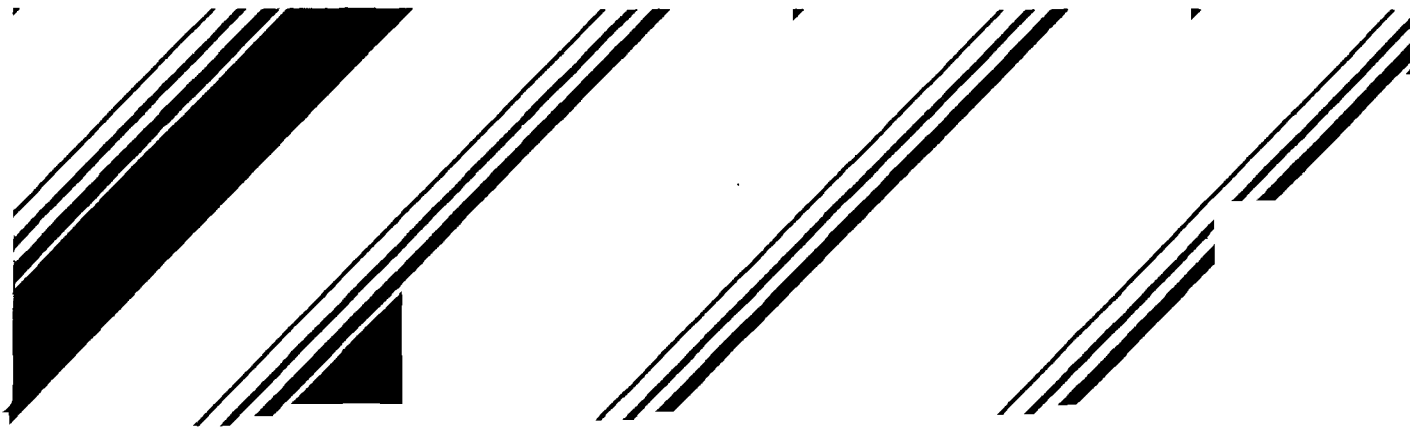


Toxic Substances



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Asbestos Content In Bulk Insulation Samples: Visual Estimates and Weight Composition



**EPA 560/5-88-011
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**ASBESTOS CONTENT IN BULK INSULATION SAMPLES:
Visual Estimates and Weight Composition**

By

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INTRODUCTION:

In April 1973, the U.S. Environmental Protection Agency (EPA) issued the National Emissions Standards for Hazardous Air Pollutants (NESHAP) for asbestos (38 FR 8820). The NESHAP regulation governs the removal, demolition, and disposal of asbestos-containing bulk wastes. An asbestos-containing product, as stated by the regulation, was defined for the first time to be a product with greater than 1% asbestos, by weight. The intent of the 1% limit was:

...to ban the use of materials which contain significant quantities of asbestos, but to allow the use of materials which would: (1) contain trace amounts of asbestos which occur in numerous natural substances, and (2) include very small quantities of asbestos (less than 1 percent) added to enhance the material's effectiveness. (38 FR 8821)

It must be clearly understood that the EPA NESHAP definition of 1% by weight was not established to be a health-based standard.

In May 1982, EPA issued a regulation which required schools to inspect and sample suspect friable surfacing materials for their asbestos content. EPA maintained consistency in its definition of an asbestos-containing material (ACM) by defining it as 1% by weight. At that time, the Agency investigated the available methodologies for measurement of asbestos fibers. The regulation included an interim methodology entitled "Interim Method for the Determination of Asbestos in Bulk Insulation Samples" (47 FR 23376). The polarized light microscope (PLM) protocol issued by the Agency was prepared by expert mineralogists and has been generally accepted by the analytical community as the appropriate analytical tool for measurement of asbestos content in bulk samples.

The interim method includes a description of its quantitation procedure. This procedure employs a technique called "point counting" to provide a determination of the area percent of asbestos in the sample. Based on a measurement made by point counting, the 1982 rule states "...reliable conversion of area percent to dry weight is not currently feasible unless the specific gravities and relative volumes of the material are known." EPA amended this statement in a correction to the regulation in September 1982 (47 FR 38535). EPA altered paragraph 1.7.2.4 of Appendix A of the rule by stating, "Paragraph 1.7.2.4 of Appendix A of the rule was intended to provide for a point counting procedure or an equivalent estimation method for determining the amount of asbestos in bulk samples." This

correction, acknowledged the practical and economic limitations of the point counting method and permitted the use of the visual estimation methodology. Visual estimation methodology is employed by most PLM laboratories and gives results which are very similar to a volume percentage.

In the following discussion, the validity of the assumptions that are made in extrapolating an area/volume percentage estimation to a weight percentage estimation of the asbestos content of insulation and other building materials will be examined. The reader should note that this discussion considers only the expected variation from the true weight percentage as is found when applying the visual estimate technique to determine the asbestos content in a bulk sample. The questions of laboratory/analyst variability of such visual estimations are not considered in this discussion.

