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MODIFIED ELUTRIATOR METHOD FOR THE DETERMINATION OF ASBESTOS IN SOILS AND BULK MATERIAL

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TABLE OF CONTENTS

TABLE OF CONTENTS (CONT.)

1.0 INTRODUCTION

This elutriator method is a sampling and analysis method for the determination of asbestos in soils and bulk materials. It is specifically designed to provide measurements that can be related to exposure and risk.

Measurements derived using this method can be combined with published dust emission and dispersion models to provide predictions of airborne asbestos concentrations that may result when asbestos-containing bulk materials are disturbed by human activities or natural phenomena. The resulting predictions are sufficiently accurate to support risk assessment and risk management (see, for example, Berman 2000). Moreover, the distribution of the sizes and shapes of the asbestos structures that are released when bulk materials are disturbed is largely preserved by the procedures described in this method for sample collection, handling, preparation, and analysis. Therefore, concentrations reported using this method preserve the size and shape information that is required to assess the risks associated with predicted exposures (see Berman and Crump 1999a).

The elutriator method documented in this report provides estimates of the concentration of asbestos structures that satisfy the dimensional constraints of a particular exposure index, which is the specific index defined in a companion protocol for conducting asbestos risk assessments (Berman and Crump 1999b). An exposure index is a concisely defined set of structure sizes, shapes, and mineralogy that represent the range of asbestos structures that are biologically active and therefore contribute to risk.

> Note, even if there is a desire to evaluate asbestos-related risks based on theories that rely on exposure indices that differ from the one defined herein, this method can still be used to provide the measurements required to predict exposure and risk. The only change needed would be to modify the counting rules (presented in Section 10.1.1), which define the sizes, shapes, and mineralogy of asbestos structures to be included in the determination of asbestos concentrations.

This method was adapted from the “Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials” (Berman and Kolk 1997) and incorporates several cost-saving modifications and other refinements. In both methods, samples are collected in a manner suitable for providing representative measurements of the concentration of asbestos in the matrix sampled, prepared using a dust generator, and analyzed by transmission electron microscopy (TEM). Modifications incorporated into this method include changes to the design and operation of the required dust generator, changes in the manner in which asbestos filters that are collected in the dust generator are prepared for TEM analysis, changes in the counting

rules defined for conducting the analysis, and changes in the manner in which the resulting concentrations are calculated and reported. Specifications for the required modifications to the dust generator are also provided. Design specifications for the original dust generator are provided in the Superfund Method (Berman and Kolk 1997).

Procedures defined for sample collection and field preparation in this method yield 40 g to 70 g samples of bulk materials that are unmodified other than limited coarse crushing (for consolidated materials) and coarse sieving to separate and remove particles larger than 1 cm (3/8ths in.) in diameter. These samples are typically (but not necessarily) derived by sub-sampling a homogenized composite of multiple, large-volume (approximately 1 kg) samples that are collected from an array designed to provide a representative sample of the bulk matrix of interest. The final 40 g to 70 g sub-samples are then sent to the laboratory for analysis.

> Note: smaller volume (mass) samples can also be analyzed by this method. It is simply recommended that samples smaller than approximately 40 g be diluted with a measured mass of washed, play sand (Section 8.3) prior to analysis.

Samples received by the laboratory are placed in the tumbler of a dust generator, which is designed to entrain the fine fraction of the sample in an air stream. The air stream is then passed through the vertical elutriator of the dust generator, which concentrates the asbestos by removing everything except the respirable fraction of fines from the sample. The resulting respirable fraction (which includes the respirable asbestos structures) is then deposited on a filter. The filter is weighed to determine the mass of respirable dust that is deposited on the filter and grid specimens are prepared for TEM analysis from selected portions of the filter using a direct transfer technique. An optional, indirect transfer technique is also described. Results are then reported as the ratio of the number of asbestos structures (within the size range of interest) per unit mass of respirable dust. This is precisely the manner in which asbestos concentrations need to be reported when they are used as inputs to published dust models for predicting airborne asbestos concentrations (Berman 2000).

As reported in this document, this method focuses on requirements for the collection, preparation, and analysis of single samples that are obtained from the field. During a site investigation (as previously indicated), samples will typically be collected from multiple locations that are arranged in an array designed to provide measurements suitable for deriving a representative (i.e. unbiased) estimate of the concentration of asbestos over the sampled matrix as a whole. Thus, proper design of a comprehensive sampling strategy, which includes a detailed design for the array of sampling locations, is also critical to the success of an investigation. However, design of a sampling strategy is necessarily site specific and site-specific considerations are beyond the scope of this document. For further guidance on developing appropriate sampling strategies, see Berman and Chesson (unpublished).

The sensitivity and precision of this method are now well defined (see Sections 2.4 and 2.5). Although the validity of the overall approach for predicting exposure has only been demonstrated in a single study (Berman 2000) heretofore, it is evident from that study that the limitations to the accuracy of such predictions lie primarily with the emission and dispersion models that need to be coupled with analytical results from this method. Nevertheless, a limited number of additional validation studies (to test the accuracy of predictions over a broader range of bulk matrices and release scenarios) would serve to better validate this method.

2.0 BACKGROUND

As indicated previously, this method was specifically developed to provide measurements suitable for supporting risk assessment. To support risk assessment, the method satisfies certain requirements including the need to:

- provide concentration measurements for the specific set of asbestos structure sizes and shapes that contribute to adverse biological effects;
- provide bulk concentration measurements reported in units that are appropriate for inputs to emission and transport models, which can be used to predict airborne exposure concentrations associated with release and dispersion scenarios of interest;
- achieve sufficient accuracy to support adequately accurate exposure (and risk) predictions;
- achieve adequate sensitivity to allow measurement of asbestos over the entire range of concentrations that potentially pose unacceptable risk;
- achieve adequate precision over the range of asbestos concentrations of interest;
- incorporate procedures for sample handling, preparation, and analysis that are insensitive to subjective application so that they are readily reproducible within and between laboratories that may offer the method commercially; and
- readily accommodate a broad range of (natural and anthropogenic) bulk materials for which determining the concentration of asbestos may be of interest.

An additional consideration addressed during the development of this method is the need to control sampling and analysis costs, which is also addressed below.

2.1 BIOLOGICALLY RELEVANT ASBESTOS STRUCTURES

Asbestos poses a threat to human health when dust is released and the resulting structures are inhaled. Whether asbestos also poses a threat to human health when it is ingested is less clear. There is no direct human evidence that ingested asbestos causes disease and the existing animal studies are equivocal (U.S. EPA 1986). Therefore, the focus of this method is to provide bulk measurements that can ultimately be used to predict airborne (inhalation) exposures.

There is strong evidence and general agreement within the scientific community that the risk of asbestos-related disease attributable to a particular exposure is a function of dose (measured in terms of the number of inhaled structures). Further, in addition to structure number concentration, both the mineralogy and sizes and shapes of the inhaled structures determine the magnitude of the risk (see, for example, Berman and Crump 1999a). Therefore, bulk measurements that are collected to support asbestos risk assessment must enumerate and distinguish asbestos structures within relevant size categories and identify their mineralogy.

In the recently completed protocol for assessing asbestos risks (Berman and Crump 1999b), a specific exposure index was defined that is expected to capture the range of asbestos structure sizes and shapes that contribute to risk. This exposure index was developed based on an extensive review of the literature and results from supplemental studies. The exposure index is concisely defined as the weighted sum of the concentrations of asbestos structures in two, related size categories:

$$C_{opt} = 0.003C_S + 0.997C_L \qquad (2.1)$$

where:

"C_{opt}" is the concentration of asbestos expressed in terms of the recommended exposure index. Such concentrations are expected to closely relate to risk;

"C_S" is the concentration of asbestos structures between 5 and 10 µm in length that are also thinner than 0.5 µm; and

"C_L" is the concentration of asbestos structures longer than 10 µm that are also thinner than 0.5 µm.

To provide measurements that can be used to derive concentration estimates reported in terms of the above-described exposure index, it is necessary to separately enumerate asbestos structures that are longer than 5 µm and thinner than 0.5 µm, to distinguish those also longer than 10 µm, and to identify the mineralogy of all such structures. Structures that satisfy the dimensional constraints defined above have been termed, "protocol structures." The rules for counting and characterizing asbestos structures during analysis that are incorporated into this method (Section 10.1) are designed specifically for enumerating protocol structures.

> Note, it has also been determined, based on the literature review and supplemental studies (Berman and Crump 1999a), that samples prepared for TEM analysis by a direct transfer technique relate best to risk. Therefore, a direct transfer technique has been incorporated into this method (Section 9.1). However, an optional, indirect transfer technique is also presented (Section 9.2).

This is because analytical results derived from samples prepared by indirect transfer may sometimes correlate with results from paired samples prepared by direct transfer (within specific matrices at specific sites). Due to the ability to optimize loading, such analyses may occasionally prove less expensive than analyses of samples prepared by direct transfer.

2.2 BULK MEASUREMENTS FOR EMISSION AND DISPERSION MODELING

As indicated previously, bulk measurements derived using this method are intended to be combined with published dust emission and dispersion models to predict airborne asbestos concentrations that may result when asbestos-containing bulk materials are disturbed by human activities or natural phenomena. Based on a dimensional analysis of such models (Berman 2000), a basic requirement for bulk measurements used for this purpose is that results be reported in units of the number of asbestos structures per mass of respirable dust (S/g_{PM10})[1]. Therefore, this method incorporates procedures for simultaneous determination of the number of asbestos structures and the respirable particulate mass that are liberated from a sample during dust generation (Section 8.5). and results are reported as the required ratio: S/g_{PM10}.

Experience gained from use of this method indicates that reporting asbestos concentrations as the ratio of asbestos to dust (which is required in this method) offers an additional, substantial advantage over other, more traditional methods for measuring asbestos in bulk materials. This relates to the ability to account for the efficiency with which sample preparation liberates the asbestos from a bulk sample so that it is available (visible) for analysis.

When samples are analyzed chemically, typically, they are first dissolved. Dissolution degrades the sample quantitatively to its component molecules. Thus, the total number of molecules of interest that were present in the original sample are available for detection during analysis. In contrast, bulk asbestos samples must typically be degraded mechanically to liberate asbestos for analysis. Commonly, such mechanical degradation is performed to achieve some pre-defined, but arbitrary size specification. However, the corresponding efficiency with which the asbestos structures in the sample have been liberated so that they are available (visible) is never known. Moreover, mechanical degradation can never be sufficiently comprehensive to assure liberation of all the asbestos structures in a sample, because such aggressive action would unavoidably alter and degrade the asbestos structures themselves. It is thus

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[1] "PM_{10}" is an abbreviation that is commonly used to represent respirable dust, which is defined as particulate matter exhibiting an aerodynamic equivalent diameter less than 10 µm (Raabe 1984). The aerodynamic equivalent diameter of a particle is the diameter of a hypothetical spherical particle of unit density that exhibits the same settling velocity in air as the particle of interest.

impossible to quantitatively liberate asbestos structures from a bulk sample by mechanical means.

When bulk samples are (naturally or mechanically) degraded and liberate asbestos, fines (including respirable dust) are unavoidably produced in the process. Further, the quantity of respirable dust that is produced is a direct function of the degree of such degradation. Thus, measuring the quantity of respirable dust present in a sample provides at least a rough measure of the efficiency with which asbestos may have been liberated from the sample so that it is available for detection during analysis. Therefore, dividing counts of the number of asbestos structures liberated from a sample by the mass of respirable dust that is simultaneously liberated normalizes the reported asbestos concentrations to account for the degree of degradation of the sample achieved during preparation and analysis.

By reporting asbestos concentrations as the ratio of asbestos to dust, this method (and its predecessor the Superfund Method) provides a measure of asbestos that is an inherent property of the material analyzed, much as a chemical concentration is an inherent property of the material analyzed. Such a measure of asbestos concentration is robust (i.e. relatively insensitive) to the manner in which a sample is handled and prepared. This facilitates reproducibility when the method is applied because subjective effects attributable to differences in the way that individuals or laboratories handle or prepare samples should not affect the results of analyses performed using this method. The same cannot be said of other, more traditional methods commonly used for the determination of asbestos in bulk materials.

That reporting measurements as the ratio of asbestos structure number to respirable dust mass normalizes reported concentrations so that they are robust to subjective handling and preparation (as well as natural in-place weathering) is indicated by comparing results from a recent (unpublished) study in which measurements were collected from ground and unground soil samples.

In the recent study, paired splits were prepared from three soil samples known to contain tremolite asbestos. One split of each pair was prepared as recommended in this method (i.e. by sieving to remove particles larger than 1 cm). The split was then weighed and placed directly in the tumbler of the dust generator for conditioning, dust generation, and analysis (Sections 8.5 and 10.1). The second split of each pair was ground so that the entire sample passed through a 200-mesh sieve prior to weighing, conditioning in the tumbler, dust generation, and analysis.

Asbestos concentrations were measured in the above-described sample splits using this method and results were reported for each of two structure size categories: (1) protocol structures and (2) phase contrast microscopy equivalent (PCME) structures. PCME structures are those that are longer than 5 µm, thicker than 0.25 µm,

and exhibit an aspect (length to width) ratio greater than 3:1. Both sets of results are presented in the following table.

Table 2-1:
Asbestos Concentrations Measured in Paired Sample Splits that Were Ground and Unground, Respectively

	Asbestos Protocol Structure Concentrations (S/g_{PM10})	
Sample Number	**from Unground Split**	**from Ground Split**
Sample Number 1	2.64×10^7	1.27×10^7
Sample Number 2	2.17×10^7	1.33×10^7
Sample Number 3	1.38×10^7	1.10×10^7

	Asbestos PCME Structure Concentrations (S/g_{PM10})	
Sample Number	**from Unground Split**	**from Ground Split**
Sample Number 1	6.40×10^7	1.08×10^8
Sample Number 2	1.20×10^8	1.28×10^8
Sample Number 3	1.39×10^8	5.95×10^7

The concentrations reported in Table 2-1 each represent the mean of duplicate (or triplicate) analyses. Each analysis is derived from one of a set of filters collected at differing time intervals over the course of a dust generator run for each sample.

To determine whether grinding affects the analytical results for a sample, an analysis of variance (ANOVA) was conducted. The purpose of the ANOVA was to test whether the observed differences in analytical results between ground and unground pairs was greater than the variation observed between replicate analyses of filters derived from each of the individual (ground or unground) samples. Measured concentrations derived from each replicate analysis of each individual sample are reported in Table 2-2.

Table 2-2:
Asbestos Concentrations Measured in Replicate Preparation of Selected Samples

	Asbestos Protocol Structure Concentrations (S/g_{PM10})		
Sample Number	**Concentration in Replicate 1**	**Concentration in Replicate 2**	**Concentration in Replicate 3**
Sample Number 1 (unground)	2.30×10^7	2.03×10^7	3.60×10^7
Sample Number 1 (ground)	1.94×10^7	5.90×10^6	----
Sample Number 2 (unground)	1.88×10^7	2.46×10^7	----
Sample Number 2 (ground)	2.06×10^7	6.09×10^6	----
Sample Number 3 (unground)	2.17×10^7	5.88×10^6	----
Sample Number 3 (ground)	1.90×10^7	3.90×10^6	----

	Asbestos PCME Structure Concentrations (S/g_{PM10})		
Sample Number	**Concentration in Replicate 1**	**Concentration in Replicate 2**	**Concentration in Replicate 3**
Sample Number 1 (unground)	6.5×10^7	7.3×10^7	5.4×10^7
Sample Number 1 (ground)	1.04×10^8	1.11×10^8	----
Sample Number 2 (unground)	9.17×10^7	1.48×10^8	----
Sample Number 2 (ground)	1.65×10^8	9.13×10^7	----
Sample Number 3 (unground)	1.19×10^8	1.59×10^8	----
Sample Number 3 (ground)	7.7×10^7	4.2×10^7	----

It is clear from a visual comparison of the measurements presented in Tables 2-1 and 2-2 that variation between ground and unground pairs is no greater than the variation observed between sample replicates. These observations are confirmed formally from the results of the ANOVA in which the variation between ground and unground samples

is shown to be no greater than variation among sample replicates[2]. Thus, measured concentrations for ground and unground pairs do not significantly differ.

Whether measured concentrations differ for ground and unground sample splits was also tested using a more rigorous procedure. In this latter procedure, the individual asbestos structures observed during each analysis were evaluated to determine whether the counts observed among the ground and unground split from each sample could be considered to have been derived from a single Poisson distribution (i.e. could both splits be considered to have come from Poisson distributions exhibiting the same mean, after adjusting for differences in the mass of material deposited on each filter).

This latter evaluation relies on the assumption that asbestos structures are randomly distributed across each sample filter. When this assumption is true, the number of structures observed while scanning a filter is described by a Poisson distribution with a mean equal to the total number of structures on the filter multiplied by the fraction of the surface area of the filter scanned during analysis. Thus, if the only source of variation that contributes to a measurement is the chance of encountering asbestos structures while scanning a fixed area of a filter, the variation observed among repeated measurements over such a filter would be described by a Poisson distribution. This is the type of variation that is observed, for example, among a set of measurements derived by repeatedly scanning randomly selected areas of uniform size on a single filter. Further discussion of Poisson distributions and counting statistics is provided in Section 2.5.

Because the replicate measurements presented in Table 2-2 are derived from multiple filters, additional sources of variation can potentially be introduced by differences in the manner in which asbestos was deposited on each filter. Moreover, ground and unground splits are two physically separate samples that have been separately handled prior to analysis, which suggests further opportunities for introducing variation between such samples. Nevertheless, due to the design of this method, it is expected that the variation between replicate filters (and even sample splits) will be dominated by variation due to counting statistics, which are described by a Poisson distribution (as discussed above).

