NISTIR 5358

Airborne Asbestos Method: Standard Practice for Recording Transmission Electron Microscopy Data for the Analysis of Asbestos Collected onto Filters Version 1.0

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and Technology
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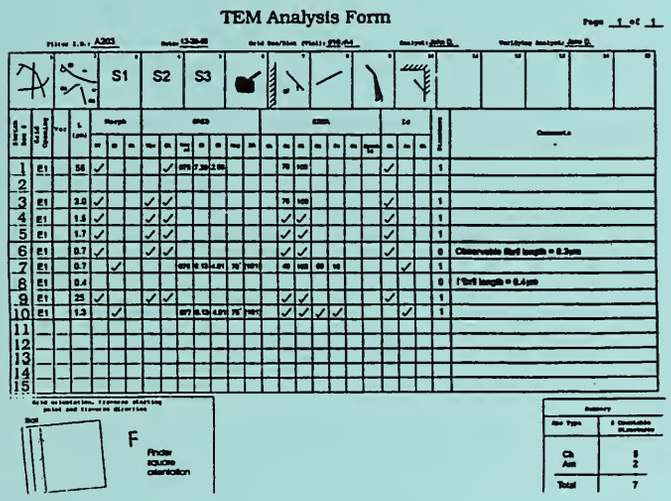
TEM Analysis Form

NIST

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U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Arati Prabhakar, Director

Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

Acknowledgment

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1. **Scope**

1.1 This standard practice describes the procedure for recording information obtained during a Transmission Electron Microscope (TEM) analyses of asbestos. The TEM Analysis Form serves as a work sheet for the analyst.

1.2 Forms completed according to this practice allow for the verification of asbestos structures determined by two or more analysts (standard method 2.1.2)

2. **Referenced Documents**

2.1 Standard Methods:

2.1.1 Turner, S. and Steel, E. B., Airborne Asbestos Method: Standard Test Method for High-Precision Counting of Asbestos Collected onto Filters - Version 1.0. NISTIR, in press, 1994.

2.1.2 Turner, S. and Steel, E.B., Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0. NISTIR, in press, 1994.

3. **Terminology**

3.1 *Definitions*

3.1.1 *TEM*--transmission electron microscope

3.1.2 *Particle*--an isolated collection of material deposited on a grid or filter. Carbon substrate and sample preparation artifacts must be ignored in identifying a particle.

3.1.3 *Aspect ratio*--ratio of fibril length to fibril width

3.1.4 *Fibril*--a single crystal of a regulated (asbestos) mineral that has an aspect ratio greater than or equal to 5:1.

3.1.5 *Structure*--a fibril or larger particle containing at least one fibril that can be observed to have a length greater than 0.5 μm.

3.1.6 *Grid*--a substrate, containing holes that are commonly squares, used for the deposition of material to be analyzed by TEM.

3.1.7 *Grid opening, grid square*--an area on the grid used for the analysis of asbestos by TEM

3.1.8 *Verifying analyst*--a person that compares the analyses of a grid square by two or more TEM analysts.

3.2 *Description of Terms Specific to This Standard*

3.2.1 *Sketch box*--location on the TEM Analysis Form where structures (or possible structures) are sketched.

3.2.2 *Ver (Verification)*--verification of structures reported on the TEM Analysis Form.

3.2.3 *L (Length)*--maximum linear dimension of a structure

3.2.4 *Morph (Morphology)*--form or visible structure of fibril(s)

3.2.5 *HT (Hollow Tube)*--a cylindrically shaped fibril that has an open axial core.

3.2.6 *PS (Parallel Sided or Prismatic)*--fibrils commonly have a blocky or prismatic habit consisting of crystals with parallel sides (faces).

3.2.7 *Selected area electron diffraction (SAED)*--a technique used in transmitted electron microscopy to view the electron diffraction pattern.

3.2.8 *Vis (Visual)*--a visual examination of the electron diffraction pattern

3.2.9 *St (Streaking)*--smearing or streaking of the electron diffraction spots into linear features

3.2.10 *Rec Id (Recording Identification)*--label used to identify uniquely a recorded image of a diffraction pattern.

3.2.11 *D1 and D2*--d-spacings of two unique diffraction maxima in a diffraction pattern

3.2.12 *Ang (Angle)*--the angle between the two vectors connecting the central spot to the reflections used to determine D1 and D2.

3.2.13 *ZA*--zone axis of the diffraction pattern

3.2.14 *EDXA (Energy dispersive x-ray analysis)*--a technique used to determine the chemical elements present in a particle.

3.2.15 *Spect Id (Spectrum Identification)*--the identification given to the energy dispersive x-ray spectrum.

3.2.16 *TEM Analysis Form*--the form shown in Figure 1

NOTE 1--For more detailed discussion or definitions concerning electron microscopy and electron diffraction, see the references listed in section 8.

4. **Summary of Practice**

4.1 All analysts performing an asbestos analysis orient and traverse the grid opening in the same manner.

4.2 Asbestos containing particles are sketched with enough detail so that other analysts can locate these particles on the referenced grid square.

4.3 Analytical information is recorded on the TEM Analysis Form (Figure 1).

5. **Significance and Use**

5.1 The practice provides a record of an asbestos analysis. A form is completed by each analyst performing the analysis.

5.2 The TEM Analysis Form is intended for use with the TEM methods for asbestos analysis developed by NIST (standard methods listed in sections 2.1.1 and 2.1.2)

5.3 The standard is designed to give a uniform recording format so that analytical data may be compared among laboratories. This standard may be particularly useful for interlaboratory comparisons, proficiency testing, and regulatory methods where comparable data recording is desired.

6. **Use of the TEM Analysis Form**

NOTE 2--An example of how to fill out the TEM Analysis Form is given in figure 2.

6.1 Record the header information at the top of the form. This section includes details concerning the analysis that do not include analytical data.

6.1.1 Filter Id: Record the identification of the original filter from which the TEM grids were made.

6.1.2 Date: The date the TEM analysis is performed.

6.1.3 Grid box/slot (vial): Record the label of the grid box and the slot from which the grid is taken. If the grid is stored in a vial rather than a grid box, record the vial identification.

6.1.4 Analyst: The name or initials of the person performing the TEM asbestos analysis.

6.1.5 Verifying Analyst: The name or initials of the person performing the verified analysis. This information is filled out only if a verified analysis is performed.

6.2. Record the analytical information in the table located in the central portion of Figure 1 according to the following directions:

6.2.1 Sketch boxes: The 15 boxes located at the top of the table are the sketch boxes. Analysts use these boxes to sketch each possible structure encountered while traversing the grid square. The first structure (or suspected structure) encountered is sketched in box #1, the second in box

#2, etc., until the entire grid square has been traversed.

6.2.1.1 The sketch should contain enough detail to allow the structure to be located by other analysts. Features located close to the structure are included in the sketch. These features may consist of grid bars, other particles, and tears, holes or other imperfections on the carbon replica.

6.2.1.2 Often during a traverse, analysts encounter several structures very close to one another. When this occurs, the structures should all be sketched in the same box and labeled as structure 1 (s1), structure 2 (s2), structure 3 (s3), etc. Sketch boxes immediately following ones containing multiple structures should contain the label of one of the multiple structures (s1 for example). An example of this is found in sketch boxes #2 - #5 on the example TEM Analysis Form (figure #2). Here three structures occur in close proximity and are therefore sketched in the same box (box #2). The structures are labeled s1, s2, and s3. The following 3 boxes (#3, #4 and #5) are filled with the designations s1, s2 and s3, respectively.

6.2.1.3 Recorded images, such as electron micrographs or electronic images, may be used in place of the sketches. If these are used, then a label that uniquely identifies the image is placed in the sketch box.

6.2.2 Sketch Box #: This column contains the numbers 1 through 15. These numbers correlate each row in the TEM Analysis Form with its corresponding sketch box above. Each row in the TEM Analysis Form therefore contains only the analysis of the structure that is sketched or listed in its corresponding sketch box. The only exception occurs when there are multiple structures sketched in one box. Then, the row corresponding to this sketch box is left blank (see row 2 on the example TEM Analysis Form; Figure 2).

6.2.3 Grid Opening: The identification of the grid opening is recorded for each possible structure being analyzed. The grid opening label must unambiguously identify the grid square that is being traversed.

6.2.4 Ver (Verification): This column is left blank during the asbestos analysis. If a verified analysis is performed, then this column should be filled out with one of the designations listed in the Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy (reference item 2.1.2).

6.2.5 L (Length): Record the maximum dimension of the asbestos containing particle. This length measurement is used as a scale factor for the structure in the sketch box.

NOTE 3-- This recorded length may not be the length of the largest observable fibril. As an example see the analysis for the particle in sketch box #6. Here the length of the largest observable fibril (0.3 μm) is less than the length of the asbestos containing particle (0.7 μm). Often, uncertainty may arise as to whether a particle containing asbestos should be recorded as a structure. This frequently happens when the largest observable fibril has a length close to 0.5 μm. Under these conditions, the analyst should measure the longest observable fibril as accurately as possible and record it along with any pertinent discussion in the Comments column.

6.2.6 Morph (Morphology): Place a check mark in the column(s) that describe the morphology of the fibrils.

6.2.6.1 HT (Hollow Tube): Fibrils of chrysotile asbestos often have a hollow tube morphology. If this morphology is observed then a check mark should be placed in this column.

6.2.6.2 PS (Parallel Sided, Prismatic): Amphibole asbestos generally contains prismatic or long parallel sided fibrils. Place a check mark under this column when this morphology is observed.

6.2.6.3 Ot (Other): Place a check mark in this column if the asbestos fibrils have neither hollow tube nor prismatic morphology. If a check mark is placed in this column then the fibril morphology should be described under the Comments column.

6.2.7 SAED (Selected Area Electron Diffraction): When selected area electron diffraction is performed, the analyst may not see a diffraction pattern. Note in the Comments column if no pattern is observed or if the pattern is of poor quality and can not be interpreted.

6.2.7.1 Vis (Visual): Place a check mark in this column when only a visual examination or verification of an electron diffraction pattern is performed.

6.2.7.2 St (Streaking): This column refers to the streaking of the (110) and the (130) reflections in the electron diffraction pattern of chrysotile asbestos. When this streaking is observed, place a check mark in this column. If streaking of any other reflections in an asbestos diffraction pattern are noteworthy, list the streaked reflections and describe the streaking under the Comments column.

6.2.7.3 Rec Id (Recording Identification): An image of the electron diffraction pattern may be recorded. The identification assigned to this image is recorded in this column.

6.2.7.4 D1 and D2 (D-spacings): Record the d-spacings determined from electron diffraction maxima. These d-spacing values should be recorded in nanometers (nm).

6.2.7.5 Ang (Angle): Measure and record the angle between the two vectors connecting the central spot with the spots used to determine D1 and D2 above.

6.2.7.6 ZA (Zone Axis): If determined, record the zone axis of the diffraction pattern.

6.2.8 EDXA (Energy Dispersive X-ray Analysis): The columns under EDXA list the major chemical elements that may be present in asbestos. The following columns should be filled out with either a check mark to indicate their presence in the spectrum or with an intensity ratio determined relative to silicon.

6.2.8.1 Na (Sodium)

6.2.8.2 Mg (Magnesium)

6.2.8.3 Si (Silicon)

6.2.8.4 Ca (Calcium)

6.2.8.5 Fe (Iron)

6.2.8.6 Ot (Other): Other major elements identified in the spectrum. If other major elements are identified, place a check mark in this column and record the other elements (along with concentration ratios, if necessary) in the Comments column.

6.2.8.7 Spect Id (Spectrum Identification): If the EDXA spectrum is saved for future reference then its identification is placed in this column.

6.2.9 Id (Identification): The analyzed particle should be identified in this column.

6.2.9.1 Ch (Chrysotile Asbestos): Mark this column if the analyzed particle is determined to be chrysotile.

6.2.9.2 Am (Amphibole): Mark this column if the analyzed particle is determined to be an amphibole. This column includes all the EPA regulated asbestos minerals other than chrysotile.

6.2.9.3 Ot (Other): Mark this column when an analyzed particle is determined not to be a regulated asbestos mineral. If the identity of the particle is determined, record it in the Comments column.

6.2.10 Structure: Place a 1 in this column if the particle has been identified as chrysotile or amphibole asbestos and also meets the dimensional requirements for a structure. If the analyst determines that the particle analyzed is not a structure then a 0 should be recorded in this column.

6.2.11 Comments: This column contains any comments the analyst may have about the particle being analyzed. Also, if a check mark has been placed in any column labeled Ot (other) then this should be explained in the Comments column.

6.3 Record the grid orientation and traverse direction

6.3.1 The box at the lower left corner of the TEM Analysis Form is for recording grid

orientation, traverse starting point and traverse direction. It is important that all operators analyzing a grid square orient and traverse the grid square in the same manner. This allows reported structures to be matched by a verifying analyst. An example of how to complete this box is recorded in figure #2. An asymmetric indicator is used to define the orientation of the grid at the time of the analysis. In the example, the grid orientation is recorded by drawing (in the orientation viewed on the TEM) the indexed grid letter "F". Also, the analyst has drawn the grid square and indicates that the traverse began at the upper left hand corner of the square. The traverse direction is indicated by arrows drawn on the grid square.

6.4 Record the summary of the analysis in the summary box.

6.4.1 The summary box is located in the lower right corner of the TEM Analysis Form. The analyst uses the summary box to record the number of structures for each asbestos type identified during the analysis. In addition the total number of structures is determined and recorded.

6.5 Customizing the form

6.5.1 The bottom center of the form is left blank for laboratories to add information that is useful to their particular laboratory practice.

7. **Key words**

7.1 analysis form; asbestos; transmission electron microscopy; verified analysis

8. **References**

Useful electron microscopy and electron diffraction references include:

Beeston, B.E.P., Horne, R.W., and Markham, R. (1972), Electron Diffraction and Optical Diffraction Techniques (North-Holland/American Elsevier Publishing Co., London/New York)

Andrews, K. W., Dyson, D.J., Keown, S.R. (1971), Interpretation of Electron Diffraction Patterns (Hilger and Watts, London).

Williams, D. B. (1987), Practical Analytical Electron Microscopy in Materials Science (Phillips Electronic Instruments, Inc. Mahwah, New Jersey)

Hirsch, P. B. et al. (1977), Electron Microscopy of Thin Crystals (Krieger, Florida)

Joy, D.C., Romig, A. D., and Goldstein, J. I. (1986), Principles of Analytical Electron Microscopy (Plenum, New York)

Figure 1. Form for recording the data from analysis of asbestos samples

TEM Analysis Form

Page ____of ____

Filter I.D.:_____ Date:_____ Grid Box/Slot (Vial):____/____ Analyst:_____ Verifying Analyst:_____

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15

Sketch Box #	Grid Opening	Ver	L (µm)	Morph			SAED							EDXA							I.D.			Structure	Comments
				HT	PS	Ot	Vis	St	Rec Id	D1	D2	Ang	ZA	Na	Mg	Si	Ca	Fe	Ot	Spect Id	Ch	Am	Ot		
1																									
2																									
3																									
4																									
5																									
6																									
7																									
8																									
9																									
10																									
11																									
12																									
13																									
14																									
15																									

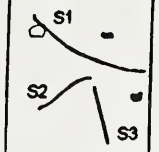
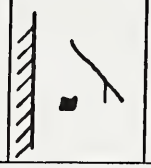
Grid orientation, traverse starting point and traverse direction

Summary	
Abs Type	# Countable Structures

Figure 2. Example of completed TEM analysis form

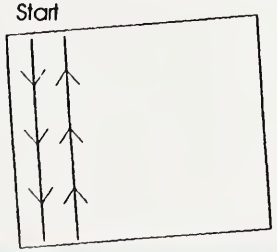
TEM Analysis Form

Filter I.D.: A203 Date: 12-20-93 Grid Box/Slot (Vial): #10/A4 Analyst: John D. Verifying Analyst: Jane D.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
		S1	S2	S3										

Sketch Box #	Grid Opening	Ver	L (µm)	Morph			SAED							EDXA								Id			Structure	Comments
				HT	PS	Ot	Vis	St	Rec id	D1	D2	Ang	ZA	Na	Mg	Si	Ca	Fe	Ot	Spect Id	Ch	Am	Ot		
1	E1		55	✓				✓	075	7.30	2.65				70	100					✓			1	
2																									
3	E1		3.0	✓			✓	✓							75	100					✓			1	
4	E1		1.5	✓			✓	✓							✓	✓					✓			1	
5	E1		1.7	✓			✓	✓							✓	✓					✓			1	
6	E1		0.7	✓			✓	✓							✓	✓					✓			0	Observable fibril length = 0.3µm
7	E1		0.7		✓				076	9.13	4.91	75°	[101]		40	100	60	10				✓		1	
8	E1		0.4																					0	Fibril length = 0.4µm
9	E1		25	✓			✓	✓							✓	✓					✓			1	
10	E1		1.3		✓				077	9.13	4.91	75°	[101]		✓	✓	✓	✓				✓		1	
11																									
12																									
13																									
14																									
15																									

Grid orientation, traverse starting point and traverse direction



F Finder square orientation

Summary	
Abs Type	# Countable Structures
Ch	5
Am	2
Total	7