NISTIR 5433

Proficiency Tests for the NIST Airborne Asbestos Program - 1992

Shirley Turner
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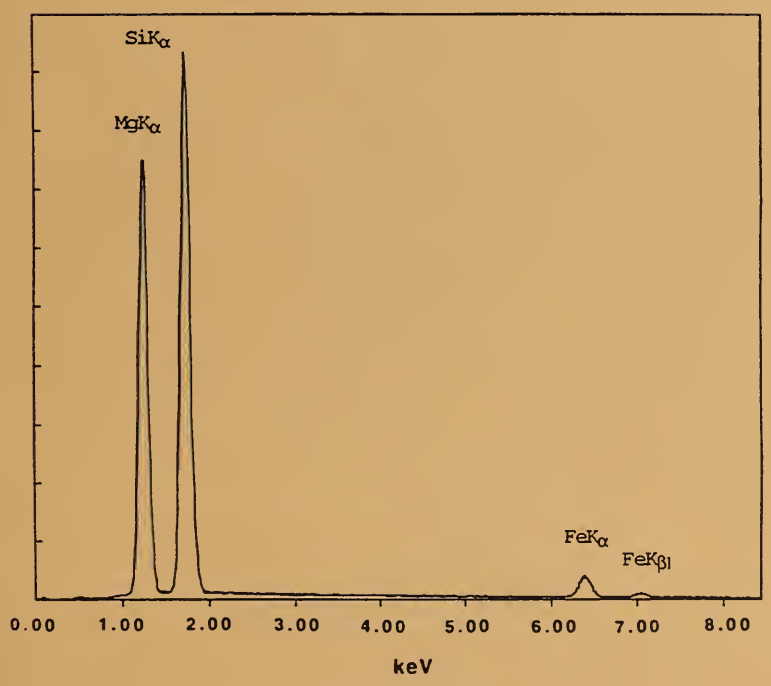
Stacy S. Doorn*
Steve B. Burris*



chrysotile energy dispersive spectrum

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards
and Technology
Microanalysis Research Group
Surface and Microanalysis Science Division
Chemical Science & Technology Laboratory
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May 1994

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U.S. DEPARTMENT OF COMMERCE
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Preface

The National Voluntary Accreditation Program (NVLAP) at the National Institute of Standards and Technology (NIST) has since 1990 had a program to accredit those laboratories involved in the analysis of airborne asbestos by transmission electron microscopy. As a part of that program, laboratories are sent proficiency tests twice yearly to evaluate their ability to correctly analyze samples and to test the general knowledge of laboratory personnel. The results of the tests are sent to the participating laboratories in the form of a summary report. This NIST Internal Report (NISTIR) contains the instructions and summary reports issued for the proficiency tests in 1992 (PT92-1, PT92-2). This NISTIR is one of a series covering the years of proficiency testing in the airborne asbestos accreditation program. The NISTIRs provide a historical record of materials sent to the laboratories for proficiency testing so that they can be referenced in other publications and so that background material can be given to those laboratories entering the accreditation program. The materials can also be used as educational aids. The material in the IRs are copies of the instructions and summary reports sent to the laboratories - if comments are warranted they are given on the chapter title page for the instructions or summary report.

Acknowledgements

We thank John M. Phelps and Diane J. Hues of NIST for their assistance in portions of these proficiency tests.

Table of Contents

I. Instructions for PT92-1

The objectives of the EDS analysis portion of the 92-1 proficiency test are the following:

- to determine the elements present in the test material
- to identify the x-ray lines present for each of the elements. Examples of x-ray lines are Kα, Kβ, Lα, etc.
- to determine the peak integrals for each of the elements before and after the background has been removed

Note: This is a two-part test. The data collected in this test will be used in a future proficiency test to determine k-values for the elements.

Please read the following instructions and examine the figures and reporting form carefully before beginning.

1. In the test packet you will find two grid boxes attached to a pressboard sheet. One of the grid boxes is labelled: NIST 92-1 I. This grid box contains a carbon-coated copper grid on which particles are deposited. The location of the grid is circled on the grid box cover. Place the grid in the electron microscope in the same manner and under the same instrument conditions used for asbestos analysis. If the grid has been damaged during shipment, or is otherwise not conducive to analysis, please contact RTI.

2. The following procedure should be followed for each of ten particles:

   a. Select a particle on the grid that: 1) is approximately 0.1 μm in width and 2) does not contain aluminum.

   b. Acquire a spectrum from the particle. Allow the acquisition to continue until the largest peak from the particle (not copper) reaches a minimum of ~10,000 background-subtracted counts. Save the spectrum.

   c. Identify the elements present. Record the element symbol and the corresponding x-ray line on Form 1 in ascending order from low to high energy (see example given in Figure 1 a, b). Include the peaks corresponding to Cu Kα, Cu Kβ and Cu Lα (if detected).

   d. For each peak determine the gross counts and the net (background subtracted) counts and record this information in the designated spaces on Form 1.

3. After the data from the ten particles have been collected, complete the last three lines on Form 1 by determining the average net (background subtracted) counts and the standard deviation for each peak.

4. Make a hard copy of one of the ten spectra. Label the peaks with the element type and x-ray line as shown in Figure 1a.

5. Save the grid for future testing and for a reference specimen. Also save the files containing the spectra.

6. