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# Environmental Asbestos Assessment Manual

## Superfund Method for the Determination of Asbestos in Ambient Air

## Part 2: Technical Background Document

INTERIM VERSION

## *DISCLAIMER*

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## 1. INTRODUCTION

A sampling and analysis method for the determination of asbestos in air is presented in Part 1 of this report, under separate cover. This method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, although it is applicable to a wide range of ambient air situations. Considerations addressed during the development of the method are presented in this companion technical background document. Also, in the interest of facilitating the use and interpretation of analytical results derived from the method presented in Part 1, recommended procedures for manipulating such data as part of a site evaluation are provided in Section 7 of this document.

Asbestos presents a complex challenge to investigators evaluating risks at Superfund sites. Unlike the majority of other chemicals frequently monitored at hazardous wastes sites, asbestos exposures can not be adequately characterized by a single concentration parameter. This is because the different size ranges of airborne asbestos particles, even when they are of the same mineral variety, exhibit different dose/response relationships. Thus a more accurate characterization of asbestos exposure is that arising from a family of substances independently contributing to toxicity rather than that of exposure to a single chemical. Therefore, proper characterization of asbestos exposure requires that the relative contributions from each of the many components of exposure be defined.

Existing equipment and methods used to measure asbestos are limited in their ability to fully characterize asbestos exposures. In addition, the toxicity of asbestos is currently a subject of scientific debate. Consequently, monitoring asbestos in a manner that satisfies the needs of a risk assessment requires innovations that tax the limits of available technology. Several variations were considered during development and the method presented in Part 1 of this report represents a workable compromise among several technical constraints.

The purpose for documenting the data and assumptions used to develop the method proposed in Part 1 is to facilitate critical evaluation while highlighting the needs for additional research and for better documentation of existing analytical results. Considerations addressed in this report that have been documented in the literature are cited accordingly. Considerations that remain largely a subject of conjecture are also noted. Due to the current level of interest and activity provoked by asbestos, further improvements in asbestos sampling, analysis, and evaluation are anticipated.

## 2. OVERVIEW

Development of the method presented in Part 1 of this report began with consideration of the kinds of data required to perform a risk assessment. Because the primary objective is to provide analysis results that reflect potential health risks, factors contributing to the biological activity of asbestos were considered to identify specific asbestos exposure parameters that relate to risk assessment objectives.

Published risk factors are expressed in terms of airborne concentrations as determined by phase contrast microscope (PCM) counts. However, a range of dimensional parameters have been shown to relate to biological activity in addition to PCM counts. Therefore, to address persisting controversies in the interpretation of asbestos biological activity, several parameters were selected for characterization by the method presented in this report. To maximize flexibility, sampling and analysis results will be recorded in this method so as to allow for re-interpretation of results without the need to re-evaluate the original sample specimens.

The characteristics of the kinds of environmental samples likely to be collected and analyzed by the proposed method were considered in order to identify the sampling and analysis criteria, which are required to satisfy the objectives of a risk assessment. The morphologies of asbestos structures and total dusts typically found in these samples were considered in order to determine requirements for distinguishing and characterizing components that potentially relate to biological activity.

To determine the required level of analytical sensitivity for the method, concentrations typical of environmental samples were estimated from the limited published data. A range of typical concentrations was defined from measured background and concentrations expected to be observed in the immediate vicinity of asbestos sources. Precision criteria defined for the method were developed by considering the requirements for delineating spatial and temporal trends in environmental asbestos concentrations as they apply to risk assessment.

After the dimensional parameters to be characterized had been established and the criteria for the sensitivity and precision of the method had been defined, the available sampling and analysis technologies were evaluated to determine what combinations would be capable of providing the required information. Because published risk factors are expressed in terms of PCM counts, phase contrast microscopy was considered for use as the analysis technique but this was rejected for a variety of severe, inherent limitations.

Despite the relationship between PCM measurements and existing risk factors, measurement of the PCM equivalent fraction of asbestos in environmental samples has been shown to be less important for assessing risks

than characterizing fractions of asbestos that are more directly associated with biological activity (Chesson et al 1989a). Therefore, transmission electron microscopy (TEM) was selected as the appropriate tool for analysis of environmental samples.

Because sensitivity and precision are functions both of sample characteristics and of rules for analysis, alternate combinations of sampling procedures, sample preparation techniques, and analysis methods were evaluated to identify the most cost-effective combination capable of providing the kinds of analytical results necessary to support a risk assessment. Published TEM analytical methods were evaluated for their applicability to this problem. Combined with several specific modifications, many of the features of the published analytical methods were incorporated into the method developed in this report. For sampling, alternate types of collection filters and the degree of filter loading were addressed. For TEM specimen preparation, both a direct and an indirect preparation technique were considered. For analysis, the area of the filter to be scanned was defined as a function of sample loading and the required analytical sensitivity. In addition, counting rules and recording rules were modified to assure that data would be preserved in a manner allowing detailed re-interpretation after analysis was completed. Finally, a two-phased approach for sample collection and analysis was adopted for cost-effectiveness.

## 3. BACKGROUND

Available data from published studies of asbestos exposure and biological activity were reviewed to define method requirements as they relate to risk assessment objectives. Existing sampling and analysis technologies were considered to identify an approach that best satisfies the method requirements.

### 3.1. BIOLOGICAL ACTIVITY

It is generally recognized that health risks posed by exposure to asbestos dusts depend predominantly on the concentrations and physical dimensions of the individual fibrous structures in the dust. Fibrous structures may include fibers, bundles, clusters or matrices because airborne asbestos is often found as a mix of such complex structures in addition to single fibers. Asbestos structures may also be found aggregated with equant (non-asbestos) particles. Somewhat arbitrarily, fibrous structures have been defined as those exhibiting aspect ratios greater than 3:1 to distinguish them from isometric particles (Walton, 1982). Isometric particles have not been shown to exhibit the same types of biological activity as fibrous structures (Elmes, 1982). However, a cutoff in aspect ratio below which biological activity can be considered insignificant has not been formally established.

Although it has been shown qualitatively that a relationship exists between the physical dimensions of fibrous structures and biological activity, the form of the relationship appears to be complex (see for example: USEPA 1986a). The relationship between certain types of structures and biological activity has been investigated in a series of animal studies. Based on these studies, asbestos toxicity varies directly with the length and inversely with the width (thickness) of asbestos fibers. Thus, the longest and thinnest fibers tend to be the most potent. However, there has not been general agreement on a minimum length below which the biological activity of asbestos can be considered insignificant (USEPA 1986a). Therefore, the exact shape of the relationship between length, diameter, and potency is still a subject of controversy.

Unlike fibers, the biological activities of bundles, clusters, and matrices have not been investigated directly. Some investigators (see for example: Nicholson, 1988) believe that biological activity correlates best with the mass of the structure. However, other investigators do not share this view (see for example: Bertrund and Pezerat, 1980). Although, various individuals have proposed theories concerning the relationship between aggregates (bundles, clusters, and matrices) and biological activity, due to the lack of data, such theories currently remain in the realm of conjecture.

Several studies (see for example: Bonneau et al, 1986) indicate that the mineralogy of a structure also plays a role in determining the biological activity of the structure. The majority of studies relating biological activity to physical dimensions indicate that structures bearing appropriate dimensions all tend to exhibit similar biological activity provided that the

structures are durable in vivo (See for example: USEPA 1986a). In contrast, the Bonneau paper is one of a small number of papers that reports that structures with identical dimensions that are composed of different minerals exhibit different levels of biological activity.

Based on a review of the published studies, it is apparent that the characteristics of an asbestos dust that best relate to biological activity are still a subject of scientific debate. The principle outstanding issues are:

(a) whether the total structure count adequately tracks the biological activity of asbestos or whether a specified, minimum length can be defined below which contributions to biological activity may be considered unimportant;

(b) whether asbestos aggregates (bundles, clusters, and matrices) should be counted as single entities or weighted in proportion to the number of individual fibers present in the aggregate to properly represent their contributions to risks;

(c) whether biological activity correlates with the overall mass of an asbestos structure;

(d) whether durable, non-asbestos fibers with similar morphologies to asbestos are biologically active;

(e) whether to employ different criteria for different asbestos mineral types or for different exposure circumstances (eg. exposure to mine tailings verses exposure to textile wastes, etc.).

Ideally, the results of an analytical method should be sufficiently flexible to allow for interpretation based on any of the prevailing theories concerning biological activity so that such results will retain validity even as the understanding of asbestos dose/response relationships matures.

### 3.2. RISK FACTORS

Although animal studies provide an indication of the qualitative relationship between physical characteristics and potency, they are not useful for quantifying dose/response relationships for humans. Such risk factors have been derived for asbestos from existing epidemiological studies in which the exposures of the cohorts evaluated are based on a combination of analytical methods including, primarily, midget impinger and phase contrast microscopy (USEPA, 1986a). However, phase contrast microscopy (PCM) and midget impinger measurements provide only crude indices of exposure and do not necessarily track characteristics representing the biological activity of asbestos.

The traditional methods of characterizing exposure used in existing epidemiology studies have not proven adequate to develop a dose/response relationship that uniformly applies under all exposure circumstances. Published risk factors vary by a factor of 50 depending on the specific exposure setting studied (USEPA, 1986a). This may be due to one or a combination of several factors that include errors and uncertainty in the quantification of mortality, errors and uncertainty in the quantification of exposure, differences in the size distributions of structures, and differences in the way that the various analytical techniques respond to dusts containing varied structure size distributions. Due to these limitations, such risk factors should be viewed as order-of-magnitude estimates at best. Despite such limitations, however, published risk factors (properly modified by considering other factors that have been shown to define biological activity) represent the only reasonable tools currently available for estimating risks from measured asbestos exposures.

Published risk factors are generally expressed in terms of PCM counts. When data from different types of analytical techniques were combined in the existing epidemiology studies, results were generally normalized and converted to PCM equivalent counts before completing the evaluation. However, despite the fact that published risk factors are generally expressed in terms of PCM equivalent counts, quantification of the PCM visible fraction of asbestos in current measurements does not provide the best index for comparing current asbestos measurements to existing risk factors.

It has been shown that the use of identical methods for measuring asbestos exposure in two different settings (such as two different factories) is not sufficient to assure the direct comparability of the two measurements, at least in terms of comparing risks (Chesson et al 1989a). Measurements from two different exposure settings, where the characteristics of asbestos dusts may vary, only indicate relative risk when the characteristics of asbestos that determine biological activity are measured directly. Other measures of asbestos exposure that do not relate directly to biological activity may not remain proportional to the characteristics that determine biological activity in different exposure settings. Therefore, PCM counts and other measures of exposure that do not relate directly to biological activity do not provide results from different exposure settings that are proportional to risk.

The best approach for monitoring current asbestos exposures to estimate risks is to measure the characteristics that relate directly to biological activity and to compare the results to existing risk factors using adjustment factors that relate PCM counts from the historical studies to proper exposure characteristics representative of the historical exposures. Such adjustment factors may be estimated from published asbestos characterizations determined using transmission electron microscopy (TEM) by pairing characterizations from specific exposure settings with existing risk factors derived from similar exposure settings. Alternately, exposure settings (selected for their similarity to settings originally studied to derive the existing risk factors) could be re-characterized using the method presented in this report to provide improved adjustment factors.

The approach recommended in the last paragraph for estimating risks presupposes a concise definition of asbestos characteristics that relate to biological activity. Because the definition of such characteristics remains a subject of controversy, the method presented in this report is designed to retain sufficient information from each analysis to span the range of characteristics currently considered likely to impact biological activity. Thus, results may be easily re-evaluated in the future.

### 3.3. MORPHOLOGY OF ASBESTOS DUSTS

The size distributions of asbestos structures are not the only characteristics of asbestos exposure that are potentially critical to biological activity. This is because asbestos structures will always be accompanied by other materials as components of dust, even in the workplace.

The major components of dust in all environments (occupational and environmental) are nonfibrous, isometric particles. Fibrous structures consistently represent a minor fraction of total dust. In addition, even among fibrous structures, asbestos represents a variable fraction of the total present. The relationship between these fractions is depicted in Figure 3.1.

Figure 3.1 is a Venn diagram depicting the universe of particles present in dust. Fibrous structures represent a minor fraction of total dust. Long fibrous structures represent a subset of total fibrous structures. Optically visible fibrous structures, those that are visible using a phase contrast microscope (optical microscope), represent a subset of long fibrous structures. The importance of the optical fraction of asbestos dusts is addressed in Section 3.2. Although not depicted, each of the subsets of fibrous structures could potentially be further subdivided into individual fibers, bundles, clusters, and/or matrices.

Particles composed of asbestiform minerals represent an independent subset of total particles that is distinct from fibrous materials. The overlap between asbestiform minerals and fibrous structures represents the fraction of fibrous structures that are asbestos. Correspondingly, subsets of long, fibrous structures and optically visible, fibrous structures (the PCM equivalent fraction) are also composed of asbestos.

The large square in Figure 3.1 represents the fraction of respirable particles. Subsets of asbestiform particles, and the three fractions of fibrous structures are also respirable. The large circle at the bottom of the diagram represents the fraction of particles traditionally counted by midget impinger. Note the limited overlap with fibrous structures. The shaded area of Figure 3.1 represents the fraction of respirable structures that are fibrous. The cross-hatched area represents the fraction of asbestos dusts (the long structures) currently believed to be the most biologically active.

Figure 3.1 is a qualitative representation. The relative size and orientation of the various fractions depicted will change as a function of the occupational or environmental setting considered. For example, the overlap

between asbestiform structures and fibrous structures is expected to be much greater in asbestos occupational settings than in environmental settings. In this manner, Figure 3.1 is probably more representative of an environmental setting.

Only a very limited number of available studies address the issue of the fraction of dust particles composed of asbestos. This is because in general, either the analytical technique used in a study has been incapable of distinguishing asbestos from non-asbestos or, if the analytical technique was capable of distinguishing asbestos from non-asbestos, only asbestos particles are traditionally characterized. Table 3.1 presents data from the few studies where the fraction of asbestos particles in dust was considered.

Unfortunately, much of the data in Table 3.1 provide an indication of the fraction of total particles composed of asbestos, while it is the fraction of fibers composed of asbestos that is of principle interest. In some exposure settings, a measurement of the fraction of asbestos in total particles may simply reflect the fraction of total fibers in total particles with no manner of distinguishing asbestos from non-asbestos fibers.

The data presented in the table from Cherrie et al (1987) and from Altree-Williams and Preston (1985) indicate that, while the vast majority of fibers in an asbestos (textile) factory may be composed of asbestos (which may be representative of occupational settings), the fraction of fibers composed of asbestos in other exposure settings (including environmental settings) may vary over a wide range.

## 3.4. PUBLISHED SIZE DISTRIBUTIONS

In any exposure setting, airborne asbestos exists as a series of simple fibers and complex structures of varying length, width, and breadth. Historically, available analytical techniques were capable of detecting only a fraction of the population of asbestos structures existing in an exposure setting. Thus, only an index of exposure could be measured.

In the last 15 or 20 years, measurements with the superior resolving power and magnification of the transmission electron microscope (TEM) have allowed complete characterization of the distribution of sizes and morphologies of asbestos structures that exist within an exposure population. Comprehensive characterization of asbestos exposures by scanning electron microscope (SEM) have also been reported, although the visibility of SEM (resulting from the combined constraints on resolution and contrast) is not generally sufficient to detect the smallest and finest asbestos structures (Walton 1982).

Table 3.2 presents published asbestos size distributions that were characterized by electron microscopy. The first column of the table lists the exposure setting in which the distribution was measured. The second column provides the type of asbestos monitored. The next several columns provide

FIGURE 3.1: COMPONENTS OF RESPIRABLE PARTICLES

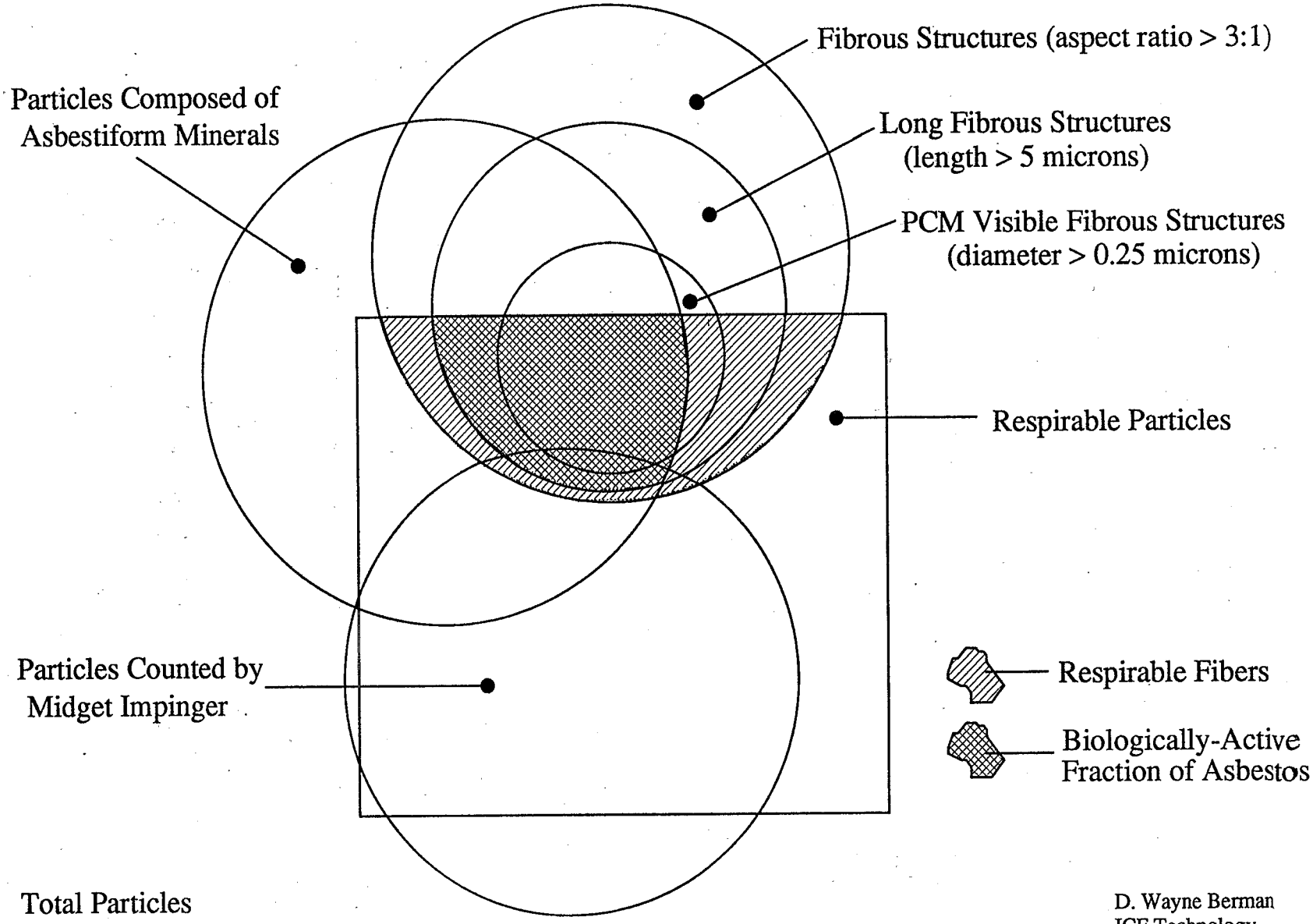


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TABLE 3.1: PUBLISHED MEASUREMENTS OF THE FRACTION OF ASBESTOS IN DUST

| Exposure Setting | Fiber Type | Analytical Method | Preparation Technique | Fraction of Asbestos in Shape Categories (% by count) of total particles | of total fibers | of optical fibers | Reference |
|---|---|---|---|---|---|---|---|
| Cape/Mine | Crc | PCM & K & TP | | 41 | | | (1) |
| Cape/Mill | Crc | | | 51 | | | |
| Tran/Mine | Crc | | | 28 | | | |
| Tran/Mill | Crc | | | 59 | | | |
| Mine | Ams | | | 33 | | | |
| Mill | Ams | | | 55 | | | |
| Mine | Chr | | | 45 | | | |
| Mill | Chr | | | 52 | | | |
| Txt | Chr&Crc | TEM | D/M | | 82 | 94 | (2) |
| Txt/Out | PChr | | | | 21 | 41 | |
| Clearance | PAms | | | | 48 | 59 | |
| Bldg w/Insl | PAms | | | | 15 | 36 | |
| Urban | No re | | | | 0 | 0 | |
| Bldg | Chr | | | | | 20 | (3) |

Table 3.1 (continued)

Key

Exposure Settings:

| | |
|---|---|
| AC | Dust from asbestos/cement sanding |
| AP | Asbestos production plant |
| ATB | Dust from asbestos tape and board |
| BC | Dust from a brake and clutch plant |
| Bag | Dust from bagging |
| Cape | Capetown, South Africa |
| Cstr | Dust from construction |
| CP | Asbestos/cement pipe manufacture |
| Card | Dust from carding |
| Crsh | Dust from crushing |
| Cut | Dust from cutting |
| Dump | Dust from raw material dumping |
| Dry | Dust from drying |
| Fin | Dust from fiber finishing |
| FP | Friction products plant |
| Fprp | Dust from fiber preparation |
| Form | Dust from forming |
| Grnd | Dust from grinding |
| Insl | Site where insulation is applied |
| Mill | Dust from asbestos milling |
| Mine | Dust from asbestos mining |
| Mix | Dust from fiber mixing |
| Orest | Dust from ore storage |
| Out | Outside Plant |
| Pinsl | Pipe insulation manufacturers |
| Que | Quebec, Canada |
| RR | Railroad factory |
| Spec | Nonstandard clearance, etc. |
| Spin | Dust from fiber spinning |
| Tran | Transvaal, South Africa |
| Twst | Dust from fiber twisting |
| Txt | Dust from textile plant |
| UICC | Sample of UICC standards |
| Weav | Dust from weaving |

Fiber Types:

| | |
|---|---|
| Actn | Actinolite |
| Amph | Non-amosite amphibole |
| Ams | Amosite |
| Chr | Chrysotile |
| Crc | Crocidolite |
| Pr | Predominantly |

Preparation Techniques:

| | |
|---|---|
| D | Direct |
| I | Indirect |
| M | Mixed cellulose ester filter |
| N | Polycarbonate filter |
| Spec | Nonstandard preparation |

Analytical Methods:

| | |
|---|---|
| PCM | Phase contrast microscopy |
| K | Konimeter |
| MI | Midget impinger |
| SEM | Scanning electron microscopy |
| TEM | Transmission electron microscopy |
| TP | Thermal precipitator |

Miscellaneous:

| | |
|---|---|
| ( ) | Extrapolated from data |
| (T) | Total fibers |

TABLE 3.1 (continued)

REFERENCES

1. Gibbs and du Toit, 1979
2. Cherrie et al., 1987
3. Altree-Williams and Preston, 1985

information on the techniques used in the study to generate the size data. These columns list, respectively, the method used to prepare the samples, the type of microscope used to determine the size distribution, and the magnification employed. The next two columns present reported median lengths and median diameters of the fibers in the distribution characterized.