RELATIONSHIPS BETWEEN AREA, VOLUME, AND WEIGHT PERCENTAGE

The principles of stereology are well documented (see, for example, "Quantitative Stereology," Underwood)¹ and will not be reiterated here other than to state that in classical stereology, with the assumption of a homogeneous distribution of phases within a solid, there is a direct relationship between the volume fraction of a phase present in the solid and the area fraction of that phase observed in a section taken through the solid.

That is to say,

$$\frac{V_p}{V} = \frac{A_p}{A}$$

where V_p refers to the volume of the phase p present in the total volume V , and A_p represents the area projection of that phase in a planar section of that solid of total area A . It should be noted that, for the classical rules of stereology to apply in a transmission sample, the section through the sample should be no thicker than the thickness or diameter of the smallest component.

The point counting method has been criticized as a technique for observing ACM because it does not take into consideration the fact that the asbestos fibers present may be comparatively thin in the Z direction relative to the other components present. Thus, if the volume percentage of asbestos present is extrapolated from the projected area obtained by the point counting technique, the volume percent of asbestos present will generally be

¹ Underwood, E.E., *Quantitative Stereology*, Addison-Wesley Publishing Company, (1970)

overestimated. As a result, the majority of laboratories analyzing ACM have adopted a visual estimate which allows a certain amount of latitude on the part of the microscopist to compensate for this thickness factor when examining samples on the microscope slide. In most instances, the visual estimation of asbestos content is made on a stereomicroscope with which the microscopist may more readily estimate the third dimension. Therefore, these estimates may be more readily extrapolated to a volume percentage than those from the point count method. This technique is essentially that which is proposed in the Interim American Society for Testing and Materials (ASTM) Method. Currently, this method is being considered for adoption by the National Institute of Standards and Technology (formerly the National Bureau of Standards) as part of its National Voluntary Laboratory Accreditation Program for the determination of bulk asbestos in samples. This procedure will provide a measurement of the asbestos in the sample which may be easily extrapolated to a volume measurement.

CURRENTLY ACCEPTED EXPERIMENTAL METHOD

The currently accepted and most generally used methodology for the identification of asbestos in building materials is compatible with both the EPA interim method and the proposed ASTM method. Identification of the asbestos type present using polarized light microscopy follows accepted mineralogical practices. The quantification of the asbestos content by visual estimation which is used is acceptable under the amendment to the 1982 Regulation published in the Federal Register and is substantially the same as that recommended in the ASTM method. It can be seen that there is continuity of approach and direct correlation between existing data and that which may be produced under the ASTM procedure.

While the visual estimation procedure is generally called the polarized light microscopy method, the microscopist, in fact, uses a combination of a low magnification stereomicroscope for preliminary examination and estimation of the percentage of each fiber type, followed by a detailed examination, using the polarized light microscope, of individual fibers removed from the bulk material. The procedure has been outlined in a draft to ASTM Committee D22.05 dated January 14, 1988—"Standard Method of Testing for Asbestos-Containing Materials by Polarized Light Microscopy."

The method calls for bulk samples of building materials to be first examined with a low power binocular microscope. By use of such a microscope, the following observations can be made.

- (1) The fibers can be detected.
- (2) The homogeneity of the material can be determined.
- (3) A preliminary identification of the fibers present can be made.
- (4) An estimate of fiber content by volume can be made.
- (5) Fibers may be separated from the matrix for more detailed analysis of subsamples with the polarized light microscope.

The method has been used, essentially in its present form, by the majority of the participants in the EPA Bulk Sample Analysis Round Robin program. These results indicate generally good reproducibility and good accuracy in assessing the volume percentage of an asbestos mineral present in an insulating material. The accuracy of such an analysis does not differ very greatly from the expected inhomogeneity (or homogeneity) of the material being analyzed (manufacturers' specifications generally show a range of composition for any one product which frequently was additionally modified at the point of application). In the ASTM technique, quantification of asbestos content is discussed in the following terms: "A quantitative estimate of the amount of asbestos present is most readily obtained by visual comparison of the bulk sample in slide preparations to other slide preparations and bulk samples with known amounts of asbestos present in them." The document goes on to state that estimates of the quantity of asbestos obtained by the method are neither volume nor weight percent estimates, but are based on estimating the projected area, from observation, of the distribution of particles over the two dimensional surface of the glass slide, and on an observation of bulk material, and that a basis for correcting to a weight or volume percent has not been established. It is this latter aspect which will be discussed more fully in this document. The ASTM method, however, provides for the percentage to be first assessed from the bulk material as observed on the stereomicroscope; it would seem, therefore, that this percentage is a closer approximation to a volume percentage rather than a projected area one. In addition the ASTM document states, "However, the error introduced by assuming that the estimates are equivalent to weight percent is probably within the precision of the visual estimate technique."

