Level II—morphology; visual SAED; and elemental analysis.

Level III—morphology; visual SAED; a selected number of SAED micrographs of zone-axis patterns; and elemental analysis.

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LIST OF ABBREVIATIONS

AEM	analytical electron microscope
EDS	energy dispersive spectrometer
EM	electron microscope
JCPDS	Joint Committee on Powder Diffraction Standards
LTA	low-temperature ashing
NIOSH	National Institute of Occupational Safety and Health
PLM	polarized light microscopy
QC/QA	quality control/quality assurance
SAED	selected area electron diffraction
SEM	scanning electron microscope
STEM	scanning transmission electron microscope
TEM	transmission electron microscope
TSP	total suspended particulates
UICC	Union Internationale Contre le Cancer
XRD	x-ray diffraction
XRF	x-ray fluorescence

SECTION 1

INTRODUCTION

Asbestos is recognized as a health hazard, especially if inspired into the alveolar region of the respiratory tract. Asbestos may be present in air samples, water samples, biological or clinical samples, and other miscellaneous bulk samples, such as ores and food. These various types of samples require different collection methodologies and diverse preparation techniques.

Asbestos analysis methodologies may be categorized as bulk-material analyses, or those providing concentration information, and single-fiber analyses, or those providing morphology, size distribution, and concentration. Bulk-material analysis techniques, which include infrared spectroscopy, differential thermal analysis, and x-ray diffraction analysis (XRD), are limited by an inability to analyze concentrations of less than 1 μg, and by an inability to differentiate between fibrous and nonfibrous forms of minerals.

Single-fiber analysis techniques include optical microscopy and electron microscopy. Optical microscopy employing phase contrast has been promulgated into a monitoring method for the workplace environment (NIOSH-P&CAM 239). In addition, promulgation of a monitoring method for bulk-material asbestos samples (building insulation) using polarized light microscopy (PLM) is presently being considered. However, optical microscopic techniques cannot determine fibers of less than approximately 1 μm in diameter, and phase contrast cannot differentiate between asbestos and nonasbestos fibers.

The electron microscope (EM) provides particle morphology and size, and a degree of identification. A comprehensive study of various EM procedures (Samudra et al., 1977) was conducted in development of a provisional methodology manual, Electron Microscope Measurement of Airborne Asbestos Concentrations (Samudra, 1978). Three EM methods are available: the scanning electron microscope (SEM), the transmission electron microscope (TEM), and the analytical electron microscope (AEM). The SEM, with an x-ray energy-dispersive spectrometer (EDS), permits visual characterization (analogous to reflection optical microscopy) and fiber identification by elemental analysis. The TEM, providing an increased data-acquisition capability, permits visual characterization (in the transmitted mode) and fiber identification by crystal structure analysis. The AEM is a TEM with an EDS, and with the added capability of SEM/STEM (scanning transmission electron microscope) operation, which permits visual characterization (morphology and size) as well as fiber identification using both crystal structure by selected area electron diffraction (SAED) and elemental analysis by EDS.

The original EM methodology was developed for the U.S. Environmental Protection Agency (EPA) for measuring airborne asbestos concentrations, specifically for ambient air and for use as a "screening" tool. Development guidelines included attainable precision and accuracy of results; relative rapidness in use; cost-effectiveness; applicability to a large number of laboratories possessing a TEM (at that time, very few laboratories had TEM's with x-ray analysis capability or an AEM); and procedural steps to be independent of unique or exceptional in-house capabilities of a single laboratory (that is, interlaboratory precision rather than intralaboratory).

In usage, the EM method was successful within its prescribed limitations--that is, the precision and accuracy of results between laboratories using the complete method was good. However, problems that had been recognized in the study developing the methodology (Samudra et al., 1977) arose in the areas of (1) interpretation of airborne, (2) sample collection, (3) need for more exacting identification of asbestos, especially of amphibole type, and (4) use of only part of the methodology.

The present study was undertaken to refine the methodology. The problem areas and related criticisms were addressed within the underlying goals and guidelines set for optimizing the methodology. Protocols similar to a cookbook were not possible since basic knowledge or training was required regarding (1) sample collection, (2) preparation of samples for EM, (3) use of the TEM-AEM, and (4) diffraction pattern analysis. The refined methodology is based on an assumption that each intended user of a particular level of analysis has the necessary background and training to use it.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

The EM methodology for measuring the concentration of airborne asbestos fibers has been refined and specified, and is recommended for field evaluation. The methodology is based on a TEM analytical protocol that is divided into three levels of effort: Level I, for screening many samples; Level II, for regulatory action; and Level III, for confirmatory analysis of controversial samples. The three-level analytical methodology is cost-effective, and will provide the required results for proper assessment of asbestos.

SECTION 3

GUIDELINES FOR UNDERSTANDING THE METHODOLOGY

The methodology is divided into separate protocols. The step-by-step procedures for each protocol are nearly identical, so that cumulative data can be obtained and uncertainties, especially in asbestos identification, can be clarified. These operational steps are:

(1) Type of Sample—Source

(2) Sample Collection and Transport

(3) Sample Preparation for Analysis—Grid Transfer

(4) TEM Examination and Data Collection

(5) Data Reduction and Reporting of Results

(6) Quality Control/Quality Assurance (QC/QA).

The analytical protocol under the TEM examination and data collection procedure is subdivided into three levels of increasing analytical effort in terms of requiring an instrument of greater capability, an electron microscopist with greater expertise, and a longer analytical time. Level I, a monitoring or screening methodology, resembles the present EPA provisional methodology (Samudra et al., 1978; Anderson and Long, 1980). Level II is a regulatory method requiring additional analytical criteria to establish asbestos identification limits, and to provide guidance for Level I analyses by confirming or clarifying visual SAED patterns. Level III, the most sophisticated and the costliest of the methods, is intended for confirming asbestos identification, especially in judicial controversies and other special situations.

In Sections 4, 5, and 6, the protocols for each of the three levels of analysis are presented independently of each other, and thus procedures common to each are repeated. All figures are presented in Appendix A.

Section 7 describes modifications for using the methodology on archival samples, which are samples collected on nonprocedural filter substrates, or samples collected without regard to filter loading levels. Section 8 describes analysis of inorganic sources in bulk-air samples or in bulk form. Section 9 concludes the report with a discussion of analytical aids pertaining to the limits of detection, preparation of blanks, use of computers, magnification calibration, and statistical methodology.

General guidelines for understanding the methodology are discussed in the following paragraphs.

LEVEL OF ANALYSIS

Knowledge of the history, source, and location of the sample, and the purpose and objective of the analysis aids in selecting the correct level of analytical effort. Simply "grinding the samples out" neither is cost-effective nor produces the best results, especially for Level II and Level III analyses. Instead of all Level I, all Level II, or all Level III, the majority of the analyses may be Level I, followed by some Level II. Level III could be used in its entirety or only at the analytical phase. If the source is known to contain no amphibole-type interference, or if chrysotile is of interest, gold-coating can be eliminated.

If a legal proceeding is anticipated, Level III analysis will be required where a chain-of-custody record is kept from collection, transport to the laboratory, preparation, analysis, data reduction, and reporting of results. EM finder grids must be used for grid transfer. In addition, for quality assurance, a second laboratory must be available for analyzing a portion of the sample using the same degree of custodial care. QC/QA protocols must be observed and records kept.

Whenever possible, and especially for unknown source samples, 10 to 20% of each set of samples should be analyzed by Level II analysis prior to using Level I as a screening procedure.

Level I is a relatively rapid procedure, and can be used by many laboratories with access to a conventional TEM. However, Level I results should not be used in legal proceedings. If "positives" or "false positives" are found, especially in areas where asbestos is known to be absent, and the field blank and laboratory blank have been checked, Level II analysis, and possibly Level III analysis, should be performed.

ORDER OF ANALYSIS

The order of analysis is (1) field blanks, (2) laboratory blanks (if needed), and (3) field samples.

COLLECTION AND REPORTING

The counting rule, "minimum 100 fibrous structures per known area (complete grid opening) or 10 grid openings, whichever is first," is a minimum rule for cost limitation. For very low asbestos presence, or for asbestos contamination studies, where particulate loading is high and asbestos presence very low, counting 20 grid openings from each of 2 grids (10 per grid) is recommended.

The EM magnification factor is very high or, conversely, the area of deposit examined is very small. Therefore, although the electron microscopist may report a zero count, the notation "Below Detectable Level" is more appropriate in the sample report. Along the same lines, the electron microscopist

should report observations, measurements, and conclusions as objectively as possible, realizing the subjective nature of his decision-making, such as parallel-sided, 3:1 aspect ratio, number count, size measurements, recognition-discrimination of SAED patterns, and categorizing of asbestos structure.

Data reduction and reporting of results must be consistent and stated. Dimensions of X-fibers (unknown length since complete fiber is not visible) may be doubled, not counted at all, or presented separately. Doubling of the visible portion is recommended, and should be so stated in the report.

Mass or conversion of size measurements to an assumed shape-volume-density relationship, is calculated, and thus is the least reliable of the data, especially for X-fibers, bundles, clusters, and matrices.

Although morphology, SAED, and XRF either singly or in combination will provide identification of asbestos, not all structures will be identified. The nature of the asbestos structure prevents analysis of all structures by SAED and/or by elemental analysis with EDS. Such factors as specimen thickness, orientation, and proximity to other particulates or to the grid wire will prevent attainment of good SAED patterns and limit the effectiveness of chemical analysis.

COSTS

Levels I, II, and III analyses are estimated to require 200, 400, and 1200 min per analysis, respectively. Additional costs will result from collection, preparation, and reporting of results. The equivalent monetary costs will depend on the laboratory rates of the personnel involved.

APPLICATION TO NONAIRBORNE SOURCES

Although the methodology has been developed for airborne asbestos, other types of samples from different sources can be analyzed if the samples are finely divided and placed with proper loading and uniform distribution either on a polycarbonate membrane filter or on a carbon-coated EM grid. Of course, the limitations of the collection and preparation steps must be known and accounted for to prevent inaccuracies in comparing results.

GEOGRAPHICAL CONSIDERATIONS

In some parts of the country, such as the Upper Great Lakes area, the possibility of misidentification is much greater because some nonamphibole minerals have visual SAED patterns that closely resemble those of amphiboles. Gold-coating and Level II analysis will help in differentiating between these minerals.

LABORATORY CONDITIONS

Asbestos analysis involves sustained microscopy for periods of 3 to 7 hours with unscheduled rest breaks. Subjective decisions regarding such factors as morphology, size measurement, visual identification, and possible

EDS make it difficult to break a manipulative physical routine or rhythm. Therefore, a professional environment for the microscopist is essential for effective asbestos analysis. In particular, such factors as unnecessary or redundant procedural steps, lack of personal recognition, and unreasonable deadlines may contribute to poor precision.

SECTION 4

LEVEL I ANALYSIS

SUMMARY OF PROTOCOL

Level I analysis is a monitoring or screening technique. It assesses the amount and type of asbestos structures in the atmosphere through the following steps:

(1) A known volume of air is passed through a polycarbonate membrane filter (pore diameter, 0.4 μm; filter diameter, 37 or 47 mm) to obtain approximately 5 to 10 μg of particulates per cm^2 of filter surface.

(2) The particulate-laden filter is transported in its own filter holder.

(3) The filter is carbon-coated in the holder.

(4) The particulates are transferred to an EM grid using a refined Jaffe wick washer.

(5) The EM grid, containing the particulates, is gold-coated lightly.

(6) The EM grid is examined under low magnification (250X to 1000X) followed by high-magnification (16,000X on the fluorescent screen) search and analysis.

(7) A known area (measured grid opening) is scanned, and the fibrous structures (fibers, bundles, clusters, and matrices) are counted, sized, and identified as to asbestos type (chrysotile, amphibole, ambiguous, or no identity) by morphology and by observing the SAED pattern.

(8) The observations are recorded—a minimum of 100 fibrous structures or 10 grid openings, whichever is first.

(9) The data are reduced and the results reported.

EQUIPMENT, FACILITIES, AND SUPPLIES

The following items are required for Level I analysis:

(1) An 80 or 100-kV TEM with a fluorescent viewing screen inscribed with graduations for estimating the length and width of fibrous particulates.

(2) A vacuum evaporator with a turntable for rotating specimens during coating, for such uses as carbon-coating polycarbonate filters, gold-coating EM grids, and preparing carbon-coated EM grids.

(3) An EM preparation room adjacent to the room housing the EM. This room should either be a clean-room facility, or contain a laminar-flow class-100 clean bench to minimize contamination during EM grid preparation. Filter handling and transfer to EM grids should be performed in a clean atmosphere. Laboratory blanks should be prepared and analyzed weekly to ensure quality of work.

(4) Several refined Jaffe wick washers for dissolving membrane filters.

(5) Miscellaneous EM supplies and chemicals, including carbon-coated 200-mesh copper grids, grid boxes, and chloroform.

(6) Sample collection equipment, including 37-mm-diameter or 47-mm-diameter filter holders, 0.4-μm (pore size) polycarbonate filters, 5.0-μm (pore size) cellulose ester membrane filters for back-up, a sampling pump with ancillary equipment, a tripod, critical orifices or flow meters, and a rain/wind shield.

DESCRIPTION OF METHODOLOGY

1. Type of Samples—Source

This protocol was originally developed for the EPA for measuring airborne asbestos (Samudra et al., 1977; Samudra et al., 1978). A broad interpretation of airborne has been to apply the term to samples obtained from ambient air (the original purpose), aerosolized source materials (such as the asbestos workplace environment, and fugitive dust emissions), bulk-air material (such as total suspended particulate (TSP) samples, dust, and powders) and any other type of sample obtained by nonrestrictive use of (1) collection of a volume of air, (2) separation from the air, and (3) concentration of the particulates onto a substrate. The airborne protocol has also been applied to samples collected in the regulatory areas of the EPA, as compared with, for example, the workplace environment (National Institute of Occupational Safety and Health), mining activities (U.S. Bureau of Mines), and shipboard atmosphere (Federal Maritime Administration).

The present methodology has been optimized for application specifically to samples collected from a volume of air in which the asbestos concentration is considered a minor component of the total particulate loading (other analytical methods are available for samples known to contain high concentrations of asbestos); and in which the particles are less than 15 μm in diameter, since particles greater than 15 μm either are not inhaled or are deposited in the upper respiratory tract and expelled, and preferably less than 10 μm in diameter as recommended by the Clean Air Scientific Advisory Committee (Hileman, 1981), since particles up to 10 μm can be absorbed by the alveolar

region of the lung. These concentration and size restrictions will preclude many air samples collected in an asbestos-processing environment and in bulk-air material from the complete methodology. However, such samples can still be examined with the TEM, within the limitations of the instrument, through changes in preparation techniques--provided the effects on the final results, such as fractionation of size and representativeness of the sample, are carefully considered.

2. Sample Collection and Transport

Sample Collection—

Sampling procedures vary depending on the nature of the sample, purpose of collection, analytical method to be used, sample substrate, and time and cost of sample collection relative to the total analytical effort. Nevertheless, the primary objective of sample collection always is to obtain a representative, unbiased sample.

Impingers, impaction devices, electrostatic precipitators, and thermal precipitators have been used in sample collection, but each has limitations. Presently, the preferred substrates are membrane filters, which are manufactured from different polymeric materials, including polycarbonate, mixed esters of cellulose, polystyrene, cellulose acetate, and cellulose nitrate. Polycarbonate membrane filters differ from the others in being thin, strong, and smooth-surfaced, and in having sieve-like construction (circular pores from top surface to the bottom). The other membrane filters are thicker, have irregular-surfaces, and have depth-filter construction (tortuous paths from top surface to bottom).

Consequently, polycarbonate filters have been selected for airborne asbestos analysis. The collection of small-sized particles (prefer less than 10 μm in diameter), the light loading of particulates, the uniform distribution of particulates attainable using a depth-type backing filter, the smooth surface and circular holes (which aid in determining size and instrument tilt axis), and the relative ease in grid transfer (thin and strong) minimize disadvantages of lack of retention and/or movement of large particles during handling. Other membrane materials, such as the cellulose ester type, are recommended for phase contrast and PLM, heavy particle loadings, and physical retention of large particles.

In microscopical analysis, uniformity of particulate distribution and loading is critical to success. Air samples are taken on 37-mm-diameter or 47-mm-diameter, 0.4-μm (pore size) polycarbonate membrane filters using the shiny, smooth side as the particle-capture surface. Cellulose ester-type membrane filters (pore size, 5.0 μm) are used to support the polycarbonate filter on the support pad (37-mm-diameter personal sampler) or on the support plate (47-mm-diameter holder).

Air monitoring cassettes (37-mm-diameter) of three-piece construction are available from several manufacturers. As with the 47-mm-diameter filters, loading the cassettes with the support pad, back-up filter, and 0.4 μm (pore size) polycarbonate filter should be carefully performed on a class-100 clean bench. Since the filters are held in place by pressure fit rather than by

screw tightening, air must not enter from the sides of the unit; a plastic band or tape (which can double as a label) should be used as a final seal.

Collecting airborne samples with proper loading requires experience. Each of the following techniques is useful in collecting airborne samples for direct microscopy, preserving representative sizes, without diluting particulate deposits:

(1) For long-term sampling at a site, test samples should be returned to the laboratory by express mail service, or air express service or by being hand-carried, and should then be analyzed by scanning electron microscopy.

(2) The estimated particulate loading (deposit is barely visible to the naked eye) should be bracketed by varying the filtration rate and using the same time, or by varying the time and using the same filtration rate.

(3) An automatic particle counter, such as a light-scattering instrument (0.3-μm detection) or a real-time mass monitor (0.1-μm detection), should be used to obtain an approximate particulate-loading level of the area.

Although any one of the three techniques will work, the suggested technique is to take the samples as a set, varying the sampling rates and using the same time so as to obtain filter samples with different particulate loadings. Each set is composed of a minimum of four 37-mm-diameter or 47-mm-diameter filter units--three for different particulate loadings (low, medium, high), and the fourth for a field blank. Suggested sampling rates are 0 for the field blank, 2.48 L/min for the low loading, 7.45 L/min for the medium, and 17.62 L/min for the high, for a 30 min sampling period using a 47-mm-diameter filter holder. Simultaneous sampling will provide at least one sample with a particulate loading suitable for direct EM analysis.

TSP's range from 10 $\mu g/m^3$ in remote, nonurban areas, to 60 $\mu g/m^3$ in near-urban areas, to 220 $\mu g/m^3$ in urban areas. However, for heavily polluted areas, TSP levels may reach 2000 $\mu g/m^3$. A loading of 5 to 10 μg per cm^2 of filter is adequate for EM analysis; values beyond 20 to 25 μg per cm^2 require a dilution treatment. As an example, for 47-mm-diameter filters at face velocities of 3.0 cm/s (2.48 L/min), 9.0 cm/s (7.45 L/min), and 21.2 cm/s (17.62 L/min), respectively, air volumes of 74.4 L, 223.5 L, and 528.6 L are sampled in 30 min. For a TSP level of 200 $\mu g/m^3$, 14.88 μg (1.07 $\mu g/cm^2$), 44.7 μg (3.23 $\mu g/cm^2$), and 105.7 μg (7.63 $\mu g/cm^2$), respectively, would be collected on 47-mm-diameter filters (which would have effective filtration areas of 13.85 cm^2). The sampling time could be increased to 60 min for areas having lower TSP levels, or reduced in a heavily polluted area (source emissions).

Airborne samples from emission sources contain coarse particles (above the respirable size) of large matrix structures, binder materials, road dust, clay minerals, fillers, and other materials. For these samples, a fifth filter unit can be added that has a size-selective inlet (cyclone, impactor, or elutriator) attached prior to the filter unit. The flow pattern and flow

rates of the tandem sampling arrangement must be checked before use. A satisfactory, tested combination presently used in California is a cyclone-filter unit with a D_{50} cut-off of 2.5 μm at 21.7 L/min, and a D_{50} cut-off of 3.5 μm at 15.4 L/min (John and Reischl, 1980). Additional sampling devices, such as impingers (used in biological sampling), impactors, and other designated filter units (for TSP, XRD, or x-ray fluorescence (XRF), for example) can be added to the system to obtain supplementary as well as interrelated data.

This expandable multifilter sampling unit, designated Hydra, offers the following advantages:

(1) It is small, inexpensive, and compact, so that an adult can easily handle it.

(2) It is efficiently designed, and includes a tripod, sampling pump, manifold, critical orifices, and a row of preloaded 37-mm-diameter or 47-mm-diameter filter holders. A rain/wind shield, size-selective cyclone-filter units, tubing, and other extras can be added as needed.

(3) Its sample preparation steps and handling are minimized.

(4) It allows complementary as well as supplementary analysis (TSP, size fractionation, bacteria, and XRF, for example), although additional air sampling capacity is required.

(5) It accommodates ambient air and source emission samples, with or without a size-selective inlet.

(6) It allows synchronous sampling in several places in the vicinity following the same sampling procedure, thereby accommodating particulate concentration fluctuations.

(7) It includes filter holders that serve as transport and storage units.

Hydra's disadvantages are a short sampling period, which may record an episode; a small sampling quantity or volume, which may not indicate the presence of asbestos fibers; and a detection limit of 2×10^4 fibers/m^3 for sampling 1 m^3 of air with the 47-mm-diameter filter.

Using 8 inch x 10 inch, or 102-mm-diameter filter sizes, is not recommended. The sampling units are designed for purposes other than microscopy. Interchanging the type of sample substrate filter (glass fiber or paper to polycarbonate) does not correct the inherent problems of filter size and sampling unit.

Sample Storage and Transport—

Once the sample is acquired, its integrity must be assured, and contamination and loss of fibers prevented, until it is examined under the EM. The low cost and small size of the 37-mm-diameter and 47-mm-diameter filter holders enables them to be used as combination storage and transport containers. The filter holders should be maintained in a horizontal position

during storage and transport to the laboratory so that the particulate-loaded filters can be removed under optimally controlled conditions in the laboratory.

For 47-mm-diameter holders (open-face) to be used in transport or storage, the screw cap is carefully removed, and the shiny, waxy, stiff separator paper used to keep the polycarbonate filters apart is carefully placed on the retaining ring. The cap is then carefully screwed back on so that the separator paper seals and protects the particulate-loaded filter without touching it. The 37-mm-diameter, three-piece filter holder (aerosol monitor) is used in its open-face position, and capped after usage for transport and storage.

When the more expensive 47-mm-diameter holder is to be re-used immediately, the particulate-loaded filter should be carefully removed and placed in a 47-mm-diameter Petri-slide (such as that manufactured by the Millipore Corp.*) This transfer takes place in the field rather than in the laboratory, so that the Petri-slide should be taken into the field. The 37-mm-diameter filter holder or the 47-mm-diameter holder/Petri-slide should be secured and all necessary sample identification marks and symbols applied to the holder.

3. Sample Preparation for Analysis—Grid Transfer

Carbon-Coating the Filter—

The polycarbonate filter, with the sample deposit and suitable blanks, should be coated with carbon as soon as possible after sampling is completed. To begin this procedure, the particulate-loaded 47-mm-diameter polycarbonate filter is removed from the holder and transferred carefully to an open-faced 47-mm-diameter Petri-slide for carbon-coating in the vacuum evaporator (see Figure A1, Appendix A). If the 47-mm-diameter filter is already in the Petri-slide, the cover is replaced with an open-face cover, minimizing filter disruption. The 37-mm-diameter filter is left in the holder, but the upper lid is removed to create an open-faced filter. The open-faced holders are placed on the rotating turntable in the vacuum evaporator for carbon-coating. Figure A2 shows the multiple-coating arrangement in the evaporator; Figure A3 shows a close-up of the 37-mm-diameter and the modified 47-mm-diameter holders for carbon-coating.

For archival filters and those of larger sizes, portions of about 2.5 cm x 2.5 cm should be cut midway between the center and edge using a scalpel. The portions are then attached with cellophane tape to a clean glass microscope slide and placed on the turntable in the vacuum evaporator for coating.

Any high-vacuum carbon evaporator may be used to carbon-coat the filters (CAUTION: carbon sputtering devices should not be used). Typically, the electrodes are adjusted to a height of 10 cm above the level of the filters. A spectrographically pure carbon electrode sharpened to a neck of 0.1 cm x 0.5 cm is used as the evaporating electrode. The sharpened electrode is

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* Millipore Corp., 80-T Ashby Rd., Bedford, Mass. 01730

placed in its spring-loaded holder so that the neck rests against the flat surface of a second carbon electrode.

The manufacturer's instructions should be followed to obtain a vacuum of about 1.33×10^{-3} Pa (1×10^{-5} torr) in the bell jar of the evaporator. With the turntable in motion, the neck of the carbon electrode is evaporated by increasing the electrode current to about 15 A in 10 s, followed by 20 to 25 A for 25 to 30 s. If the turntable is not used during carbon evaporation, the particulate matter may not be coated from all sides, resulting in an undesirable shadowing effect. The evaporation should proceed in a series of short bursts until the neck of the electrode is consumed. Continuous prolonged evaporation should be avoided, since overheating and consequent degradation of the polycarbonate filter may occur, impeding the subsequent step of dissolving the filter. The evaporation process may be observed by viewing the arc through welders goggles (CAUTION: never look at the arc without appropriate eye protection). Preliminary calculations show that a carbon neck of 5 mm^3 volume, when evaporated over a spherical surface 10 cm in radius, will yield a carbon layer that is 40 nm thick.

Following carbon-coating, the vacuum chamber is slowly returned to ambient pressure, and the filters are removed and placed in their respective holders or in clean, marked Petri dishes for storage on a clean bench.

Transfer of the Sample to the EM Grid—

Transferring the collected particulates from the carbon-coated polycarbonate filter to an EM grid is accomplished in a clean room or on a class-100 clean bench. The transfer is made in a Jaffe wick washer, which is usually a glass Petri dish containing a substrate to support the EM grid/carbon-coated membrane filter combination. Solvent is added to a level to just wet the combination and cause gentle dissolution of the membrane with minimum loss or dislocation of the particulates, resulting in a membrane-free EM grid with particles embedded in the carbon film coating. The substrate support can be stainless steel mesh bridges, filter papers, urethane foams, or combinations of these.

The refined Jaffe wick washer is described as follows:

(1) The glass Petri dish (diameter, 10 cm; height, 1.5 cm) is made airtight by grinding the top edge of the bottom dish with the bottom of the cover dish, with water and Carborundum* powder (80 mesh); this creates a ground-glass seal (closer fit) and minimizes the need to refill the Petri dish with added solvent. (The usual glass Petri dish was found not to retain the solvent for long periods of time, and unless the wicking substrate is kept continuously wet, poor solubility of the membrane filter results, leading to a poor-quality EM grid).

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* Carborundum is a registered trademark of the Carborundum Co., Carborundum Center, Niagara Falls, N.Y. 14302.

(2) A combination of foam and a single sheet of 9-cm filter paper is used as the substrate support. A 3-cm x 3-cm x 0.6-cm piece of polyurethane foam (the packing in Polaroid film boxes) is cut and placed in the bottom dish. A 0.5-inch V-shaped notch is cut into the filter paper; the notch is oriented in line with the side of the foam, creating a well for adding solvent. Spectrographic-grade chloroform (solvent) is poured into the Petri dish through the notch until it is level with the top of the foam (also level with the paper). The foam will swell, and care is needed to avoid adding solvent above the filter paper.

(3) On top of the filter paper, pieces of 100-mesh stainless steel screen (0.6 cm x 0.6 cm) are placed, usually in two rows, to make several grid transfers at one time (for such uses as replicas), and to facilitate maintenance of proper identity of each transfer.

(4) A 3-mm section (usually midway between the center and edge) of the carbon-coated polycarbonate filter is cut in a rocking motion with a scalpel. The section may be a square, rectangle, or triangle, and should just cover the 3-mm EM grid.

(5) A section is laid carbon-side down on a 200-mesh carbon-coated EM grid. (Alternatively, Formvar-coated* grids or uncoated EM grids may be used. Here, the carbon coating on the polycarbonate filter forms the grid substrate.) Minor overlap or underlap of the grid by the filter section can be tolerated, since only the central 2-mm portion of the grid is scanned in the microscope. The EM grid and filter combination is picked up at the edges with the tweezers and carefully laid on the damp 100-mesh stainless steel screen. The EM grid-filter combination will immediately "wet out" and remain on the screen.

(6) Once all specimens are placed in the washer, more solvent is carefully added through the notch to maintain the liquid level so that it just touches the top of the paper filter. Raising the solvent level any higher may float the EM grid off the mesh or displace the polycarbonate filter section.

(7) The cover is placed in the washer and oriented in place over the specimen, and a map of the filter/grid/screen arrangement is made on the glass cover and in the logbook.

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* Formvar is a registered trademark of the Monsanto Company, 800 N. Lindbergh Blvd., St. Louis, Mo.

(8) Solvent (chloroform) is added periodically to maintain the level within the washer until the filter is completely dissolved by the wicking action (24 to 48 h).

(9) The temperature in the room must remain relatively constant to minimize condensation of solvent on the bottom of the cover and subsequent falling of solvent drops on the EM grid. Should day-night or other temperature differentials occur, solvent condensation on the under-surface of the cover can be minimized by placing the Jaffe washer at a slight tilt (three glass slides under one edge of the Petri dish parallel to the row of grids) to allow the condensation drops to flow toward the lower edge rather than fall on the EM grids. At temperatures lower than 20°C (68°F), the complete filter solution may take longer than 72 h.

(10) After the polymer is completely dissolved, the stainless steel mesh screen with the EM grid is picked up while wet and set on lens paper tacked to the bottom of a separate Petri dish. The EM grid is then lifted from and placed next to the screen to dry. When all traces of solvent have evaporated, the grid is stored in a grid box and identified by location and grid box in the logbook.

Figure A4 illustrates the Jaffe wick washer method; Figure A5 shows the washer. The foam/filter combination is currently preferred, as is use of a closely fitted (by means of the ground-glass seal) Petri dish.

Gold Coating—

An additional step will aid in subjectively evaluating the SAED pattern. This step is required for specimens from the upper Great Lakes area and for those of unknown origins. After the particulates on the filter are transferred to the EM grid, the grid is held to a glass slide with double stick tape for gold-coating in the vacuum evaporator. Several EM grids may be taped to the glass slide for coating at one time. Approximately 10 mm of 0.015-cm-diameter (0.006-inch) pure gold wire is placed in a tungsten basket (10 cm from the rotating table holding the EM grids) and evaporated onto the grid.

The thin gold-coating establishes an internal standard for SAED analysis. For some mineral species, an internal standard will clarify visual identification of the pattern of a fibrous particulate as being or not being an amphibole species (for example, minnesotaite as opposed to amosite). With experience, differentiation in SAED patterns can be observed. For samples of known geographic origins, gold-coating is optional, since the additional coating hinders observation and identification of small-diameter chrysotile fibers.

4. TEM Examination and Data Collection

Low Magnification Examination of Grids—

Figure A6 shows a modern TEM. The grid is observed in the TEM at magnifications of 250X and 1000X to determine its suitability for detailed

study at higher magnification. The grid is rejected and a new grid used if: (1) the carbon film over a majority of the grid openings is damaged and not intact; (2) the specimen is dark due to incomplete dissolution of the polycarbonate filter; or (3) the particulate loading is too light (unless a blank) or too heavy with particle-particle interactions or overlaps.

TEM Analysis (Morphology and SAED)—

The following guidelines are observed for consistency in the analytical protocol:

(1) Magnification at the fluorescent screen is determined by calibration with a diffraction-grating replica in the specimen holder.

(2) A field of view or "gate" is defined. On some microscopes, the central rectangular portion of the fluorescent screen, which is lifted for photographic purposes, is convenient to use. On others, a scribed circle or the entire circular screen may be used as the field of view. The area of the field of view must be accurately measurable.

(3) The grid opening is selected on a random basis.

(4) The analysis, morphology, and SAED are performed at a tilt angle of 0°.

(5) The recommended instrument settings are: accelerating voltage, 100 kV; beam current, 100 μA; film magnification, 20,000X (which is equivalent to 16,000X on the fluorescent screen for this instrument); and concentric circles of radii 1, 2, 3, and 4 cm on the fluorescent screen.

(6) The grid opening is measured at 1000X.

(7) Since asbestos fibers are found isolated as well as with each other or with other particles in varying arrangements, the fibrous particulates are characterized as asbestos structures:

Fiber (F) is a particle with an aspect ratio of 3:1 or greater, with substantially parallel sides.

Bundle (B) is a particulate composed of fibers in a parallel arrangement, with each fiber closer than the diameter of one fiber.

Cluster (Cl) is a particulate with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

Matrix (M) is a fiber or fibers with one end free and the other end embedded or hidden by a particulate.

Combinations of structures, such as matrix and cluster, matrix and bundle, or bundle and cluster, are categorized by the dominant fiber quality--cluster, bundle, or matrix.

(8) Counting rules for single fibers are:

(a) Particulates meeting the definition of fiber are isolated by themselves. With this definition, edge view of flakes, fragments from cleavage planes, and scrolls, for example, may be counted as fibers.

(b) Count as single entities if separation is equal to or greater than the diameter of a single fiber.

(c Count as single entities if three ends can be seen.

(d) Count as single entities if four ends can be seen.

(e) In general, fibers that touch or cross are counted separately.

(f) Two or more fibers are counted as a bundle if the distances between fibers are less than the diameter of a single fiber, or if the ends cannot be resolved.

(g) Fibrils attached longitudinally to a fiber are counted as part of the fiber and the size (width) is estimated based on the fiber-to-fibril relationship.

(h) A fiber partially hidden by grid wires (one or two ends) is counted, but labeled as an X-fiber. If the number of X-fibers is more than 20% of the fibers identified as asbestos, a larger-mesh EM grid should be used, such as 100 mesh (about 200 μm wide).

(9) Sizing rules for asbestos structures are:

(a) For fibers, widths and lengths are obtained by orienting the fibers to the inscribed circles on the fluorescent screen. Since estimates are within ±1 mm, small-diameter fibers have greater margins of error. Fibers less than 1 mm at the fluorescent screen magnification level are characterized as being 1 mm. A cylindrical shape is assumed for fibers. X-fibers are sized by measuring their entire visible portions in the grid opening.

(b) Bundles and clusters are sized by estimating their widths and lengths. The sum of individual diameters is used to obtain the total width, and an average length for the total length. A laminar-sheet shape

is assumed, with the average diameter of the individual fiber as the thickness.

(c) Matrices are sized by adding the best estimates of individual fiber components. A laminar or sheet structure is assumed for volume calculation.

(10) The method of sizing is as follows:

(a) An asbestos structure is recognized, and its location in the rectangular "gate" relative to the sides, inscribed circles, and other particulates, is memorized.

(b) The structure is moved to the center for SAED observation and sizing.

(c) Sizing is performed using the inscribed circles. If the structure, such as a fiber, extends beyond the rectangular "gate" (field of view), it is superimposed across the series of concentric circles (several times, if necessary) until the entire structure is measured.

(d) The structure is returned to its original location by recall of the location, and scanning is continued.

Figure A7 illustrates some of the counting and morphology guidelines used in determining asbestos structures.

TEM Procedure—

The TEM procedure is as follows:

(1) EM grid quality is assessed at 250X.

(2) Particulate loading is assessed at 1000X.

(3) A grid opening is selected at random, examined at 1000X, and sized.

(4) A series of parallel traverses is made across the grid opening at the film magnification of 20,000X. Starting at one corner, and using the tilting section of the fluorescent screen as a "gate" or "chute," the grid opening is traversed. Movement through the "gate" is not continuous, but rather is a stop/go motion. On reaching the end of one traverse, the image is moved the width of one "gate," and the traverse is reversed. These parallel traverses are made until the entire grid opening has been scanned.

(5) Asbestos structures are identified morphologically and counted as they enter the "gate."

(6) The asbestos structure is categorized as fiber (with or without X-) bundle, cluster, or matrix, and sized through use of the inscribed circles.

(7) The structure (individual fiber portion) is centered and focused, and the SAED pattern is obtained through use of the field-limiting aperture.

(a) SAED patterns from single fibers of asbestos minerals fall into distinct groups. The chrysotile asbestos pattern has characteristic streaks on layer lines other than the central line, and some streaking also on the central line. Spots of normal sharpness are present on the central layer line and on alternate lines (that is, 2nd, 4th etc.) The repeat distance between layer lines is about 0.53 nm.

(b) Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines also of about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(c) Transmission electron micrographs and SAED patterns obtained with asbestos standard samples should be used as guides to fiber identification. An example is the "Asbestos Fiber Atlas" (Mueller et al., 1975).

(8) From visual examination of the SAED pattern, the structure is classified as belonging to one of four categories: (1) chrysotile, (2) amphibole group (includes amosite, crocidolite, anthophyllite, tremolite, and actinolite), (3) ambiguous (incomplete spot patterns), or (4) no identification. SAED patterns cannot be inspected for some fibers. Reasons for the absence of a recognizable diffraction pattern include contamination of the fiber, interference from nearby particles, fibers that are too small or too thick, and nonsuitable orientation of the fiber. Some chrysotile fibers are destroyed in the electron beam, resulting in patterns that fade away within seconds of being formed. Some patterns are very faint and can be seen only under the binocular microscope. In general, the shortest available camera length must be used, and the objective lens current may need to be adjusted to give optimum pattern visibility for correct identification. A 20-cm camera length and a 10X binocular are recommended for inspecting the SAED pattern on the tilted screen.

(9) Additional grid openings are selected, scanned, and counted until either the total number of structures counted exceeds 100 per known area or a minimum of 10 grid openings has been scanned, whichever is first.

(10) The TEM data should be recorded in a systematic form so that it can be processed rapidly. Sample information, instrument parameters, and the sequence of operations should be tabulated for ease in data reduction and subsequent reporting of results. Figure A8 shows an example of a data sheet used in Level I analysis.

Figure A9 illustrates the method of scanning a full-grid opening. The "field of view" method of counting previously included in the provisional methodology, which is based on randomly selected fields of view, has been discontinued. Originally, the method was recommended for medium loading level on the filter (50 to 300 fibers per grid opening). However, if samples are collected at three different loading levels and the optimum is selected, this medium loading on the filter will not be used. Samples with grid openings containing 50 to 300 fibers may be used as laboratory fiber preparations or selected source samples, but in field samples the particulate loading is usually of much higher concentration than the fiber. Filter loading is characterized by the particulate concentration, not by fiber concentration.

5. Data Reduction and Reporting of Results

Data Reduction—

From the data sheet, size measurements are converted to microns (16,000X screen magnification), mass of asbestos structure is calculated, and other characterizing parameters are calculated through use of a hand calculator or computer. (Appendix B, an example of a computer printout from Level I analysis, shows reduced data--that is, what was found on the specified number of grid openings or area examined.) These measurements are summarized and related to the volume of air sampled and the total effective filtration area (area of deposit). Size measurements of X-fibers may be doubled and noted, or kept as a separate category.

Fiber number concentration is calculated from the equation

$$\text{Fibers/m}^3 = \frac{\text{Total no. of fibers}}{\text{No. of EM fields}} \times \frac{\text{Total effective filter area, cm}^2}{\text{Area of an EM field, cm}^2} \times \frac{1}{\text{Volume of air sampled, m}^3}$$

The number of X-fibers, bundles, clusters, and matrices are calculated in a similar manner. X-fibers may be included with fibers if they are few in

number. Similarly, their corresponding mass (from their size measurements) may be included.

Fiber mass for each type of asbestos (chrysotile or amphibole) in the sample is calculated by assuming that both chrysotiles and amphiboles have circular cross-sections (cylindrical shape) and that the width measurements are one diameter. The density of chrysotile is assumed to be 2.6 g/cm^3, and of amphiboles to be 3.0 g/cm^3. The individual mass is calculated from the equation

$$\text{Mass, } \mu g = \frac{\pi}{4} \times (\text{length, } \mu m) \times (\text{diameter, } \mu m)^2 \times (\text{density, } g/cm^3) \times 10^{-6}$$

The total mass concentration of fibers for each type of asbestos is then calculated from the total mass of all the individual fibers of that type.

The individual masses of bundles, clusters, and matrices are calculated by assuming a laminar or sheet-like structure with an average thickness of the fiber make-up of the structure. Again, the density of chrysotile is assumed to be 2.6 g/cm^3, and of amphiboles to be 3.0 g/cm^3. The individual masses are calculated from the equation

$$\text{Mass, } \mu g = (\text{length, } \mu m) \times (\text{width, } \mu m) \times (\text{thickness, } \mu m) \times (\text{density, } g/cm^3) \times 10^{-6}$$

The total mass for each type of structure for each type of asbestos is the sum of all the individual masses.

Other characterizing parameters of the asbestos structures are: (1) length and width distribution of fibers, (2) aspect ratio distribution of fibers, and (3) relationships of fibers, bundles, clusters, and matrices.

Reporting of Results—

The data in their acquired and reduced forms are reported as summarized, or, depending on the purpose of the analysis, are further reduced to present the interrelationships of the various characterizing parameters. Again, the Level I methodology is a monitoring or screening technique, and its limitations, such as the possibility of "false positives" and misidentification, should be noted.

6. Quality Control/Quality Assurance

Sampling procedures will vary depending on the type of sample, objectives of the sampling, and time/cost factors. The primary goals of sampling are to obtain a representative sample at the location and time of sampling, and to maintain sample integrity. The sampling team will have written sampling

procedures, and the field chief and/or designated individual will be responsible for all record-keeping (including sample identification, labeling, logging of data, site description, and meteorological conditions), pre- and post-collection checks, and continuous sample custody and sign-outs until the sample is delivered to the laboratory and transferred to the appropriate quality assurance officer (QAO). Verification of sampling times, flow rates, equipment calibration, and taking of field blanks will be checked and recorded in the field logbook.

Samples are turned over to the QAO for logging into a project logbook. Each sample is carefully examined for gross features, such as tears, breaks, and overall condition of container. The QAO registers the as-received sample number and other designated information, and assigns a simple internal code number that will accompany the sample through the preparation stage, grid transfer, grid analysis, data reduction, and reporting of results.

After being logged into the project logbook, the sample is transferred to the custody of the electron microscopy staff, where every precaution is taken to maintain sample integrity and to prevent contamination and loss of collected particulates. During storage and transport, the filters in their respective holders are maintained in a horizontal position at all times.

The sample logging, handling, and storing procedures ensure that all samples can be readily located and identified throughout the course of a program. The QAO has divisional responsibility for QC/QA activities, and must see that the laboratory maintains high standards. He must be aware of current standards of analysis, and must ensure that internal quality control standards, instrument calibration, and records of samples and completed analyses are kept for ease of later retrieval and use.

For quality control, internal laboratory blanks are analyzed at least once a week, which may or may not coincide with a sample batch blank. In addition, a magnification calibration of the EM using a carbon grating replica (2,160 lines per mm) is performed once a week. The results are recorded in an EM instrument log, along with other routine instrumental performance checks. All photographs, TEM, SEM, and STEM images are recorded in a photo log. These QC results are documented for inspection by the QAO.

SECTION 5

LEVEL II ANALYSIS

SUMMARY OF PROTOCOL

Level II analysis is a regulatory technique consisting of Level I analysis plus chemical elemental analysis. Morphology, size, SAED pattern, and chemical analysis are obtained sequentially. By a process of elimination, mineral fibers are identified as chrysotile, amphibole, ambiguous, or "no-identity" by morphology and SAED pattern. X-ray elemental analysis is used to categorize the amphibole fibers, identify the ambiguous fibers, and confirm or validate chrysotile fibers.

Level II analysis is summarized as follows:

(1) A known volume of air is passed through a polycarbonate membrane filter (pore diameter, 0.4 μm; filter diameter, 37 or 47 mm) to obtain approximately 5 to 10 μg of particulates per cm^2 of filter surface.

(2) The particulate-laden filter is transported in its own filter holder.

(3) The filter is carbon-coated in the holder.

(4) The particulates are transferred to an EM grid using a refined Jaffe wick washer.

(5) The EM grid, containing the particulates, is gold-coated lightly.

(6) The EM grid is examined under low magnification (250X to 1000X) followed by high-magnification (16,000X on the fluorescent screen) search and analysis.

(7) A known area (measured grid opening) is scanned, and the fibrous structures (fibers, bundles, clusters, and matrices) are counted, sized, and identified as to asbestos type (chrysotile, amphibole, ambiguous, or no identity) by morphology and by observing the SAED pattern; and finally by elemental analysis using EDS.

(8) The observations are recorded—a minimum of 100 fibrous structures or 10 grid openings, whichever is first.

(9) The data are reduced and the results reported.

EQUIPMENT, FACILITIES, AND SUPPLIES

The following items are required for Level I analysis:

(1) A modern 100-kV TEM equipped with an EDS. A scanning accessory as found in a STEM will increase the versatility and analytical capability for very small fibers and for fibers adjacent to other particulate matter. The microscope should be equipped with the fluorescent viewing screen inscribed with graduation of known radii to estimate the length and width of fibrous particulates.

(2) A vacuum evaporator with a turntable for rotating specimens during coating, for such uses as carbon-coating polycarbonate filters, gold-coating EM grids, and preparing carbon-coated EM grids.

(3) An EM preparation room adjacent to the room housing the EM. This room should either be a clean-room facility, or contain a laminar-flow class-100 clean bench to minimize contamination duing EM grid preparation. Filter handling and transfer to EM grids should be performed in a clean atmosphere. Laboratory blanks should be prepared and analyzed weekly to ensure quality of work.

(4) Several refined Jaffe wick washers for dissolving membrane filters.

(5) Miscellaneous EM supplies and chemicals, including carbon-coated 200-mesh copper grids, grid boxes, and chloroform.

(6) Sample collection equipment, including 37-mm-diameter or 47-mm-diameter filter holders, 0.4-μm (pore size) polycarbonate filters, 5.0-μm (pore size) cellulose ester membrane filters for back-up, a sampling pump with ancillary equipment, a tripod, critical orifices or flow meters, and a rain/wind shield.

DESCRIPTION OF METHODOLOGY

1. Type of Samples—Source

This protocol is an expansion of the method originally developed for the EPA for measuring airborne asbestos (Samudra et al., 1977; Samudra et al., 1978). A broad interpretation of airborne has been to apply the term to samples obtained from ambient air (the original purpose), aerosolized source materials (such as the asbestos workplace environment, and fugitive dust emissions), bulk-air material (such as total suspended particulate (TSP) samples, dust, and powders) and any other type of sample obtained by nonrestrictive use of (1) collection of a volume of air, (2) separation from the air, and (3) concentration of the particulates onto a substrate. The airborne protocol has also been applied to samples collected in the regulatory areas of the EPA, as compared with, for example, the workplace environment (National

Institute of Occupational Safety and Health), mining activities (U.S. Bureau of Mines), and shipboard atmosphere (Federal Maritime Administration).