Size distributions are presented in Table 3.2 as the number percent of total asbestos structures for structures in each of a series of size ranges that are listed in the headings of the next several columns. The column headed "percent with lengths less than 5 $\mu$m" represents the fraction of short structures detected within the particular distribution listed. The next two columns list structures with lengths greater than 5 $\mu$m. The second of these columns presents the fraction of structures with diameters (widths) greater than 0.25 $\mu$m, which represent the fraction of PCM equivalent structures detected. Since 0.25 $\mu$m is the limit of resolution generally quoted for phase contrast microscopy (PCM), this column represents the fraction of structures that would be detected by optical microscopy[1]. The first of the two columns representing structures longer than 5 $\mu$m lists structures that are too thin to be resolved by optical microscopy: the long, thin structures. The sum of PCM equivalent structures and long, thin structures represent the fraction of structures defined as long structures.

The four columns to the right of long, thick structures on Table 3.2, provide number percent fractions for two other ranges of length within the component represented by long structures. These are shown because it is possible that the most biologically active asbestos structures may be more restricted than indicated in Section 3.1 and 3.2 and Figure 3.1. Each length range is also subdivided into those resolvable and those unresolvable by optical microscopy (PCM).

Because chrysotile dusts have been characterized most frequently among the published fiber size distributions, the data for chrysotile (grouped by exposure setting) are presented in Table 3.3. Table 3.3 includes size distributions for dusts characterized both as "chrysotile" and as "predominantly chrysotile", that includes approximately half of all of the distributions presented in Table 3.2. The format of Table 3.3 is identical to that used in Table 3.2.

Although data are compressed and extrapolated in Table 3.3 (and Table 3.2) so that results from each study could be presented in a common format, the gross features of chrysotile size distributions can be summarized based on the detailed results of the studies listed. Where such studies provide sufficient detail, similar features are found for all chrysotile dusts

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[1] Note that the limit of resolution of PCM depends on a variety factors including the type and the condition of the instrument. The typical ranges of resolution quoted for PCM is between 0.2 and 0.4 $\mu$m. The impact of such variation on the interpretation of results is considered in later sections.

TABLE 3.2: STRUCTURE SIZE DISTRIBUTIONS MEASURED IN VARIOUS ENVIRONMENTS FOR SEVERAL ASBESTOS MINERALS

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Magnification | Median Length | Median Diameter | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Cape/Mine | Crc | PCM | D/N | – | 0.95 | 0.08 | 96 | 3.72 | 0.7 | 3.02 | 0.57 | 0.7 | 0.13 | (1) |
| Cape/Bag | Crc | & | | | 1.16 | 0.08 | 93 | 5.88 | 1.2 | 5.1 | 1.09 | 0.78 | 0.11 | |
| Tran/Mine | Ams | TEM | | | 1.83 | 0.26 | 88 | 3.85 | 8.33 | 3.42 | 7.03 | 0.43 | 1.30 | |
| Tran/Bag | Ams | | | | 2.53 | 0.26 | 77 | 5.56 | 17.8 | 4.42 | 12.6 | 1.14 | 5.2 | |
| Que/Mine | Chr | | | | 0.34 | 0.06 | 99 | 0.40 | 0.2 | 0.36 | 0.7 | 0.04 | 0.13 | |
| Que/Bag | Chr | | | | 0.55 | 0.06 | 96 | 2.06 | 1.72 | 1.56 | 1.31 | 0.5 | 0.41 | |
| AP/Dump | Ams | SEM | D/N | 2500 | 3.9 | 0.41 | 54 | (3) | (43) | (3) | (27) | – | (16) | (2) |
| Ins/App | Ams | | | 2500 | 2.5 | 0.37 | 69 | (2) | (29) | (1.5) | (23) | – | (6) | |
| AP/Dump | Crc | | | 6000 | 2.5 | 0.25 | 79 | 3 | (18) | (3) | (14) | – | (4) | |
| Mill/Dry | Chr | | | 6000 | 1.25 | 0.17 | 88.9 | 1.5 | 10 | (1.5) | (7) | – | (3) | |
| Mill/Bag | Chr | | | 6000 | 1.35 | 0.16 | 95.2 | 1 | (4) | – | (3) | (1) | (1) | |
| Txt/Card | Chr | | | 6000 | 1.0 | 0.15 | 97.7 | 0.40 | (2.1) | (0.4) | (1) | – | (1) | |
| Cape/Mine | Crc | TEM | D/M,N | 400 | 0.95 | 0.07 | 95.9 | 3.33 | 0.71 | 2.57 | 0.56 | 0.76 | 0.15 | (3) |
| Cape/Orest | | & | | to | 1.07 | 0.10 | 93.6 | – | – | – | – | – | – | |
| Mill/Crsh | | SEM | | 15,000 | 1.00 | 0.08 | 96.4 | – | – | – | – | – | – | |
| Mill/Bag | | & | | | 1.16 | 0.09 | 92.9 | 5.89 | 1.21 | 5.11 | 1.1 | 0.78 | 0.11 | |
| AP/Dump | | PCM | | | 0.91 | 0.08 | 98.1 | 1.41 | 0.44 | 1.27 | 0.38 | 0.14 | 0.06 | |
| AP/Mix | | | | | 1.25 | 0.10 | 93.9 | – | – | – | – | – | – | |
| AP/Cut | | | | | 0.50 | 0.04 | 99 | – | – | – | – | – | – | |

TABLE 3.2 (CONTINUED)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Magnification | Median Length | Median Diameter | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Special | Ams | PCM & SEM | D/Spec | 400 to 10,000 | | | 21-40 | 52 | 81 | 26 | 37 | 26 | 44 | (4) |
| Special | | | | | | | 57 | (43) | | 26 | | 17 | | |
| Special | | | | | | | 50 | 50 | | 26 | | 24 | | |
| UICC | | | | | | | 77 | 23 | | 21 | | 2 | | |
| Cape/Mine | Crc | PCM & KON | – | – | | | 88 | | | | | | | (5) |
| Cape/Mill | Crc | | | | | | 84 | | | | | | | |
| Tran/Mine | Crc | | | | | | 82 | | | | | | | |
| Tran/Mill | Crc | | | | | | 73 | | | | | | | |
| Mine | Ams | | | | | | 91 | | | | | | | |
| Mill | Ams | | | | | | 65 | | | | | | | |
| Mine | Chr | | | | | | 93 | | | | | | | |
| Mill | Chr | | | | | | 87 | | | | | | | |
| Txt/Fprp | Chr | PCM & TEM | – | – | 1.4 | | 96 | | | | | | | (6) |
| Txt/Spin | | | | | 1.0 | | 98 | | | | | | | |
| FP/Mix | | | | | 0.9 | | 98 | | | | | | | |
| FP/Grnd | | | | | 0.8 | | 98 | | | | | | | |
| CP/Mix | | | | | 0.9 | | 98 | | | | | | | |
| CP/Fin | | | | | 0.7 | | 99 | | | | | | | |

TABLE 3.2 (CONTINUED)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Magnification | Median Length | Median Diameter | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Txt/Fprep | Chr | PCM | D/M | 900 | | | 79 | 7.9 | 12.8 | 4.5 | 5.7 | 3.4 | 7.1 | (7) |
| Txt/Twst | | & | | to | | | 83.4 | 8.0 | 8.6 | 3.6 | 2.9 | 4.3 | 5.7 | |
| Txt/Weav | | TEM | | 17,000 | | | 81.2 | 9.0 | 9.7 | 4.9 | 3.1 | 4.2 | 6.6 | |
| FP/Mix | | | | | | | 90.4 | 6.3 | 3.4 | 2.9 | 1.2 | 3.4 | 2.2 | |
| FP/Form | | | | | | | 90.7 | 5.9 | 3.4 | 3.2 | 1.5 | 2.8 | 2.0 | |
| FP/Fin | | | | | | | 83.8 | 6.3 | 9.9 | 4.2 | 3.4 | 2.0 | 6.5 | |
| CP/Mix | | | | | | | 87.6 | 6.0 | 6.4 | 3.3 | 3.1 | 2.7 | 3.3 | |
| CP/Form | | | | | | | 90.7 | 6.4 | 2.9 | 3.8 | 1.4 | 2.5 | 1.6 | |
| CP/Fin | | | | | | | 95.0 | 3.0 | 2.1 | 2.0 | 0.8 | 0.9 | 1.2 | |
| Pinsl/Mix | Ams | | | | | | 59.2 | 8.9 | 32.0 | 5.9 | 15.0 | 2.9 | 16.9 | |
| Pinsl/Form | Ams | | | | | | 63.2 | 9.4 | 27.4 | 6.1 | 11.9 | 3.2 | 15.5 | |
| Pinsl/Fin | Ams | | | | | | 65.1 | 6.6 | 28.4 | 6.1 | 14.4 | 0.4 | 14.0 | |
| Que/ Out/Mine | Chr | TEM | I | | | | 92.86 | 7.14 | 0.00 | | | | | (8) |
| | Chr | TEM | D | | | | 84.54 | 3.54 | 11.92 | | | | | |
| BC | Chr | PCM | D/M | 500 | | | 44 | (<6.1) | (49.9) | | | | | (9) |
| RR/ATB | | & | | to | | | 75 | (<2.5) | (22.5) | | | | | |
| RR/AC | | TEM | | 10,000 | | | 78 | (<2.4) | (19.6) | | | | | |

TABLE 3.2 (CONTINUED)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Magnification | Median Length | Median Diameter | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Cstr | Chr | PCM | D/M | | | | 86.2 | 8.7 | 5.1 | | | | | (10) |
| Bag | | & | | | | | 94.3 | 1.9 | 3.8 | | | | | |
| Cut | | STEM | | | | | 53.4 | 13.3 | 33.3 | | | | | |
| Cut | | | | | | | 87.5 | 10.2 | 2.4 | | | | | |
| Mill/Crsh | | | | | | | 91.2 | 2.0 | 6.8 | | | | | |
| Mill/Crsh | | | | | | | 97.9 | 0.2 | 1.9 | | | | | |
| Mill/Crsh | | | | | | | 88.9 | 4.6 | 6.5 | | | | | |
| Crsh | | | | | | | 83.1 | 4.8 | 12.1 | | | | | |
| CP/Mix | | | | | | | 97.8 | 0.2 | 2.0 | | | | | |
| Clearance | PAms | | | | | | 68.9 | 11.7 | 19.5 | | | | | |
| Clearance | PAms | | | | | | 65.2 | 11.6 | 23.2 | | | | | |
| Insl/Rmvl | PChr | | | | | | 64.2 | 10.2 | 25.6 | | | | | |
| Insl/Rmvl | PAms | | | | | | 66.1 | 12.7 | 21.2 | | | | | |
| Mine | Amph | | | | | | 96.9 | 0 | 3.1 | | | | | |
| Mine | Amph | | | | | | 85.3 | 0 | 14.7 | | | | | |
| Crsh | Actn | | | | | | 87.4 | 0 | 12.6 | | | | | |
| Crsh | Actn | | | | | | 87.6 | 0 | 12.4 | | | | | |

TABLE 3.2 (CONTINUED)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Magnification | Median Length | Median Diameter | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Laboratory | ChrB | SEM | I/M | | | | 69 | | | | | | | (11) |
| | ChrB | TEM | | | | | 89 | | | | | | | |
| | Ams | SEM | | | | | 74 | | | | | | | |
| | Ams | TEM | | | | | 84 | | | | | | | |
| | Crc | SEM | | | | | 88 | | | | | | | |
| | Crc | TEM | | | | | 93 | | | | | | | |
| | Trm | SEM | | | | | 50 | | | | | | | |
| | Trm | TEM | | | | | 59 | | | | | | | |
| Txt | Chr & Crc | SEM | D/M | | | | 78 (T) | | | | | | | |
| Txt | | TEM | | | | | 60 (T) | | | | | | | |
| Txt/Out | PChr | SEM | | | | | 81 (T) | | | | | | | |
| Txt/Out | PChr | TEM | | | | | – | | | | | | | |
| Clearance | PAms | SEM | | | | | 66 (T) | | | | | | | |
| Clearance | PAms | TEM | | | | | 82 (T) | | | | | | | |
| Bldg w/Insl | PAms | SEM | | | | | 63 (T) | | | | | | | |
| Bldg w/Insl | PAms | TEM | | | | | (91)(T) | | | | | | | |
| Urban | None | SEM | | | | | 69 (T) | | | | | | | |
| Urban | None | TEM | | | | | – | | | | | | | |

TABLE 3.2 (CONTINUED)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Magnification | Median Length | Median Diameter | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Mine | Ams | PCM | | 10,000 | | | 35.5 | | | | | | | (12) |
| Crsh | | & | | | | | 46.4 | | | | | | | |
| Bag | | EM | | | | | 55.8 | | | | | | | |
| Assay | | | | | | | 66.1 | | | | | | | |
| Mine | Chr | | | | | | 56.6 | | | | | | | |
| BC | | | | | | | 45.5 | | | | | | | |
| Mill | | | | | | | 61.4 | | | | | | | |
| Mine | Crc | | | | | | 77.1 | | | | | | | |
| Mill | Crc | | | | | | 63.7 | | | | | | | |
| Mill | Chr | PCM | | | | | 93.7 | (6.2) | (0.11) | | | | | (13) |
| Mill | | & | | | | | 96.1 | (3.9) | (0.02) | | | | | |
| Mill | | TEM | | | | | 98.5 | (1.5) | (0.01) | | | | | |
| Mill | | | | | | | 98.6 | (1.4) | (0.06) | | | | | |
| BC/Card | PChr | | | | | | 98.7 | (1.3) | (0.04) | | | | | |
| BC | | | | | | | 97.1 | (2.9) | (0.05) | | | | | |
| CP | | | | | | | 98.6 | (1.4) | (0.03) | | | | | |
| CP | | | | | | | 97.5 | (2.5) | (0.06) | | | | | |
| Txt | | | | | | | 87.5 | (12.4) | (0.1) | | | | | |
| Txt | | | | | | | 94.4 | (5.5) | (0.08) | | | | | |
| Txt | | | | | | | 93.7 | (6.2) | (0.1) | | | | | |

Table 3.2 (continued)

Key

Exposure Settings:

| | |
|---|---|
| AC | Dust from asbestos/cement sanding |
| AP | Asbestos production plant |
| ATB | Dust from asbestos tape and board |
| BC | Dust from a brake and clutch plant |
| Bag | Dust from bagging |
| Cape | Capetown, South Africa |
| Cstr | Dust from construction |
| CP | Asbestos/cement pipe manufacture |
| Card | Dust from carding |
| Crsh | Dust from crushing |
| Cut | Dust from cutting |
| Dump | Dust from raw material dumping |
| Dry | Dust from drying |
| Fin | Dust from fiber finishing |
| FP | Friction products plant |
| Fprp | Dust from fiber preparation |
| Form | Dust from forming |
| Grnd | Dust from grinding |
| Insl | Site where insulation is applied |
| Mill | Dust from asbestos milling |
| Mine | Dust from asbestos mining |
| Mix | Dust from fiber mixing |
| Orest | Dust from ore storage |
| Out | Outside Plant |
| Pinsl | Pipe insulation manufacturers |
| Que | Quebec, Canada |
| RR | Railroad factory |
| Spec | Nonstandard clearance, etc. |
| Spin | Dust from fiber spinning |
| Tran | Transvaal, South Africa |
| Twst | Dust from fiber twisting |
| Txt | Dust from textile plant |
| UICC | Sample of UICC standards |
| Weav | Dust from weaving |

Fiber Types:

| | |
|---|---|
| Actn | Actinolite |
| Amph | Non-amosite amphibole |
| Ams | Amosite |
| Chr | Chrysotile |
| Crc | Crocidolite |
| Pr | Predominantly |

Preparation Techniques:

| | |
|---|---|
| D | Direct |
| I | Indirect |
| M | Mixed cellulose ester filter |
| N | Polycarbonate filter |
| Spec | Nonstandard preparation |

Analytical Methods:

| | |
|---|---|
| PCM | Phase contrast microscopy |
| K | Konimeter |
| MI | Midget impinger |
| SEM | Scanning electron microscopy |
| TEM | Transmission electron microscopy |
| TP | Thermal precipitator |

Miscellaneous:

| | |
|---|---|
| ( ) | Extrapolated from data |
| (T) | Total fibers |

TABLE 3.2 (continued)

REFERENCES


1. Gibbs and Hwang, 1980.
2. Gibbs and Hwang, 1975.
3. Hwang and Gibbs, 1981.
4. Beckett and Jarvis, 1979.
5. Gibbs and du Toit, 1979.
6. Lynch et al., 1970.
7. Dement and Harris, 1979.
8. Sebastien et al., 1984.
9. Marconi et al., 1984.
10. Snyder et al., 1987.
11. Cherrie et al., 1987.
12. Rendall and Skikne, 1980.
13. Winer and Cossette, 1979.

analyzed. Briefly, the distribution of structure lengths is unimodal and skewed with the mode occurring between 0.8 and 1.2 $\mu$m. The tail of the distribution extends out so that longer structures are present, but at decreasing frequencies. Structures longer than 5 $\mu$m constitute no more than 25% of the total and frequently constitute less than 5%. Structures longer than 10 $\mu$m constitute no more than half of the concentration of structures longer than 5 $\mu$m and frequently represent less than 2% of the total structures counted. Thus, representative counts of longer structures can only be guaranteed if counting procedures direct that long structures be counted independently from short structures and that 5 to 10 times as much area is scanned for the count of long structures. Such a procedure is termed statistically-balanced counting (Sebastien et al, 1982).

The reported magnitude of the fraction of short structures needs to be addressed with caution. Because the distribution of structures peaks in the vicinity of 1 $\mu$m and on the order of one half of the short structures are shorter than 1 $\mu$m, the relative number of short structures in a total distribution is extremely sensitive to the minimum size of the structures characterized. For example, counting all structures longer than 0.2 $\mu$m in length is likely to yield a structure size distribution where structures longer than 5 $\mu$m constitute less than 5% of the total while the same distribution truncated at 1 $\mu$m minimum length will yield contributions from long structures of 20%. The lower length limit counted is not reported in the majority of these studies. However, those where the limit is known all show short structure contributions greater than 90% (with corresponding contributions by long structures of less than 10%).

Diameters for chrysotile structures also vary. The median diameter of a typical structure less than 5 $\mu$m in length lies between 0.02 and 0.03 $\mu$m and virtually all of the structures less than 5 $\mu$m long have diameters less than 0.05 $\mu$m. Mean diameters increase with increasing length, but the increase is not proportional, so that the aspect ratios of long chrysotile structures are much larger than the aspect ratios of short structures. The thinnest chrysotile structures exhibit diameters on the order of 0.02 $\mu$m.

The data in Tables 3.2 and 3.3 must be interpreted carefully. Although the majority of the listed studies employed TEM to derive structure size distributions, lack of standardization of sample preparation techniques, of rules for counting and characterizing structures, and of the criteria used for establishing the mineralogy of counted structures potentially contribute to analytical variation between the studies (see Sections 6.1 and 6.4). For this reason, comparisons of distributions within a study are more reliable than comparisons between studies. In fact, the data in Table 3.2 clearly indicate that at least 2 studies are quantitatively different than the other studies. For the same fiber type in similar exposure settings, Rendall and Skikne (1980) and Marconi et al (1984) consistently report distributions containing a greater fraction of long structures than the other studies. Unfortunately, none of the studies contain sufficient documentation of the methods used to derive size distributions to determine the cause of the apparent discrepancies.