CORRELATION OF WEIGHT PERCENTAGE WITH VOLUME PERCENTAGE

To correlate the weight fraction of the phase to its area or volume fraction, it is necessary, as is pointed out in the The EPA Test Method, that the specific gravities and relative volume fractions of all the phases present in the material are known.¹

In any multicomponent system consisting of n components, the weight percent of component i is given by the following formula:

$$\frac{P_i \times V_i \times 100}{\sum_{i=1}^{i=n} P_i \times V_i} \quad (1)$$

where P_i is the specific gravity of the i th component and V_i is the volume of the i th component. From this formula, it is clear that if the volume percent and the density of each individual element in a bulk insulation sample is known, it would be possible to obtain a weight percentage for any particular component and specifically for those components which are classed as asbestos. To determine this information experimentally would, however, be extremely time consuming, requiring the separate identification of each component in the matrix, determining its specific gravity from reference tables, and applying these factors in the formula.

An alternative conversion is therefore suggested in which an average density is assumed for the nonasbestos matrix. In this model, the weight percentage, W_a , of a particular asbestos type present at a volume percentage of V_a and having a density of P_a present in a matrix of density P_m is given by the formula

$$W_a = \frac{P_a \times V_a \times 100}{(100 - V_a) \times P_m + (V_a \times P_a)} \quad (2)$$

The density value ascribed to the nonasbestos matrix should be selected taking into consideration the major constituents of the matrix but, for a large range of commonly encountered inorganic matrices, a value of 2.5 g/cm^3 may be assumed..

¹ Interim Method for The Determination of Asbestos in Bulk Insulation Samples
EPA 600/MA-82-020, December, 1982.

PRACTICAL APPLICATION

These formulas will be applied to a range of samples. In applying formula 1 to determine actual weight percentages, published values for the several components were used. To determine the weight percentages using the model described by formula 2, a matrix density of 2.5 g/cm³ was assumed.

Sample 1 Acoustical Material

Sample 1 is a sample of an acoustical material taken from an actual ceiling treatment.

<u>Component</u>	<u>Vol%</u>	<u>Wt% (Actual)</u>	<u>Wt% (Model)</u>
Chrysotile	15.0	15.12	15.51
Glass Fiber	60.0	60.47	
Carbonate	10.0	10.85	
Cement	3.0	3.26	
Clay	10.0	8.53	
Gypsum	2.0	1.78	

(Appendix 1 shows in detail how these weight percentages are calculated.)

Sample 2 Round Robin Sample from Independent QC Ring

Sample 2 is from an independent round robin sample series in which four laboratories participated. Reported values for amosite content were 30%, 30-40%, 45%, and 15-20%. The results from the second laboratory were taken using the midpoint of the reported compositional range (the midpoint of the reported range for sample two was selected as most probably representing the actual composition, lying between the reported values of one and three, with four regarded as an outlier).

<u>Component</u>	<u>Vol%</u>	<u>Wt% (Actual)</u>	<u>Wt% (Model)</u>
Amosite	35.0	38.82	41.55
Carbonate	35.0	32.94	
Cement	30.0	28.24	

Sample 3 Sample A EPA Bulk Sample Analysis Round Robin No. 16

Sample 3 is sample A from the EPA Bulk Sample Analysis Round Robin series, Round number 16.

<u>Component</u>	<u>Vol%¹</u>	<u>Wt% (Actual)</u>	<u>Wt% (Model)</u>
Amosite	3.0	4.04	3.92
Glass	87.0	92.29	
Cellulose	10.0	3.67	

¹ Volume percentage data for samples 3, 4, 5 and 6 are averages taken from EPA Round Robin reports and would not normally be reported to this level of significance.

Sample 4 Sample D EPA Bulk Sample Analysis Round Robin No. 16

Sample 4 is Sample D from the EPA Bulk Sample Analysis Round Robin series, Round Number 16.

<u>Component</u>	<u>Vol%</u>	<u>Wt% (Actual)</u>	<u>Wt% (Model)</u>
Chrysotile	3.0	3.53	3.12
Clay	97.0	96.47	

Sample 5 Sample D EPA Bulk Sample Analysis Round Robin No. 17

Sample 5 is Sample D from the EPA Bulk Sample Analysis Round Robin series, Round Number 17.

<u>Component</u>	<u>Vol%</u>	<u>Wt% (Actual)</u>	<u>Wt% (Model)</u>
Chrysotile	2.9	2.56	3.01
Amosite	30.7	34.40	36.90
Cement	66.3	63.04	

Sample 6 Sample A EPA Bulk Sample Analysis Round Robin No. 17

Sample 6 is Sample A from the EPA Bulk Sample Analysis Round Robin series, Round Number 17.

<u>Component</u>	<u>Vol%</u>	<u>Wt% (Actual)</u>	<u>Wt% (Model)</u>
Crocidolite	97.0	97.52	97.78
Cement	3.0	2.48	

It is clear from these data that, for most samples, the weight percentage of the asbestos content is not substantially different from the volume percentage which is normally reported and is within the expected variation both of the analytical procedure and the sample homogeneity. A close estimate of the weight percentage can be derived from a simple model which assumes an average matrix density of 2.5 g/cm³.

Plots of the difference between observed volume percentage and calculated weight percentage for chrysotile, density 2.6 g/cm³, (Figure 1) and crocidolite, density 3.4 g/cm³, (Figure 2) are shown calculated using this model. The maximum deviation between the numerical values of weight and volume percentage occurs near the 50% mark and, in the worst case (crocidolite), is less than 10%.

Exceptions will be found in samples whose matrices have significantly higher or lower densities than the asbestos observed. Figure 3 presents the extreme case of crocidolite (density 3.4 g/cm³) in a matrix of cellulose with an estimated average density of 0.9 g/cm³.

The magnitude of the discrepancy in the critical region near 1% is shown in figure 4. If only the volume percentage estimate is used, mass percentages as high as 3% would be reported as below the definition of ACM. In this case, a conversion to weight percentage is necessary if the weight percentage is not to be grossly underestimated.

SAMPLE TREATMENT

Some samples, for example floor tiles, roofing felts, and some cementitious products, may require special treatment (ashing, solvent or acid extraction) to separate the asbestos from other materials in order to facilitate analysis. In such cases, the resulting weight loss of the sample due to treatment must be recorded and any volume to weight percentage correction applied to the remaining material must be further corrected to take this weight loss into consideration. For example, if 30% asbestos is detected in a sample after processing which resulted in a 25% weight loss, then the corrected asbestos content is $0.75 \times 30 = 22.5\%$

CONCLUSIONS AND RECOMMENDATIONS

An assessment has been made of the validity of extrapolating to a weight percentage the area or volume percentage of asbestos present in a sample as determined by polarized light microscopy. A model has been presented which can be applied to area or volume percentage data to give a more accurate estimation of the weight percentage. *With the exception of asbestos-containing materials having a substantial density differential between matrix and asbestos, generally low density cellulosic or perlitic matrices, the magnitude of this correction is smaller than the expected variability imposed by both the analytical variation and the inhomogeneity of the sample. As a result, the weight percentage of asbestos present can generally be equated with the observed area or volume percentage.*

The following recommendations are made:

- 1) For samples whose approximate average matrix density is close to that of the asbestos species observed (within 0.5 g/cm^3), assume equivalence of weight and area or volume percentage.
- 2) For samples whose approximate average matrix density differs from that of the asbestos species present by more than 0.5 g/cm^3 , convert the observed area or volume percentage to weight percentage using formula 2, using a matrix density consistent with the principal matrix components.

TABLE I

Calculated relationship between weight percentage and volume percentage of chrysotile (density 2.6 g/cm³) in matrix of average density of 2.5 g/cm³.

VOLUME %	WEIGHT %	DIFFERENTIAL (WEIGHT%-VOLUME %)
0	0.00	0.00
5	5.17	0.19
10	10.36	0.36
15	15.51	0.51
20	20.63	0.63
25	25.74	0.74
30	30.83	0.83
35	35.90	0.90
40	40.94	0.94
45	45.97	0.97
50	50.98	0.98
55	55.97	0.97
60	60.94	0.94
65	65.89	0.89
70	70.82	0.82
75	75.73	0.73
80	80.62	0.62
85	85.49	0.49
90	90.35	0.35
95	95.18	0.18
100	100.00	00.00

These values were used to produce Figure 1.

TABLE II

Calculated relationship between weight percentage and volume percentage of crocidolite (density 3.4 g/cm³) in a matrix of average density 2.5 g/cm³.

VOLUME %	WEIGHT %	DIFFERENTIAL WEIGHT %-VOLUME %
0	0.00	0.00
5	6.68	1.68
10	13.13	3.13
15	19.35	4.35
20	25.37	5.37
25	31.19	6.19
30	36.82	6.82
35	42.27	7.27
40	47.55	7.55
45	52.67	7.67
50	57.63	7.63
55	62.44	7.44
60	67.11	7.11
65	71.64	6.64
70	76.04	6.04
75	80.31	5.31
80	84.47	4.47
85	88.51	3.51
90	92.45	2.40
95	96.27	1.27
100	100.00	0.00

These values were plotted to produce the curve of Figure 2.

TABLE III

Calculated relationship between weight percentage and volume percentage of crocidolite (density 3.4 g/cm³) in a matrix of average density 0.9 g/cm³.

VOLUME %	WEIGHT %	DIFFERENTIAL WEIGHT %-VOLUME%
0	0.00	0.00
5	16.59	11.59
10	29.57	19.57
15	40.00	25.00
20	48.57	28.57
25	55.74	30.74
30	61.82	31.82
35	67.04	32.04
40	71.58	31.58
45	75.56	30.56
50	79.07	29.07
55	82.20	27.20
60	85.00	25.00
65	87.52	22.52
70	89.81	19.81
75	91.89	16.89
80	93.79	13.79
85	95.54	10.54
90	97.14	7.14
95	98.63	3.63
100	100.00	0.00

These values were plotted to produce the curve of Figure 3.

TABLE IV

Calculated relationship between weight percentage and volume percentage of crocidolite (density 3.4 g/cm³) in a matrix of average density 0.9 g/cm³ over the range 0 to 2 volume%.

VOLUME %	WEIGHT %	DIFFERENTIAL WEIGHT %-VOLUME%
0.0	0.00	0.00
0.1	0.38	0.28
0.2	0.75	0.55
0.3	1.12	0.82
0.4	1.49	1.09
0.5	1.86	1.36
0.6	2.23	1.63
0.7	2.59	1.89
0.8	2.96	2.16
0.9	3.32	2.42
1.0	3.68	2.68
1.1	4.03	2.93
1.2	4.39	3.19
1.3	4.74	3.44
1.4	5.09	3.69
1.5	5.44	3.94
1.6	5.79	4.19
1.7	6.13	4.43
1.8	6.48	4.68
1.9	6.82	4.92
2.0	7.16	5.16

These values were plotted to produce the curve of Figure 4.