Results from the more rigorous test to determine whether paired ground and unground splits could be considered to be derived from the same Poisson distribution are somewhat mixed. Among measurements of protocol structures, each pair of ground and unground splits from the three samples can be adequately described by a single Poisson. Among measurements of PCME structures, the ground and unground pair

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[2] The F-test statistics (which are the ratios of the variance between ground and unground samples relative to the variance within ground and unground samples, respectively) from the ANOVA are: F = 2.55, P = 0.15 (not significant) for protocol structures and F = 0.14, P = 0.72 (also not significant) for PCME structures.

representing Sample No. 2 can be adequately described by a single Poisson. However, the pairs representing Sample Nos. 1 and 3, respectively, cannot be adequately described by a single Poisson.

The reason for deviation from Poisson counting statistics among the splits for Sample Nos. 1 and 3 (based on counts of PCME structures) is unclear. It may be due to additional contributions to variation (from the kinds of sources described above) for these specific samples. This would make the test for compliance with Poisson counting statistics too severe for the purpose intended here. Overall, however, the results of the statistical tests described above indicate that grinding a sample during preparation has little or no effect on the outcome of a measurement derived using this method.

Given the above, it is recommended for this method that mechanical modification of bulk materials of interest be limited to the minimum that is required to generate 40 g to 70 g samples containing individual particles no larger than approximately 1 cm (3/8ths in.) in diameter. Thus, unconsolidated material need only be sieved (to separate out particles larger than 1 cm), homogenized, and split (Chapter 7). Rocks or other bulk solids may additionally need to be coarse crushed so that the majority of the fragments can pass through a 1 cm sieve.

It is expected that, as long as mechanical crushing, grinding, or other disaggregation is not so severe as to cause degradation of the embedded asbestos structures themselves and as long as any such mechanical actions are performed in a manner allowing capture and preservation of the resulting fines (as part of the sample), then preparation employing such activities should not substantially affect analytical results derived using this method. However, additional studies of such effects over a broader range of sample types and conditions than those reported above would certainly improve our understanding of the capabilities of this method.

2.3 ACCURACY

Because there are no bulk standards available for asbestos, it is not currently possible to determine the accuracy of this method by direct means. However, an indirect procedure exists that can be used to provide a bounding estimate of the accuracy of measurements derived using this method.

Controlled experiments in which bulk materials in the field are subjected to mechanical disturbance and the airborne asbestos concentrations that result from such disturbance are measured, provide an independent set of measured airborne concentrations that can be compared with concentrations predicted by combining bulk measurements using this method with appropriately matched emission and dispersion models. The degree of agreement between measured and predicted airborne concentrations then provides

a measure of the accuracy of such predictions. In turn, the accuracy of such predictions represent bounding estimates for the accuracy of this method.

Such a study has been reported in a recent publication (Berman 2000). Results from this study suggest that airborne asbestos concentrations predicted based on single measurements of bulk concentration (derived from a composite of samples collected from an appropriately designed sampling array) are likely to be accurate to within a factor of three or four. Moreover, the accuracy of predictions for long-term average exposure concentrations (which is what is required to support risk assessment) should be even better. The precision of predictions derived from method measurements can also be improved by basing such predictions on the mean of multiple bulk measurements, rather than single measurements (see Section 2.5).

Importantly, although the results discussed above are suggestive, they are limited. The Berman (2000) study is a single study of two serpentine surfaced roads. The range of conditions examined in this study was limited. Among published dust models that might be coupled with measurements using this method to predict airborne asbestos concentrations, the dust model employed for unpaved roads (U.S. EPA 1985) in the Berman study is among the most highly developed, best tested, and most precise. Moreover, the study was conducted under conditions that were particularly favorable to control of meteorological effects and other potentially confounding variables. Thus, the accuracy of predictions derived for other exposure scenarios may not be as good. Nevertheless, it is apparent from the Berman (2000) study that the accuracy of exposure predictions that are derived in the manner described above will be limited primarily by the accuracy of the dust model used for deriving the predictions, rather than from the input measurements derived using this method.

Additional studies similar to Berman (2000) are recommended in which a broader range of release scenarios are evaluated under a broader range of meteorological conditions. Such studies would allow better characterization of the performance of this method and the predictions derived by modeling that employ measurements using this elutriator method.

2.4 SENSITIVITY

A range of target sensitivities for this method were estimated from calculations presented in the feasibility study (Berman 1990) for the original Superfund Method (Berman and Kolk 1997). However, such targets were based on assumed preparation by an indirect transfer procedure, which is no longer recommended. The original sensitivity calculations were also based on procedures for evaluating asbestos-related risks that are not consistent with the new protocol (Berman and Crump 1999b).

Therefore, a new set of target sensitivities has been estimated based on recent calculations (Berman unpublished). In these calculations, acceptable airborne concentrations for protocol structures were separately estimated for chrysotile and the amphiboles based on a target risk level of 10^{-5} (one in one hundred thousand) and the procedures recommended for relating exposure and risk that are presented in the new protocol (Berman and Crump 1999b).[3]

Based on the unpublished calculations, a target sensitivity of 3×10^6 S/g_{PM10} is likely to adequately bound the range of concentrations of potential concern for the vast majority of emission and dispersion scenarios of interest for risk management. However, to assure adequate sensitivity for specific projects, it is highly recommended that preliminary calculations be performed to identify a level of sensitivity that will adequately support risk management decisions that are project specific. Therefore, the target sensitivity presented here is intended to be illustrative only.

> Note that the target sensitivity provided in the last paragraph is based on calculations assuming amphibole asbestos protocol structures. Because chrysotile appears to be substantially less potent than the amphiboles, a target sensitivity on the order of 5×10^7 S/g_{PM10} may generally prove sufficient for chrysotile asbestos. Again, however, sensitivity for this method should be set based on project-specific requirements and the stopping rules for analysis (Section 10.1.2) should then be adjusted accordingly.

As indicated in Section 10.1.2, achieving the lower (more severe) of the two sensitivities listed above should ultimately require scanning a maximum of approximately 150 grid openings (distributed over 5 grid specimens), when filter preparation during dust generation is optimized. Because scanning need only be performed at a maximum magnification of 10,000x, the cost for performing such an analysis is expected to remain reasonable (see Section 2.8).

2.5 PRECISION

The original Superfund Method was shown to achieve a level of precision that is adequate for distinguishing acceptable asbestos concentrations from potentially hazardous concentrations with a resolution that is considered acceptable for supporting risk management decisions (Berman et al. 1994). During the pilot study, the counting of 50 structures (during the analysis of paired splits from each of several homogenized,

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[3] There are six different mineral types included in the definition of asbestos that are grouped into two mineralogical categories: serpentine asbestos (chrysotile) and the amphiboles, which include the other five types of asbestos. Asbestos structures in each of the two mineralogical categories have been found to exhibit differing potency (Berman and Crump 1999a). That is why the new risk assessment protocol incorporates differing risk factors for chrysotile and the amphiboles (Berman and Crump 1999b).

bulk samples) produced an average relative percent difference (RPD) across duplicate sample pairs of 47%. Of 40 paired counts (five separate counts of each of a pair of filters from each of four sets of paired samples by each of two laboratories), 14 (35%) exceeded an RPD of 50% and 6 (15%) exceeded an RPD of 100%. However, 8 of the 14 RPD's exceeding 50% derive from a single set of paired splits so it is conceivable that special problems developed during the preparation of this one pair of splits. For further discussion of this issue, see Berman (2000).

Importantly, the results presented above were derived based on analysis of samples prepared using an indirect transfer technique. Because this method relies primarily on the analysis of samples prepared by direct transfer, additional tests were recently performed to determine the achievable precision for the analysis of samples prepared for TEM analysis by direct transfer (Berman unpublished).

In a recent series of studies, replicate filters were collected during dust generator runs from each of a series of 10 samples. The filters were then prepared for TEM analysis using a direct transfer technique and each of two types of asbestos structures (protocol structures and PCME structures) were counted. Measurements were then evaluated to determine whether counts derived from replicate filter pairs could be adequately described by a single Poisson distribution (after adjusting for differences in the mass deposited on each replicate).

The results of this evaluation indicate that, for protocol structures, counts derived from replicate filters cannot be distinguished from one another (i.e. they can be adequately described by a single Poisson distribution) for 8 of the 10 pairs tested. Although the replicates from the remaining two samples could be distinguished at the 95% level of significance, they were indistinguishable at the 99% level of significance (P = 0.039 and 0.045 for the two pairs, respectively). Thus, the dominant source of variation apparent among these replicates is that attributable to counting (see Section 2.2), which is the minimum variation achievable for asbestos analyses that require the enumeration of individual asbestos structures.

Results for the 9 available pairs of replicates analyzed for PCME structures showed somewhat greater variability, but generally support the same conclusion. Of the PCME structure counts derived from replicate filters 6 of 9 replicate pairs cannot be distinguished from one another (i.e. each pair of replicates can be adequately described by a single Poisson distribution). Two of the remaining three pairs of replicates can be distinguished at the 95% level of significance but not at the 99% level of significance (P = 0.042 and 0.016, for the two pairs, respectively). The two filters of the last replicate pair exhibit structure concentrations that are clearly distinguishable from one another (they cannot reasonably be described by a single Poisson, P = 0.008). It is not known why counts of PCME structures appear to be somewhat more variable than counts of protocol structures. Overall, however, it is reasonable to

conclude from these data that the variability observed between replicate measurements derived from this method is dominated by that attributable to counting. This means that the precision of the method approaches the optimum achievable for a method that involves the enumeration of individual asbestos structures.

> Note: the above described test of precision (for samples prepared by direct transfer) is based on the analysis of replicate filters rather than paired splits of homogenized samples (which would address additional sources of variation potentially introduced by the handling of bulk samples prior to dust generation). It is expected, however, that the achievable precision for paired sample splits will be comparable to that demonstrated for replicate filters (i.e. variation will be dominated by the limitations of counting statistics and will therefore approach the minimum possible). This is indicated by the results of the original pilot study (Berman et al. 1994), which demonstrate that variation introduced by bulk sample handling in this method is trivial compared to the variation associated with filter generation and the variation introduced by counting statistics during analysis. Both of the latter sources of variation are adequately addressed by the comparison of results from replicate filters.

2.6 OBJECTIVE PROCEDURES FOR SAMPLE HANDLING AND ANALYSIS

This method is designed to be adopted and applied broadly. Therefore, with one exception, equipment and supplies required to implement this method are commercially available and in common use among laboratories offering analyses for the determination of asbestos by TEM.

The one exception involves the need to construct a dust generator to conduct the required sample preparation. However, even the dust generator is designed to be constructed from commercially available materials and precise specifications for its construction have been published in the Superfund Method (Berman and Kolk 1997). The specifications for the modifications required to adapt the dust generator for use with this method are incorporated within this document (Section 6.3). The cost for constructing the required dust generator has proven to be modest and should not be a hindrance to any laboratory wishing to offer commercial analyses using this method.

So that laboratories offering analysis using this method will generate comparable results, the procedures incorporated into the method were designed and selected to facilitate objective implementation and are robust (i.e. relatively insensitive) to subjective application that might otherwise introduce variability within and between laboratories. The data and evaluation presented in Section 2.2 provides a good indication of the degree to which the procedures incorporated into this method for

sample preparation are robust to subjective application. Procedures employed for analysis are also intended to limit the effects of subjective application.

While it is unavoidable that the counting rules employed to determine the manner in which asbestos structures are to be enumerated during analysis are subject to analyst interpretation and this introduces some variation, it is expected that such variation will be somewhat more limited for this method due to the constraints in the range of structure sizes that are included for analysis. Structures counted during analysis by this method are limited to those defined as protocol structures (Section 2.1). At the same time, subjective variation introduced by the interpretation of counting rules is a limitation that is common to all TEM methods used for the determination of asbestos structure concentrations.

The need to produce filters during dust generation with dust deposits that are demonstrably uniform must also be considered when addressing the reproducibility of repeated analyses and the comparability of analyses within and between laboratories.

To test this ability initially, five filters were selected for evaluation from among filters generated during a series of recent studies. Multiple grid specimens (up to nine for some filters) were then prepared from locations on the filter selected in systematic arrays designed to representatively cover the entire surface of each filter. Asbestos structures were then enumerated during scans of multiple grid openings (up to 50) on each of the grid specimens that were prepared from each filter. The resulting structure counts were then evaluated to determine whether the counts across grid openings within each grid specimen and, separately, whether the total counts across grid specimens from each filter could be adequately described by a single Poisson distribution.

Results from this evaluation indicate that counts on grid openings within every grid specimen tested could always be adequately described by a single Poisson distribution. However, only three of the five filters yielded a set of grid specimens that could be adequately described by a single Poisson. This was not a problem as long as samples were being prepared by an indirect transfer procedure. However, direct transfer is now preferred, due to research indicating superior correlation with risk (Berman and Crump 1999a).

After further study, it was found that lack of uniformity on the filters prepared for asbestos analysis using the dust generator was primarily due to the lack of complete mixing between the air stream exiting the tumbler of the dust generator and the air stream entering from the open tube at the bottom of the elutriator (in the original configuration of the dust generator). In hindsight, the laminar flow regime of the elutriator is not conducive to efficient mixing of such air streams.

Based on these results, the dust generator was reconfigured by eliminating the open tube at the bottom of the elutriator and redesigning to assure adequate flow velocities at the bottom of the elutriator[4] to assure that respirable particles are transferred quantitatively from the tumbler to the elutriator (see Section 6.3). Based on visual inspection of filters that are intentionally overloaded so that the deposit on the filter is readily visible, it appears that the reconfigured dust generator produces adequately uniform deposits on filters so that there should be no problem demonstrating that structures observed on such filters are Poisson distributed. Further analysis of asbestos structure counts are in progress.

2.7 THE CHARACTERISTICS OF BULK MATERIALS REQUIRING ANALYSIS

The asbestos content of a broad range of bulk materials (both natural and anthropogenic) are expected to provoke interest. These may include, for example:

- rock or soils containing naturally occurring asbestos;
- soils, fills, or sediments containing asbestos-containing debris;
- asbestos-containing construction materials; and
- asbestos-containing, settled dust.

This method is designed to be applied broadly and the procedures provided herein should allow the method to be adapted for application to any of the kinds of materials listed above. For descriptions of adaptations that may be required for some of the materials listed, see Sections 2.2, 8.2, and 8.3.

To date, the original Superfund Method and this modified elutriator method have been applied to a broad range of asbestos containing materials of interest including:

- crushed serpentine rock variously containing naturally occurring chrysotile and tremolite asbestos;
- soils variously containing naturally occurring chrysotile and tremolite asbestos; and

[4] Air stream velocities at the bottom of the elutriator must be higher than velocities in the main body of the elutriator at all points in the air stream so that respirable particles are not lost prior to sorting in the elutriator. This assures quantitative transfer of respirable material from the tumbler to the filters where dust and asbestos are collected for analysis.

- soils and fills variously containing chrysotile, amosite, anthophyllite asbestos, tremolite asbestos, and actinolite asbestos in construction debris.

The concentrations of asbestos that have been observed in samples analyzed using this method to date (excluding non-detects) range between 1 x 10^6 S/g_{PM10} and 8 x 10^8 S/g_{PM10} for protocol structures and as high as 4 x 10^9 S/g_{PM10} for total structures.

2.8 COST CONSIDERATIONS

Per-sample costs for analysis using the original Superfund Method tend to run between $800 and $1,500, depending on the target sensitivity and precision requested for a particular analyses. Given the modifications adapted for this method, a target sensitivity of 3 x 10^6 S/g_{PM10}, and counts that are restricted to protocol structures, per-sample costs for analysis using this method have been running between $500 and $800 (approximately half of per-sample costs for the original method). As experience with this method increases and as competition increases with additional laboratories offering this method commercially, it is also expected that per-sample costs for this method will decrease further.

Importantly, per-sample costs for analysis using this method are not expected to represent a driving factor for the cost of site characterization employing this method. The procedures incorporated into this method for sample collection and field preparation are amenable to substantial compositing so that, when an investigation is properly designed, only a very limited number of analyses using this method should be required to provide adequate characterization of the bulk materials of interest. In fact, the process of combining results from this method with appropriately selected dust emission and dispersion models to predict airborne exposure concentrations is expected to be less expensive than characterizing airborne exposures by direct measurement (with a comparable degree of accuracy) for all but the simplest cases of potential interest.

3.0 OVERVIEW OF METHOD

Samples are collected in the field according to a pre-defined sampling plan identifying the number of samples to be collected and the locations from which samples are to be collected. Procedures for designing such a plan are beyond the scope of this document but are reported elsewhere (see, for example, Berman and Chesson, unpublished).

Any of a variety of commercially available sampling equipment (i.e. trowels, shovels, augers, corers, etc.) may be used to collect samples for this method. However, they must have been specified in the pre-defined sampling plan based on the nature of the material being sampled and the depths over which samples are to be collected. Whatever sampling technique is employed, the *minimum* size sample to be collected at each location shall be 1 kg.

Once collected, each sample is brought to a central location for field preparation. Field preparation steps are listed in Figure 3-1 and discussed further in Chapter 7 and in greater detail in Chapter 8 of Berman and Kolk (1997). Each sample is first weighed. Then the sample is sieved using a screen with 3/8th in. (1 cm) openings to separate a coarse and fine fraction. The material placed on the sieve is worked with gloved hands to assure that all friable components pass through the screen.

The coarse fraction, composed of material that is retained by the screen, is transferred to a bucket and weighed prior to discarding on site. The fine fraction is also weighed. As indicated in Figure 3-1, the fine fraction is then homogenized. The procedure recommended in this method for homogenization is repetitive splitting using a riffle splitter with the split halves of the sample being re-combined at the end of each split. Studies indicate that five to seven iterations are typically sufficient to achieve adequate homogenization.

Once homogenized, the fine fraction is then sub-sampled using the riffle splitter (Figure 3-1). During sub-sampling, the one-half of the sample from one of the two receiving trays is discarded after each split and the second half of the sample is then re-split. The process is repeated until sub-samples weighing between 40 g and 70 g are produced in each of the two receiving trays. The material in each tray is then transferred quantitatively to a sample bottle, packaged and shipped to the laboratory.

Sample handling, preparation, and analysis in the laboratory is depicted in Figure 3-2 and described in detail in Chapter 8 (of this document). Once sub-samples weighing between 40 and 70 g are obtained, they can be separately prepared and analyzed (Section 8.5).

To prepare samples, as indicated in Figure 3-2, first load the sample into the tumbler of a dust generator. The design, construction, and operation of a dust generator suitable for use with this method is provided in Appendix A of Berman and Kolk (1997) with required modifications provided in Section 6.3 of this document. The sample is then conditioned by flowing humidity-controlled air through the tumbler and over the sample for several hours.

Once the sample is conditioned, the tumbler of the dust generator is started and a sample run is initiated. During each run, a series of filters is collected continuously from the top of ME openings of the dust generator and these are weighed and plotted to determine when dust generation has stabilized sufficiently to collect filters for asbestos analysis.

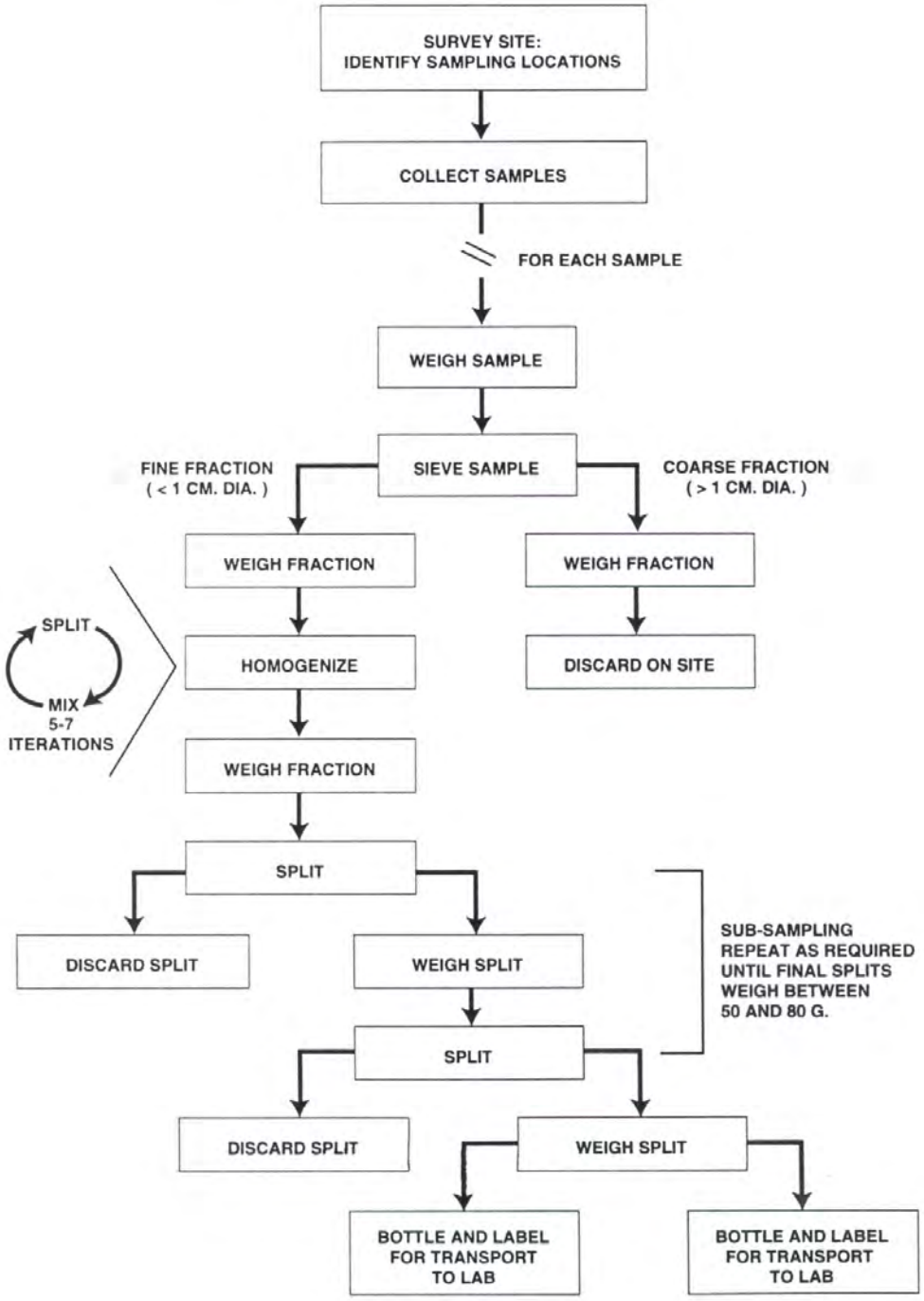
While the dust generator is operating, a second set of filters is also collected over the IST opening of the dust generator, which articulates with an isokinetic sampling tube. These are collected such that loading is appropriate for specimen grid preparation using a direct transfer technique.

Next, as indicated in Figure 3-2, TEM specimen grids are prepared using a direct transfer technique from the filters collected from atop the isokinetic sampling tube of the elutriator. Before specimen grid preparation, however, the filters must be weighed to determine the mass of dust deposited on the filter. Specimen grids are then analyzed using the counting and identification rules of the International Standards Organization (ISO) Method for the determination of asbestos in air using an indirect transfer technique (ISO 10312) with the counting rules modified for determining only protocol structures (defined in Section 10.1.1) and with the stopping rules modified as indicated in Section 10.1.2.

Dust mass estimates are then combined with asbestos counts to allow reporting of the concentration of asbestos protocol structures per unit mass of respirable dust in the sample (Figure 3-2).

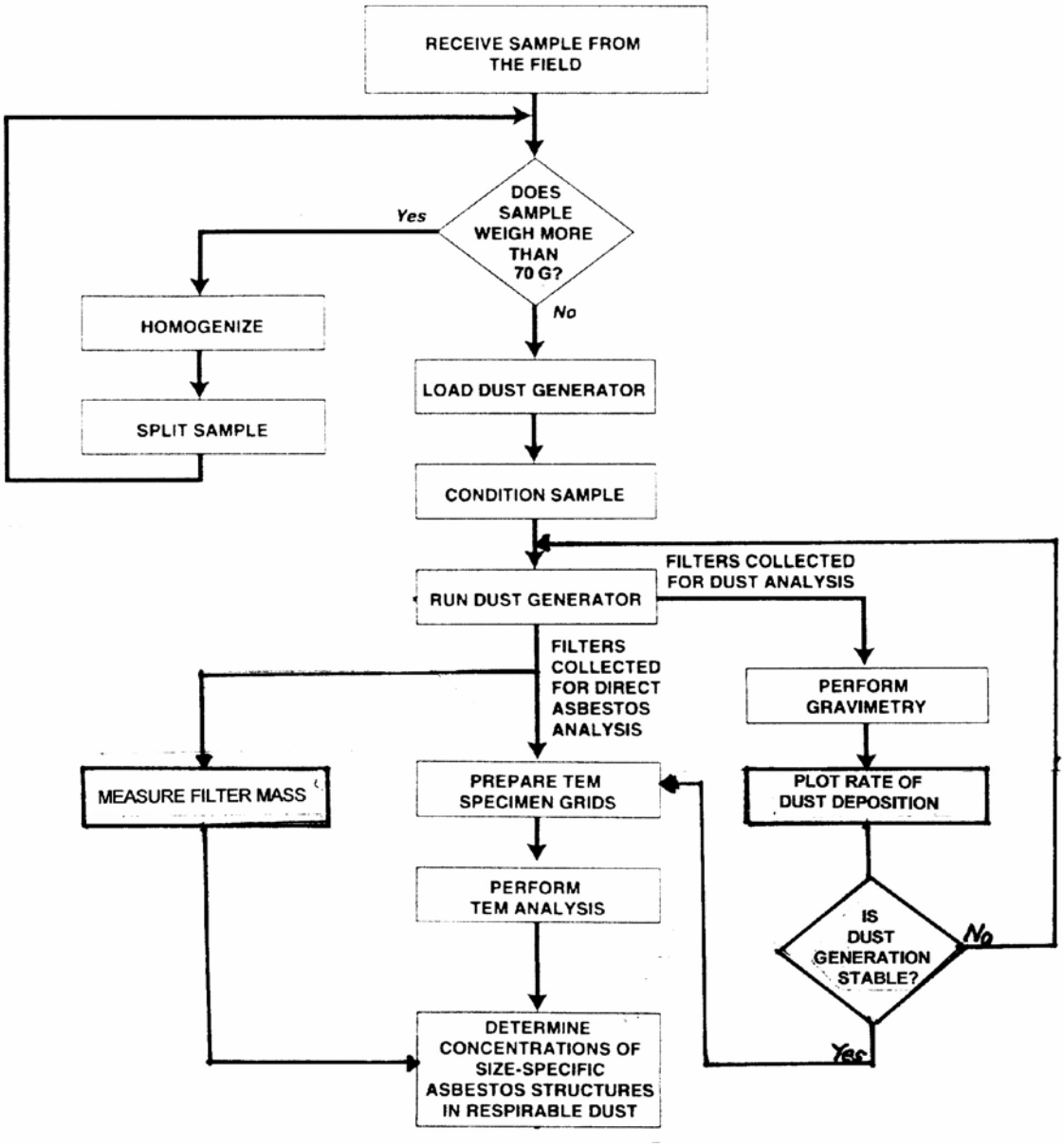
FIGURE 3-1

SAMPLE COLLECTION AND FIELD PREPARATION



J94-102.3 Mac 4

LABORATORY PREPARATION AND ANALYSIS



4.0 DEFINITIONS

Amphibole: a group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula:

$$A_{0-1}B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where:

A = K, Na;

B = Fe^{2+}, Mn, Mg, Ca, Na;

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+};

T = Si, Al, Cr, Fe^{3+}, Ti.

In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124° (Hodgson 1965).

Amphibole Asbestos: amphibole in an asbestiform habit.

> Note, based on work performed to define the relationship between structure morphology and health effects (Berman and Crump 1999a), it appears to be primarily the overall dimensions of an asbestos structure that determine it's potential to contribute to disease, not whether that particular structure is strictly "asbestiform" in habit (see the definition of asbestiform below). Therefore, prudence dictates that all isolated amphibole asbestos structures that exhibit dimensions consistent with those defined as "protocol structures" should be included in the determination of risk-related concentrations (see the definition of protocol structures).

Analytical Sensitivity: the calculated asbestos concentration in soil or a bulk matrix, in asbestos structures per gram of respirable dust (S/g_{PM10}), equivalent to counting of one asbestos structure in the analysis.

Asbestiform: a specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

> Note, the term, "asbestiform" is primarily a geology term that is intended to distinguish deposits of a particular mineral that exhibit high fibrosity *as a bulk property* in contrast to massive deposits in which the material is variously described as columnar, platy, or granular (Kraus et al. 1959). The term was not originally intended to distinguish the potential for a material to contribute to health effects. Therefore, this distinction should not be applied to isolated asbestos structures when determining risk-related concentrations (see the note under the definition for amphibole asbestos). While it appears there is generally good correlation between an asbestos mineral's habit and the potential to induce adverse health effects (see, for example, OSHA 1992), the correlation may not be perfect (see the definition of protocol structures).

Asbestos: a term applied to a group of fibrous silicate minerals that readily separate into thin, strong fibers that are flexible, heat resistant and chemically inert. The term is generally applied to asbestiform serpentine and the non-aluminous amphibole minerals (Kraus et al. 1959).

> Note, the term, "asbestos" is a geology term that was originally intended to distinguish the bulk properties of deposits with potential commercial value (due to fibrosity) from those that do not exhibit such properties. It was not initially intended to define materials that present a risk for inducing adverse health effects (when inhaled). When assessing health effects, the term asbestos should be considered to include all isolated structures of the proper mineralogy that exhibit a morphology consistent with that defined below as "protocol structures."

Asbestos Component: a term applied to any individually identifiable asbestos sub-structure that is part of a larger asbestos structure.

Asbestos Structure: a term applied to any contiguous grouping of asbestos fibers, with or without equant particles.

Aspect Ratio: the ratio of the length to width of a particle.

Blank: a fiber count made on TEM specimen grids prepared from an unused filter (or a filter through which asbestos-free air or water has been passed), to determine a background measurement. Blanks may include equipment blanks, field blanks, filter or lot blanks, and laboratory blanks, and/or method blanks.

Bundle: a fiber composed of parallel, smaller diameter fibers attached along their lengths (ISO 10312).

Chrysotile: the asbestiform habit of a mineral of the serpentine group that has the nominal composition:

$$Mg_3Si_2O_5(OH)_4$$

In some varieties of chrysotile, the silicon may be partially substituted by Al or less commonly by Fe. The magnesium may be partially substituted by Fe, Ni, Mn or Co. Some varieties contain Na, Cl or both. Chrysotile is a highly fibrous and silky variety and constitutes the most prevalent type of asbestos (Hodgson 1965). Also, see notes under the definitions for asbestiform, asbestos, and protocol structures when considering the health effects potentially attributable to chrysotile.

Cluster: an assembly of randomly oriented fibers (see Chatfield 1993).

Component Count: for any sample, a tally that includes the individually identified components of complex asbestos structures and each single asbestos structure with no identifiable components.

Conditioning Filters: a collected at the beginning of a run while a sample is being conditioned. Under such circumstances, the tumbler is loaded with sample but is not running. However, humidity controlled air is being passed through the tumbler, through the elutriator, and into the filter mounts.

Elutriator: a device in which differential flow through a fluid (gas or liquid) against an opposing force (i.e. gravity) is employed to separate particles by size.

Equant Particle: as used in this document, a non-asbestos particle bound to, or overlapping with, asbestos structures observed on a TEM specimen grid.

Equipment Blank: a filter collected from a mount over one of the openings atop the elutriator while air is passed through an empty tumbler assembly and the elutriator.

Fiber: an elongated particle that has parallel or stepped sides. In this method, fibers that potentially contribute to biological activity are restricted to those exhibiting dimensions consistent with protocol structures (see Berman and Crump 1999b).

Fibril: a single fiber of asbestos that cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearance.

Fibrous Structure: a contiguous grouping of fibers, with or without equant particles.

Field Blank: a bulk material known to be free of asbestos that is packaged in such a manner so as to be indistinguishable from other field samples (for a particular project) and is sent to the laboratory for preparation and analysis using this method.

Filter Blank: an unused filter that is analyzed to determine the background asbestos structure count on the filter matrix.

Friable: as used in this document, capable of being crushed or deformed with the hand with the attendant release of fibers.

Habit: the characteristic crystal form or combination of forms of a mineral, including characteristic irregularities.

Identify: during asbestos analysis, the use of a sequential set of procedures to determine and confirm the mineralogy of a structure.

Isokinetic Sampling: sampling air in such a manner so as not to disturb the direction or velocity of air flow at the point sampled.

Isokinetic Sampling Tube: a tube placed in the air flow of the vertical elutriator portion of the dust generator used in this method, which samples the air at the top of the elutriator isokinetically.

Laboratory Blank: an unused filter that is analyzed along with sample filters to determine the background asbestos structure count in laboratory air.

Lot Blank: see filter blank.

Matrix:[5] A connected assembly of asbestos fibers with particles of another species (non-asbestos) (ISO 10312).

Method Blank: a filter collected in a mount over one of the openings atop the elutriator while washed, play sand is tumbling under conditions appropriate for a routine sample run.

PCM Equivalent Structure: a structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a mean diameter between 0.2 µm and 3.0 µm for a part of its length greater than 5 µm. PCME structures also must contain at least one asbestos component (see Berman and Chatfield 1999a).

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[5] The above definition applies when used to describe an asbestos structure. The term is also used in this document to describe a heterogeneous bulk solid.

Note that the definition of PCME structures is provided here primarily for historical perspective.

Protocol Structure: an isolated structure or a component of a complex structure that is longer than 5 µm and thinner than 0.5 µm. When determining risk-related concentrations, protocol structures longer than 10 µm must be distinguished and separately enumerated because such structures are assigned a greater potency than protocol structures with lengths between 5 µm and 10 µm (see Section 2.1). When counting protocol structures to assess health effects, both isolated parent structures exhibiting the appropriate dimensions and qualifying components of more complex structures should be included in the count.

Note, it has been observed that only a small fraction of the isolated particles produced by the degradation of minerals found in a massive habit qualify as protocol structures. In contrast, minerals found in an asbestiform habit tend to yield large numbers of protocol structures when mechanically or naturally disturbed. Therefore, the relative hazard associated with the asbestiform versus massive habits of a mineral may be related largely to the relative rate of release of (and the attendant levels of exposure to) protocol structures when such materials are disturbed under comparable conditions. Moreover, under conditions typically associated with quarrying and mining of materials containing small quantities of the massive forms of the asbestos-related minerals, the exposures attendant to the disturbance of such materials are generally not considered sufficient to warrant concern other than as nuisance dusts (OSHA 1992). Incidentally, protocol structures are defined in a manner that is consistent with the requirements recommended by Wylie to OSHA for distinguishing among structures that derive from asbestiform and non-asbestiform habits, respectively (OSHA 1992, P. 24329).

Respirable Dust: particulate matter in a size range capable of penetrating the deep lung and being deposited in terminal bronchioles and alveoli (beyond the reach of the ciliary escalator, see Berman and Crump 19991). This is further defined as particulate matter exhibiting an aerodynamic equivalent diameter less than 10 µm (Raabe 1984). The aerodynamic equivalent diameter of a particle is the diameter of a hypothetical spherical particle of unit density that exhibits the same settling velocity in air as the particle of interest. Due its size range, respirable dust is commonly abbreviated as PM_{10}.

Riffle Splitter: a device composed of a hopper and multiple, uniform, parallel chutes that alternately feed from the hopper to opposing receiving trays.

Scrubber: a device for removing particles from an air stream by passing the air stream through a super-saturated vapor in which the particles serve as nucleation centers for

condensation and are thus captured. The resulting droplets (containing the trapped particles) then fall back into a central reservoir of boiling liquid.

> Note that among the modifications to the dust generator from the Superfund Method that is defined in this modified elutriator method is to remove the scrubber from the dust generator assembly and to seal the ports for the scrubber with stoppers (see Section 6.3). Therefore, this definition is provided primarily for historical reference.

Serpentine: a group of common rock-forming minerals having the nominal formula:

$$Mg_3Si_2O_5(OH)_4$$

Serpentine deposits often contain chrysotile asbestos (which is serpentine in an asbestiform habit).

Silt: the fine material found in a bulk matrix. Silt is defined formally as material less than 70 µm in diameter.

Structure Count: for any sample, a tally of each individually identified asbestos structure regardless of whether the structure contains identifiable components. This is equivalent to a count of the total number of separate asbestos entities encountered on the sample.

Vertical Elutriator: see Elutriator.

Tumbler: a device that is rotated to provide continuous agitation to a bulk material placed inside. In the dust generator employed in this method, air is blown through a tumbler containing sample to carry away the dust generated during agitation by the tumbler.

> Note that the tumbler incorporated into the dust generator that is required for sample preparation in this method is designed primarily to facilitate capture of respirable size particles in the air stream that flows through the tumbler. It is not intended to alter the bulk characteristics of the sample.

Washed, Play Sand: a commercially available material composed of well-sorted sand that contains virtually nothing fine enough to be considered respirable.

5.0 SYMBOLS AND ABBREVIATIONS

5.1 SYMBOLS

A_f - the area of a filter from which a specimen grid is prepared (mm^2).

A_{go} - the average area of a specimen grid opening (mm^2).

C_{dust} - the concentration of asbestos structures (of a defined size and type) in the respirable dust from a sample (S/g_{pm10})

C_{mtrx} - the concentration of asbestos structures (of a defined size and type) in the original field matrix that was sampled for analysis using this method.

C_{smpl} - the concentration of asbestos structures (of a defined size and type) in a soil or bulk sample (S/g).

cm - centimeter (10^{-2} meter).

cm^2 - square centimeter.

cm^3 - cubic centimeter.

cm^3/min- cubic centimeter per minute.

d - the density of a particle (g/cm^3).

D_{rate} - the estimated rate of deposition on a specific filter (g/s).

^oC - degrees centigrade.

^oK - degrees Kelvin.

ΔM_f - the mass of respirable dust collected on a single filter during the interval Δt (g).

ΔM_s - the mass of respirable dust released from the sample during the interval Δt (g).

Δt - a short time interval (no more than 20 minutes).

η - the dynamic viscosity of air (g/cm*s).

eV - electron volt.

F_c - the percent of airflow (i.e. the % of the volumetric flow rate) through the top exit (ME) opening of the elutriator that does not pass through the isokinetic sampling tube (%).

F_d - the percent of airflow (i.e. the % of the volumetric flow rate) through the top exit (IST) opening of the elutriator that passes through the isokinetic sampling tube (%).

ft - foot.

g - gram.

g - the acceleration due to gravity (cm/s^2), when used as a variable in an equation.

g/L - gram per liter.

g/cm^3 - gram per cubic centimeter.

hp - horsepower.

k - the first order rate constant (s^{-1}).

kg - kilogram (10^3 gram).

kV - kilovolt.

in - inch.

L - liter.

L/min - liters per minute.

M_{coarse} - the mass of the coarse fraction of a matrix sampled in the field.

M_{DEP} - the target deposition loading for an analytical filter.

M_f - the cumulative mass of respirable dust collected on filters from the start of a run to time, t (g).

M_{fine} - the mass of the fine fraction of a matrix sampled in the field.

M_o - the mass of respirable dust in a sample at the start of a run (g).

M_r - the cumulative mass of respirable dust released from a sample from the start of a run to time, t (g).

M_s - the mass of respirable dust remaining in a sample during a run but after time, t (g).

M_{sample}- the mass of a sample introduced into the dust generator (g).

M_{tot} - the total mass of respirable dust estimated to reside in a sample (g).

ml - milliliter (10^{-3} L).

mm - millimeter (10^{-3} meters).

mm^2 - square millimeter.

µg - microgram (10^{-6} grams).

µm - micrometer (10^{-6} meters).

N_{go} - the number of grid openings counted during a scan (#).

nm - nanometer (10^{-9} meter).

P_f - the pressure measured at a flowmeter (torr).

P_t - the pressure estimated at an elutriator opening (torr).

%RD - the mass percent of respirable dust in a sample (%).

r - the radius of a particle (cm).

r^2 - the coefficient of determination (also defined as the correlation coefficient squared).

$R_{a/d}$ - the ratio of the number of asbestos structures (of a defined size and type) to the mass of respirable dust (S/g_{PM10}).

R_f - the flow reading from a flowmeter (cm/s).

R_{slt} - the mass fraction of silt in a bulk matrix (g_{silt}/g_{matrix})

S - the number of asbestos structures.

S_c - the number of asbestos structures (of a defined size and type) counted during a scan (#).

S_d - the number of asbestos structures that must be detected during a TEM scan for asbestos to be defined as detected (#).

S_{anal} - the required analytical sensitivity for this method (defined separately for total and long protocol structures) (S/g_{PM10}).

s - second.

S/g - structures per gram.

S/g_{PM10} - structures per gram of respirable dust.

S/L - structures per liter.

S/mm^2 - structures per square millimeter.

t - time (s).

t_{opt} - the time required to deposit a mass of M_{dep} on a filter (s).

T_f - the temperature at a flowmeter (^oK).

T_t - the temperature at an exit opening of the elutriator (^oK).

V_l - linear air flow rate (cm/s).

V_v - the volumetric air flow rate (cm^3/s).

W - watt.

5.2 ABBREVIATIONS

ANOVA- Analysis of Variance

ED - Electron diffraction

EDXA - Energy dispersive X-ray analysis

FWHM - Full width at half maximum

HEPA - High efficiency particle absolute

IST - refers to the opening at the top of the elutriator that is associated with the *isokinetic sampling tube*

MCE - Mixed cellulose ester

ME - refers to the *main exit* opening at the top of the elutriator, which is *not* associated with the isokinetic sampling tube

PC - Polycarbonate

PCM - Phase contrast optical microscopy

PCME- Phase contrast microscopy equivalent

PLM - Polarized light microscopy

PM_{10} - Respirable dust

RPM - Revolutions per minute

SAED - Selected area electron diffraction

TEM - Transmission electron microscopy

TSP - Total suspended particulate

UICC - Union Internationale Contre le Cancer

6.0 FACILITIES AND EQUIPMENT

6.1 SAMPLE COLLECTION EQUIPMENT AND CONSUMABLE SUPPLIES

To complete field sampling per this method, the following field equipment is mandatory:

- survey equipment appropriate to the manner in which sample locations are to be defined per the sampling plan;
- appropriate trowels, shovels, augers, or corers for sample collection per the sampling plan;
- (when sampling surface materials) a 12 in square aluminum template with an 8 in square hole in the center;
- a minimum of three 3-gal plastic buckets;
- a brass or steel sieve with 3/8 in. (1 cm) openings;
- a field balance (with a capacity of 40 kg and capable of achieving a precision of ± 10 g);
- a field balance (with a capacity of 2 kg and capable of achieving a precision of ± 0.2 g)[6]
- a riffle splitter with a minimum of 24, 3/4 in. (minimum size) chutes and three sample trays;
- one L plastic sample containers;
- sufficient plastic coolers to store and ship samples at ice temperature;
- equipment for cleaning sampling tools, including:
  - large buckets and tubs;
  - a container of asbestos-free water;
  - garden sprayers;
  - bio-degradable detergent;
  - assorted asbestos-free rags, sponges, etc.;
  - an air compressor with HEPA filter (optional, for drying equipment);
- field logbook and appropriate custody forms and sample labels;

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[6] If appropriate equipment is available, it is advantageous to use a single field balance to achieve both sets of capacity and precision requirements for field weighing.

- assorted garbage bags, paper towels, and tape;
- Tyvek suits and protective gloves; and
- appropriate equipment for respiratory protection.

6.2 LABORATORY FACILITIES

Laboratories wishing to adopt this method must develop and maintain the following facilities:

- a properly ventilated room for bulk sample handling that is isolated from other room(s) in which air samples are handled and analyzed by TEM. All such facilities must be sufficiently well ventilated to allow preparation of blanks that yield background determinations satisfying the requirements of Section 10.6 of the Superfund air method (Chatfield and Berman 1990); and
- a glove box or equivalent isolation chamber of sufficient size to house a riffle splitter (or other equipment) required for the homogenization and sub-sampling of samples for this method. The glove box or isolation chamber must provide ample room for handling kg size soil or bulk samples while maintaining background concentrations in the outside room air at levels considered acceptable as defined in Section 10.6 of Chatfield and Berman (1990).

6.3 LABORATORY EQUIPMENT

Implementing this elutriator method requires use of the following equipment::

- a dust generator-elutriator constructed per the specifications provided in Appendix A of the Superfund Method (Berman and Kolk 1997) and modified as follows:

– reduction of the size of the exit tube leading from the tumbler to the elutriator (through the tumbler bearing). Insert a 1.75 in. length of standard 0.25 in. O.D. copper tube as indicated in Figure 6-1. The tubing is sealed in place with a 1.5 in. sleeve constructed of 0.25 in. I.D. Tygon tubing, as depicted in the figure. Place the Tygon sleeve flush against inner face of the aluminum end plug. The copper tube extends approximately 0.25 in. into the tumbler body (beyond the end of the sleeve);

– placement of a conical shaped plug that fits into the hole at the bottom of the elutriator, as depicted in Figure 6-2. The plug should fit snugly but remain sufficiently loose to be easily removed;

– extension of the tube leading from the tumbler through the elutriator wall and bending downward toward the bottom of the elutriator. Using a removable attachment, extend the length of the 1.5 in. diameter X 0.625 in. wall stainless steel tubing so that it terminates no more than 0.5 in. from the bottom of the elutriator, as depicted in Figure 6-2. The extension may extend so that it just overlaps the top of the plug, but should leave ample clearance for free flow of air around the lip of the tube and up the outside of the tube into the main body of the elutriator; and

– plugging of the exit tube at the bottom of the elutriator and the side ports that originally led to the scrubber, as depicted in Figure 6-3. Place a No. 4 rubber stopper in each of the two exit ports of the elutriator, as depicted in the figure. Use a No. 8 rubber stopper to plug the hole at the bottom of the elutriator, as depicted in the figure.

Note that plugging the hole at the bottom of the elutriator obviates the need for the glass cup that was mounted over the bottom hole in the original configuration of the device (Figure A-6 of Berman and Kolk 1997). This also eliminates the small opening in the cup that served as a second source of air into the elutriator in the original configuration (Figure A-1 of Berman and Kolk 1997).

Note further that all modifications are designed such that the tumbler and elutriator can be returned to their original configuration (described in Berman and Kolk 1997) within a matter of minutes;

- a TEM operating at an accelerating potential of 80-120 kV, with a resolution better than 1.0 nm and a magnification range of approximately 300 to 100,000. The ability to obtain a direct screen magnification of about 100,000 is necessary for inspection of fiber morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. The TEM shall also be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 175 eV (FWHM) on the MnK_{α} peak. For requirements concerning screen calibration and SAED and ED performance, see Chatfield and Berman (1990);

- a computer system for recording analytical results. As indicated in the section addressing reporting requirements (see Chapter 12), analytical results are to be provided on computer disk (standard, high density 3.5 in or a readable/writeable CD) in a file format that is compatible with EXCEL™. ASCII files are acceptable;

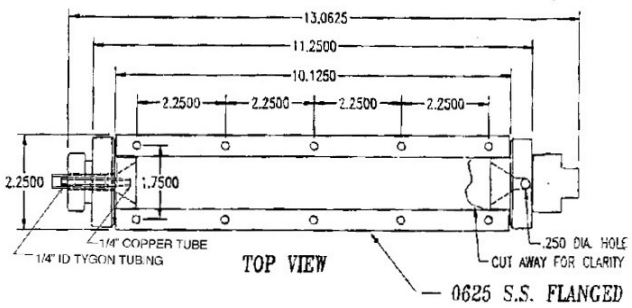
- a laboratory balance capable of mass determination with a resolution of 0.00001 g (1×10^{-5} g) with a capacity of 30 g;

- a laboratory balance capable of mass determination with a resolution of 0.000001 g (1×10^{-6} g) with a capacity of 200 mg;
- a laboratory oven, minimum 1 cu. ft. capacity; and
- a dessicator.

6.4 LABORATORY SUPPLIES

- One No. 8 rubber stopper.
- Two No. 4 rubber stoppers.
- One No. 3 rubber stopper with a three in. length of 1/4 in. copper tubing inserted through it and connected to about 5 ft. of 1/4 in. Tygon tubing.
- Five pounds of $CaNO_3 \cdot 4H_2O$.
- Mixed cellulose ester (MCE) filters: 25 mm dia., 0.47 µm pore size.
- Polycarbonate (PC) filters: 25 mm dia., 0.2 µm pore size.
- Plastic petri dishes for storing 25 mm filters

Figure 6-1: Modification for Exit Hole of Tumbler



Based on Figure A-14 of Berman and Kolk (1997).

FIGURE 6-2:
Elutraitor Bottom Modifications

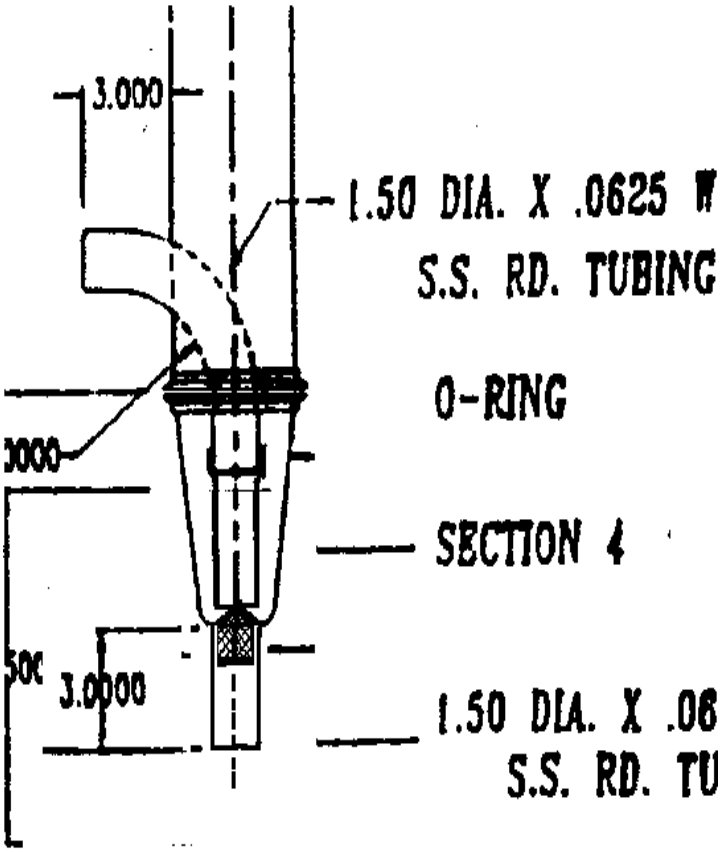
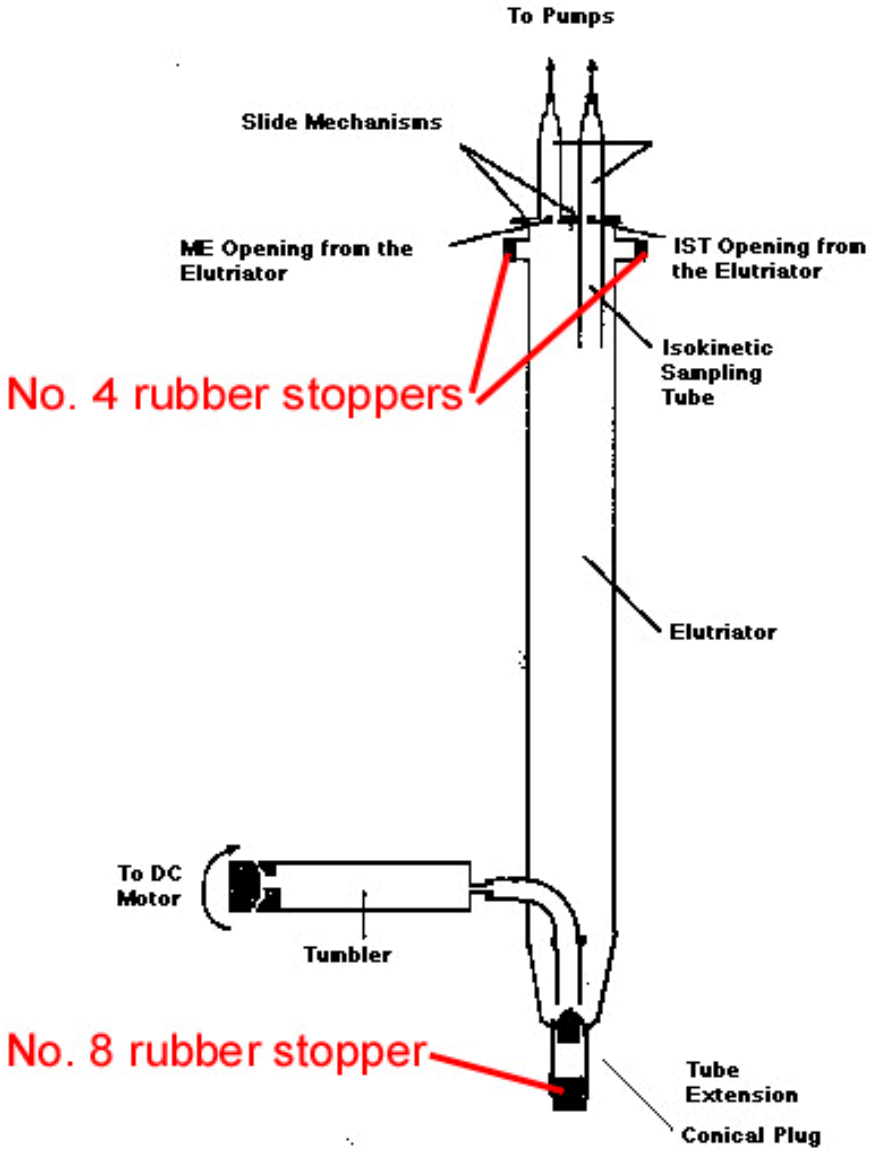


Figure 6-3:
Dust Generator Modifications



From Figure A-1 of Berman and Kolk (1997)

7.0 SOIL OR BULK SAMPLE COLLECTION

Sample collection procedures adopted for this method are flexible to allow adequate sampling of a broad variety of matrices. The method also incorporates several field preparation steps that are designed to preserve sample representativeness while reducing the mass of the samples sent to a laboratory for analysis. Controlling the mass of the samples sent to a laboratory from the field is a cost saving measure; the less material that a laboratory needs to handle as hazardous waste, the lower the total cost of analysis.

WARNING:

MOST OF THE SAMPLE COLLECTION PROCEDURES AND FIELD PREPARATION PROCEDURES DISCUSSED IN THIS DOCUMENT ARE INHERENTLY DUSTY OPERATIONS. THEREFORE, WHEN HANDLING SOILS OR BULK MATERIALS THAT ARE KNOWN TO CONTAIN OR POTENTIALLY CONTAIN ASBESTOS, IT IS IMPERATIVE THAT PROPER RESPIRATORY PROTECTION BE WORN WHILE CONDUCTING THESE PROCEDURES.

As indicated previously (see Section 2.2), measurements derived using this method serve as inputs for emission and dispersion models to predict airborne exposures attendant to disturbance of the sampled matrix. Such models also typically require measurement of the silt content of the sampled matrix. Therefore, it will generally prove prudent to collect at least a subset of samples for simultaneous characterization of the asbestos and silt content of each bulk matrix of interest.

Importantly, however, asbestos concentrations and silt contents serve distinct functions within the emission and dispersion models that are used to predict exposure. For this reason, optimal strategies for sampling a matrix may differ when the goal is to determine asbestos or silt content, respectively. For example, silt content measurements are input into the mathematical terms of these models that reflect activity-specific relationships between the characteristics of the matrix and the magnitude of emissions. It is therefore important that silt content measurements be collected in a manner assuring that they are representative of the effects of the specific release activities being modeled. For example, silt content should be determined for the specific product of a crushing or grinding operation that needs to be modeled.

In contrast, asbestos concentrations determined using this method are expected to reflect an inherent property of each of the materials sampled, which is largely independent of the effects of the release activities of interest (see Section 2.2). Therefore, locations from which soil or bulk samples are to be collected for determination of asbestos concentration shall be selected formally as part of a comprehensive strategy that is designed to provide a representative (unbiased) set of measurements for characterizing the overall mean concentration.

Although further consideration of such issues is required to identify appropriate sampling locations as part of the design of an effective field sampling plan, such considerations are addressed elsewhere (see, for example, Berman and Chesson unpublished) but are beyond the scope of this document. Rather, the remainder of this Chapter focuses on requirements for the collection and handling of individual samples at a site.

7.1 SAMPLE COLLECTION

Any variety of commercially available field sampling equipment (trowels, shovels, augers, corers, etc.) may be used to collect samples for this method. The equipment and procedure(s) selected should be based on the nature of the material being sampled and the depths over which samples are to be collected. Two common examples are presented in the original Superfund Method (Berman and Kolk 1997) so that only a general overview is provided here. Importantly, whatever equipment and procedures are chosen for sampling a particular matrix, they should be applied consistently and invariably at each sampling location within that matrix.

Whatever technique is chosen, the target *minimum* size sample to be collected at each sampling location shall be 1 kg[7]. Larger samples may be required, however, if particularly large (i.e. larger than a 4 or 5 cm in diameter) rocks or debris are present in the material being sampled. To assure representativeness, the largest component sampled should occupy no more than a few percent of the volume of the sample collected (Berman and Kolk 1997). Procedures for compositing samples, if this is desirable, have also been previously presented (Berman and Kolk 1997).

All sampling equipment shall be washed thoroughly with water and detergent between collection of each sample. Sampling equipment shall then be rinsed thoroughly with filtered, distilled water and allowed to air dry. Forced air may be used to expedite drying. If forced air is to be used to facilitate drying, however, such air must be passed through a HEPA filter to prevent delivery of any potential contamination.

Record the identification number, the date, time, and method of collection for each sample in a field notebook. Record the locations from which each sample is collected in the field notebook. Note in the logbook any changes between the sampling locations proposed in the sampling strategy and the actual locations sampled. As indicated previously (Berman and Kolk 1997), such changes are to be avoided to the extent

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[7] It may be impossible to collect kg-size samples for certain types of matrices (e.g. settled dust or manufactured asbestos products) that may be sampled for analysis using this method. The method can handle samples as small as 10 g, although samples smaller than 40 g require special preparation (see Section 8.3). When samples smaller than 1 kg are collected for field preparation and then analysis using this method, the interpretation of results requires special care. For small samples, for example, it is critical to address the consequent limitations that may be associated with compositing or sample homogenization, which may in turn affect representativeness.

possible. If changes are absolutely necessary, clearly document the rationale behind each change.

Supplement written documentation with photographs of each sampling location. This is particularly important if the sampling locations are not laid out on a formal, documented sampling grid that is tied to a permanent field marker.

7.2 FIELD PREPARATION

The specific procedures to be employed for field (and laboratory) preparation will differ depending on the nature of the matrix sampled. Consolidated solids (such as rock samples), for example, will need to be coarse crushed so that the majority of the sample ultimately passes through a 1 cm (3/8 in.) sieve. Such crushing must also be performed in a manner assuring that any fines produced during crushing will be preserved as part of the sample. Alternately, samples that are composed exclusively of fine material to begin with (such as settled dust) can likely be weighed, packaged, labeled, and sent directly to the laboratory without field preparation.

Most field samples will ultimately be handled as unconsolidated, heterogeneous solids. Such materials need to be weighed, separated into a coarse and fine fraction by passing the sample through a 1 cm (3/8 in) sieve, separately weighing the resulting coarse and fine fractions, homogenizing the fine fraction, splitting the fine fraction to produce samples weighing between 40 and 70 g each, and packaging, labeling, and shipping such sample splits to the laboratory for analysis. Detailed procedures for weighing, sieving, homogenizing, splitting, packaging, labeling, and shipment have previously been provided (Berman and Kolk 1997).

8.0 SAMPLE PREPARATION BY DUST GENERATION

The primary purpose for sample preparation by dust generation that is described in this section is to generate dust-laden filters that can be prepared (by direct transfer) for analysis by TEM to determine the concentration of protocol structures (see Chapter 2). The mass of respirable dust, which is deposited on such filters in coincidence with the asbestos, is also determined by weighing the filters. Dust and asbestos measurements are then combined so that asbestos concentrations can be appropriately reported as structures per mass of respirable dust (S/g_{PM10}).

A detailed description of the apparatus employed for dust generation and its theory of operation is provided in Appendix A of Berman and Kolk (1997). Specifications and construction drawings are also provided in that document. Modifications to the original dust generator, which are required to support measurement using this modified elutriator method, are described in Section 6.3 of this document.

8.1 SAMPLE RECEIVING AND STORAGE

All samples received from the field are to be wiped clean with a damp cloth prior to storage or other handling. Samples to be prepared using the dust generator are to be inspected for the presence of free water. If a sample contains free water or if the sample appears visibly moist, it shall be dried at low temperature. Dry the sample in an open, shallow container in an oven that is maintained at a temperature *below* 60˚ C until the sample comes to constant weight. Note that oven-dried samples may require additional time for conditioning (Section 8.5.2) because the moisture content of the sample will need to be increased to bring it into equilibrium with conditions prevailing in the dust generator.

Once dry, samples between approximately 40 g and 70 g can be loaded directly into the tumbler of the dust generator (Section 8.5.1)[8]. Larger samples must be homogenized and split, as described in Section 8.2, prior to being placed in the tumbler. Smaller samples need to be diluted by mixing with a known mass of washed, play sand prior to loading into the tumbler of the dust generator (see Section 8.3).

8.2 SAMPLE HOMOGENIZATION AND SPLITTING IN THE LABORATORY

Samples received from the field that are larger than approximately 70 g must be dried, as described above, and homogenized and split as described in this section.

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[8] Generally, the denser the sample, the larger the mass that can be tolerated. Ideally, the tumbler should be one-quarter full. Higher volume samples will need to be split prior to loading the tumbler. Lower volume samples will need to be diluted with washed, play sand. The optimum volume for a sample is approximately 36 cm^3.

WARNING:

BECAUSE ASBESTOS CONTAINING DUSTS MAY BE GENERATED FROM THE HANDLING AND PREPARATION OF BULK SAMPLES, ALL OF THE FOLLOWING PREPARATION STEPS SHALL BE PERFORMED IN A PROTECTIVE ENCLOSURE (I.E. A HEPA FILTERED GLOVE BOX OR AN APPROVED FUME HOOD THAT IS DESIGNED TO MINIMIZE EXPOSURE TO LABORATORY PERSONNEL).

As with field homogenization and splitting (described in Section 8.2.3 of Berman and Kolk 1997), either of two options may be selected for homogenization and splitting in the laboratory. When performed in the laboratory, however, such equipment must fit within an appropriately designed, protective enclosure, which is why field preparation may be cost-effective.

Homogenize large volume samples in precisely the same manner as described in Section 8.2.3 of Berman and Kolk (1997). Once samples are homogenized, split samples in precisely the same manner as described in the same section of that document. Continue splitting until a paired set of samples are produced that each contain between approximately 40 g and 70 g of material (or a target volume of 36 cm^3). Record in a laboratory notebook the final masses and identification numbers of the samples that are homogenized and split.

8.3 DILUTING SAMPLES WITH WASHED, PLAY SAND

Samples that are smaller than approximately 40 g shall be diluted with washed, play sand prior to loading in the tumbler.

Weigh out a mass of sand such that the sum of the mass of sand and the mass of the sample of interest will total approximately 70 g. The sand and sample can be mixed directly in the tumbler. Layer half of the sand into the tumbler (in the manner described in Section 8.5.1) so that the material is deposited evenly across the tumbler's bottom. The sample shall then be layered over the sand so that it too is uniformly spread across the sand at the bottom of the tumbler. Finally, layer the rest of the sand over the top of the sample material so that it too is deposited uniformly across the bottom of the tumbler. It is anticipated that the rotation of the tumbler during the initial 1 hour or so of a run, prior to the start of filter collection for asbestos analysis (see Section 8.5.4), will be adequate to homogenize the mix of sand and sample. Alternately, the sand and sample may be thoroughly mixed (by shaking together in a sealed plastic bag) prior to placing the mix in the tumbler. This latter approach may be preferable because it may lessen the time required for dust generation to stabilize (see Section 8.5.4).

> Note, the washed, play sand that is commercially available is generally free of asbestos and contains minimal respirable material. Prior to using such material in this method, however, the sand must be analyzed to confirm its "purity." To analyze the material, place approximately 70 g of candidate sand in the tumbler

of the dust generator. Then, follow all of the steps described in this chapter for generating filters (including sample conditioning and dust generation). As with normal runs, let the dust generator run for about an hour before collecting filters for measurement (Section 8.5.2).

Collect any asbestos (and the associated dust) generated from the sand on filters mounted on the IST opening of the elutriator. Continue passing air through such filters for a period at least *five times* as long as anticipated to be required for loading filters from actual asbestos samples of interest (see Section 8.5.5). The mass of respirable dust deposited on filters over the IST opening of the elutriator from such a run (with the candidate sand) shall then be weighed. Sand can only be used to dilute low volume samples for this method if it exhibits no detectable dust (i.e. less than 5 µg) based on weighing of the filters generated as defined above.

The filters generated from this run with the candidate sand must then be prepared by direct transfer for TEM analysis and analyzed for the determination of asbestos content. Asbestos loading on the sand filters shall be demonstrated to be less than 1 structure in 200 grid openings. Two random samples shall be collected from each 25 kg (50 lb) bag of washed, play sand and analyzed as described above to qualify the sand for use.

Importantly, the largest size particles that can be tolerated on smaller volume samples (to assure adequate homogeneity) are somewhat smaller than the 1 cm target maximum that has been defined for this method. For example, although 1 cm size particles are probably tolerable for samples approaching 40 g total mass, particles no larger than approximately 0.5 cm should be tolerated in samples weighing only 10 g. Field preparation techniques recommended for preparing samples that are shipped to the laboratory for analysis using this method should be adjusted to assure adequate homogenization (that preserves the representativeness of such samples) when the availability of or access to bulk matrices of interest limit the size of samples that can be collected.

8.4 DUST GENERATOR SETUP

Prior to using the dust generator, a supply of at least 15 MCE filters must be conditioned and stored for use. The constant humidity chamber must be loaded with the appropriate solution, and air flow within the dust generator must be calibrated and adjusted.

8.4.1 Conditioning a Stock of Filters

A stock of at least 15 filters (0.45 µm pore size, 25 mm diameter), all from the same filter lot, must be conditioned in a desiccator overnight to bring them into equilibrium with the relative humidity at which they will be used during a run. Place the 15 MCE

filters in a desiccator containing *moist* salt of the same variety as that selected to fill the pans in the humidity control chamber of the dust generator (Section 8.4.2). For most applications, this will be $CaNO_3 \cdot 4H_2O$ (see Appendix A of Berman and Kolk 1997).

After storing the filters overnight in the desiccator, pre-weigh each filter to a minimum precision of ±0.00002 g. Each filter shall then be placed in a separate, covered Petri dish with its weight marked on the top of the container. The lids shall also be numbered sequentially and the filters shall all be used during the run in the order numbered.

Ten polycarbonate filters of either 0.1 or 0.2 µm pore size shall also be weighed on a microbalance with a precision of ±0.000002 g. The polycarbonate filters are not sensitive to humidity changes in the range of 40 to 55% RH. This range can be achieved in an air conditioned office environment. The 0.2 µm pore size is suitable for collecting protocol structures, as required by this method. The 0.1 µm pore size filters are recommended, if fibers as short as 0.5 µm in length are to be collected and counted.

8.4.2 Initiating Humidity Control

Use asbestos-free (filtered, distilled) water to make a 2 L solution of saturated salt. As indicated previously, for most applications, use $CaNO_3 \cdot 4H_2O$, but other salts may be used for specific applications (see Appendix A of Berman and Kolk 1997).

Prepare the solutions by placing 1000 g of the $CaNO_3 \cdot 4H_2O$ into a one L container and adding distilled water to fill the container. The container should be kept capped except when adding additional water or salt on succeeding days, as necessary, until it appears that the solution has stabilized (i.e. no changes in the relative amounts of water and salt are apparent from one day to the next). The solution may also be agitated to accelerate dissolution of the salt and the approach to equilibrium. No water or salt shall be added less than 24 hours from the time that the mixture is to be used, because adequate time is required for the salt solution to come to stable equilibrium.
To load the salt solution, open the top of the humidity control chamber and remove the two pans. Fill each pan with the saturated salt solution being sure that about 25% by volume undissolved salt is also transferred to each pan. Replace the pans and replace the front panel of the plastic enclosure; air should enter the enclosure primarily from the top opening.

8.4.3 Adjusting the Initial Air Flow

The air flow within the various components of the dust generator must be adjusted so that flow within the vertical elutriator will properly separate and pass only respirable particles. Based on the discussion presented in Appendix A (Section A.2.3), the proper linear flow rate in the elutriator shall be set at 0.31 cm/s, which is 5% greater than the Stokes' velocity estimated for the largest spherical, respirable particles (i.e. those with an aerodynamic diameter of 10 µm and unit density).

Next, calculate the required volumetric air flow, V_v, within the elutriator using Equation 8-1:

$$V_v = 81.1 * V_l \qquad (8\text{-}1)$$

where:

V_l is the estimated linear flow rate required to separate respirable particles (i.e. 0.31 cm/s); and

V_v is the corresponding volumetric flow rate (cm^3/s) through the elutriator.

The coefficient, "81.1", in Equation 8-1 corresponds to the cross-sectional area of the elutriator (in cm^2) with the internal diameter given in the drawings supplied in the Superfund Method (Berman and Kolk 1997)[9].

> Note: the following discussion, which describes procedures both for adjusting air flow within the dust generator and for checking for leaks, assumes familiarity with the component pieces of the dust generator. It is therefore recommended that the reader familiarize themselves with the operation of the dust generator by reading Chapter 9 and Appendix A of Berman and Kolk (1997), prior to proceeding with the rest of this section.

8.4.3.1 Determining proper air flow for a dust generation run. Using Equation 8-1, V_v is calculated to be 25.14 cm^3/s. This is the volumetric flow rate that must be maintained in the elutriator to prepare filters suitable for analysis by this method. This flow must then be divided between flow out of the ME opening of the elutriator and the IST opening of the elutriator.

Given the specifications indicated in the construction drawings for the elutriator (Figures A-15 and A-17 in Appendix A of Berman and Kolk 1997), the cross-sectional area of the isokinetic sampling tube is 4.7% of the total internal cross-section of the elutriator. Therefore, during a run, flow must be adjusted so that 4.7% of the flow in the elutriator (1.19 cm^3/s or 72 ml/min) passes through the IST opening of the elutriator and the remaining 95.3% (23.9 cm^3/s or 1,430 ml/min) passes through the ME opening.

> Note, if the design of the elutriator being used for this method differs from that defined in Berman and Kolk 1997 (and modified as described of Section 6.3 of this document), air flow requirements will have to be adjusted accordingly.

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[9] If the design of the dust generator being used for this method differs from the that defined in Berman and Kolk 1997, Equation 8-1 will need to be adjusted accordingly.

To prepare for a run, the elutriator must first be properly set up. Filter cassettes should be attached to the flowmeters and the pumps of the dust generator through the stopcocks as shown in Figure 8-1[10]. Appropriate filters must then be mounted in each of the four cassettes: 0.45 µm MCE filters (over backing pads) are mounted in the filter cassettes over the ME opening of the elutriator and 0.2 µm polycarbonate filters (with 0. 45 µm MCE filters as backing pads) are mounted in each filter cassette of the IST filter assembly.[11]

> **A FILTER CASSETTE CONTAINING A 0.45 µm MCE FILTER AND BACKING PAD IS ALWAYS PLACED BETWEEN EACH PRECISION FLOWMETER AND ITS CORRESPONDING PUMP FOR SAFETY REASONS. THIS IS TO PREVENT ASBESTOS FIBERS FROM BEING PUMPED INTO LABORATORY AIR SHOULD ONE OF THE FILTER CASSETTES ON THE DUST GENERATOR FAIL OR SHOULD SOMEONE INADVERTANTLY FORGET TO MOUNT A FILTER INTO ONE OF THE SLIDER CASSETTES DURING OPERATION.**

When the elutriator is being run, each stopcock is positioned so that the active filter on each slider is the only one connected to its pump. Just before an active filter is switched, stopcock(s) are adjusted so that flow is now directed through both the active and the inactive filter on a slider. The slider is then repositioned to place the new filter over the opening of the elutriator. Stopcock(s) are then adjusted again so that flow to the old (previously active filter) is shut off and all of the flow is now directed through the new, active filer.

> Note: when a slider is being repositioned with both stopcocks open (or a corresponding, three-way stopcock adjusted to allow flow to both filters), the flow out of the elutriator is never completely shut off because the space between the two filter mounts on the slider is less than the diameter of the hole into the elutriator.

Once the elutriator is properly configured, the flow meters calibrated, and the tumbler loaded (Section 8.5.1), the dust generator is ready for use. Turn on the pumps and adjust flow so that the required 1,430 ml/min pass through the ME opening of the elutriator (with a loaded filter mounted over the opening) and the required 72 ml/min pass through the IST opening and the associated filter cassette[12]. Check for variation

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[10] One modification to the setup depicted in Figure 8-1 that has proven helpful is to replace each assembly of multiple stopcocks and glass "Y" tubes depicted in the figure with single, three-way stopcocks.

[11] The indicated configuration is appropriate for measurement of protocol structures, which are all longer than 5 µm. If there is a need to count shorter structures, use 0.1 µm pore size PC filters in the cassettes over the IST opening of the elutriator.

[12] It has been observed that increasing the flow through the IST opening increases the efficiency of deposition on filters mounted over this opening. If necessary to facilitate collection, flow through the IST

in flow as sliders and valves are adjusted (as when "changing out" filters) during a run and correct accordingly. Flow through the ME opening should vary by no more than ± 10% from the required value as flow is switched between the two filter mounts on the associated slider. Flow through the IST opening should vary by no more than -0% to +20% as flow is switched between the two filter mounts on the associated slider. Importantly, the tolerance range is adjusted upward for the IST opening so that the *minimum* acceptable volume flow rate is the target value. It is, however, acceptable, for air flow in the IST opening to slightly exceed the target value.

8.4.3.2 Checking for leaks. The filter assembly over the IST opening of the elutriator shall first be checked for leaks prior to assembling the dust generator for a run because the low flow rate in this filter assembly (i.e. 72 ml/min) makes it very sensitive to small leaks. Similar leaks do not affect the performance of the filter assembly over the ME opening of the elutriator because flow in this assembly is so much higher under normal operating conditions.

To check the integrity of the filter assembly over the IST opening, remove the top section off the elutriator and insert the No. 3 stopper with the Tygon tubing into the bottom of the isokinetic tube. Place a backing pad and filter into each cassette on the slider. Next open the stopcock(s) that connect one filter of this assembly to the entrance end of one of the 375 ml flowmeters. The exit end of this flowmeter (which is the flowmeter that will read air flow through the IST opening during actual dust generator runs) is then connected through a filter cassette to a pump.

> **A FILTER CASSETTE CONTAINING A 0.45 µm MCE FILTER AND BACKING PAD IS ALWAYS PLACED BETWEEN THE FINAL FLOWMETER AND THE PUMP FOR SAFETY REASONS. THIS IS TO PREVENT ASBESTOS FIBERS FROM BEING PUMPED INTO LABORATORY AIR SHOULD ONE OF THE FILTER CASSETTES ON THE DUST GENERATOR FAIL OR SHOULD SOMEONE INADVERTANTLY FORGET TO MOUNT A FILTER INTO ONE OF THE SLIDER CASSETTES DURING OPERATION.**

Position the filter with the open stopcock(s) so that it is aligned directly over the IST opening on the elutriator top. Connect the tubing from the stopper at the bottom of the isokinetic sampling tube to the exit end of another 375 ml flowmeter, the entrance to which is left open to the air.

When the pump connected to the filter assembly in the manner described above is turned on, the flow path is from the air, through the first flowmeter, into the bottom of the isokinetic sampling tube, through the IST opening at the top of the elutriator, through the active filter cassette, through the second flowmeter (which is the flowmeter that will read air flow during actual dust generator runs), through the safety filter cassette, and to the pump.

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opening can be increased by as much as 70% over its nominal value. This has virtually no effect on the overall operation of the elutriator.

Turn the pump on and adjust the flow through the filter to the 72 ml/min level on the second flowmeter (i.e. the one that will be used to monitor flow during actual runs). The first flowmeter should also read the same flow within 7%. If the difference is greater than 7%, the system shall be checked for leaks. When both flowmeters read 72 ml/min plus or minus 7%, the stopcocks should be switched so that flow now occurs through both filters on the slider and the readings again compared. Both should still read 72 ml/min ± 7%.. The slider shall then be positioned with the second filter over the IST opening and the stopcock(s) adjusted to direct flow only through the second filter. The readings on the two flowmeters should remain the same, within the stated tolerance.

It is prudent also to check for leaks in the assembly over the ME opening of the elutriator. To accomplish this, remount the isokinetic tube onto the opening for the ME filters and repeat the above-described procedure at the same *low* flow rate to check for leaks in the filter assembly over the ME opening. If no leaks are detected in that slider assembly, the isokinetic tube is returned to its original position (under the IST opening) in preparation for a run.

Once leak detection is complete, the elutriator can be reassembled in preparation for a run.

8.5 DUST GENERATOR OPERATION

To prepare asbestos samples using the dust generator: load the tumbler, condition the bulk sample, wait for dust generation to stabilize, and collect appropriately loaded filters for asbestos analysis by TEM following preparation by direct transfer.

8.5.1 Loading the Tumbler

Detach the tumbler from its drive motor and the vertical elutriator and remove it from the plastic enclosure at the bottom of the dust generator (see Appendix A of Berman and Kolk 1997). Place the tumbler on a support surface and open the top for loading. Be sure that the tumbler is clean prior to loading.

Introduce a sample by holding the sample container against the inner lip of the tumbler and tilting the container so that the sample pours smoothly into the tumbler. Move the sample container back and forth along the length of the tumbler to facilitate uniform deposition of the sample in the tumbler. When pouring is complete, tap the sample container vigorously so that the quantitative transfer is complete. The masses of samples introduced into the tumbler shall range between 40 g and 70 g. Larger samples shall be homogenized and split prior to loading as described in Section 8.2. Samples smaller than 40 g can also be run by first diluting them with washed, play sand (Section 8.3).

Shake the tumbler gently to assure uniform deposition of the sample within the tumbler, which should be no more than about one quarter full. Be sure that the rubber gasket on the tumbler is in good repair and properly seated. Replace the gasket if it is worn. Secure the top of the tumbler with 10 screws and replace the tumbler within the plastic enclosure at the bottom of the dust generator. Reattach the elutriator entrance tube and D.C. motor to the tumbler (see Appendix A of Berman and Kolk 1997).

8.5.2 Conditioning the Sample

Before conditioning the sample, be sure that the dust generator has been properly set up. This means, check that:

- the pans in the constant humidity chamber have been filled with saturated salt solution;
- appropriate size and type filters have been mounted in each of the four cassette mounts on the slide mechanisms atop the elutriator;
- air flow valves have been properly set; and
- all air lines between the dust generator, flow valves, and pumps are properly configured (see Section A.1.1 of Appendix A of Berman and Kolk 1997).

To condition the sample, turn on all pumps and begin the flow of air through the dust generator. ***DO NOT TURN ON THE TUMBLER MOTOR.*** Allow the flow of air to continue for a minimum of two hours before beginning a run.

8.5.3 Initiating a Run

Once the sample has been conditioned. set the tumbler drive motor to 30 rpm and turn it on. Simultaneously, move the two slide mechanisms at the top of the elutriator so that new, clean filters are now aligned over both the ME and IST openings of the elutriator. Be sure to change the valve orientations on the lines leading to the filters so that air flow is directed through both filter cassettes of each slider while the sliders are moved and then to the filter cassettes that are newly aligned with the elutriator openings.

Replace the filters originally aligned over the elutriator openings (but no longer aligned) with clean filters and weigh and store the old filters in labeled Petri dishes. These filters are conditioning filters and may be useful for isolating a problem should corrective actions be required.

8.5.4 Monitoring to Confirm Stable Dust Generation

The rate of respirable dust generation is monitored at the start of a sample run to indicate when conditions have stabilized[13]. Filters to be used for determination asbestos concentration shall only be collected after dust generation has stabilized. Dust generation is monitored by recording the deposited mass on each of a set of filters that are sequentially changed out of the filter mounts over the ME opening of the elutriator at defined, regular intervals.

Initially, change out the filters aligned over the ME opening of the elutriator at intervals of between ten and twenty minutes. The change is accomplished by moving the slide mechanism to switch a new filter into alignment at the same time that the old filter is switched out of alignment. Prior to sliding the filters, be sure to adjust the stopcock(s) so that flow is simultaneously directed to both the active filter (i.e. the one over the opening to the elutriator) and the inactive filter (i.e. the one that is out of the line of air flow).

After sliding the new filter into alignment, turn the associated stopcock(s) so that the air flow is directed only through the new filter. Dismount and replace the old filter (the one previously exposed to the air stream from the elutriator) with a fresh filter and place the exposed filter in the Petri dish in which it was initially stored after the determination of its tare mass. Repeat this process at regular intervals to generate a series of filters that represent dust sequentially collected from the air flow in the generator. If required to better understand this process for monitoring dust generation, a more detailed description is provided in Section 9.4.4 of Berman and Kolk (1997).

Along with the proper identifier, record the times during which air flow is started and halted for each filter. Weigh each filter after dismounting. Record the initial and final masses of the filter and the net mass of dust deposited on the filter (i.e. the difference between the initial tare mass and the final mass). Determine the net amount of time during which dust was being deposited on the filter by subtracting the start time from the stop time (in seconds). Then compute the average rate at which dust was deposited on the filter by finding the ratio of the net mass deposited to the net time over which deposition occurred.

After collecting dust on the first two or three filters, the interval over which dust is collected on each filter may be optimized. The ideal mass of dust to be deposited on each filter is between 0.002 and 0.006 g. Estimate the interval of time required to deposit approximately 0.006 g and exchange later filters at the optimum rate. When adjusting the time for dust deposition, however, be sure to avoid depositing more than 0.006 g of dust on any filter because the probability that a portion of the deposit will be lost during handling increases as the mass of the deposit on the filter increases.

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[13] Dust generation can also be monitored and modeled for extended periods, if there is a desire either to estimate the absolute concentration of respirable dust in the original sample or to explore the dynamics of dust generation for the particular matrix being analyzed (see Berman and Kolk 1997 for details).

Once dynamic equilibrium is achieved, so that dust generation stabilizes, the rate of release of dust from a sample in the tumbler has been shown to decrease slowly and regularly with time as the reservoir of dust within the sample is depleted. Such decay has been shown to follow first-order kinetics (see Section A.1 of the Appendix).

That dust generation has stabilized will be apparent when the computed rate of dust deposition on each subsequent filter that is collected over the ME opening of the elutriator indicates a slow, monotonic decrease with time[14]. Rate estimates for individual filters should not vary from their nominal value (predicted from a linear trend) by more than 5%. When this pattern is observed, filters can then be collected over the IST opening of the elutriator for determination of the concentration of asbestos (Section 8.5.5). Typically, about an hour of run time is required before conditions stabilize sufficiently to allow collection of filters for asbestos analysis.

8.5.5 Collecting Filters for Asbestos Analysis

In preparation for collecting filters for asbestos analysis, estimate the time that will be required for optimal deposition. Estimate the optimum time required to collect dust on the filters to be used for asbestos analysis using Equation 8-2.

$$t_{opt} = 20*M_{dep}/D_{rate} \qquad (8\text{-}2)$$

where:

t_{opt} is the time required to collect a filter deposit of mass M_{dep} (s);

M_{dep} is the target mass of the deposit on the filter (g); and

D_{rate} is the estimated rate of deposition for the specific run over the time interval of interest (g/s).

The derivation of Equation 8-2 can be found in Section A.2.2 of the Appendix. Equation 8-2 is simply a recapitulation of Equation A-10, with the terms redefined.

Recent experience with this method (Kolk unpublished) indicates that the optimum loading for filters to be prepared for asbestos analysis using this method is approximately 100 µg of respirable dust. By substituting this value for M_{dep}, Equation 8-2 can be further simplified:

$$t_{opt} = 0.002/D_{rate} \qquad (8\text{-}3)$$

where all terms have been previously defined.

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[14] In some cases the rate of decrease is so small that the rate of dust deposition appears steady.

The D_{rate} in Equation 8-3 is estimated simply from the deposition rates estimated from the last set of filters collected over the ME opening of the elutriator. t_{opt} can then be derived accordingly. To expedite asbestos measurement using this method, it is recommended (but not required) that a set of multiple filters be collected, which bracket the optimum collection time estimated using Equation 8-3. After superficial examination of gird specimens prepared from such filters, the optimally loaded filter can then be selected for final analysis.

> Note: to collect optimally loaded filters over the IST opening, the flow rate through the opening may be increased by as much as 70% over its nominal value. Although this will have no impact on elutriator performance, dust deposition over the IST opening has been found to be very sensitive to minor changes in this flow rate.

Dust is collected on filters mounted over the IST opening in the same manner previously described for filters mounted over the ME opening of the dust generator. Polycarbonate (PC) filters shall be used for collecting asbestos samples because PC filter masses have been found to be less affected by humidity changes than MCE filters (Kolk unpublished). Filters should be left in line with the air flow over the IST opening for the period of time approximately equal to t_{opt} (estimated using Equation 8-3).

Following dust collection, each PC filter shall be weighed on the microbalance to determine whether the target dust mass was successfully collected. Unfortunately, a substantial electrostatic charge builds up on these filter during deposition and this charge must be neutralized before an accurate weight can be obtained.

Cahn, a manufacturer of microbalances, sells radioactive sources that can be placed in the weighing chamber and also sells solutions of radioactive salts that can be used to coat the inside of the weighing chamber. There is a technical note available from Cahn indicating how to minimize the effects of electrostatic charge. Care must be taken when eliminating static charges to assure that particles are not loss from the filter surface. Another way to eliminate charges on a filter is to place the filter in a covered Petri dish lined with a conducting material that is ground and that is maintained at a high relative humidity, but not sufficiently high to cause condensation. The filter is left for several hours to allow the charge to dissipate. Although this latter method is slower than using a radioactive source to neutralize charge, it has been found to be less problematic (with much lower potential for disturbing the dust deposit on the filter). It is this latter procedure that was used during method tests and found to be satisfactory.

It is recommended that approximately five filters be collected for asbestos analysis during each run with the loading on each such filter increased sequentially by increasing deposition time. If there appears to be difficulty obtaining adequate deposits on these filters, the air flow through the isokinetic sampling tube can be increased by as much as 70% beyond nominal to facilitate particle collection. This will have virtually no impact on the elutriator's ability to separate respirable and non-

respirable particles. The filter chosen for preparation and TEM analysis shall be the filter that exhibits a deposited mass closest to 100 µg.

After the five filters are collected, the run may be terminated. However, it is recommended that the dust generator assembly be left undisturbed until the PC filters are determined to be satisfactory for TEM examination.

When the static charge has dissipated to the extent that it does not interfere with weighing, the PC filters shall be weighed. If one or more of the filters is within 10% of the target weight of deposit (100 µg), the tumbler can be emptied, the remaining material archived, and the elutriator cleaned.

8.6 CLEANING THE DUST GENERATOR

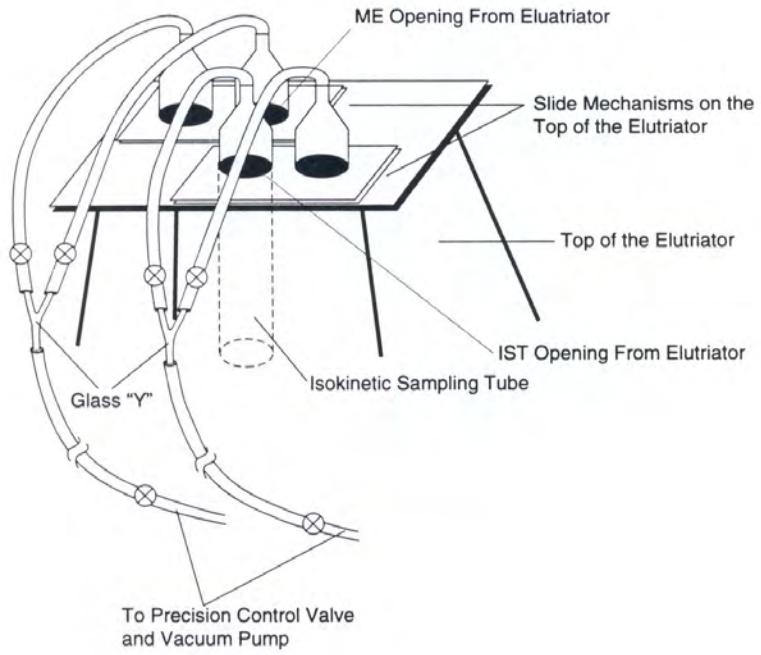
8.6.1 Cleaning the Elutriator

The dust generator is designed for quick and easy assembly and disassembly to facilitate cleaning. Most of the joints are simple friction couplings or ring clamp couplings. To clean the dust generator, carefully disconnect and disassemble the tumbler, remove the plug and dust collector system from the elutriator, decouple the sections of the elutriator tube, disassemble the slide mechanisms of the dust collector. Be careful to not disturb or spill any of the dust on the bottom and sides of the elutriator. Conduct the transfer of all material left in the elutriator to a container that can be capped. The metal pieces of the dust generator may then be washed with biodegradable detergent and rinsed with asbestos-free water. The pieces may then be left to dry in room air or may be dried with a forced, HEPA-filtered air stream. Discard asbestos containing waste according to applicable regulations.

8.6.2 Optional Method for Disassembling the Elutriator

An optional cleaning procedure for soils that are believed to be heavily contaminated with respirable asbestos is as follows. Use a 1/2 gallon size pressurized tank garden spray to wet the inside of the elutriator. The ME filter holder is removed to expose the opening at the top of the elutriator and the mist spray is injected into the opening until the contents of the elutriator are lightly wetted. The spray is also applied to the connection to the tumbler while detaching the tumbler from the elutriator. The spray is then directed into the tube leading into the bottom of the elutriator and the inside lightly misted. The elutriator may then be disassembled and cleaned. The contents of the tumbler can be archived and the soil removed from within the elutriator can also be dried and archived if desired. Again any contaminated waste should be given proper disposal.

Figure 8-1:
Tubing Connections for Filter Cassettes Mounted On the Elutriator



⊗ Stop Cock Valves

From Figure A-11 of Berman and Kolk (1997)

94-102.4 Mac 4

9.0 PREPARATION OF SPECIMEN GRIDS FOR TEM ANALYSIS

Prepare speciment grids from filters collected over the IST opening of the elutriator.

Although this method specifies that filters collected over the IST opening of the elutriator shall be prepared using a direct transfer technique, an indirect transfer technique is also described, as an option for certain applications. The primary purpose for considering indirect transfer is that it may prove cost-effective under special circumstances. Importantly, however, one must also consider that concentration measurements derived from indirectly prepared samples are not generally good predictors of risk (see Berman and Crump 1999a). Thus, indirect preparation should be used in association with this method only after appropriate consideration of the consequences.

9.1 SPECIMEN GRID PREPARATION USING A DIRECT TRANSFER TECHNIQUE

Filters collected over the IST opening of the elutriator (as described in Section 8.5.5) shall be prepared using the direct transfer technique that is described in Section 10.5 of the IS0 Method (ISO 10312). The loading calculation described in Section 8.5.5 Equation 8-3) is for a filter loading of 100 µg. If a different loading is desired, the appropriate target mass needs to be substituted into the equation to estimate the appropriate time for collection. A loading of over 150 µg is not recommended.

From each filter that has been collected over the IST opening, prepare four specimen grids from locations on the filter that are each separated by 90° radially and also prepare one grid from the center of the filter. The four outer grids should be about 75% of the distance from the center to the active edge of the filter (i.e.about 8.3 mm from the center of the filter). Thus, a total of 5 grid specimens shall be prepared from each filter.

9.2 SPECIMEN GRID PREPARATION USING AN INDIRECT TRANSFER TECHNIQUE

As an option to the procedure described in Section 9.1 above (for special applications *only),* filters collected over the IST opening of the elutriator (as described in Section 8.5.5) may also be prepared using the indirect transfer technique that is described in Sections 10.3 to 10.5 of the IS0 Method (ISO 10312). For this option, multiple sections of the most highly loaded filter obtained from the dust generator shall be prepared using a range of dilutions to allow selection of the optimally loaded specimen grids for final, detailed analysis.

Alternately the more highly loaded filters collected over the ME opening of the elutriator (to monitor the stability of dust generation as described in Section 8.5.4) may also be prepared by indirect transfer for asbestos analysis.

10.0 PROCEDURES FOR ASBESTOS AND DUST ANALYSIS

10.1 PROCEDURES FOR ASBESTOS ANALYSIS

Specimen grids prepared as described in Chapter 9 are to be analyzed using transmission electron microscopy (TEM). Follow the procedures for analysis described in the ISO Method (ISO 10312) including procedures for:

- examining specimen grids to determine acceptability for analysis;
- structure counting by TEM (except that counts are limited to parent structures and components that qualify as protocol structures and the determination of the stopping point is modified);
- structure morphological classification;
- structure mineralogical identification; and
- blank and quality control determinations.

The stopping points for the analyses conducted in support of this method are a function of the required sensitivity for the method and are defined in Section 10.1.2 below.

Begin by examining one of each set of specimen grids derived for a defined loading from a particular run of the dust generator and select the optimally loaded set for analysis. Use the criteria for determining the acceptability of specimen grids (from the ISO Method) to define optimal loading.

When performing detailed analysis, be sure to distribute asbestos counts evenly over the entire set of specimen grids prepared from a particular filter at a defined (optimal) loading. Record the morphology and mineral type of asbestos structures as described in the ISO Method (ISO 10312). Importantly, because protocol structures are limited to structures longer than 5 µm, scans for counts of protocol structures can be performed at a magnification of 10,000x, which should expedite analysis relative to higher magnification scans.

10.1.1 Counting Rules for Asbestos

Because this method is focused specifically on deriving counts of asbestos protocol structures, count only structures longer than 5 µm that are also thinner than 0.5 µm. Qualifying structures that are longer than 10 µm also need to be distinguished for separate enumeration. Include in the count all isolated structures that exhibit the required dimensions and identifiable components of complex structures that also exhibit qualifying morphology. Chrysotile and amphibole structures must be separately enumerated. For amphiboles, identify the specific mineral type.

10.1.2 Analysis of Specimen Grids Prepared from Filters Collected Over the IST Opening of the Elutriator

Prior to initiating a detailed analysis of specimen grids, the stopping rules for the analysis must be defined. Assuming these specimen grids have been prepared using a direct transfer technique (as discussed in Section 9.1), define the stopping rules for the detailed analysis as follows.

First, calculate the maximum number of grid openings that will have to be scanned during the analysis from the relationship:

$$N_{go} = S_d * A_f * / (S_{smpl} * A_{go} * \Delta M_f) \qquad (10\text{-}1)$$

where:

N_{go} is the maximum number of grid openings to be scanned;

S_d is the number of structures required to define detection using the analysis (defined here as 1);

A_f is the total area of the filter from which the specimen grids were prepared (mm^2). A typical value for the surface area of a filter is 385 mm^2;

S_{smpl} is the required analytical sensitivity for the method (s/g_{PM10}). A target analytical sensitivity of $3x10^6$ s/g_{PM10} is defined in Section 2.4, which should be adequate for most studies ;

A_{go} is the area of a single grid opening (mm^2). A typical value for the area of a grid opening is 0.01 mm^2; and

ΔM_f is the mass of respirable dust collected on the filter from which the specimen grids were prepared (g). As indicated in Section 8.5.5, a reasonable target loading is $1x10^{-4}$ g.

Given a target sensitivity of $3x10^6$ s/g_{PM10} and typical values for each of the parameters of Equation 10-1 (listed above), approximately 150 grid openings will need to be counted for a typical sample. Importantly, to count protocol structures, such scans need to be performed at a magnification of only 10,000x.

The number of grid openings to be scanned for specific analyses shall be determined by substituting case-specific values for the above listed parameters into Equation 10-1.

Stop the counting, characterization, identification, and recording of asbestos structures on a particular analysis when one of the following obtains:

- the scan is completed for the grid opening on which the 25th asbestos protocol structure that is longer than 10 µm is counted; or
- a sufficient number of grid openings are scanned to achieve the target analytical sensitivity.

Note, because a total of 5 grid specimens are to be prepared from each filter, it is practical to modify the above-listed stopping rules so that they can be applied to each of the individual grid specimens from a particular filter. Thus, stop counting, characterization, identification, and recording of asbestos structures on a particular grid specimen when:

- the scan is completed for the gird opening on which the 5^{th} asbestos protocol structure that is longer than 10 µm is observed on the particular grid specimen being scanned; or

- one fifth of the total number of grid openings required to achieve the target analytical sensitivity are scanned on the particular grid specimen.

The above rules must then be applied to each of the five grid specimens that are prepared from a particular filter.

10.2 EVALUATING THE RATE OF RELEASE OF RESPIRABLE DUST

10.2.1 Determining the Mass of Respirable Dust on Filters to be Prepared for Asbestos Analysis

To support the determination of asbestos concentrations using this method, it is necessary to determine the mass of dust that is simultaneously deposited on the filter. This is because, as indicated in Section 2.2, asbestos concentrations are to be reported as the ratio of asbestos to dust (S/g_{PM10}).

For filters collected over the IST opening of the elutriator (or filters collected over the ME opening, if these are to be used for determination of asbestos concentrations using the optional indirect transfer technique described in Section 9.2), determine the dust mass on the filter by weighing the filter directly. As indicated in Section 8.5.5, a microbalance is required to determine the net weight of the PC filters collected over the IST opening because the mass of dust on such filters will not be more than 150 µg.

10.2.2 Optional Procedure for Determining the Mass of Respirable Dust in a Bulk Sample

Although not required in this modified elutriator method, an optional procedure is presented here for determining the mass of respirable material in bulk materials that are analyzed for asbestos content using this method. This procedure is modified from the one originally reported in Section 11.2 of the Berman and Kolk (1997) to account for the modifications to the configuration of the dust generator that are incorporated into this method.

10.2.2.1 Evaluating the rate of release of respirable dust. The rate of release of respirable dust from a sample prepared using the dust generator is estimated from measurements of the mass of dust collected over time on the set of filters mounted over the ME opening of the elutriator. The measurements used specifically are from those filters that are collected while the tumbler is operating at the *highest* rotation rate employed for the sample (see Section A.2.1 of Appendix A).

Begin by plotting the cumulative mass collected on the filters as a function of time. To derive the cumulative mass for a particular time interval, add the mass of dust measured on the filter collected from that time interval to the sum of the masses measured on the set of filters collected earlier in the run. Typical curves are depicted in Figures 11-1 and 11-2 of Berman and Kolk (1997). Next, calculate the cumulative mass released from the sample over time from the cumulative mass collected on filters over time using the relationship developed in Section A.2.1 of Appendix A:

$$M_r = 1.0523 * M_f \qquad (10\text{-}2)$$

where:

M_r is the cumulative mass of dust released from a sample between the start of a run and time "t" (g); and

M_f is the cumulative mass collected on filters between the start of a run and time "t" (g).

Equation 10-2 is appropriate to use to relate the mass of dust collected on filters to the mass released from the sample when air flow in the dust generator is setup as indicated in Section 8.4.3. If different air flow conditions are established for a particular experiment, the relationship between M_r and M_f will have to be derived using Equation A-4 from Appendix A.

The total mass of dust in the sample at the beginning of the run must next be estimated using the relationship developed in Appendix A. Based on the relationship (see Section A.2.1):

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad (10\text{-}3)$$

where:

M_o is the mass of dust in the sample at the start of the run (g);

k is the first-order rate constant for the release of dust from the sample (s^{-1}); and

t is the time since the start of the run (s).

A plot of $\ln(M_o - M_r)$ versus t should be a straight line with a slope equal to the rate constant for the release of dust from the sample and an intercept equal to the natural logarithm of the mass of dust in the sample at the start of the run. Derive estimates of "M_o" and "k" by programming Equation 10-3 into a spreadsheet and running a regression[15].

Input a range of guesses for the value of M_o into the spreadsheet and run a regression to fit a value for k and to calculate a value for the regression coefficient, "r^2" for each value of M_o. Plot the regression coefficient, "r^2" as a function of M_o. An example of such a plot is presented in Figure 11-3 of Berman and Kolk (1997). The value of M_o that provides the fit with the largest regression coefficient (i.e. with r^2 closest to 1) shall be reported as the correct value for the mass of dust in the sample at the start of the run and shall be reported with the corresponding k value as the estimated rate constant for dust release from the sample during the run.

10.2.2.2 Determining the content of respirable dust. To determine the mass percent of respirable dust in the original sample, first determine the total mass of respirable dust in the sample *at the start of a run* for the last run completed on the sample, which is derived as described in the last section.

If the dust generator run analyzed as described in Section 10.2.2 was preceded by a run with the tumbler speed set at 30 rpm, which will generally be the case when the goal is to determine the mass of respirable dust in the bulk sample (see Section 9.4.4 of Berman and Kolk 1997), to estimate the total mass of dust present in the sample, it is necessary to sum the masses collected on the ME filters during all of the earlier runs

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[15] Any of several commercial spreadsheet programs (including, for example, EXCEL™ or LOTUS™) contain the necessary capabilities and may be employed to derive optimum values for "M_o" and "k."

from the same sample, call this M_E. Then adjust this mass for the additional mass of dust that would simultaneously have passed through the IST opening of the elutriator:

$$M_{OE} = 1.053 * M_E \qquad (10\text{-}4)$$

where:

M_{OE} is the cumulative mass of respirable dust released from a sample during all runs completed prior to the current run (g); and

M_E is the cumulative mass of respirable dust measured on filters collected over the ME opening during all runs completed prior to the current run (g).

Calculate the total mass of dust originally present in the sample, M_{tot}, by summing the mass released during all earlier run(s) with the mas of dust estimated to have resided in the sample at the beginning of the final (usually higher rpm) run, M_o. M_o is equal to the mass of respirable dust remaining in the sample at the end of the earlier run(s). M_o will have been derived as described in Section 10.2.2.1:

$$M_{tot} = M_{rOE} + M_o \qquad (10\text{-}5)$$

where all parameters have been previously defined.

Finally, estimate the mass percent of respirable dust in the bulk sample as follows:

$$\%RD = 100 * M_{tot}/M_{sample} \qquad (10\text{-}6)$$

where:

%RD is the mass percent of respirable dust in the sample (%); and

M_{sample} is the mass of the original sample placed in the tumbler (g).

10.3 DETERMINING THE CONTENT OF ASBESTOS

As previously indicated (Section 2.2), the asbestos concentrations determined using this method are reported as the ratio of asbestos to dust (S/g_{PM10}). Also as previously indicated (Section 2.1), concentrations of protocol structures longer than 10 µm must be separately enumerated from structures between 5 and 10 µm in length. Concentrations for these two size categories shall be reported separately.

Whatever length (or other size) category of structures is to be determined, calculate concentrations as follows:

$$C_{dust} = S_c * A_f / (N_{go} * A_{go} * \Delta M_f) \qquad (10\text{-}7)$$

where:

C_{dust} is the concentration of asbestos per unit mass of respirable dust (S/g_{PM10});

S_c is the number of structures (of a defined size and type) that are counted during the scan (#);

A_f is the area of the filter on which the asbestos (and dust) were deposited for analysis (mm^2);

N_{go} is the total number of grid openings scanned during the analysis (#);

A_{go} is the area of a grid opening (mm^2); and

ΔM_f is the mass of respirable dust deposited on the filter.

11.0 QUALITY ASSURANCE QUALITY CONTROL REQUIREMENTS

The quality assurance/quality control (QA/QC) requirements indicated in the ISO Method (ISO 10312) shall be considered relevant and appropriate when using this method. In addition, the following blank and duplicate/replicate schedule shall be employed when running samples using this method.

11.1 BLANKS

The following blanks shall be collected routinely in concert with use of this method:

- *lot blanks or filter blanks*. Two filters from each lot of 50 filters obtained from the manufacturer shall be prepared using a direct transfer procedure and analyzed to assure that background contamination on the filters does not exceed 0.2 S/mm^2. Only filters from lots whose blanks pass the defined criterion shall be used in support of this method;

- *laboratory blanks*. A sufficient number of laboratory blanks shall be collected, prepared using a direct transfer technique and analyzed to show that the room in which bulk samples are handled and prepared satisfy the requirements defined in Section 10.6 of Chatfield and Berman (1990). When laboratory blanks indicate that room air is out of compliance with the stated criterion, use of this method is to cease until appropriate corrective actions are completed;

- *field blanks.* Field blanks shall be collected during any sample collection activities performed in association with use of this method. The number of such blanks to be collected and the schedule for their analysis shall be determined based on the complexity of the anticipated sampling scheme and shall be defined as part of the sampling plan for the site. QC criteria for field blanks will also be set as part of the planning for the study;

- *method blanks.* Method blanks should be collected and analyzed at a minimum frequency of one for every 20 samples analyzed using this method. The cumulative loading of asbestos structures observed on method blanks must not exceed 0.2 S/mm^2. Note that it may prove advantageous to collect method blanks more frequently than one every 20 samples. The additional blanks can then be stored and analyzed at a later date, if needed to troubleshoot for corrective action;

- *equipment blanks.* Equipment blanks and method blanks can be considered to be interchangeable. However, method blanks offer the advantage of simultaneously evaluating the laboratory's supply of washed sand. As long as no problem exists with the washed sand, run method blanks in lieu of running equipment blanks. However, should a problem develop with the washed sand, equipment blanks can be substituted for method blanks with no loss of method

performance (other than preventing the analysis of small volume samples for lack of appropriately qualified washed sand); and

- *conditioning filters.* Conditioning filters are collected at the beginning of every run. These filters do not need to be prepared and analyzed routinely but should be stored because they may prove useful for troubleshooting should corrective actions be required.

11.2 DUPLICATES AND REPLICATES

A fixed fraction (5 to 10%) of the samples collected in the field in support of this method shall be collected as spatial duplicates (two samples collected at immediately adjacent locations or two composites composed of an independent, inter-located set of samples representing the same area or volume). These shall be labeled and sent to the laboratory in such a manner so as to assure that laboratory personnel cannot identify them as duplicates. The frequency of collection of spatial duplicates shall be defined as part of the sampling plan for the site. Comparison of the results of the analysis of such samples provides a measure of all of the components of total precision except population variability.

As indicated previously (Section 8.2.1), 100% of the samples shipped from the field are to be shipped as duplicate pairs. The laboratory shall randomly select 2 or 3% of the duplicate samples shipped from the field and shall analyze both samples of the pairs so selected. Comparison of the results of the analysis of such samples, which are homogenized splits of the same sample, provides an indication of the precision achieved by sample preparation and analysis.

Should analysis of duplicate pairs indicate an unacceptable degree of variability (i.e. a relative percent difference greater than 50%), replicate counts shall be performed on designated samples by multiple analysts in the laboratory (or by the same analyst on different days). Laboratory management shall assign such counts so as to assure that analysts cannot determine which counts are replicates. Results of such replicate counts shall serve to distinguish whether the major source of variability observed among duplicate pairs is due to analysis or to sample preparation. Appropriate corrective actions may then be devised.

11.3 INTER-LABORATORY PERFORMANCE PROGRAMS

For large projects, it is highly recommended that analyses be performed by multiple laboratories (at least two) and that a well-crafted, formal inter-laboratory proficiency program be instituted to gauge the relative performance of the laboratories. In addition to the standard components of such a program (such as laboratory audits and consensus standards), such a program should include blind field replicates sent occasionally for comparison both between laboratories and within each laboratory.

12.0 REPORTING REQUIREMENTS

This chapter indicates field and laboratory reporting requirements.

12.1 FIELD AND LABORATORY NOTEBOOKS

Over the course of the project, information critical to the proper reporting and interpretation of each sample analysis will be developed both in the field and in the laboratory. Formal procedures are required to preserve such information and to allow for the documentation of attendant information that, while not employed directly in the calculation of results, may provide insight into the interpretation of such results.

Details of the reporting requirements for field and laboratory notebooks are provided in Section 13.1 of Berman and Kolk (1997).

12.2 FIELD ACTIVITIES REPORT

To assure that the field information required to complete estimation of dust and asbestos concentrations and release rates are provided to the data users, a field activities report must be completed and must be submitted to the laboratory along with the corresponding samples. Laboratory personnel are then to attach this report directly to their batch report, which shall cover the corresponding batch of samples.

Details of the reporting requirements for field activities reports are provided in Section 13.2 of Berman and Kolk (1997).

12.3 SAMPLE ANALYSIS REPORT

The sample analysis report for each sample shall include the following information, at a minimum:

(a) reference to this method;

(b) reference to the sample identification and batch number for the sample;

(c) the date and site from which the sample was collected;

(d) the weights and identities of the coarse and fine fractions of the sample and the sub-sample of the fine fraction sent for analysis;

(e) the weights and identities of any splits or other fractions of the sample generated during laboratory preparation;

(f) the weight and identity of the sub-sample placed in the dust generator;

(g) the relative flow rates through the IST and ME openings of the elutriator during each run of the dust generator for the sample;

(h) the measured mass of respirable material on each filter that is collected over the IST opening (or ME Opening) of the elutriator and prepared for asbestos analysis;

(I) if other than protocol structures, a statement identifying the structure size and type categories of interest for the study (see Chatfield 1993);

(j) a statement of the minimum acceptable identification category and the maximum identification category attempted for asbestos structures counted and characterized during analysis (see Chatfield 1993);

(k) a statement specifying which identification categories and which structure categories have been included in the counts of structures employed to estimate concentrations (see Chatfield 1993);

(l) the counts of asbestos protocol structures (separately, for total protocol structures and protocol structures longer than 10 µm and separately for chrysotile and amphibole structures);

(m) the active area of the filter prepared for asbestos analysis along with the average area of grid openings on specimen grids prepared from each filter and the number of grid openings scanned during analysis;

(n) the estimated concentration of asbestos protocol structures (separately, for total protocol structures and protocol structures longer than 10 µm and separately for chrysotile and amphibole structures). Concentrations are to be reported as S/g_{PM10};

(o) the analytical sensitivity achieved for a particular analysis; and

(p) 95% confidence limits for the concentrations reported.

An example of the format to be employed for a sample analysis report is presented in Figure 12-1.

FIGURE 12-1
SAMPLE ANALYSIS REPORT FORMAT

Laboratory Name
Laboratory Address
Laboratory Contact
Telephone Number

Report Date
Project Name (Optional)

METHODS:
(reference this method)

Date Analysis Started (M/D/Yr)
Date Analysis Completed (M/D/Yr)
Analyst(s) Initials

Laboratory Sample No.
Field Sub-Sample Identification No.
Field Preparation Technique (**Attach a Copy of the Relevant Field Activities Report**)
Additional Laboratory Preparation Procedures (*describe any employed*)
Sample Drying
Sample Splitting
Other

TEM Analysis:
Effective Area of Analytical Filter (sq mm)
(*Indicate whether from ME or from IST Opening*)
Magnification
Grid Opening Area (sq mm)
Number of G.O. Scanned
Asbestos Struc;ture Size and Type Categories of Interest (*see Chaffield 1993*)
Minimum Acceptable Structure Identification Category (*see Chaffield 1993*)

Dust Generator
Mass of Sample Tumbled (g)
Air Flow Rate Through ME Opening of Dust Generator (ml/min)
Air Flow Rate Through IST Opening of Dust Generator (ml/min)
Estimated Total Air Flow Rate Through Elutriator (ml/min)

Filters from the Isokinetic Sampling Tube (IST) Opening of the Elutriator
Mass of respirable dust on filter (g)

FIGURE 12-1
SAMPLE ANALYSIS REPORT FORMAT (Cont.)

Laboratory Name Report Date

Laboratory Sample No.

	No. of Protocol Structures	
	Total	Long (> 10 µm)
Asbestos Analysis Results[16]:		
No. of Chrysotile Asbestos Structures	XXX	XXX
No. of Amphibole Asbestos Structures	XXX	XXX
(Indicate Amphibole Mineral Type)		

ESTIMATED ASBESTOS CONCENTRATIONS (S/g_{PM10})

	Concentrations	
	Mean	95% UCL
Total Chrysotile Protocol Structures	XXX	XXX
Long Chrysotile Protocol Structures	XXX	XXX
Total Amphibole Protocol Structures	XXX	XXX
Long Amphibole Protocol Structures	XXX	XXX
Total Asbestos Protocol Structures	XXX	XXX
Long Asbestos Protocol Structures	XXX	XXX
Estimated Analytical Sensitivity: *(structures/g_{PM10})*	*XXX*	*XXX*

(Attach a Copy ot the TEM Raw Data Sheets)

[16] Protocol structure counts shall include all qualifying parent structures and qualifying components of complex structures (enumerated per the counting rules of the ISO Method)

13.0 REFERENCES

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APPENDIX A:
CONSTRUCTION AND OPERATION OF A DUST GENERATOR/ ELUTRIATOR FOR ISOLATING AND CONCENTRATING RESPIRABLE DUST AND ASBESTOS FROM SOILS AND OTHER BULK MATERIALS OF INTEREST

The dust generator/elutriator incorporated into this Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Materials is designed to isolate and concentrate respirable material and asbestos that is present within soils or bulk materials of interest. A detailed description of the device (including specifications and construction drawings) is provided in Berman and Kolk (1997) along with a discussion of the theory of operation for the device. Changes to the design of the original device that are required to support this modified method are described in Section 6.3. A brief description of the motivation for each of the changes is provided below, followed by a refined and abbreviated discussion of the theory of operation of the new device.

A.1 DUST GENERATOR MODIFICATIONS

A.1.1 Elimination of the Scrubber

The purpose of the scrubber in the initial design was to provide a slurry of respirable particles from which filters could be prepared for TEM examination using an indirect transfer procedure. Since it has now been shown that TEM analysis of samples prepared by direct transfer best correlate with risk (Berman and Crump 1999a), this modified method incorporates TEM specimen grid preparation by direct transfer, which obviates the need for the scrubber. In fact, even though an optional indirect transfer procedure is incorporated into this modified method, the preferred alternative is to use filters collected over the ME opening of the elutriator as the source of material for indirectly prepared samples, rather than rely on a scrubber. Thus, the scrubber has been removed from the modified design for the device (see Figure 6-3).

A.1.2 Elimination of the Secondary Air Source

In the original configuration of the device, a side inlet to the cup at the bottom of the elutriator was left open as a secondary source of air. This initially allowed air flow within the tumbler to be varied independent of flow in the elutriator. It was also thought that a source of air providing upward flow at the bottom of the elutriator would minimize the chance that respirable particles would be lost from the air stream from the tumbler by passing through quiescent sections of the flow regime at the bottom of the elutriator.

It was later discovered, however, that the laminar flow regime within the elutriator did not allow for adequate mixing of the two air streams (the one from the tumbler and the one from auxiliary entrance at the bottom of the elutriator). This was not a problem when TEM filters were being prepared by indirect transfer from scrubber material. However, it creates non-uniform deposits on filters collected over the IST opening of the elutriator, which limits their utility for analysis following direct transfer. Therefore,

the auxiliary opening at the bottom of the elutriator is sealed in the modified design. Moreover, the detailed design of the bottom of the elutriator has also been modified (see Section 6.3) to assure that flow velocities between the tumbler and the elutriator never fall below the minimum velocity in the main body of the elutriator. This assures that respirable material is not inadvertently lost, even in the absence of the auxiliary flow of air.

A.1.3 Reduction in the Size of the Exit Hole from the Tumbler

When the tumbler operates for a few hours with air constantly flowing in one direction, it was found that there is gradual movement of the mass of the material in the tumbler toward the exit hole. This is expected because, as the particles in the tumbler are tossed into the air stream, they are always pushed toward the exit, regardless of their size. Eventually, the level of the material in the tumbler at the end near the exit hole would rise to the point where some of the material would be forced out through the exit hole. This problem was minimized by reducing the diameter of the exit hole and extending the tube that serves as the exit hole a small distance into the tumbler so that it is no longer flush with the wall of the tumbler (see Figure 6-1).

A.1.4 Modifications to Humidity Control

As indicated in Berman and Kolk (1997), optimum generation of dust occurs at a relative humidity of approximately 50%. In the original method, the salt solution recommended for maintaining constant humidity contains potassium carbonate dihydrate, which actually provides control at a relative humidity of 43% at 20° C. Calcium nitrate tetrahydrate is recommended in the current method because, at equilibrium with a saturated solution, humidity is maintained at a value very close to 50%. It turns out that control of relative humidity that is closer to the ideal is more critical for this modified method to help mitigate the effects of static charging, which are much more pronounced on the required polycarbonate (PC) filters than on MCE filters. As indicated in Section 8.5.5, static electricity must be controlled to minimize problems during weighing of the PC filters on the microbalance.

A.2 THEORY OF OPERATION

A.2.1 The Dynamics of Dust Generation

The dynamics of the release of dust from a sample during a run using the dust generator have been evaluated so that the rate of release and mass of dust in the sample can be derived from measurements of the mass of dust deposited over time on the set of filters collected over the ME opening of the elutriator. Analysis of data obtained from several different types of samples during the pilot study for this method (Berman et al.1994) indicate that the rate of release of mass from a sample in the dust generator is well described by a first-order rate equation:

$$-dM_s/dt = k*M_s \qquad \text{(A-1)}$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" (g);

t is the time since the start of the run (s); and

k is the first-order rate constant for the release (s^{-1}).

The minus sign in this equation indicates that mass is lost with time.

Equation A-1 can be integrated to yield:

$$\ln(M_s) = \ln(M_o) - kt \qquad \text{(A-2)}$$

where:

M_o is the mass of respirable dust in the sample at the start of the run (i.e. at time t = 0) (g).

Given that "M_s" can also be expressed as the difference between "M_o" and "M_r" the cumulative mass released up to time "t," Equation A-2 can also be expressed as:

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad \text{(A-3)}$$

where:

M_r is the cumulative mass released between the start of a run and time "t" (g).

The relationship presented in Equation A-3 indicates that a plot of the natural logarithm of the quantity ($M_o - M_r$) versus time should be a straight line with a slope equal to the rate constant for dust release, k, and an intercept equal to the initial mass of dust in the sample at the start of the run, M_o. The cumulative mass of dust released from the sample over time, M_r, can be derived from measurements of dust collected on filters during the run. However, because M_o also appears as part of one of the parameters that must be plotted to evaluate the relationship expressed in Equation A-3, the value of M_o must be optimized using regression, as described in Section 10.2.2.1 of the main text of this method.

The cumulative mass released from a sample at time "t" during a run, "M_r" is directly proportional to the cumulative mass measured on filters collected during the run:

$$M_r = M_f*(F_d + F_c)/F_c \qquad \text{(A-4)}$$

where:

M_f is the cumulative mass measured on filters collected from filters mounted over the top of the elutriator up to time "t" (g);

F_d is the percent of airflow through the IST opening of the elutriator (%); and

F_c is the percent of airflow through the ME opening of the elutriator (%).

Because F_d plus F_c must sum to unity and F_d will typically have been set at 4.75% during the initial setup of the dust generator[17] (see Section 8.4.3.1), Equation A-4 reduces to:

$$M_r = 1.05*M_f \qquad \text{(A-5)}$$

As indicated above, values for M_o must be derived by performing a regression analysis of the relationship described by Equation A-3. This can be accomplished by using any of several commercially available spreadsheet programs (such as, for example, EXCEL™ or LOTUS™). The procedure to be followed to derive estimates of M_o and k are described in Section 10.2.2.1.

A.2.2 The Time Dependence of Dust Collection

As indicated in Section A.2.1 above, the generation of dust from the tumbler is well described by the first order rate equation:

$$-dM_s/dt = k*M_s \qquad \text{(A-1)}$$

However, experience gained during the pilot study for this method (Berman et al. 1994) further indicates that the rate of change of M_s is sufficiently slow in most cases such that, for periods of no more than 20 minutes, M_s can be considered constant. Thus, for estimating such things as the time required to load individual filters in the dust generator, a simpler form of Equation A-1 can be used (in which M_s is considered constant):

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[17] This assumes that the design specifications for the elutriator being used by the laboratory conform to those defined in Berman and Kolk 1997.

$$\Delta M_s = k*M_s*\Delta t = k'\Delta t \qquad \text{(A-6)}$$

where:

M_s is still the mass of respirable dust remaining in the sample at time "t" but it is assumed constant over the short interval of time "Δt" (g);

ΔM_s is the mass of respirable dust released from the sample over the short time interval "Δt" (g);

Δt is a relatively short time interval (no more than 20 minutes) during which the release of dust is being estimated (s);

k is still the first-order rate constant for the release (s^{-1}); and

k' is an empirically derived, linear rate constant that is appropriate for any particular, short time interval (s^{-1}).

Due to the geometry of the dust generator, the mass of respirable dust deposited on a filter in the dust generator in any short time interval, call this ΔM_f, is simply the product of the dust released from the sample, ΔM_s, and the fraction of the flow through the dust generator that is also directed through that filter. Thus, for filters collected over the isokinetic sampling tube (the IST opening of the elutriator):

$$\Delta M_{f\ (IST)} = 0.047*\Delta M_s = 0.047*k'*\Delta t \qquad \text{(A-7)}$$

Similarly, for the ME opening over the elutriator:

$$\Delta M_{f(ME)} = 0.953*\Delta M_s = 0.953*k'*\Delta t \qquad \text{(A-8)}$$

Comparison of Equations A-7 and A-8 shows that the k' constants for the two equations must be identical. Moreover, it is possible to estimate k' (without knowledge of M_s) from dust mass measurements made on filters collected over one opening of the elutriator, which can then be extrapolated to estimate the amount of time required to collect a defined mass of dust on filters collected over the other opening of the elutriator. Thus, by solving both Equations A-7 and A-8 for k' and substituting one into the other, the following relationship obtains:

$$\frac{\Delta M_{f(ME)}}{0.953\Delta t} = \frac{\Delta M_{f(IST)}}{0.047\Delta t} \qquad \text{(A-9)}$$

Combining terms and re-arranging yields the following relationship for determining the time required to collect a defined mass on a filter over the IST opening of the elutriator, given an estimated deposition rate that is defined based on dust mass measurements made on filters collected over the ME opening of the elutriator:

$$\frac{20 * \Delta M_{f(IST)}}{\left(\frac{\Delta M_{f(ME)}}{\Delta t}\right)} = \Delta t \qquad \text{(A-10)}$$

Where:

$\Delta M_{f(IST)}$ is a target mass of dust to be deposited on a filter mounted over the IST opening of the elutriator (g);

Δt is the time required to deposit a mass of dust equal to $\Delta M_{f(IST)}$ on a filter mounted over the IST opening of the elutriator (s);

$\left(\frac{\Delta M_{f(ME)}}{\Delta t}\right)$ is the rate of deposition of dust on a filter estimated as the ratio of the net dust deposited on a filter mounted over the ME opening of the elutriator to the net time required to deposit the observed mass (g/s).

A.2.3 Size Separation Using the Vertical Elutriator

The operation of the vertical elutriator has been described in detail in Section A.2.3 of the original Superfund Method (Berman and Kolk 1997).