TABLE 3.3: STRUCTURE SIZE DISTRIBUTIONS MEASURED IN VARIOUS ENVIRONMENTS FOR CHRYSOTILE ASBESTOS

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Median Length | Median Diameter | Magnification | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| Que/Mine | Chr | PCM/TEM | D/ | 0.34 | 0.06 | | 99 | 0.40 | 0.2 | 0.36 | 0.7 | 0.04 | 0.13 | 1 |
| Mine | | PCM/K, TP | D/ | | | | 93 | | | | | | | 5 |
| Que | | TEM | I | | | | 92.86 | 7.14 | 0.00 | | | | | 8 |
| Out/Mine | | TEM | D | | | | 84.54 | 3.54 | 11.92 | | | | | 8 |
| Mine | | PCM/EM | | | | 10,000 | 56.6 | | | | | | | 12 |
| Mill/Dry | | SEM | D/N | 1.25 | 0.17 | 6000 | 88.9 | 1.5 | 10 | (1.5) | (7) | | (3) | 2 |
| Mill/Crsh | | PCM/STEM | D/M | | | 10-70,000 | 91.2 | 2.0 | 6.8 | | | | | 10 |
| Mill/Crsh | | PCM/STEM | D/M | | | | 97.9 | 0.2 | 1.9 | | | | | 10 |
| Mill/Crsh | | PCM/STEM | D/M | | | | 88.9 | 4.6 | 6.5 | | | | | 10 |
| Crsh | | PCM/STEM | D/M | | | | 83.1 | 4.8 | 12.1 | | | | | 10 |
| Mill | | PCM/K,TP | D/ | | | | 87 | | | | | | | 5 |
| Mill | | PCM/TEM | | | | | 93.7 | (6.2) | (0.11) | | | | | 13 |
| Mill | | | | | | | 96.1 | (3.9) | (0.02) | | | | | 13 |
| Mill | | | | | | | 98.5 | (1.5) | (0.01) | | | | | 13 |
| Mill | | | | | | | 98.6 | (1.4) | (0.06) | | | | | 13 |
| Mill | | PCM & EM | | | | 10,000 | 61.4 | | | | | | | 12 |
| Lab | ChrB | TEM | I/M | | | | 89 (T) | | | | | | | 11 |
| Lab | ChrB | SEM | I/M | | | | 69 (T) | | | | | | | 11 |

TABLE 3.3 (CONTINUED)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Median Length | Median Diameter | Magnification | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5 μm | L > 5 μm D < 0.25 μm | L > 5 μm D > 0.25 μm | L 5 - 10 μm D < 0.25 μm | L 5 - 10 μm D > 0.25 μm | L > 10 μm D < 0.25 μm | L > 10 μm D > 0.25 μm | |
| FP/Mix | Chr | PCM/TEM | D/M | | | 900 | 90.4 | 6.3 | 3.4 | 2.9 | 1.2 | 3.4 | 2.2 | 7 |
| FP/Form | | | D/M | | | to | 90.7 | 5.9 | 3.4 | 3.2 | 1.5 | 2.8 | 2.0 | 7 |
| FP/Fin | | | D/M | | | 17,000 | 83.8 | 6.3 | 9.9 | 4.2 | 3.4 | 2.0 | 6.5 | 7 |
| (FP)/BC | | | D/M | | | 10,000 | 44 | (<6.1) | (49.9) | | | | | 9 |
| FP/Mix | | | | 0.9 | | | 98 | | | | | | | 6 |
| FP/Grnd | | | | 0.8 | | | 98 | | | | | | | 6 |
| BC/Card | | | | | | | 98.7 | (1.3) | (0.04) | | | | | 13 |
| BC | | | | | | | 97.1 | (2.9) | (0.05) | | | | | 13 |
| BC | | PCM & EM | | | | 10,000 | 45.5 | | | | | | | 12 |
| RR/ATB | | PCM/TEM | D/M | | | 10,000 | 75 | (<2.5) | (22.5) | | | | | 9 |
| RR/AC | | | D/M | | | 10,000 | 78 | (<2.4) | (19.6) | | | | | 9 |
| CP/Mix | | | D/M | | | 900 | 87.6 | 6.0 | 6.4 | 3.3 | 3.1 | 2.7 | 3.3 | 7 |
| CP/Form | | | D/M | | | to | 90.7 | 6.4 | 2.9 | 3.8 | 1.4 | 2.5 | 1.6 | 7 |
| CP/Fin | | | D/M | | | 17,000 | 95.0 | 3.0 | 2.1 | 2.0 | 0.8 | 0.9 | 1.2 | 7 |
| CP/Mix | | | | 0.9 | | | 98 | | | | | | | 6 |
| CP/Fin | | | | 0.7 | | | 99 | | | | | | | 6 |
| CP/Mix | | PCM/STEM | D/M | | | | 97.8 | 0.2 | 2.0 | | | | | 10 |
| CP | | PCM/TEM | | | | | 98.6 | (1.4) | (0.03) | | | | | 13 |
| CP | | PCM/TEM | | | | | 97.5 | (2.5) | (0.06) | | | | | 13 |

TABLE 3.3 (Continued)

| Exposure Settings | Fiber Type | Analytical Methods | Preparation Techniques | Median Length | Median Diameter | Magnification | Fraction of Asbestos Fibers in Size Categories (% by Count) | | | | | | | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L < 5μm | L > 5μm D < 0.25μm | L > 5μm D > 0.25μm | L 5 - 10μm D < 0.25μm | L 5 - 10μm D > 0.25μm | L > 10μm D < 0.25μm | L > 10μm D > 0.25μm | |
| Bag | Chr | PCM/STEM | D/M | | | 10-70,000 | 94.3 | 1.9 | 3.8 | | | | | 10 |
| Que/Bag | | TEM/PCM | D/ | 0.55 | 0.06 | | 96 | 2.06 | 1.72 | 1.36 | 1.31 | 0.50 | 0.41 | 1 |
| Mill/Bag | | SEM | D/N | 1.35 | 0.16 | 6000 | 95.2 | 1 | (4) | | (3) | (1) | (1) | 2 |
| Cut | | STEM/PCM | D/M | | | 10-70,000 | 53.4 | 13.3 | 33.3 | | | | | 10 |
| Cut | | STEM/PCM | D/M | | | 10-70,000 | 87.5 | 10.2 | 2.4 | | | | | 10 |
| Cstr | | STEM/PCM | D/M | | | 10-70,000 | 86.2 | 8.7 | 5.1 | | | | | 10 |
| Insl/Rmvl | PChr | STEM/PCM | D/M | | | | 64.2 | 10.2 | 25.6 | | | | | 10 |
| Txt/Card | Chr | SEM | D/N | 1.0 | 0.15 | 6000 | 97.7 | 0.4 | (2.1) | (0.4) | (1) | | (1) | 2 |
| Txt/Fprp | | TEM/PCM | D/M | | | 900 | 79 | 7.9 | 12.8 | 4.5 | 5.7 | 3.4 | 7.1 | 7 |
| Txt/Twst | | | | | | to | 83.4 | 8.0 | 8.6 | 3.6 | 2.9 | 4.3 | 5.7 | 7 |
| Txt/Weav | | | | | | 17,000 | 81.2 | 9.0 | 9.7 | 4.9 | 3.1 | 4.2 | 6.6 | 7 |
| Txt/Fprp | | | | 1.4 | | | 96 | | | | | | | 6 |
| Txt/Spin | | | | 1.0 | | | 98 | | | | | | | 6 |
| Txt/Out | PChr | SEM | | | | | 81(T) | | | | | | | 11 |
| Txt/Out | PChr | TEM | | | | | --- | | | | | | | 11 |
| Txt | Chr | TEM/PCM | | | | | 87.5 | (12.4) | (0.1) | | | | | 13 |
| Txt | | | | | | | 94.4 | (5.5) | (0.08) | | | | | 13 |
| Txt | | | | | | | 93.7 | (6.2) | (0.1) | | | | | 13 |

Table 3.3 (continued)

Key

Exposure Settings:

| | |
|---|---|
| AC | Dust from asbestos/cement sanding |
| AP | Asbestos production plant |
| ATB | Dust from asbestos tape and board |
| BC | Dust from a brake and clutch plant |
| Bag | Dust from bagging |
| Cape | Capetown, South Africa |
| Cstr | Dust from construction |
| CP | Asbestos/cement pipe manufacture |
| Card | Dust from carding |
| Crsh | Dust from crushing |
| Cut | Dust from cutting |
| Dump | Dust from raw material dumping |
| Dry | Dust from drying |
| Fin | Dust from fiber finishing |
| FP | Friction products plant |
| Fprp | Dust from fiber preparation |
| Form | Dust from forming |
| Grnd | Dust from grinding |
| Insl | Site where insulation is applied |
| Mill | Dust from asbestos milling |
| Mine | Dust from asbestos mining |
| Mix | Dust from fiber mixing |
| Orest | Dust from ore storage |
| Out | Outside Plant |
| Pinsl | Pipe insulation manufacturers |
| Que | Quebec, Canada |
| RR | Railroad factory |
| Spec | Nonstandard clearance, etc. |
| Spin | Dust from fiber spinning |
| Tran | Transvaal, South Africa |
| Twst | Dust from fiber twisting |
| Txt | Dust from textile plant |
| UICC | Sample of UICC standards |
| Weav | Dust from weaving |

Fiber Types:

| | |
|---|---|
| Actn | Actinolite |
| Amph | Non-amosite amphibole |
| Ams | Amosite |
| Chr | Chrysotile |
| Crc | Crocidolite |
| Pr | Predominantly |

Preparation Techniques:

| | |
|---|---|
| D | Direct |
| I | Indirect |
| M | Mixed cellulose ester filter |
| N | Polycarbonate filter |
| Spec | Nonstandard preparation |

Analytical Methods:

| | |
|---|---|
| PCM | Phase contrast microscopy |
| K | Konimeter |
| MI | Midget impinger |
| SEM | Scanning electron microscopy |
| TEM | Transmission electron microscopy |
| TP | Thermal precipitator |

Miscellaneous:

| | |
|---|---|
| ( ) | Extrapolated from data |
| (T) | Total fibers |

TABLE 3.3 (continued)

REFERENCES


1. Gibbs and Hwang, 1980.
2. Gibbs and Hwang, 1975.
3. Hwang and Gibbs, 1981.
4. Beckett and Jarvis, 1979.
5. Gibbs and du Toit, 1979.
6. Lynch et al., 1970.
7. Dement and Harris, 1979.
8. Sebastien et al., 1984.
9. Marconi et al., 1984.
10. Snyder et al., 1987.
11. Cherrie et al., 1987.
12. Rendall and Skikne, 1980.
13. Winer and Cossette, 1979.

The extent that the published size distributions presented in Tables 3.2 and 3.3 are representative of the exposure settings examined also needs to be considered when comparing results between studies. In most cases, each of the distributions presented is derived from a single grab sample. Since it is not known to what extent the size characteristics of an asbestos dust in a particular exposure setting vary over time, it is unclear how to relate single grab samples to the general features of an asbestos dust in a particular exposure setting. Nevertheless, this is currently the only data available for providing an indication of the distribution of particle size in (environmental and occupational) asbestos dusts.

Despite the above caveats, the data in Table 3.2 clearly indicate that different asbestos mineral types tend to produce dusts with distinctive size characteristics. Amosite dusts appear to contain the greatest fraction of long and thick structures. Averaged over the 26 distributions for amosite and predominantly amosite dusts, 34% of total amosite structures are longer than 5 $\mu$m. This compares with 10% of the total structures in the 53 chrysotile and predominantly chrysotile dusts and 12% of the 18 crocidolite dusts. The difference between amosite and the other two mineral types is further exaggerated if Marconi et al (1984) and Rendall and Skikne (1980) are removed from the data set.

NOTE

The absolute magnitudes of the fraction of long structures in a distribution all represent upper limits and may be revised downward depending on the minimum lengths counted in each of the studies. However, the relative values (between mineral type) are less likely to be affected because the observed differences are also apparent within individual studies, including the Rendall and Skikne study.

Although chrysotile and crocidolite dusts appear to contain comparable length distributions, the data in Table 3.2 indicate that chrysotile dusts tend to contain thicker structures than crocidolite. For example, the fraction of dusts visible by optical microscopy (those thicker than 0.25 $\mu$m) represents an average of 9% of chrysotile dusts with a range of 1 to 50%. Only an average 4% of crocidolite dusts are visible by optical microscopy (range 1 to 18%). The range of diameters across chrysotile dusts may actually be narrower, if inter-study variation is considered. Removing the Marconi et al (1984) data and one apparent outlier from Snyder et al (1987), the range of the fraction of structures visible by optical microscopy in chrysotile dusts is reduced to between 1 and 18% with an average of 8%. Note that the Winer and Cossette (1979) data were not included in the above averages because thick structures were counted by optical microscopy in this study and such results cannot be compared directly with the TEM data presented for counts of total long structures.

As indicated previously, amosite dusts appear to contain the greatest fraction of thick structures. Between 8 and 43% of total amosite structures are thick enough to be visible by optical microscopy with an average 25% among

amosite dusts. Interestingly, the median diameters reported for dusts do not appear to track the fractions of dusts represented by specific ranges of thicknesses. This is not surprising and simply indicates that size ranges are heavily skewed so that the mean and the median of the distributions are widely separated.

Overall, the fraction of long structures in crysotile and crocidolite typically represent less than 25% of total structures. Approximately 20 to 70% of long chrysotile structures or 1 to 18% of total chrysotile structures are visible by optical microscopy. Although the data is sparse for crocidolite, approximately 10 to 50% of long crocidolite structures are visible by optical microscopy. In contrast, the majority of long amosite structures are visible optically and long structures typically represent up to 50% of the total number of amosite structures present. In general, therefore, the fraction of amosite dust visible by optical techniques is up to 5 times higher than for chrysotile or crocidolite, although all three show a wide range.

As indicated earlier, the magnitude of the range and average fractions of long structures and structures visible by optical microscopy reported for the various size distribution may be revised downward subject to the minimum size counted in the distribution. For example, assuming published size distributions could be adjusted so that structures longer than 0.2 $\mu$m were included uniformly, long structures likely represent less than 10% of total structures in chrysotile size distributions and 4% may represent a better average fraction of structures in chrysotile that are visible by optical microscopy. This is in contrast to the numerical averages for these size fractions (25% and 8%, respectively), which were calculated directly from the data and presented above. Similarly, only 2% of crocidolite structures may fall within the range visible by optical microscopy.

It must be emphasized that the published size distributions presented in Tables 3.2 and 3.3 do not generally indicate whether aggregates are included or excluded from the distribution presented. However, aggregates likely represent major components of all asbestos dusts found in the environment (see for example: Chatfield, 1985b). In other examples, data generated at the Atlas and Coalinga Superfund sites in California indicate that 50% of the structures collected in the vicinity of the Atlas and Coalinga mines are matrices or other aggregates. Data from the South Bay Superfund site in California indicate that aggregates represent between 10 and 30% of the structures found in fibrous dust at this site. In two studies, Sebastien et al (1984 and 1986) report that aggregates constitute 40% of the fibrous structures in asbestos dusts found in the vicinity of the asbestos mines in Quebec.

One observation concerning size distributions that relates to biological activity address the spread in apparent risk factors derived from different exposure settings (see Section 3.2). Variations in fiber size distributions in dusts from different exposure settings have been identified as one of several possible causes of the observed spread in reported risk factors from

published epidemiology studies (see for example: USEPA 1986a). However, such variations are not readily apparent in Table 3.3. Overall, a slight trend toward increasing length for structures in dusts from mining and milling (except for bagging), through bagging, to textiles appears when distributions for these exposure settings are averaged. Structures in dusts from friction products and asbestos/cement pipe both appear shorter than textiles, although they cannot be distinguished from either mining, milling, or bagging.

Within individual studies, trends relating asbestos structure dimensions to exposure setting should be easier to distinguish since inter-study effects do not interfere. The clearest trend appears in the data reported by Dement and Harris (1979) with structures in dusts increasing in length from asbestos/cement pipe, through friction products, to textiles. Such trends are not apparent in the data of Winer and Cossette (1979) or Snyder (1987). However, the representativeness of the samples in terms of the types of dusts generated in the exposure settings examined has not been addressed in any of the studies except for Dement and Harris. None of the other studies listed in Tables 3.2 and 3.3 contain sufficient data for a single mineral type over a broad enough range of exposure settings to address this question. To resolve issues concerning the quantification of the risk associated with asbestos exposure, additional structure size characterizations may need to be developed using a standardized analytical method.

## 3.5. REPRESENTATIVE ASBESTOS CONCENTRATIONS

A limited number of published studies contain measurements of ambient concentrations of asbestos. Unfortunately, the utility of the data from these studies is restricted by the use of different analytical techniques and methods of reporting so that the results from different studies generally are not directly comparable (Chatfield, 1983b). Another problem with the available database is that several of the studies report results in mass equivalent concentrations and it has been shown that mass concentrations do not generally correlate with structure counts at ambient concentrations (Chatfield, 1985c). The high mass measurements that sporadically occur within any set of ambient samples are frequently due to the presence of a single, large aggregate on the sample filter, while the overall structure count remains low.

Available ambient asbestos measurements are presented in Tables 3.4 and 3.5. Table 3.4 presents representative background concentrations in various environmental settings and Table 3.5 presents several representative concentrations near sources of contamination. To distinguish ranges of concentrations that likely represent natural background from anthropogenic contamination, both the range of concentrations and the median concentrations reported in each study are presented in the tables. The median concentrations probably are better to base judgments upon since they tend to smooth the effects of outliers that potentially skew the limits of the reported ranges.

To the extent that the data are available in each study, concentrations are expressed in the tables in three formats: total asbestos structures per

unit air volume, asbestos structures visible by optical microscopy (PCM equivalent) per unit air volume, and asbestos mass per unit air volume. As addressed in later sections of this report, each of these formats has particular advantages and disadvantages. As indicated in the first paragraph of this section, however, the concentration formats are virtually independent variables; correlations between these formats that have been reported in the literature have been weak at best.

Comparisons between results presented in each of these tables should be made only with extreme care. Although the data presented is entirely from studies employing transmission electron microscopy, comparisons between studies must address the type of preparation (direct or indirect) employed and the specific size fraction of asbestos counted. Unfortunately, documentation for several of the studies is insufficient to determine precisely what size fraction was counted and whether aggregates are included or excluded from the count.

A few of the studies referenced in Tables 3.4 and 3.5 address the question of aggregates and provide a qualitative indication of size distributions. Sebastien et al (1986) indicate that in and around the mines of Quebec, 14% of structures longer than 5 $\mu$m are aggregates. This corresponds closely to the fraction of the long structures that represents the PCM equivalent fraction. Several authors report that asbestos structures encountered as background at remote locations are all short (less than 5 $\mu$m) chrysotile fibers (see, for example, Chatfield, 1985c). It is unclear, however, whether the detection of only short structures is solely due to statistical limitations in the number of structures encountered (longer structures are one to two orders of magnitude less abundant than short structures in most chrysotile distributions) or whether such observations represent a valid phenomenon indicating that the detection of contamination representative of a anthropogenic source can be limited to searching for long structures.

Due to the severe limitations of this database, it is difficult to identify reasonable target concentrations that represent the boundary between natural background and anthropogenic contamination. However, certain generalizations are apparent. For example, the data in Tables 3.4 and 3.5 suggest that analysis of samples prepared by an indirect-transfer technique generally yield results that are higher than analyses of samples prepared by a direct-transfer technique. However, because the concentration of indirectly prepared samples can be optimized to facilitate analysis, it is the limiting concentrations observed on directly prepared samples that is of principle concern for development of the method.

Of the results identified as derived from directly prepared samples in Table 3.4, the range of median concentrations reported for total structures spans from less than 0.4 s/L to 6 s/L. Note that this range does not include the median of less than 0.01 reported in one study (Tuckfield et al 1988), which appears to lie outside the range of the majority of other studies. Results reported in studies where the preparation technique was not specified

## TABLE 3.4: PUBLISHED BACKGROUND CONCENTRATIONS OF ASBESTOS

| Environmental Setting | Median of Reported Concentrations [a] | | | Range of Reported Concentrations [b] | | | Conversion | Analytical Sensitivity [c] | Preparation Technique | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| | s/l | PCME/l | ng/m$^3$ | s/l | PCME/l | ng/m$^3$ | (PCME/l to ng/m$^3$) | | | |
| Outside Schools | 25 | (0.05) [d] | 0.12 | 0-2000 | — | 0-8 | — | — | indirect | ① |
| Outside Schools | 10 | 0.3 [d] | 0.08 | 0-100 | 0-8 [d] | 0-0.9 | 0.1-100, (4) [e,f] | — | indirect | ② |
| Outside Schools | 4 | — | 0.02 | 0-10 | — | 0-0.07 | — | — | indirect | ③ |
| Outside Schools | — | — | 0.5 | — | — | 0-100 | — | — | — | ④ |
| Outside Public Buildings | <0.01 | — | — | — | — | — | — | — | direct | ⑤ |
| Urban | 2 | <2 | 0.03 | 0-8 | 0-4 | 0-20 | 0.4 [g] | 2 | direct | ⑥ |
| Urban Toronto | 6 | <2 [h] | 0.07 | 0-45 | 0-4 [h] | 0-0.3 | — | 2 | direct | ⑥ |
| Urban Paris | — | — | 0.4 | — | — | 0.1-9 | — | — | indirect | ⑦ |
| Urban | — | — | 1 | — | — | 1-10 | — | — | — | ⑧ |
| Urban | — | — | — | 0-10 [i] | — | — | — | — | — | ⑨ |
| New York | — | — | 10 | — | — | 0-100 | — | — | — | ⑩ |
| US Cities | — | — | 1 | — | — | 0-50 | — | — | — | ⑪ |
| US Cities | — | — | 3 | — | — | 0-15 | — | — | — | ⑫ |
| Canadian Cities | — | 0.7 [h,j] | — | — | 0.6-0.9 [h,k] | — | — | — | indirect | ⑬ |
| Canadian Cities | — | 0.1 [h,j] | 1 [j] | — | 0-3 [h] | 0-6 | 0.1 | — | — | ⑭ |
| English Cities | — | — | — | — | — | 0.1-1 | — | — | — | ⑮ |
| German Cities | 3 [g] | — | — | 0-10 | — | — | — | — | — | ⑯ |
| Urban Switzerland | — | 0.4 [h,j] | 0.75 [h,j] | — | — | — | 0.5 | — | — | ⑰ |
| Upwind of Asbestos Plants | 0.2 | — | 0.03 | 0-11 | — | 0-170 | — | 0.3 | — | ⑱ |

TABLE 3.4 (CONTINUED)

| Environmental Setting | Median of Reported Concentrations [a] | | | Range of Reported Concentrations [b] | | | Conversion | Analytical Sensitivity [c] | Preparation Technique | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| | s/l | PCME/l | ng/m$^3$ | s/l | PCME/l | ng/m$^3$ | (PCME/l to ng/m$^3$) | | | |
| Suburban | 0.6 | <0.6 [d] | 0.003 | 0-6 | 0-2 | 0-9 | 0.2 [g] | 0.6 | direct | ⑥ |
| Remote | <0.4 | <0.4 | — | 0-0.4 | <0.4 | 0-0.008 | — | 0.4 | direct | ⑥ |
| Remote | — | — | 0.3 | — | — | 0.1-2 | — | — | — | ⑲ |
| Remote (FRG) | — | — | — | 0.03-0.9 | — | — | — | — | direct | ⑳ |
| Rural Ontario | 2 | <2 [h] | 0.002 | 0-30 | <2 [h] | 0-0.2 | — | 2 | direct | ⑥ |
| Rural | — | — | — | 0-0.3 [i] | — | — | — | — | — | ⑨ |
| Rural Austria | — | <0.1 [h,j] | — | — | — | — | — | — | — | ㉑ |

TABLE 3.4 (CONTINUED)

FOOTNOTES:

a. Values in these columns represent estimated median values for the range of concentrations reported in the study. In some cases, due to the form of data presentation, values presented in this table represent the median of a range of averages. In other cases, which are marked accordingly, only mean values could be derived from the study.

b. The lowest and highest values reported in each study are presented here as the limits of the range of reported concentrations. In some cases, due to the form of data presentation, the values presented in this table represent a range of average values from multiple locations.

c. Values in this column represent an estimated average of the analytical sensitivities reported for each measurement in the study.

d. These values are based on PCM analyses rather than TEM analyses.

e. This is a range derived from the manipulation of paired PCM, TEM analyses.

f. This value is simply the quotient of the median values for the parameters indicated.

g. These values are derived from a single measurement.

h. These values represent total structures longer than 5 um rather than PCM equivalent structures.

i. These values are estimated values.

j. These values are the mean of a range of concentrations rather than the median.

k. This is a range of means of multiple samples from several locations.

TABLE 3.4 (continued)

REFERENCES


1. Tuckfield, et al., 1988.
2. Chesson, et al., 1985.
3. Chesson, et al., 1986.
4. Constant, et al., 1983.
5. Hatfield, et al., 1988.
6. Chatfield, 1983b.
7. Sebastien et al., 1979.
8. Nicholson, et al., 1979.
9. Steen, et al., 1983.
10. Nicholson, et al., 1971.
11. Nicholson, et al., 1975.
12. U.S. EPA, 1974.
13. Sebastien, et al., 1986.
14. Nicholson, 1988.
15. Rickards, 1972.
16. Friedrichs, et al., 1983.
17. Litistorf, et al., 1985.
18. John, et al., 1976.
19. Sebastien, 1985.
20. Spurny and Stober, 1978.
21. Felbermeyer, 1983.

TABLE 3.5: PUBLISHED ASBESTOS CONCENTRATIONS PROXIMAL TO ASBESTOS SOURCES

| Environmental Setting | Median of Reported Concentrations [a] | | | Range of Reported Concentrations [b] | | | Conversion | Analytical Sensitivity [c] | Preparation Technique | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| | s/l | PCME/l | ng/m$^3$ | s/l | PCME/l | ng/m$^3$ | (PCME/l to ng/m$^3$) | | | |
| In Buildings: | | | | | | | | | | |
| -with asbestos | <5 | <1 | <0.1 | 0-40 | 0-2 | 0-40 [d] | | | direct | ① |
| -with damaged asbestos | 0.6 | — | — | — | — | — | | | — | ② |
| -with asbestos | 0.4 | — | — | — | — | — | | | — | ② |
| -with no asbestos | 0.1 | — | — | — | — | — | | | — | ② |
| -with asbestos | — | — | 12 | — | — | 0-750 | | | — | ③ |
| Toronto subways | 25 | — | 0.2 | 15-150 | 0-15 [e] | 0.015-50 | 0.3 [e,f] | | direct | ④ |
| Toronto subways | 200 | — | 15 | 40-3000 | 0-55 [e] | 0.2-200 | 0.9 [e,g] | | indirect | ④ |
| Near mines | — | 40 [e,h] | — | — | 15-150 [e,i] | — | | | indirect | ⑤ |
| Near waste piles | 60 | 7 [h] | — | 13-340 [i] | 0.5-65 [i] | — | | | direct | ⑥ |
| Paraoccupational | — | — | — | — | 0-100 [e] | — | | | — | ⑦ |

TABLE 3.5 (CONTINUED)

FOOTNOTES:

a. Values in these columns represent estimated median values for the range of concentrations reported in the study. In some cases, due to the form of data presentation, values presented in this table represent the median of a range of averages. In other cases, which are marked accordingly, only mean values could be derived from the study.

b. The lowest and highest values reported in each study are presented here as the limits of the range of reported concentrations. In some cases, due to the form of data presentation, the values presented in this table represent a range of average values from multiple locations.

c. Values in this column represent an estimated average of the analytical sensitivities reported for each measurement in the study.

d. The highest mass concentration in this range is reported for a sample composed largely of amphibole. The highest mass concentration reported for a chrysotile sample in this data set is 24 $ng/m^3$.

e. These values represent total structures longer than 5 um rather than PCM equivalent structures.

f. These values are derived from a single measurement.

g. This value is the mean of three measurements.

h. These values are the mean of a range of concentrations rather than the median.

i. This is a range of means of multiple samples from several locations.

TABLE 3.5 (continued)

REFERENCES


1. Burdett and Jaffrey, 1986.
2. Hatfield, et al., 1988.
3. Sebastien, et al., 1979.
4. Chatfield, 1983b.
5. Sebastien, et al., 1986.
6. Ruch and Serper, 1977.
7. Steen, et al., 1983.

appear to be in general agreement with this range, as opposed to the range of medians reported for samples prepared by an indirect technique: 4 to 25 s/L. The range of background concentrations in urban environments appear higher than exhibited at rural locations. As indicated by the data presented in Table 3.5, the lower range of concentrations reported in the vicinity of asbestos sources appears similar to the range reported in Table 3.4 for background.

Deriving a range of background concentrations for PCM equivalent structures is more problematic. For directly prepared samples from urban settings, the reported medians range between 0.1 and less than 2 s/L. However, for rural locations, PCM equivalent structures were largely undetected on directly prepared samples. Interestingly, median concentrations for PCM equivalent structures reported for indirectly prepared samples appear to span a lower range than for directly prepared samples: 0.05 to less than 2 s/L. Thus, the true range of background concentrations for PCM equivalent structures may not have been identified in this dataset.

Rounded to the nearest half order of magnitude, a reasonable range for background asbestos concentrations appears to span 0.5 to 5 s/L for total asbestos structures. The lower end of the range appears to be closer to the median background for rural locations. Although a representative range for background concentrations of PCM equivalent structures (or long structures) is difficult to estimate from the available literature, such values may be estimated from the range reported for total structures given the data presented in Section 3.4. Conservatively, long structures (those longer than 5 $\mu$m) represent approximately 4% of total structures in most chrysotile size distributions encountered. Thus an estimate of the range of background concentrations for long structures spans 0.02 to 0.2 s/L. Presumably, PCM equivalent structures would be some major fraction of these concentrations.

## 3.6. REPRESENTATIVE TOTAL DUST CONCENTRATIONS

As addressed in Section 6.2.1, the concentration of total particulate in a dust affects the ability to characterize the asbestos fraction. Concentrations of total suspended particulate (TSP) vary over several orders of magnitude depending on location, time of day, and weather. Urban and agricultural sites tend to exhibit significantly higher concentrations of TSP than rural locations. In addition to variation in overall concentration, the composition of TSP also varies significantly as a function of location. At Urban sites and specific rural locations, the TSP tends to be composed principally of organic matter that can be ashed or inorganic substances that are soluble in acidified media. Agricultural locations and other rural locations frequently exhibit higher concentrations of refractory silicate particles. Due to the wide spatial and temporal variation in TSP concentrations, a general rule for estimating levels at a site can not be provided without data from a comprehensive survey.

## 3.7. ANALYTICAL TECHNIQUES USED TO MONITOR ASBESTOS

Analytical methods traditionally used to monitor asbestos include midget impinger (MI), phase contrast microscopy (PCM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Each method differs in its ability to detect the various size fractions typical of asbestos dusts including those most likely to relate to biological activity.

In the occupational studies used to develop dose/response relationships (addressed in section 3.2), asbestos dusts were analyzed by counting structures on a slide or filter using optical microscopes. Until the early 1970's the procedure involved collecting workplace dust with a midget impinger using a light microscope at a magnification of 100 to count the number of particles collected.

The midget impinger is a device in which a stream of contaminated air is forced through a restricted opening into a liquid (alcohol) where it emerges as a jet of bubbles that disperse the asbestos. The particles suspended in the liquid are than transferred to a 1 mm deep well and counted using a light microscope.

Because the counting rules employed during the examination of midget impinger samples require that all observed particles be counted, the resulting concentrations represent coarse total dust counts and have little direct relationship to the quantity of fibrous structures present. In Figure 3.1, the lack of significant overlap between the midget impinger circle and the circle representing fibrous particles illustrates the limited relationship between analytical results from this method and potentially important asbestos fibers. Thus, midget impingers provide only an indirect index of asbestos exposure.

In the 1970's the midget impinger was replaced by the membrane filter method, which has become the standard technique for monitoring asbestos in industry. This latter method involves collecting a sample of airborne dust on a membrane filter, rendering the filter transparent with an appropriate solvent, and counting fibers using a phase contrast microscope (PCM) at magnifications between 400 and 900.

Because of the increased magnification and because phase-contrast lighting increases the sensitivity to narrow objects in the field of view, it is possible to delineate the general morphology of the particles being counted so that measurements can be restricted to fibrous structures (defined as those longer than 5 $\mu$m, exhibiting aspect ratios greater than 3:1, and having largely parallel sides). However, the limitations imposed are somewhat arbitrary and not based on health effects (Chatfield, 1979). In addition, PCM techniques were standardized only recently so that counting procedures and attendant results changed over the period spanned by existing epidemiology studies (Chatfield, 1985d).

Due to the limited resolution of PCM, the internal components of complex structures (bundles, clusters, or matrices) are not generally distinguishable so that all such structures appear as single, solid particles. Consequently, all structures visible by PCM that satisfied the appropriate dimensional criteria were counted as individual structures (fibers). The term "structure" is used here for consistency, because frequently a specific "fiber" viewed by PCM is observed to be a complex structure when viewed with the increased resolution afforded by transmission electron microscopy. The purpose is to preserve the terminology used to describe a specific particle that may be viewed using any of several microscopic techniques.

Due to a combination of resolution and contrast, the minimum width of structures visible in the phase contrast microscope reportedly ranges between 0.2 and 0.4 $\mu$m, depending on the configuration and condition of the instrument. The most frequently quoted average limit to visibility is 0.25 $\mu$m. Partially by convention and partially due to the practical limitations associated with classifying structures with aspect ratios less than 10:1, structure counts were further limited to those longer than 5 $\mu$m (See for example: Chatfield, 1985d).

As indicated in Figure 3.1, structures visible by phase contrast microscopy correspond to a range that encompasses a significant portion of the structures believed to be biologically active. However, structures believed to be the most biologically active (the longest, thinnest structures) are not counted by this technique. Additionally, because the method is incapable of distinguishing asbestos from non-asbestos minerals, non-asbestos structures (to the extent that they are present in an exposure setting under study) are included in PCM counts. Although it is assumed that the vast majority of fibrous structures encountered in an occupational setting are asbestos, this is not the case for environmental samples. Consequently, these two stated limitations of PCM render it unsuitable for monitoring environmental asbestos.

More recently, techniques based on the electron microscope have been introduced. The scanning electron microscope (SEM) and the transmission electron microscope (TEM) count, respectively, asbestos structures on membrane filters or structures transferred from the filters to electron microscope grids.

Magnifications typically achieved with SEM range between 2,000 and 10,000, while TEM magnifications can easily reach 100,000. Thus, TEM is capable of resolving even the finest fibrous structure. Although the magnification implies that the resolution of SEM should be significantly better than PCM, in practice, the visibility of structures in the SEM is limited by a combination of contrast and electronic noise (Small et al 1983). Consequently, the minimum structure width visible in the SEM is only slightly better than PCM. Under optimum conditions, the minimum width of structures visible in the SEM is perhaps a factor of 5 better than PCM (Walton 1982). Given the increased expense and other instrumental constraints, however, there appears to be little advantage to SEM over PCM except for the ability to distinguish asbestos from non-asbestos structures. At the same time, SEM does

not appear to retain a significant cost advantage over TEM for the analysis of environmental samples. Therefore, SEM is not likely a useful technology for environmental analysis of asbestos.

Most electron microscopes (both TEMs a SEMs) are equipped to allow mineralogical and elemental analysis to confirm the composition of the structures counted. Thus, TEM is clearly capable of identifying populations currently believed to represent the most biologically active fraction of asbestos, the shaded area of Figure 3.1 (see Sections 3.1, 3.2, and 3.3). In fact, TEM is theoretically capable of distinguishing whatever appropriate size or mineralogical fraction that is likely to be linked to asbestos biological activity in the future.

## 3.8. EXISTING TEM ANALYTICAL METHODS

Several researchers have published analytical methods based on TEM for the analysis of asbestos samples collected on membrane filters. The principal features of the Yamate method (1984), NIOSH 7402 (NIOSH 1986), Hayward's method (Hayward, no date), and the method in the AHERA regulations (USEPA 1987), are summarized in Table 3.6. A PCM analysis method, NIOSH 7400 (NIOSH 1985) is also included in Table 3.6 for comparison. It should be emphasized, however, that procedures employed for PCM analysis have changed over time. Therefore, the NIOSH method for PCM presented in the table, which is a relatively recent method, differs in detail from the various procedures likely employed to collect the data used in published epidemiology studies. Nonetheless, it is included to allow comparison between TEM methods and the general features of a PCM method.

As indicated in Table 3.6 the methods incorporate procedures for preparation, counting rules, and rules for identifying and characterizing asbestos mineralogy. Generally, the methods also define a fixed area of the specimen grid to be counted. By comparing the requirements of a method suitable to support risk assessment to the features of the methods presented in Table 3.6, it is possible to determine which of the methods may be applicable to Superfund field investigations.

Except for differences in procedural details, the TEM methods presented in Table 3.6 share many common features. All rely on direct preparation, although the Yamate method contains a protocol for an optional indirect preparation should filters prove too loaded to analyze the filters when prepared by a direct technique. All of the TEM methods incorporate structure characterization at a magnification of 20,000. The principle differences between the methods involve counting rules and the inclusion in the Hayward method of a low magnification scan designed to provide a statistically balanced count of large asbestos structures (longer than 5 $\mu$m). Because long structures appear to play a major role in determining the biological activity of asbestos dusts (see Sections 3.1 and 3.2), the use of a low magnification scan to count long structures in a statistically balanced manner is incorporated in the method presented in Part 1 of this report.

## TABLE 3.6: COMPARISON OF AVAILABLE ASBESTOS ANALYTICAL TECHNIQUES

| METHOD: | NIOSH 7400 | NIOSH 7402 | YAMATE | HAYWARD | AHERA |
|---|---|---|---|---|---|
| **Analytical Technique** | PCM | TEM | TEM | TEM | TEM |
| **Preparation Methodology** | Direct | Direct | Direct (Optional Indirect) | Direct | Direct |
| **Magnification** | 450x | 10,000x | 20,000x | High Magnification: 20,000x - 50,000x<br>Low Magnification: 400x - 4000x | 15,000x - 20,000x |
| **Dimensions ($\mu$m)** | | | | | |
| Length (l): | l > 5 | l > 1 | l > 0.06 [a] | High Magnification:<br>l > 0.06 [a] | l > 0.5 |
| Width (w): | w > 0.25 [b] | 3.0>w>0.04 [c] | w > 0.02 [c] | w > 0.02 [c] | w > 0.02 [c] |
| Aspect Ratio (ar): | ar > 3 | ar > 3 | ar > 3 | ar > 3 | ar > 5 |
| | | | | Low Magnification:<br>l > 5<br>w > 0.25<br>ar > 3 | |
| **Reported Sensitivity** | | | | | |
| s/cm$^3$ | --- | --- | --- | --- | 0.005 s/cm$^3$ |
| s/mm$^2$ | --- | --- | --- | --- | 70 s/mm$^2$ |

TABLE 3.6 (continued)

| METHOD: | NIOSH 7400 | NIOSH 7402 | YAMATE | HAYWARD | AHERA |
|---|---|---|---|---|---|
| **Counting Rules:** | | | | | |
| Structures | Count all structures exhibiting l > 5$\mu$m, w < 3.0 $\mu$m, and aspect ratios > 3. | Count all structures exhibiting l > 1$\mu$m, w < 3.0$\mu$m, and aspect ratio > 3. Note PCME fraction within count. | Count all structures exhibiting an aspect ratio > 3. | Count all structures exhibiting an aspect ratio > 3. Use low magnification scan to count PCME fraction. | Count all structures longer than 0.5$\mu$m that exhibit an aspect ratio > 5. Record individual fibers within all groupings with fewer than 3 intersections. Count structures longer than 5$\mu$m separately (PCME). |
| Bundles [d] | Bundles meeting overall dimensional criteria generally counted as single fibers unless up to 10 individual fiber ends can be distinquished within the bundle (representing 5 individual fibers). | Bundles meeting overall dimensional criteria generally counted as single fibers. | Bundles meeting overall dimensional criteria generally counted as single entities and noted as bundles on the count sheet. | Bundles meeting overall dimensional criteria generally counted as single entities and noted as bundles on the count sheet. | Bundles of 3 or more fibers that meet the overall dimensional criteria are counted as single entities and noted as bundles on the count sheet. |

TABLE 3.6 (continued)

| METHOD: | NIOSH 7400 | NIOSH 7402 | YAMATE | HAYWARD | AHERA |
|---|---|---|---|---|---|
| Clusters[e] | Within a cluster, count up to 10 individual fiber ends from (up to 5) fibers that meet the overall dimensional criteria. Otherwise, count a cluster as a single entity. | Within a cluster, count up to 3 individual fibers that meet the overall dimensional criteria. Otherwise, clusters that contain more than 3 fibers that meet the overall dimensional criteria are counted as single clusters. | Within a cluster, count up to 3 individual fibers that meet the overall dimensional criteria. Otherwise, clusters that contain more than 3 fibers that meet the overall dimensional criteria are counted as single clusters. | Within a cluster, count up to 3 individual fibers that meet the overall dimensional criteria. Otherwise, clusters that contain more than 3 fibers that meet the overall dimensional criteria are counted as single clusters. Clusters are defined in the low magnification scan by their overall dimensions. | A collection of fibers with more than 2 intersections where at least one individual projection meets the overall dimensional criteria is counted as a single cluster. |
| Matrices[f] | Count up to 5 fibers emanating from a clump (matrix). Each individual fiber must meet the dimensional criteria. | Count individually identifiable fibers within a matrix. Fibers must individually meet the dimensional criteria. | Count as a single matrix, all matrices with at least one protruding or embedded fiber that meets the dimensional criteria. | Count as a single matrix, all matrices with at least one protruding or embedded fiber that meets the dimensional criteria. | Count as a single matrix, all matrices with at least one protruding fiber such that the protruding section meets the dimensional criteria. |
| Maximum Number Counted | 100 structures | 100 structures | 100 structures | 100 structures at high magnification and 100 long structures at low magnification. | 50 structures |

TABLE 3.6 (continued)

| METHOD: | NIOSH 7400 | NIOSH 7402 | YAMATE | HAYWARD | AHERA |
|---|---|---|---|---|---|
| Area Scanned | 100 fields | 100 openings [g] | 10 openings | 15 openings[h] at high magnification distributed over 4 grids from each sample and 100 openings at low magnification distributed over 4 grids from each sample. | Blanks: 10 openings Samples: 10 openings (given the defined sensivity and the recommended air volumes). |
| Mineralogy Determined? | no | yes | yes, except matrix particles | yes, except matrix particles | yes, except matrix particles |
| Statistical Balanced Counting? | no | no | no | yes | no |

a. The minimum fiber length to be counted has not been defined in this method. Presumably the minimum fiber length counted would correspond to 3 times the resolution limit of the width (due to the aspect ratio requirement). Since the presumed resolution limit is 0.001$\mu$m, the corresponding minimum length fiber that would be counted under this method is 0.003$\mu$m.

b. Width restrictions for PCM are due to limits in resolution. A width of 0.25 $\mu$m represents the average resolution reported for PCM.

c. The width restriction reported for TEM is based on an estimate of the resolution limit associated with typical magnification settings for the technique. The presumed limit of resolution at a typical magnification of 20,000x reported for this technique is 0.001$\mu$m.

d. Bundles are defined as a parallel arrangement of fibers separated by distances smaller than one fiber diameter.

e. Clusters are a collection of fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

f. Matrices (termed "mats" by Hayward) are one or more fibers embedded within or protruding from another particle.

g. The 100 grid opening limit for scanning is not stated directly in this method but can be inferred from the information provided.

h. This has been converted to a 200 mesh equivalent to be consistent with all of the other criteria presented in this row.