**Mass - Volume Percent Differential
Chrysotile In Matrix Of S.G. = 2.5**

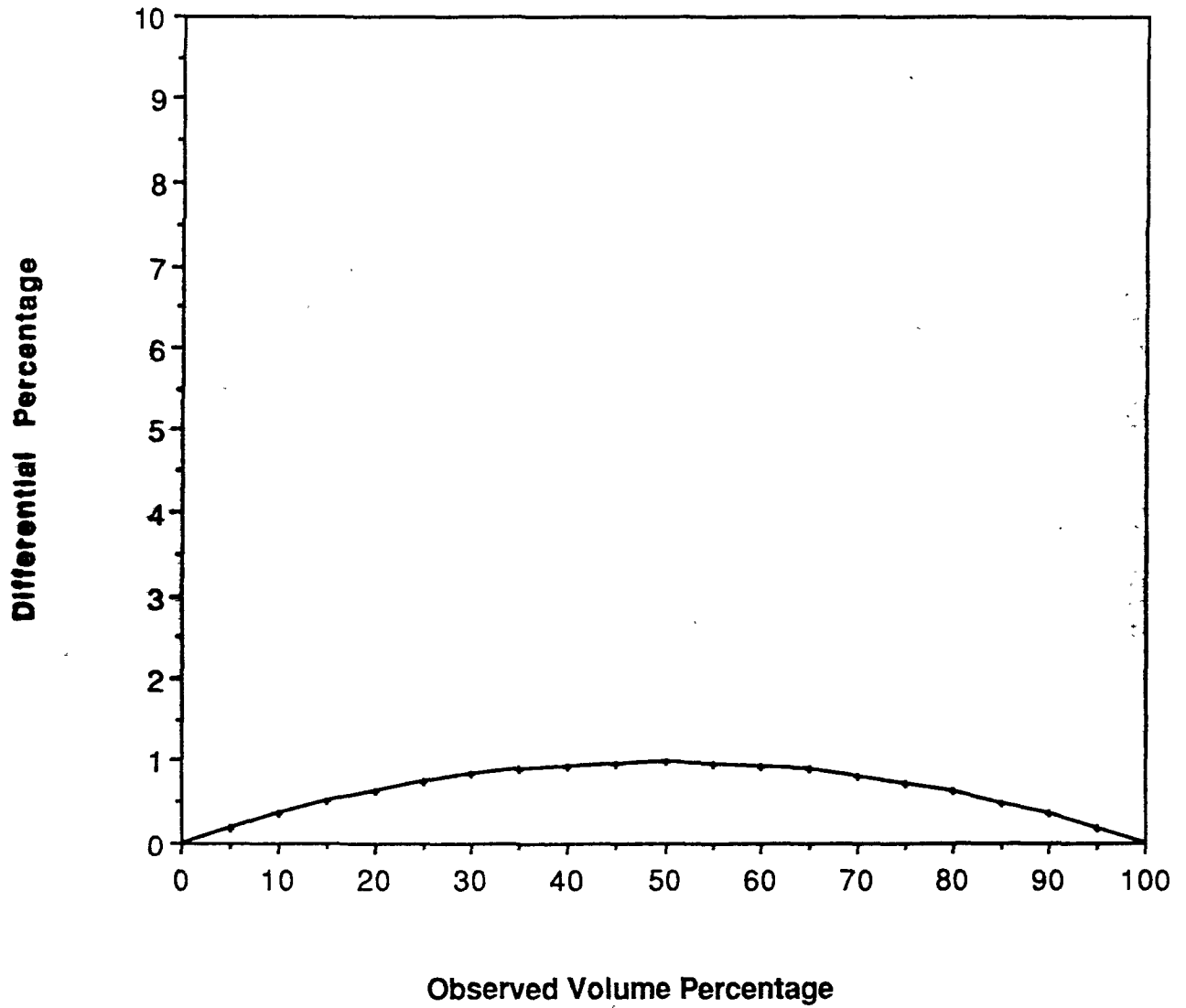


Figure 1.

**Mass - Volume Percent Differential
Crocidolite In Matrix Of S.G. = 2.5**

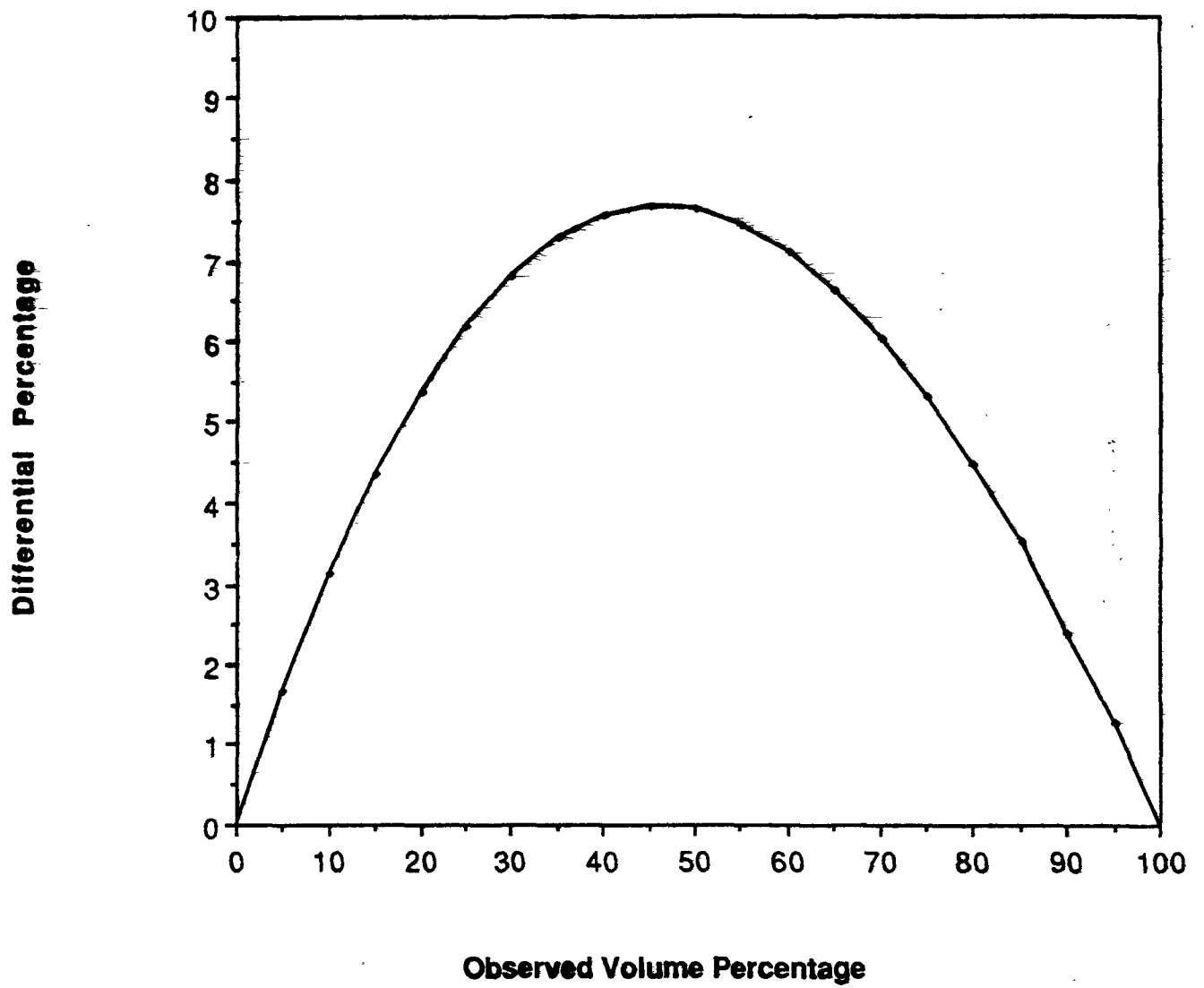


Figure 2.