The present methodology has been optimized for application specifically to samples collected from a volume of air in which the asbestos concentration is considered a minor component of the total particulate loading (other analytical methods are available for samples known to contain high concentrations of asbestos); and in which the particles are less than 15 μm in diameter, since particles greater than 15 μm either are not inhaled or are deposited in the upper respiratory tract and expelled, and preferably less than 10 μm in diameter as recommended by the Clean Air Scientific Advisory Committee (Hileman, 1981), since particles up to 10 μm can be absorbed by the alveolar region of the lung. These concentration and size restrictions will preclude many air samples collected in an asbestos-processing environment and in bulk-air material from the complete methodology. However, such samples can still be examined with the TEM, within the limitations of the instrument by changes in preparation techniques--provided the effects on the final results, such as fractionation of size and representativeness of the sample, are carefully considered.

2. Sample Collection and Transport

Sample Collection—

Sampling procedures vary depending on the nature of the sample, purpose of collection, analytical method to be used, sample substrate, and time and cost of sample collection relative to the total analytical effort. Nevertheless, the primary objective of sample collection always is to obtain a representative, unbiased sample.

Impingers, impaction devices, electrostatic precipitators, and thermal precipitators have been used in sample collection, but each has limitations. Presently, the preferred substrates are membrane filters, which are manufactured from different polymeric materials, including polycarbonate, mixed esters of cellulose, polystyrene, cellulose acetate, and cellulose nitrate. Polycarbonate membrane filters differ from the others in being thin, strong, and smooth-surfaced, and in having sieve-like construction (circular pores from top surface to the bottom). The other membrane filters are thicker, have irregular-surfaces, and have depth-filter construction (tortuous paths from top surface to bottom).

Consequently, polycarbonate filters have been selected for airborne asbestos analysis. The collection of small-sized particles (prefer less than 10 μm in diameter), the light loading of particulates, the uniform distribution of particulates attainable using a depth-type backing filter, the smooth surface and circular holes (which aid in determining size and instrument tilt axis), and the relative ease in grid transfer (thin and strong) minimize disadvantages of lack of retention and/or movement of large particles during handling. Other membrane materials, such as the cellulose ester type, are recommended for phase contrast and PLM, heavy particle loadings, and physical retention of large particles.

In microscopical analysis, uniformity of particulate distribution and loading is critical to success. Air samples are taken on 37-mm-diameter or 47-mm-diameter, 0.4-μm (pore size) polycarbonate membrane filters using the shiny, smooth side as the particle-capture surface. Cellulose ester-type membrane filters (pore size, 5.0 μm) are used to support the polycarbonate filter on the support pad (37-mm-diameter personal sampler) or on the support plate (47-mm-diameter holder).

Air monitoring cassettes (37-mm-diameter) of three-piece construction are available from several manufacturers. As with the 47-mm-diameter filters, loading the cassettes with the support pad, back-up filter, and 0.4 μm (pore size) polycarbonate filter should be carefully performed on a class-100 clean bench. Since the filters are held in place by pressure fit rather than by screw tightening, air must not enter from the sides of the unit; a plastic band or tape (which can double as a label) should be used as a final seal.

Collecting airborne samples with proper loading requires experience. Each of the following techniques is useful in collecting airborne samples for direct microscopy, preserving representative sizes, without diluting particulate deposits:

(1) For long-term sampling at a site, test samples should be returned to the laboratory by express mail service, or air express service or by being hand-carried, and should then be analyzed by scanning electron microscopy.

(2) The estimated particulate loading (deposit is barely visible to the naked eye) should be bracketed by varying the filtration rate and using the same time, or by varying the time and using the same filtration rate.

(3) An automatic particle counter, such as a light-scattering instrument (0.3-μm detection) or a real-time mass monitor (0.1-μm detection), should be used to obtain an approximate particulate-loading level of the area.

Although any one of the three techniques will work, the suggested technique is to take the samples as a set, varying the sampling rates and using the same time so as to obtain filter samples with different particulate loadings. Each set is composed of a minimum of four 37-mm-diameter or 47-mm-diameter filter units--three for different particulate loadings (low, medium, high), and the fourth for a field blank. Suggested sampling rates are 0 for the field blank, 2.48 L/min for the low loading, 7.45 L/min for the medium, and 17.62 L/min for the high, for a 30 min sampling period using a 47-mm-diameter filter holder. Simultaneous sampling will provide at least one sample with a particulate loading suitable for direct EM analysis.

TSP's range from 10 $\mu g/m^3$ in remote, nonurban areas, to 60 $\mu g/m^3$ in near-urban areas, to 220 $\mu g/m^3$ in urban areas. However, for heavily polluted areas, TSP levels may reach 2000 $\mu g/m^3$. A loading of 5 to 10 μg per cm^2 of filter is adequate for EM analysis; values beyond 20 to 25 μg per cm^2 require a dilution treatment. As an example, for 47-mm-diameter filters at face velocities of 3.0 cm/s (2.48 L/min), 9.0 cm/s (7.45 L/min), and 21.2 cm/s

(17.62 L/min), respectively, air volumes of 74.4 L, 223.5 L, and 528.6 L are sampled in 30 min. For a TSP level of 200 $\mu g/m^3$, 14.88 μg (1.07 $\mu g/cm^2$), 44.7 μg (3.23 $\mu g/cm^2$), and 105.7 μg (7.63 $\mu g/cm^2$), respectively, would be collected on 47-mm-diameter filters (which would have effective filtration areas of 13.85 cm^2). The sampling time could be increased to 60 min for areas having lower TSP levels, or reduced in a heavily polluted area (source emissions).

Airborne samples from emission sources contain coarse particles (above the respirable size) of large matrix structures, binder materials, road dust, clay minerals, fillers, and other materials. For these samples, a fifth filter unit can be added that has a size-selective inlet (cyclone, impactor, or elutriator) attached prior to the filter unit. The flow pattern and flow rates of the tandem sampling arrangement must be checked before use. A satisfactory, tested combination presently used in California is a cyclone-filter unit with a D_{50} cut-off of 2.5 μm at 21.7 L/min, and a D_{50} cut-off of 3.5 μm at 15.4 L/min (John and Reischl, 1980). Additional sampling devices, such as impingers (used in biological sampling), impactors, and other designated filter units (for TSP, XRD, or x-ray fluorescence (XRF), for example) can be added to the system to obtain supplementary as well as interrelated data.

This expandable multifilter sampling unit, designated Hydra, offers the following advantages:

(1) It is small, inexpensive, and compact, so that an adult can easily handle it.

(2) It is efficiently designed, and includes a tripod, sampling pump, manifold, critical orifices, and a row of preloaded 37-mm-diameter or 47-mm-diameter filter holders. A rain/wind shield, size-selective cyclone-filter units, tubing, and other extras can be added as needed.

(3) Its sample preparation steps and handling are minimized.

(4) It allows complementary as well as supplementary analysis (TSP, size fractionation, bacteria, and XRF, for example), although additional air sampling capacity is required.

(5) It accommodates ambient air and source emission samples, with or without a size-selective inlet.

(6) It allows synchronous sampling in several places in the vicinity following the same sampling procedure, thereby accommodating particulate concentration fluctuations.

(7) It includes filter holders that serve as transport and storage units.

Hydra's disadvantages are a short sampling period, which may catch an episode; a small sampling quantity or volume, which may not indicate the presence of asbestos fibers; and a detection limit of 2×10^4 fibers/m^3 for sampling 1 m^3 of air with the 47-mm-diameter filter.

Using 8 inch x 10 inch, or 102-mm-diameter filter sizes, is not recommended. The sampling units are designed for purposes other than microscopy. Interchanging the type of sample substrate filter (glass fiber or paper to polycarbonate) does not correct the inherent problems of filter size and sampling unit.

Sample Storage and Transport—

Once the sample is acquired, its integrity must be assured, and contamination and loss of fibers prevented, until it is examined under the EM. The low cost and small size of the 37-mm-diameter and 47-mm-diameter filter holders enables them to be used as combination storage and transport containers. The filter holders should be maintained in a horizontal position during storage and transport to the laboratory so that the particulate-loaded filters can be removed under optimally controlled conditions in the laboratory.

For 47-mm-diameter holders (open-face) to be used in transport or storage, the screw cap is carefully removed, and the shiny, waxy, stiff separator paper used to keep the polycarbonate filters apart is carefully placed on the retaining ring. The cap is then carefully screwed back on so that the separator paper seals and protects the particulate-loaded filter without touching it. The 37-mm-diameter, three-piece filter holder (aerosol monitor) is used in its open-face position, and capped after usage for transport and storage.

When the more expensive 47-mm-diameter holder is to be reused immediately, the particulate-loaded filter should be carefully removed and placed in a 47-mm-diameter Petri-slide (such as that manufactured by the Millipore Corp.*). This transfer takes place in the field rather than in the laboratory, so that the Petri-slide should be taken into the field. The 37-mm-diameter filter holder or the 47-mm-diameter holder/Petri-slide should be secured and all necessary sample identification marks and symbols applied to the holder.

3. Sample Preparation for Analysis—Grid Transfer

Carbon-Coating the Filter—

The polycarbonate filter, with the sample deposit and suitable blanks, should be coated with carbon as soon as possible after sampling is completed. To begin this procedure, the particulate-loaded 47-mm-diameter polycarbonate filter is removed from the holder and transferred carefully to an open-faced 47-mm-diameter Petri-slide for carbon-coating in the vacuum evaporator (see Figure A1, Appendix A). If the 47-mm-diameter filter is already in the Petri-slide, the cover is replaced with an open-face cover, minimizing filter disruption. The 37-mm-diameter filter is left in the holder, but the upper lid is removed to create an open-faced filter. The open-faced holders are placed on the rotating turntable in the vacuum evaporator for carbon-coating. Figure A2 shows the multiple-coating

* Millipore Corp., 80-T Ashby Rd., Bedford, Mass. 01730

arrangement in the evaporator; Figure A3 shows a close-up of the 37-mm-diameter and the modified 47-mm-diameter holders for carbon-coating.

For archival filters and those of larger sizes, portions of about 2.5 cm x 2.5 cm should be cut midway between the center and edge using a scalpel. The portions are then attached with cellophane tape to a clean glass microscope slide and placed on the turntable in the vacuum evaporator for coating.

Any high-vacuum carbon evaporator may be used to carbon-coat the filters (CAUTION: carbon sputtering devices should not be used). Typically, the electrodes are adjusted to a height of 10 cm above the level of the filters. A spectrographically pure carbon electrode sharpened to a neck of 0.1 cm x 0.5 cm is used as the evaporating electrode. The sharpened electrode is placed in its spring-loaded holder so that the neck rests against the flat surface of a second carbon electrode.

The manufacturer's instructions should be followed to obtain a vacuum of about 1.33×10^{-3} Pa (1×10^{-5} torr) in the bell jar of the evaporator. With the turntable in motion, the neck of the carbon electrode is evaporated by increasing the electrode current to about 15 A in 10 s, followed by 20 to 25 A for 25 to 30 s. If the turntable is not used during carbon evaporation, the particulate matter may not be coated from all sides, resulting in an undesirable shadowing effect. The evaporation should proceed in a series of short bursts until the neck of the electrode is consumed. Continuous prolonged evaporation should be avoided, since overheating and consequent degradation of the polycarbonate filter may occur, impeding the subsequent step of dissolving the filter. The evaporation process may be observed by viewing the arc through welders goggles (CAUTION: never look at the arc without appropriate eye protection). Preliminary calculations show that a carbon neck of 5 mm^3 volume, when evaporated over a spherical surface 10 cm in radius, will yield a carbon layer that is 40 nm thick.

Following carbon-coating, the vacuum chamber is slowly returned to ambient pressure, and the filters are removed and placed in their respective holders or in clean, marked Petri dishes for storage on a clean bench.

Transfer of the Sample to the EM Grid—

Transferring the collected particulates from the carbon-coated polycarbonate filter to an EM grid is accomplished in a clean room or on a class-100 clean bench. The transfer is made in a Jaffe wick washer, which is usually a glass Petri dish containing a substrate to support the EM grid/carbon-coated membrane filter combination. Solvent is added to a level to just wet the combination and cause gentle dissolution of the membrane with minimum loss or dislocation of the particulates, resulting in a membrane-free EM grid with particles embedded in the carbon film coating. The substrate support can be stainless steel mesh bridges, filter papers, urethane foams, or combinations of these.

The refined Jaffe wick washer is described as follows:

(1) The glass Petri dish (diameter, 10 cm; height, 1.5 cm) is made airtight by grinding the top edge of the bottom dish with the bottom of the cover dish, with water and Carborundum* powder (80 mesh); this creates a ground-glass seal (closer fit) and minimizes the need to refill the Petri dish with added solvent. (The usual glass Petri dish was found not to retain the solvent for long periods of time, and unless the wicking substrate is kept continuously wet, poor solubility of the membrane filter results, leading to a poor-quality EM grid).

(2) A combination of foam and a single sheet of 9-cm filter paper is used as the substrate support. A 3-cm x 3-cm x 0.6-cm piece of polyurethane foam (the packing in Polaroid film boxes) is cut and placed in the bottom dish. A 0.5-inch V-shaped notch is cut into the filter paper; the notch is oriented in line with the side of the foam, creating a well for adding solvent. Spectrographic-grade chloroform (solvent) is poured into the Petri dish through the notch until it is level with the top of the foam (also level with the paper). The foam will swell, and care is needed to avoid adding solvent above the filter paper.

(3) On top of the filter paper, pieces of 100-mesh stainless steel screen (0.6 cm x 0.6 cm) are placed, usually in two rows, to make several grid transfers at one time (for such uses as replicas), and to facilitate maintenance of proper identity of each transfer.

(4) A 3-mm section (usually midway between the center and edge) of the carbon-coated polycarbonate filter is cut in a rocking motion with a scalpel. The section may be a square, rectangle, or triangle, and should just cover the 3-mm EM grid.

(5) A section is laid carbon-side down on a 200-mesh carbon-coated EM grid. (Alternatively, Formvar-coated† grids or uncoated EM grids may be used. Here, the carbon coating on the polycarbonate filter forms the grid substrate.) Minor overlap or underlap of the grid by the filter section can be tolerated, since only the central 2-mm portion of the grid is scanned in the microscope. The EM grid and filter combination is picked up at the edges with the tweezers and carefully laid on the damp 100-mesh

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* Carborundum is a registered trademark of the Carborundum Co., Carborundum Center, Niagara Falls, N.Y. 14302.

† Formvar is a registered trademark of the Monsanto Company, 800 N. Lindbergh Blvd., St. Louis, Mo.

stainless steel screen. The EM grid-filter combination will immediately "wet out" and remain on the screen.

(6) Once all specimens are placed in the washer, more solvent is carefully added through the notch to maintain the liquid level so that it just touches the top of the paper filter. Raising the solvent level any higher may float the EM grid off the mesh or displace the polycarbonate filter section.

(7) The cover is placed in the washer and oriented in place over the specimen, and a map of the filter/grid/screen arrangement is made on the glass cover and in the logbook.

(8) Solvent (chloroform) is added periodically to maintain the level within the washer until the filter is completely dissolved by the wicking action (24 to 48 h).

(9) The temperature in the room must remain relatively constant to minimize condensation of solvent on the bottom of the cover and subsequent falling of solvent drops on the EM grid. Should day-night or other temperature differentials occur, solvent condensation on the under-surface of the cover can be minimized by placing the Jaffe washer at a slight tilt (three glass slides under one edge of the Petri dish parallel to the row of grids) to allow the condensation drops to flow toward the lower edge rather than fall on the EM grids. At temperatures lower than 20°C (68°F), the complete filter solution may take longer than 72 h.

(10) After the polymer is completely dissolved, the stainless steel mesh screen with the EM grid is picked up while wet and set on lens paper tacked to the bottom of a separate Petri dish. The EM grid is then lifted from and placed next to the screen to dry. When all traces of solvent have evaporated, the grid is stored in a grid box and identified by location and grid box in the logbook.

Figure A4 illustrates the Jaffe wick washer method; Figure A5 shows the washer. The foam/filter combination is currently preferred, as is use of a closely fitted (by means of the ground-glass seal) Petri dish.

Gold Coating—

An additional step will aid in subjectively evaluating the SAED pattern. This step is required for specimens from the upper Great Lakes area and for those of unknown origins. After the particulates on the filter are transferred to the EM grid, the grid is held to a glass slide with double-stick tape for gold-coating in the vacuum evaporator. Several EM grids may be taped to the glass slide with double-stick tape for gold-coating in the vacuum evaporator. For comparison, one-half of the EM grids may be coated and the other one-half not coated; recognition of the gold-coating is helpful in searching and x-ray analysis. Several EM grids may be taped to the glass slide for

coating at one time. Approximately 10 mm of 0.015-cm-diameter (0.006-inch) pure gold wire is placed in a tungsten basket (10 cm from the rotating table holding the EM grids) and evaporated onto the grid.

The thin gold-coating establishes an internal standard for SAED analysis. For some mineral species, an internal standard will clarify visual identification of the pattern of a fibrous particulate as being or not being an amphibole species (for example, minnesotaite as opposed to amosite). With experience, differentiation in SAED patterns can be observed. For samples of known geographic origins, gold-coating is optional, since the additional coating hinders observation and identification of small-diameter chrysotile fibers.

4. TEM Examination and Data Collection

Figure A10 shows a modern TEM with capabilities for elemental analysis with an EDS. The grid is observed in the TEM at magnifications of 250X and 1000X to determine its suitability for detailed study at higher magnification. The grid is rejected and a new grid used if: (1) the carbon film over a majority of the grid openings is damaged and not intact; (2) the specimen is dark due to incomplete dissolution of the polycarbonate filter; or (3) the particulate loading is too light (unless a blank) or too heavy with particle-particle interactions or overlaps.

TEM Analysis (Morphology, SAED, and X-Ray Analysis)—

The following guidelines are observed for consistency in the analytical protocol:

(1) Magnification at the fluorescent screen is determined by calibration with a diffraction-grating replica in the specimen holder.

(2) A field of view or "gate" is defined. On some microscopes, the central rectangular portion of the fluorescent screen, which is lifted for photographic purposes, is convenient to use. On others, a scribed circle or the entire circular screen may be used as the field of view. The area of the field of view must be accurately measurable.

(3) The grid opening is selected on a random basis.

(4) The analysis, morphology, and SAED are performed at a tilt angle of 0°.

(5) The recommended instrument settings are: accelerating voltage, 100 kV; beam current, 100 μA; film magnification, 20,000X (which is equivalent to 16,000X on the fluorescent screen for this instrument); and concentric circles of radii 1, 2, 3, and 4 cm on the fluorescent screen.

(6) The grid opening is measured at low magnification (about 1000X).

(7) Since asbestos fibers are found isolated as well as with each other or with other particles in varying arrangements, the fibrous particulates are characterized as asbestos structures:

Fiber (F) is a particle with an aspect ratio of 3:1 or greater with substantially parallel sides.

Bundle (B) is a particulate composed of fibers in a parallel arrangement, with each fiber closer than the diameter of one fiber.

Cluster (Cl) is a particulate with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

Matrix is a fiber or fibers with one end free and the other end embedded or hidden by a particulate.

Combinations of structures, such as matrix and cluster, matrix and bundle, or bundle and cluster, are categorized by the dominant fiber quality--cluster, bundle, and matrix.

(8) Counting rules for single fibers, which are illustrated in Figure A7 are as follows:

(a) Particulates meeting the definition of fiber are isolated by themselves. With this definition, edge view of flakes, fragments from cleavage planes, and scrolls, for example, may be counted as fibers.

(b) Count as single entities if separation is equal to or greater than the diameter of a single fiber.

(c) Count as single entities if three ends can be seen.

(d) Count as single entities if four ends can be seen.

(e) In general, fibers that touch or cross are counted separately.

(f) Two or more fibers are counted as a bundle if the distances between fibers are less than the diameter of a single fiber, or if the ends cannot be resolved.

(g) Fibrils attached longitudinally to a fiber are counted as part of the fiber and the size (width) is estimated based on the fiber-to-fibril relationship.

(h) A fiber partially hidden by grid wires (one or two sides of the grid opening) is counted, but labeled as an X-fiber (X-F) in the structure column. If the number of X-fibers is high enough to affect the size distribution (mass, etc.), a large-mesh EM grid should be used, such as 100 mesh (about 200 μm wide).

(9) Sizing rules for asbestos structures are:

(a) For fibers, widths and lengths are obtained by orienting the fibers to the inscribed circles on the fluorescent screen. Since estimates are within ±1 mm, small-diameter fibers have greater margins of error. Fibers less than 1 mm at the fluorescent screen magnification level are characterized as being 1 mm. A cylindrical shape is assumed for fibers. X-fibers are sized by measuring their entire visible portions in the grid opening.

(b) Bundles and clusters are sized by estimating their widths and lengths. The sum of individual diameters is used to obtain the total width, and an average length for the total length. A laminar-sheet shape is assumed, with the average diameter of the individual fiber as the thickness.

(c) Matrices are sized by adding the best estimates of individual fiber components. A laminar or sheet structure is assumed for volume calculation.

(10) The method of sizing is as follows:

(a) An asbestos structure is recognized, and its location in the rectangular "gate" relative to the sides, inscribed circles, and other particulates is memorized.

(b) The structure is moved to the center for SAED observation and sizing.

(c) Sizing is performed using the inscribed circles. If the structure, such as a fiber, extends beyond the rectangular gate (field of view), it is superimposed across the series of concentric circles (several times, if necessary) until the entire structure is measured.

(d) The structure is returned to its original location by recall of the location, and scanning is continued.

Analytical Procedure—

The analytical procedure is as follows:

(1) EM grid quality is assessed at 250X.

(2) Particulate loading is assessed at 1000X.

(3) A grid opening is selected at random, examined at 1000X, and sized.

(4) A series of parallel traverses is made across the grid opening at the film magnification of 20,000X. Starting at one corner, and using the tilting section of the fluorescent screen as a "gate" or "chute," the grid opening is traversed. Movement through the "gate" is not continuous, but rather is a stop/go motion. On reaching the end of one traverse, the image is moved the width of one "gate," and the traverse is reversed. These parallel traverses are made until the entire grid opening has been scanned.

(5) Asbestos structures are identified morphologically and counted as they enter the "gate."

(6) The asbestos structure is categorized as fiber (with or without X-) bundle, cluster, or matrix, and sized through use of the inscribed circles.

(7) The structure (individual fiber portion) is centered and focused, and the SAED pattern is obtained through use of the field-limiting aperture.

(a) SAED patterns from single fibers of asbestos minerals fall into distinct groups. The chrysotile asbestos pattern has characteristic streaks on layer lines other than the central line, and some streaking also on the central line. Spots of normal sharpness are present on the central layer line and on alternate lines (that is, 2nd, 4th etc.) The repeat distance between layer lines is about 0.53 nm.

(b) Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and have repeat distances between layer lines also of about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(c) Transmission electron micrographs and SAED patterns obtained with asbestos standard samples should be used as guides to fiber identification. An example is the "Asbestos Fiber Atlas" (Mueller et al., 1975).

(8) From visual examination of the SAED pattern, the structure is classified as belonging to one of four categories: (1) chrysotile, (2) amphibole group (includes amosite, crocidolite, anthophyllite, tremolite, and actinolite), (3) ambiguous (incomplete spot patterns), or (4) no identification. SAED patterns cannot be inspected for some fibers. Reasons for the absence of a recognizable diffraction pattern include contamination of the fiber, interference from nearby particles, fibers that are too small or too thick, and nonsuitable orientation of the fiber. Some chrysotile fibers are destroyed in the electron beam, resulting in patterns that fade away within seconds of being formed. Some patterns are very faint and can be seen only under the binocular microscope. In general, the shortest available camera length must be used, and the objective lens current may need to be adjusted to give optimum pattern visibility for correct identification. A 20-cm camera length and a 10X binocular are recommended for inspecting the SAED pattern on the tilted screen.

(9) The specimen holder is tilted for optimum x-ray detection (40° tilt for the JEOL* 100C instrument's Tracor Northern† NS 880 analyzer and Kevex‡ detector). The categorized asbestos structure is maintained in its centered position for x-ray analysis by means of the Z-control.

(10) The spot size of the electron beam is reduced and stigmated to overlap the fiber. As an option for STEM instruments, the electron beam may be used in the spot mode and the x-ray analysis performed on a small area of the structure.

(11) The EDS is used to obtain a spectrum of the x-rays generated by the asbestos structure.

(12) The profile of the spectrum is compared with profiles obtained from asbestos standards; the best (closest) match identifies and categorizes the structure. The image of the spectrum may be photographed, or the peak heights (Na, Mg, Si, Ca, Fe) recorded for normalizing at a later time. No background spectra or constant acquisition time is required since the shape of the spectrum (profile) is the criteria. Acquisition of x-ray counts may be to a constant time; to a constant peak height for a selected element, such as silicon (1.74 keV); or just

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* JEOL (U.S.A.) Inc., 11 Dearborn Road, Peabody, Mass. 01960

† Tracor Northern Inc., 2551-T.W. Beltway Hwy., Middleton, Wis. 53562

‡ Kevex Corp., Chess Dr., Foster City, Calif. 94404

long enough to get an adequate idea of the profile of the spectra, and then aborted. Figure A11 illustrates spectra obtained from various asbestos standards and used as referenced profiles.

(13) The specimen holder is returned to 0° tilt to examine other asbestos structures.

(14) Scanning is continued until all structures are identified, measured, analyzed, and categorized in the grid opening.

(15) Additional grid openings are selected, scanned, and counted until either the total number of structures counted exceeds 100 per known area, or a minimum of 10 grid openings has been scanned, whichever is first.

(16) The TEM data should be recorded in a systematic form so that they can be processed rapidly. Sample information, instrument parameters, and the sequence of operations should be tabulated for ease in data reduction and subsequent reporting of results. Figure A12 shows an example of a data sheet used in Level II analysis.

Figure A9 illustrates the method of scanning a full-grid opening. The "field of view" method of counting, which is based on randomly selected fields of view, has been discontinued. Originally, the method was recommended for medium loading level on the filter (50 to 300 fibers per grid opening). However, if samples are collected at three different loading levels and the optimum is selected, this medium loading on the filter will not be used. Samples with grid openings containing 50 to 300 fibers may be used as laboratory fiber preparations or selected source samples, but in field samples, the particulate loading is usually of much higher concentration than the fiber. Filter loading is characterized by particulate concentration, not by fiber concentration.

EDS is relatively time-consuming, and becomes redundant if used as repetitive analysis for a confirmatory check on chrysotile fibers. Chrysotile identity by morphology and visual SAED analysis is not as controversial as amphibole identification and categorization.

The following rules are recommended for EDS analysis (Level II):

(1) For chrysotile structure identification, the first five are analyzed by EDS, then one out of every 10.

(2) For amphibole structure identification, the first 10 are analyzed by EDS, then one out of every 10.

(3) For amphibole structure identification and categorization, all confirmed amphiboles are analyzed by EDS.

(4) For ambiguous structure identification and categorization, all are analyzed by EDS.

Energy dispersive x-ray analysis as used in asbestos analysis is semiquantitative at best. X-ray analyzer manufacturers may claim quantitative results based on calibration standards and sophisticated computer software, but such claims are based on stoichiometric materials and extension of work with XRF instrumentation. Asbestos has a varying elemental composition. The electron beam in an EM is of varying size, and not all instruments are equipped to measure the beam current hitting the specimen. The size of the specimen has an effect on the x-ray output, and nearby materials may fluoresce and add to the overall x-ray signals being generated. Moreover, specimen tilting results in a loss of x-ray acquisition from particles hidden by grid wires or by other particles.

The only consistency in x-ray analysis is that the intensity of the output, within restrictions, is proportional to the mass, therefore providing the semiquantitative analytical possibility. Asbestos minerals have been found to have a characteristic profile, although not an exact duplicate of each other. For example, the Mg:Si ratio of chrysotile may vary from 5:10 to 10:10, averaging about 7:10. The ratio can be used to confirm the morphology and visual SAED analysis.

Table 1 illustrates the phenomena of variability with resemblance for some of the amphibole fibers. Peak heights and profile measurements were taken.

To aid in the visual perspective of the spectrum profile, the peak heights were normalized to a silicon value of 10, resulting in a five-number series that is relatively easy to visualize--as in the following examples:

chrysotile	~ 0-7-10-0-0
tremolite	~ 0-4-10-3-<1
crocidolite	~ 1-1-10-0-6
anthophyllite	~ 0-3-10-0-1
amosite	~ 0-2-10-0-7

These relationships are approximate, since chrysotile can vary from 0-5-10-0-0 to 0-10-10-0-0. However, for the others, the variation is only about one point, such that the profile (shape) of the five elements (Na, Mg, Si, Ca, Fe) is recognizable.

5. Data Reduction and Reporting of Results

Data Reduction--

From the data sheet, size measurements are converted to microns (16,000X screen magnification), mass of asbestos structure is calculated, and other characterizing parameters are calculated through use of a hand calculator or computer. (Appendix C, an example of a computer printout from Level II analysis, shows reduced data--that is, what was found on the specified number of grid openings or area examined.) These measurements are summarized and related to the volume of air sampled and the total effective filtration area

TABLE 1. PROFILE COMPARISON OF ASBESTOS STANDARDS

Asbestos Type	Size, μ	Na	Mg	Si	Ca	Fe	Profile
Amosite (GP-38A)	0.19 x 1.44 (stigmate)		182	497		386	0-4-10-0-8
	0.19 x 0.75 (STEM)		186	528		387	0-4-10-0-7
	0.19 x 1.25		181	352		289	0-5-10-0-8
	0.19 x 0.88 (100 s)		226	870		674	0-3-10-0-8
	0.25 x 1.81 (100 s)		576	4207		3338	0-1-10-0-8
	0.12 x 1.56		253	2049		1515	0-1-10-0-7
	0.31 x 2.38		256	2127		1613	0-1-10-0-8
	0.19 x 1.56		276	1696		1116	0-2-10-0-7
	Repeat		477	2945		1959	0-2-10-0-7
Anthophyllite (AP-45)	0.56 x 2.38 (stigmate)		631	2577		349	0-2-10-0-1
	0.31 x 2.38 (stigmate)		640	1670		71	0-4-10-0-0
	0.31 x 5.19 (stigmate)		1064	3610		466	0-3-10-0-1
	0.19 x 1.56 (stigmate)		507	2191		309	0-2-10-0-1
	0.19 x 1.88 (stigmate)		787	2286		257	0-3-10-0-1
Crocidolite (CR-37)	0.19 x 0.81 (stigmate)	131	100	885		501	2-1-10-0-6
	0.06 x 0.50 (stigmate)	28	28	205		115	1-1-10-0-6
	0.06 x 0.69 (stigmate)	37	35	171		96	2-2-10-0-6
	0.12 x 1.00 (stigmate)	44	53	379		204	1-1-10-0-5
	Repeat (STEM)	70	64	612		333	1-1-10-0-5
	0.12 x 0.62 (stigmate)	56	65	479		260	1-1-10-0-5
	0.12 x 1.12 (stigmate)	53	56	326		166	2-2-10-0-5
	0.19 x 1.56 (stigmate)	78	83	735		421	1-1-10-0-6
	0.06 x 1.69 (stigmate)	45	48	290		159	2-2-10-0-6
	Repeat (STEM)	72	85	892		463	1-1-10-0-5
	Repeat (STEM)	35	42	373		237	1-1-10-0-6
	Repeat (STEM)	16	22	166		104	1-1-10-0-6
Tremolite (T-79)	0.38 x 2.19 (stigmate)		138	368	93		0-4-10-2-0
	0.38 x 2.19 (spot)		114	327	80		0-4-10-2-0
	0.25 x 1.75 (stigmate)		80	197	65		0-4-10-3-0
	0.25 x 1.75 (spot)		95	252	62		0-4-10-2-0
	Repeat (stigmate)		70	211	51		0-3-10-2-0
	(STEM-100 s)		376	1118	245		1-3-10-2-0
	(STEM-100 s)		135	364	72		0-4-10-2-0
	(STEM-100 s)		1454	4810	1235		0-3-10-3-0
	(STEM-100 s)		64	191	48		0-3-10-2-0
	(STEM-100 s)		1072	3114	882		0-3-10-3-0
	(STEM-40 s)		46	113	27		0-4-10-2-0
	(STEM-40 s)		123	333	94		0-4-10-3-0

(area of deposit). Size measurements of X-fibers may be doubled and noted, or kept as a separate category.

Fiber number concentration is calculated from the equation

$$\text{Fibers/m}^3 = \frac{\text{Total no. of fibers}}{\text{No. of EM fields}} \times \frac{\text{Total effective filter area, cm}^2}{\text{Area of an EM field, cm}^2} \times \frac{1}{\text{Volume of air sampled, m}^3}$$

The number of X-fibers, bundles, clusters, and matrices are calculated in a similar manner. X-fibers may be included with fibers if they are few in number. Similarly, their corresponding mass (from their size measurements) may be included.

Fiber mass for each type of asbestos (chrysotile or amphibole) in the sample is calculated by assuming that both chrysotiles and amphiboles have circular cross-sections (cylindrical shape) and that the width measurements are one diameter. The density of chrysotile is assumed to be 2.6 g/cm^3, and of amphiboles to be 3.0 g/cm^3. The individual mass is calculated from the equation

$$\text{Mass, } \mu\text{g} = \frac{\pi}{4} \times (\text{length, } \mu\text{m}) \times (\text{diameter, } \mu\text{m})^2 \times (\text{density, g/cm}^3) \times 10^{-6}$$

The total mass concentration of fibers for each type of asbestos is then calculated from the total mass of all the individual fibers of that type.

The individual masses of bundles, clusters, and matrices are calculated by assuming a laminar or sheet-like structure with an average thickness of the fiber make-up of the structure. Again, the density of chrysotile is assumed to be 2.6 g/cm^3, and of amphiboles to be 3.0 g/cm^3. The individual masses are calculated from the equation

$$\text{Mass, } \mu\text{g} = (\text{length, } \mu\text{m}) \times (\text{width, } \mu\text{m}) \times (\text{thickness, } \mu\text{m}) \times (\text{density, g/cm}^3) \times 10^{-6}$$

The total mass for each type of structure for each type of asbestos is the sum of all the individual masses.

Other characterizing parameters of the asbestos structures are: (1) length and width distribution of fibers, (2) aspect ratio distribution of fibers, and (3) relationships of fibers, bundles, clusters, and matrices.

Reporting of Results—

The data and their subsequent reduction are reported as summarized, or can be further reduced to present the interrelationships of the various characterizing parameters. Figure A13 is an example of the EM data report; Figure A14 is an example of the sample summary report.

The methodology can establish the limits of identity for unknown samples, act as a QC/QA method for Level I analysis, and satisfy most of the identification criteria for asbestos.

6. Quality Control/Quality Assurance

Sampling procedures will vary depending on the type of sample, objectives of the sampling, and time/cost factors. The primary goals of sampling are to obtain a representative sample at the location and time of sampling, and to maintain sample integrity. The sampling team will have written sampling procedures, and the field chief and/or designated individual will be responsible for all record-keeping (including sample identification, labeling, logging of data, site description, and meteorological conditions), pre- and post-collection checks, and continuous sample custody and sign-outs until the sample is delivered to the laboratory and transferred to the appropriate quality assurance officer (QAO). Verification of sampling times, flow rates, equipment calibration, and taking of field blanks will be checked and recorded in the field logbook.

Samples are turned over to the QAO for logging into a project logbook. Each sample is carefully examined for gross features, such as tears, breaks, and overall condition of container. The QAO registers the as-received sample number and other designated information, and assigns a simple internal code number that will accompany the sample through the preparation stage, grid transfer, grid analysis, data reduction, and reporting of results.

After being logged into the project logbook, the sample is transferred to the custody of the electron microscopy staff, where every precaution is taken to maintain sample integrity and to prevent contamination and loss of collected particulates. During storage and transport, the filters in their respective holders are maintained in a horizontal position at all times.

The sample logging, handling, and storing procedures ensure that all samples can be readily located and identified throughout the course of a program. The QAO has divisional responsibility for QC/QA activities, and must see that the laboratory maintains high standards. He must be aware of current standards of analysis, and must ensure that internal quality control standards, instrument calibration, and records of samples and completed analyses are kept for ease of later retrieval and use.

For quality control, internal laboratory blanks are analyzed at least once a week, which may or may not coincide with a sample batch blank. In addition, a magnification calibration of the EM using a carbon grating replica (2,160 lines per mm) is performed once a week. The results are recorded in an EM instrument log, along with other routine instrumental performance checks. All photographs, TEM, SEM, and STEM images are recorded in a photo log. These QC results are documented for inspection by the QAO.

SECTION 6

LEVEL III ASBESTOS ANALYSIS

DISCUSSION OF PROTOCOL

The Level III protocol is an extension of the Level II analysis procedures described in Section 5. This extension may be necessitated by the need for positive identification of the specific amphibole species in situations where (1) fundamental disagreements between parties involved in a litigation require further clarification; (2) for identification purposes, e.g., as causative agents in medical diagnosis or studies; (3) for quality control of Level II analysis in special situations, and/or; (4) for source samples whether as bulk material or bulk-air type where a legal judgment is anticipated.

Since an SAED pattern may be considered as a signature of the crystal structure of the diffracting crystal (mineral fiber or particulate), the mineral giving the pattern can be identified by comparison of measured and standard sets of d-spacings and interplanar angles (θ) from SAED patterns obtained in near-exact zone axis orientations. Such identification, however, may not be absolute without the provision of SAED patterns from more than one zone-axis orientation.

The Level III analysis is an objective, confirmatory-type analysis and consists of Level II analysis plus quantitative SAED analysis from two different near-exact zone-axis orientations on a selected number of fibers identified for detailed SAED analysis during the course of Level II analysis.

The Level III analytical procedure consists of locating the selected fibers contained in gold-coated grid openings (for internal calibration); photographing the fibers under bright-feld illumination; obtaining (by tilting) and recording two zone-axis SAED patterns from each selected fiber; and obtaining (recording and photographing) representative EDS spectra from the subject fiber.

The present Level III protocol is based on the following guidelines:

(1) Maintenance of procedural continuity so that results of Level II analytical effort will aid in conducting the Level III effort.

(2) Since detailed SAED analysis on all the fibers measured in Level II analysis is not possible due to time and cost restraints, a selection criterion is needed to assure representative analyses.

(3) The primary emphasis in Level III analysis is on the positive identification of the amphibole type.

(4) The present protocol is designed to allow greater flexibility and freedom of decision for the microscopist in determining the selection criteria since, due to practical constraints (position, orientation, contamination, etc.), all fibrous particulates may not be suitable for detailed SAED work.

(5) It is recommended that approximately 20% (at least 10%) of the fibers examined in Level II analysis be selected for Level III SAED analysis. Fibers which would be classified as "amphiboles" or "ambiguous" in Level II analysis should be more often included for Level III analysis as compared to those fibers which could be readily identified as "not asbestos." In cases where the majority of the fibers in Level II belong to a single, easily-identifiable species (e.g., chrysotile), fibers that are different should be more often selected for detailed Level III analysis. This flexibility in selection criteria will maximize the gain (meaningful information) from Level III effort beyond what would be achieved from the analysis of 10-20% randomly selected fibrous particulates.

(6) The electron microscope grids used in Level III analysis (also Level II if Level III is anticipated) should be finder grids so that location of fibers examined could be referenced for quantitative SAED and for future rechecking.

(7) Level III analysis should always be conducted by or under the close supervision of a professional electron microscopist knowledgeable in crystallography, SAED analysis, mineralogy, plus Level I and Level II asbestos analyses. If such expertise is not available in-house, an outside consultant should be retained.

(8) If enforcement proceedings and possible legal involvement may be part of the analytical procedure, the sample collection procedure entails additional record-keeping to maintain sample integrity. The field crew chief or a designated individual initiates, in addition to normal QC/QA activities, a chain-of-custody record. The sample is collected by the field team or by a representative of the adversary party in the presence of each other, and is sealed and signed for with the date and time. The designated individual acknowledges receipt of the collected sample. In transferring the sample, the designate signs a release of the sample in the presence of the new recipient, who notes the date and time, and signs for acceptance in the designate's presence. The chain of custody ensures that only responsible personnel have access to and control of the sample, thereby avoiding the possibility of

contamination before and after transport to the laboratory. At the laboratory, the QAO has first access to the sealed sample container, and signs for it after obtaining a signed release by the hand-carrier.

SUMMARY OF PROTOCOL

(1) An EM grid is prepared as directed in Level II analysis using finder or locator grids instead of regular 200-mesh grids.

(2) The particulate-loaded grid is then one-half or completely coated with a thin layer of gold.

(3) The gold-coated grid is placed in a tilt-rotation or a double-tilt specimen holder, and examined in the AEM or STEM.

(4) At low magnification the specimen grid is examined, and a grid opening is selected and identified for reference.

(5) Fibers identified for detailed Level III SAED work during Level II analysis, employing the selection criteria described under Level III guidelines, are now examined one at a time.

(6) A bright-field image of the fiber is taken at 0° tilt and at the magnification of analysis (20,000X).

(7) With the tilt-rotation or double-tilt combination, well-defined SAED patterns of two different zone-axis orientations are observed and photographed. The fiber location with respect to the edges of the grid opening or to other particulates may prevent more than one zone-axis orientation from being obtained for some fibers.

(8) X-ray elemental analysis is taken of the fiber after the SAED patterns. The EDS analysis also may be affected by proximity of the fiber to the edge of the grid opening or to other particles if tilting of the specimen is required for efficient use of the EDS. An image of the spectra is taken along with a record of the peak heights (the presence of grid peaks, such as Cu or Ni, as well as gold-coating may serve as markers).

(9) As explained earlier, due to time and cost considerations, at least 10% (preferably 20%) of the fibers examined in Level II are analyzed in Level III work.

(10) Those fibers whose EDS elemental analysis points to a possible amphibole identification are selected for SAED pattern indexing.

(11) Parameters of interest obtained form zone-axis SAED patterns are: the camera constant, CC (obtained from the gold ring); the diffraction spot spacing d_1 (along the slant vector), d_2 (along a row); the inter-row spacing, R; and the interplanar angle $\theta_{1,2}$. See Figure A18 for details.

(12) The reciprocal lattice values of the d-spacings d_1 and d_2 and the inter-row spacing (R) are converted into direct lattice spacings and then d_1, d_2, R, and $\theta_{1,2}$ are compared to those of standard amphibole species listed in JCPDS Powder Diffraction Files, values computed from lattice parameters and crystal structures, or SAED Standard Pattern File developed internally from known amphibole minerals regulated by EPA.

EQUIPMENT, FACILITIES, AND SUPPLIES

Essential items required for a Level III analysis are:

- A 100-kV AEM equipped with the fluorescent viewing screen inscribed with graduations of known radii to estimate the lengths and widths of fibrous particulates; or a modern 100-kV TEM equipped with an EDS. A scanning accessory as found in an STEM will increase the versatility and analytical capability for very small fibers or for fibers adjacent to other particulate matter. This microscope should also be equipped with the fluorescent viewing screen inscribed with graduations of known radii to estimate the lengths and widths of fibrous particulates.
- A specimen holder with tilt-rotation or double-tilt capability to obtain diffraction patterns at different zone-axis orientations.
- Darkroom facilities for developing negatives, making enlarged prints of patterns, and facilitating measurement of distances, spots, lines, and circles.
- A vacuum evaporator with a turntable for rotating specimens during coating, for such uses as carbon-coating polycarbonate filters, gold-coating EM grids, and preparing carbon-coated EM grids.
- An EM preparation room adjacent to the room housing the EM. This room should either be a clean-room facility, or contain a laminar-flow class-100 clean bench to minimize contamination during EM grid preparation. Filter handling and transfer to EM grids should be performed in a clean atmosphere. Laboratory blanks should be prepared and analyzed weekly to ensure quality of the work. In addition, a sample preparation room with a laminar-flow class-100 clean bench should be available for handling bulk-air

samples, ashing procedures, sedimentation, ultrasonification, filtration, and other prefilter activities.

- Several modifed Jaffe wick washers for dissolving membrane filters.
- Miscellaneous supplies and chemicals, such as membrane filters, EM grids, films, gold wire, chloroform, and carbon rods.
- Sample collection equipment, such as filter holders, sampling pumps, critical orifices, and tripods.

DESCRIPTION OF METHODOLOGY

A detailed discusson of the morphology, crystallography and chemistry of asbestos minerals, electron microscopy, and SAED analysis is outside the scope of the present protocol. Basic knowledge in these areas and an adequate level of comprehensive knowledge of TEM and SAED are prerequisites for the microscopists participating in asbestos analysis, especially at Level III stage.

Since Level III analysis is an extension of Level II analysis, common methodological details dealing with type of samples (source), sample collection and transport, sample preparation, TEM examination and data collection, data reduction and reporting of results, and quality control/quality assurance (QC/QA) program, which were discussed in detail in Section 5 (Level II Asbestos Analysis) will not be repeated here and the users are advised to refer to Section 5 for details in these areas. Differences, if any, between Level II and Level III protocols in common areas have been dealt with earlier under "Guidelines" and "Summary of Protocol."

The following provides brief descriptions of some of the essential areas of the Level III protocol that were not covered under Level II protocol.

1. Crystallography and Morphological Properties

Both crystallographic and morphological characteristics of asbestos minerals can help considerably in asbestos indentification and analysis. Chrysotile displays a unique narrow tubular morphology. The amphibole asbestos minerals have very similar morphologies—they are elongated along the z-axis (the chain direction) and generally lie with (100) planes approximately perpendicular to the electron beam. All varieties of amphiboles exhibit these Wadsley faults parallel to the length of the fiber.

Chrysotile possesses a cylindrical lattice which produces a unique SAED pattern. All the amphiboles, except anthophyllite, which is orthorhombic, have a monoclinic crystal structure. The amphiboles are double-chain silicates in which the fiber axis, z, has a repeat of 0.53 nm (inter-row spacing 'R' in real space, Figure A18). Since the other lattice parameters are also very similar, detailed zone-axis SAED analysis in more than one orientation is needed for positive identification. The non-asbestos forms of amphiboles have properties very similar to their asbestos counterparts, thus they must be distinquished from asbestos on the basis of morphology alone.

2. Chemical Properties—Elemental Analysis by EDS

Amphiboles are nonstochiometric minerals and often contain substitutional cations in varying amounts. Therefore, precise determination of their chemistry is difficult and positive identification based on chemistry alone is not reliable. This may be particularly pertinent when dealing with asbestos minerals present as minor constituents in mineral samples.

Elemental ratios, which are sometimes used to distinquish between asbestos types, often vary over wide ranges even in standard samples. The presence of gold coating, which would tend to preferentially absorb x-rays from lighter elements more than heavier elements, may make the situation even worse. In view of these ambiguities, and due to inherent practical difficulties in obtaining representative quantitative EDS elemental analyses from submicroscopic fibers, the present Level II and Level III protocols specify the use of only qualitative EDS spectra, which are often very valuable for screening purposes in the identification procedure. For example, in distinguishing between tremolite and actinolite type of amphibole, actinolite usually contains Fe, but tremolite does not.

3. Selected Area Electron Diffraction (SAED)

The method of obtaining an SAED pattern of a randomly oriented specimen is usually described in the EM instruction manual. The general directions for using the instrument to obtain and photograph SAED patterns are:

(1) Select the image magnification for the selected area.

(2) Bring the desired field of view to the center of the screen.

(3) Insert the appropriate field-limiting aperture (according to the desired field of view) into the beam path.

(4) Obtain the sharpest field-limiting aperture shadow.

(5) Confirm that the desired field of view is in the field-limiting aperture.

(6) Focus the specimen image; a photograph of the selected area image can be taken.

(7) Obtain the SAED pattern, remembering to retract the objective lens aperture from the beam path. The SAED pattern will be observed on the fluorescent screen.

(8) Select the desired camera length (the shorter the length, the better for SAED patterns of asbestos taken at high magnification).

(9) Focus the SAED pattern sharply. The beam stopper is used to intercept the bright center spot.

(10) For photography, the illumination is expanded (condenser reduced) after focusing the pattern, so that the pattern becomes barely visible (indistinct). A manual time exposure of approximately 20 to 30 s (maybe more depending on such factors as specimen and film) is required. The beam stopper can be left in place or removed from the beam path 1 to 2 s before closing the shutter. A double exposure of the specimen image and the SAED pattern can be taken if particle-to-particle spacing is adequate.

4. Use of Tilting to Acquire Exact Zone-Axis SAED Patterns

Determination of the Tilt Axis—

In the side-entry type electron microscopes, the instrument tilt axis is always fixed. However, the position of the tilt axis on the viewing screen shifts with magnification. Also, there is always an angular rotation between the image and the SAED pattern. It is highly desirable to know the location of the tilt axis on the viewing screen and its relationship vis-a-vis SAED pattern under the operating conditions to make effective use of specimen tilting for obtaining exact zone-axis orientations. The following steps can be used to locate the position of the tilt axis:

(1) A gold-coated EM grid with a standard asbestos mineral specimen on a polycarbonate replica film is placed in a tilt-rotation or double-tilt holder and inserted at 0° tilt into an aligned TEM set at 100 kV, 100 μA, 20,000X magnification, and 20-μm camera length operation.

(2) The image is focused on the fluorescent screen, which is at approximately 16,000X magnification.

(3) A circular hole in the polycarbonate replica is positioned in the center of the field of view.

(4) On tilting, the circular feature changes to an ellipse with the major axis unchanged, and indicates the position (direction) of tilt axis at that magnification. The minor axis shows the perpendicular direction to the tilt axis. A high tilt angle defines the tilt axis more accurately than a small tilt angle. Figure A15 illustrates the effect of tilt.

(5) A double-exposure photograph at 0° tilt and at some high tilt angle, such as 30°, is taken of the focused circular hole for reference.

Tilting—for zone-axis SAED Patterns—

Quantitative SAED requires knowledge of crystallography to obtain useful zone axis diffraction patterns from which precise measurements can be made for comparison with known asbestos standards on file. Thus the method of obtaining the visual SAED pattern of randomly oriented specimens, as in Level I and II analysis, is modified for quantitative SAED pattern analysis. It requires tilting of the specimen to align major crystallographic directions with the

electron beam. The zone axis is a line parallel to a set of intersecting crystal planes and nearly parallel to the electron beam. A zone-axis pattern thus gives regular repeat distances and even intensities of spots throughout the pattern.

Either a double-tilt or a tilt-rotation type specimen holder can be used for obtaining zone-axis patterns. A double-tilt holder is often preferred because tilt-rotation combination involves translational movement of the fiber during tilting, necessitating constant adjustment of the specimen-positioning controls to keep the specimen centered in the SAED aperture. On the other hand, it is much easier to obtain an accurate measure of the degree of tilt and perform systematic tilting with the tilt-rotation specimen holder. It is only necessary to rotate the specimen (fiber) until the tilt axis (as determined earlier) coincides with a major row of spots and then tilt until a major zone axis is parallel to the incident electron beam. Alternately, fiber axis of the fiber can be oriented either parallel or perpendicular to the tilt axis and then further tilting is used to obtain exact zone-axis orientations.

In order to avoid flip-flopping between image and diffraction modes while tilting, a recommended procedure is to defocus the diffraction pattern (the aperature becomes visible and the specimen/fiber can be seen in it) so that a double image of fiber in aperture can be seen with a poorly focused diffraction pattern. The movement of the fiber can then be tracked in relation to the spot pattern during tilting and kept centered in the SAED aperture by use of the specimen-positioning controls (knobs) of the microscope. Sometimes a larger aperture aids in the tracking-pattern recognition process.

An experienced electron microscopist can readily recognize the geometrical features like Kikuchi lines or Laue zones in the SAED pattern and use these to obtain the exact zone-axis SAED patterns. A detailed discussion of Kikuchi patterns and Laue zones and their utility in tilting experiments may be found in any standard text book on electron microscopy. Use of the double-tilt specimen holder is very helpful and less tedious in tilting experiments. However, all laboratories may not have both types of specimen holders available. A skilled microscopist can use either specimen holder without much difficulty. Experience and skill are more important factors in SAED analysis than the type of specimen holder used.

5. Characteristics of SAED Patterns Encountered in Asbestos Analysis

Successful application and exploitation of SAED analysis in asbestos analysis needs prior knowledge of the general appearance and distinguishing characteristics of other SAED patterns which are often encountered. The following discussion summarizes some of the observed SAED features of asbestos and other related minerals. This discussion is by no means comprehensive and assumes that the reader is familiar with general crystallography and the nomenclature pertaining to varous aspects of SAED patterns.

Minnesotaite and Stilpnomelane—

These iron-rich non-asbestos layer minerals are often encountered in asbestos analysis of specimens from certain geographic locations. Particulates of these minerals lie near their basal (001) planes. Stilpnomelane and

minnesotaite both possess large superlattices and their commonly observed SAED patterns are easily distinguishable from amphibole patterns. The spacing (in reciprocal space) is about half (for minnesotaite) or less than that for most amphiboles. These minerals can be readily distinguished in Level I or Level II analyses if a gold coating (optional) is applied to the specimen grids. A visual inspection of the number of rows of spots inside the (111) gold ring is sufficient to distinguish minnesotaite and stilpnomelane from amphiboles.

Chrysotile—

Due to the cylindrical lattice of chrysotile the SAED pattern is unique. The SAED pattern observed is symmetrical about the cylinder axis, x, and the spacing of the rows of spots is proportional to 1/a, where a is 0.53 nm. The most distinguishing features of the pattern are the flared spots of the type (130) which occur in the firt layer line. The flaring is due to the cylindrical lattice. A typical EDS spectra shows the presence of only Mg and Si (Figure A11).

Amphiboles—Systematic Absences, Twinning, and Double Diffraction—

The most commonly observed row of diffraction shots found in SAED patterns in amphiboles is in the y* or b* direction, representing the shortest reciprocal spacing between the spots (18.4 Å in real space). There are many strong zone axis orientations containing the y* row of spots. The lattice of amosite, crocidolite, tremolite, and actinolite is c-centered, and for such a lattice the h + k odd spots are absent along the y* or b* row. In practice, however, weak spots may be present in forbidden positions due to the presence of thin multiple twinning on (100), which cause streaking parallel to a*. Often, reciprocal nets from both twins are present in the same SAED pattern. In a twinned crystal, the number of important diffraction nets containing b* is doubled, leading to the observation that the diffraction patterns appear insensitive to tilt.

In some cases SAED patterns can contain spots from both twin individuals which overlap. However, not all the spots present in the composite SAED patterns are generated by the overlapping nets; some spots may be present because of double diffraction where a diffracted beam from one twin becomes the transmitted beam when it enters the other twin.

The purpose of the above discussion is to point out that although many complications exist in the analysis of SAED patterns, these can be overcome; in a good goniometric tilting stage most amphiboles can be identified by SAED analysis.

Amosite—

The nearest reciprocal lattice section to the (100) direct lattice plane in amosite is (301)* and it is also the most commonly observed section. Due to the presence of the thin (100) twins, this section closely resembles (100)*.

Typical EDS spectra from amosite fibers (Figure A11) show mainly Si and Fe with smaller amounts of Mg and Mn. Mn is frequently observed as a substitutional cation in amosite.

Crocidolite—

Most of the commonly observed patterns are asymmetrical and cannot be indexed easily. However, they all show rows of spots separated by a reciprocal repeat (R) corresponding to the fiber axis (0.53 nm).

The main elements observed in typical EDS analysis are Mg, Si, Ca, and Fe. Na, which is usually present in crocidolite, may not be detected in gold-coated specimens because of absorption, or because of overlapping secondary peaks from the copper grid.

Tremolite-Actinolite—

Temolite and actinolite show a variety of SAED patterns which have very similar appearances. In actinolite some of the Mg is replaced by Fe, with the result that interplanar d-spacings of actinolite are slightly larger than tremolite. In both tremolite and actinolite, the main elemental constituents are Mg, Si, and Ca. Actinolite also contains some Fe.

Anthophyllite—

Even though anthophyllite has an orthorhombic crystal structure, its commonly observed patterns are similar to the monoclinic amphiboles. Anthophyllite fibers dehydrate more easily in an electron beam and are, therefore, more difficult to study.

EDS elemental analysis shows the main constituents to be Si and Mg with a small amount of Fe.

6. Determination of Camera Constant and SAED Pattern Analysis

As mentioned earlier, a thin film of gold is evaporated on the specimen EM grid to obtain zone-axis SAED patterns superimposed with a ring pattern from the polycrystalline gold film. Since d-spacings corresponding to identifiable gold rings are known, these can be used as an internal standard in measuring unknown d-spacings on an SAED pattern from a fiber. The precision of measurement is as good as the quality of the photograph (or negative) and usually the measurements should be in the order of 0.1-0.2 mm with an angular tolerance of 0.5-1.5 degrees. The measurements can be made by several methods: manually with a ruler, with a mechanical aid, or a densitometer, etc. The patterns can be read directly on the developed negative or on an enlarged non-glossy print.

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 SAED 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 interst in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis SAED patterns.

7. Determination of Camera Constant Using Gold Rings

An average camera constant using multiple gold rings can be determined as explained below. However, in practice, in most cases determination of the

average camera constant is not necessary and thicker gold films are not desirable. The camera constant, CC, is 1/2 the diameter, D, of the rings times the interplanar spacing, d, of the ring being measured and is expressed as:

$$CC(mm\text{-}Å) = \frac{D(mm)}{2} \times d(Å)$$

The value of d for each ring can be obtained from the JCPDS file.

(a) Measure the diameters (two perpendicular locations) of the gold rings in mm as precisely as possible (see Figure A16).

(b) Measure as many distinct rings as possible to minimize systematic errors.

(c) Example: if the measured values in mm are D_1, D_2, D_3, D_4, and D_5, these will represent, respectively, d-spacings of

$$\frac{4.079}{\sqrt{3}}, \frac{4.079}{2}, \frac{4.079}{\sqrt{8}}, \frac{4.079}{\sqrt{11}}, \text{ and } \frac{4.079}{\sqrt{12}} \text{ Å}$$

(d) The camera constants will be:

$$CC_1 = \frac{D_1}{2} \times \frac{4.079}{\sqrt{3}} = \frac{D_1}{2} \times 2.355$$

$$CC_2 = \frac{D_2}{2} \times \frac{4.079}{2} = \frac{D_2}{2} \times 2.04$$

$$CC_3 = \frac{D_3}{2} \times \frac{4.079}{\sqrt{8}} = \frac{D_3}{2} \times 1.442$$

$$CC_4 = \frac{D_4}{2} \times \frac{4.079}{\sqrt{11}} = \frac{D_4}{2} \times 1.23$$

$$CC_5 = \frac{D_5}{2} \times \frac{4.079}{\sqrt{12}} = \frac{D_5}{2} \times 1.178$$

(e) The camera constant for the SAED pattern is the average of CC_1, CC_2, CC_3, CC_4, and CC_5. Table 2 presents an example of camera-constant determination.

TABLE 2. DETERMINATION OF CAMERA CONSTANT (EXAMPLE)

Ring No.	D_i readings (mm)	Mean D_i (mm)	d-spacing, d_i (Å)	Camera constant $C_i = D_i/2 \times d_i$
1	23.0, 22.0	22.5	2.355	26.5
2	27.4, 27.6	27.5	2.04	28.0
3	37.8, 38.2	38.0	1.44	27.4
4	44.6, 45.4	45.0	1.23	27.7

Mean Value of Camera Constant $= \frac{\Sigma C_i}{n} = \frac{26.5 + 28.0 + 27.4 + 27.7}{4} = 27.4$ (mm-Å)

8. Measurement of d-Spacings and Interplanar Angles

The gold film, because of its small, randomly oriented crystallites, produces a ring pattern superimposed on the SAED pattern from the fibers. The diameters of the gold rings correspond to known values of d-spacings, and this provides an internal standard to correct for inherent uncertainties present due to variations in instrumental and/or operating conditions. Since the d-spacings of interest on SAED patterns are usually the ones that lie closest to the center spot (transmitted beam), a camera constant measured from the first gold ring in the direction of measurement of d-spacings will usually give better accuracy in computed spacings than the use of an average camera constant. This method will account for any distortions in the symmetry of the gold ring pattern. The zone-axis SAED pattern usually has several rows of spots within the circular pattern of the gold rings. These rows of spots contain information about the two sets of planes in the crystal structure and the angle between them. The following procedure outlines the steps necessary to obtain the distances between planes (d-spacings) and the corresponding interplanar angle, θ (see Figure A17):

(1) From the spot pattern, determine the row with spots most closely spaced, and designate this as a horizontal row. Draw a fine line to show the row through the origin, and designate this the zeroeth row. Draw fine lines to show the first and succeeding horizontal rows. For a few horizontal rows, measure the mean spacing between adjacent spots (or the minimum vector):

$$X_i = \frac{\text{Distance between spots m units apart}}{m}$$

where m is chosen as an optimum number to minimize measurement errors. The mean horizontal spot distance, X,

equals the summation of X_1 divided by the number, n, of rows measured. The d-spacing in Å corresponding to this vector is the camera constant divided by X, and is labeled d_2. Table 3 presents an example of spot spacing measurement within a horizontal row.

(2) The perpendicular distance between two adjacent horizontal rows is similarly measured. This interrow spacing, Z, is the mean separation between horiziontal rows, and equals the distance between a number of rows divided by the number of spaces. This distance is an additional vector for comparison that coincides with the slant vector, d_1-spacing, when angle $\theta_{1,2}$ is 90°. The row-spacing (R) equals the camera constant divided by Z. Table 3 presents an example of perpendicular spacing between horizontal rows; Figure A17 illustrates spot and row spacing.

(3) To obtain the d_1-spacing and corresponding angle $\theta_{1,2}$, a perpendicular is drawn to the zeroeth horizontal row through the origin. A line is drawn to the first spot to the right of the perpendicular in the first row and extended through the succeeding rows. This line, called the slant vector, forms the acute angle $\theta_{1,2}$. The mean spacing, Y, between spots on the slant vector can be measured by dividing the maximum distance between spots by the number of spaces between them, or by calculating from the interrow spacing:

$$Y = \frac{R}{\sin \theta_{1,2}}$$

The d-spacing in Å corresponding to this vector is the camera constant, CC, divided by Y and labeled d_1.

$$d_1(Å) = \frac{CC \times \sin \theta_{1,2}}{R} = \frac{CC}{Y}$$

Figure A18 illustrates the relationship of d_1, d_2, $\theta_{1,2}$ and R. In some cases, the interplanar angle $\theta_{1,2}$ may be more than 90 degrees (not shown in Figure A18).

Summary of Data from Each SAED Pattern:

(a) The camera constant, CC, as determined from the gold rings, normalizes the distances on the SAED pattern regardless of such factors as magnification and tilting.

TABLE 3. DETERMINATION OF SPOT SPACINGS (EXAMPLES)

Reading	Separation (mm)	Units	Mean spacing, X_i (Å)
Spot spacing within a horizontal row, d_2:			
1	49	16	3.006
2	42.7	14	3.05
3	—	—	—
			3.028 = Mean

$$\text{d-spacing} = \frac{27.4}{3.028} = 9.05 \text{ Å}$$

Perpendicular spacing between horizontal rows, R:			
1	43	8	5.0375
—	—	—	—
—	—	—	—
			5.0375 = Mean

$$\text{d-spacing, R} = \frac{27.4}{5.0375} = 5.44 \text{ Å}$$

Note: It is preferable that the camera constant values used in computing d-spacings are measured from the first one or two gold ring diameters in the direction of d-spacing measurement.

(b) The parameters of interest are:

- d-spacing of spots in a horiziontal row: $CC/X = d_2$
- d-spacing of spots in the slant vector: $CC/Y = d_1$
- angle $\theta_{1,2}$ formed between a horizontal row and slant vector
- d-spacing corresponding to row separation as an additional parameter of interest: $CC/Z = R$.

It should be noted that the use of camera constant in the form used here in calculating d_1, d_2, and R, which are measured in reciprocal space on SAED patterns, automatically converts the calculated numbers into real space spacings, which are then compared to those from a suitable standard file.

9. Identification of Unknown Fibers

Unknown d-spacings (d_1 and d_2), interrow spacing (R), and interplanar angles (θ) measured from zone-axis SAED patterns of unknown fibers are compared with corresponding known values tabulated in JCPDS powder diffraction files, or those computed using lattice parameters and crystal structures of candidate asbestos minerals, or with the values contained in an internally developed file from standard specimens of candidate minerals. Table 4 is an example of the IITRI standards file (Jones et al., 1981). Figures A19 to A22 are examples of zone-axis SAED patterns.

Unknowns are matched as closely as possible to the file parameters for positive identification. However, considerable care and competent judgment are required in Level III confirmatory analysis. For example, amphiboles are usually nonstochiometric minerals, and thus a perfect match may not be possible between the d-spacings and interplanar angles determined from unknown fibers and those available from standard minerals. JCPDS Powder Diffraction files do not list interplanar angles. Since amphiboles have low-symmetry crystal structures, tabulated values of d-spacings and interplanar angles would be extensive and very expensive to generate, and to get an accurate match may not be possible because these tables are derived assuming certain lattice parameters which may not be the same as those of the unknown fibers being analyzed. Given these inherent uncertainties, it would seem that use of internally developed SAED files consisting of several readily accessible orientations (by virtue of natural habit of amphibole fibers) from standard amphibole species could eliminate a lot of tedious unnecessary work and yet provide reliable data for comparison and identification of unknown fibers.

In practice, SAED analysis combined with qualitative EDS analysis may help resolve certain cases where a close match in d-spacings and interplanar angles is not possible. For difficult specimens or SAED patterns of controversial nature, a second opinion may be necessary, especially if a legal case is involved.

TABLE 4. COMPARISION OF d-SPACINGS FROM SAED FILE AND POWDER DIFFRACTION FILE (EXAMPLE)

		Internal Standard File Data				Powder Diffraction File Data (1975)		
Amphibole type	Zone axis	d_1 (Å)	d_2 (Å)	θ (deg)	Interrow spacing, R (Å)	d_1 (Å)	d_2 (Å)	File index no.
Amosite	[100]	5.3	9.14	90.0	5.3	5.22	9.20	17-725
	[30$\bar{1}$]	1.79	9.26	84.0	—	1.76	9.20	17-725
	[101]	4.88	9.23	74.0	5.17	4.84	9.20	17-725
	[$\bar{1}$01]	4.14	9.11	78.0	4.21	4.10	9.20	17-725
	[$\bar{3}$10]	5.22	5.13	95.0	—	5.22	5.12	17-725
Crocidolite	[100]	5.22	8.97	90.0	5.22	5.20	9.02	19-1061
	[101]	4.94	9.05	75.0	5.19	5.89	9.02	19-1061
	[$\bar{1}$10]	4.79	8.19	79.0	5.23	4.89	8.40	19-1061
	[30$\bar{1}$]	1.75	8.97	83.5	—	1.76	9.02	19-1061
	[$\bar{3}$10]	5.12	5.12	96.0	—	—	—	19-1061
Tremolite	[100]	5.04	9.03	90.0	—	5.07	8.98	13-437
	[101]	4.83	9.03	75.0	—	4.87	8.98	13-437
	[$\bar{2}$0$\bar{1}$]	2.59	8.97	80.5	—	2.59	8.98	13-437
	[30$\bar{1}$]	1.72	8.98	83.5	—	1.69	8.98	13-437
Anthophyllite	[100]	—	—	90.0	5.24	5.28	8.90	9-455
	[$\bar{1}$42]	4.56	4.56	60.0	—	4.50	4.50	9-455

SECTION 7

ARCHIVAL SAMPLES

DISCUSSION OF PROTOCOL

Samples that have been collected on filter substrates other than polycarbonate, or that have been collected without regard to filter loading levels, are referred to as archival samples. These samples were usually collected for other analytical objectives, such as for optical microscopy or gravimetric analysis, for defined sampling periods without regard to concentration levels in the air, or for collection of particles larger than 10 µm in diameter. Such samples were historically collected, and are of value and interest in determining the presence of asbestos fibers and/or structures. Filter substrates designated as archival samples include glass fiber filters; cellulose or modified paper filters; cellulose ester filters; other organic polymeric membranes, such as polystyrene, nylon, and polyvinyl chloride; and all overloaded organic polymeric membrane filters.

The purpose of the preparation step is to transfer particles from a filter surface to an EM grid with a minimum of distortion in morphology and size distribution. The nature of non-polycarbonate filter substrates or particle loading makes it sometimes necessary to transfer a satisfactory quantity of particles to a polycarbonate filter prior to transfer to the EM grid. At present, only transfer to an EM grid from a polycarbonate filter has been standardized.

A modified preparation technique is recommended for archival samples, followed by the analytical methodology using Level I, Level II, or Level III effort—with the understanding that these samples will indicate the presence of asbestos, and secondarily the number, size, distribution, and morphology. The results from sample to sample are less precise due to problems in standardizing the preparation procedures used for archival samples.

The archival filter samples are prepared for analysis based on the information sought, type of filter material, and particle loading on the filter. The various preparation techniques for these filters include:

(1) Individual particle picking and/or reverse washing of the filter, with subsequent filtration of the filtrate using a polycarbonate filter.

(2) Collapsing the membrane filter structure by exposure to solvent vapor (surface fusion), to produce a more uniform substrate for replication and grid transfer.

(3) Solubilizing filter material in selected solvents, followed by separation of particulates.

(4) Low temperature ashing (LTA).

Two preparation methods are recommended based on filter loading and type of filter material: surface fusion, and LTA.

DESCRIPTION OF METHODOLOGY

Because the greatest number of archival samples have cellulose ester substrates, this type of filter material is used in examples describing the methodology.

1. Samples with Adequate Loading

Discussion—

As an example, samples collected on cellulose ester filters have been received by a laboratory. Direct transfer of the particulates to the EM grid is possible using acetone as the solvent in a modified Jaffe wick washer. However, a question arises concerning indeterminate particle loss in the transfer. Carbon-coating the cellulose ester filter prior to grid transfer minimizes particle loss. However, this improvement in particle count is offset by difficulty in visually observing and counting the fibrous particles against a replica background of the uneven surface topography of the cellulose ester filter, and by indeterminate loss of very small particles hidden in the crevices of the uneven filter surface. The NIOSH method of surface fusion (Zumwalde and Dement, 1977) is relatively reliable, and produces a more consistent result, although the question of loss of the very small particles has not been resolved. LTA, described later in this section, may also be used for these samples.

Procedure—

The NIOSH technique, a modification of a particle-transfer technique developed at Los Alamos Scientific Laboratory (Ortiz and Isom, 1974), is described as follows:

(1) A section of the membrane filter is cut with a scalpel, and placed on a clean microscope slide with the sampled side facing up.

(2) The cut section is fastened on all sides to the slide with narrow strips of transparent tape.

(3) The slide, with the cut section, is exposed to acetone vapor (not liquid) for approximately 10 min. The acetone vapor collapses the structure of the filter and produces a fused, relatively smooth-surfaced film. The size of the acetone vapor bath and time of filter response to the vapors are critical in obtaining the desired smooth, fused surface; each laboratory must determine its own optimum conditions.

(4) The fused filter section is placed on the rotating stage of the vacuum evaporator for carbon-coating.

(5) A 3-mm-diameter portion of the carbon-coated filter is transferred to a carbon-coated EM grid in the modified Jaffe wick washer.

(6) Acetone is used in dissolving the fused membrane filter.

(7) Transfer to the grid and options for analytical efforts were described previously.

2. Samples with Heavy Loading

Discussion—

As an example, samples collected with a heavy deposit of particulates have been received by a laboratory. These particulates may be organic in nature (for example, pollen or soot), or of mineral matter. LTA is used to remove the organic material (filter as well as particulates), leaving the inorganic residue. The residue is gently resuspended and dispersed in filtered distilled water by low-wattage, short-time ultrasonification. The resuspension is then filtered onto a 0.1-μm (pore size) polycarbonate filter. The dry, particulate-loaded polycarbonate filter is then carbon-coated and transferred to EM grids for analysis as described previously.

Low temperature ashers are available with one, two, or four chambers. The following modifications minimize contamination in using these units:

(1) A single chamber is dedicated for ashing samples for EM analysis.

(2) An in-line filter is placed in the oxygen supply between the regulator and entry into the asher.

(3) For models with direct access to ambient laboratory air on completion of ashing and return to ambient pressure, a filter is placed in the inlet line to prevent laboratory air from being sucked into the chamber.

In using the single-chamber method, a blank test tube and the sample tubes (up to four, for a total of five in a 10-cm-diameter chamber) are placed in the chamber lengthwise, with the opening facing the door.

The filtration step is also used in diluting the initial heavy particulate loading. Filtration of aliquots is not recommended to obtain different levels of loading on the new filters; a representative sample from each aliquot in the filtration of suspensions is difficult to obtain. Instead, for heavy loadings, different known areas of filter segments (one-eighth, one-fourth, or one-half of the filter) should be ashed so that the entire contents of the resuspension tube can be filtered onto either a 25-mm-, 37-mm-, or 47-mm-diameter polycarbonate filter for the desired dilution.

Distilled water is filtered through a 0.1-μm (pore size) polycarbonate filter prior to use. All glassware is washed with soap and water, rinsed with acid, and then rinsed with particle-free distilled water. The dedicated asher chamber is carefully wiped with damp lens paper.

LTA Procedure—

The LTA manufacturer's instructions are followed since the power required for one, two, or four chambers, the mass (glassware plus sample) placed in the chamber, and the desired rate of ashing all vary. In general, the following steps are performed:

(1) Each filter segment with a known deposit area is carefully placed in a clean test tube (13 mm x 80 mm) using a clean tweezer.

(2) With forceps, the tubes containing the sample, and one lab blank (unused filter segment of the same size and type of filter material as the sample) are placed lengthwise, side by side in the chamber, with the mouths of the tubes facing the open end (door) of the asher chamber. The tubes are laid in the center of the chamber within the region of the coils surrounding the chamber. Up to four sample tubes and one blank can be laid like logs inside the chamber.

(3) The power is slowly and carefully increased to prevent "flashing" of the filter, which would result in loss of sample.

(4) The filter membrane vanishes in about 30 min; ashing is continued for another 2 to 3 h to ensure complete ashing. The chamber is slowly allowed to reach ambient pressure.

(5) The test tubes are carefully removed and placed in a beaker, covered, and stored on a class-100 clean bench for resuspension.

Resuspension (Sonification) Procedure—

Ultrasonification is used in resuspending and redispersing the ash resulting from the LTA. The superiority of a probe-type ultrasonic device over a bath-type device has not been demonstrated. However, the criteria of low energy and minimum sonification time appear valid. The probe-type instrument is more readily calibrated (desired reproducibility), but requires a larger volume of suspension to work with. The bath-type instrument is more difficult to calibrate, and is usually of fixed wattage. A generalization is that probes are used for dispersing, baths for cleaning. Recently, a bath-type ultrasonic unit (Ladd Research Industries, Inc., Burlington, Vermont) with a variable power source and timer has become available that appears to have the advantages of both types of ultrasonic devices.

The resuspension procedure is as follows:

(1) 10 mL of filtered distilled water is added to each test tube.

(2) Each tube is placed in a 100-mL beaker containing 50 mL of water.

(3) The beaker, with the tube, is placed in the low-energy ultrasonic bath.

(4) Ultrasonic energy of about 50 to 60 W (60%) is applied for 3 min.

Filtration Procedure—

Liquid filtration of suspensions for EM examination using polycarbonate filters is one of the more difficult procedures to standardize. Variations in the nature of the filter material, geometry and distribution of pores, and method of manufacturing make it difficult to obtain a uniform deposit of particulates on the filter. The following procedure is used for consistency in the filtration procedure:

(1) A filtering apparatus having a filter size adequate for the desired dilution--preferably 25-mm or 47-mm diameter--is assembled. A polycarbonate filter (0.1-μm pore size) is used shiny side up for the deposit, with a cellulose ester filter (5-μm pore size) as a backing filter on the glass frit.

(2) While dry, the filters are centered and suction is applied. The filter funnel is mounted on the centered, perfectly flat filters with the vacuum on.

(3) The vacuum is then turned off. A 2 mL amount of particle-free distilled water is added to the filter funnel, followed by careful addition of all the water in the test tube containing the dispersed ash. The test tube is rinsed twice with particle-free distilled water, and the contents are carefully added to the filter funnel.

(4) Suction is then applied; neither rinsing the filter funnel nor adding extra liquid is permitted during the entire filtration process.

(5) At the end of filtration, suction is stopped.

(6) If possible, the filter is dried on a glass slide or holder that can be placed directly in the vacuum evaporator for carbon-coating.

(7) The dry filter is stored in a disposable Petri dish (taped on a glass slide), or in the special holder, until ready for carbon-coating, grid transfer, and EM analysis.

(8) The effective area of the redispersion filter and the area of original filter deposit cut for ashing must be recorded (ashing factor) for inclusion in analytical data reduction and reporting.

SECTION 8

BULK-SAMPLE ANALYSIS

DISCUSSION OF PROTOCOL

Bulk samples may be air samples collected in large volumes using electrostatic precipitators, bag collectors, or high-volume samplers, for example; or they may be original pieces of source material containing asbestos, such as insulation, asbestos paper products, and asbestos cement products.

Efficient usage of the three levels of analysis requires effective communication between those requesting an analysis and those responsible for conducting the analysis. Personnel requesting an analysis must understand the limitations of each level of analysis by EM. For example, requesting EM analysis of a bulk-material (solid) sample, where there is marked disagreement regarding the presence of asbestos (amphibole), and using Level I (screening) analysis, are incompatible. Bulk-material samples require grinding for analysis; grinding requires care to minimize such problems as contamination, change in size of the asbestos fiber, increase in fragments that morphologically meet the criteria of a fiber, possible change in the relationship of asbestos to nonasbestos components, and possible destruction of asbestos fiber crystallinity.

Following grinding, bulk-material samples should first be analyzed by PLM, followed by XRD, if necessary. XRD provides information on samples having asbestos concentration levels of at least 2%. PLM provides information on asbestos and nonasbestos components, as well as on the size of the asbestos fibers in the solid-bulk phase. The additional information aids in EM analysis of these samples at the selected level of analysis.

DESCRIPTION OF METHODOLOGY

1. Polarized Light Microscopy

Analysis of bulk samples, such as insulation material, for component identification and for determination of the type and concentration of asbestos present is best accomplished by PLM. With the polarized light microscope, particle properties--such as color, morphology, refractive index, birefringence (which indicates a crystalline substance rather than an amorphous substance), surface texture, reflectivity, and magnetism--can be observed and determined. Determination of such a large number of particle properties allows identification of specific particle types, in most cases. For example, amorphous slags and crystalline minerals are common nonfibrous filler components of insulation materials that can be easily distinguished by PLM.

2. X-Ray Diffraction Analysis

XRD has been successfully used to measure asbestos content in both aerosol samples and bulk samples. The reported values for sensitivity and accuracy vary depending on the exact technique, but recent reports quote sensitivity values of about 1% chrysotile, whereas 5% was more common when the technique was first demonstrated. Much of the improved sensitivity derives from sample preparation techniques, which are important in XRD, but digital data collection and use of such accessories as x-ray monochromators to reduce background are also important.

While 1% sensitivity has not been demonstrated for asbestos materials other than chrysotile, the same sample preparation procedures are applicable to other mineral forms, with comparable sensitivities expected in cases with serious interfering lines. Interferences would hinder the analysis for chrysotile as well as for other asbestos minerals. Considering that the high-sensitivity procedures have been only partially demonstrated at the 1% level, a sensitivity of, say, 2% is probably a more realistic expectation for asbestos minerals in general.

3. Electron Microscopy

For bulk-air samples, asbestos analysis by EM entails an addition to the sample preparation procedure to attain a representative powder sample at a suitable concentration level to be placed on the EM grid. This additional step is similar to the method used in preparing standards of known asbestos. The finely divided powder samples are split into representative fractions, and a small, weighed portion is suspended in a known volume of filtered distilled water containing 0.1% Aerosol OT.* A mild ultrasonic treatment is used to disperse the particles. Different known volumes of suspension are filtered through a 0.1-μm (pore size), 25-mm-diameter Nuclepore† membrane filter. The dried Nuclepore filter is then carbon-coated and transferred to an EM grid using the refined Jaffe wick technique described previously.

Bulk-solid samples are gently and slowly ground to a powder for EM analysis to minimize localized heating; the powder is then prepared for the EM grid by the method described for bulk-air samples.

For EM analysis of bulk-air samples, a weighed portion is suspended in filtered distilled water, deagglomerated in an ultrasonic bath, transferred to a volumetric flask, and brought to volume with filtered distilled water. An aliquot is then filtered onto a 0.1-μm (pore size) polycarbonate filter using a 5.0-μm (pore size) cellulose ester filter as a back-up filter on the filtration apparatus. The dried polycarbonate filter is then carbon-coated. A 3-mm x 3-mm portion of the carbon-coated filter is then directly transferred to a 200-mesh carbon-coated copper EM grid using the refined Jaffe wick washer technique. EM analysis based on Level I, II, or III effort is then performed.

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* Fisher Scientific Co. (Cat. no. 50-A-292), 711 Forbes Ave., Pittsburgh, Pa.

† Nuclepore Corporation, 7035 Commerce Circle, Pleasanton, Calif.

SECTION 9

NUMERICAL RELATIONSHIPS AND ANALYTICAL AIDS

The fibrous structures (fibers, bundles, clusters, and matrices) in an air sample are to be counted, sized, and identified as asbestos or non-asbestos. An air sample ranging from 1 to 5 m^3, depending on its total suspended particulates (TSP) content (in ambient air, the average TSP is between 30 and 300 $\mu g/m^3$) is drawn through a 37-mm filter (effective filtration area of 8.6 cm^2) or a 47-mm filter (effective filtration area of 9.6 cm^2). The asbestos content, unlike a prepared laboratory standard or sample, is a very small percentage (less than 1%) of the particulate loading (TSP content) collected on the filter surface.

Two small circular sections of the filter of approximately 3-mm diameter are transferred to EM grids for transmission electron microscopy. Either one or both EM grids are examined for asbestos content; 10 random grid openings are examined for each EM grid. Each grid opening measures approximately 85 µm x 85 µm. At 20,000X magnification, a field of view of 4.5 µm x 5.0 µm is used in the examination. This approximates to about 300 fields per grid opening or 3000 fields of view per grid examined (6000 fields for two EM grids) if less than 100 asbestos structures had been found. Unlike a field blank or laboratory blank, the statistical significance of obtaining a low asbestos count in the midst of atmospheric clutter needs to be recognized.

LIMITS OF DETECTION

The minimum detection limit of the EM method for counting airborne asbestos fibers varies depending on the amount of total extraneous particulate matter in the sample, and on the contamination level in the laboratory environment. This limit also depends on the air sampling parameters, loading level, and EM parameters used. For example, assuming that a fiber count has an accuracy of ±1 fiber, when 10 full-grid openings are scanned, each grid opening having an average area of 0.72×10^{-4} cm^2, the detection limit is determined from the equation

$$\text{Detection limit} = \frac{1}{10} \times \frac{\text{Area of filter } (cm^2)}{0.72 \times 10^{-4}\ (cm^2)} \times \frac{1}{\text{Volume of air } (m^3)}$$

The minimum detection limit, then, is lower for very dilute samples. Examining full-grid openings leads to a lower value of the minimum detection limit because of the large area scanned, as compared with the field of view method. With a given sample, the detection limit can be lowered considerably, but the required experimental effort increases. The guideline of using 10

full-grid openings represents a judicious compromise between a reasonable experimental effort and a fairly low value of the detection limit. However, using two or more TEM grids reduces the detection limit further and improves the precision of the estimates.

STATISTICAL METHODOLOGY

Several statistical strategies have been used to characterize airborne asbestos distributions and estimate the abundance of asbestos fibers in a given sample. These methods range from simple tabulation of observed frequencies of fibers across grid opening samples to the fitting of statistical probability distribution such as the Poisson. This statistical section outlines a general methodology for fitting observed data to a statistical probability distribution (either Poisson or normal depending on the fit of the former). The mean and 95% confidence interval are then estimated and used for the purpose of sample description and drawing inference regarding the abundance of airborne asbestos fibers in the environment in which the samples were obtained.

As an illustration, consider the hypothetical data in Table 5.

The expected number of grid openings with no fibers is:

$$Ne^{-\hat{u}} = (98)\ (e^{-3.0204}) = 4.7806$$

The expected number of grid openings with 1, 2, 3, ... fibers are found by multiplying $Ne^{\hat{u}}$ by $\hat{u}/r$, i.e., 4.7806 x 3.0204, 4.7806 x 3.0204/2, 4.7806 x 3.0204/3, successively.

To test the fit of the Poisson distribution to the observed data we compute a chi-square statistic, $\chi^2 = \Sigma$ (observed - expected)^2/expected = 8.26.

Since there are nine different observed frequencies (i.e., numbers of fibers) there are 9 - 2 = 7 degrees of freedom (since we estimate one parameter). The probability of $\chi_a^2 = 8.26$ is p = 0.4; therefore, we conclude that the Poisson distribution fits the observed data.

In certain cases, the observed frequencies will not have a Poisson distribution (as determined by the previously described chi-square statistic). In this case we estimate the mean ($\bar{X}$), variance S^2, and 95% confidence limits

TABLE 5. HYPOTHETICAL DATA

No. of fibers on grid 1 opening (r)	Observed* frequency (f)	Expected frequency		Observed - expected	Probability (r)
0	3	4.78		-1.78	.049
1	17	14.44		+2.56	.149
2	26	21.81		+4.19	.224
3	16	21.96		-5.96	.224
4	18	16.58		+1.42	.168
5	9	10.03		-1.02	.101
6	3	5.04		-2.04	.050
7	5	2.18		+2.84	.022
8	0	0.82			
9	1†	0.27	1.20	-0.20	.003
10	0	0.08			
11 or more	0	0.03			
Total	98	98.0			

* The number of grid openings showing that number of fibers.

† We combine adjacent frequencies to get a minimum of 1 fiber per group. Assuming a Poisson distribution, the mean is $\hat{u} = \Sigma fr/\Sigma f = 296/98 = 3.0204$. That is, the sum of the product of the observed frequencies and number of fibers divided by the sum of the frequencies.

assuming normality. The 95% confidence limits are computed as a function of the sample variance and t distribution.

$$S^2 = \frac{n \sum_{i=1}^{k} X_i^2 - [\sum_{i=1}^{n} X_i]^2}{n(n-1)}$$

where k is the number of grid openings, n is the total number of fibers found, and X_i is the number of fibers found in grid opening i.

The 95% confidence limits are given by

$$\bar{X} \pm \frac{ts}{\sqrt{n}}$$

where t is the value of the two-tailed t distribution for probability $p < .025$ and n - 1 degrees of freedom.

1. 95% Confidence Limits for a Poisson Variate

To generate a level of confidence regarding our estimate of the number of asbestos fibers per grid opening, a 95% confidence limit can be derived. For counts of 0 through 20, Table 6 may be used.

For example, if out of 20 grid openings 15 fibers are found, the 95% confidence limits are obtained by taking the lower and upper bounds from Table 6 as 8.40 and 24.74. Then per grid opening, the 95% confidence limit is 8.40/20 to 24.74/20, or 0.42 to 1.237 fibers per grid opening.

For counts greater than 20, a simple normal approximation is computationally convenient. The normal approximation is $\lambda = L \pm \chi^* (S_L)$ where L is the observed count, S_L is $\sqrt{L}$, and χ^* is 1.96 or 2.58 for the 95% or 99% confidence limits, respectively.

For example, if 35 fibers were observed from inspection of 20 grid openings, $L = 35$, $S_L = \sqrt{35} = 5.91$, $\lambda = 35 \pm 1.96\ (5.91) = 23.4$ to 46.6 or 23.4/20 = 1.38 to 46.6/20 = 2.74 fibers per grid opening.

2. Comparison of Two Poisson Variates

In certain cases, a new test sample is compared to a "blank" or "control" sample. Table 7 gives those differences between control and test samples that are significant at the 5% level.

Inspection of Table 7 reveals that the minimal detectable difference between test and control samples is 5 fibers in the test sample and 0 fibers in the blanks. Typically, inspection of 20 grid openings for a blank control reveals between 0 and 5 fibers. At the upper bound (i.e., 5 fibers in a

TABLE 6. 95 PERCENT CONFIDENCE LIMITS

No. of Fibers	95% Limits Lower	Upper
0	0.000	3.69
1	0.0253	5.57
2	0.242	7.22
3	0.619	8.77
4	1.09	10.24
5	1.62	11.67
6	2.20	13.06
7	2.81	14.42
8	3.45	15.76
9	4.12	17.08
10	4.80	18.39
11	5.49	19.68
12	6.20	20.96
13	6.92	22.23
14	7.65	23.49
15	8.40	24.74
16	9.15	25.98
17	9.90	27.22
18	10.67	28.45
19	11.44	29.67
20	12.22	30.89

TABLE 7. CONTROL AND TEST SAMPLE DIFFERENCES

Fiber Count		Fiber Count	
Control	Test sample	Control	Test sample
	1	6	21
	2	6	22
	3	7	23
	4	7	24
0	5	7	25
0	6	8	26
0	7	8	27
1	8	9	28
1	9	9	29
1	10	10	30
2	11	10	31
2	12	10	32
3	13	11	33
3	14	11	34
3	15	12	35
4	16	12	36
4	17	13	37
5	18	13	38
5	19	13	39
5	20	14	40

control sample), 18 fibers in the test sample are required for statistical significance. This estimate of 18 fibers may prove useful for establishing an asbestos detection limit criterion.

MAGNIFICATION CALIBRATION

The following steps should be performed to calibrate the magnification of the EM:

(1) Align the EM using the manufacturer's instructions.

(2) Insert mag-calibration grating replica* (with 54,864 lines per inch, or 2160 lines per mm) in the specimen holder.

(3) Switch on the beam, obtain the image of the replica grating at 20,000X magnification (or at the magnification at which the asbestos samples will be analyzed), and focus.

(4) If the fluorescent screen has scribed circles of known diameters, align one line tangentially to the circumference of one circle using stage control. Count the number of lines in a diameter perpendicular to the lines. In most cases, the other end of the diameter will be between the nth and $n^{th} + 1$ line. The fractional spacing can be estimated by eye. Alternatively, the separation between lines can be estimated using the scribed circles.

(5) If X line spacings span Y mm on the fluorescent screen using this grating replica, the true magnification, M, is given by

$$M = \frac{Y \times 2160}{X}$$

The readings should be repeated at different locations on the replica, and the average of about six readings should be taken as the representative or true magnification for that setting of the EM, as in the following example:

* For example, Cat. no. 1002, E. F. Fullam Co., Schenectady, N.Y.

Line Spacings, X	mm on Screen, Y	Magnification, M
9.5	83	18871
9.3	80	18580
7.0	60	18514
8.8	80	19636
9.0	80	19200
9.0	80	19200
	Average:	19000

On most EM's with large (18-cm diameter) fluorescent screens, the magnification is substantially constant only with the central 8- to 10-cm-diameter region. Therefore, calibration measurements should be made within this small region and not over the entire screen.

PREPARATION OF BLANKS

Even after taking the utmost precautions to avoid asbestos contamination, the possibility of some contamination cannot be ruled out. Contamination should be checked periodically by running field blank samples in addition to laboratory blanks. Field blanks should be analyzed prior to laboratory blanks. A blank sample may consist of a clean filter subjected to all the processing conducted for an actual air sample. This processing may include ashing, resuspension, redeposition, carbon-coating, transfer to a TEM grid, and TEM examination.

When analyses of blank samples show significant background levels of asbestos, these should be subtracted from the values obtained for field samples. Also, the minimum detection limit may be calculated as twice or three times the standard deviation of the blank or background value.

USE OF COMPUTERS

Data reduction is facilitated by computers. Computer printouts can be used in reports. Each laboratory should develop software suitable for its needs as well as to maintain basic information, such as fiber, areas examined, volume/mass of sample, and size distribution, for possible interlaboratory comparison.

Appendixes B and C present sample printouts from Level I and Level II analyses, respectively.

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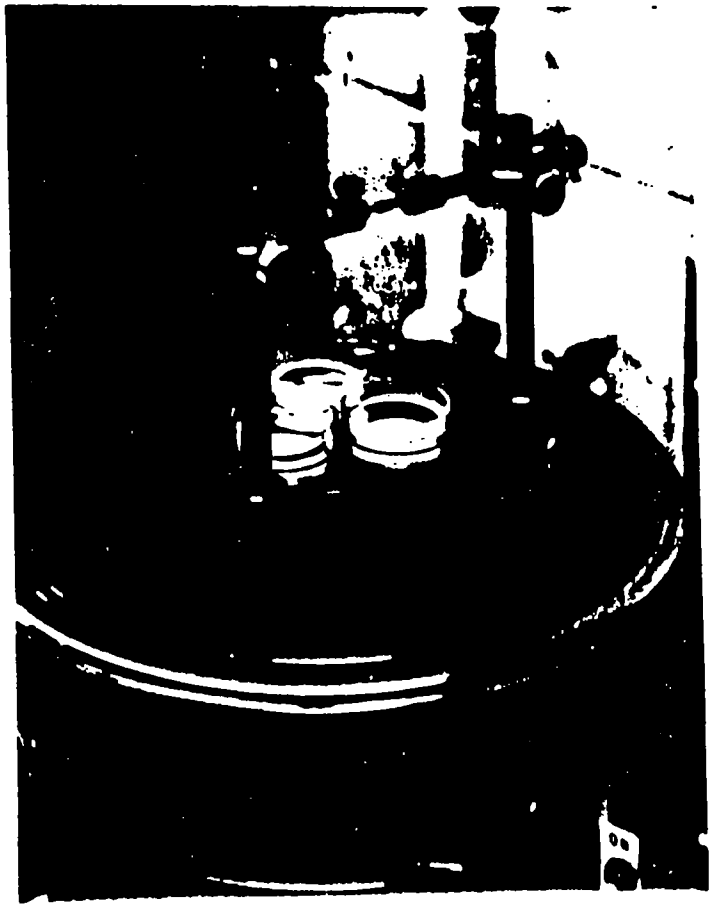
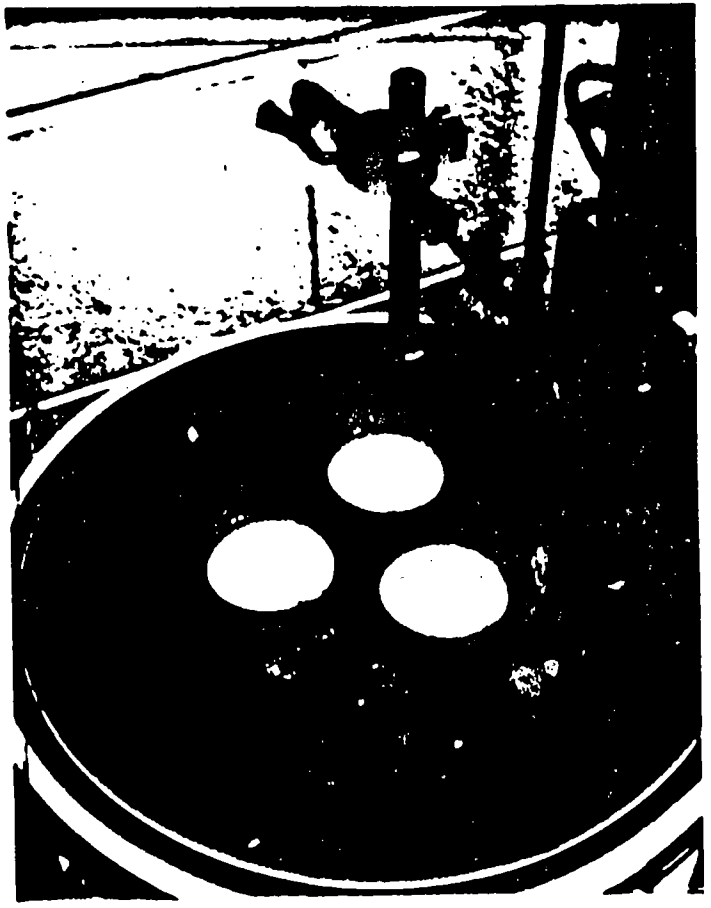
APPENDIX A

FIGURES

Figure A1. Vacuum evaporator.