TABLE 3.6 (continued)

| METHOD | REFERENCES |
|---|---|
| 1. NIOSH 7400 | Niosh, 1985. |
| 2. NIOSH 7402 | Niosh, 1986. |
| 3. YAMATE | Yamate, et al., 1984. |
| 4. HAYWARD | Hayward, no date. |
| 5. AHERA | US EPA, 1987. |

Changes in counting rules as the methods evolved (each one building on its predecessors) were designed to minimize subjective decisions by the analyst using the method. This serves to minimize observer-dependent variation, one of the major sources of variation associated with TEM analysis. Of the published methods, the counting rules presented in AHERA provide the best defined counting procedures for minimizing observer-dependent variation. However, the recording of structure dimensions is not required under AHERA, which potentially limits the ability to use such results in a risk assessment. A method designed to support a risk assessment must count structures in a manner that is consistent with the characteristics that relate to biological activity, which depend on structure size. Therefore, the counting rules defined in AHERA were modified for this method to better detect and record the range of characteristics that potentially relate to biological activity (see Sections 3.1 and 3.2).

One of the limitations common to most of the TEM methods presented in Table 3.6 is that the sensitivity of the analytical method cannot be defined. This is a consequence of defining a fixed area of the specimen to scan without simultaneously defining a fixed volume of air to be collected during sampling. One of the critical elements of this method is that the target sensitivity is defined and the combined effect of sample loading and the area to be scanned during analysis are addressed (see Section 6.2).

Another critical modification incorporated into this method that is only partially addressed by the TEM methods presented in Table 3.6, is a recording procedure that preserves sufficient information to allow extensive and flexible interpretation of the results without the necessity to re-analyze the specimen.

## 4. RISK ASSESSMENT OBJECTIVES AND METHOD REQUIREMENTS

To support a risk assessment, a method must address three objectives:

(a) to provide measurements that relate to the biological activity of asbestos;

(b) to provide sufficient sensitivity to measure asbestos at the low concentrations typically found in the environment near potential sources;

(c) to provide sufficient precision to elucidate spatial and temporal trends in asbestos concentrations.

An additional consideration to be addressed is the need to control sampling and analysis costs. Each of these objectives imposes a set of technical constraints on a method for asbestos sampling and analysis. Such constraints served as a basis for defining performance criteria for this method.

### 4.1. CONSIDERATION OF BIOLOGICAL ACTIVITY

Although published risk factors are expressed in terms of PCM measurements, other studies indicate that the biological activity of asbestos is a function of a broader range of the distribution of asbestos structure types and sizes, which PCM is not capable of distinguishing (see Sections 3.1, 3.2, and 3.7). Therefore, to properly address current concepts concerning the biological activity of asbestos, this method is designed to:

(a) track asbestos structures of all lengths (defined as all entities with components exhibiting an aspect ratio exceeding 5:1 that are longer than 0.5 $\mu$m);

(b) separately track asbestos structures longer than 5 $\mu$m;

(c) track such entities at sufficient magnification to resolve the thinnest asbestos structures (approximately 0.02 $\mu$m in diameter);

(d) distinguish among asbestos fibers, bundles, clusters, and matrices and independently track their concentrations;

(e) characterize the mineralogy of each structure counted;

(f) record the lengths and widths of each structure counted to allow for later selection and tracking of size fractions with particular emphasis on long structures (longer than 5 $\mu$m);

(g) provide a system for recording analytical results that preserves sufficient information concerning the mineralogical determination and morphological characteristics of each structure to allow for later re-interpretation without the need to re-evaluate the original specimen.

Regarding aggregates (bundles, clusters, and matrices), it is also important to estimate and record, if possible, the number of asbestos components present within each aggregate.

NOTE

Although it appears that the size fraction represented by "b" above can be derived from the count of total structures by selecting those exhibiting the appropriate length and width criteria, it is listed separately to emphasize the need to count long structures separately for statistical validity.

Recording the length and width of each structure preserves the ability to select size fractions out of the total structure count that may be of special interest. Examples of size fractions likely to be of special interest include the PCME fraction (defined as structures longer than 5 $\mu$m and thicker than 0.25 $\mu$m) or "Stanton" fibers (defined as structures longer than 8 $\mu$m and thinner than 0.25 $\mu$m).

## 4.2. PRECISION REQUIREMENTS

To support a risk assessment, the precision of this method should be sufficient to delineate spatial and temporal trends in the field data collected at a Superfund site. Primarily, this means distinguishing among environmental concentrations of asbestos attributable to local sources at a site from concentrations associated with general background. In the absence of site-specific information, it is assumed desirable to distinguish with high confidence a factor of five difference (half an order of magnitude) between concentrations. The purpose of this assumption is simply to define a target performance requirement from which the method could be developed. However, numerous sources of variation associated with asbestos measurement potentially contribute to the uncertainty of an analytical result so that a specific, desired level of precision may be difficult to achieve.

In practice, the precision of a method for the determination of asbestos is limited by several factors including the distribution of asbestos on sample filters, the characteristics of sampling and analysis tools, and the subjectivity of the analyst. In the absence of a database sufficient to establish levels of uncertainty (and, hence, the ability to quantify precision), a lower limit to the number of structures that must be counted in a measurement to achieve a desired level of precision may be estimated from consideration of the distribution of asbestos on an analytical filter, ignoring variation introduced by other factors. The variation potentially introduced by these other factors would tend to increase the minimum number of

structures that must be counted to achieve a desired level of precision. A more detailed discussion of the factors affecting precision can be found in the literature (see, for example, ISO 1981).

Assuming, as suggested above, that it may be important to distinguish concentrations that differ by a factor of five, the minimum number of structures that must be counted to achieve the defined level of precision can be estimated as follows. If it is assumed that structures deposited on a filter exhibit a Poisson distribution and that analytical contamination is zero so that observed asbestos represents sampled asbestos, the test statistic for evaluating whether two means ("m" and "n") can be distinguished is:

$$t = (m - n)/(m + n)^{1/2}.$$

The object is to find the lowest value of the two parameters (m and n) such that $m = 5n$ and the difference between m and n is significant based on the standard normal distribution to the Poisson (Miller and Freund 1965). For $n = 1$ and $m = 5$, $t = 1.6$ which is not quite significant at the 5% level (where the cutoff is 1.65). For $n = 2$ and $m = 10$, $t = 2.3$, which is significant at the 5% level. Thus, since the variability in structure counts is probably larger than that predicted by the Poisson distribution due to contributions from other factors, it is reasonable to assume that a minimum of 10 structures need to be counted at the concentration of interest to distinguish concentrations that differ by a factor of five.

Determinations based on multiple samples can be based on fewer counts per sample as long as the precision of the aggregate array of samples is comparable to the precision indicated above for single sample determinations. This requirement applies both to the count of asbestos structures of all lengths and to the count of asbestos structures longer than 5 $\mu$m. However, these two size fractions must be counted separately to provide a statistically balanced representation (Sebastien et al, 1982).

NOTE

Although the foundation for the above assumptions may be a subject of some debate, the practical impact of the application of these assumptions to the development of the method were minimal (see Section 4.3).

As indicated above, method precision is also limited by instrument characteristics and analyst subjectivity, among other factors. To minimize the extent of variation contributed by these two factors, instrument settings and characteristics must be specified in the method and counting rules must be specified sufficiently to minimize the opportunity for subjective decisions by the analyst.

## 4.3. SENSITIVITY REQUIREMENTS

Analytical sensitivity is defined as the estimated airborne concentration corresponding to the observation of one asbestos structure. The required analytical sensitivity for this method may be defined by considering the range of concentrations over which measurement is likely to be required.

Based on a qualitative evaluation of the data presented in Section 3.5, estimates of median background concentrations range between 0.5 and 5 s/L for total asbestos structures and 0.02 to 0.2 s/L for asbestos structures longer than 5 $\mu$m. Such median concentrations are reasonable targets for analysis because they likely represent the concentrations above which contributions from local sources may be distinguished.

To achieve the desired precision, as indicated in section 4.2, the assumed requirement for this method is the detection of 10 structures at the target concentrations specified. However, this requirement must be tempered against the probability that a particular asbestos concentration measured in the environment includes contributions from anthropogenic contamination over local background. At the low end of the range of median concentrations reported for background, it is highly unlikely that such a concentration may be attributed to anthropogenic contamination. At the high end of the range of background concentrations, however, the probability that the measurement of such a concentration represents contamination from anthropogenic sources is high. At the high end of the range for background, therefore, it may be important to distinguish among small changes in measured concentrations such as might occur between upwind and downwind samples in the vicinity of a potential source.

Setting the analytical sensitivity for this method at 0.5 s/L for total structures and 0.02 s/L for structures longer than 5 $\mu$m, achieves the duel purposes of providing sufficient sensitivity to measure concentrations down to levels at which anthropogenic contamination is unlikely while providing that at least 10 structures will be counted when measured concentrations fall into a range where it is important to distinguish among small differences in concentrations. However, it is also important to consider analytical background.

Should asbestos be observed during analysis, it is generally important to distinguish whether such asbestos originated in the sampled medium or if it was introduced as contamination during analysis. Asbestos that can be attributed to the sampled medium is generally considered to have been "detected". Thus, a detection limit is defined as the smallest measurement that is unlikely (probability less than a specified value) to be due entirely to contamination from sources other than the air being sampled. Detection limits are generally quantified by considering the magnitude and frequency of occurrence of the analytical background associated with a particular method.

However, unless the distribution of analytical background is known, detection limits are difficult to quantify. Consequently, an alternate method to account for analytical background is incorporated into this method: a statistical test to distinguish blank measurements from sample measurements. Factors affecting analytical background are addressed in Section 6.3.

As addressed in Section 4.2, it should be emphasized that the desired analytical sensitivity defined above does not have to be achieved for individual samples if such samples are part of a set of multiple samples that were collected so that they are representative of the same sampling environment. Under such conditions, it is sufficient that the desired analytical sensitivity be achieved by the aggregate of the set of samples as a whole. For example, assuming that 10 samples were collected in a manner assuring that they represent the same sampling environment, analyzing each individual sample so that the analytical sensitivity of the measurement is 5 s/L, yields an analytical sensitivity for the arithmetic mean of the 10 samples of 0.5 s/L.

### 4.4. REPORTING REQUIREMENTS

It is necessary to understand the potential sources of variation in a measurement before the results of an analysis can be properly interpreted. Sources of variation that potentially contribute to the result of an asbestos measurement include particularly the location of the subsection selected for analysis on the TEM specimen, the characteristics of the instrument used in the analysis, and the subjectivity of the analyst. Variation introduced by each of these factors means that there is a probability that the results of two or more consecutive measurements obtained from the same sample may differ within a finite range. The degree of variation introduced by such factors to the results of a measurement obtained using a specific method is usually represented by specifying a set of confidence limits in association with each reported measurement result. Consequently, rules for constructing appropriate confidence limits are incorporated as part of this method (see Appendix E of The Method, Part 1 of this report).

In addition to factors that potentially contribute to variation in the result of the measurement of a specific specimen, asbestos observed during the analysis may have originated either in the air sampled or may have been introduced from contamination during any of several phases of sample handling, preparation, and analysis (see Section 4.3). Therefore, it is important to distinguish contributions to a measurement that may be attributed to sampled asbestos from contributions that may be attributed to analytical background. Consequently, rules for conducting a statistical test to distinguish sampled asbestos from analytical background are incorporated as part of this method (see Appendix E of The Method, Part 1 of this report).

## 4.5. METHOD SPECIFICATIONS

The method presented in Part 1 of this report is a method where samples are collected on membrane filters and analyzed by TEM. None of the published TEM methods satisfy all of the above requirements; accordingly, several alternatives had to be evaluated to develop a procedure that is capable of satisfying the entire set of method requirements defined above. The following procedures for sample collection, preparation, handling, and analysis were combined.

## 5. OVERVIEW OF METHOD

In the method presented in Part 1 of this report, samples are collected and prepared for TEM examination by either one of two techniques. The majority of air samples (denoted Phase 1 samples) will be analyzed using an indirect procedure for preparation of TEM specimens, optimized to provide the required analytical sensitivity and precision. A small number of samples (denoted Phase 2 samples) will be collected in such a way that they can be analyzed using both indirect and direct procedures for preparation of TEM specimens, to allow comparisons to be made between the results from the two specimen preparation procedures. TEM examination procedures used for the two sets of samples also differ.

### 5.1. SAMPLE COLLECTION

A sample of airborne particle is collected by drawing a measured volume of air through a 25 mm diameter, 0.45 $\mu$m pore size MCE membrane filter by means of a pump. Air volumes collected on Phase 1 samples will be maximized. Air volumes collected on Phase 2 samples will be limited to provide optimum loadings for filters to be prepared by a direct-transfer procedure.

### 5.2. SAMPLE PREPARATION

TEM grids will be prepared according to either 5.2.1 or 5.2.2 below.

#### 5.2.1. Indirect TEM Specimen Preparation

This preparation will be applied to all Phase 1 and Phase 2 samples. Half of the filter is ashed in a low-temperature plasma asher. The residual ash is ultrasonically dispersed in freshly-distilled water. The suspension is acidified using hydrochloric acid, and immediately filtered through a 25 mm diameter, 0.1 $\mu$m pore size MCE filter. The filter is dried and the filter structure is collapsed using a mixture of dimethyl formamide, acetic acid and water. A thin film of carbon is evaporated onto the collapsed filter surface and small areas are cut from the filter. These areas of filter are supported on TEM specimen grids and the filter medium is dissolved away by a solvent extraction procedure.

NOTE

An alternate procedure for indirect preparation, which incorporates washing the deposit off of MCE filters and ashing of the wash-suspended deposit, may be substituted into this method subject to the results of a pilot study.

#### 5.2.2. Direct TEM Specimen Preparation

One quarter of the remaining filter sections from all Phase 2 samples will be prepared by this procedure. The quarter filter is collapsed using a mixture of dimethyl formamide, acetic acid and water. The collapsed filter is

etched for a short time in a low temperature plasma asher to remove the surface layer of filter polymer which may have encapsulated asbestos structures during the collapsing procedure. A thin film of carbon is evaporated onto the collapsed filter surface and small areas are cut from the filter. These areas of filter are supported on TEM specimen grids and the filter medium is dissolved away by a solvent extraction procedure.

## 5.3. ANALYSIS

The TEM specimen grids are examined at both low and high magnifications to check that they are suitable for analysis before carrying out a quantitative examination on randomly-selected grid openings. In addition to isolated fibers, ambient air samples often contain more complex aggregates of fibers, with or without equant particles. Some particles are composites of asbestos fibers with other materials. Individual fibers and more complex structures are collectively referred to as "asbestos structures". A coding system is used to record the type of fibrous structure, and to provide the optimum description of each of these complex structures.

The method requires that separate examinations be made for asbestos structures of all sizes (incorporating asbestos fibers with lengths greater than 0.5 $\mu$m) and for asbestos structures longer than 5 $\mu$m. In both cases, asbestos structures are defined as structures containing components exhibiting mean aspect ratios equal to or greater than 5:1. This TEM examination procedure allows for specification of a lower analytical sensitivity for the measurement of the concentration of asbestos structures longer than 5 $\mu$m.

In the TEM analysis, electron diffraction (ED) is used to examine the crystal structure of a fiber, or fibrous components of complex structures, and the elemental composition is determined by energy dispersive X-ray analysis (EDXA). For a number of reasons, it is not possible to identify (confirm the mineralogy of) each structure unequivocally and structures are classified according to the techniques that have been used to identify them. A simple code is used to record the manner in which each structure is classified.

The classification procedure is based on successive inspection of the morphology, the electron diffraction pattern, and the energy dispersive X-ray spectrum. Confirmation of the identification of chrysotile is only by quantitative ED, and confirmation of amphibole is by a combination of quantitative EDXA and quantitative zone-axis ED.

Several levels of analysis are specified, the higher levels providing a more rigorous approach to the identification of fibers. The procedure permits a minimum required asbestos identification procedure to be defined on the basis of previous knowledge, or lack of it, about the particular sample. Attempts are then made to achieve this defined minimum procedure for each asbestos structure, and the degree of success is recorded for each. The two codes remove from the microscopist the requirement to interpret observations made during the TEM examination, and allow this evaluation to be made later without the requirement for re-examination of the TEM specimens.

The lengths and widths of all classified asbestos structures are recorded. The number of asbestos structures found on a known area of the TEM specimen grids, together with the equivalent volume of air filtered through this area, are used to calculate the airborne concentration in asbestos structures/liter of air.

This method specifies minimum analytical sensitivities of 0.5 s/L and 0.02 s/L for the measurements of asbestos structures of all sizes (incorporating structures longer than 0.5 $\mu$m) and asbestos structures longer than 5 $\mu$m, respectively. In both cases, asbestos structures are defined as structures containing components exhibiting mean aspect ratios equal to or greater than 5:1.

It will not always be possible to achieve the defined analytical sensitivities, because the volume of air that can be sampled is dictated by the nature and concentration of the suspended particulate in the atmosphere being sampled. To some degree, this limitation can be overcome by selective concentration of asbestos structures during the specimen preparation procedures and by examination of a larger area of the TEM specimens. However, the ease and cost of achieving a specific value for the analytical sensitivity will vary from sample to sample.

## 6. METHOD COMPONENTS

The detailed protocols incorporated in this method were selected based on the state of knowledge of their presumed capabilities and limitations. In some cases, data are lacking so that additional laboratory work is needed to properly evaluate the efficacy of the proposed procedures.

### 6.1. FACTORS AFFECTING CHARACTERIZATION OF ASBESTOS STRUCTURES

The proper characterization of asbestos structures requires use of an analytical technique capable of resolving the thinnest asbestos structure and capable of distinguishing asbestos from non-asbestos minerals. Counting rules must be designed to facilitate distinguishing fibers, bundles, clusters, and matrices. Various fractions of potential interest must be easily extracted from analysis results and counts must be recorded in sufficient detail to allow later reinterpretation.

#### 6.1.1. Analytical Technique

Because one of the goals of environmental sampling and analysis for the determination of asbestos is to provide a measurement that is comparable with published risk factors, which are expressed in terms of PCM counts, an obvious question that arises is whether to simply use PCM as the analytical technique for the analysis of environmental samples. However, environmental samples can not be properly characterized using PCM due to a combination of the limitations of PCM and the characteristics of environmental dusts. It is unfortunate that PCM can not be used to evaluate environmental samples because PCM is significantly less expensive than TEM.

PCM is inherently less sensitive than TEM at detecting asbestos structures. The sensitivity of PCM is limited both by increasing obscuration as filters become more loaded and by increasing observer-dependent variation as fibrous structures become less concentrated on the filter (Chatfield, 1985a). In one round-robin study of PCM laboratories (Crawford, 1985), observer-dependent variation approached a factor of 300.

Observer-dependent variation may be due to a combination of instrument limitations, differences in preparation techniques, and the subjective judgments of the analyst. In common with any technique in which measurements are made close to the lower limits of sensitivity, PCM results vary as a function of the condition of the instrument. Differences may be due to such factors as misalignment of the phase ring, failure to scan the full depth of focus, and differences in the interpretation of irregular fibers. Evidence for such variation is provided in several studies including the following:

(a) fiber counts made on identical samples were shown to increase by a factor of two if the count was made at twice the magnification (Lynch et al 1970);

(b) counting with a graticule verses full field counting increases counts by a factor of three (Beckett et al, 1976);

(c) for chrysotile, novice counters frequently count only 25% of the fibers observed by experienced counters (Beckett and Attfield 1974);

(d) there are large inter-laboratory differences in counts of the same sample (Beckett and Attfield 1974).

In contrast to the last observation, inter-laboratory agreement among TEM results has been achieved without the extensive inter-laboratory discussions that have been required to normalize the optical work.

The practical limit of sensitivity for PCM is approximately 10 s/L at the maximum total dust loading that can be tolerated in occupational samples (Chatfield, 1985d). PCM can be applied in occupational settings because contaminated work sites tend to exhibit elevated asbestos concentrations in relationship to total dust concentrations. This allows asbestos to be deposited on the filter at concentrations within the range of the sensitivity limits of PCM at the same time that dust deposited on the filter remains below the level at which obscuration precludes a proper PCM count.

In contrast, the ratio of asbestos structures to total dust particles is generally lower in environmental samples than in occupational samples. The absolute concentration of asbestos also tends to be lower in environmental samples. The lower ratio of asbestos to dust limits PCM sensitivity so that the typical asbestos concentrations found in environmental samples (ranging between 0.01 and 0.1 PCME s/L) can not be detected. The net result, in the absence of other factors, is the creation of large numbers of false negative analyses when PCM is used to analyze environmental samples.

PCM analysis of environmental samples can create false positive results due to the inability of PCM to distinguish asbestos structures from non-asbestos structures. Non-asbestos fibers (such as cellulose and gypsum) are ubiquitous in many environmental settings. In fact, asbestos generally constitutes a minority of the total fibrous structures typically present in environmental samples (Chatfield, 1985d). Because they can not be distinguished from asbestos, non-asbestos fibers that exhibit the appropriate dimensional criteria will be included as asbestos in a PCM count, creating falsely inflated asbestos counts.

The inability of PCM to track asbestos concentrations in environmental samples has been documented in numerous studies. In a comparison of four sampling and analysis methods (Gibbs et al, 1980), three EM methods generated data that showed trends in asbestos concentrations, which decreased with distance from known sources. The one PCM method included in the study was not capable of distinguishing between high environmental asbestos concentrations

near sources and low concentrations at background locations. This is presumably because the majority of structures observed by PCM were not asbestos.

Asbestos measurements by TEM and PCM were also compared in a series of studies of asbestos abatement projects (Tuckfield et al, 1988; Chesson et al, 1985; and Karaffa et al, 1986). Both indoor air and outdoor (ambient air) were monitored in these studies. In none of these studies was PCM capable of reliably determining indoor or ambient concentrations. In fact, it was reported in one study (Chesson et al, 1985) that PCM counts appeared to relate to human activity rather than total asbestos (as measured by TEM), suggesting that non-asbestos fibers were interfering with the analysis.

Perhaps the most important limitation associated with PCM is the inability to detect asbestos characteristics that best relate to biological activity and, therefore, determine risk (see Sections 3.1 and 3.2) Due to the limited resolution of PCM, only structures longer than 5 $\mu$m with diameters exceeding 0.25 $\mu$m can be counted. Therefore, the long, thin asbestos structures (which are believed to constitute the most biologically active structures) can not be resolved by PCM. In addition, PCM is incapable of resolving the internal detail of the structures counted so that it is frequently not possible to distinguish aggregates from simple fibers. Thus, PCM is not capable of characterizing many of the aspects of asbestos exposures generally believed to affect biological activity.

Over the range of conditions typical of environmental samples, PCM is not capable of providing measurements of asbestos in environmental settings that relate meaningfully to risk factors in occupational settings. PCM can not be used to properly evaluate environmental samples because:

(a) it is not sufficiently sensitive to detect asbestos at concentrations typical of environmental samples;

(b) it suffers from observer-dependent variation to a degree that is unacceptable for the level of precision required for environmental samples;

(c) it is not capable of distinguishing asbestos from non-asbestos structures, a requirement that is critical to the proper analysis of environmental samples; and

(d) it is not capable of distinguishing among the various types and sizes of asbestos structures that impact the biological activity of asbestos.

Therefore, TEM is the only analytical technique capable of characterizing asbestos exposures in a manner that is consistent with the method requirements defined in Section 4. TEM is the analytical technique to be used for the determination of asbestos in this method.

NOTE

SEM was also evaluated and eliminated from consideration for use in this method (see Section 3.7).

### 6.1.2. Magnification/Resolution

To properly characterize asbestos as dictated by the method requirements presented in Section 4, it is necessary to resolve all asbestos structures present in an airborne dust. The thinnest asbestos fibrils that retain the recognizable crystalline character of asbestos (chrysotile fibrils) generally range in diameter between 0.02 and 0.04 $\mu$m (see Section 3.4). Therefore, the resolution of the method must exceed 0.02 $\mu$m in order to detect all of the asbestos structures on a sample specimen. A magnification of 20,000 is more than sufficient to resolve asbestos structures over the total range of lengths and diameters common to asbestos. A magnification of 10,000 is sufficient to resolve TEM structures longer than 5 $\mu$m over the entire range of diameters common to asbestos. The advantage of conducting the scan for long structures at the lower magnification is the decreased time required and the corresponding cost savings. Thus, magnifications of 20,000 and 10,000 are employed in this method to scan for asbestos structures of all lengths and asbestos structures longer than 5 $\mu$m, respectively.

Method requirements presented in Section 4 also indicate that the width of detected asbestos structures must be properly delineated. At a magnification of 10,000, structures 0.02 $\mu$m in diameter appear as thick as 0.2 mm on the viewing screen of the TEM, which is easily seen, but which can not be easily distinguished from dimensions that vary by factors of less than 2. Thus, although structures of such dimensions may be detected at the magnification specified, structure dimensions must be characterized at higher magnifications to adequately distinguish among diameters. The objective is to discriminate among diameters that differ by 0.05 $\mu$m. Such precision can be obtained at magnifications of 20,000 and greater. The primary purpose for the clear delineation of diameters is to facilitate later classification of candidate size fractions of potential interest for assessing risks from among the structures recorded.

NOTE

The possibility of employing lower magnification (less than 10,000) to detect the long (greater than 5 $\mu$m) structure fraction was considered during method development. However, this tends to increase variation in the results due to a combination of instrument variation and differences in operator experience.

Asbestos counts derived at lower magnifications (500 - 2000) appear to vary as a function of magnification and other microscope characteristics. For example, at magnifications typically employed in a PCM analysis, small changes that affect the average visibility of asbestos structures potentially yield large changes in the number of structures

that can be detected. The visibility of small asbestos structures varies from instrument to instrument and with changes in operating conditions. For PCM, a visibility test slide is available for standardizing the visibility of each instrument despite differences in operating conditions. Use of the slide is specified in the method. However, no such test procedure is available for TEM. Consequently, if the TEM is to be used at these low magnifications, there is no mechanism for standardizing the visibility of structures to be counted.

Due to the increased variation in results that generally accompany the counting of asbestos structures at reduced magnification, such an approach for determining the longer structure fractions were not incorporated in this method. Rather, longer fractions of asbestos are characterized by counting structures longer than 5 $\mu$m at a magnification of 10,000, characterizing their diameters at increased magnifications, and distinguishing the various fractions of interest in the count by selecting the structures listed on the count sheet that exhibit appropriate lengths and diameters, with diameters determined to a precision of +/- 0.05 $\mu$m.

### 6.1.3. Rules for Counting, Characterization, and Recording

Counting, characterization, and recording rules for this method are designed to provide:

(a) a count of total asbestos structures (individual entities) whose distribution is best described by Poisson statistics;

(b) a count of structure components that is not skewed by an arbitrary cutoff for the number of individually recognizable components present in each structure; and

(c) counts of structures in the range of dimensional fractions that are believed to best relate to biological activity.

To achieve these objectives, several modifications to published procedures are incorporated in this method. First, the criterion for meeting specified counting limits is defined in terms of asbestos structures of all lengths, which represents a count of individual asbestos entities. Structure components are not counted individually. This is to assure appropriate statistical validity. Second, when structure components are counted (to establish an estimate of the total equivalent number of fibers) the count is not arbitrarily truncated at 5 components per structure. Rather, structures exhibiting more than 5 individually recognizable components are specially noted on the count sheet.

## 6.2. FACTORS AFFECTING SENSITIVITY

For TEM analysis, analytical sensitivity (the concentration corresponding to the observation of a single asbestos structure) is limited by the amount of asbestos that can be deposited on a given area of filter.

The amount (loading) of asbestos that can be deposited on a filter is limited by two factors: sampling constraints and the limit at which a filter becomes too loaded to allow for proper analysis. Although the second of these factors is generally more restrictive than the first, it is also influenced by the type of filter preparation employed. This latter limitation is due, primarily, to the concentration of total dust deposited on the filter in coincidence with the asbestos. Thus, the volume of air that can be usefully collected for asbestos analysis is restricted by the concentration of dust in the air and the quantity of dust that can be tolerated on the filter before analysis is prevented. This limit is higher for filters prepared by an indirect technique than for filters prepared by a direct technique.

The analytical sensitivity is a combined function of the volume of air sampled, the total area of the analytical filter, the dilution or concentration factor introduced during specimen preparation, and the area of the TEM specimen over which asbestos structures are counted. The interrelationship between each of these latter factors determines the relative cost of sampling and analysis.

The relationship between analytical sensitivity, the volume of air sampled, the total area of the analytical filter, the dilution or concentration factor introduced during specimen preparation, and the area of the TEM specimen over which asbestos structures are counted can be summarized by the following relationship:

$$C_s = A_f/(N \times A_g \times V \times F) \qquad (6\text{-}1)$$

where:

$C_s$ = Analytical sensitivity in structures/liter
$A_f$ = Total area of the analytical filter in $mm^2$
$A_g$ = Area of a TEM specimen grid opening in $mm^2$
N = Number of grid openings examined
V = Volume of air sampled in liters
F = Concentration factor

The concentration factor, F, simply reflects the dilution or concentration of asbestos structures resulting from an indirect preparation. During indirect preparation, asbestos structures deposited on the original sample filter are dispersed in water and redeposited on the analytical filter. The density of structures on the final filter is a function of the relative area of the two filters and the fraction of the total dispersion that is refiltered:

$$F = A_a \times V_f/(A_f \times V_r) \qquad (6\text{-}2)$$

where:

$A_a$ = Area of filter ashed in $mm^2$
$A_f$ = Area of analytical filter in $mm^2$

$V_r$ = Volume of water used to redisperse ashed particles in liters
$V_f$ = Volume of dispersion filtered through analytical filter in liters

For samples prepared by direct preparation, the concentration factor is always unity.

Actually, equation 6-1 is a contraction of a more general equation relating the number of structures counted on a filter to the total airborne asbestos concentration:

$$C = A_f \times S/(N \times A_g \times V \times F) \quad (6\text{-}3)$$

where:

C = Airborne concentration of asbestos in structures/liter
S = Number of structures counted
$A_f$ = Total area of the analytical filter in $mm^2$
$A_g$ = Area of a TEM specimen grid opening in $mm^2$
N = Number of grid openings examined
V = Volume of air sampled in liters
F = Concentration factor

The original equation 6-1 can be obtained from equation 6-3 simply by setting the number of structures counted equal to one and substituting the analytical sensitivity for the airborne concentration of asbestos.

NOTE

The required analytical sensitivities for total asbestos structures and for structures longer than 5 $\mu$m were derived as indicated in Section 4.3 using equation 6-3 to relate target concentrations to analytical sensitivity. The range of target concentrations were derived as indicated in Section 3.5.

### 6.2.1. Filter Loading

TEM analysis can proceed as long as the majority of the particles on a filter are deposited directly on the filter and do not overlap other particles present. Both asbestos and non-asbestos structures may potentially obscure observation of other asbestos structures, but at concentrations typical of environmental samples, non-asbestos structures (total suspended particulate) generally determine the upper limit to filter loading.

Based on experience, analysis of a TEM grid becomes impossible at a relative coverage of 25%. Analysis is difficult when the fraction of the specimen grid covered by particles exceeds 10%. Based on private conversations with several microscopists, experience suggests that a loading of 10 $\mu g/cm^2$ on

a filter will result in 10% of the filter area being covered with particles. The data of Steen et al (1983) is in general agreement with this estimate but indicates a range of potentially appropriate values. Steen et al (1983) report that the maximum tolerated dusts on filters are in the range of 50 $\mu g/cm^2$ for filters prepared by direct preparation and 250 $\mu g/cm^2$ for filters prepared by indirect preparation. Higher loadings can be tolerated by indirect preparation because much of the interfering particle is removed during filter preparation.

The reported variation in tolerable loading is not surprising. The fraction of filter covered per unit weight of deposit varies as a function of the average size and density of the particles. For spherical particles, the fraction of coverage expected as a function of the size of the particles can be estimated by taking the quotient of the calculated mass of a particle (the product of volume and density) and the calculated circumference of the particle. The majority of the non-asbestos fraction of any dust found in the environment is expected to be composed primarily of particles that are approximately spherical. Based on such calculations, 10% of the filter will be covered at a loading of 10 $\mu g/cm^2$ when the average particle diameter on the filter is greater than approximately 1 $\mu m$ at an average particle density of 2.3 $g/cm^3$ (the average density of many silicates). However, in different environments, this value may vary significantly. Generally, the smaller the particles, the less the mass that can be tolerated before the filter becomes overloaded. Much of the variation in loadings reported as tolerable is likely due to differences in the average size of the particles present in the dust.

### 6.2.2. Sampled Air Volumes

The volume of air that can be collected during asbestos analysis is limited both by the technical constraints of the sampling system and by the tolerable limit of total dust that may be deposited before asbestos analysis becomes impossible due to overloading (as described in Section 6.2.1). The volume of air that can be collected per unit area of filter is related to the tolerable loading by the airborne concentration of total suspended particulate (see Section 3.6). However, because of the extent of variation in the concentration of airborne particulate (TSP), it is difficult to derive usable general averages suitable for deriving the tolerable upper limit to the volume of air that can be sampled for an asbestos analysis. At the same time, some data are available from limited published studies that provide an indication of reasonable upper limits.

(a) In a series of studies of airborne asbestos both in the vicinity of the Quebec mines and at remote urban and rural locations, Sebastien et al (1984 and 1986) report that on the order of 1 $m^3$ of air may be collected per $cm^2$ of filter for filters that are prepared by a direct technique. The tolerable limit for indirectly prepared samples is approximately an order of magnitude higher.

(b) In a study of building atmospheres (Chatfield, ed. 1985) an optimum loading of 0.3 $m^3$ of air per $cm^2$ of filter was recommended when filters were to be prepared by a direct technique.

(c) In one of a series of abatement studies, Tuckfield et al (1988) report that 2 of 24 filters could not be analyzed due to overloading when 0.6 $m^3$ of air was collected per $cm^2$ of filter and the filters were prepared by a direct technique.

(d) In a study of rural, suburban, and urban air, Chatfield (1983b) reports that filters prepared by direct analysis were loaded to 1.2 $m^3/cm^2$.

(e) At the South Bay Superfund site in California, only a small number of filters out of a total of more than 60 filters could not be analyzed due to overloading when 1.1 $m^3$ of air was collected per $cm^2$ of filter and the filters were prepared using a direct technique (unpublished results).

(f) Burdett (1985a) reports from a study in England that directly prepared samples of amosite and crocidolite from abatement sites could be loaded for analysis to a level of 0.5 $m^3/cm^2$.

The above data indicate that slightly more than 1 $m^3$ of air can be collected per $cm^2$ of filter before environmental samples become overloaded so that analysis is prevented for filters prepared by a direct technique. The corresponding limit for abatement samples appears to be on the order of 0.5 $m^3$ per $cm^2$ of filter. Better estimates for optimum loading can be derived from data on local TSP concentrations, when such information is available.

### 6.2.3. Area to be Scanned for Analysis

Equation 6-1 may be graphed to depict the combination of sampling requirements and analytical requirements necessary to obtain a particular analytical sensitivity. The two curves presented in Figure 6.1 depict the relationship between the volume of air to be sampled and the number of grid openings to be counted to achieve an analytical sensitivity of 0.5 s/L and 0.02 s/L. These, in turn, represent the analytical sensitivities required to monitor, respectively, total asbestos structures and long asbestos structures in environmental samples (see Section 4.3). To generate the figure, it is assumed that an average grid opening size is 0.0081 $mm^2$, the area of the analytical filter is 25 $mm^2$, and the concentration factor is unity. The vertical axis for the curve representing an analytical sensitivity of 0.02 s/L is presented on the right vertical axis of the figure while the vertical axis for the 0.5 s/L curve is presented on the left axis.

Based on cost considerations, it is optimal to minimize the number of grid openings to be counted during analysis. Experience also indicates that analysts tire more rapidly when forced to count an excessive number of grid openings on a single sample, so that precision suffers. It is recommended

that no more than 20 grid openings be counted at the magnification of 20,000 recommended for characterizing total asbestos structures. Similarly, 100 grid openings is a recommended target for the 10,000 magnification employed to characterize long asbestos structures.

Based on the information presented in Figure 6.1, the recommended limit for counting grid openings can be satisfied by collecting a sample volume of 5,000 liters for characterizing total asbestos structures. For a 25 mm filter, this corresponds to 1.2 $m^3/cm^2$, which in most cases probably will not cause excessive overloading of filters prepared by direct analysis. However, a sample volume of 25,000 liters would be necessary to remain within the recommended grid opening count for long asbestos structures, corresponding to a loading of 6.5 $m^3/cm^2$. This clearly exceeds the recommended maximum loadings defined above for filters prepared by direct techniques. Therefore, indirect preparation is recommended to keep the number of grid openings that must be counted within reason (see Section 6.2.4).

NOTE

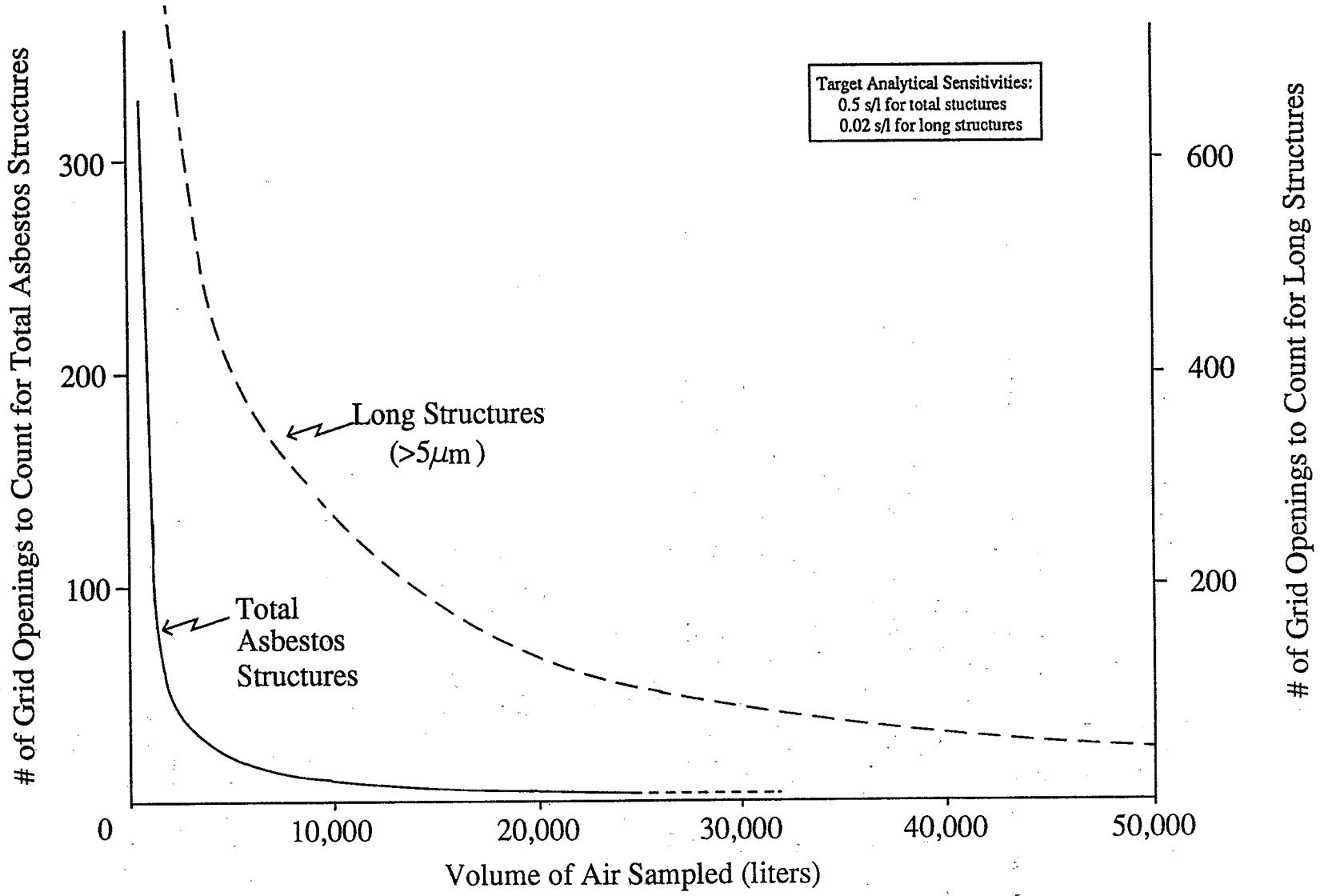
These calculations ignore the limitations imposed by filter blank contamination, which are addressed in Section 6.3.1.

### 6.2.4. Filter Preparation

To provide the required analytical sensitivity at asbestos levels typically found in the environment, it is necessary either to selectively concentrate asbestos, or to count structures over a much greater area of a TEM specimen grid than has traditionally been required. To the extent that it can be employed, selective concentration is the least costly of the alternatives for increasing sensitivity. The desired degree of concentration requires filter loadings in excess of what can generally be analyzed using direct-transfer methods for TEM specimen preparation. Consequently, an indirect technique for TEM specimen preparation is employed in this method.

In most ambient environments, a proportion of suspended particulate is organic, consisting of soot, spores, pollens and other debris from vegetation. Organic materials such as these can be removed from the analysis by low-temperature ashing. It is also common to find substantial numbers of calcium sulfate fibers (gypsum) in airborne particles collected in buildings and urban environments, and particularly in samples collected where demolition or construction work is in progress. The fibers are readily released when plasters and cement products are disturbed. Gypsum is also generated on air filters as a consequence of the chemical reaction between collected calcium carbonate particles and atmospheric sulfur dioxide. Gypsum can be removed by dissolution in water. Another major component of exterior atmospheres is limestone, either as calcite or dolomite. Such carbonates, along with some oxide species, can be removed by extraction with hydrochloric acid.

FIGURE 6.1: THE RELATIONSHIP BETWEEN ANALYTICAL SENSITIVITY, SAMPLED AIR VOLUME, AND THE NUMBER OF GRID OPENINGS

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NOTE

Since the mineralogy of airborne dusts is related to local geological and meteorological conditions, the ability to concentrate asbestos from dusts by removal of soluble and ashable components varies as a function of location.

Removal of a large proportion of the suspended particulate by use of these techniques permits the asbestos present in the sample to be concentrated on to a smaller area of the TEM specimen. Consequently, the area of the TEM specimen that must be examined to achieve a particular analytical sensitivity is proportionately reduced. Also, many of the non-asbestos fibrous structures normally found in a directly-prepared TEM specimen, each of which must be identified and rejected from the asbestos structure count, do not appear on the indirectly-prepared TEM specimen.

A selective concentration of the asbestos structures, incorporating low temperature ashing, re-suspension in water, and acidification with HCl is therefore capable of removing substantial amounts of interfering particle from the analysis, and, for a particular analytical sensitivity, reduces the area of the TEM specimens that must be examined. If these procedures are carried out correctly, no chemical or crystallographic degradation of asbestos can be detected by routine methods of TEM analysis.

Indirect TEM specimen preparation techniques offer several advantages over direct preparation:

(a) air samples can be collected without regard to the amount of deposit on the filter surface. The filter loading can be adjusted in the laboratory to provide satisfactory TEM specimens;

(b) interfering particulate material can be completely or partially removed through a combination of dissolution and oxidation (ashing);

(c) the uniformity of distribution of asbestos structures on the filters to be analyzed is improved.

The middle of the above advantages has been addressed above. The first of the above advantages is addressed in greater detail in Section 6.2.1. The last of the advantages listed is addressed in Section 6.4.3.

Despite the advantages, use of indirect preparation techniques must be considered carefully because they also suffer from the following disadvantages:

(a) the size distribution of asbestos structures is modified;

(b) there is increased opportunity for fiber loss or introduction of extraneous contamination;

(c) when sample collection filters are ashed, any fiber contamination in the filter medium is concentrated.

The second of the above disadvantages can be minimized by maintaining a clean laboratory and following proper experimental procedures (see Section 6.3). The last of the above considerations is addressed in Section 6.3.1. The extent that structure size distributions are modified during the indirect preparation of a TEM specimen has been addressed in several studies.

(a) In a set of studies, Sebastien et al (1984 and 1986) found that samples prepared by an indirect technique exhibit 20 to 50 times as many short fibers as paired specimens prepared by a direct technique. Changes in the number of structures longer than 5 $\mu$m were not significant. It was noted in the study that the absolute concentration of aggregates encountered remained the same whether samples were prepared by the indirect or the direct technique. However, the average dimensions of the aggregates encountered on the indirectly prepared specimens appeared to be smaller than those observed on the directly prepared specimens. Because of the increase in the number of short structures, the fraction of total structures represented by long structures decreased. Possible sources of the increased number of short structures presented in the paper are losses during analysis of the directly prepared specimens due to obscuration, liberation of short structures from soluble matrices dissolved during indirect preparation, and disassociation of short structures loosely bound to the larger aggregates.

(b) Chatfield (1985d) in a comparison of specimens prepared by the direct and the indirect techniques found that structures shorter than 2.5 $\mu$m were 4 to 20 times more plentiful on specimens prepared by the indirect technique than on specimens prepared by the direct technique. The conversion factor for structures longer than 2.5 $\mu$m is less than a factor than 2. He also noted agreement in results between direct preparations of polycarbonate and MCE filters and indirect preparations of polycarbonate and MCE filters. For the indirect preparations, the deposits on polycarbonate filters were washed into suspension while deposits on MCE filters were ashed. Data were not reported for the process of washing MCE filters.

(c) In another study, Chatfield (1985b) reports that for single fibril suspensions of chrysotile, there is no difference in results obtained from specimens prepared by direct and indirect techniques either on polycarbonate or MCE filters. For suspensions that contain aggregates, which are more representative of the types of samples likely to be encountered in the environment, PCME counts were comparable for direct and indirect preparations on both polycarbonate and MCE filters but counts of structures shorter than 2.5 $\mu$m increased on indirectly prepared specimens. Chatfield

did not comment on the fate of long, thin structures in this study. It is unknown whether the source of the additional short structures observed following indirect preparation is from disaggregation or disintegration.

(d) In a study reported by Cook and Marklund (1980), the ratio of amphibole asbestos structure counts on paired specimens prepared, respectively, directly and indirectly varied between 0.3 and 5.1 with a mean of 2. Results from the direct and indirect preparations correlated to 95% significance. Cook and Marklund also report that results with chrysotile are much more problematic. Chrysotile samples that contained a high fraction of aggregates exhibited counts varying by four orders of magnitude from different laboratories (possibly due to lack of standardization). Unfortunately, there is insufficient documentation in this paper to determine the exact conditions under which specimens were prepared. However, they do report significant increases in the total mass of chrysotile observed on indirect preparations subjected to "harsh" preparation conditions.

NOTE

There may be a range of problems contributing to the observations presented in this study. At the time the study was conducted, little was known of the effects of organic materials on aqueous suspensions of asbestos so that four orders of magnitude variation could well have resulted from loss of asbestos from suspension onto container walls, etc.

(e) In a series of studies of abatement sites, Tuckfield et al (1988), Hatfield et al (1988), and Chesson et al (1985), directly prepared specimens consistently generated lower structure counts than indirectly prepared samples. Too few structures were counted on any of the preparations to derive size specific information. However, they did report that counts from paired direct and indirect preparations did not correlate.

(f) Chesson et al (1989b) performed a statistical evaluation of data from five independent studies where samples prepared, respectively, by direct-transfer and by indirect-transfer techniques could be paired for comparison. Results indicate that samples prepared by an indirect technique uniformly exhibit higher total structure counts and higher total fiber counts. Unfortunately, the data were not sufficient to determine the effect of indirect preparation on specific size fractions such as the long structures.

The Chesson et al study also indicates that, although counts on directly and indirectly prepared specimens are not strictly proportional across studies, they both track similar trends in

concentrations so that the choice of preparation method is unlikely to affect comparisons between samples from the same study. A statistically significant relationship was found in all five studies between the two sets of specimens (direct and indirect preparations). Quantitative relationships between samples prepared by the two techniques are expected to depend both on the characteristics of the dusts sampled and on the specifics of the sampling and analysis protocols employed in a particular study.

(g) In a study of amosite, Burdett (1985b) reported a high correlation between samples prepared by a direct technique and those prepared by an indirect technique. The observed ratio of indirect to direct varied between 1.7 to 1. The slight increase in counts on the indirectly prepared specimens was attributed to splitting of aggregates. Correspondingly, the average length and diameter measured for structures observed on indirect preparations were reported to decrease slightly. It is noted that surfactant was used during indirect preparation in this study.

Based on these findings, specimens prepared by an indirect technique are appropriate for comparing relative asbestos concentrations at a site. In addition, such specimens are likely to yield representative counts of long structures that correspond to counts on samples prepared by direct transfer, provided that gentle conditions are employed during the preparation. However, counts of asbestos structures of all sizes (which include short structures) or counts of individual components within complex asbestos structures of any size fraction likely vary when performed on specimens prepared by the indirect technique compared to specimens prepared by the direct technique. In either case, quantitative relationships between directly and indirectly prepared specimens are likely to be study specific due to a dependence on the sampling and analysis protocols employed.

The magnitude of differences between counts of long, thin structures (potentially the most biologically active) performed on directly prepared and indirectly prepared specimens has not been adequately addressed. Relationships between counts of asbestos structures, other than single, long fibers, obtained from specimens prepared by a direct technique or an indirect technique, respectively, appear to depend on the fraction of aggregates present in the asbestos dust. Therefore, they are likely to be site specific as well as study specific.

The question as to whether direct or indirect specimen preparation yields the "correct" result (in terms of representing biological activity) is currently unresolved. It can be argued that the direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, the indirect methods can be considered to

yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

Because of critical limitations imposed by the sensitivity requirements for sampling environmental asbestos and because there is little reason to believe that direct preparations track relative risks better than indirect preparations, an indirect preparation technique is incorporated in the method presented in Part 1. However, assessing absolute risks requires that current measurements be compared with risk factors derived from historical measurements (PCM) that correspond to direct preparations. Therefore, the method also incorporates a procedure for evaluating the relationship between results from directly and indirectly prepared samples for any study employing this method.

A small subset of sample filters will be split so that one half of each filter may be prepared by a direct and one half by an indirect preparation technique to allow detailed evaluation of the effects of preparation. The total loading on this subset of filters will be restricted to assure that the level of dust can be tolerated by analysis of the directly prepared filter section. Consequently, a larger number of grid openings will be counted on these samples to achieve desired sensitivity and precision.

**NOTE**

The effect of an indirect preparation is heavily dependent on the specific procedures employed. A detailed protocol has been incorporated into this method.

As indicated by the Burdett study (1985b), variation in fiber counts between samples prepared by direct techniques versus indirect techniques appear to occur primarily for preparations of chrysotile. Preparations of amphiboles likely are relatively unaffected by the choice of preparation technique.

### 6.2.5. Existing Analytical Methods

Equation 6-3 is graphed in Figure 6.2 to illustrate the relationship between sampling and analysis constraints and the concentrations of airborne asbestos. In the log-log plot of Figure 6.2, the vertical axis represents the volume of air sampled, the horizontal axis represents the number of grid openings counted, and the diagonal lines represent the airborne concentrations (analytical sensitivities) of asbestos at which at least 1 structure can be expected to be encountered during analysis. Because it is a log-log plot, the hyperbolic curves depicted in Figure 6.1 are represented by straight lines in Figure 6.2. The upper curve corresponds to the required analytical sensitivity for structures longer than 5 $\mu$m (0.02 s/L) and the lower curve corresponds to the analytical sensitivity required to characterize asbestos

structures of all sizes in environmental samples (0.5 s/L). As Figure 6.1, it is assumed that an average grid opening is 0.0081 $mm^2$, that the analytical filter is 25 $mm^2$, and the concentration factor is unity.

The horizontal line representing 5 $m^3$ has been emphasized in Figure 6.2 because this volume of air corresponds to 1.2 $m^3/cm^2$ on a 25 mm filter, which is the maximum volume of air that can be tolerated for a direct preparation (see Section 6.2.1). Published TEM methods discussed in Section 3.8 are graphed along the 5 $m^3$ line at points corresponding to the number of grid openings specified to be counted in each method. Thus, each of these points represent the effective analytical sensitivities for the methods at the maximum reasonable volume of air that can be collected on a 25 mm filter. The Hayward method is depicted twice to represent the analytical sensitivities corresponding, respectively, to the high magnification scan and the low magnification scan defined in this method.

As is readily apparent from the figure, only NIOSH 7402 yields sufficient sensitivity to detect asbestos structures of all sizes over the range of concentrations expected to be found in environmental samples. The Yamate method, the method specified in AHERA, and the high magnification scan of the Hayward method would not provide sufficient sensitivity to detect the entire range of concentrations expected for environmental asbestos. None of the methods depicted in Figure 6.2 (including the low magnification scan of the Hayward method) are sufficiently sensitive to characterize the expected range of concentrations of long structures (longer than 5 $\mu$m) in environmental samples. In the method presented in Part 1 of this report, the number of grid openings to be counted is determined by specifying the analytical sensitivity, specifying the volume of air to be collected, and calculating the required number of grid openings to be counted using equation 6-1.

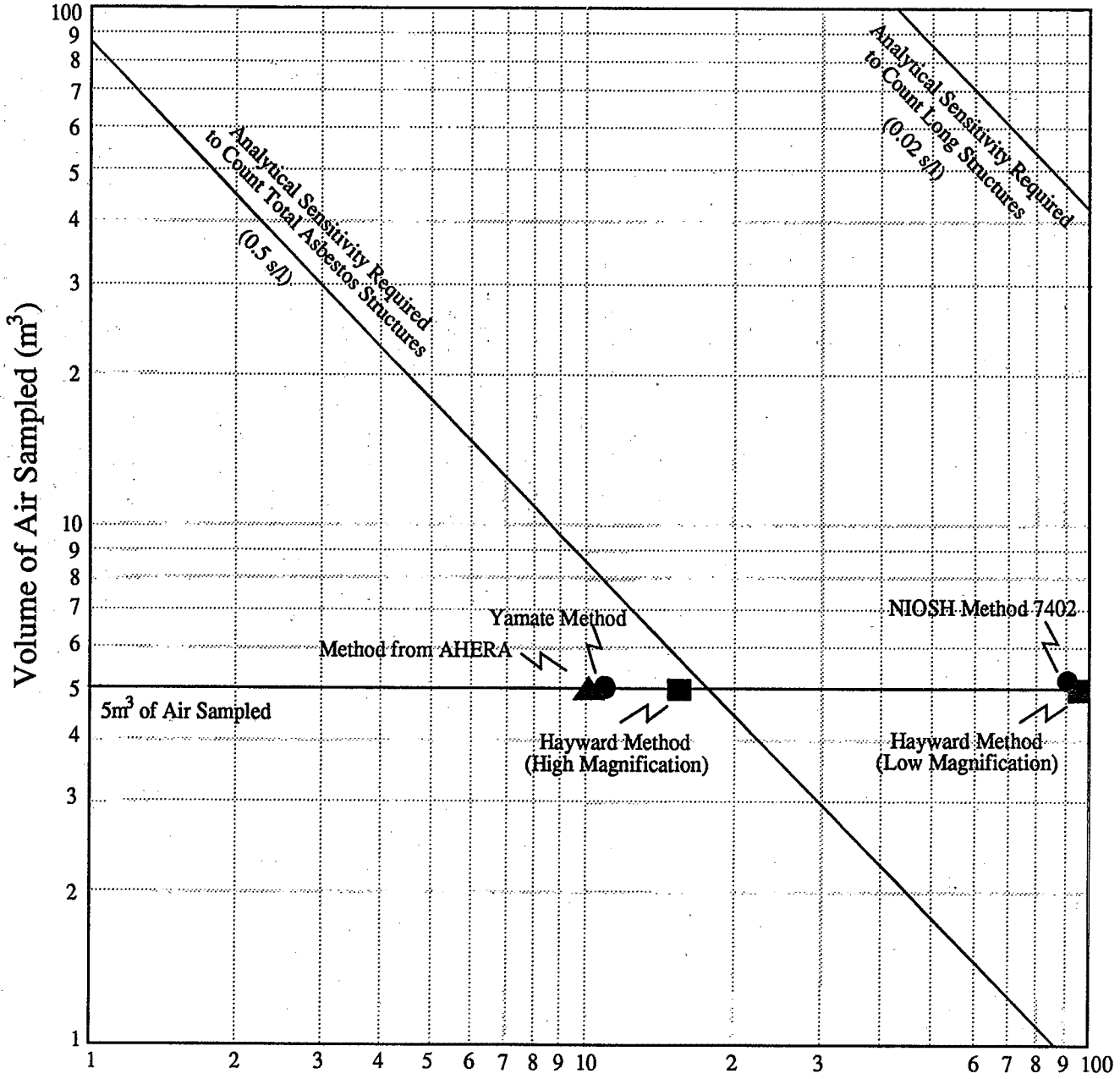
### 6.2.6. Analytical Technique

TEM is potentially capable of achieving the level of analytical sensitivity required for this method.

### 6.2.7. Magnification/Resolution

As noted in Section 3.4 and 4.2, total asbestos structures and asbestos structures longer than 5 $\mu$m are counted separately in this method because of the disparity in their abundance. Based on the size distributions discussed in Section 3.4, the probability of encountering an asbestos structure longer than 5 $\mu$m in an asbestos dust is only one tenth to one hundredth as likely as encountering an asbestos structure of any size. Given the disparity, the most cost-effective manner for analyzing the population of total structures and long structures representatively is to count them separately. If they were to be counted together and the limit for total structures was employed as the defined level of sensitivity, then the chance of encountering even 1 long structure while counting 10 total structures is very small. On the other hand, using the median for long structures as the defined limit of sensitivity

FIGURE 6.2: THE RELATIVE ANALYTICAL SENSITIVITIES OF PUBLISHED TEM METHODS

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means that likely 500 total asbestos structures would have to be counted at the same time that 10 long structures are identified. Characterizing such a large number of structures is extremely time consuming and expensive.

## 6.3. FACTORS AFFECTING THE LIMIT OF DETECTION

The limit of detection is defined as the smallest measurement that is unlikely to be due entirely to contamination from sources other than the air being sampled. More simply, it is the minimum environmental concentration that can be distinguished (with specified probability) from analytical background. To this point, the discussion of performance requirements (in terms of the analytical sensitivity) have been developed assuming zero analytical background. However, several published studies indicate that the analytical background for this method is potentially a critical factor affecting method performance.

Analytical background may derive both from filter blank contamination and from contamination contributed by the method of filter preparation. During an indirect preparation, for example, background contamination can arise from within the asher chamber, the containers in which the filters are ashed, the distilled water supply used for re-dispersing the ash, the pipettes used for transfer of the dispersion for filtration, and the filtration funnel[2]. However, while contamination introduced during filter preparation can be minimized by adherence to a rigid program of laboratory cleanliness, filter blank contamination is simply a function of the quality of commercially available filters.

### 6.3.1. Filter Blank Contamination

Asbestos contamination observed on membrane filters has varied historically. The ranges of filter blank contamination also differs for MCE and polycarbonate filters and may differ for different size filters of each type. In addition, the extent to which asbestos contamination on such membrane filters may contribute to background contamination during analysis depends on the procedure employed for preparing the sample. Unfortunately, studies reporting levels of asbestos filter blank contamination frequently omit critical information concerning either the filter type, size, date of purchase, or the preparation technique used in the analysis. The available

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[2] Some microscopists also suggest that asbestos losses may occur during sample collection and transport when asbestos structures are shaken off of the filter and redeposited on the cowl or cassette. Others contend that this is not a significant problem and that the data used to identify it is flawed by not controlling for the effects of the differences between direct and indirect preparation. The problem has yet to be confirmed in a published study.

data appear contradictory. Consequently, it is difficult to quantify a representative range of asbestos contamination levels for filter batches that are available today.

#### 6.3.1.1. Contamination on MCE Filters

Prior to 1975, the level of contamination on MCE filters prepared by the direct method was reported in some batches to be sufficient to preclude use in standard asbestos analysis (Chatfield, 1985a). Prescreening of filter batches was therefore recommended. According to current consensus, levels of contamination detected on MCE filters prepared by a direct preparation now appear to be minimal and these filters are acceptable for most asbestos work. However, published data is limited and published data addressing 25 mm filters specifically is lacking.

(a) In a study of indoor asbestos contamination, Hatfield et al (1988) report 1 chrysotile asbestos fiber detected on 1 of 19 MCE filter blanks (37 mm, 0.45 $\mu$m pore size). This corresponds to an upper limit to background of less than 12 s/mm$^2$.

(b) The EPA study on filter blank contamination (USEPA, 1986b) concluded with the observation that contamination on MCE filters appeared to be composed predominantly of short chrysotile structures and that MCE filters are recommended for asbestos sampling and analysis.

(c) Chatfield (1985d) also reports that most of the contamination on filter blanks is chrysotile and that current levels of blank contamination are acceptable. This paper also suggests that short fibers predominate, although this observation may be due simply to lack of a statistically balanced count.

For MCE filters prepared by an indirect technique, the situation is less clear. General consensus indicates that levels of contamination are acceptable when MCE filters are ashed. But published data, especially for recent batches of 25 mm filters are limited. Published data that address background contamination resulting from the washing of MCE filters are lacking.

(a) In a study of abatement techniques, Chesson et al (1985) report detection of no asbestos contamination on laboratory blanks and field blank MCE filters (47 mm, 0.45 $\mu$m pore size) prepared by ashing the filter and resuspending the deposit in distilled water by sonication. This means that background contamination on the filters is less than 12 s/mm$^2$ based on the analysis performed. Note that the filtrate was redeposited on a polycarbonate filter (25 mm, 0.2 $\mu$m pore size) so that the background could also represent an upper limit to contamination on polycarbonate filters subjected to direct preparation.

(b) In a followup study of abatement techniques, Chesson et al (1986b) report levels ranging between 0 and 58 s/mm$^2$ with a mean of 30 s/mm$^2$ on laboratory blank MCE filters (47 mm, 0.45 $\mu$m pore size) prepared by ashing the filter and resuspending the deposit in distilled water by sonication. Field blanks appear to exhibit a slightly higher average asbestos contamination level than the laboratory blanks. Note that the filtrate was redeposited on a polycarbonate filter (25 mm, 0.2 $\mu$m pore size) so that the background could also represent contamination due to direct preparation of a polycarbonate filter.

(c) In another followup to the series of abatement papers, Tuckfield et al (1988) report levels ranging between 0 and 50 s/mm$^2$ with a mean of 20 s/mm$^2$ on 30 field and laboratory blank MCE filters (47 mm, 0.45 $\mu$m pore size) prepared by ashing the filter and resuspending the deposit in distilled water by sonication. Note that the filtrate was redeposited on a polycarbonate filter (25mm, 0.2 $\mu$m pore size) so that the background observed could also represent contamination due to direct preparation of polycarbonate filters.

(d) Individual microscopists (for example, G. Dunmeyer, 1988) have indicated in private conversations that indirect preparation by washing MCE filters results in efficient transfer of asbestos and that blank contamination is low (less than 15 s/mm$^2$). Filters considered were 25 mm, 0.8 $\mu$m pore size MCE filters. However, there are no published data to confirm such reports.

#### 6.3.1.2. Asbestos Contamination on Polycarbonate Filters

The level of asbestos contamination on polycarbonate filters has historically been reported to be higher than MCE filters. Available data addressing current levels of contamination on polycarbonate filters is inconclusive for filters prepared by direct preparation and published data addressing background contamination on polycarbonate filters prepared by an indirect technique (either ashing or washing) appears to be lacking. General consensus indicates that background contamination on current batches of polycarbonate filters is generally higher than MCE filters when both are prepared by the direct technique. However, published data are limited.

(a) In an abatement study, Tuckfield et al (1988) prepared 47 mm polycarbonate filters (pore size not reported) by a direct technique and measured the background on a series of laboratory and field blanks. Levels reported range between 0 and 40 s/mm$^2$ with an arithmetic mean of 7 s/mm$^2$. Note that an amphibole fiber was detected on one of the blanks.

(b) In the workshop on blank contamination (USEPA, 1986b), it was reported that polycarbonate filters prepared by a direct technique exhibit variable levels of blank asbestos contamination. Levels

appear to average about 2-6 structures/10 grid openings (25-75 structures/$mm^2$) but particular lots may contain 10 times this amount. It was recommended that filter lots with more than 5 structures/10 grid openings (60 s/$mm^2$) be returned to the manufacturer. In the same study, use of MCE filters was recommended because polycarbonate blank contamination appears to include both chrysotile and amphibole and a significant fraction of structures longer than 5 $\mu m$ were encountered.

(c) Chatfield (1985b) reports that although contamination of MCE filters appears to have been minimized, contamination of polycarbonate filters still appears to be a problem.

(d) The studies reported in Section 6.2.1.1 indicate that an upper limit to filter blank contamination on polycarbonate filters prepared by a direct technique is 30 s/$mm^2$ based on contamination observed following an indirect preparation of MCE filters in which polycarbonate filters were used to filter the final suspension.

(e) There appears to be a consensus among several microscopists that at least one group of microscopists consistently report lower values for polycarbonate filter contamination. However, there is no published data addressing this discrepancy and it has not been resolved.

#### 6.3.1.3. Estimating Filter Blank Contamination Levels

Although qualitative conclusions may be drawn from the available data, additional research is recommended in this area. General consensus indicates that most current batches of MCE filters, when prepared by a direct technique, should yield filter blank contamination levels below 30 s/$mm^2$. MCE filters prepared by ashing should also yield background below 30 s/$mm^2$. Washing of polycarbonate filters yields comparably low levels of background contamination. Published data, however, is too sparse to support any of these numbers as typical. Therefore, at a minimum, duplicate filter blanks from every batch used in a study should be prepared and analyzed as per the technique to be used in the study to quantify the level of background.

If the range of filter blank contamination found during a preliminary evaluation is not clearly distinguishable from typical levels expected to be encountered during the study, either the filter batch should be returned for a new one (which must also be tested) or the study protocol should be redesigned. It is assumed for this method that filter blank contamination can be maintained below 30 s/$mm^2$. It is further assumed that the size distribution of asbestos appearing as filter blank contamination parallels typical chrysotile distributions as presented in Section 3.4. Therefore, if the count of structures of all sizes exceeds background, the count of any size fraction will also exceed the level of background corresponding to that

particular size fraction. To minimize interference with filter blank contamination, 60 s/mm$^2$ is set as a target minimum concentration for this method.

NOTE

The potential impact of asbestos contamination contributed by the membrane filter can be minimized by maximizing the volume of air collected per unit area of filter. However, collected air volumes must be limited to prevent overloading, especially for filters to be prepared by a direct-transfer technique (see Sections 6.2.1 and 6.2.4).

Within the range of the maximum air volume that can be tolerated on a filter due to constraints associated with filter loading, there is also the problem of the limit of the rate at which air can be pumped through a filter given the capabilities of available pumps (see Section 2.2 of The Method, Part 1 of this report). If the capabilities of the pump are limiting in a given situation, the total volume of air to be collected can theoretically be minimized without compromising analytical sensitivity by selecting the smallest filter (25 mm) that is practical for sample collection.

### 6.3.2. Conclusions

Although specification of a detection limit for this method requires knowledge of the range and variation of analytical background and is therefore study specific, general conclusions concerning the ability to achieve a reasonable limit of detection may be drawn from a comparison of the relative contributions to asbestos counts from environmental and analytical sources. For filters prepared for TEM analysis in a particular study, the absolute limit to detection is unlikely to be lower than the concentration at which the density of structures deposited on the filter from the air is just equal to the mean density of structures present on the blank filter (30 s/mm$^2$). Ideally, however, sampled asbestos should be deposited on the filter at several times the concentration of asbestos present due to analytical background so that distinguishing sampled asbestos from analytical contamination is trivial.

Given the upper range of the target concentrations derived in Section 3.5 (5 s/L for total asbestos structures and 0.2 s/L for long asbestos structures), an air volume of 1 m$^3$ of air per cm$^2$ of filter will deposit 50 asbestos structures of all sizes per mm$^2$ of filter and 2 long structures per mm$^2$ of filter respectively. The fact that the first of these numbers exceeds projected filter blank contamination levels by less than a factor of 2 indicates that preparing these filters by a direct technique, which is subject to the 1 m$^3$/cm$^2$ limitation, likely presents a marginally acceptable method for characterizing asbestos structures of all lengths, at best. Direct preparation is clearly unacceptable for characterizing the concentrations of long structures, because the major contribution to observed asbestos will be

from analytical background. Therefore, an indirect preparation protocol is incorporated in this method. The consequences of the selection of an indirect preparation procedure are addressed in Section 6.2.4.

NOTE

At a limit of 30 s/mm$^2$ on a 25 mm filter with 1 m$^3$/cm$^2$ air collected, 1 structure observed corresponds a concentration (analytical sensitivity) of 3 s/L for structures of all sizes and 0.1 s/L for structures longer than 5 $\mu$m. Clearly, limiting the quantity of air that can be collected to 1 m$^3$/cm$^2$ (as is the case for a direct preparation) is not sufficient to achieve the required analytical sensitivities defined for this method (see Sections 4.3 and 6.2).

## 6.4. FACTORS AFFECTING PRECISION

To maximize precision, potential sources of variation within the method must be controlled. To minimize systematic variation during sampling and analysis, the method specifies detailed procedures. Random variation is minimized by maximizing the number of structures to be identified and counted, which includes increasing the probability of encountering asbestos structures during analysis, as discussed in Section 6.2. In addition, detailed and unambiguous counting rules and rules for identification are specified in the method to minimize variation due to subjective interpretation. Specific factors that must be controlled to maximize precision are:

(a) generating a uniform deposit on the analytical filter;

(b) minimizing subjectivity in the interpretation of counting rules;

(c) minimizing variation introduced by small changes in instrument performance; and

(d) minimizing statistical variation from counting too small a number of structures.

### 6.4.1. Analytical Technique

TEM is the analytical tool selected for this method so that analysis can be performed at sufficient magnification to minimize variation due to small differences in the performance characteristics of the instrument or the experience of the analyst. However, several performance criteria must be specified to minimize the impact on precision.

In a comparison study, Steel and Small (1985) report that TEM instruments vary with age and type. Differences between instruments that contribute the most to analytical variation include image quality (resolution), contrast, and brightness. Electron beam dose and mechanical stage operation are also important. Resolution was judged by the ability to distinguish the hollow core of chrysotile fibrils. Contrast and brightness

are controlled by adjusting the voltage and aperture sizes of the instrument. In the study, not all instruments could be configured to perform equally well. Some of the oldest TEM instruments could not be adjusted to provide sufficient resolution to observe the central canals of chrysotile fibrils clearly.

The beam dose effects the counting process by causing damage. If too high a beam current is required by an instrument to achieve the required brightness or resolution, than the potential to damage the sample may become so great that the ability to scan properly may be lost. This is particularly true when attempting to generate SAED spectra.

The operation of the mechanical stage is crucial to achieve good precision in counts. Approximately 20-30 traverses are needed to complete a scan of a single grid opening. Thus, the extent that a stage wanders during a traverse may affect the time necessary and the ability to precisely count all fibers in a grid opening. Some of the stages tested by Steel and Small wandered by 30 $\mu$m during a single traverse. However, the TEM used for the operator tests in the study wandered by less than 1 $\mu$m in a 100 $\mu$m linear traverse.

Based on the work of Steel and Small, it is recommended that TEM instruments to be used with this method exhibit a resolution at 20,000 magnification that is sufficient to clearly resolve the central canal of the finest chrysotile fibril. In addition, the mechanical stage of the instrument must be shown to wander by less than 1 $\mu$m per 100 $\mu$m linear traverse. Finally, it is recommended that specific performance criteria addressing beam dose, brightness, and contrast be developed for instruments to be used with this method.

### 6.4.2. Magnification/Resolution

The precision of this method is affected by the magnification selected to the extent that the ability to detect asbestos structures lies close to the limit of the associated resolution. Microscopists vary in their ability to detect very small asbestos structures at magnifications used for scanning in this method, 10,000 and 20,000, (Steel and Small, 1985). It has been reported that only 50% of asbestos structures shorter than 0.5 $\mu$m were detected by all analysts in a round-robin study. Below this length, the fraction of analysts who regularly detect such structures decreases rapidly. At the same time, proper characterization of the size fractions present in an asbestos dust requires that the mode of the distribution be included in the count. The published size distributions presented in Section 3.4 indicate that the mode of asbestos length distributions in most dusts falls within a range from 0.8 $\mu$m to 1.2 $\mu$m. Therefore, as a reasonable compromise allowing the detection of the mode of asbestos distributions while addressing the limited ability of analysts to detect short structures, a minimum length of 0.5 $\mu$m has been defined as the shortest fiber to be incorporated in the reported results for this method.

The minimum detectable width for asbestos structures counted in this method is determined by the ability of an operator to detect them in a routine examination at the specified magnification of the method. The minimum magnification specified in this method (10,000) is more than sufficient to assure visibility of the thinnest chrysotile asbestos fibrils.

### 6.4.3. Uniformity of Filter Deposit

To meaningfully extrapolate structure counts performed over a limited area to the concentration of structures on an entire filter, the nature of the distribution of structures on the filter must be known and the distribution must be reasonably uniform. Chatfield (1985b) has shown that the distribution of structures on analytical filters prepared by a direct technique are not uniform. Using a chi-squared test, Chatfield found that 100% of the directly prepared filters that exhibited more than 30 structures/ 10 grid openings had to be rejected as non-uniform at a 1% level of significance. The fraction of filters that passed the chi-squared test for uniformity increased as the loadings on the filters decreased, but this was attributed to the presence of an insufficient number of structures to properly determine a distribution. In contrast, nearly all of the filters prepared by indirect preparation techniques exhibited uniform deposits at a 1% level of significance and 100% of the indirectly prepared samples could be shown to be uniform at reduced levels of significance.

Due to the apparent characteristics of air deposited asbestos, directly prepared filters do not exhibit uniform deposits. Consequently, indirect preparation has been incorporated into this method.

### 6.4.4. Rules for Counting, Characterization, and Recording

Based on a study by Steel and Small (1985), operator error is one of several potential sources of variation. The problem is worse if counting procedures are not standardized. It is also reported that this variation can be reduced when counting rules are standardized and specified in detail. Performance also varies as a function of observer experience and the degree of professional time pressure under which an analysis is performed. To account for these observations, detailed standardized counting rules have been incorporated in this method. It is also recommended that minimum requirements for experience be developed for the analysts. This may include, for example, mandatory participation by analysts in round-robin studies.

Once the concentration of structures collected on a filter is sufficient to be observed, the number of structures detected affects the precision of the measurement. Precision tends to increase as the square root of the number of structures counted. This implies that the incremental improvement in precision derived from each individual structure counted decreases as the total number of structures counted increases.

Burdett (1985a) got significant improvement in precision by increasing counts between 20 and 50 structures. He reports, however, that the incremental improvement in precision for structures counted beyond 50 are minimal. The characterization of structures to be counted in this method includes distinguishing among counts of several sub-types (bundles, clusters, matrices, and fibers). Based on Burdett's data, improvements in the precision of the count of each sub-type is expected until 50 structures of each sub type are counted. However, this would require counting more than of 400 total asbestos structures, an excessive requirement. Therefore, an upper limit to the count of total asbestos structures of 50 is defined for this method and a lower level of precision is considered acceptable for the count of each sub-type. A similar strategy is adopted for structures longer than 5 $\mu$m.

NOTE

A count of 100 total asbestos structures and 100 structures longer than 5 $\mu$m is defined in this method for Phase 2 samples because of a need for increased precision in the counts of sub-types in these samples (see Section 6.4.2.

## 7. RECOMMENDED PROCEDURES FOR MANIPULATING ANALYTICAL DATA

Because the principle objective of the sampling and analysis method presented in Part 1 of this report is to provide results suitable for supporting a risk assessment, this section addresses the kinds of data manipulation typically performed to complete a risk assessment. The procedures presented here are recommended for general application. It is recognized, however, that alternate valid approaches are also available so that the recommended procedures should not be viewed as strict requirements. In addition, specific circumstances may present special computational needs beyond the scope of what is addressed here. To preserve the integrity and the intent of data derived from this method, it is recommended that a statistician be consulted prior to the adoption of alternate computational procedures for manipulating data as part of a site evaluation.

In a risk assessment, field results are typically combined to provide average concentrations representative of a given location or a given period of time. Frequently, long-term (multiple-year) time averages are desired because lifetime rather than short-term risks are generally addressed. Depending on the particular purpose for the computation, average concentrations may be derived either from homogeneous samples (samples intended to represent the same sampling environment) or from non-homogeneous samples (samples collected in different sampling environments). Procedures for deriving averages from homogeneous and non-homogeneous sets of samples are provided in Section 7.1 along with instructions for constructing confidence limits around averages of homogeneous samples (Section 7.2) and non-homogeneous samples (Section 7.3).

Once average concentrations are computed, they are generally compared to distinguish anthropogenic contamination from background or to distinguish among levels of contamination potentially contributed from different sources. Such comparisons examine the relative differences of various measurements. However, long-term average exposure concentrations are also typically compared to existing risk factors to establish the magnitude of absolute risk. This last use of analytical data relies on the absolute concentration derived from a particular set of measurements. A procedure for conducting a statistical test to distinguish among the average concentrations derived from various sample sets is presented in Section 7.4.

The significance of considering the need for a relative verses an absolute measurement is to determine whether analytical results must be adjusted to account for analytical background contamination (see Section 11.4 of Part 1 of this report). While unnecessary and not recommended for the manipulation of relative values, the adjustment for analytical background is required to derive an absolute measurement. A procedure for adjusting measurements to account for analytical background is provided in Section 7.5.

NOTE

Statistical tests should all be performed on <u>unadjusted</u> analysis results.

## 7.1. COMPUTING AVERAGE CONCENTRATIONS FROM MULTIPLE SAMPLES

As indicated above, it is frequently helpful to summarize results from a set of samples by computing an average. For several reasons, it is recommended that arithmetic averages be used exclusively. First, averages are often used to judge risk to health and such risk is more likely to be a function of the arithmetic mean than, say, a geometric mean (e.g., the risk from breathing 0.2 f/ml 12 hours per day and 0.00002 f/ml 12 hours per day is probably closer to the risk from constantly breathing 0.1001 f/ml [the arithmetic mean] than breathing 0.002 f/ml [the geometric mean]). Second, the geometric mean is zero whenever one of the individual samples results is zero, and ad hoc adjustments to address this problem (such as replacing zero estimates by detection limits) can introduce serious biases.

If samples are considered to be homogeneous (designed to represent the same sampling environment), it is recommended that simple un-weighted arithmetic averages be calculated. For example, denote the calculated airborne concentrations from a group of K samples by $C_1$, ..., $C_K$. The estimated average concentration, $\overline{C}$, would then be

$$\overline{C} = (C_1 + \ldots + C_K)/K.$$

If the analytical sensitivities of the samples to be averaged differ considerably, it may be appropriate to compute a weighted average with weights set equal to the inverses of the analytic sensitivities. This is equivalent to computing the average by dividing the total number of structures found in all of the samples by the total volume of air passed through all of the sample filters.

Frequently, it will also be necessary to compute an average for a collection of non-homogeneous samples. For example, one might wish to calculate the average exposure of an individual who frequents several areas with varying estimated airborne concentrations of asbestos. It is recommended that such an average be obtained as follows. First, calculate an average concentration from each homogeneous area by averaging the set of samples collected in each such area as indicated above. Averages derived for the homogeneous areas should then be combined in a weighted average, with weights equal to the proportions of time the individual spends in each of the various areas. Thus, if $\overline{C}_1$, $\overline{C}_2$,...$\overline{C}_K$ are the average concentrations computed from K groups of homogeneous samples using the previous expression, then the overall average exposure concentration experienced by the individual under consideration, based on the respective weights $W_1$,...$W_K$, would be

$$C_W = (W_1\overline{C}_1 + \ldots + W_K\overline{C}_K)/(W_1 + \ldots + W_K).$$

If desired, one can test whether groups of samples are homogeneous using the Wilcoxon and Kruskal-Wallis tests (see Section 7.4).

## 7.2. COMPUTING CONFIDENCE LIMITS FOR AVERAGES OF HOMOGENEOUS SAMPLES

If there are no more than 30 structures in a combined group of K samples then the confidence limits for the average concentration is computed based on the Poisson distribution. Let x be the total number of structures on the combined filters (the number of structures in a single sample is the estimated concentration divided by the analytical sensitivity). Let $x_U$ be the 95% upper confidence limit on the total count x, calculated from the Poisson distribution as described in detail in Section E.4.2 (Appendix E of The Method, Part 1 of this report). The upper 95% limit on the average concentration, $C_U$, is then $x_u$ divided by the sum of the reciprocals of the analytical sensitivities, i.e.,

$$C_U = x_U/(1/S_1 + \ldots + 1/S_K).$$

The lower limit is determined from the same formula except that a suitably defined lower limit $x_L$ replaces $x_U$.

If more than 30 structures are found in a combined group of K samples then confidence limits are also calculated by the following procedure and the most extreme values are reported. Let $C_i$ be the calculated concentration for the ith sample, let $\bar{C}$ be the arithmetic average, and let $s_c^2$ be the corresponding sample variance,

$$s_c^2 = \frac{1}{k-1} \sum_{i=1}^{k} (C_i - \bar{C})^2.$$

Then the 95% confidence limits on the average concentration are computed as

$$C_U = \bar{C} + 1.65\ s_c$$

and

$C_L$ equal to the larger of $\bar{C} - 1.65\ s_c$ or zero.

## 7.3. COMPUTING CONFIDENCE LIMITS FOR AVERAGES OF NON-HOMOGENEOUS SAMPLES

Suppose $\bar{C}_1, \ldots \bar{C}_K$ are average concentrations computed from K groups of homogenous samples, $s_{c1}^2, \ldots s_{cK}^2$ are the corresponding variance terms, to compute confidence limits for the weighted average concentration estimated by

$$C_W = (W_1\bar{C}_1 + \ldots + W_K\bar{C}_K)/(W_1 + \ldots + W_K).$$

Define

$$s_W^2 = (W_1^2 s_{c1}^2 + \ldots + W_K^2 s_{cK}^2)/(W_1 + \ldots + W_K)^2.$$

The 95% upper and lower limits, respectively, are then

$$C_{Wu} = C_W + 1.65\ s_W$$

and $C_{Wl}$ equal to the larger of

$$C_W - 1.65\ s_W \text{ or zero.}$$

## 7.4. TESTING FOR DIFFERENCES BETWEEN CONCENTRATIONS DERIVED FROM DIFFERENT SETS OF SAMPLES

It is recommended that the Wilcoxon non-parametric test (Hollander and Wolfe 1973) be used to test whether airborne concentrations measured from one set of samples are significantly different from those measured by another set. Such a test can be used, for example, to determine whether concentrations are higher in one environmental area than another (e.g., near an asbestos disposal site verses a more distant control area). The test is also recommended to distinguish "sampled" asbestos from contamination attributed to analytical background.

The test should be applied directly to the unadjusted means reported for measurements of airborne concentrations. A mean of zero, "0", should be used in place of all measurements reported as "NF" for not found. A detailed description of the application of the Wilcoxon Test is provided in Section E.5 (Appendix E of The Method, Part 1 of this report).

Care should be taken to exclude possible sources of systematic bias when applying the Wilcoxon test or similar tests. Such bias can result, for example, if:

(1) different laboratories analyzed the groups of samples being compared;

(2) the groups of samples were analyzed by the same laboratory but by distinct analysts;

(3) the samples were analyzed by the same analysts at the same laboratory but before and after a modification to laboratory procedure was adopted;

(4) the samples from the two groups were collected on different types or batches of filters; or

(5) the groups of samples were collected or prepared using radically different procedures.

Such sources of bias can be minimized by keeping experimental conditions as similar as possible when collecting groups of samples intended for comparison, randomizing filters among laboratories or among analysts whenever multiple laboratories or analysts within laboratories are used to analyze filters, and withholding from analysts the identity of the source locations of the filters.

To test for non-homogeneities among airborne concentrations from several sites the non-parametric Kruskal-Wallis test can be used (Hollander and Wolfe 1973). This test is a generalization of the Wilcoxon test that can be applied to more than two groups of data. If analyses involve samples analyzed at separate laboratories, it is strongly suggested that laboratory be used as a blocking factor (Lehmann 1975), so that direct comparisons are made only among samples analyzed at the same laboratory.

### 7.5. ADJUSTING MEASURED CONCENTRATIONS FOR CONTRIBUTIONS FROM ANALYTICAL BACKGROUND CONTAMINATION

When it becomes necessary to determine the absolute magnitude of a measured asbestos concentration (as when measured concentrations are compared to existing risk factors, to establish the magnitude of risk), mean measurements of airborne concentrations must be adjusted to account for contributions from analytical background contamination. As addressed in Section E.5 (Appendix E to The Method, Part 1 of this report), it is strongly recommended that such adjustments be applied to averages of groups of samples rather than individual sample analysis results.

To adjust an average concentration computed for a group of samples weighted by the inverse of their individual analytical sensitivities, proceed as follows. Let x be the total number of structures detected in the used filters, $A_T$ the total area examined from these filters, and BC the surface concentration of structures obtained from an appropriate set of blanks. The adjusted airborne concentration is calculated as

$$\frac{(x/A_T - BC)\, A_T}{1/S_1 + \ldots + 1/S_K} .$$

As indicated in Section E.5, blank analyses may be obtained either from filters analyzed from the same batch used for sample collection or from a cumulative database of historical blank analyses maintained by the laboratory performing the analyses. Depending on the given application, one or the other of these data sets may be considered most appropriate. When adjusting sample analysis results for contributions from analytical background, the source of the blank data must be specified.

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