**Mass - Volume Percentage Differential
Crocidolite In Matrix Of S.G. = 0.9**

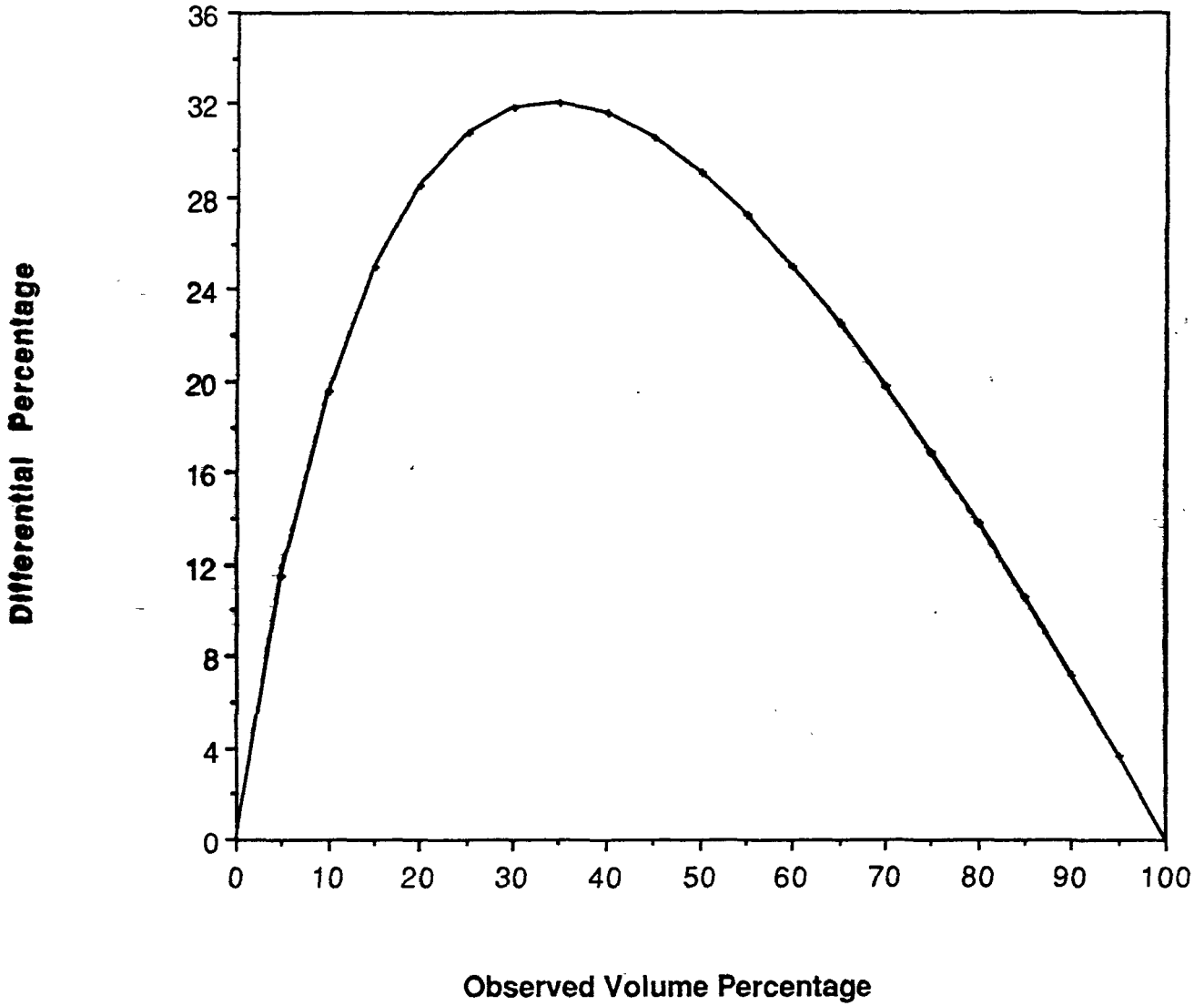


Figure 3.

Crocidolite In Cellulose Matrix

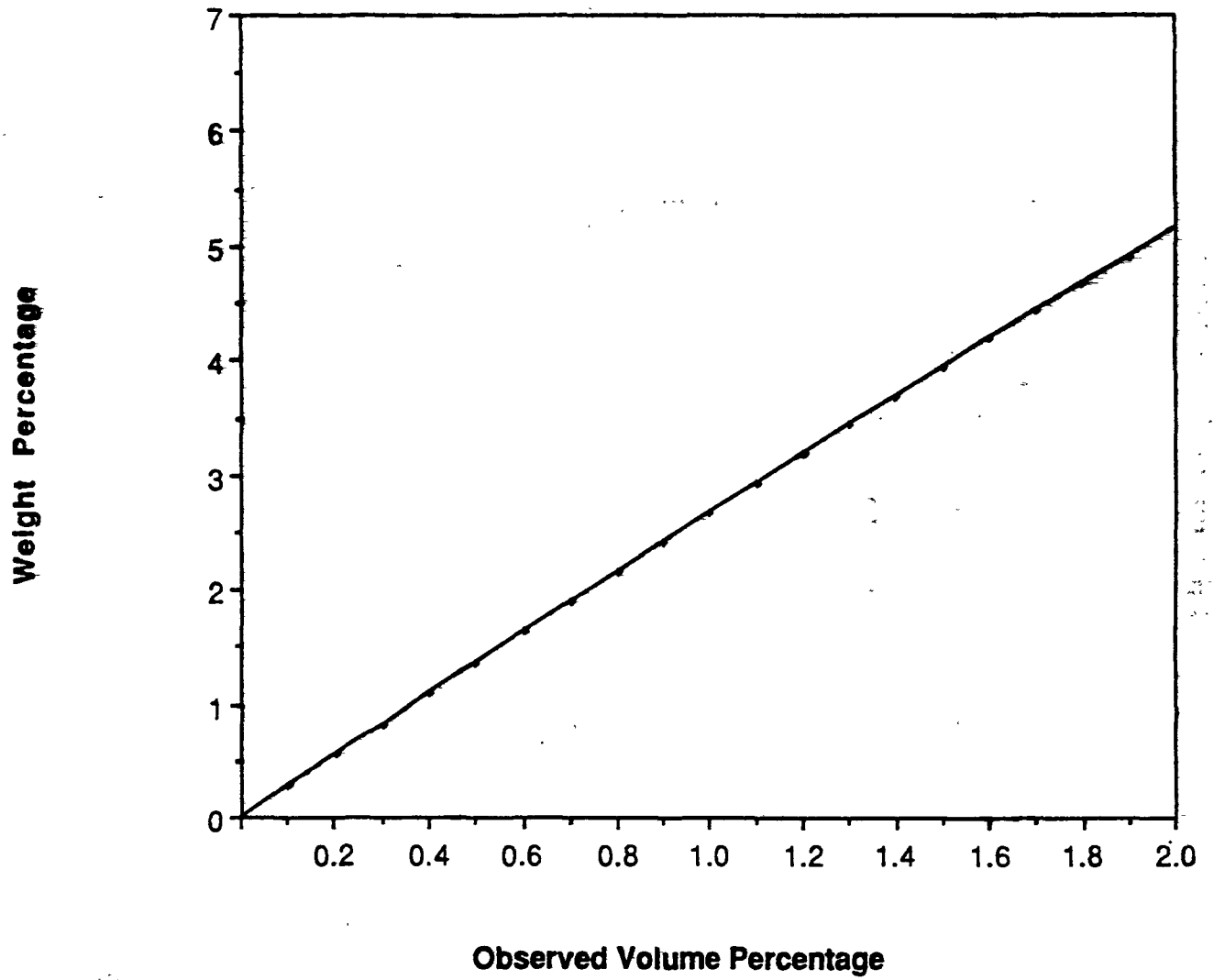


Figure 4.

APPENDIX I

Examples of Calculations

a) Actual Weight Percentages

Sample #1

Compound	Vol. %	Density (g/cm ³)	Relative Weight	$\frac{\text{Wt. \%}}{\text{Total Rel. Wt.}} \times 100$
Chrysotile	15.0	2.6	15 x 2.6 = 39.0	15.12
Glass Fiber	60.0	2.6	60 x 2.6 = 156.0	60.47
Carbonate	10.0	2.8	10 x 2.8 = 28.0	10.85
Cement	3.0	2.8	3.0 x 2.8 = 8.4	3.26
Clay	10.0	2.2	10.0 x 2.2 = 22.0	8.53
Gypsum	2.0	2.3	2.0 x 2.3 = 4.6	1.78
TOTALS	100.0	--	258	100.01

b) Approximate weight percentages based on a model with assumed 2.5 g/cm³ density.

Sample #1

	Vol. %	Rel. Wt.	Approx. Wt. %
Chrysotile	15.0	15 x 2.6 = 39.0	15.51
Non-asbestos matrix	85.0	85 x 2.5 = 212.5	
TOTALS	100.0	251.5	

Sample #5

Sample 5 contains both chrysotile and amosite. The approximate weight percentage is calculated separately for each asbestos type as follows:

	Vol. %	Rel. Wt.	Approx. Wt. %
Chrysotile (density 2.6 g/cm ³)	2.9	2.9 x 2.6 = 7.54	3.01
Non-chrysotile matrix	97.1	94.1 x 2.5 = 242.75	
Chrysotile totals	100.0	250.29	
Amosite (density 3.3 g/cm ³)	30.7	101.31	36.90
Non-chrysotile matrix	69.3	173.25	
Amosite totals	100.0	274.56	

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