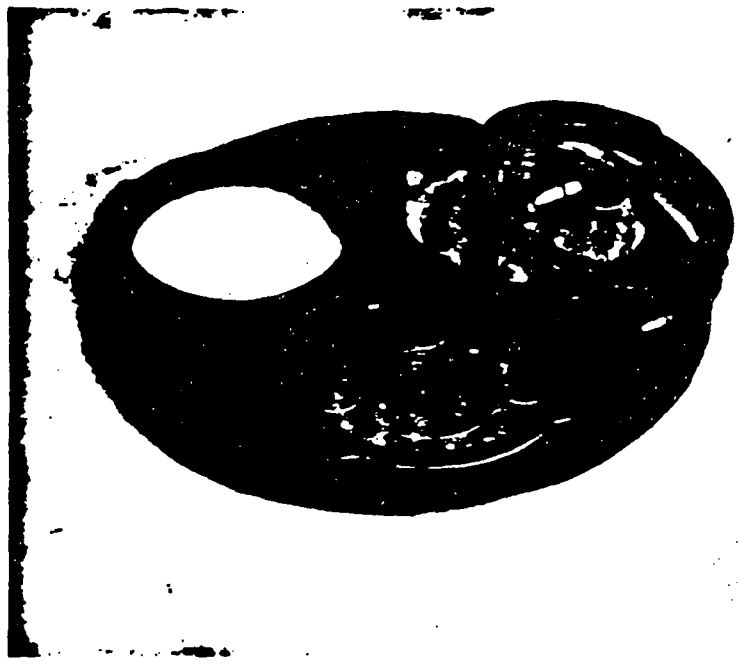


(a) 37-mm diameter.



(b) 47-mm diameter.

Figure A2. Multiple coating arrengement in evaporator.



(a) Modified 47-mm-diameter Petri-slides.

(b) 37-mm-diameter cassette.

Figure A3. Close-up of multiple coating arrangement.

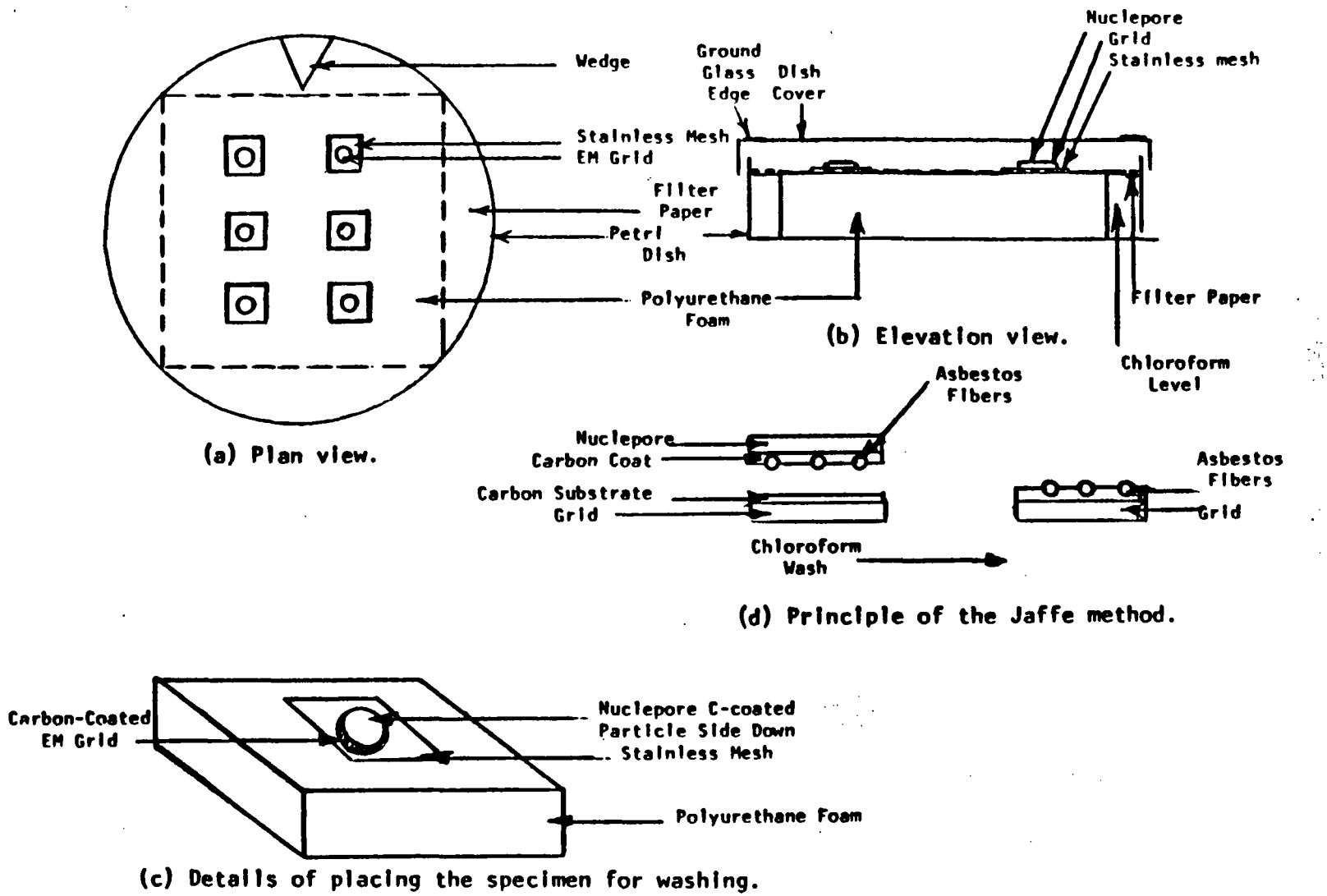


Figure A4. Modified Jaffe wick washer method (sketch).

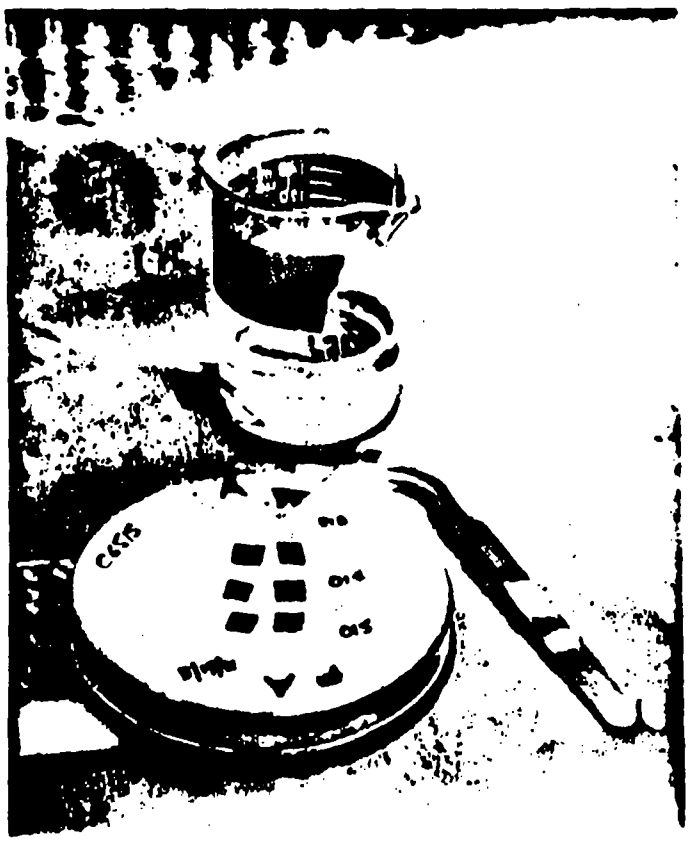


Figure A5. Modified Jaffe wick washer.

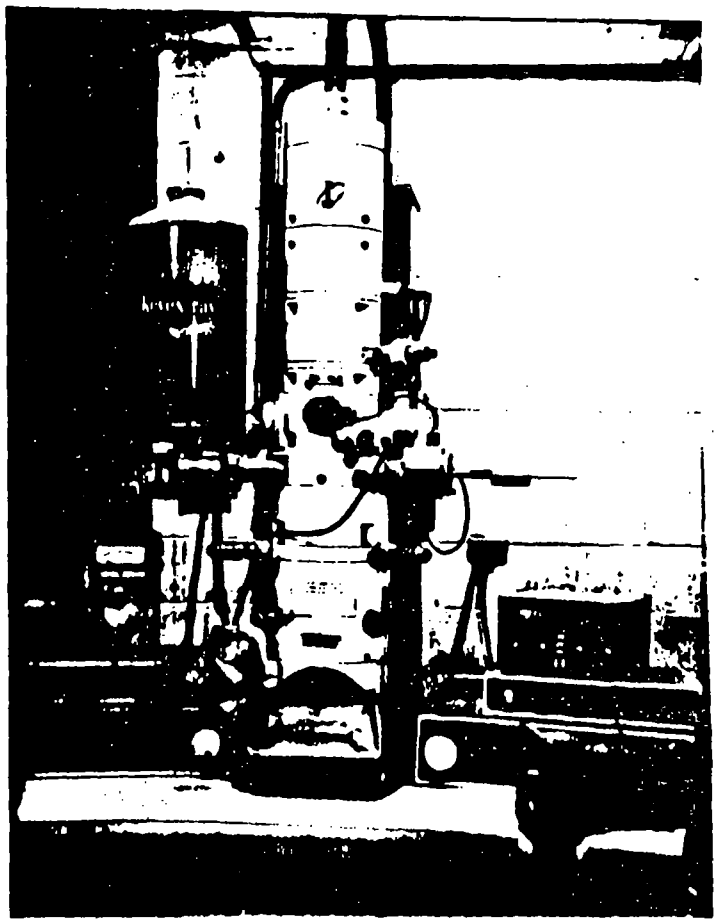
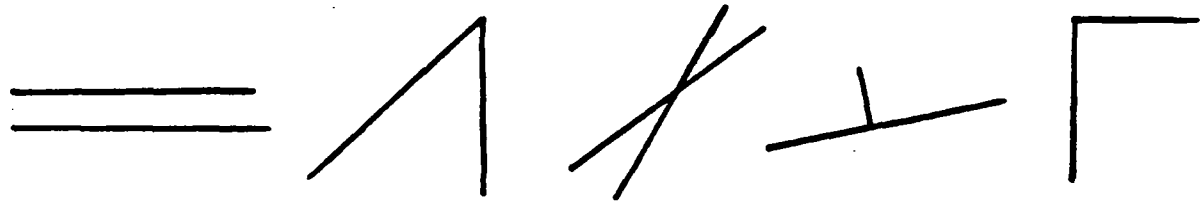


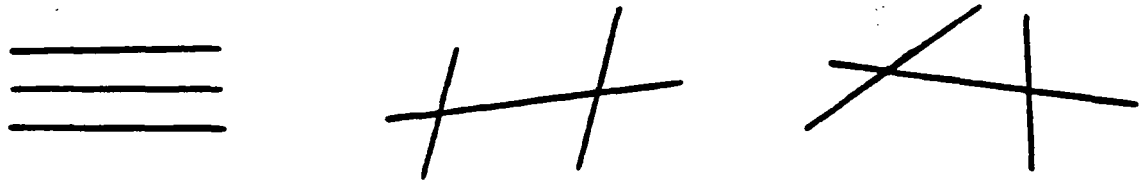
Figure A6. Transmission electron microscope.

Count as one fiber:

Count as two fibers (space between fibers greater than the width of one fiber):

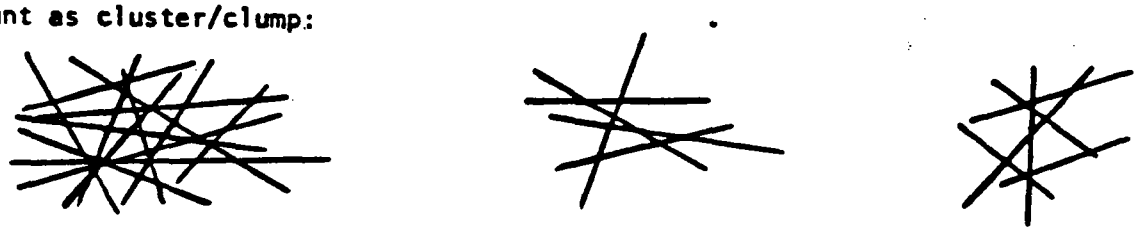


Count as three fibers:



Count as bundles:

Count as cluster/clump:



Count as matrix/debris:

Figure A7. Morphology and counting guidelines used in determining asbestos structures.

Sample No. C06470-5-4A1
Filter Type Nuclepore
Filter Area 962 cm²
Grid Opening Area .0984725 cm²

Acc. Voltage 100 KV
Beam Current 100 μA
Magnification 20,000 X
Comments:

Date 8/5/81
Grid Box 8
Grid Location C-5

GO	Struct. #	Struct.	Dimension Width	Dimension Length	SAED Observation Chrys	Amph	Ambig	No-ID	GO	Struct. #	Struct.	Dimension Width	Dimension Length	SAED Observation Chrys	Amph	Ambig	No-ID
1	1	F	1	33	✓				4	26	M	1	7	✓			
	2	F	1	19	✓					27	F	1	16				✓
	3	F	1	20	✓					28	F	1	7	✓			
	4	F	1	7			✓			29	M	1	26	✓			
	5	M	1	9	✓					30	M	1	10			✓	
	6	B	2	11	✓					31	F	1	11				✓
2	7	F	4	20			✓		5	32	M	1	41	✓			
	8	F	1	10	✓					33	B	4	22	✓			
	9	F	1	14	✓					34	M	1	6	✓			
	10	M	1	6	✓					35	M	1	6			✓	
	11	M	1	56	✓					36	M	1	13	✓			
	12	M	1	25	✓					37	M	1	17				
	13	M	1	23	✓					38	F	1	29				
	14	M	1	7			✓			39	M	1	10				
	15	M	2	12	✓				6	40	F	1	24				
	16	F	1	13	✓					41	B	4	205	✓			
	17	M	1	6	✓					42	F	5	15		✓		
3	18	M	1	15				✓		43	F	1	11	✓			
	19	M	1	19			✓			44	F	1	17	✓			
	20	F	1	17	✓				7	45	M	1	9	✓			
	21	M	1	34			✓			46	F	1	20			✓	
	22	M	1	15			✓			47	F	1	15	✓			
	23	M	1	15			✓			48	F	1	13	✓			
4	24	F	1	7				✓		49	F	1	9				✓
	25	M	3	13	✓					50	F	1	41	✓			

Figure A8. Level I data sheet (example).

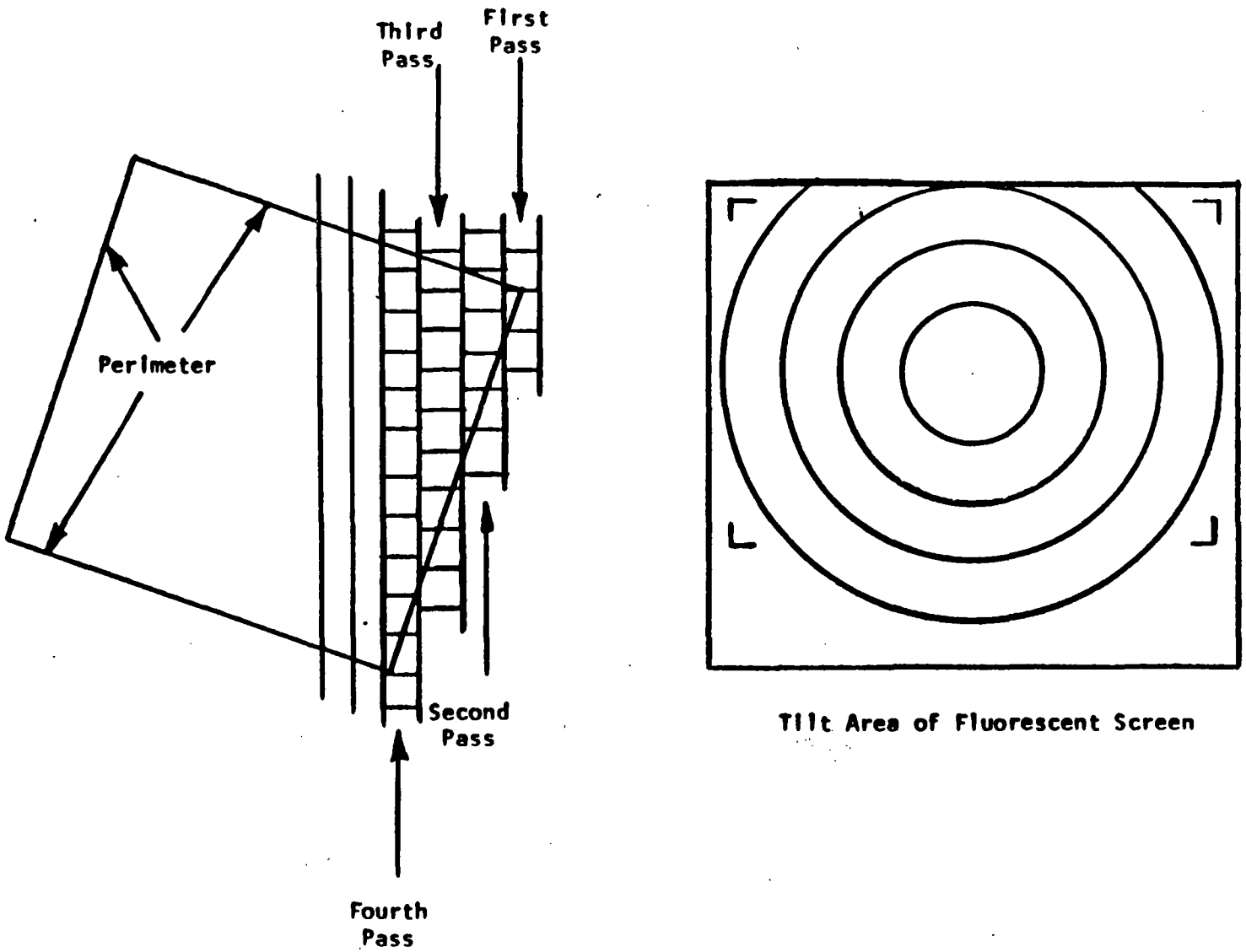


Figure A9. Scanning of full-grid opening.

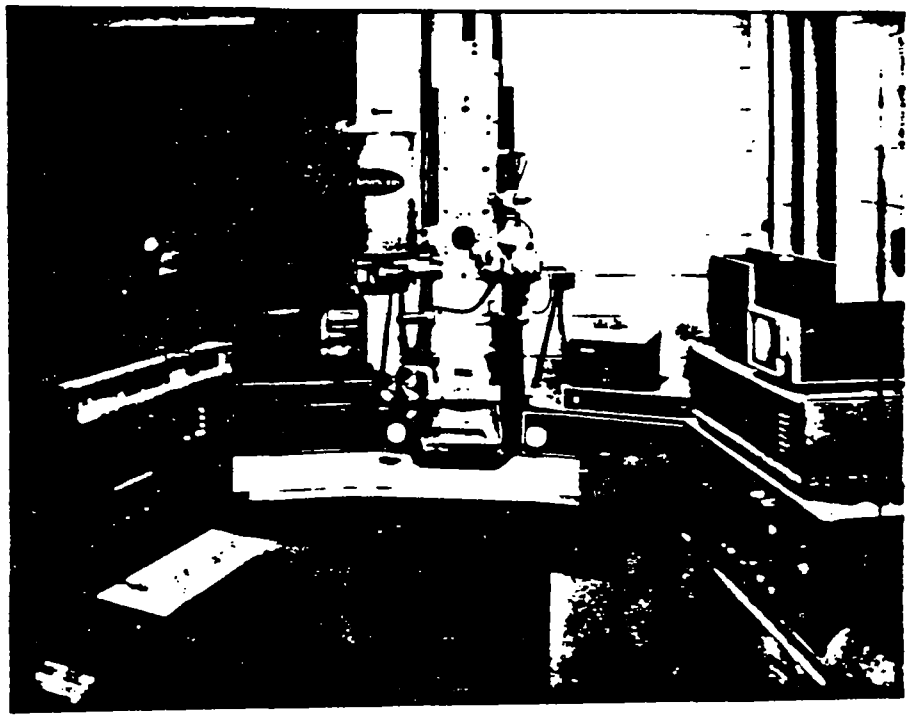
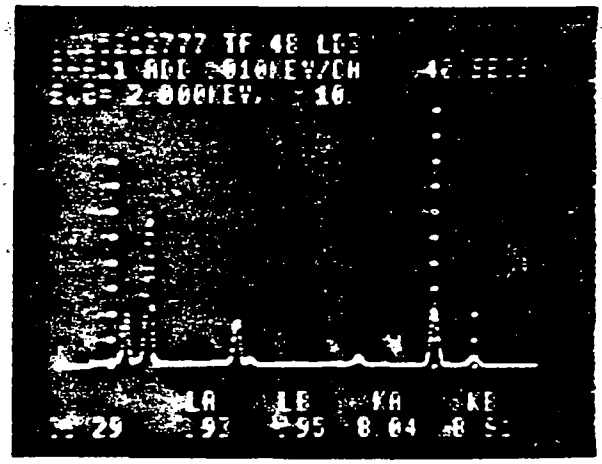
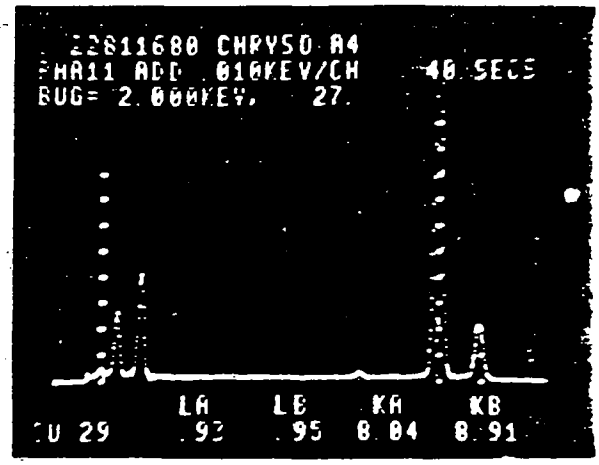


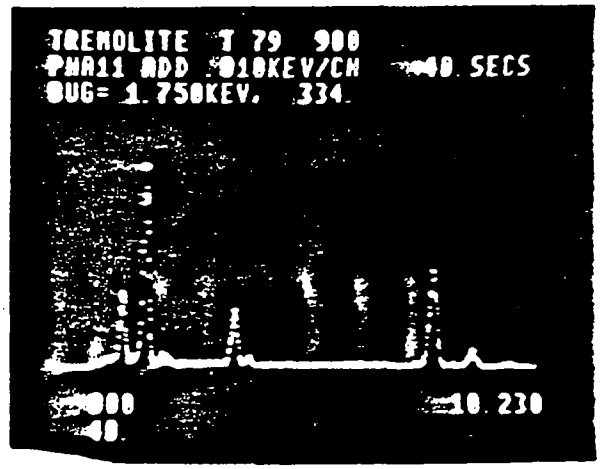
Figure A10. Transmission electron microscope with energy dispersive spectrometer.



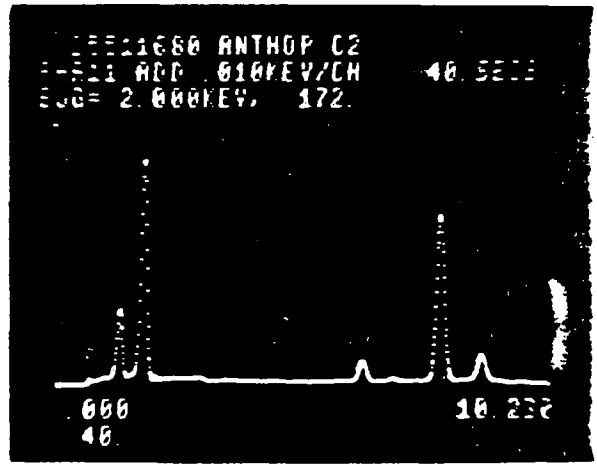
0-4-10-3-<1



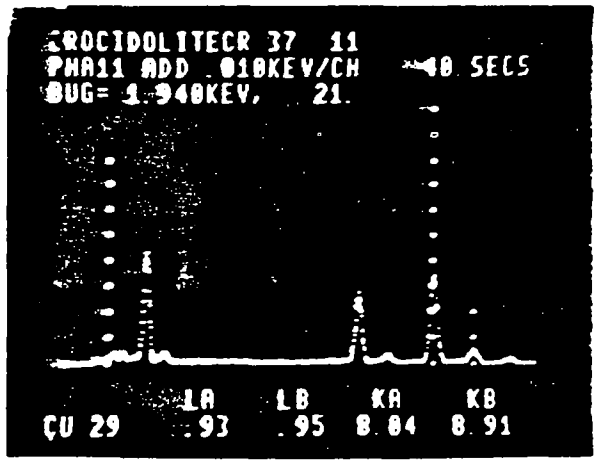
0-7-10-0-0



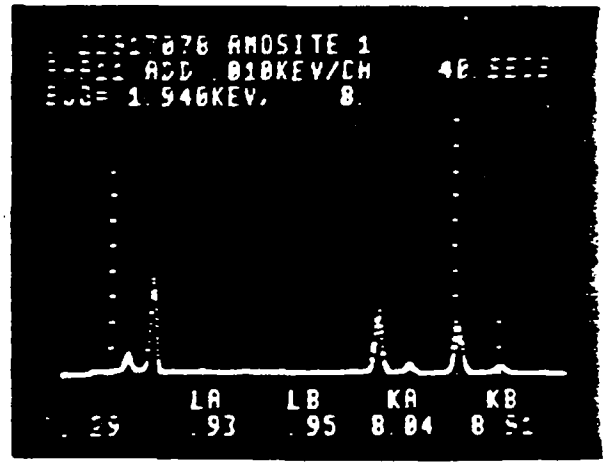
0-4-10-3-<1



0-3-10-0-1



1-1-10-0-6



0-2-10-0-7

Figure A11. Spectra profiles of asbestos standards.

Sample No. C6470-119-6513 Ex
Filter Type Nuclepore
Filter Area 0.6 cm²
Grid Opening Area 72.25 x 10⁻⁶ cm²

Acc. Voltage 100 KV
Beam Current 100 μA
Magnification 20,000 x - Film
Comments Good distribution of particulates - Few extra large particles

Date 4/13/81
Grid Box 22681
Grid Location D-7

GO	Struct. #	Struct.	Dimension Width	Dimension Length	SAED Observation Chyrs	Amph	Ambig	Non-A	No-P	SAED/Image 0°	EDS Na	Mg	Si	Ca	Fe	ID
1	1	F	2	26		✓					—	9	19	7	7	TR
	2	"	9	30		✓										TGW
2	0	"														
3	3		10	85		✓					—	35	110	35	17	TR
	4	"	6	65		✓					—	19	59	22	21	TR
4	5	"	4	23		✓					—	66	152	—	28	AN
5	6	"	1	8				✓								
	7	"	3	48		✓	✓				—	57	170	43	16	TR
6	8	"	2	30				✓								
7	9	"	2	11				✓								✓
	10	"	15	80				✓								✓
	11	"	10	80				✓								✓
	12	"	5	45				✓								✓
8	13	"	6	43			✓	✓								✓
	14	"	3	25		✓	✓				—	15	51	18	6	TR
9	15	"	7	26				✓								
10	16	"	4	25				✓								
	17	"	4	38				✓								

Figure A12. Level II data sheet (example).

EM DATA REPORT

Sample Number: R09-2865 Date Analyzed: 3/26/81
IITRI Sample No.: C010-1859 Date Sample Received: 1/30/81
Sample Type: Bulk, Air, Water, Misc. (circle one)
Filter Type: Nuclepore Area of Filter Deposit (cm^2): 8.6
Volume of Fluid Sampled: NA Mass Deposited: NA

1. Total Number of Structures: 101
2. Total Number of Asbestos Structures: 93
   2.1 Chrysotile 88
   2.2 Amphibole 5
      Crocidolite 5 Anthophyllite
      Tremolite Actinolite
      Amosite Non-Identity
   2.3 Non-Identity 8
3. Asbestos Structure Description
   3.1 Total Number of Fibers: 85 Mass (ng) .0016
      3.1.1 Chrysotile 80 Mass (ng) .0010
         Fiber Length; Range (μm) .31 - 2.25 Mean (μm) 0.78
         Fiber Diameter; Range (μm) .06 - .25 Mean (μm) 0.07
         Aspect Ratio; Range (μm) 3.5 - 31.0 Mean (μm) 11.00
      3.2.2 Amphibole 5 Mass (ng) .0006
         Fiber Length; Range (μm) .31 - 2.19 Mean (μm) 1.00
         Fiber Diameter; Range (μm) .06 - .31 Mean (μm) 0.15
         Aspect Ratio; Range (μm) 5.0 - 10.0 Mean (μm) 6.40
   3.2 Total Number of Bundles: 5 ,Mass (ng) .0007
   3.3 Total Number of Clusters/Clumps: 2 ,Mass (ng) .0002
   3.4 Total Number of Matrix/Debris: 1 ,Mass (ng) negligible
4. Area of Filter Sample Analyzed, (cm^2): .0007225
5. Total Mass of Asbestos Analyzed (ng): .0025
6. Number of Pictures Attached: 2
7. Qualitative Description of Non-Asbestos Particles Few, small particles--non-descriptive
8. Comments: Particulate loading OK.

Figure A13. EM data report (example).

SAMPLE SUMMARY REPORT

Sample Number: R09-2865 Date of Report: 4/1/81

IITRI Sample No.: C010-1859 Date Sample Received: 1/30/81

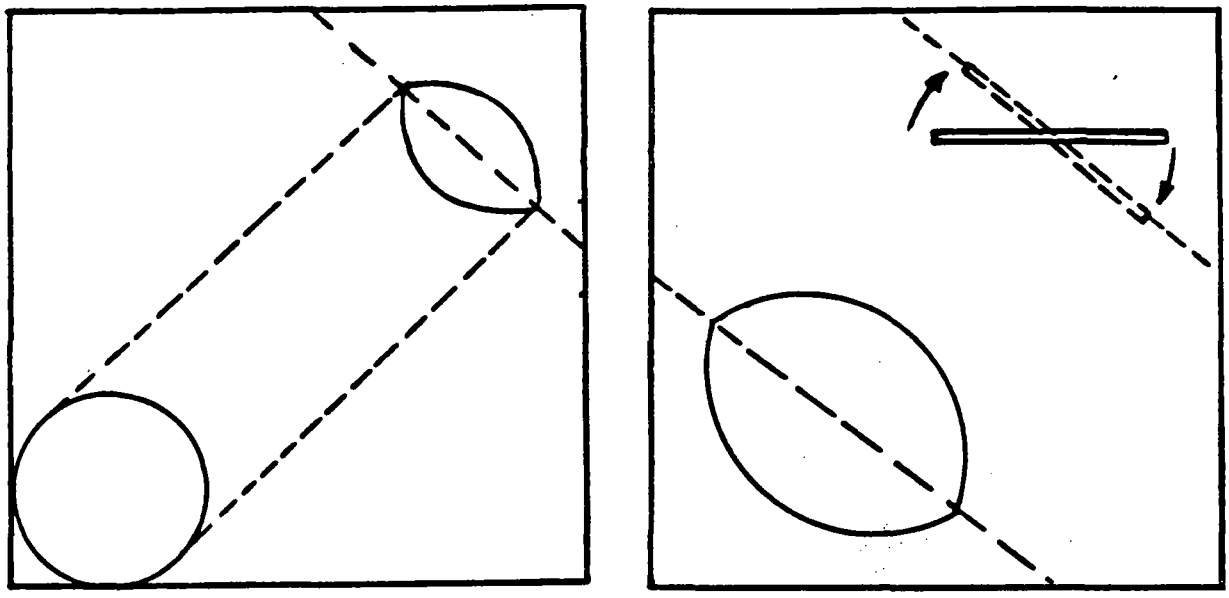
Sample Type: Bulk, (Air) Water, Misc. (circle one)

Filter Type: Nuclepore Area of Filter Deposit (cm^2): 8.6

Volume of Fluid Sampled: NA Mass Deposited: NA

1. Total Number of Structures: 1,202,215
2. Total Number of Asbestos Structures: 1,106,990
   - 2.1 Chrysotile 1,047,474
   - 2.2 Amphibole 59,516
     - Crocidolite 59,516 Anthophyllite
     - Tremolite Actinolite
     - Amosite Non-Identity
   - 2.3 Non-Identity 95,225
3. Asbestos Structure Description
   - 3.1 Total Number of Fibers: 1,011,765 Mass (ng) 19.00
     - 3.1.1 Chrysotile 952,249 Mass (ng) 11.90
       - Fiber Length; Range (μm) .31 - 2.25 Mean (μm) 0.78
       - Fiber Diameter; Range (μm) .06 - .25 Mean (μm) 0.07
       - Aspect Ratio; Range (μm) 3.5 - 31.0 Mean (μm) 11.00
     - 3.2.2 Amphibole 59,516 Mass (ng) 7.14
       - Fiber Length; Range (μm) .31 - 2.19 Mean (μm) 1.00
       - Fiber Diameter; Range (μm) .06 - .31 Mean (μm) 0.15
       - Aspect Ratio; Range (μm) 5.0 - 10.0 Mean (μm) 6.40
   - 3.2 Total Number of Bundles: 59,516 ,Mass (ng) 8.30
   - 3.3 Total Number of Clusters/Clumps: 23,806 ,Mass (ng) 2.10
   - 3.4 Total Number of Matrix/Debris: 11,903 ,Mass (ng) 0.10
4. Area of Filter Sample Analyzed, (cm^2): .0007225
5. Total Mass of Asbestos Analyzed (ng): 29.5
6. Number of Pictures Attached: 2
7. Qualitative Description of Non-Asbestos Particles Few, small particles--Non-descriptive
8. Comments: Particulate loading OK.

Figure A14. Sample summary report (example).



(a) Effects of tilting. (b) Fiber alignment.

Figure A15. Effects of tilting and alignment of fiber.

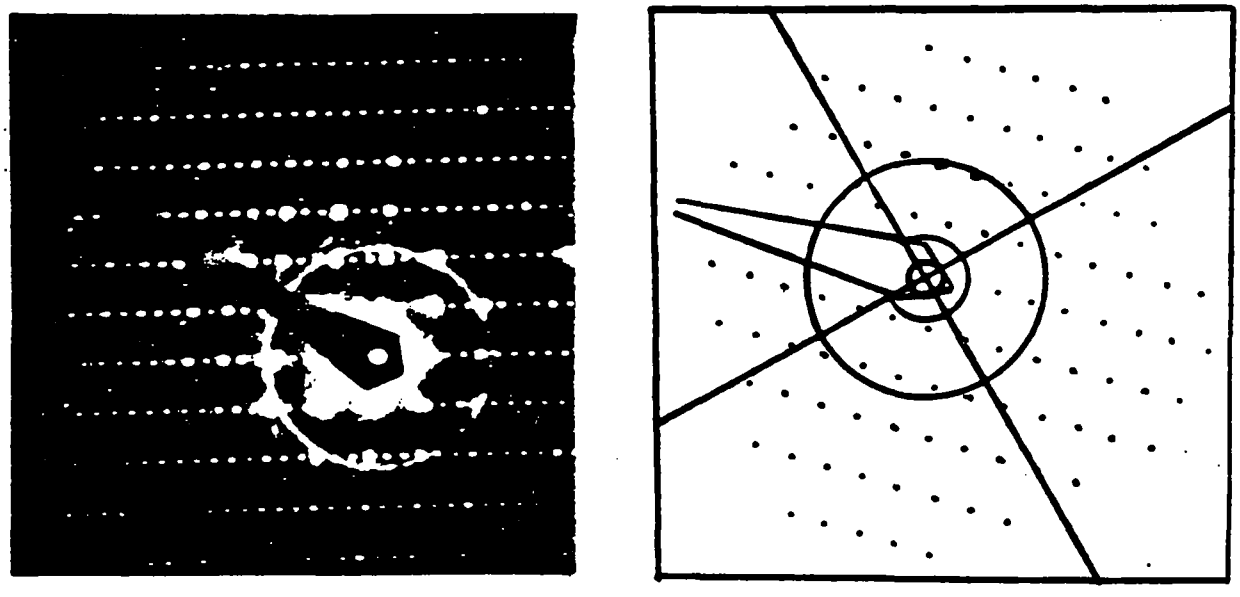
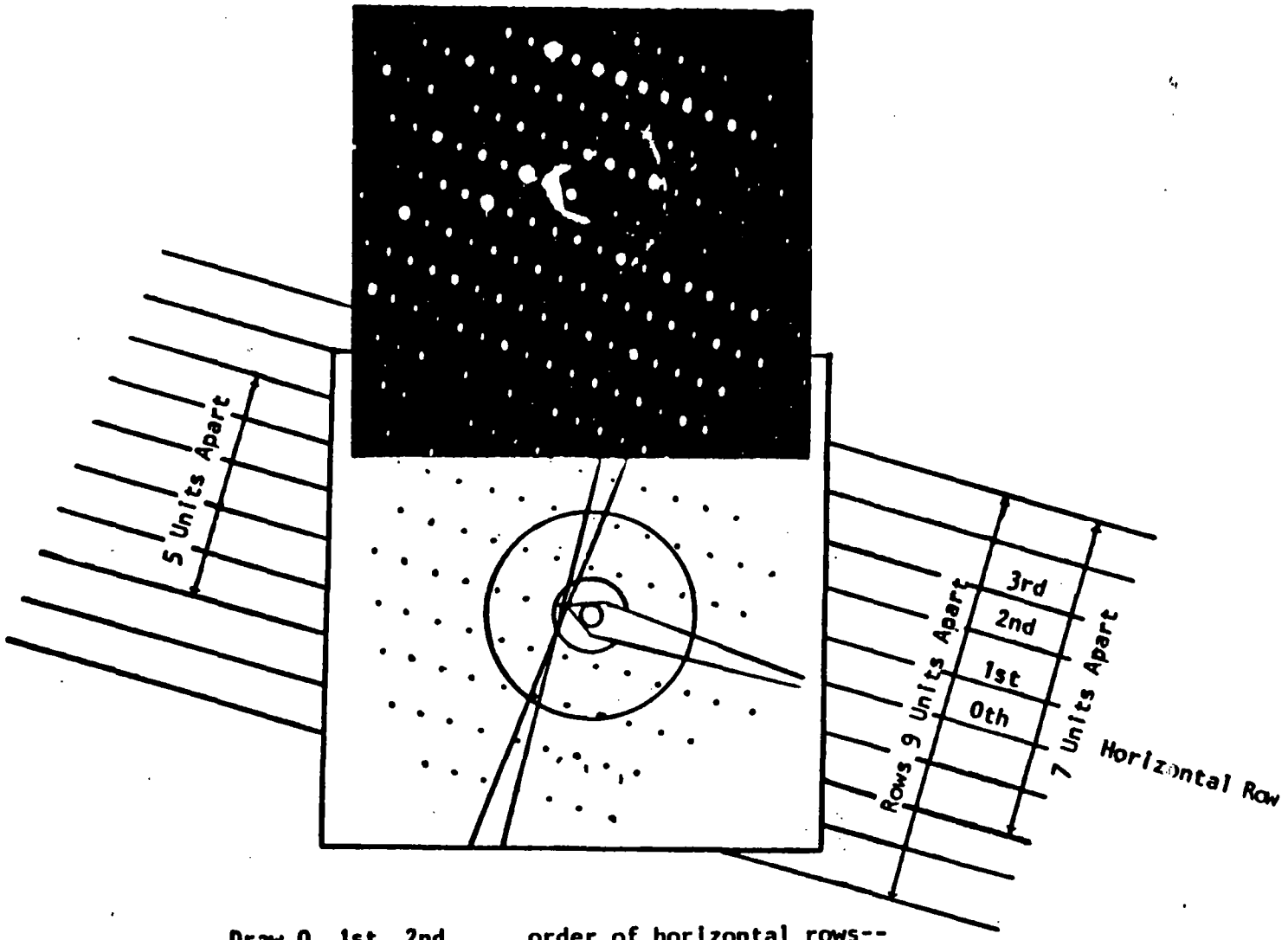
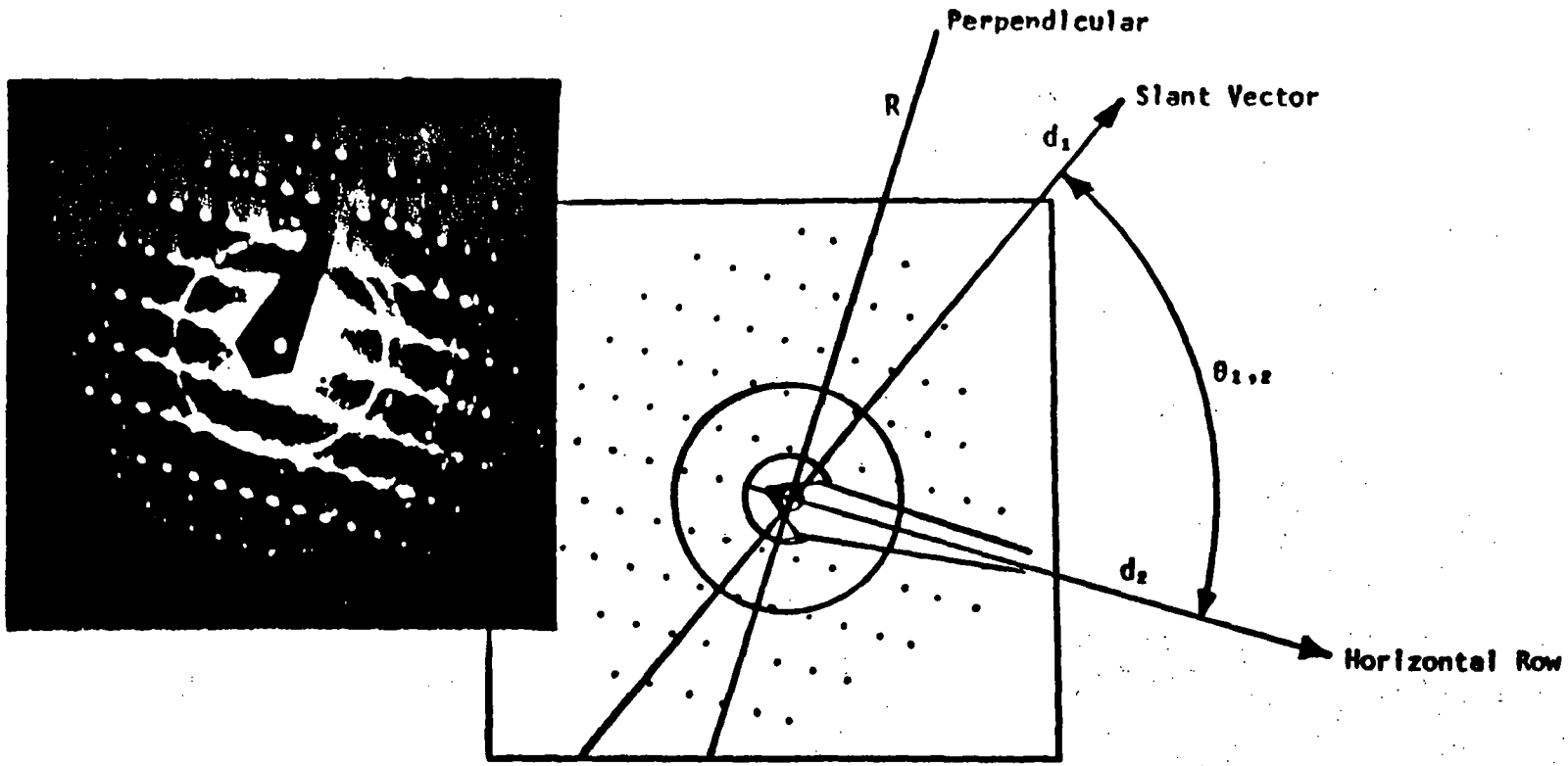


Figure A16. Method of measuring two perpendicular diameters for each ring.



Draw 0, 1st, 2nd, ... order of horizontal rows--
perpendicular separation between horizontal rows.

Figure A17. Method of recognizing a horizontal row of spots.

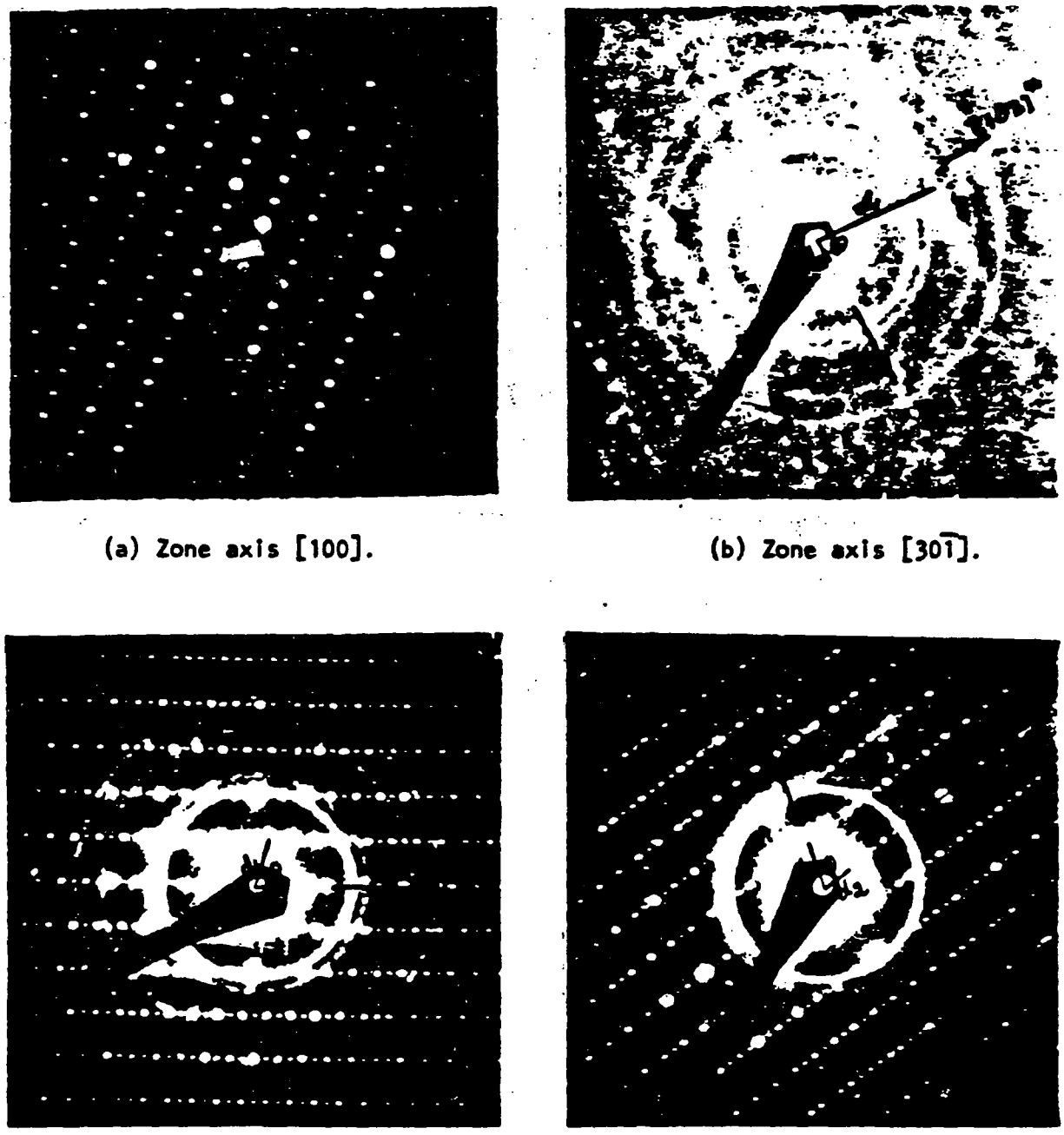


Draw the 0th horizontal row.

Draw a perpendicular through the origin.

Join the origin to the first spot to the right of the perpendicular in the 1st row and extend the line to measure the acute angle θ of this line from the 0th row.

Figure A18. Relationship of d_1, d_2, $\theta_{1,2}$, and R.



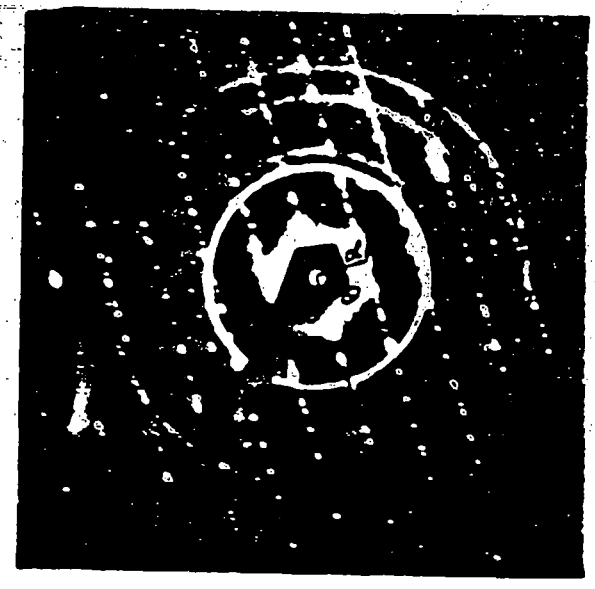
(a) Zone axis [100].

(b) Zone axis [30$\bar{1}$].

(c) Zone axis [101].

(d) Zone axis [$\bar{1}$01].

Figure A19. Typical Zone-axis SAED patterns from amosite standard specimen. (Jones et al., 1981)



(a) Zone axis [100].

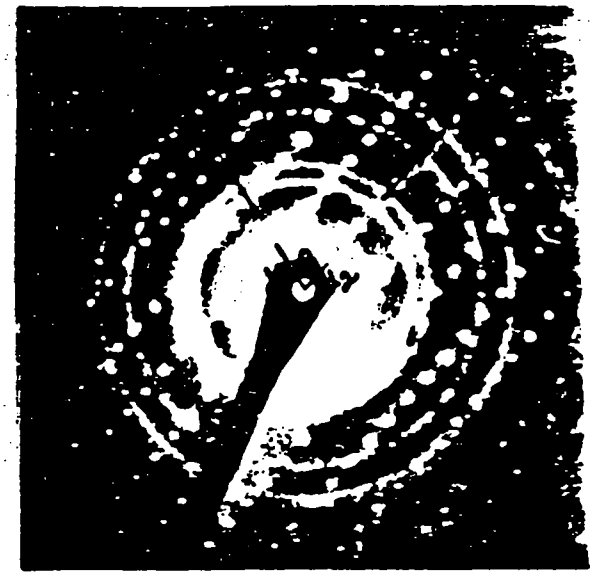
(b) Zone axis [101].

(c) Zone axis [$\bar{1}10$].

(d) Zone axis [$30\bar{1}$].

Figure A20. Typical zone-axis patterns from crocidolite standard specimen. (Jones et al., 1981)

(a) Zone axis [100].



(b) Zone axis [101].

(c) Zone axis $[\bar{2}0\bar{1}]$.

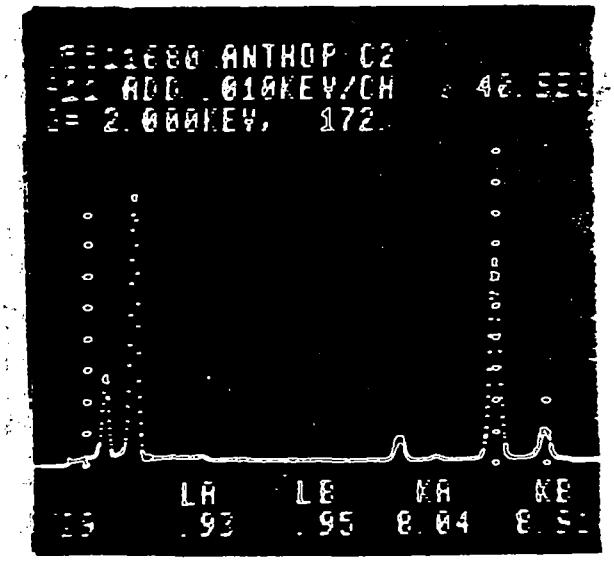
(d) Zone axis $[30\bar{1}]$.

Figure A21. Typical zone-axis patterns from tremolite standard specimen. (Jones et al., 1981)

(a) Zone axis [100].

(b) Zone axis [$\bar{1}42$].

(c) Commonly observed orientation.



(d) EDAX.

Figure A22. Typical SAED patterns and EDAX spectra from anthophyllite standard specimen. (Jones et al., 1981)

APPENDIX B

COMPUTER PRINTOUT OF LEVEL I ANALYSIS (EXAMPLE)

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
INDIVIDUAL OBJECT DATA TABLE (F=FIBER, B=BUNDLE, C=CLUSTER, M=MATRIX)
TABLE PREPARATION DATE: 21-APR-81

SAMPLE CODE:

Grd Opn	Obj	Str	Size (Micron) Depth	Width	Length	Ratio	Mass (Picogram) Chrysotile	Amphibole	Ambig	Not Asbe	No Patt	X-Raw
.1	1	F	0.000	0.187	1.38	7.3	.	.	.	X	.	
1	2	F	0.000	0.125	0.75	6.0	.	.	.	X	.	
2	3	F	0.000	0.500	8.12	16.3	.	4.786	.	.	.	
2	4	F	0.000	0.437	2.81	6.4	.	1.268	.	.	.	
2	5	F	0.000	0.250	0.94	3.7	.	.	.	X	.	
3	6	F	0.000	0.375	2.19	5.8	.	.	.	X	.	
3	7	F	0.000	0.750	3.25	4.3	.	4.307	.	.	.	
3	8	F	0.000	0.625	7.81	12.5	.	.	.	X	.	
4	9	F	0.000	0.937	3.00	3.2	.	.	.	X	.	
4	10	F	0.000	0.125	1.25	10.0	.	0.046	.	.	.	
7	11	F	0.000	0.250	9.37	37.5	.	1.380	.	.	.	
7	12	F	0.000	0.812	6.00	7.4	.	.	.	X	.	
7	13	F	0.000	0.312	3.63	11.6	.	.	.	X	.	
8	14	F	0.000	0.625	5.94	9.5	.	5.464	.	.	.	
8	15	F	0.000	0.625	3.12	5.0	.	.	.	X	.	
9	16	F	0.000	0.250	2.50	10.0	.	.	.	X	.	
12	17	F	0.000	0.187	0.94	5.0	.	.	.	X	.	
12	18	F	0.000	0.625	3.44	5.5	.	3.164	.	.	.	
12	19	F	0.000	0.312	1.87	6.0	.	.	.	X	.	
13	20	F	0.000	0.187	1.00	5.3	.	0.083	.	.	.	
13	21	F	0.000	0.437	3.88	8.9	.	.	.	X	.	
13	22	F	0.000	0.312	5.00	16.0	.	1.150	.	.	.	
14	23	F	0.000	0.562	3.06	5.4	.	.	.	X	.	
14	24	F	0.000	0.062	0.69	11.0	.	.	.	X	.	
14	25	F	0.000	0.187	3.38	18.0	.	0.280	.	.	.	
15	26	F	0.000	1.562	5.00	3.2	.	.	.	X	.	
15	27	F	0.000	0.937	3.75	4.0	.	.	.	X	.	
16	28	F	0.000	0.750	6.38	8.5	.	.	.	X	.	
16	29	F	0.000	0.250	1.56	6.2	.	.	.	X	.	

APPENDIX B (Continued)

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
SINGLE SAMPLE SUMMARY TABLES
SAMPLE CODE: TABLE PREPARATION DATE: 21-APR-81

Aerosol Object Count And Calculated Object Mass Characteristics

Object Structure	Type	Actual Object Count	Number Concen. (Number Per Cu M)	Mass Concen. (Picogram Per Cu M)	Average Width (Micron)	Average Length (Micron)	Average Length To Width Ratio
Fiber	Chrysotile	0.	0.	0.0	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
	Amphibole	12.	8304.	19155.4	0.38 ± 0.21	5.20 ± 4.50	15.35 ±13.02
	Other	22.	15225.		0.46 ± 0.37	2.87 ± 1.96	7.81 ± 5.03
	All Fiber	34.	23529.		0.43 ± 0.32	3.69 ± 3.24	10.47 ± 9.27

Sample Collection and Preparation Data

Air Volume = 1.00 Cu M
Deposit Area = 1.00 Sq Cm
Ashed Area = 1.00 Sq Cm
Redeposit Area = 1.00 Sq Cm

Grid Data

Grid ID: 22501/D-8,D-9
Individual Grid Opening = 0.000072 Sq Cm
Number of Grid Openings = 20
Film Magnification = 20000

APPENDIX B (Continued)

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
INDIVIDUAL OBJECT DATA TABLE (F=FIBER, B=BUNDLE, C=CLUSTER, M=MATRIX)
TABLE PREPARATION DATE: 21-APR-81

SAMPLE CODE:

Grd Opn	Obj	Str	Size (Micron) Depth	Width	Length	Ratio	Mass (Picogram) Chrysotile	Amphibole	Ambis	Not Asbe	No Patt	X-Ray
16	30	F	0.000	0.375	16.88	45.0	.	5.591	.	.	.	
17	31	F	0.000	0.250	1.56	6.2	.	.	.	X	.	
19	32	F	0.000	0.187	1.44	7.7	.	.	.	X	.	
19	33	F	0.000	0.062	1.69	27.0	.	.	.	X	.	
19	34	F	0.000	0.187	1.94	10.3	.	0.160	.	.	.	
			Total Mass (Picogram)=				0.000	27.680				
			Total Count =				0.	12.	0.	22.	0.	

APPENDIX C

COMPUTER PRINTOUT OF LEVEL II ANALYSIS (EXAMPLE)

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
INDIVIDUAL OBJECT DATA TABLE (F=FIBER, B=BUNDLE, C=CLUSTER, M=MATRIX)
TABLE PREPARATION DATE: 21-APR-81

SAMPLE CODE:

Grd Opn	ObJ	Str	Size (Micron) Depth	Width	Length	Ratio	Mass (Picogram) Chrysotile	Amphibole	Ambig	Not Asbe	No Patt	X-Ray
1	1	F	0.000	0.125	1.63	13.0	.	0.0598	.	.	.	MG(9) SI(19) CA(7) FE(7)
1	2	F	0.000	0.562	1.87	3.3	.	1.3977	.	.	.	TCGW
3	3	F	0.000	0.625	5.31	8.5	.	4.8892	.	.	.	MG(35) SI(110) CA(35) FE(17)
3	4	F	0.000	0.375	4.06	10.8	.	1.3460	.	.	.	MG(19) SI(59) CA(22) FE(21)
4	5	F	0.000	0.250	1.44	5.8	.	0.2117	.	.	.	MG(66) SI(152) FE(28)
5	6	F	0.000	0.062	0.50	8.0	.	.	.	X	.	
5	7	F	0.000	0.187	3.00	16.0	.	0.2485	.	.	.	MG(57) SI(170) CA(43) FE(16)
6	8	F	0.000	0.125	1.87	15.0	.	.	.	X	.	
7	9	F	0.000	0.125	0.69	5.5	.	.	.	X	.	
7	10	F	0.000	0.937	5.00	5.3	.	.	.	X	.	
7	11	F	0.000	0.625	5.00	8.0	.	.	.	X	.	
7	12	F	0.000	0.312	2.81	9.0	.	.	.	X	.	
8	13	F	0.000	0.375	2.69	7.2	.	.	.	X	.	
8	14	F	0.000	0.187	1.56	8.3	.	0.1294	.	.	.	MG(15) SI(51) CA(18) FE(6)
9	15	F	0.000	0.437	1.63	3.7	.	.	.	X	.	
10	16	F	0.000	0.250	1.56	6.2	.	.	.	X	.	
10	17	F	0.000	0.250	2.38	9.5	.	.	.	X	.	
			Total Mass (Picogram)=				0.0000	8.2822				
			Total Count =				0.	7.	0.	10.	0.	

IIT RESEARCH INSTITUTE STRUCTURE ANALYSIS DATA
SINGLE SAMPLE SUMMARY TABLES
SAMPLE CODE: TABLE PREPARATION DATE: 21-APR-81

Aerosol Object Count And Calculated Object Mass Characteristics

Object Structure	Type	Actual Object Count	Number Concen. (Number Per Cu M)	Mass Concen. (Picogram Per Cu M)	Average Width (Micron)	Average Length (Micron)	Average Length To Width Ratio
Fiber	Chrysotile	0.	0.	0.0	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
	Amphibole	7.	9689.	11463.3	0.33 ± 0.20	2.70 ± 1.50	9.39 ± 4.29
	Other	10.	13841.		0.35 ± 0.27	2.41 ± 1.56	7.75 ± 3.11
	All Fiber	17.	23529.		0.34 ± 0.23	2.53 ± 1.49	8.42 ± 3.61

Sample Collection and Preparation Data

Air Volume = 1.00 Cu M
Deposit Area = 1.00 Sq Cm
Ashed Area = 1.00 Sq Cm
Redeposit Area = 1.00 Sq Cm

Grid Data

Grid ID: 22581/D-7
Individual Grid Opening = 0.000072 Sq Cm
Number of Grid Openings = 10
Film Magnification = 20000

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO
4. TITLE AND SUBTITLE Methodology for the Measurement of Airborne Asbestos by Electron Microscopy		5. REPORT DATE July 1984 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) George Yamate, Satish C. Agarwal, Robert D. Gibbons		8. PERFORMING ORGANIZATION REPORT NO C06470
9. PERFORMING ORGANIZATION NAME AND ADDRESS IIT Research Institute 10 West 35th Streeet Chicago, Illinois 60616		10. PROGRAM ELEMENT NO. 11. CONTRACT/GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Monitoring Systems Laboratory Office of Research and Development U.S. Environmental Protection Agency Research Triangle Park, North Carolina 27711		13. TYPE OF REPORT AND PERIOD COVERED 14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

The provisional electron microscope methodology for measuring the concentration of airborne asbestos fibers was refined. The methodology is divided into separate protocols. The step-by-step procuedures for each protocol are nearly identical, so that cumulative data can be obtained and uncertainties, especially in asbestos identification, can be clarified. The operational steps encompass (1) type of sample, (2) collection and transport, (3) sample preparation, (4) examination under the transmission electron microscope (TEM) and data collection, (5) data reduction and reporting of results, and (6) quality control-quality assurance.

The TEM analytical protocol is subdidvided into three levels of analysis: Level I, for screening many samples; Level II, for regulatory action; and Level III, for confirmatory analysis of controversial samples. Because identification of asbestos structures is critical, the level of analysis is directly related to the information sought:

Level I--morphology and visual selected area electron diffraction (SAED) pattern recognition.
Level II--morphology; visual SAED; and elemental analysis.
Level III--morpholgy; visual SAED, a selected number of SAED micrographs of zone-axis patterns; and elemental analysis.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field Group

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AIR SAMPLE
TEM ANALYSIS METHOD
Developed by Steven Hayward (DHS)
Containing low-magnification scan step to
identify large fibers used in Phase II
duplicate analysis

METHODOLOGY FOR THE ANALYSIS OF AMBIENT LEVELS OF AIRBORNE ASBESTOS BY TRANSMISSION ELECTRON MICROSCOPY

Prepared by

Steven B. Hayward
Research Specialist
Air and Industrial Hygiene Laboratory
Department of Health Services
Berkeley, CA 94704-9980

AIHL METHOD (Draft)

Limited Distribution
Not for Publication

ASBESTOS IN AMBIENT AIR

Analyte: Asbestos (asbestos fibers/m^3, free asbestos fibers/m^3, asbestos structures/m^3, asbestos mass/m^3 and "LOM countable" asbestos fibers/m^3)

Application: Indoor and Outdoor Air Pollution

Matrix: Air

Procedure: Transmission Electron Microscopy (TEM) with Electron Diffraction and Energy Dispersive X-ray Spectrometry (EDS)

Date Issued: July 1984

Range: $7x10^3$ - $7x10^6$ fibers (free fibers, structures)/m^3; 300 - $7x10^6$ LOM countable fibers/m^3; 0.008 - ? ng/m^3

Precision: Coefficient of variation (CV) ranges from approximately 44% for 5 free asbestos fibers (structures) counted to approximately 11% for 100 free fibers (structures) counted. The CV of the total fiber count increases from these values with the proportion of fibers attached to air particles.

Collaboratively Tested: No

1. GENERAL DISCUSSION

1.1 BACKGROUND

Asbestos has come to be recognized as a ubiquitous environmental contaminant. Its presence in water, soil, and especially air has become evident in the last few decades. In the past, attention has focused primarily on asbestos exposure in the workplace. It is in this setting that much has been learned about the health effects of inhaling asbestos fibers.

Through the monitoring efforts of the Occupational Safety and Health Administration (OSHA), much is known about present day asbestos exposures in industries where asbestos is used commonly. These monitoring efforts have used the NIOSH recommended method, i.e., cellulose ester filter sampling with phase contrast light microscopy for analysis. (Ref. 4.1 and 4.2). In these methods it is stated explicitly that the fiber count is an index of exposure only. For the latest method (Ref. 4.2), the lower limit of sensitivity is approximately 0.1 fibers/cm^3 air. This level is considered quite low for the asbestos industries. However, it appears that in the absence of local sources, ambient levels of airborne asbestos are many orders of magnitude lower than this level. Environmental health professionals are faced with the task of evaluating ambient air levels which may be significantly elevated with respect to this "background" level, due to the presence of a local source, but which may still be lower than 0.1 fibers/cm^3 when analyzed by the NIOSH phase contrast method. According to recent risk estimates by OSHA and the Federal Consumer Products Safety Commission (CPSC), lifetime

exposures to airborne levels below 0.1 fibers/cm^3 can still result in many additional cancer cases per 100,00 population. (Ref. 4.3 and 4.4). Thus such exposures, if they do occur, can still be of substantial public health concern.

The problem of measurement sensitivity of the NIOSH method has been overcome in some individual cases by simply increasing the volume of air sampled, and/or increasing the area of the filter analyzed. However, the phase contrast method has two other fundamental limitations. First, only those fibers thicker than some value between 0.2 and 0.4 micrometers (due to the resolution limitation of the light microscope) can be detected, and second, fibers below 1 or 2 micrometers in thickness cannot be positively identified and so must be counted whether they are asbestos or not.

The best alternative to NIOSH's phase contrast method for environmental analysis is transmission electron microscopy (TEM) following sampling through a polycarbonate membrane filter. This method overcomes the two principal drawbacks of the phase contrast method: all sizes of fibers can be detected, and all asbestos fibers can be distinguished from other types of fibers. Several versions of TEM methodologies for airborne asbestos analysis have been published. (Ref. 4.5, 4.6 and 4.7). This method incorporates aspects of all of these, although the primary reference is the method developed by Yamate under contract to the EPA (Ref. 4.6). The major improvement incorporated herein is the low magnification analysis designed to mimic counts obtained by the NIOSH phase contrast method. Results using low magnification analysis have not yet been directly compared to results using the NIOSH method on replicate real world samples. However, internal consistency within

the TEM low magnification analysis (see Ref. 4.8) is all that is required for results to be compared from sample to sample. Results of future work comparing this analysis with the phase contrast method in cases where all fibers are known to be asbestos will determine how to compare TEM results with phase contrast results, allowing them to be used to assess health risks.

Sampling will not be discussed in this method. Instead, the sampling protocol developed for Alviso, CA has been included as Appendix A. It is assumed that the sample will have been acquired on a 0.2 or 0.4 pore size polycarbonate filter (37 mm diam.) and has been transported to the laboratory in the polypropylene (dichotomous sampler) filter cassette enclosed in a plastic petri dish.

This method covers all aspects of sample preparation, analysis, computation, and reporting of results. Precision based on comparisons of counts in different grid windows is discussed. However, accuracy is more difficult to assess. It is expected that interlaboratory comparisons will be carried out on a subset of all samples.

1.2 Limit defining parameters

Throughout this protocol, the analyte air concentrations listed are based on minimum and maximum air volumes of 1.8 m^3 (flowrate = 15 L/min, 2 hour sampling time and 9.6 m^3 (flowrate = 20 L/min, 8 hour sampling time), filter area of 855 mm^2 (37 mm filter holder), and 300 mesh electron microscope grids (grid opening area approx. 0.0032 mm^2). Other mesh size grids can be substituted, as long as the total area analyzed is kept approximately the same.

1.2.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 5 fibers (structures) in all grid windows counted. This is the number at which the lower 95% confidence limit due simply to Poisson counting statistics is equal to one fiber.

1.2.2 Detection limit of the overall procedure

The detection limits of the overall procedure are approximately 7×10^3 asbestos fibers (or free fibers or asbestos structures)/m^3 and 3×10^2 NIOSH equivalent fibers/m^3. (See Sections 3.2.2.7 for definitions). These are the values equivalent to the detection limit of the analytical procedure assuring a maximum air volume of 9.6 m^3. The mass concentration detection limit is approximately 0.008 ng/m^3. This assumes, in addition to the above assumptions, that all fibers are chrysotile, having a diameter of 0.04 µm (a typical chrysotile fibril diameter) and a length of 0.4 µm.

1.2.3 Reliable quantitation limit

The reliable quantitation limit is unknown because the intralaboratory precision is unknown. However, if the precision due simply to Poisson counting statistics is required to give CV = 12.5%, then 64 fibers must be counted. Assuming the maximum air volume, the reliable quantitation limit based solely on counting precision is 9×10^4 asbestos fibers (free fibers, structures)/m^3 and 3.6×10^3 NIOSH equivalent fibers/m^3.

1.2.4 Precision

Data from the analyses of seven air samples from Alviso, CA were used to evaluate the precision of the method. Total fiber counts ranged from 13 to 164, and asbestos structure counts ranged from 6 to 63. Based upon the standard deviation σ of counts among n grid windows, the 67%(1σ) confidence limits as a percentage of the average grid window count N was calculated. This coefficient of variation, CV, was estimated as

$$(1) \qquad CV = \frac{t\sigma}{z\sqrt{n}/N} \times 100\%$$

where t is the student's t statistic for the number n. (this value approaches 2 for large n).

Based upon an assumed Poisson distribution, the coefficient of variation due simply to counting statistics (CV_1) should be related to n•N, the total number of fibers counted, by

$$(2) \qquad CV_1 = \frac{\sqrt{nN}}{nN} \times 100\% = \frac{100\%}{\sqrt{nN}}$$

In Figure 1, CV is plotted as a function of nN (on a log•log plot) for this theoretical counting variation. The CV ranges from 45% at a total count of five fibers to 10% for 100 fibers. Also shown in the figure are the seven estimates of CV for the the actual asbestos structure counts, as well as the estimated CV for a count from a

reference filter material obtained from the National Bureau of Standards, and from a proposed standard reference filter material, also from the National Bureau of Standards.

These data were fit with a straight line as shown in the figure. This line corresponds fairly well with that for the theoretical counting errors at low counts, but diverges slightly at high counts. By 100 structures counted, the line fit to the data predicts a CV of approximately 11%. This result is to be expected if other errors less dependent on total fiber count are present, since the counting error should predominate at low counts. An alternative interpretation is that the line fit to the data points is not significantly different from the theoretical line, especially in view of the fact that the fit line dips below the theoretical one for low counts. In any case, for counts less than 200, counting errors will clearly dominate the method's precision.

The data points for the two reference material counts do not appear to be inconsistent with the structure counts. Again, counting statistics are expected to dominate the method's precision.

The estimated coefficients of variation for the total count data are plotted in Figure 2, along with the theoretical curve for Poisson counting statistics. Here the least-squares fit predicts a CV ranging from 65% for 5 total fibers to 24% for 100 fibers. This suggests another significant source of imprecision. Since many of the fibers

counted were attached to air particles, the variation in the number of fibers attached to each particle is expected to be responsible for the increase in the CV for these counts. The results for the reference material sample total fiber counts support this assumption, since they are composed principally of free fibers or fiber bundles, and their CV's lie close to the theoretical line. The precision for total fiber concentrations is thus likely to improve as the proportion of free fibers increases.

1.2.5 Accuracy

The accuracy of the method is unknown, although a negative bias is expected due to obscuring of fibers by particles. Interlaboratory comparisons have yet to be carried out using this method.

1.3 General Comments

1.3.1 Estimated EM times of 600 and 120 minutes for high and low magnification analyses respectively appear to be reasonable. Times for high magnification analysis can be significantly longer if the loading of particles with many fibers attached is high. Additional costs are incurred on sample preparation on analyzing count data, and reporting of results.

1.3.2 EM analysis for asbestos is tedious work, and places great demands on the microscopist. As a result, the microscopist should be accorded

latitude in the scheduling of breaks to coincide with natural stopping places in the analysis (such as finishing a grid opening). Lack of consideration of operator discomfort or fatigue will result in less reliable analyses.

1.3.3 Although this methodology has been developed for asbestos in ambient air, it can be used for analysis of asbestos in other media such as water or soil with suitable modifications. The most important modification is that the sample be finely divided and filtered uniformly through a polycarbonate membrane filter.

1.3.4 The order of analysis is field blanks, then laboratory blanks (if applicable), and then field samples.

1.3.5 The counting rule of 100 asbestos structures or 20 300 mesh grid openings, (or 40 400 mesh grid openings) whichever comes first is a minimum to keep costs down. More extensive counts are desirable, especially when structure counts are low.

1.3.6 Mass concentrations are the least reliable results reported, since the total mass is often dominated by a few large fibers, for which the precision is poor, and whose volume is often difficult to determine accurately. Although mass concentration should be reported, estimating confidence intervals is virtually impossible.

1.3.7 The order of analysis as specified in this method is

1) High magnification analysis (Sect. 3.2.2.2), and

2) Low magnification analysis (Sect. 3.2.2.10).

This order may be reversed at the operator's discretion. In fact, the low magnification analysis can be used as a screening tool to select particular samples for high magnification analysis.

2. LABORATORY REQUIREMENTS

2.1 APPARATUS

2.1.1 A modern 100kV transmission electron microscope (TEM) with energy dispersive x-ray spectrometer (EDS) is needed for the analysis. It is desirable for the microscope to have a scanning attachment for secondary electron imaging (SEI) so that particle surfaces can be examined. It should be capable of obtaining selected area diffraction patterns on areas 100 nm in diameter, or preferably it should be capable of obtaining "microdiffraction" patterns from the same size area. For fiber length and width measurements, the microscope should be equipped with a fluorescent viewing screen which is inscribed with graduated circles of known radii, or the eyepiece of the binoculars should contain a reticule with a scale.

2.1.2 A vacuum evaporator with a turntable for rotating specimens is necessary for carbon-coating samples. It can also be used for preparing carbon films, and for gold-coating of EM grids.

2.1.3 An EM preparation room adjacent to the room containing the electron microscope should either be a clean room or contain a laminar flow class 100 clean bench to minimize contamination during sample preparation.

2.1.4 A microbalance accurate to a few micrograms should be located in a temperature and humidity controlled chamber for gravimetric analysis if it is desired.

2.2 SUPPLIES

2.2.1 Modified Jaffe Wick Washers are required for dissolving polycarbonate membrane filters (See Ref. 4.5 or 4.6).

2.2.2 Miscellaneous EM supplies and chemicals needed are 300 or 400 mesh copper grids, plastic petri dishes, scalpels, tweezers, carbon rods, carbon grating replica, chloroform, and EM grid storage boxes.

3. ANALYTICAL PROCEDURE

3.1 SAMPLE PREPARATION

3.1.1 If gravimetric analysis is required, post-weighing of the sample filters should be carried out following receipt in the laboratory. (Pre-weighing is discussed in Appendix A - Sampling). Filters should be equilibrated overnight in the environmental chamber, and neutralized with a polonium source before weighing.

3.1.2 The weighed polycarbonate sample filters and suitable blanks should be coated with carbon as soon as possible. The filter is placed in the bottom half of a 100 mm plastic petri dish, and taped down around the edges with transparent tape. The petri dish is then placed on the turntable of the vacuum evaporator, the evaporator is evacuated and approximately 40 nm thickness of carbon is evaporated (according to the manufacturer's instructions), while the turntable is rotated. The evaporation should be carried out in short bursts to prevent overheating and consequent degradation of the filter material. Such degradation makes dissolution of the filter virtually impossible.

After carbon-coating, the vacuum chamber is slowly returned to atmospheric pressure, a clear cover is put on the plastic petri dish, and the marked dish is stored in the clean bench.

3.1.3 The particles on the filter surface along with their embedding carbon coat are then transferred to 300 or 400 mesh electron microscope grids in the clean room or clean bench by means of the modified Jaffe Wick Washer (See Ref. 4.5 or 4.6 for details). Four or five sections should be cut from widely separate portions of the filter so than an assessment can be made of the uniformity of the filter deposit. Dissolution should be carried out for 72 hours so that it goes to completion. At temperatures lower than 68°F, complete dissolution may take longer than 72 hours. After the grids have been removed from the washer, they should be placed on clean filter paper inside clean, 50 mm plastic petri dishes, or inside a grid storage box. The locations of the grids should be noted in the logbook.

3.2 TEM Analysis

3.2.1 TEM Analysis Guidelines

3.2.1.1 The magnification at the fluorescent screen must be determined by calibration with a diffraction grating replica in the specimen holder. If sizing of fibers is carried out using binocular viewing (to facilitate diffraction pattern viewing), the calibration should be carried out (with the fluorescent screen tilted) in both horizontal and vertical directions because of vertical stretching due to the screen tilt. In this case, fiber lengths should be measured in both

vertical and horizontal components, the magnification corrected separately on the two components, and the two components combined by the Pythagorean Theorem to yield the total length. Measured widths should also be corrected based on the angle of the fiber from the vertical θ, and the two magnifications Mh and Mv according to the formula

$$(3) \qquad W_T = W^2 \frac{\cos^2\theta}{M_v^2} + \frac{\sin^2\theta}{M_h^2}$$

where W_T is the true fiber width, and W is the apparent width on the screen.

3.2.1.2 The operator should define a "gate" through which the window is scanned. This can be the rectangular central portion of the fluorescent screen. If binocular viewing is used, the area will be much smaller, and may be defined by an enclosed figure scribed on a reticule.

3.2.1.3 Selection of the grid opening should be on a semi-random basis, that is to say, several widely dispersed areas of the grid should first be systematically selected, and then one opening should be selected randomly within each area. This helps to ensure detection of uneven distributions across large areas of the grid.

3.2.1.4 The average grid opening for a batch of grids should be determined by low magnification micrography of several openings. For this measurement, light, scanning electron, or transmission electron microscopy can be used, but the measurement must be done with an instrument for which the magnification used has been independently calibrated. Account should be taken of the rounding of the corners of the openings. Finally, during the actual asbestos analysis, only grid openings of the standard (square with round corners) shape should be selected for detailed analysis. The analyst should beware of selecting openings of irregular shape due to gross grid imperfections.

3.2.1.5 Suggested instrument settings are:

a) Accelerating voltage: 100kV

b) Magnification: 200 - 1000x for assessing the quality of the grid and the particulate loading, as well as for selecting grid openings to be analyzed, 400 - 4000x for low magnification analysis, and 20,000 - 50,000x (total with or without binocular viewing) for detailed analysis (high magnification scanning).

c) Brightness: Beam brightness should be adjusted during the scanning procedure such that features are just clearly

visible. High brightness is undesirable for two reasons. First, brightness (electron flux) should be minimized to prevent radiation damage to chrysotile fibers, and second, viewing a high brightness screen reduces operator dark adaptation, compromising the ability to see weak electron diffraction patterns.

d) Diffraction: If possible, microdiffraction should be used rather than selected area diffraction. Microdiffraction is carried out by exciting all lenses below the specimen in the same way as for selected area diffraction. The condenser lens system is adjusted so that a narrow, coherent beam is incident on the sample. This can be accomplished by highly exciting the first condenser lens, and using a small (10-60 μm) second condenser aperture. The extent of the beam determines the diffracting area, and can be made as small as a fibril diameter if desired. An image can be viewed in this mode by expanding the beam slightly by underexciting the second condenser lens, and slightly overexciting the intermediate (diffraction) lens. Once the beam is centered on the desired fiber, the second condenser lens can be used to converge the beam onto the fiber. The diffraction pattern will then be visible.

If selected area diffraction must be used, care must be taken to adjust the objective lens current for proper focus.

Microdiffraction is preferred over selected area diffraction because the focus is not critical for visibility of the pattern. Furthermore, contamination of the pattern with spots from closely adjacent material can be prevented by careful location of the beam.

3.2.2 TEM PROCEDURE

3.2.2.1 Low magnification sample assessment. At a magnification of 200 - 1000x, assess the quality of the carbon film, the clearing of the filter, and the uniformity of the particle loading. Obviously non-uniform grids or grids with many broken or folded windows should be rejected.

3.2.2.2 High magnification analysis. At a total magnification of 20,000 - 50,000x, a series of parallel traverses should be made across the grid opening. Starting at one corner, traverse the opening by moving it through the "gate" already established. Movement through the gate is stop-and-go, rather than continuous, to allow the operator time to visually inspect each area of the opening as it enters the gate. At the end of each traverse, the sample is moved one gate width, and a parallel traverse is made in the opposite direction. This process is continued until the entire grid opening has been scanned.

3.2.2.3 Fiber identification. When a suspected asbestos-containing particle is detected, scanning is stopped. The presence of asbestos is confirmed by electron diffraction (See 3.2.2.5 below). If the possible asbestos is suspected to be an amphibole, or if it is suspected chrysotile for which diffraction gives an ambiguous result, energy dispersive x-ray analysis should be employed for confirmation (See 3.2.2.6 below).

3.2.2.4 Particle sizing. Confirmed asbestos structures are categorized as fibers, bundles, or mats, and their length and width is measured and recorded. (If measurement is done through the binoculars, vertical and horizontal components of the length are measured.) If the asbestos is attached to other particles, the size of these particles should also be measured and recorded. Specific counting rules are detailed in 3.2.2.7 and 3.2.2.8 below.

3.2.2.5 Electron diffraction. Electron diffraction is used for positive identification of chrysotile asbestos. Once chrysotile has been tentatively identified by morphology (tubular appearance, diameter approximately 35 to 50 nm), a diffraction pattern should be obtained. For training purposes, it is useful to have micrographs of typical electron diffraction patterns of chrysotile. These should include patterns of both helical and right-cylindrical clino- and ortho-chrysotile (See Ref. 4.9

and 4.10 and Figures 3 and 4). For positive identification, the following characterisitics of the diffraction pattern should be observed:

1) The pattern should be streaky in appearance, with mirror-symmetric layer lines perpendicular to the fiber axis at the (silicate) spacing of 1/5.32 A. (See Fig. 3).

2) Odd-numbered layer lines should contain streaks (hko reflections, h, k odd) whose intensity varies with radius as cylindrical Bessel functions symmetrically placed about the line parallel to the fiber axis. There should be no diffraction intensity at this center point, since this would be h00, and k must be odd. For helical polytypes, these streaks are split into two streaks above and below the layer lines. (See Fig. 4). The vertical splitting increases with increasing k.

3) The zero layer line contains spots at a spacing of 1/7.3A (00ℓ reflections) together with streaks corresponding to the same lattice as is seen on the odd layer lines (0k0 reflections with k even) at a spacing of a 1/4.6A. For helical fibers, the 0k0 reflections will also be split.

4) The even-numbered layer lines contain either pairs of split spots for the clinochrysotile polytype (h0ℓ and h0ℓ,

h = even, ℓ = odd) or single spots spaced 1/14.6 A apart for orthochrysotile (h0ℓ, h = even). The central spots on the second layer line are often missing, due to slight tilt of the fiber axis from perpendicular to the electron beam.

3.2.2.6 Energy Dispersive X-ray Spectroscopy. Although suspected amphiboles should be checked for morphology (long, thin, straight-sided crystals) and electron diffraction, neither of these criteria is sufficient for positive identification of amphiboles. Ideally, double-tilting should be done to obtain two or three zone-axis diffraction patterns (Ref. 10), but the skill and/or training necessary for such an analysis can be prohibitive, even when a double tilt stage is available. Instead, the present method accepts the use of energy dispersive x-ray spectroscopy (EDS) for amphibole identification as well as for identification of chrysotile for which no distinct diffraction pattern can be discerned.

The microscope manufacturer's instructions for obtaining spectra should be followed, and the spectra should be compared to library spectra obtained from standard preparations (See Figures 5, 6, 7, 8, and 9 and Ref. 4.6). Care should be taken to obtain spectra from portions of the fibers well separated from other particulate matter if possible, to avoid the superposition of interfering spectra.

3.2.2.7 Fiber Nomenclature. Fibers are first classified as positive chrysotile (chrysotile morphology, chrysotile diffraction pattern, or EDS), tentative chrysotile (chrysotile morphology, ambiguous diffraction, ambiguous EDS, usually due to overlapping particles), positive amphibole with type (amphibole morphology, amphibole EDS), tentative amphibole (amphibole morphology, ambiguous EDS due to overlapping particles), or non-asbestos. Asbestos fibers should then be classified according to the state of aggregation, either as single fibers, bundles (two or more fibers in a parallel arrangement, with fibers closer together than one fiber thickness), and mats (two or more fibers in a random arrangement, but which are separated from at least one other fiber in the mat by less than a fiber diameter somewhere along their length). See Ref. 4.6, Figure A-7 for specific fiber-counting guidelines. Note: The term "mat" should be substituted for "cluster/clump". The term "matrix/debris" in the figure is used to denote fiber, bundle, or mat attached to a non-asbestos fiber.

3.2.2.8 Additional Fiber Information. All fibers, bundles, and mats attached to non-asbestos particles are to be counted and sized separately, and the particle itself is also to be sized (See Fig. 10 for a suggested sample count sheet). For fibers on particle surfaces, a scanning attachment with secondary electron imaging capabilities is useful. However, for fibers

of which part of the length is obscured, lengths should be increased from that actually observed according to the following rules:

1) If the obscuring particle or grid bar is large enough for the actual length of the fiber to be twice that observed, record a value equal to twice the observed length.

2) If the obscuring particle or grid bar is too small for the actual length to be twice that observed, record a value equal to the length of the fiber plus the size of the particle or grid bar.

3) Lengths estimated in this way should be flagged on the count sheet.

4) Fibers that extend over more than one grid opening should be counted only once.

3.2.2.9 Filter Area to be Analyzed. Enough grid windows should be analyzed for detection of at least 100 asbestos "structures", i.e., free fibers, free bundles, free mats, or particles with asbestos attached. However, no greater than 20 300 mesh or 40 400 mesh grid windows should be analyzed. The analysis should be distributed evenly among the four grids.

3.2.2.10 Low Magnification Analysis. At a total magnification of 400-4000x, approximately 50 (300 mesh) or 100 (400 mesh) intact grid openings in each of the four grids should be examined. (This analysis is to be used for the detection of "NIOSH equivalent" fibers). Methods for asbestos identification, fiber classification, and fiber sizing are identical to those used for the high magnification analysis. The differences between this analysis and the high magnification analysis are as follows:

1) The total filter area analyzed is at least 10 times that analyzed at high magnification.

2) Small fibers (as free fibers), in bundles or mats, or attached to particles, may not be detected. However, the diameter of these missed fibers will be far below the resolving power of the phase contrast light microscope. The exact diameter below which detection is impossible will depend on the magnification.

3) The fibers detected at low magnification should be selected by the data processing to conform closely to phase contrast detection criteria. Only fibers with diameter $\geq$ 0.2 or 0.3 µm, with length $\geq$ 5 µm, and with aspect ratio $\geq$ 3:1 should be retained as "NIOSH equivalent" fibers.

4) It is unlikely that, in 200 (300 mesh) grid windows, greater than 100 fibers satisfying the NIOSH size criteria will be detected. However, if this is the case, the count may be terminated at this point.

3.3 DATA REDUCTION

3.3.1 Data reduction is greatly facilitated by access to a computer. The program should use as input the following quantities: The total air volume filtered (V_a), the effective filter area (A_f), the area of a grid opening (A_g), and the magnification at which fibers were sized M (or the horizontal and vertical magnification [M_H and M_V] if fibers were sized with the screen tilted). The fiber counts read by the program should include all information entered on the count sheet, including grid opening number, fiber type (positive or tentative chrysotile, positive or tentative amphibole), fiber classification (fiber, bundle, or mat), structure classification (free or attached to particle), length flag (length augmented due to obscuring of an end), and size information (length L_i and width W_i, or width with horizontal and vertical components L^H_i and L^Y_i of length if the screen is tilted, and estimated thickness of mat). Sizes of particles which have asbestos attached should also be entered, as well as some code to designate which fibers, bundles, or mats are attached to each particle.

3.3.2 Calculated quantities should include the concentration of total asbestos fibers (C_T) with 95% confidence limits in fibers/m^3, the concentration of asbestos structures (C_S) with 95% confidence limits in structures/m^3, the concentration of free fibers (C_F) with 95% confidence limits in free fibers/m^3, asbestos mass concentration (C_M) in ng/m^3, and NIOSH equivalent fiber concentration (C_{NE}) with 95% confidence limits in fibers/m^3 > 0.3 µm diameter, > 5 µm length. For example, C_T is calculated as,

$$(4) \quad C_T(\text{fibers}/m^3) = \frac{N_T A_f}{n A_g V_a (m^3)}$$

where N_T is the total number of fibers detected n grid openings. A similar calculation is carried out for C_S, C_F, and C_{NE}. Confidence limits should only be calculated if the fiber (or structure) counts are randomly distributed among grid windows (See Ref. 4.12 for statistical tests for random distributions and confidence interval calculation). Calculations should be done on each of the four fiber types, as well as for total asbestos (positive and tentative chrysotile, and positive and tentative amphiboles).

Mass concentration in ng/m^3 should be calculated from the total estimated volume V_i (in units of 10^{-9} cm^3 of each asbestos fiber, bundle, or mat, assuming specific gravities (SG) of 2.5 for chrysotile and 3.25 for amphiboles), by

$$(5) \quad C_m = \frac{SG\, A_f \sum_i V_i}{n A_g V_a (m^3)}$$

Means, standard deviations, geometric means, and minus one and plus one geometric standard deviation values should be calculated for lengths and widths. Also, it is desirable to have some graphical or tabular representation of length and width distributions. (See Figure 11 for a sample report sheet).

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4.12 Anderson, C.H. and Long, J.M. Interim Method for Determining Asbestos in Water, EPA-600/4-80-005, U.S. Environmental Protection Agency, Environmental Research Laboratory, Athens, GA (1980).

FIGURE CAPTIONS

1. Plot of coefficient of variation (CV) versus structure count.

2. Plot of CV versus total figure count.

3. Electron diffraction (microdiffraction) pattern of right cylindrical orthochrysotile fiber. Silicate spacing is indicated.

4. Microdiffraction pattern of helical clinochrysotile fiber.

5. Energy dispersive x-ray spectrum (EDS) of chrysotile fiber. Copper peaks are from copper grid.

6. EDS of amosite fiber.

7. EDS of crocidolite fiber. Titanium peaks are from titanium grid.

8. EDS of tremolite fiber.

9. EDS of actinolite fiber.

10. Suggested count sheet format.

11. Sample report sheet.

APPENDIX A

Sampling Protocol — Asbestos in Ambient Air

1. Introduction

This protocol is designed for sampling ambient air for asbestos fibers. Since analysis is assumed to be by electron microscopy, polycarbonate membrane filters are specified. In order to obtain enough material in a four to eight hour period, a medium volume (15-20 liter per minute) air flow through a 37 mm filter is specified. The use of a size selective inlet is optional, since physical sizing is done in the microscope. If such an inlet is used, it is desirable for it to operate isokinetically, and to have a 10 μm aerodynamic diameter cutpoint at the specified flow rate.

2. Apparatus

The sampling equipment consists of 1) a sampling unit containing a pump, flowmeter, flow controller, skiptimer, and sample cassette holder, and 2) a filter and backing pad installed in a filter cassette and carried to the site in a plastic petri dish. Devices for measuring meteorological variables, such as wind-speed and direction, will also be available somewhere in the sampling grid.

2.1 Sampling unit.

This unit can be purchased from a company such as Sierra Anderson. It is usually self-contained, and requires 110V AC electrical power. It should include the following:

2.1.1. Pump.

The pump must be capable of sampling at a rate of 15-20 liters per minute (Lpm) through a 37 mm diameter polycarbonate membrane filter, pore size 0.2 µm.

2.1.2. Flowmeter and controller.

A flowmeter reading from 0 to 20 Lpm and a flow controller should allow the flow rate to be controlled to the desired value (15-20 Lpm) and maintained up to a pressure drop of 15 in Hg.

It is also desirable for the pump exhaust to have an exposed fitting, thus allowing the attachment of a dry test meter to calibrate the internal flowmeter.

2.1.3. Skiptimer.

A timer should be available to start and stop the sampling at preset times. Total sampling times of 4-8 hours should be possible.

2.1.4. Sample cassette holder.

The sample cassette holder will accomodate the plastic cassettes which are used to hold the polycarbonate membrane filters and the backing pads. It should be mounted at a height of 1½-2 meters above the ground level. Adjustable wrenches are often required to tighten airline connections and set the sample height, and compressed freon is useful for blowing off the inlet.

2.2 Filter cassette assembly.

The filter and backing pad will be preloaded into the cassette after preweighing of the filter, and the cassette into a clean plastic petri dish, in the laboratory, in order to minimize handling in the field. The petri dish should have a gummed label on the top for recording the filter number and sample identification information. The filter number will be recorded at the time of weighing, which will be carried out after a period of 24 hours of equilibration in the environmental chamber.

3. Sampling Procedure

3.1 Setting up the sampler

Locate the sampler assembly where power is available. Blow out the sample cassette holder inlet with compressed freon or wipe it out with a lintless

cloth. Remove the filter cassette from the petri dish and mount it in the cassette holder. Turn on the sampler manually annd set the flow rate at the desired value (15-20 Lpm). Turn it off and set the timer to turn it on at the desired time. Make sure the inlet cover (rainhat) is removed from the sample cassette holder inlet. Number the petri dish, and record the following in a logbook:

1. Filter number

2. Sampler location and height

3. Pump start time and date

4. Starting flow rate

5. Meteorological information, if available.

The sampler should be checked periodically during the sampling period for proper operation, and the flow rate should be recorded each time. If the wind speed and direction are known to change dramatically, sampling should be terminated.

3.2 Sampling times

Optimum sampling times are 4-8 hours if the flow rate is 15-20 Lpm through a 0.2 μm pore size polycarbonate filter. The sampling time should be

selected based upon the predicted meteorological conditions. However, if the flow rate shows a sharp reduction (to below 10 Lpm) during the sampling period, sampling should be terminated to prevent damage to the pump and overloading of the filter.

3.3 End of Sampling period

Check the flow rate just before sampling ends. If the step-timer has shut off the sampler at a preset time, manually switch on the pump momentarily to note the flow rate. When the pump is shut off, remove the cassette from the cassette holder, being careful not to disturb the filter. Replace the cassette in the petri dish, recording in the logbook

1. The time of sample termination,

2. The flow rate, and

3. The meteorological conditions (if known).

3.4 Blanks

On each sampling day, extra loaded cassettes should be carried to the sampling sites in petri dishes. These serve two purposes:

1. If a filter is deemed to have been contaminated before sampling, a spare will be available to replace it, and

2. One of the spare cassettes should be removed from the petri dish, mounted in the cassette holder of one of the samplers, dismounted, and returned to the petri dish. This should then be labeled as the field blank.

3.5 Shipping

All filter cassette petri dishes, including the field blank, should be carried in person to the laboratory. Samples should be protected from shock, and kept upright at all times.

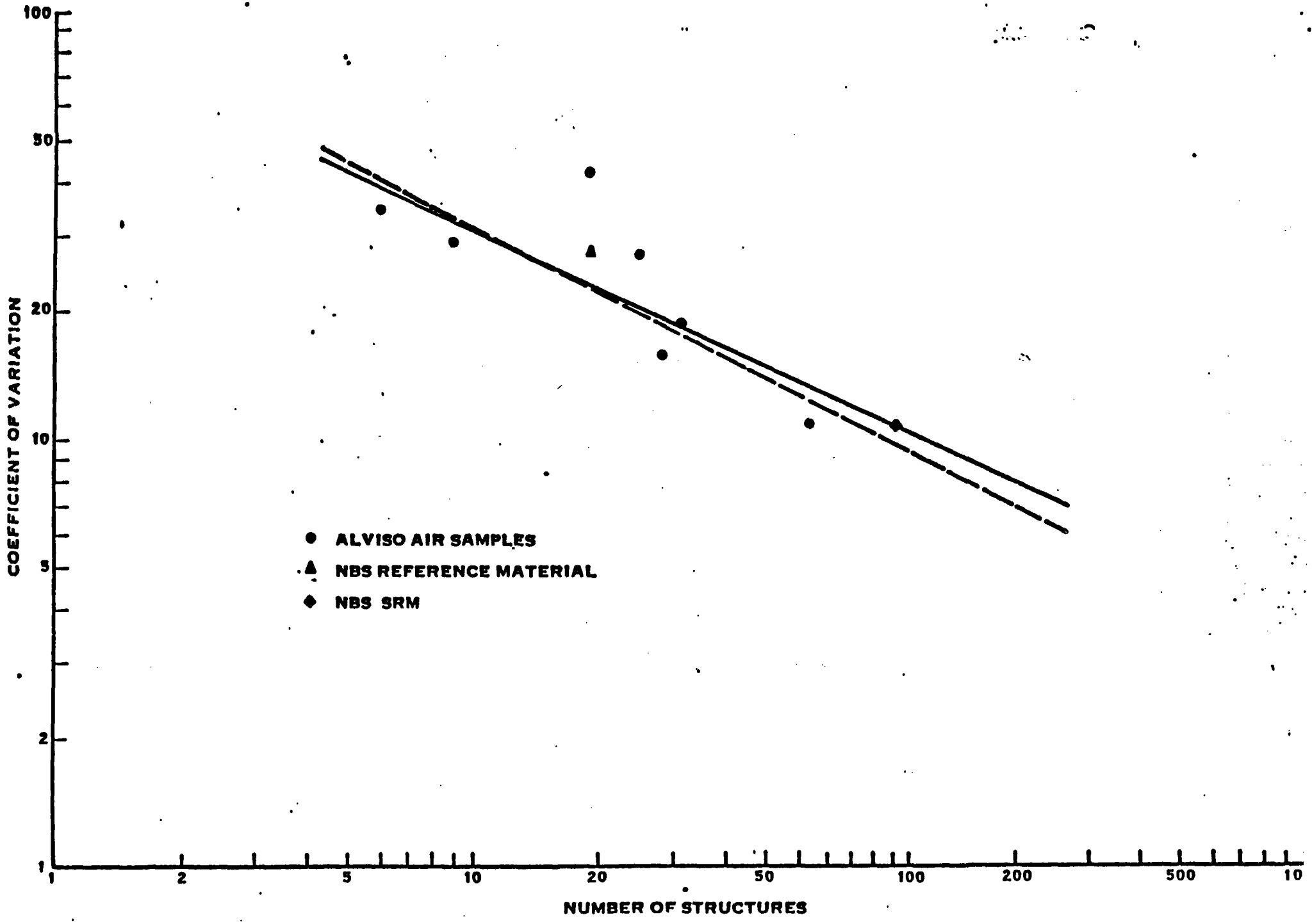
100
50
20
10
5
2
1
COEFFICIENT OF VARIATION
ALVISO AIR SAMPLES
NBS REFERENCE MATERIAL
NBS SRM
1
2
5
10
20
50
100
200
500
10
NUMBER OF STRUCTURES

Figure 1

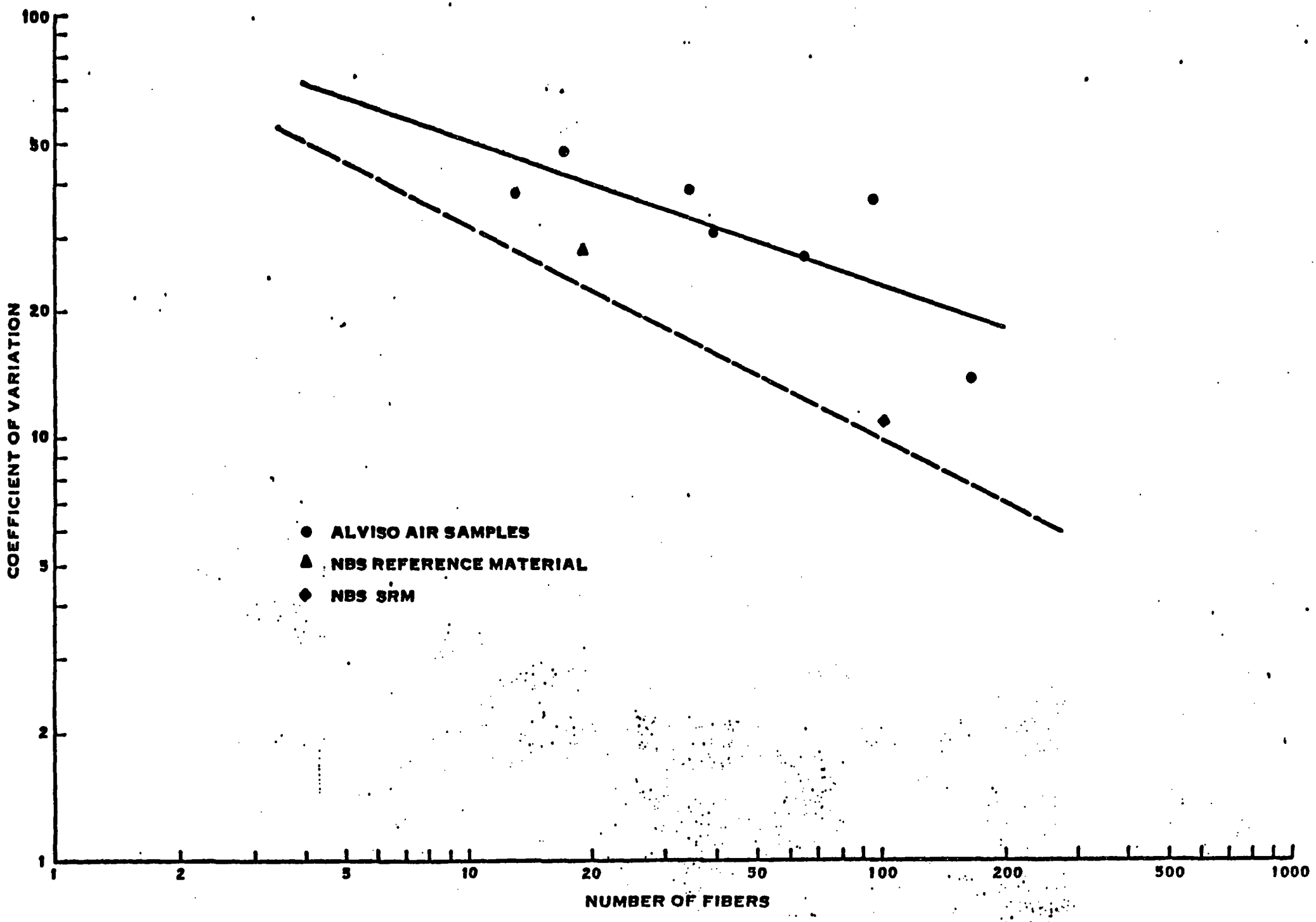
100
50
20
10
5
2
1
COEFFICIENT OF VARIATION
ALVISO AIR SAMPLES
NBS REFERENCE MATERIAL
NBS SRM
1
2
5
10
20
50
100
200
500
1000
NUMBER OF FIBERS

Figure 2

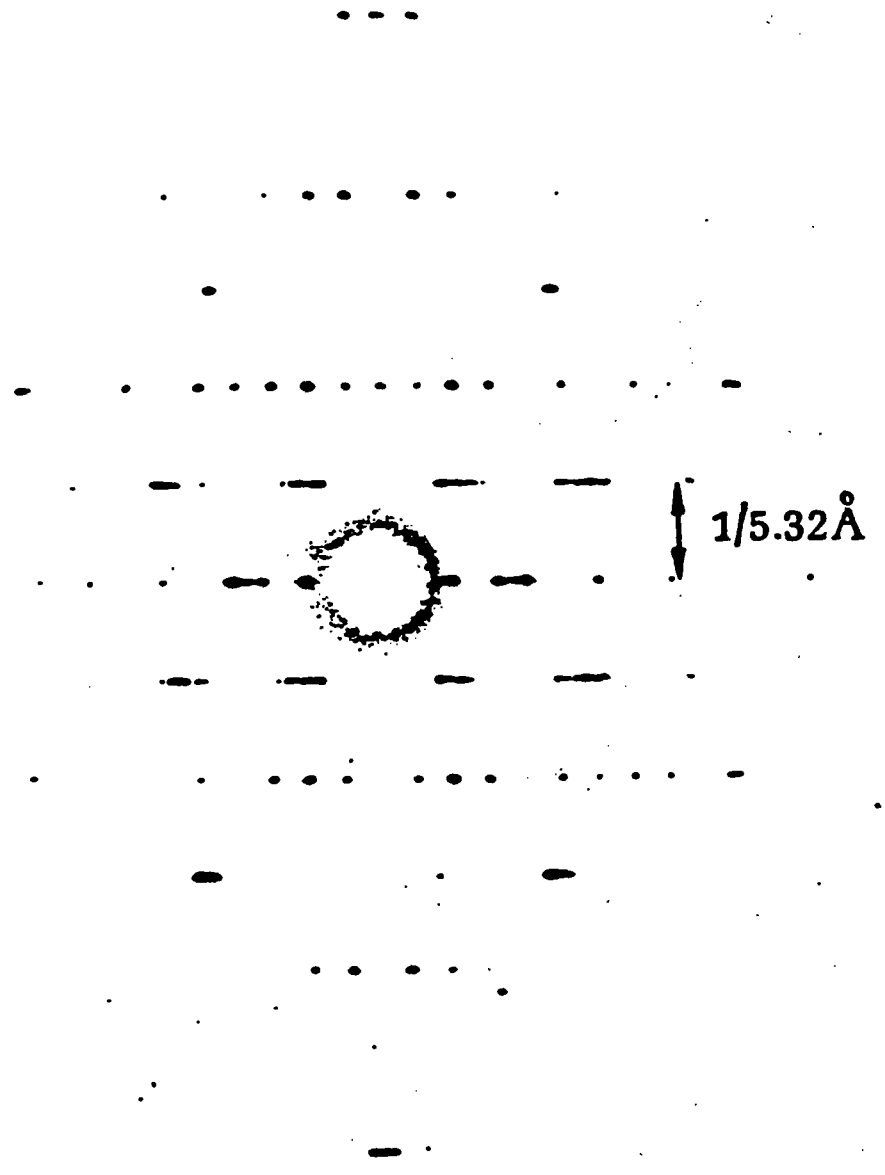

Figure 3. Electron diffraction (microdifraction) pattern of right cylindrical orthochrysotile fiber. Silicate spacing is indicated.

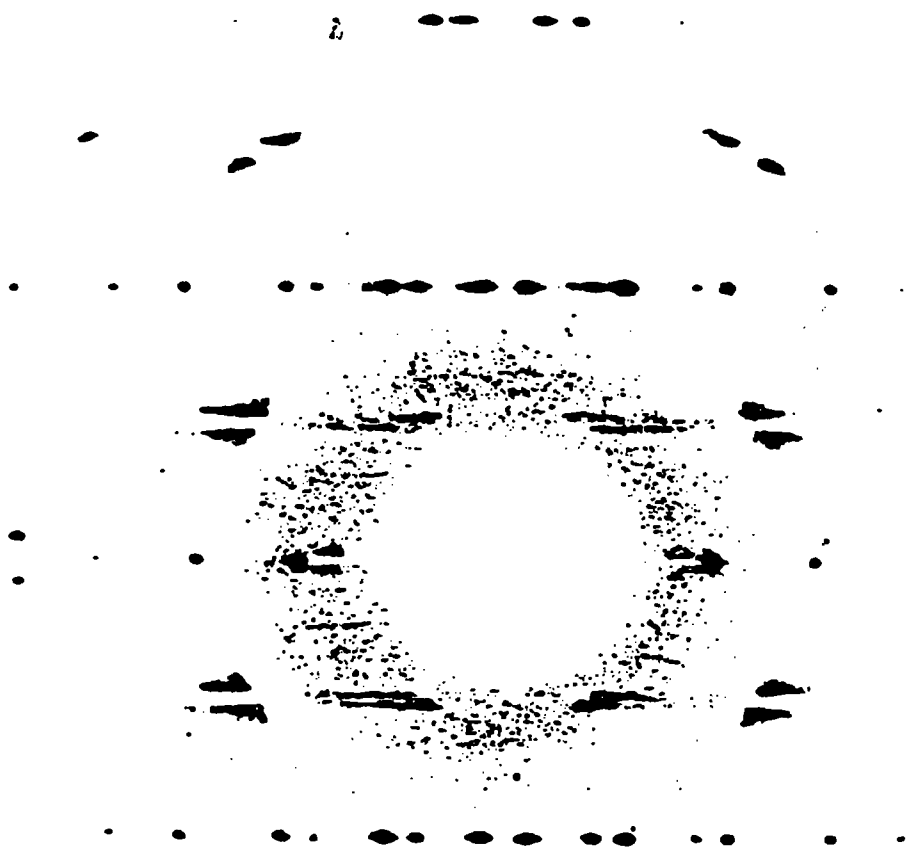

Figure 4. Microdiffraction pattern of helical clinochrysotile fiber.

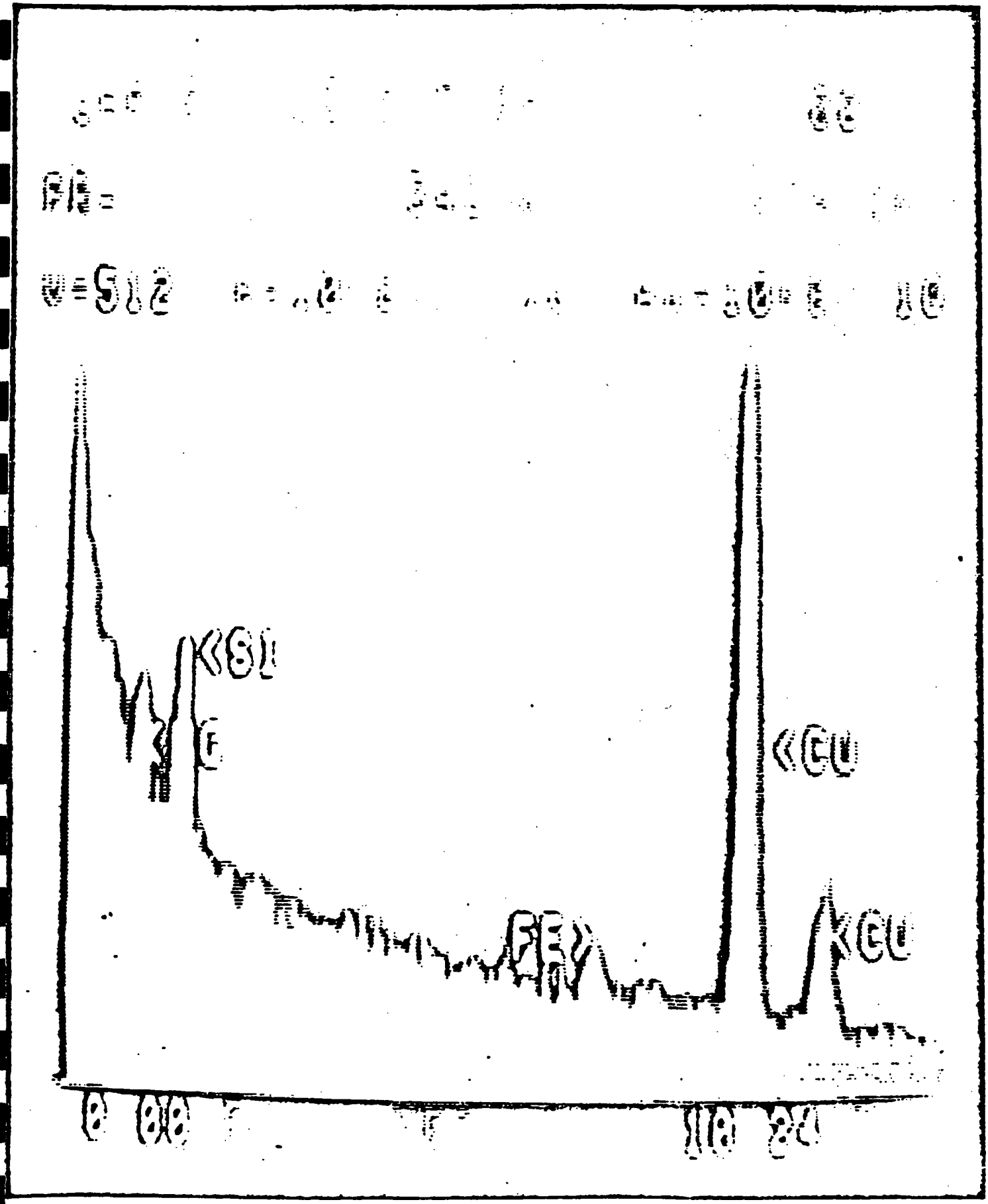

Figure 5. EDS of chrysotile fiber.

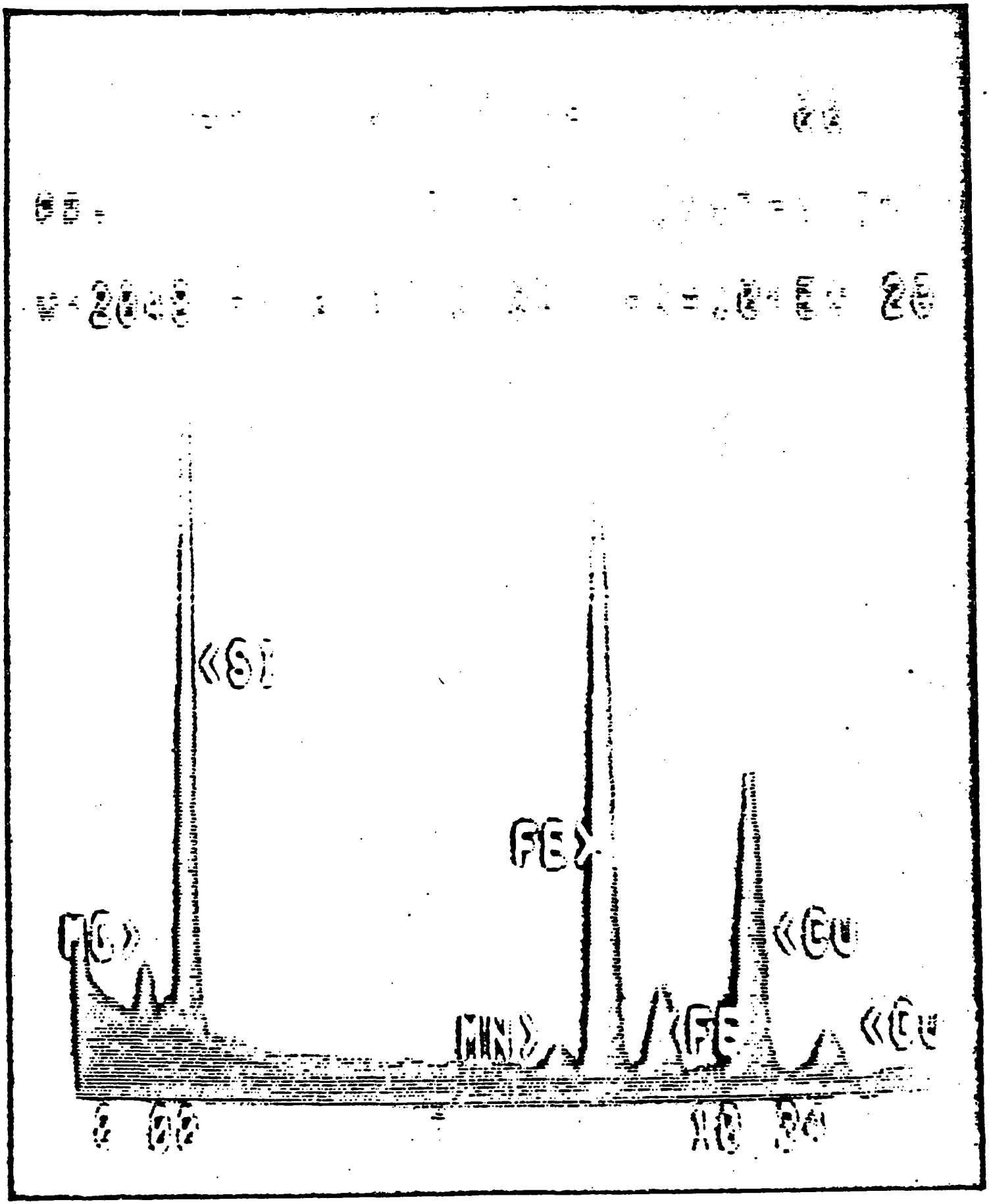

Figure 6. EDS of amosite fiber.

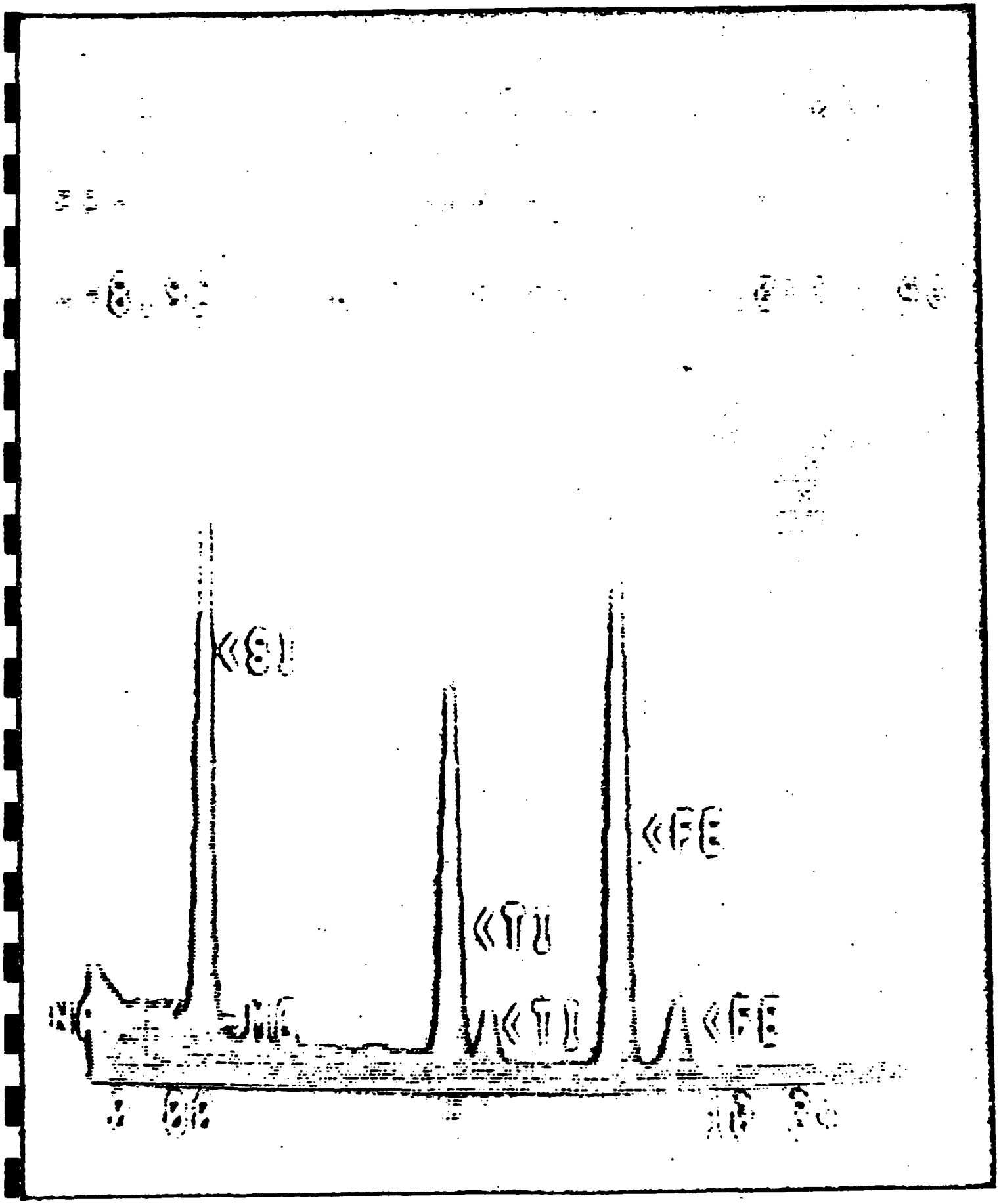

Figure 7. EDS of crocidolite fiber.

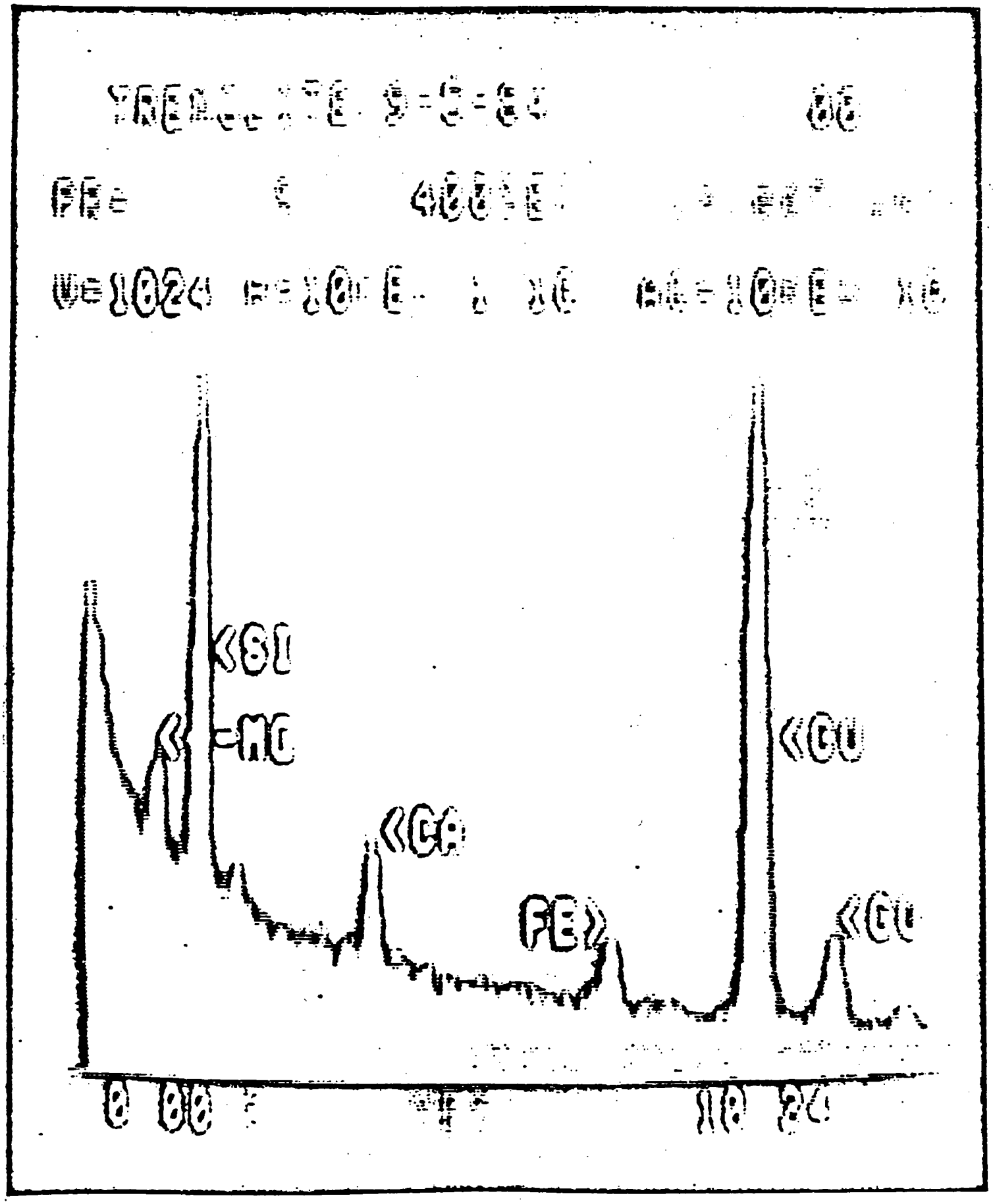

Figure 8. EDS of tremolite fiber.

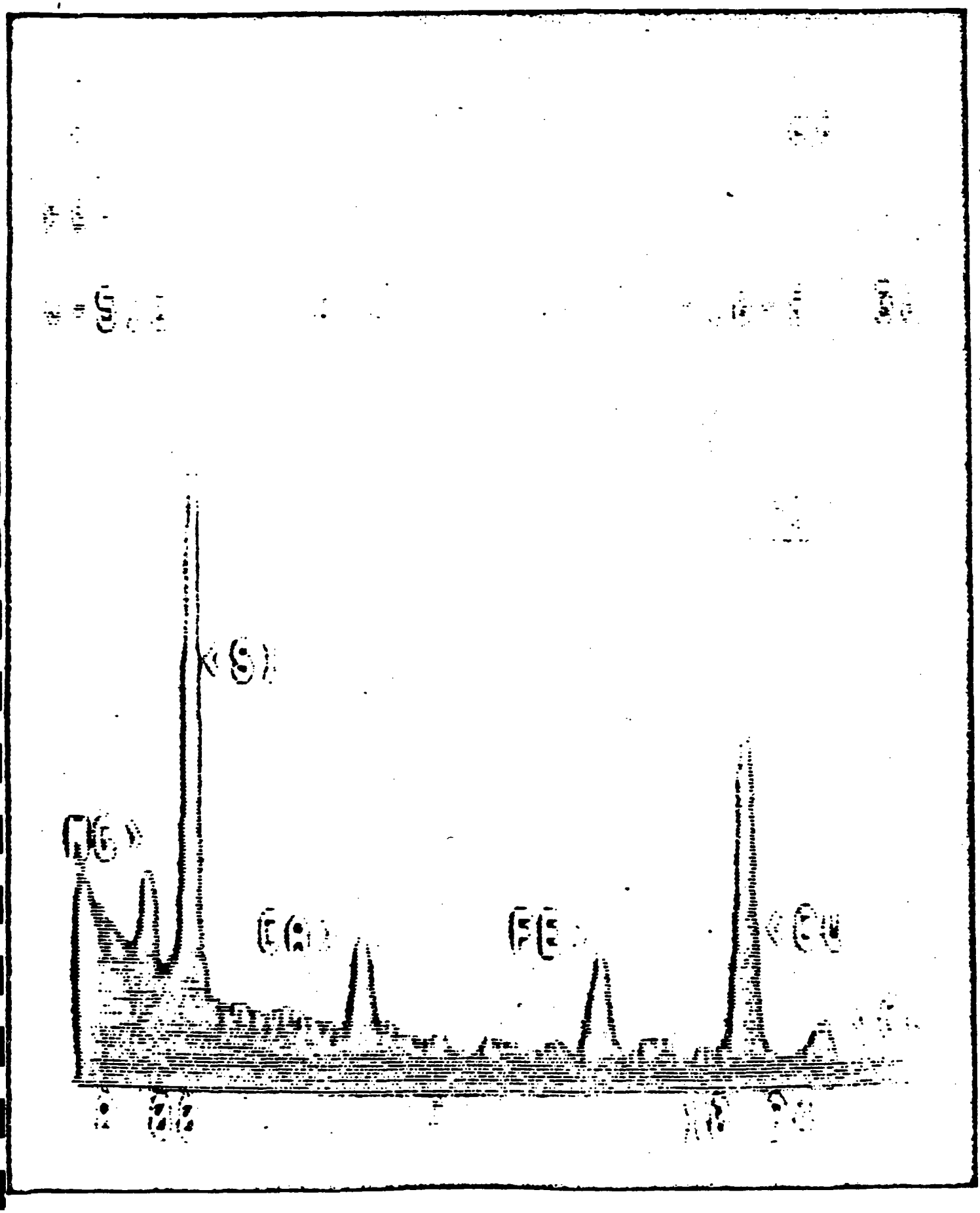

Figure 9. EDS of actinolite fiber.

ASBESTOS COUNT SHEET

	STRUCT-URES	FREE FIBERS	TOTAL FIBERS
HIGH MAG. / CT. SHEET			
LOW MAG.(optically countable fibers) / CUMULATIVE			

CUMULATIVE WINDOW NUMBER__________

TE___/___/___

ME ___:___to___:___=___:___

I__________ FN__________

L.FILTERED________

ALYST________________

SCANNING MAG.____________

SIZING MAG.____________

EFF. FILTER AREA__________MM**2

GRID HOLE AREA__________MM**2

GRID NUMBER____________

HOLE NUMBERS____________

undle Code: 1=fibril,2=bundle,3=matte,4=fibers attached to particle,
5=bundles on part.,6=matte on part.

D Code(morph,diff): 1=chrys,2=amph,3=cell,4=ambis

iber ID(XES): 1=chrys,2=cell,3=amos,4=croc,5=trem,6=other amph,7=other fiber

iber Code: 1=positive,2=tentative

ber=1 rticle NI =0	Bun-dle Code	ID Code morph	diff	Fiber ID XES	Fiber Code	Length vertical	horiz.	Width	Matte thick-ness	Obsc. yes=1 no=0
						.	.	.	.	
						.	.	.	.	
						.	.	.	.	
						.	.	.	.	
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Figure 10. Suggested count sheet format.

AIR AND INDUSTRIAL HYGIENE LABORATORY
BERKELEY,CALIFORNIA 94704

RESULTS OF
ASBESTOS IN AIR ANALYSIS

DATE OF REPORT: 13-SEPT-84
LAB NUMBER:
FIELD NUMBER: 1

GENERAL COMMENTS

THIS IS AN ANALYSIS OF AIRBORN ASBESTOS FIBERS SAMPLED OUTSIDE THE RESIDENCE IN ,CALIFORNIA.

GENERAL ANALYSIS PARAMETERS

1. AIR VOLUME FILTERED: 4.2E+03 LITERS
2. FILTER AREA: 855 SQ. MM.
3. GRID WINDOW AREA: 0.0016 SQ. MM.
4. MAGNIFICATION: 2800X
5. VERTICAL DISTORTION: 1.4

Figure 11. Sample report sheet.

DATA RESULTS FOR TOTAL NUMBER OF FIBERS WITH MASS INFORMATION

	CHRYSOTILE POSITIVE	CHRYSOTILE TOTAL	AMPHIBOLE POSITIVE	AMPHIBOLE TOTAL	TOTAL FIBERS
FIBER COUNT	119	119	0	0	119
UNIFORM DISTRIBUTION	NO	NO	YES	YES	NO
FIBER CONCENTRATION (FIBERS/M^3)	0.69E+06	0.69E+06	0.00E+00	0.00E+00	0.69E+06
95% CONFIDENCE INTERVAL FIBERS/M^3)	0.24E+06	0.24E+06	0.00E+00	0.00E+00	0.24E+06
MASS CONCENTRAION (NANOGRAMS/M^3)	9.11	9.11	0.0	0.0	9.11
%BUNDLES	7.6	7.6	0.0	0.0	7.6
%MATS	27	27	0.0	0.0	27

DATA RESULTS FOR THE NUMBER OF FREE FIBERS WITHOUT MASS INFORMATION

	CHRYSOTILE POSITIVE	CHRYSOTILE TOTAL	AMPHIBOLE POSITIVE	AMPHIBOLE TOTAL	TOTAL FIBERS
FIBER COUNT	2	2	0	0	2
UNIFORM DISTRIBUTION	YES	YES	YES	YES	YES
FIBER CONCENTRATION (FIBERS/M^3)	1.2E+04	1.2E+04	0.00E+00	0.00E+00	1.2E+04
95%CONFIDENCE INTERVAL	1.7E+04	1.7E+04	0.00E+00	0.00E+00	1.7E+04
% BUNDLES	50	50	0.0	0.0	50
% MATS	0.0	0.0	0.0	0.0	0.0

Figure 11 Continued

DATA RESULTS FOR THE NUMBER OF STRUCTURES

	CHRYSOTILE		AMPHIBOLE		TOTAL
	POSITIVE	TOTAL	POSITIVE	TOTAL	FIBERS
TRUCTURE COUNT	44	44	0.0	0.0	44
N FORM ISTRIBUTION	YES	YES	YES	YES	YES
TRUCTURE CONCENTRATION STRUCTURES/M^3)	2.5E+05	2.5E+05	0.00E+00	0.00E+00	2.5E+05
5 CONFIDENCE NTERVAL	8.9E+04	8.9E+04	0.00E+00	0.00E+00	8.9E+04

DATA RESULTS FOR THE NUMBER OF N.I.O.S.H. EQUIVALENT FIBERS

	CHRYSOTILE		AMPHIBOLE		TOTAL
	POSITIVE	TOTAL	POSITIVE	TOTAL	FIBERS
IBER COUNT	2	2	0	0	2
N FORM ISTRIBUTION	YES	YES	YES	YES	YES
IBER CONCENTRATION FIBERS/M^3)	4.8E+02	4.8E+02	0.00E+00	0.00E+00	4.8E+02
5 CONFIDENCE NTERVAL	4.8E+02	4.8E+02	0.00E+00	0.00E+00	4.8E+02
ASS CONCENTRATION NANOGRAMS/M^3)	1.4E+01	1.4E+01	0.00E+00	0.00E+00	1.4E+01

Figure 11 Continued

REPRESENTATION
OF FIBER DIMENSIONS

WIDTH (MICRONS)	0 – .27	.27 – .54	.54 – .80	.80 – 1.1	1.1 – 1.3	1.3 – 1.6	1.6 – 1.9
.58							
.50	0	0	0	0	0	0	0
.41	0	0	0	0	0	1	0
.33	0	0	0	0	0	0	0
.25	0	0	0	0	0	0	0
.17	0	0	0	0	0	0	0
.08	0	0	0	0	0	0	0
0	1	0	0	0	0	0	0

LENGTH (MICRONS)

NUMBER OF FREE FIBERS

WIDTH (MICRONS)	0 – .49	.49 – .97	.97 – 1.5	1.5 – 1.9	1.9 – 2.4	2.4 – 2.9	2.9 – 3.4
.68							
.58	0	0	0	0	0	0	0
.48	0	0	0	0	0	0	0
.39	1	0	0	1	0	0	0
.29	2	0	1	0	0	0	0
.19	1	2	0	0	0	0	0
.10	24	1	1	0	0	0	0
0	74	8	2	0	0	1	0

LENGTH (MICRONS)

TOTAL FIBERS

Figure 11 Continued

WIDTH (MICRONS)	4.8	5.1	5.4	5.6	5.9	6.2	6.4	6.7
2.3								
2.0	0	0	0	0	0	0	0	
1.7	0	0	0	0	0	1	0	
1.4	0	0	0	0	0	0	0	
1.2	0	0	0	0	0	0	0	
.87	0	0	0	0	0	0	0	
.58	0	0	0	0	0	0	0	
.30	1	0	0	0	0	0	0	

LENGTH (MICRONS)

NIOSH FIBERS

Figure 11 Continued

SOIL SAMPLE
PLM ANALYSIS METHOD USED IN RI

United States
Environmental Protection
Agency

Environmental Monitoring Systems
Laboratory
Research Triangle Park NC 27711

Research and Development

EPA-600/M4-82-020 Dec. 1982

EPA

Test Method

Interim Method for the Determination of Asbestos in Bulk Insulation Samples*

1. Polarized Light Microscopy

1.1 *Principle and Applicability*

Bulk samples of building materials taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.[1,2] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[2] and the interpretation of point count data[3] is available.

This method is applicable to all bulk samples of friable insulation materials submitted for identification and quantitation of asbestos components.

1.2 Range

The point counting method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is less than 1 percent.

1.3 Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Spray-on binder materials may coat fibers and affect color or obscure optical characteristics to the extent of masking fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Procedures that may be used for the removal of interferences are presented in Section 1.7.2.2.

1.4 Precision and Accuracy

Adequate data for measuring the accuracy and precision of the method for samples with various matrices are not currently available. Data obtained for samples containing a single asbestos type in a simple matrix are available in the EPA report *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method.*[4]

1.5 Apparatus

1.5.1 Sample Analysis

A low-power binocular microscope, preferably stereoscopic, is used to

*An interim method is carefully drafted from available source information. This method is still under investigation and therefore is subject to revision.

examine the bulk insulation sample as received.

- *Microscope:* binocular, 10-45X (approximate)
- *Light Source:* incandescent or fluorescent
- *Forceps, Dissecting Needles, and Probes*
- *Glassine Paper or Clean Glass Plate*

Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris.

- *Polarized Light Microscope:* described above
- Objective Lenses: 10X, 20X, and 40X or near equivalent
- *Dispersion Staining Objective Lens* (optional)
- *Ocular Lens:* 10X minimum
- *Eyepiece Reticle:* cross hair or 25 point Chalkley Point Array
- *Compensator Plate:* 550 milli-micron retardation

1.5.2 Sample Preparation

Sample preparation apparatus requirements will depend upon the type of insulation sample under consideration. Various physical and/or chemical means may be employed for an adequate sample assessment.

- *Ventilated Hood* or negative pressure glove box
- *Microscope Slides*
- *Coverslips*
- *Mortar and Pestle:* agate or porcelain (optional)
- *Wylie Mill* (optional)
- *Beakers & assorted glassware* (optional)
- *Centrifuge* (optional)
- *Filtration apparatus* (optional)
- *Low temperature asher* (optional)

1.6 Reagents

1.6.1 Sample Preparation

- *Distilled Water* (optional)
- *Dilute CH_3COOH:* ACS reagent grade (optional)
- *Dilute HCl:* ACS reagent grade (optional)
- *Sodium metaphosphate* $(NaPO_3)_6$ (optional)

1.6.2 Analytical Reagents

- *Refractive Index Liquids:* 1.490-1.570, 1.590-1.720 in increments of 0.002 or 0.004
- *Refractive Index Liquids for Dispersion Staining:* high-dispersion series, 1.550, 1.605, 1.630 (optional)
- *UICC Asbestos Reference Sample Set:* Available from: UICC MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan CF6 1XW, UK, and commercial distributors
- *Tremolite-asbestos* (source to be determined)
- *Actinolite-asbestos* (source to be determined)

1.7 Procedures

Note: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

1.7.1 Sampling

Samples for analysis of asbestos content shall be taken in the manner prescribed in Reference 5 and information on design of sampling and analysis programs may be found in Reference 6. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

1.7.2 Analysis

1.7.2.1 Gross Examination

Bulk samples of building materials taken for the identification and quantitation of asbestos are first examined for homogeneity at low magnification with the aid of a stereomicroscope. The core sample may be examined in its container or carefully removed from the container onto a glassine transfer paper or clean glass plate. If possible, note is made of the orientation of top and bottom surfaces. When discrete strata are identified, each is treated as a separate material so that fibers are first identified and quantified in that layer only, and then the results for each layer are combined to yield an estimate of asbestos content for the whole sample.

1.7.2.2 Sample Preparation

Bulk materials submitted for asbestos analysis involve a wide variety of matrix materials. Representative subsamples may not be readily obtainable by simple means in heterogeneous materials, and various steps may be required to alleviate the difficulties encountered. In most cases, however, the best preparation is made by using forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope.

Alternatively, attempts may be made to homogenize the sample or eliminate interferences before further characterization. The selection of appropriate procedures is dependent upon the samples encountered and personal preference. The following are presented as possible sample preparation steps.

A mortar and pestle can sometimes be used in the size reduction of soft or loosely bound materials, though this may cause matting of some samples. Such samples may be reduced in a Wiley mill. Apparatus should be clean and extreme care exercised to avoid cross-contamination of samples. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. These procedures are not recommended for samples that contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments that have aspect ratios greater than 3:1 and will be classified as asbestos fibers. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Acid treatment may occasionally be required to eliminate interferences. Calcium carbonate, gypsum, and bassanite (plaster) are frequently present in sprayed or trowelled insulations. These materials may be removed by treatment with warm dilute acetic acid. Warm dilute hydrochloric acid may also be used to remove the above materials. If acid treatment is required, wash the sample at least twice with distilled water, being careful not to lose the particulates during decanting steps. Centrifugation or filtration of the suspension will prevent significant fiber loss. The pore size of the filter should be 0.45 micron or less. *Caution: prolonged acid contact with the sample may alter the optical characteristics of chrysotile fibers and should be avoided.*

Coatings and binding materials adhering to fiber surfaces may also be removed by treatment with sodium metaphosphate.[7] Add 10 mL of 10 g/L sodium metaphosphate solution to a small (0.1 to 0.5 mL) sample of bulk material in a 15-mL glass centrifuge tube. For approximately 15 seconds each, stir the mixture on a vortex mixer, place in an ultrasonic bath and then shake by hand. Repeat the series.

Collect the dispersed solids by centrifugation at 1000 rpm for 5 minutes. Wash the sample three times by suspending in 10 mL distilled water and recentrifuging. After washing, resuspend the pellet in 5 mL distilled water, place a drop of the suspension on a microscope slide, and dry the slide at 110°C.

In samples with a large portion of cellulosic or other organic fibers, it may be useful to ash part of the sample and examine the residue. Ashing should be performed in a low temperature asher. Ashing may also be performed in a muffle furnace at temperatures of 500°C or lower. Temperatures cf 550°C or higher will cause dehydroxylation of the asbestos minerals, resulting in changes of the refractive index and other key parameters. If a muffle furnace is to be used, the furnace thermostat should be checked and calibrated to ensure that samples will not be heated at temperatures greater than 500°C.

Ashing and acid treatment of samples should not be used as standard procedures. In order to monitor possible changes in fiber characteristics, the material should be viewed microscopically before and after any sample preparation procedure. Use of these procedures on samples to be used for quantitation requires a correction for percent weight loss.

1.7.2.3 Fiber Identification

Positive identification of asbestos requires the determination of the following optical properties.

- Morphology
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 1-1 lists the above properties for commercial asbestos fibers. Figure 1-1 presents a flow diagram of the examination procedure. Natural variations in the conditions under which deposits of asbestiform minerals are formed will produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Alternatively, dispersion staining may be used. Inexperienced operators may find that the dispersion staining technique is more easily learned, and should consult Reference 9 for guidance. Central stop dispersion staining colors are presented in Table 1-2. Available high-dispersion (HD) liquids should be used.

1.7.2.4 Quantitation of Asbestos Content

Asbestos quantitation is performed by a point-counting procedure. An ocular reticle (cross-hair or point array) is used to visually superimpose a point or points on the microscope field of view. Record the number of points positioned directly above each kind of particle or fiber of interest. Score only points directly over asbestos fibers or nonasbestos matrix material. Do not score empty points for the closest particle. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories. Point counting provides a determination of the area percent asbestos. Reliable conversion of area percent to percent of dry weight is not currently feasible unless the specific gravities and relative volumes of the materials are known.

For the purpose of this method, "asbestos fibers" are defined as having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 1-1.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forcep samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

- A cross-hair reticle and mechanical stage; or
- A reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the plane polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X or at the lowest magnification of the polarized light microscope that can effectively distinguish the sample components. Confirmation of the quantitation result by a second analyst on some percentage of analyzed samples should be used as standard quality control procedure.

The percent asbestos is calculated as follows:

$$\% \text{ asbestos} = (a/n)\ 100\%$$

where

a = number of asbestos counts,
n = number of nonempty points counted (400).

If a = 0, report "No asbestos detected." If $0 < a \leq 3$, report "<1% asbestos."

The value reported should be rounded to the nearest percent.

1.8 References

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9. Walter C. McCrone, *Asbestos Particle Atlas*, Ann Arbor: Ann Arbor Science Publishers, June 1980.

Table 1-1. Optical properties of asbestos fibers

Mineral	Morphology, color[1]	Refractive indices[2] α	γ	Birefringence	Extinction	Sign of elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1 Colorless[3], nonpleochroic.	1.493-1.560	1.517-1.562[6] (normally 1.556)	.002-.014	‖to fiber length	+ (length slow)
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635-1.696	1.655-1.729[6] (normally 1.696-1.710)	.020-.033	‖to fiber length	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654-1.701	1.668-1.717[5] (normally close to 1.700)	.014-.016	‖to fiber length	− (length fast)
Anthophyllite-asbestos	Straight, single fibers, some larger composite fibers. Anthrophyllite cleavage fragments may be present with aspect ratios < 10:1.[4] Colorless to light brown.	1.596-1.652	1.615-1676[6]	.019-.024	‖to fiber length	+ (length slow)
Tremolite-actinolite-asbestos	Tremolite-asbestos may be present as single or composite fibers. Tremolite cleavage fragments may be present as single crystals with aspect ratios < 10:1.[4] Colorless to pale green.	1.599-1.668	1.622-1.688[6]	.023-.020	Oblique extinction, 10-20° for fragments. Composite fibers show ‖extinction.	+ (length slow)

[1] From reference 5; colors cited are seen by observation with plane polarized light.
[2] From references 5 and 8.
[3] Fibers subjected to heating may be brownish.
[4] Fibers defined as having aspect ratio >3:1.
[5] ⊥ to fiber length.
[6] ‖ to fiber length.

Table 1-2. Central stop dispersion staining colors [a]

Mineral	RI Liquid	⊥	‖
Chrysotile	1.550[HD]	Blue	Blue-magenta
"Amosite"	1.680	Blue-magenta to pale blue	Golden-yellow
	1.550[HD]	Yellow to white	Yellow to white
Crocidolite[b]	1.700	Red magenta	Blue-magenta
	1.550[HD]	Yellow to white	Yellow to white
Anthophyllite-asbestos	1.605[HD]	Blue	Gold to gold-magenta
Tremolite-asbestos	1.605[HDc]	Pale blue	Yellow
Actinolite-asbestos	1.605[HD]	Gold-magenta to blue	Gold
	1.630[HDc]	Magenta	Golden-yellow

[a] From reference 9, colors may vary slightly.
[b] Blue absorption color.
[c] Oblique extinction view.

Polarized light microscopy qualitative analysis: For each type of material identified by examination of sample at low magnification. Mount spacially dispersed sample in 1.550 RI liquid. (If using dispersion staining, mount in 1.550 HD.) View at 100X with both plane polarized light and crossed polars. More than one fiber type may be present.

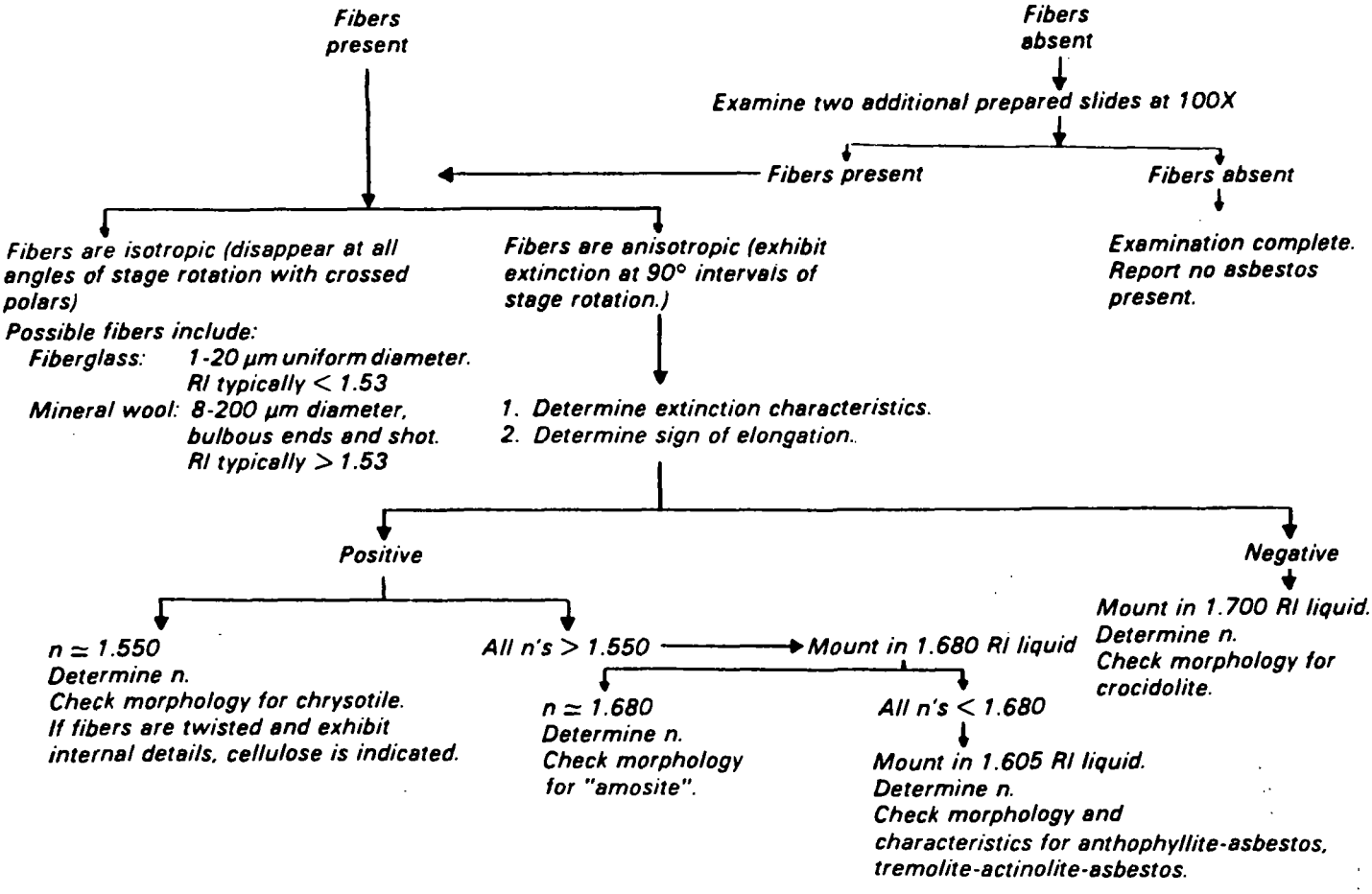

Figure 1-1. Flow chart for qualitative analysis of bulk samples by polarized light microscopy.

2. X-Ray Powder Diffraction

2.1 Principle and Applicability

The principle of X-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid, crystalline material will diffract an impingent beam of parallel, monochromatic X-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the X-ray wavelength, Å;
d = the interplanar spacings of the set of reflecting lattice planes, Å; and
θ = the angle of incidence between the X-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident X-ray beam, a diffraction pattern can be generated that, in most cases, will be uniquely characteristic of both the chemical composition and structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1). However, when used in conjunction with optical methods such as polarized light microscopy (PLM), XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For *qualitative analysis* by XRD methods, samples are initially scanned over limited diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-2). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5-10 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals are submitted to a full (5°-60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns are compared with standard reference powder diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Accurate *quantitative analysis* of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis is selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] is recommended in which, subsequent to comminution of the bulk material to ~10 μm by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods,[7,8] may be used for *semiquantitative analysis*.

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.2 Range and Sensitivity

The range of the method has not been determined.

The sensitivity of the method has not been determined. It will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected, and their relative intensities.

2.3 Limitations

2.3.1 Interferences

Since the fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally occurring materials the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials the nature of the interferences may vary greatly (Table 2-3) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-4 and include the following:

- *Chlorite* has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.36 Å) and secondary (3.66 Å) peaks for chrysotile. Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.
- *Halloysite* has a peak at 3.63 Å that interferes with the secondary (3.66 Å) peak for chrysotile.
- *Kaolinite* has a major peak at 7.15 Å that may interfere with the primary peak of chrysotile at 7.36 Å when present at concentrations of >10 percent. However, the secondary chrysotile peak at 3.66 Å may be used for quantitation.
- *Gypsum* has a major peak at 7.5 Å that overlaps the 7.36 Å peak of chrysotile when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris.
- *Cellulose* has a broad peak that partially overlaps the secondary (3.66 Å) chrysotile peak.[8]
- Overlap of major diagnostic peaks of the amphibole asbestos minerals, amosite, anthophyllite, crocidolite, and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the X-ray port.
- *Carbonates* may also interfere with quantitative analysis of the amphibole asbestos minerals, amosite,

anthophyllite, crocidolite, and tremolite. Calcium carbonate ($CaCO_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, if present, chrysotile may be partially dissolved by this treatment.[11]

- A major *talc* peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of crocidolite (3.10 Å), amosite (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interferences is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, with the result being that their diffraction patterns are characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of the orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.3.2 Matrix Effects

If a copper X-ray source is used, the presence of iron at high concentrations in a sample will result in significant X-ray fluorescence, leading to loss of peak intensity with increased background intensity and an overall decrease in sensitivity. This situation may be corrected by use of an X-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5,13,14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7,8] or filter substrate (Ag) peak.[5,6]

2.3.3 Particle Size Dependence

Because the intensity of diffracted X-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 µm.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7,8,15] For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.[12,14] However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

2.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. NIOSH has recently directed a major research effort toward the preparation and characterization of analytical reference materials, including asbestos standards;[16,17] however, these are not available in large quantities for routine analysis.

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be underestimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4 Precision and Accuracy

Precision of the method has not been determined.

Accuracy of the method has not been determined.

2.5 Apparatus

2.5.1 Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- *Mortar and Pestle:* Agate or porcelain
- *Razor Blades*
- *Sample Mill:* SPEX, Inc., freezer mill or equivalent
- *Bulk Sample Holders*
- *Silver Membrane Filters:* 25-mm diameter, 0.45-µm pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006
- *Microscope Slides*
- *Vacuum Filtration Apparatus:* Gelman No. 1107 or equivalent, and side-arm vacuum flask
- *Microbalance*
- *Ultrasonic Bath or Probe:* Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent
- *Volumetric Flasks:* 1-L volume
- *Assorted Pipet*
- *Pipet Bulb*
- *Nonserrated Forceps*
- *Polyethylene Wash Bottle*
- *Pyrex Beakers:* 50-mL volume
- *Desiccator*
- *Filter Storage Cassettes*
- *Magnetic Stirring Plate and Bars*
- *Porcelain Crucibles*
- *Muffle Furnace or Low Temprature Asher*

2.5.2 Sample Analysis

Sample analysis requirements include an X-ray diffraction unit, equipped with:

- *Constant Potential Generator; Voltage and mA Stabilizers*
- *Automated Diffractometer with Step-Scanning Mode*
- *Copper Target X-Ray Tube:* High intensity; fine focus, preferably
- *X-Ray Pulse Height Selector*
- *X-Ray Detector* (with high voltage power supply): Scintillation or proportional counter

- *Focusing Graphite Crystal Monochromator;* or *Nickel Filter* (if copper source is used, and iron fluorescence is not a serious problem)
- *Data Output Accessories:*
  Strip Chart Recorder
  Decade Scaler/Timer
  Digital Printer
- *Sample Spinner* (optional)
- *Instrument Calibration Reference Specimen:* α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent

2.6 Reagents

2.6.1 Standard Reference Materials

The reference materials listed below are intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- *Chrysotile:* UICC Canadian, or NIEHS Plastibest. (UICC reference materials available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK)
- *Crocidolite:* UICC
- *"Amosite":* UICC
- *Anthophyllite-Asbestos:* UICC
- *Tremolite Asbestos:* Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 90064 (washed with dilute HCl to remove small amount of calcite impurity); Indian tremolite, Rajasthan State, India.
- *Actinolite Asbestos:* (Source to be determined).

2.6.2 Adhesive

Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

2.6.3 Surfactant

1 Percent aerosol OT aqueous solution or equivalent.

2.6.4 Isopropanol

ACS Reagent Grade.

2.7 Procedure

2.7.1 Sampling

Samples for analysis of asbestos content shall be collected as specified in EPA Guidance Document #C0090, *Asbestos-Containing Materials in School Buildings.*[10]

2.7.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD should be used as an auxiliary method when a second, independent analysis is requested.

Note: Asbestos is a toxic substance. All handling of dry materials should be performed in an operating fume hood.

2.7.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin layer or bulk).

Bulk materials are usually received as inhomogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.7.2.2 and 2.7.2.3. *Note: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.*

2.7.2.1.1 Milling—Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[19-22] Therefore, all milling times should be kept to a minimum.

For *qualitative analysis*, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other suitable wetting agent, to minimize exposures.

For accurate, reproducible *quantitative analysis*, the particle size of both sample and standard materials should be reduced to ~10 μm (see Section 2.3.3). Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill, or equivalent) for a maximum time of 10 min is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to <10 μm.[6,16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.7.2.1.2. Low temperature ashing—For materials shown by PLM to contain large amounts of gypsum, cellulose, or other organic materials, it may be desirable to ash the samples prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,[23,24] ashing temperatures should be kept below 500°C. Use of a low temperature asher is recommended. In all cases, calibration of the oven is essential to ensure that a maximum ashing temperature of 500°C is not exceeded.

2.7.2.1.3 Acid leaching—Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.3.1), it may be necessary to remove these interferents by a simple acid leaching procedure prior to analysis (see Section 1.7.2.2).

2.7.2.2 Qualitative Analysis

2.7.2.2.1 Initial screening of bulk material—Qualitative analysis should be performed on a representative, homogeneous portion of the sample with a minimum of sample treatment using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.7.2.1.1) to a final particle size sufficiently small (~100 μm) to allow adequate packing into the sample holder.
2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. (Note: Use of a back-packing method[25] for bulk sample

preparation may reduce preferred orientation effects.)

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-2). The X-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.
4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°-60° 2θ; 1° 2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.
5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data [3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-2; common constituents of bulk insulation and wall materials are listed in Table 2-3.

2.7.2.2.2 Detection of minor or trace constituents—Routine screening of bulk materials by XRD may fail to detect small concentrations (<5 percent) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances where confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.7.2.2.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.7.2.3) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-2). Accurate transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.7.2.3 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[5] A thick-layer or bulk method involving pelletizing the sample may be used for semiquantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.7.2.1.1.
2. Dry at 100°C for 2 hr; cool in a desiccator.
3. Weigh accurately to the nearest 0.01 mg.
4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.7.2.1.2 and 2.7.2.1.3. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss, L.
5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask with approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.
6. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the sample material.
7. Dilute to volume with isopropanol.
8. Place flask on a magnetic stirring plate. Stir.
9. Place a silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)
10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.
11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-2).
12. Analyze the selected diagnostic reflection(s) by step scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a, is the difference between the peak integrated count and the total background count.
13. Determine the net count, I_{A_g}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, $I^{\circ}_{A_g}$. Scan times may be *less* for measurement of silver peaks than for sample peaks; however, they should be *constant* throughout the analysis.
14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net

count, I_r^o, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I_r^o:

$$\bar{I}_a = \frac{I_a}{I_r^o},\ \bar{I}_{Ag} = \frac{I_{Ag}}{I_r^o},\ \text{and}\ \bar{I}_{Ag}^o = \frac{I_{Ag}^o}{I_r^o}$$

2.8 Calibration

2.8.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.7.2.1.1. *Equivalent, standardized matrix reduction and sizing techniques should be used for both standard and sample materials.*
2. Dry at 100°C for 2 hr; cool in a desiccator.
3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Quantitatively transfer each to a 1-L volumetric flask with approximately 200 mL isopropanol to which a few drops of surfactant have been added.
4. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the asbestos material.
5. Dilute to volume with isopropanol.
6. Place the flask on a magnetic stirring plate. Stir.
7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.8.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.7.2.3, step 12, and the same conditions as those used for the samples.
2. Determine the normalized intensity for each peak measured, $\bar{I}_{std}^o$, as outlined in Section 2.7.2.3, step 14.

2.9 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\bar{I}_{std}^o$ on w. Poor reproducibility (±15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\bar{I}_{std}^o$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\bar{I}_{Ag}$, for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak, $\bar{I}_{Ag}^o$, of the sample filter, calculate the transmittance, T, for each sample as follows:[26,27]

$$T = \frac{\bar{I}_{Ag}}{\bar{I}_{Ag}^o}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R\,(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a},$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and
θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the appropriate calibration data and absorption corrections:

$$W_a = \frac{\bar{I}_a\, f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a\,(1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;
W_a = mass of asbestos mineral on filter, in μg;
W_T = total sample weight on filter, in μg;
L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.7.2.3).

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Table 2-1. *The asbestos minerals and their nonasbestiform analogs.*

Asbestiform	*Nonasbestiform*
Serpentine	
Chrysotile	*Antigorite, lizardite*
Amphibole	
Anthophyllite asbestos	*Anthophyllite*
Cummingtonite-grunerite asbestos ("Amosite")	*Cummingtonite-grunerite*
Crocidolite	*Riebeckite*
Tremolite asbestos	*Tremolite*
Actinolite asbestos	*Actinolite*

Table 2-2. *Principal lattice spacings of asbestiform minerals.*[a]

Minerals	*Principal d-spacings (Å) and relative intensities*			*JCPDS Powder diffraction file*[c] *number*
Chrysotile	7.37_{100}	3.65_{70}	4.57_{50}	*21-543*[c]
	7.36_{100}	3.66_{80}	2.45_{65}	*25-645*
	7.10_{100}	2.33_{80}	3.55_{70}	*22-1162 (theoretical)*
"Amosite"	8.33_{100}	3.06_{70}	2.756_{70}	*17-745 (nonfibrous)*
	8.22_{100}	3.060_{85}	3.25_{70}	*27-1170 (UICC)*
Anthophyllite	3.05_{100}	3.24_{60}	8.26_{55}	*9-455*
	3.06_{100}	8.33_{70}	3.23_{50}	*16-401 (synthetic)*
Actinolite	2.72_{100}	2.54_{100}	3.40_{80}	*25-157*
Crocidolite	8.35_{100}	3.10_{55}	2.720_{35}	*27-1415 (UICC)*
Tremolite	8.38_{100}	3.12_{100}	2.705_{90}	*13-437*[c]
	2.706_{100}	3.14_{95}	8.43_{40}	*20-1310*[c] *(synthetic)*
	3.13_{100}	2.706_{60}	8.44_{40}	*23-666 (synthetic mixture with richterite)*

[a] *This information is intended as a guide, only. Complete powder diffraction data, including mineral type and source, should be referred to, to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite, and chrysotile are available from the U.S Bureau of Mines,* Reference 4.
[b] *From Reference 3.*
[c] *Fibrosity questionable.*

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Table 2-3. Common constituents in insulation and wall materials (from Ref. 10)

A. Insulation materials	B. Spray finishes or paints
Chrysotile	Bassanite
"Amosite"	Carbonate minerals (calcite, dolomite, vaterite)
Crocidolite	Talc
*Rock wool	Tremolite
*Slag wool	Anthophyllite
*Fiber glass	Serpentine (including chrysotile)
Gypsum ($CaSO_4 \cdot 2H_2O$)	"Amosite"
Vermiculite (micas)	Crocidolite
*Perlite	*Mineral wool
Clays (kaolin)	*Rock wool
*Wood pulp	*Slag wool
*Paper fibers (talc, clay, carbonate fillers)	*Fiber glass
Calcium silicates (synthetic)	Clays (kaolin)
Opaques (chromite, magnetite inclusions in serpentine)	Micas
Hematite (inclusions in "amosite")	Chlorite
Magnesite	Gypsum ($CaSO_4 \cdot 2H_2O$)
*Diatomaceous earth	Quartz
	*Organic binders and thickeners
	Hydromagnesite
	Wollastonite
	Opaques (chromite, magnetite inclusions in serpentine)
	Hematite (inclusions in "amosite")

*Amorphous materials--contribute only to overall scattered ' radiation and increased background radiation.

Table 2-4. Interferences in XRD analysis of asbestiform minerals

Asbestiform mineral	Primary diagnostic peaks (approximate d-spacings in Å)	Interference
Serpentine		
Chrysotile	7.4	Nonasbestiform serpentines (antigorite, lizardite) Chlorite Kaolinite Gypsum
	3.7	Nonasbestiform serpentines, (antigorite, lizardite) Chlorite Halloysite Cellulose
Amphibole "Amosite" Anthophyllite Crocidolite Tremolite	3.1	Nonasbestiform amphiboles (cummingtonite-grunerite, anthophyllite, riebeckite, tremolite) Mutual interferences Carbonates Talc
	8.3	Nonasbestiform amphiboles (cummingtonite, grunerite, anthophyllite, reibeckite, tremolite) Mutual interferences

United States
Environmental Protection
Agency

Center for Environmental Research
Information
Cincinnati OH 45268

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Official Business
Penalty for Private Use $300

SOIL SAMPLE
TEM ANALYSIS METHOD USED IN RI

METHODOLOGY FOR THE ANALYSIS OF ASBESTOS IN SOIL BY TRANSMISSION ELECTRON MICROSCOPY

PREPARED BY

Steven B. Hayward
Research Specialist

and

Nora P. Lowe
Laboratory Technician

Air and Industrial Hygiene Laboratory
California Department of Health Services
Berkeley, California 94704-9980

AIHL METHOD (Draft)

Limited Publication
Not for Publication

ASBESTOS IN SOIL

Analyte: Asbestos (weight percent in soil, sediment, or sludge)

Range:

Application: Soil, sediment, and sludge contamination including hazardous waste sites.

Precision:

Matrix: Soil, sediment, sludge

Procedure: Transmission Electron Microscopy (TEM) with Energy Dispersive X-ray Spectrometry (EDS)

Date Issued:

Collaboratively Tested: No.

1. INTRODUCTION.

1.1.

1.2.

1.2.1. Detection Limit of Overall Procedure.

1.2.2. Reliable Quantitation Limit.

1.2.3. Accuracy and Precision.

1.3. GENERAL COMMENTS.

1.3.1. Sample preparation time for a single sample is estimated to be approximately eight hours. However, when groups of samples are prepared simultaneously, this time is reduced. Estimated EM times of eight and four hours for windows and field of view analyses respectively appear to be reasonable. Additional costs are incurred on analyzing count data and reporting of results.

1.3.2. EM analysis for asbestos is tedious work, and places great demands on the microscopist. As a result, the microscopist should be accorded latitude in the scheduling of breaks to coincide with natural stopping places in the analysis (such as finishing a grid opening or a field of view). Lack of consideration of operator discomfort or fatigue will result in less reliable analyses.

1.3.3. This method has been developed for analysis of asbestos in soil, sediment or sludge. It is imperative that the sample be ground sufficiently to produce individual fibrils or small fiber bundles that will be uniformly distributed when filtered through a 0.1 micron polycarbonate membrane filter.

2. LABORATORY REQUIREMENTS.

2.1. Apparatus.

2.1.1. MICROBALANCE accurate to a few micrograms should be located in a temperature and humidity controlled chamber for gravimetric analysis.

2.1.2. INFRARED LAMP for rapid evaporation of water and alcohol from the sample.

2.1.3. AUTOMATIC MORTAR GRINDER for wet comminution and homogenation of the sample.

2.1.4. EM PREPARATION ROOM containing a laminar flow class 100 clean bench to minimize contamination during sample preparation.

2.1.5. ULTRASONIC BATH necessary for dispersion of particles and fibers throughout the solution and for general cleaning.

2.1.6. VACUUM TYPE MEMBRANE FILTERING APPARATUS to filter the sample suspensions. Includes: a vacuum pump or "house" vacuum capable of providing up to 20 inches of mercury negative pressure; a filtering assembly consisting of a 1-liter side-arm flask, "Millipore" coarse-frit glass support with funnel and clamp for sample collection on a 0.1 micron pore size polycarbonate membrane filter with a 0.45 micron pore size cellulose acetate backing filter.

2.1.7. VACUUM EVAPORATOR equipped with a rotating turntable necessary to coat the polycarbonate membrane filter containing the sample with a thin layer of spectrochemically pure carbon.

2.1.8. MODIFIED JAFFE-WICK WASHER required for dissolving polycarbonate membrane filters (see references Samudra; Yamate).

2.1.9. TEM (TRANSMISSION ELECTRON MICROSCOPE) with energy dispersive X-ray spectrometer (EDS) is needed for analysis. It is desirable for the microscope to have a scanning attachment for secondary electron imaging (SEI) do that particle surfaces can be examined. It should be capable of obtaining "microbeam" diffraction patterns from the same size area. For fiber length and width measurements, the microscope should be equipped with a fluorescent viewing screen which is inscribed with graduated circles of known radii, or the eyepiece of the binoculars should contain a reticle with a square grid area, such as a micrometer net reticle subdivided into 100 squares.

2.2. Reagents.

2.2.1. 95 percent ETHYL ALCOHOL used to facilitate grinding and prevent the fibers from becoming airborne. Also used for cleaning.

2.2.2. ULTRAFILTERED DISTILLED WATER (XX dH2O) distilled water that has been filtered through a 0.1 micron filter to be used as a carrying fluid for the ground sample and for rinsing implements.

2.2.3. 0.1 percent AEROSOL O.T. SOLUTION that has been filtered through a 0.1 pore size filter used to aid dispersion of particles and fibers throughout the sample suspension.

2.2.4. CHLOROFORM, the clearing agent for polycarbonate membrane filters.

2.3. Miscellaneous.

2.3.1. Weighting paper, various spatulas, pyrex glassware, magnetic stirrer and stirbars, iron mortar and pestle, an accurate pipettor with tips for 1 milliliter aliquots, 400 mesh copper grids, various forceps, scalpels, and various storage items (transparent tape, plastic petri dishes, vials, labels, etc.).

3. ANALYTICAL PROCEDURE.

3.1. Sample Preparation.

3.1.1. GRINDING.

Manually grind an aliquot if the sample wetted with 95 percent ethyl alcohol into small enough fragments for comminution by an automatic mortar grinder. Make sure the sample remains wet with alcohol to facilitate grinding and prevent the fibers from becoming airborne. Transfer the sample to an automatic mortar grinder to be ground for 6 hours. Periodically check the slurry to be certain that a sufficient quantity of alcohol is always present. The ground

soil/alcohol mixture should then be transferred to a pyrex container. Dry the sample by radiant heat in a fume hood.

3.1.2. WEIGH AND SONICATE.

A weighed aliquot (approximately 0.01 grams) of the ground, dried soil is placed into a labeled 100 milliliter graduated cylinder or similar, Q.S. to volume with XX dH2O and add 1 milliliter of 0.1 percent aerosol O.T. solution. Mix thoroughly. Sonicate for 1 hour at maximum intensity in an ultrasonic bath.

3.1.3. FILTER.

Position a "Millipore" coarse-frit glass support upon a 1-liter side-arm flask. Wet with XXdH2O and vacuum through. Using XXdH2O rinsed forceps, place a 0.45 micron cellulose acetate filter onto the support. Allow the filter to become wet by adding more XXdH2O if necessary, then vacuum through. A 0.1 micron polycarbonate membrane filter (shiny side up) is stacked on top of the 0.45 micron filter. Complete assembly if the filtering apparatus by positioning and clamping the filter funnel. Without vacuum, pour about 25 milliliters of XXdH2O into the funnel to pretreat the filters and allow to stand a few minutes. Mix the sample thoroughly. Transfer a 1 milliliter aliquot of the solution into the prepared filter funnel. To insure an even distribution of the soil particles in the filter, move the pipette tip around while dispensing. Turn on the vacuum and filter the entire volume.

3.1.4. CARBON COAT.

Remove the membrane filter, with the sample side up, to the bottom half of a 100 millimeter plastic petri dish and tape down along the edged. Coat with carbon in a vacuum evaporator as soon as possible. Place the sample onto the turntable in the vacuum evaporator, evacuate the chamber, and deposit an approximately 40 nanometer thickness of carbon, while rotating the turntable (according to manufacturer's instructions). The evaporation should be carried out in short bursts to prevent overheating and consequent degredation of the filter material. Such degredation makes dissolution of the filter virtually impossible. Slowly return the chamber to atmospheric pressure. Remove the sample, cover with the petri dish lid, and store on the clean bench until ready to prepare the grids.

3.1.5. GRID PREPARATION.

The particles on the filter surface along with the embedding carbon coating are then transferred to 400 mesh copper electron microscope grids in the clean air hood and "cleared" by means of the Modified Jaffe-Wick Washer. Four sections are cut from widely separate portions of the filter so that an assessment can be made of the uniformity of the filter deposit. Complete dissolution of the membrane requires 72 hours at temperatures above 68 degrees Farenheit (longer at lower temperatures). After clearing, the grids are stored either in an EM grid storage box or a clean 50 millimeter plastic petri dish on clean , labeled filter paper. The grid locations should be noted in a log book.

3.2. TEM Analysis.

3.2.1. SUGGESTED INSTRUMENT SETTINGS.

3.2.1.1. Accelerating voltage: 100 KV

3.2.1.2. Magnification:

200 - 1,000X for assessing the quality of the grid and particulate loading, as well as for selection of grid openings for analysis.

20,000 - 50,000X (total with or without binocular view) for detailed analysis.

3.2.1.3. Brightness: Beam brightness should be adjusted during scanning procedure such that features are just clearly visible. High brightness is undesirable for two reasons. First, brightness (electron flux) should be minimized to prevent radiation damage to chrysotile fibers, and second, viewing a high brightness screen reduces the operator dark adaptation, compromising the ability to see weak electron diffraction patterns.

3.2.1.4. Diffraction: If possible, microbeam diffraction should be used rather than selected area diffraction. Microbeam diffraction is carried out by exciting all lenses below the specimen in the same way as for selected area diffraction. The condenser lens system is

adjusted so that a narrow, coherent beam is incident on the sample. This can be accomplished by highly exciting the first condenser lens, and using a small (10 - 60 micron) second condenser apperture. The extent of the beam determines the diffracting area, and can be made as small as a fibril diameter if desired. An image can be viewed in this mode by expanding the beam slightly by underexciting the second condenser lens, and slightly overexciting the intermediate condenser (diffraction)lens. Once the beam is centered on the desired fiber, the second condenser lens can be used to converge the beam onto the fiber. The diffraction pattern will then be visible. If selected area diffraction must be used, care must be taken to adjust the objective lens current for proper focus. Microbeam diffraction is preferred over selected area diffraction because the the focus is not critical for visibility of the pattern. Furthermore, contamination of the pattern with spots from closely adjacent material can be prevented by careful location of the beam.

3.2.2. MAGNIFICATION CALIBRATION.

The magnification at the fluorescent screen must be determined by calibration with a diffraction grating replica in the specimen holder. If sizing of the fibers is carried out using binocular viewing (to facilitate diffraction pattern viewing), the calibration should be carried out (with the fluorescent screen tilted) in both horizontal and vertical directions because of vertical stretching due to the screen tilt. In this case, fiber lengths should be measured in both vertical and horizontal components, the magnification corrected separately on the two components, and the

two components combined by the Pythagorean Theorem to yield the total length. Measured widths should also be corrected based on the angle of the fiber from the vertical 0 and the two magnifications Mh and Mv according to the formula:

$$Wt = \sqrt{W^2 \left(\frac{\cos^2 0}{Mv^2} + \frac{\sin^2 0}{Mh^2} \right)}$$

Where Wt is the true fiber width, and W is the apparent width on the screen.

3.2.3. LOW-MAGNIFICATION sample assessment.

At a magnification of 200 - 1,000X , assess the quality of the carbon film, the clearing of the filter, and the uniformity of particle loading. Obviously contaminated, non-uniform grids, or grids with many broken or folded windows should be rejected.

3.2.4. HIGH-MAGNIFICATION analysis.

At a magnification of 20,000 - 50,000X define a "gate" through which the sample will be viewed and analyzed. This can be the rectangular central portion of the fluorescent screen or if binocular viewing is used, the area will be much smaller, and may be defined by an enclosed figure scribed on a reticule. A high magnification scan of the grid should be used to determine whether the "field of view"

method or full grid "window" method shall be instituted for sample analysis. This determination is based on the average number of fibers, with an aspect ratio greater than 3:1, observed per window. As a general guideline, when scanning shows less than 20 fibers per grid window the grid is either rejected and the sample reprepped at a lower concentration level or analyzed at a higher magnification depending on the loading level.

3.2.4.1. Selection of the grid opening or field of view should be on a semi-random basis, that is to say, several widely dispersed areas of the grid should first be systematically selected, and then one opening or field of view should be selected within each area. This helps ensure detection of uneven distributions across large areas of the grid.

3.2.4.2. The size of the selected area must be accurately measurable. In the case of the "window" method, the average grid opening for a batch of grids should be determined by low magnification micrography of several openings. For this measurement, light, scanning electron, or transmission electron microscopy can be used, but the measurement must be done with an instrument for which the magnification used has been independently calibrated. Account should be taken of the rounding of the corners of the openings. Finally, during actual asbestos analysis, only grid openings of the standard shape (square with round corners) should be selected for detailed analysis. The analyst should be aware of selecting openings of irregular shape due to gross grid imperfections.

If the "field of view" method is used, the dimensions of the defined "gate" are determined by means of a mag-calibration grating replica and are used to calculate the area.

3.2.4.3. Fiber identification. When a suspected asbestos-containing particle is detected, scanning is stopped. The presence of asbestos is confirmed by electron diffraction (See 3.2.4.4. below). If the possible asbestos is suspected chrysotile for which diffraction gives an ambiguous result, energy dispersive X-ray analysis should be employed for confirmation (See 3.2.4.5.)

3.2.4.4. Electron diffraction. Electron diffraction is used for positive identification of chrysotile asbestos. Once chrysotile has been tentatively identified by morphology (tubular appearance, diameter approximately 35 to 50 nanometers), a diffraction pattern should be obtained. For training purposes, it is useful to have micrographs of typical electron diffraction patterns of both helical and right-cylindrical clino- and ortho-chrysotile (See References 4.9 and 4.10 and Figures 3 and 4). For positive identification, the following characteristics of the diffraction pattern should be observed:

1) The pattern should be streaky in appearance, with mirror-symmetric layer lines perpendicular to the fiber axis at the (silicate) spacing of 1/5.32 Å. (See Figure 3).

2) Odd-numbered layer lines should contain streaks (hko reflections, h, k, odd) whose intensity varies with radius as cylindrical Bessel functions symetrically placed about

the line parallel to the fiber axis. There should be no diffraction intensity at this center point, since this would be h00, and k must be odd. For helical polytypes, these streaks are split into two streaks above and below the layer lines. See Figure 4). The vertical splitting increases with increasing k.

3) The zero layer line contains spots at a spacing of 1/7.3 A (001 reflections) together with streaks corresponding to the same lattice as is seen on the odd layer lines (0k0 reflections with k even) at a spacing of a 1/4.6 A. For helical fibers, the 0k0 reflections will also be split.

4) The even-numbered layer lines contain either pairs of split spots for the clino-chrysotile polytype (h01 and h01, h = even, l = odd) or single spots spaced 1/14.6 A apart for ortho-chrysotile (h01, h = even). The central spots on the second layer line are often missing, due to slight tilt of the fiber axis from perpendicular to the electron beam.

3.2.4.5. Energy Dispersive X-ray Spectroscopy.

Although suspected amphiboles should be checked for morphology (long, thin, straight-sided crystals) and electron diffraction, neither of these criteria is sufficient for positive identification of amphiboles. Ideally, double-tilting should be done to obtain two or three zone-axis diffraction patterns (reference 10), but the skill and/or training necessary for such an analysis can be

prohibitive, even when a double tilt stage is available. Instead, the present method accepts the use of energy dispersive X-ray spectroscopy (EDS) for amphibole identification as well as for identification of chrysotile for which no distinct diffraction pattern can be discerned. The microscope manufacturer's instructions for obtaining spectra should be followed, and the spectra should be compared to library spectra obtained from standard preparations (See Figures 5, 6, 7, 8, and 9 and Reference 4.6). Care should be taken to obtain spectra from other particulate matter if possible, to avoid the superposition of interferring spectra.

3.2.4.6. Fiber Nomenclature.

Asbestos fibers can be observed in various states of aggregation; either as single fibers, bundles (two or more fibrils in a parallel arrangement, with fibrils closer together than one fiber thickness), and mats (two or more fibers in a random arrangement, but which are separated from at least one other fiber in the mat by less than a fiber diameter somewhere along their length). Fibers, bundles, and mats may also be observed attached to non-asbestos particles.

diff. from Yamate

3.2.4.7. Particle sizing.

Fiber lengths and widths are measured and recorded. If measurement is done through binoculars equipped with a micrometer net reticle, the vertical and horizontal components of length are measured. Confirmed asbestos structures are categorized as individual fibers whenever possible. Each fiber within a bundle or mat is sized

separately but may be counted as a single fiber. In cases where the fibrils within a bundle are tightly or mats very dense, the dimensions of the entire structure may be estimated and flagged on the count sheet. For fibers of which part of the length is obscured by a particle, lengths should be increased from that actually observed according to the following rules:

1) If the obscuring particle is large enough for the actual length of the fiber to be twice that observed, record a value equal to twice the observed length.

2) If the obscuring particle is large enough for the actual length of the fiber to be obscured, record a value equal to the length of the fiber plus the size of the particle.

3) Lengths estimated in this way should be flagged on the count sheet.

3.2.4.8. Filter area to be analyzed.

Enough grid "windows" or "fields of view" should be analyzed for detection of at least 160 asbestos fibers. However, no greater than forty 400 mesh or ten 200 mesh grid windows should be analyzed. The analysis should be distributed evenly among the four grids.

3.2.4.9. Counting rules.

1) "Window" method. At a total magnification of 20,000 - 50,000X, a series of parallel traverses should be made across the grid opening. Starting at one corner, traverse the opening by moving it through the "gate" already established. Movement through the gate is stop-and-go, rather than continuous, to allow the operator time to visually inspect each area of the opening as it enters the gate. At the end of each traverse, the sample is moved one gate width, and a parallel traverse is made in the opposite direction. This process is continued until the entire grid opening has been scanned. Fiber measurements and identification are recorded on a count sheet.

2) "Field of View" method. The area within the established "gate" is to exclusively be considered for analysis. Only fibers or portions of fibers identified as asbestos, lying within the "gate" boundaries are to be measured and counted.

3.3. Data Analysis.

3.3.1. Distribution uniformity check.

3.3.2. Total volume calculation.

3.3.3. Concentration calculation.

4. REFERENCES.

5. FIGURE CAPTIONS.

WATER SAMPLE
TEM ANALYSIS METHOD

EPA-600/4-80-005
January 1980

INTERIM METHOD FOR DETERMINING ASBESTOS IN WATER

by

Charles H. Anderson and J. MacArthur Long
Analytical Chemistry Branch
Environmental Research Laboratory
Athens, Georgia 30605

ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
ATHENS, GEORGIA 30605

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FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure specific pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of environmental contaminants, the Analytical Chemistry Branch develops and assesses new techniques for identifying chemical constituents of water and soil.

The widespread use of asbestos-containing materials gives rise to concern about exposure of the general population to low level concentrations in air, water supplies, and food. Although hazards associated with the inhalation of asbestos at high concentrations are recognized, the health significance of ingested particles is not fully understood. An integral part of the U.S. Environmental Protection Agency's research into the health effects of exposure to this naturally occurring mineral is the development of an accurate analysis method for determining its presence in water. To this end, this report presents an interim EPA procedure for analytical laboratories to follow that would result in better agreement of analytical results.

David W. Duttweiler
Director
Environmental Research Laboratory
Athens, Georgia

PREFACE

In July 1976, the Preliminary Interim Method for Determining Asbestos in Water was issued by the U.S. Environmental Protection Agency's Environmental Research Laboratory in Athens, Georgia. That method was perceived as representing the current state-of-the-art in asbestos analytical methodology. The objective of writing the method was to present a procedure that analytical laboratories could follow that would result in a better agreement of analytical results. Since that time, a significant amount of additional experimental work has generated data that provide the basis for a more definitive method than was possible previously.

This revised Interim Method reflects the improvements that have been made in asbestos analytical methodology since the initial procedure was drafted. The general approach to the analytical determination, however, remains the same as previously outlined. That is, asbestos fibers are separated from water by filtration on a sub-micron pore size membrane filter. The asbestos fibers are then counted, after dissolving the filter material, by direct observation in a transmission electron microscope.

The major change in the initial procedure is the elimination of the condensation washer as a means of sample preparation. Intra- and inter-laboratory precision data for the method are presented. Also, a suggested statistical evaluation of grid fiber counts is included.

ABSTRACT

An interim electron microscope (EM) procedure for measuring the concentration of asbestos in water samples is described. The main features of the method include filtering the samples through a sub-micron polycarbonate membrane filter, examining an EM specimen grid in a transmission electron microscope (TEM), and verifying fiber identity by selected area electron diffraction.

This interim method is a revision of the procedure issued in 1976 and reflects the improvements that have been made in asbestos analytical methodology since that time.

This report covers a period from July 1976 to December 1978 and work was completed as of December 1978.

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INTERIM METHOD
FOR DETERMINING ASBESTOS IN WATER

1. Scope and Application

1.1 This method is applicable to drinking water and water supplies.

1.2 The method determines the number of asbestos fibers per liter, the size (length and width) of the fibers, the size distribution, and the total mass. The method distinguishes chrysotile from amphibole asbestos. The detection limits are variable and depend upon the amount of total extraneous particulate matter in the sample as well as the contamination level in the laboratory environment. Under favorable circumstances, 0.01 million fibers per liter (MFL) can be detected. The detection limit for total mass of asbestos fibers is also variable and depends upon the fiber size and size distribution in addition to the factors affecting the total fiber count. The detection limit under favorable conditions is in the order of 0.1 nanogram per liter (ng/L).

1.3 The method is not intended to furnish a complete characterization of all the fibers in water.

1.4 It is beyond the scope of this method to furnish detailed instruction in electron microscopy, electron diffraction, or crystallography. It is assumed that those using this method will be sufficiently knowledgeable in these fields to understand the methodology involved.

2. Summary of Method

2.1 A variable, known volume of water sample is filtered through a 0.1 micrometer (um) Nuclepore filter to trap asbestos fibers and the filter is then carbon coated. A small portion of the carbon coated filter with deposited fibers is placed on an electron microscope grid and the filter material is removed by gentle solution in organic solvent. The

material remaining on the electron microscope grid is examined in a transmission microscope at a magnification of about 20,000X. The asbestos fibers are identified by their morphology and electron diffraction patterns and their lengths and widths are measured. The total area examined in the electron microscope is determined and the number of asbestos fibers in this area is counted. The concentration in MFL is calculated from the number of fibers counted, the amount of water filtered, and the ratio of the total filtered area/ sampled filter area. The mass per liter is calculated from the assumed density and the volume of the fibers.

3. Definitions

Asbestos - A generic term applied to a variety of commercially useful fibrous silicate minerals of the serpentine or amphibole mineral groups.

Fiber - Any particulate that has parallel sides and a length/width ratio greater than or equal to 3:1.

Aspect Ratio - The ratio of length to width.

Chrysotile - A nearly pure hydrated magnesium silicate, the fibrous form of the mineral serpentine, possessing a unique layered structure in which the layers are wrapped in a helical cylindrical manner about the fiber axis.

Amphibole Asbestos - A double chain fibrous silicate mineral consisting of S_4O_{11}, laterally linked by various cations such as aluminum, calcium, iron, magnesium, and sodium. Amphibole asbestos consists of crocidolite and amosite (the fibrous form of cummingtonite-gruenerite), and the fibrous forms of tremolite, actinolite, and anthophyllite. These minerals consist of or contain fibers formed through natural growth processes. Mineral fragments that conform to the definition of a fiber and that are formed through a crushing and milling process are analytically indistinguishable from the naturally formed fibers by this method.

Detection Limit - The calculated concentration in MFL, equivalent to one fiber above the background or blank count (Section 8.6).

Statistically Significant - Any concentration based upon a total fiber count of 5 or more in 20 grid squares.

4. Sample Handling and Preservation

It is beyond the scope of this procedure to furnish detailed instructions for field sampling; the general principles of obtaining water samples apply. Some specific considerations apply to asbestos fibers, however, because they are a special type of particulate matter. These fibers are small, and in water range in length from 0.1 um to 20 um or more. Because of the range of size there may be a vertical distribution of particle sizes. This distribution will vary with depth depending upon the vertical distribution of temperature as well as local meteorological conditions. Sampling should take place according to the objective of the analysis. If a representative sample of a water supply is required, a carefully designated set of samples should be taken representing the vertical as well as the horizontal distribution and these samples should be composited for analysis.

4.1 Containment Vessel

The sampling container shall be a clean, screw-capped, polyethylene bottle capable of holding at least 1 liter. The bottle should be rinsed at least two times with the water that is being sampled prior to sampling.

4.2 Quantity of Sample

A minimum of approximately 1 liter of water is required. Leave air space at the top of the container to allow for shaking the sample. It is desirable to obtain two samples from one location.

4.3 Sample Preservation

No preservatives should be added during sampling and the addition of acids should be particularly avoided. If the sample cannot be filtered in the laboratory within 48 hours of its arrival, sufficient amounts (1 milliliter per liter of sample) of a 2.71% solution of mercuric chloride to give a final concentration of 20 ppm of Hg may be added to prevent bacterial growth.

NOTE 1: It has been reported that the growth of algae in water samples can be prevented by storing the samples in the dark.

NOTE 2: Refrigeration of samples at about 5°C minimize bacterial and algal growth.

5. Interferences

5.1 Misidentification

The guidelines set forth in this method for counting fibrous asbestos require a positive identification by both morphology and crystal structure as shown by an electron diffraction pattern. Chrysotile asbestos has a unique tubular structure, usually showing the presence of a central canal, and exhibits a unique characteristic electron diffraction pattern. Although halloysite fibers may show a streaking similar to chrysotile, they do not exhibit chrysotile's characteristic triple set of double spots or 5.3A layer line. It is highly improbable that a non-asbestiform fiber would exhibit the distinguishing chrysotile features. Although amphibole fibers exhibit characteristic morphology and electron diffraction patterns, they do not have the unique properties exhibited by chrysotile. It is possible, therefore, though not probable for misidentification to take place.

It is important to recognize that a significant variable fraction of both chrysotile and amphibole asbestos fibers do not exhibit the required confirmatory electron diffraction pattern. This absence of diffraction is attributable to unfavorable fiber orientation and fiber sizes. The results reported will be low, therefore, as compared to the absolute number of asbestos fibers that are present.

5.2 Obscuration

If large amounts of other materials are present, some small asbestos fibers may not be observed because of physical overlapping. This will result in low values for the reported asbestos content.

5.3 Contamination

Although contamination is not strictly considered to be an interference, it is an important source of erroneous results, particularly for chrysotile. The possibility of contamination, therefore, should always be a consideration.

5.4 Freezing

The effect of freezing on asbestos fibers is not known but there is reason to suspect that fiber breakdown could occur and result in a higher fiber count than was present in the original sample. Therefore, the sample should be transported to the laboratory under conditions that would avoid freezing.

6. Equipment and Apparatus

6.1 Specimen Preparation Laboratory

The ubiquitous nature of asbestos, especially chrysotile, demands that all sample preparation steps be carried out to prevent the contamination of the sample by airborne or other source of asbestos. The prime requirement of the sample preparation laboratory is that it be sufficiently free from asbestos contamination that a specimen blank determination using 200 ml of asbestos-free water yields no more than 2 fibers in 20 grid squares of a conventional 200 mesh electron microscope grid.

In order to achieve this low level of contamination, the sample preparation area should be a separate conventional clean room facility. The room should be operated under positive pressure and have incorporated electrostatic precipitators in the air supply to the room, or as an alternative, absolute (HEPA) filters. No asbestos floor or ceiling tiles, transite heat-resistant boards, or asbestos insulation should be used in construction. Work surfaces should be stainless steel or Formica or equivalent. A laminar flow hood should be provided for sample manipulation. Disposable plastic laboratory coats and disposable overshoes are recommended. Alternatively, new shoes for all operators should be provided and retained for clean room use only. A mat (Tacky Mat, Liberty Industries, 589 Deming Road, Berlin, Connecticut 06037, or equivalent) should be placed at the

entrance to the room to trap any gross contamination inadvertently brought into the room on contaminated shoes. Normal electrical and water services, including a distilled water supply should be provided. In addition, a source of ultra-pure water from a still or filtration-ion exchange system is desirable.

6.2 Instrumentation

6.2.1 Transmission Electron Microscope. A transmission electron microscope that operates at a minimum of 80 kV and has a resolution of better than 1.0 nm and a magnification range of 300 to 100,000 is required. If the upper limit is not attainable directly it may be attained through the use of auxiliary optical viewing. It is mandatory that the instrument be capable of carrying out selected area electron diffraction (SAED) on an area of about 0.3 μm^2. The viewing screen shall have either a millimeter scale, concentric circles of known radii, or other devicer to measure the length and width of the fiber. Most modern transmission microscopes meet the requirements for magnification and resolution.

An energy-dispersive X-ray spectrometer is useful for the identification of suspected asbestiform minerals; this accessory to the microscope, however, is not mandatory.

6.2.2 Data Processor. The large number of repetitive calculations make it convenient to use computer facilities together with relatively simple computer programs.

6.2.3 Vacuum Evaporator. A vacuum evaporator is required for depositing a layer of carbon on the Nuclepore filter and for preparing carbon coated grids.

6.2.4 Low Temperature Plasma Asher. An asher is used for the removal of organic material (including the filter) from samples containing so much organic matter that asbestos fibers are obscured. The sample chamber should be at least 10 cm in diameter.

6.3 Apparatus, Supplies and Reagents

6.3.1 Jaffe Wick Washer. The Jaffe Wick Washer for dissolving Nuclepore filter is described in 8.3.1, and is illustrated in Figure 1.

6.3.2 Filtering Apparatus. A 47-mm funnel (Cat No. XX1504700, Millipore Corporation, Order Service Dept., Bedford, MA 01730) or equivalent is used to filter water samples. A 25-mm funnel (Millipore Cat No. XX1002500) or equivalent is used to filter dispersed ash samples. (617) 275-9200

6.3.3 Vacuum Pump. A pump, for use in sample filtration, should provide vacuum up to about 500 mm of mercury.

6.3.4 EM Grids. Grids of 200-mesh copper or nickel covered with formvar film for use with the Nuclepore-Jaffe sample preparation method are required. These grids may be purchased from manufacturers of electron microscopic supplies or prepared by standard electron microscopic grid preparation procedures. Finder grids may be substituted and are useful if the re-examination of a specific grid opening is desired.

6.3.5 Membrane Filters.

47-mm diameter Millipore membrane filter, type HA, 0.45-um pore size. Used as a Nuclepore filter support on top of glass frit.

47-mm diameter Nuclepore membrane filter; 0.1-um pore size (Nuclepore Corp., 7035 Commerce Circle, Pleasanton, CA 94566). Used to filter the water sample.

25-mm diameter Millipore membrane filter, type HA; 0.45-um pore size. Used as Nuclepore filter support on top of glass frit.

25-mm diameter Nuclepore membrane filter; 0.1-um pore size. Used to filter dispersed ashed Nuclepore filter.

bad news

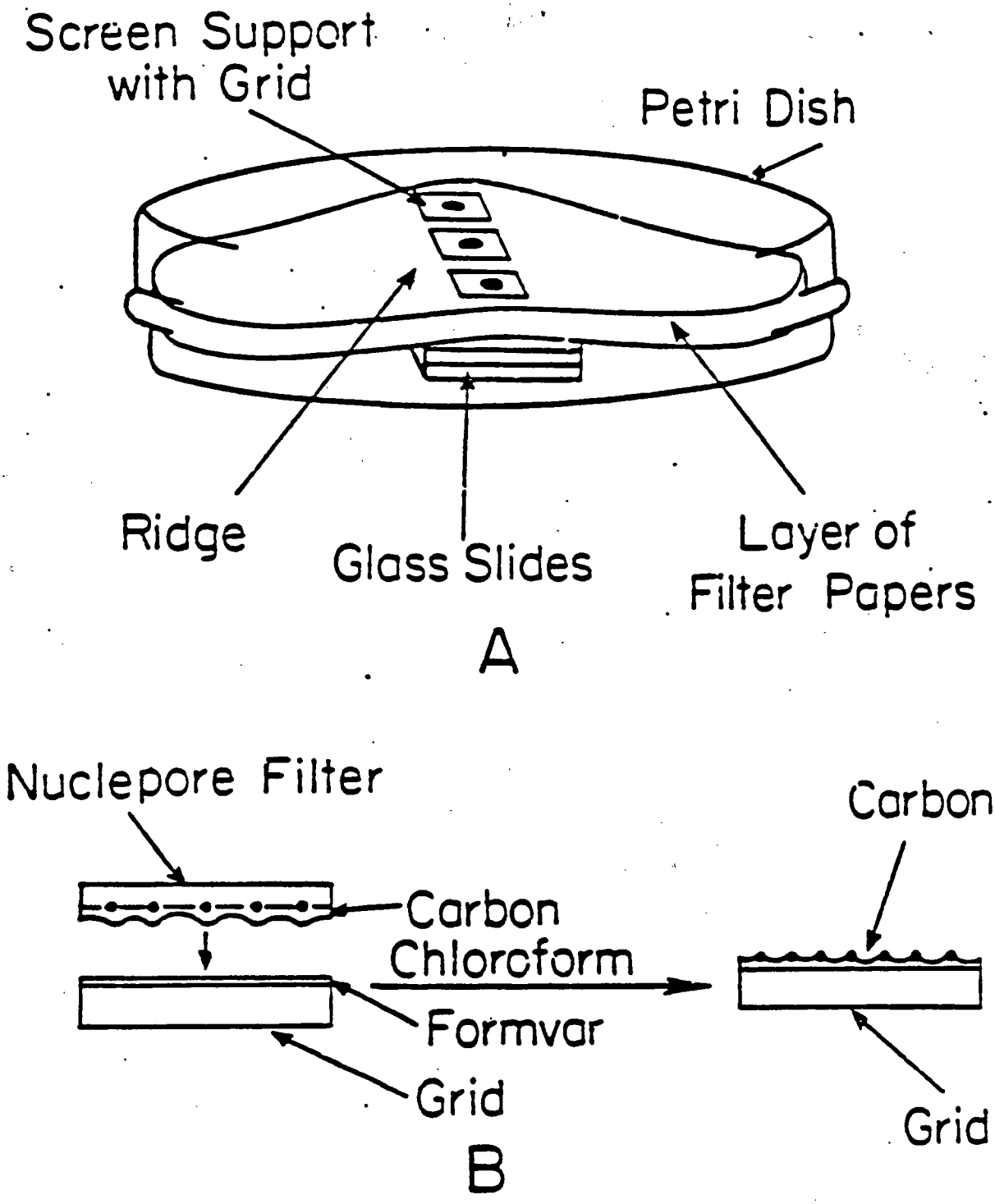

Figure 1. Modified Jaffe Wick Method.
A. Washing Apparatus
B. Washing Process

6.3.6 Glass Vials. 30-mm diameter x 80-mm long. Used to hold filter during ashing.

6.3.7 Glass Slides. Used to support Nuclepore filter during carbon evaporation.

6.3.8 Scalpels.

6.3.9 Scissors.

6.3.10 Tweezers. Several pairs are needed for the many handling operations.

6.3.11 Double-sided Tape. Used to hold filter section flat on glass slide while carbon coating is applied.

6.3.12 Disposable Petri Dishes, 50-mm diameter. Used for storing membrane filters.

6.3.13 Static Eliminator, 500 microcuries Po-210. (Nuclepore Cat. No. V090POL00101) or equivalent. Used to eliminate static charges from membrane filters.

6.3.14 Carbon Rods. Spectrochemically pure, 1/8 in. dia., 3.6 mm x 1.0 mm neck. Used for carbon coating.

6.3.15 Carbon rod sharpener. (Cat. No. 1204, Ernest F. Fullam, Inc., P. O. Box 444, Schenectady, NY 12301) or equivalent. Used for sharpening carbon rods to a neck of specified length and diameter.

6.3.16 Ultrasonic Bath. (50 watts, 55 kHz). Used for dispersing ashed sample and for general cleaning.

6.3.17 Graduated Cylinder, 500 ml.

6.3.18 Spot Plate.

6.3.19 10-µl Microsyringe. Used for administering drop of solvent to filter section during sample preparation.

6.3.20 Carbon Grating Replica, 2160 lines/mm. Used for calibration of EM magnification.

6.3.21 Filter Paper. S & S #589 Black Ribbon (9-cm circles) or equivalent absorbent filter paper. Used for preparing Jaffe Wick Washer.

6.3.22 Screen supports (copper or stainless steel) 12 mm x 12 mm, 200 mesh or equivalent. Used to support specimen grid in Jaffe Wick Washer.

6.3.23 Chloroform. Spectro grade, doubly distilled. Used for dissolving Nuclepore filters.

6.3.24 Asbestos. Chrysotile (Canadian), Crocidolite, Amosite. UICC (Union Internationale Contre le Cancer) Standards. Available from Duke Standards Company, 445 Sherman Avenue, Palo Alto, CA 94306.

6.3.25 Petri Dish. Glass dish (100 mm diameter x 15 mm high). Used for modified Jaffe Wick Washer.

6.3.26 Alconox. (Alconox, Inc., New York, NY 10003) or equivalent. Used for cleaning glassware. Add 7.5 g Alconox to a liter of distilled water.

6.3.27 Parafilm. (American Can Company, Neenah, WI) or equivalent. Used as protective covering for clean glassware.

6.3.28 Pipets. Disposable, 5 ml and 50 ml pipets are required.

6.3.29 Distilled or Deionized Water. Filter if necessary through 0.1-µm Nuclepore filter for making up all reagents, for final rinsing of glassware, and for preparing blanks.

6.3.30 Mercuric chloride, 2.71% solution w/v. Used as sample preservative. See 4.3. Add 5.42 g of reagent grade mercuric chloride ($HgCl_2$) to 100 ml distilled water and dissolve by shaking. Dilute to 200 ml with additional water. Filter through 0.1-µm Nuclepore filter paper before using.

7. Preparation of Standards

Reference standard samples of asbestos that can be used for quality control for a quantitative analytical method are not available. It is, however, necessary for each laboratory to prepare at least two suspensions, one of chrysotile and another of a representative amphibole. These suspensions can then be used for intra-laboratory control and to furnish standard morphology photographs and diffraction patterns.

7.1 Chrysotile Stock Suspension.

Grind about 0.1 g of UICC chrysotile to a powder in an agate mortar. Transfer 10 mg to a clean 1-liter volumetric flask, add several hundred ml of filtered distilled water containing 1 ml of a stock mercuric chloride solution and then make up to 1 liter with filtered distilled water. To prepare a working solution, transfer 10 ml of the above suspension to another 1-liter flask, add 1 ml of a stock mercuric chloride solution and make up to 1 liter with filtered distilled water. This suspension contains 100 ug per liter. Finally transfer 1 ml of this suspension to a 1-liter flask, add 1 ml of a stock mercuric chloride solution and make up to volume with filtered distilled water. The final suspension will contain 5 to 10 MFL and is suitable for laboratory testing.

7.2 Amphibole Stock Suspension.

Prepare amphibole suspensions from UICC amphibole samples as in Section 7.1.

7.3 Identification Standards.

Prepare electron microscopic grids containing the UICC asbestos fibers according to Section 8 and obtain representative photographs of each fiber type and its diffraction pattern for future reference.

8. Procedure

8.1 Filtration.

The separation of insoluble material, including asbestiform minerals, through filtration and subsequent deposition on a membrane filter is a critical step in the procedure. The objective of the

filtration is not only to separate, but also to distribute the particulate matter uniformly such that discrete particles are deposited with a minimum of overlap.

The volume filtered will range from 50 to 500 ml. In an unknown sample, the volume can not be specified in advance because of the presence of variable amounts of particulate matter. In general, sufficient sample is filtered such that a very faint stain can be observed on the filter medium. The maximum loading that can be tolerated is 10 μg/cm², or about 200 μg on a 47-mm diameter filter; 5 μg/cm² is near optimum. If the total solids content is known, an estimate of the maximum volume tolerable can be obtained. In a sample of high solids content, where less than 50 ml is required, the sample should be diluted with filtered distilled water so that a minimum total of 50 ml of water is filtered. This step is necessary to allow the insoluble material to deposit uniformly on the filter. The filtration funnel assembly must be scrupulously cleaned before each filtration. The filtration should be carried out in a laminar flow hood.

50 μg

NOTE: The following cleaning procedure has been found to be satisfactory.

Wash each piece of glassware three times with distilled water. Following manufacturer's recommendations, use the ultrasonic bath with an Alconox-water solution to clean all glassware. After the ultrasonic cleaning, rinse each piece of glassware three times with distilled water. Then rinse each piece three times with deionized water that has been filtered through 0.1-μm Nuclepore filter. Dry in an asbestos-free oven. After the glassware is dry, seal openings with parafilm.

a. Assemble the vacuum filtration apparatus incorporating the 0.1-μm Nuclepore backed with 0.45-μm Millipore filter. See 8.3.2.

b. Vigorously agitate the water sample in its container. Treatment of the sample in an ultrasonic bath may be required to evenly disperse the particulate material.

c. If the required filtration volume can be estimated, either from turbidity estimates of suspended solids or previous experience, immediately withdraw the proper volume from the container and add the entire volume to the 47-mm diameter funnel. Apply vacuum sufficient for filtration but gentle enough to avoid the formation of a vortex. If a completely unknown sample is being analyzed, a slightly modified procedure must be followed. Pour 500 ml of a well-mixed sample into a 500-ml graduated cylinder and immediately transfer the entire contents to the prepared vacuum filtration apparatus. Apply vacuum gently and continue suction until all of the water has passed through the filter. If the resulting filter appears obviously coated or discolored, another filter should be prepared in the same manner, but this time using only 200 or 100 ml of sample.

NOTE 1: Do not add more water after filtration has started and do not rinse the sides of the funnel.

NOTE 2: Nuclepore filter is basically a hydrophobic material. The manufacturer applies a detergent to the surface of the filter in order to render it hydrophilic; this process, however, does not appear to be entirely satisfactory in some batches. Pretreatment of the filter in a low temperature asher at 10 watts for 10 seconds can be used to render the surface of the filter hydrophilic. This process will significantly decrease the islands of sparse deposit that are frequently observed.

d. Disassemble the funnel, remove the filter, and dry it in a covered petri dish.

8.2 Preparation of Electron Microscope Grids.

Preparation of the grid for examination in the microscope is a critical step in the analytical procedure. The objective is to remove the organic filter material from the asbestos fibers with minimum loss and movement and with minimum breakage of the grid support film.

If the sample contains large amounts of organic matter that interfere with fiber counting and identification a preliminary ashing step is required. See 8.5.

8.3 Nuclepore Filter, Modified Jaffe Wick Technique.

8.3.1 Preparation of Modified Jaffe Washer

Place three glass microscope slides (75 mm x 22 mm) one on top of the other in a petri dish (100 mm x 15 mm) along a diameter. Place 14 S & S #589 Black Ribbon filter papers (9-cm circles) in the petri dish over the stack of microscope slides. Place three copper mesh screen supports (12 mm x 12 mm) along the ridge formed by the stack of slides underneath the layer of filter papers. Place an EM specimen grid on each of the screen supports. See Fig. 1.

NOTE: A stack of 30 to 40 S & S filters (7-cm circle), or equivalent, can be substituted for the 14 filters and microscope slides in preparing the Jaffe washer.

8.3.2 Vacuum Filtration Unit

Assemble the vacuum filtration unit. Place a 0.45-um Millipore filter type HA on the glass frit and then position a 0.1-um Nuclepore filter, shiny side up, on top of the Millipore filter. Apply suction to center the filters flat on the frit. Attach the filter funnel and shut off the suction.

8.3.3 Sample Filtration

See 8.1.

8.3.4 Sample Drying

Remove the filter funnel and place the Nuclepore filter in a loosely covered petri dish to dry. The petri dish containing the filter may be placed in an asbestos-free oven at 45° C for 30 minutes to shorten the drying time.

Alternatively, the Nuclepore filter section may be mounted on a glass slide prior to drying the filter.

8.3.5 Selection of Section for Carbon Coating

Using a small pair of scissors or sharp scalpel, cut out a rectangular section of the Nuclepore filter. The minimum approximate dimensions should be 15 mm long and 3 mm wide. Avoid selection near the perimeter of the filtration area.

8.3.6 Carbon Coating the Filter

Tape the two ends of the selected filter section to a glass slide using double-sided tape. Take care not to stretch the filter section. Identify the filter section using a china marker on the slide. Place the glass slide with the filter section into the vacuum evaporator. Insert the necked carbon rod and, following manufacturer's instructions, obtain high vacuum. Evaporate the neck, with the filter section rotating, at a distance of approximately 7.5 cm from the filter section to obtain a 30 to 50 nm layer of carbon on the filter paper. Evaporate the carbon in several short bursts rather than continuously to prevent overheating the surface of the Nuclepore filter.

NOTE 1: Overheating the surface tends to crosslink the plastic, rendering the filter dissolution in chloroform difficult.

NOTE 2: The thickness of the carbon film can be monitored by placing a drop of oil on a porcelain chip that is placed at the same distance from the carbon electrodes as the specimen. Carbon is not visible in the region of the oil drop thereby enabling the visual estimate of the deposit thickness by the contrast differential.

8.3.7 Grid Transfer

Remove the filter from the vacuum evaporator and cut out three sections somewhat less than 3 mm x 3 mm and such that the

square of Nuclepore fits within the circumference of the grid. Pass each of the filter sections over a static eliminator and then place each of the three sections carbon-side down on separate specimen grids previously placed in the modified Jaffe Washer. Using a microsyringe, place a 10-µl drop of chloroform on each filter section resting on a grid and then saturate the filter pad until pooling of the solvent occurs below the ridge formed by the glass slides inserted under the layer of filter papers. Place the cover on the petri dish and allow the grids to remain in the washer for approximately 24 hours. Do not allow the chloroform to completely evaporate before the grids are removed. To remove the grids from the washer, lift the screen support with the grid resting upon it and set this in a spot plate depression to allow evaporation of any solvent adhering to the grid. The grid is now ready for analysis or storage.

8.4 Electron Microscopic Examination

8.4.1 Microscope Alignment and Magnification Calibration

Following the manufacturer's recommendations carry out the necessary alignment procedures for optimum specimen examination in the electron microscope. Calibrate the routinely used magnifications using a carbon grating replica.

NOTE: Screen magnification is not necessarily equivalent to plate magnification.

8.4.2 Grid Preparation Acceptability

After inserting the specimen into the microscope, adjust the magnification low enough (300X-1000X) to permit viewing complete grid squares. Inspect at least 10 grid squares for fiber loading and distribution, debris contamination, and carbon film continuity.

Reject the grid for counting if:

1) The grid is too heavily loaded with fibers to perform accurate counting and diffraction operations. A new sample preparation either from a smaller volume of water or from a dilution with filtered distilled water must then be prepared.

2) The fiber distribution is noticeably uneven. A new sample preparation is required.

3) The debris contamination is too severe to perform accurate counting and diffraction operations. If the debris is largely organic the filter must be ashed and redispersed (see 8.5). If it is inorganic, the sample must be diluted and again prepared.

4) The majority of grid squares examined have broken carbon films. A different grid preparation from the same initial filtration must be substituted.

8.4.3 Procedure for Fiber Counting

Two methods are commonly used for fiber counting. In one method (A), 100 fibers contained in randomly selected fields of view are counted. The number of fields plus the area of a field of view must be known when using this method. In the other method (B), all fibers (at least 100) in several grid squares or 20 grid squares are counted. The number of grid squares counted and the average area of one grid square must be known when using this method.

NOTE: The method to use depends upon the fiber loading on the grid and it is left to the judgment of the analyst to select the optimum method. The following guidelines can be used: If it is estimated that a grid square (80 um x 80 um) contains 50 to 100 fibers at a screen magnification of 20000X, it is convenient to use the field-of-view counting method. If the estimate is less than 50, the grid square method of counting should be chosen. On the other

hand, if the fiber count is estimated to be over 300 fibers per grid square, a new grid containing fewer fibers must be prepared (through dilution or filtration of a smaller volume of water).

8.4.3A Field-of-View Method

After determining that a fiber count can be obtained using this method, adjust the screen magnification to 15,000 to 20,000X. Select a number of grid squares that would be as representative as possible of the entire analyzable grid surface. From each of these squares, select a sufficient number of fields of view for fiber counting. The number of fields of view per grid square is dependent upon the fiber loading. If more than one field of view per grid square is selected, scan the grid opening orthogonally in an arbitrary pattern that prevents overlapping of fields of view. Carry out the analysis by counting, measuring and identifying (see 8.4.4) approximately 50 fibers on each of two grids.

The following rules should be followed when using the field of view method of fiber counting. Although these rules were derived for a circular field of view they can be modified to apply to square or rectangular designs.

1) Count all fibers contained within the counting area and not touching the circumference of the circle.

2) Designate the upper right-hand quadrant as I and number in clockwise order. Count all fibers touching or intersecting the arc of quadrants I or IV. Do not count fibers touching or intersecting the arc of quadrants II or III.

3) If a fiber intersects the arc of both quadrants III and IV or I and II count it only if the greater length was outside the arc of quadrants IV and I, respectively.

4) Count fibers intersecting the arc of both quadrants I and III but not those intersecting the arc of both II and IV.

These rules are illustrated in Fig. 2.

8.4.3B Grid Square Method

After determining that a fiber count can be obtained using this method adjust the screen magnification to 15,000 to 20,000X. Position the grid square so that scanning can be started at the left upper corner of the grid square. While carefully examining the grid, scan left to right, parallel to the upper grid bar. When the perimeter of the grid square is reached, adjust the field of view down one field width and scan in the opposite direction. The tilting section of the fluorescent screen may be used conveniently as the field of view. Examine the square until all the area has been covered. The analysis should be carried out by counting, measuring, and identifying (see 8.4.4) approximately 50 fibers on each of two grids or until 10 grid squares on each of two grids have been counted. Do not count fibers intersecting a grid bar.

8.4.4 Measurement and Identification

Measure and record the length and width of each fiber having an aspect ratio greater than or equal to three. Disregard obvious biological-bacteriological fibers and diatom fragments. Examine the morphology of each fiber using optical viewing if necessary. Tentatively identify, by reference to the UICC standards, chrysotile or possible amphibole asbestos. Attempt to obtain a diffraction pattern of each fiber utilizing the shortest camera length possible. Move the suspected fiber image to the center of the screen and insert a suitable selected area aperture into the electron beam so that the fiber image, or a portion of it, is in the illuminated area. The size of the aperture and the portion of the fiber should be such that particles other than the one to be examined are

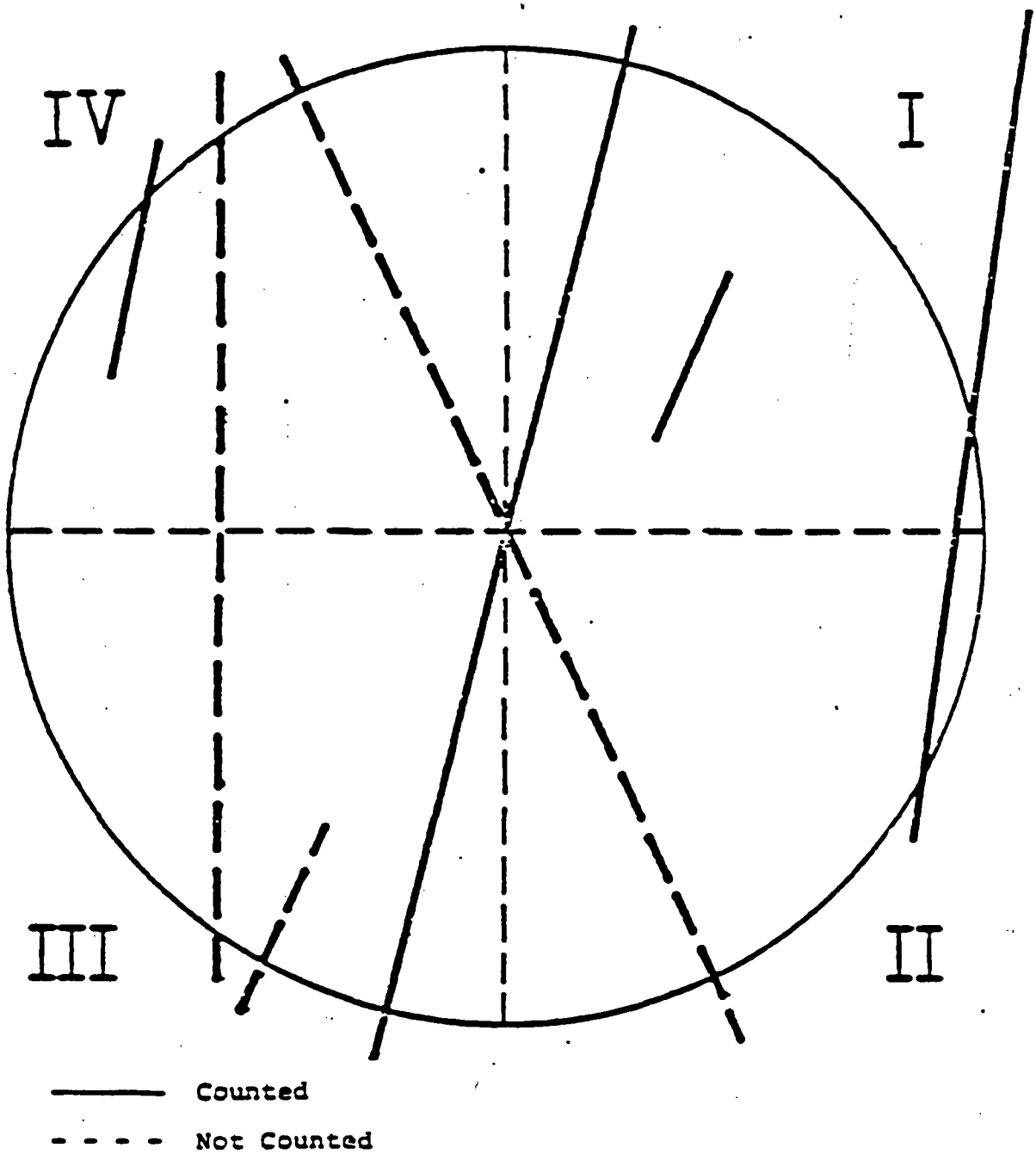

Figure 2. Illustration of Counting Rules for Field of View Method.

excluded from the selected area. Observe the diffraction pattern with the binocular attachment. If an incomplete diffraction pattern is obtained, move the particle image around in the selected area to get a clearer diffraction pattern or to eliminate possible interferences from neighboring particles.

Determine whether the fiber is chrysotile or an amphibole by comparing the diffraction pattern obtained to the diffraction patterns of known standard asbestos fibers. Confirm the tentative identification of chrysotile and amphibole asbestos from their electron diffraction patterns. Classify each fiber as chrysotile, amphibole, non-asbestos, no diffraction, or ambiguous.

NOTE 1: It is convenient to use a tape recorder during the examination of the fibers to record all pertinent data. This information can then be summarized on data sheets or punched cards for subsequent automatic data processing.

NOTE 2: Chrysotile fibers occur as single fibrils or in bundles. The fibrils generally show a tubular structure with a hollow canal, although the absence of the canal does not rule out its identification. Amphibole asbestos fibers usually exhibit a lath-like structure with irregular ends, but occasionally will resemble chrysotile in appearance.

NOTE 3: The positive identification of asbestos by electron diffraction requires some judgment on the part of the analyst because some fibers give only partial patterns. Chrysotile shows unique prominent streaks on the layer lines nearest the central one and a triple set of double spots on the second layer line. The streaks and the set of double spots are the distinguishing characteristics of chrysotile required for identification. Amphibole asbestos requires a more complete diffraction pattern to be positively identified. As a qualititative guideline,

layer lines for amphibole, without the unique streaks (some streaking may be present) of chrysotile, should be present and the arrangement of diffraction spots along the layer lines should be consistent with the amphibole pattern. The pattern should be distinct enough to establish these criteria.

NOTE 4: Chrysotile and thin amphibole fibers may undergo degradation in an electron beam; this is particularly noticeable in small fibers. It may exhibit a pattern for 1 to 2 seconds and disappear and the analyst must be alert to note the characteristic features.

NOTE 5: An ambiguous fiber is a fiber that gives a partial electron diffraction pattern resembling asbestos, but is insufficient to provide positive identification.

8.4.5 Determination of Grid Square Area

Measure the dimensions of several representative grid squares from each batch of grids with an optical microscope. Calculate the average area of a grid square. This should be done to compensate for variability in grid square dimensions.

8.5 Ashing

Some samples contain sufficiently high levels of organic material that an ashing step is required before fiber identification and counting can be carried out.

Place the dried Nuclepore filter paper containing the collected sediment into a glass vial (28 mm diameter x 80 mm high). Position the filter such that the filtration side touches the glass wall. Place the vial in an upright position in the low temperature asher. Operate the asher at 50 watts (13.56 MHz) power and 2 torr oxygen pressure. Ash the filter until a thin film of white ash remains. The time required is generally 6 to 8 hours. Allow the ashing chamber to slowly reach atmospheric pressure and remove the vial. Add 10 ml of filtered distilled water to the vial. Place the vial in an ultrasonic bath for 30 minutes to disperse the ash. Dilute the sample if required.

Assemble the 25-mm diameter filtering apparatus. Center a 25-mm diameter 0.1-um Nuclepore filter (with the 0.45-um Millipore backing) on the glass frit. Apply suction and recenter the filter if necessary. Attach the filter funnel and turn off the suction. Add the water containing the dispersed ash from the vial to the filter funnel. Apply suction and filter the sample. After drying, this filter is ready to be used in preparing sample grids as in 8.3.

NOTE 1: In specifying a 25-mm diameter filter it is assumed that the ashing step is necessary mainly because of the presence of organic material and that the smaller filtering area is desirable from the point of view of concentrating the fibers. If the sample contains mostly inorganic debris such that the smaller filtering area will result in overloading the filter, the 47-mm diameter filter should be used.

NOTE 2: It will be noted that a 10-ml volume is filtered in this case instead of the minimum 50-ml volume specified in 8.1. These volumes are consistent when it is considered that there is approximately a 5-fold difference in effective filtration area between the 25-mm diameter and 47-mm diameter filters.

NOTE 3: Cross contamination is probable when ashing more than one sample at a time.

8.6 Determination of Blank Level

Carry out a blank determination with each batch of samples prepared, but a minimum of one per week. Filter a fresh supply (500 ml) of distilled, deionized water through a clean 0.1-um membrane filter. Filter 200 ml of this water through a 0.1-um Nuclepore filter, prepare the electron microscope grid, and count exactly as in the procedures 8.1 - 8.4. Examine 20 grid squares and record this number of fibers. A maximum of two fibers in 20 grid squares is acceptable for the blank sample.

NOTE: Monitoring the background level of asbestos is an integral part of the procedure. Upon initiating asbestos analytical work, blank samples must be run to establish the initial suitability of the laboratory environment, cleaning procedures, and

reagents for carrying out asbestos analyses. Analytical determinations of asbestos can be carried out only after an acceptably low level of contamination has been established.

9. Calculations

9.1 Fiber Concentrations

Grid Square Counting Method - If the Grid Square Method of counting is employed, use the following formula to calculate the total asbestos fiber concentration in MFL.

$$C = (\bar{F} \times A_f)/(A_g \times V_o \times 1000) \qquad (1)$$

where: C = Fiber concentration (MFL)

$\bar{F}$ = Average number of fibers per grid opening

A_f = Effective filtration area of filter paper (mm^2) used in grid preparation for fiber counting

A_g = Average area of one grid square (mm^2)

V_o = Original volume of sample filtered (ml)

If ashing is involved, use the same formula but substitute the effective filtration area of the 25-mm diameter filter for A_f instead of that for the 47-mm diameter filter. If one-half the filter is ashed, multiple C by two.

Field-of-View Counting Method - If the Field-of-View Method of counting is employed, use the following formula to calculate the total asbestos fiber concentrations (MFL).

$$C = (\bar{F} \times A_f \times 1000)/(A_v \times V_o) \qquad (2)$$

where: C = Fiber concentration

$\bar{F}$ = Average number of fibers per field of view

A_f = Effective filtration area of filter paper (mm^2) used in grid preparation for fiber counting

A_v = Area of one field of view (μm^2)

V_o = Original volume of sample filtered (ml)

If ashing is involved, use the same formula but substitute the effective filtration area of the 25-mm diameter filter for A_f instead of that for the 47-mm diameter filter.

9.2 Estimated Mass Concentration

Calculate the mass (µg) of each fiber counted using the following formula.

$$M = L \times W^2 \times D \times 10^{-6}$$

If the fiber content is predominantly chrysotile, the following formula may be used.

$$M = \frac{\pi}{4} \times L \times W^2 \times D \times 10^{-6} \qquad (3)$$

where: M = Mass (µg)

L = Length (µm)

W = Width (µm)

D = Density of fibers (g/cm^3)

Then calculate the mass concentration (µg/l) employing the following formula.

$$M_c = C \times \bar{M}_f \times 10^6$$

where: M_c = mass concentration (µg/l)

C = fiber concentration (MFL)

$\bar{M}_f$ = mean mass per fiber (µg)

To calculate $\bar{M}_f$ use the following formula.

$$\bar{M}_f = \sum_{i=1}^{n} M_{i/n} \qquad (4)$$

where: M_i = mass of each fiber, respectively

n = number of fibers counted

NOTE 1: Because many of the amphibole fibers are lath shaped rather than square in cross section the computed mass will tend to be high because laths will, in general, tend to lie flat rather than on edge.

NOTE 2: Assume the following densities: chrysotile 2.5, amphibole 3.25.

9.3 Aspect Ratio

The aspect ratio for each fiber is calculated by dividing the length by the width.

10. Reporting

10.1 Report the following concentration as MFL for sample and blank using 95% confidence intervals.

a. Chrysotile

b. Amphibole

c. Total asbestos fibers

10.2 Use two significant figures for concentrations greater than 1 MFL, and one significant figure for concentrations less than 1 MFL.

10.3 Tabulate the size distribution, length and width.

10.4 Tabulate the aspect ratio distribution.

10.5 Report the calculated mass as µg/l.

10.6 Indicate the detection limit in MFL.

10.7 Indicate if less than five fibers were counted.

10.8 Include remarks concerning pertinent observations, (clumping, amount of organic matter, debris) amount of suspected though not identifiable as asbestos fibers (ambiguous).

11. Precision

11.1 Intra-Laboratory

The precision that is obtained within an individual laboratory is dependent upon the number of fibers counted. If 100 fibers are counted and the loading

is at least 3.5 fibers/grid square, computer modeling of the counting procedure shows that a relative standard deviation of about 10% can be expected.

In actual practice some degradation from this precision will be observed but should not exceed ± 15% if several grids are prepared from the same filtered sample. The relative standard deviation of analyses of the same water sample in the same laboratory will increase as a result of sample preparation errors and a relative standard deviation of about about ± 25 to 35% will occur. As the number of fibers counted decreases, the precision will also decrease approximately proportional to $\sqrt{N}$ where N is the number of fibers counted. The precision for mass concentration is generally poorer than that for fiber concentration.

Based upon the analysis of one laboratory utilizing a different analyst for each of three water samples, intra-laboratory precision data are presented in Table 1.

11.2 Inter-Laboratory

Based upon the analysis by various government and private industry laboratories of filters prepared from nine water samples, inter-laboratory precision data of the method are presented in Table 2.

12. Accuracy

12.1 Fiber Concentrations

As no standard reference materials are available, only approximate estimates of the accuracy of the procedure can be made. At 1 MFL, it is estimated that the results should be within a factor of 10 of the actual asbestos fiber content.

This method requires the positive identification of a fiber to be asbestos as a means for its quantitative determination. As the state-of-the-art precludes the positive identification of all of the asbestos fibers present, the results of this method, as expressed as MFL, will be biased on the low side and, assuming no fiber loss, represent 0.4 to 0.8 of the total asbestos fibers present.

TABLE 1. INTRA-LABORATORY PRECISION

Sample Type	Number of Sample Aliquots Analyzed	Mean Fiber Concentration MFL (millions of asbestos fibers/l)	Precision, Relative Standard Deviation	Mass Concentration (μg/l)	Precision, Relative Standard Deviation
Chrysotile (UICC)	26	23	37%	0.32	71%
Crocidolite (UICC)	20	8	36%	1.5	48%
Taconite (raw water)	20	16	24%	10.5	65%

TABLE 2. INTER-LABORATORY PRECISION

Sample Type	Number of Labs Reporting	Mean Fiber Concentration, MFL (millions of asbestos fibers/l)	Precision, Relative Standard Deviation
Chrysotile	10	877	35%
	9	119	43%
	11	59	41%
	9	31	65%
	9	28	32%
	3	25	35%
Amphibole	11	139	50%
	4	95	52%
	14	36	66%

12.2 Mass Concentrations

As in the case of the fiber concentrations, no standard samples of the size distribution found in water are available. The estimated mass concentration is often very inaccurate because of poor counting statistics associated with large fibers that are few in number but represent most of the actual mass concentration.

13. Suggested Statistical Evaluation of Grid Fiber Counts

13.1 Because the fiber distribution on the sample filter, resulting from the method of filtration, has not been fully characterized, the fiber distribution obtained on the electron microscope grids for each sample should be tested statistically against an assumed distribution and a measure of the precision of the analysis should be provided.

13.2 Assume that the fibers are uniformly and randomly distributed on the sample filter and grids. One method for confirming this assumption is given below.

Using the chi-square test, determine whether the total number of fibers found in individual grid openings are randomly and uniformly distributed among the openings using the following formula.

$$X^2 = \sum_{i=1}^{N} \frac{(n_i - np_i)^2}{np_i} \qquad (5)$$

where: X^2 = Chi-square statistic

N = Number of grid openings examined for the sample

n_i = Total number of fibers found in each respective grid opening

n = Total number of fibers found in N grid openings

p_i = Ratio of the area of each respective grid opening to the sum of the areas of all grid openings examined

NOTE: If an average area for the grid squares has been measured as outlined in 8.4.5, the term np_i represents the mean fiber count per grid square.

If the value for X^2 exceeds the value listed in statistical tables for the 0.1% significance level with N-1 degrees of freedom, the fibers are not considered to be uniformly and randomly distributed among the grid openings. In this case, it is advisable to try to improve the uniformity of fiber deposition by filtering another aliquot of the sample and repeating the analysis.

13.3 If uniformity and randomness of fiber deposition on the microscope grids has been demonstrated as in 13.2, and the fiber concentration is assumed to be normally distributed about the mean value, the 95% confidence interval about the mean fiber concentrations for chrysotile, amphibole, and total asbestos fibers may be determined using the following formulae.

$$S_c = \left[\frac{N \sum_{i=1}^{N} X_i^2 - \left(\sum_{i=1}^{N} X_i \right)^2}{N(N-1)} \right]^{1/2} \qquad (6)$$

where: S_c = Standard deviation of the chrysotile fiber count

N = Number of grid openings examined for the sample

X_i = Number of chrysotile fibers in each grid opening, respectively

Obtain the standard deviations of the fiber counts for amphibole asbestos fibers and for total asbestos fibers by substituting the corresponding value of X_i into equation (6).

$$X_u = \bar{X} + \frac{tS_c}{\sqrt{N}} \qquad (7)$$

$$X_L = \bar{X} - \frac{tS_c}{\sqrt{N}} \qquad (8)$$

where: X_u = Upper value of 95% confidence interval for chrysotile

X_L = Lower value of 95% confidence interval for chrysotile

$\bar{X}$ = Average number of fibers per grid opening

t = Value listed in t-distribution tables at the 95% confidence level for a two tailed distribution with N-1 degree of freedom

S_c = Standard deviation of the fiber counts for chrysotile

N = Number of grid openings examined for the sample

The values of X_u and X_L can be converted to concentrations in millions of fibers per liter using the formula in section 9 and substituting either X_u or X_L for the term F.

Obtain the upper and lower values of the 95% confidence interval for amphibole asbestos fibers and total asbestos fibers by substituting the corresponding values of $\bar{X}$ and S into equations (7) and (8).

Report the precision of the analysis, in terms of the upper and lower limits of the 95% confidence interval, for chrysotile, amphibole, and total asbestos fiber content. If a lower limit is found to be negative, report the value of the limit as zero.

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13135 Lee Jackson Memorial Highway
Suite 200
Fairfax, Virginia 22033
(703) 968-0900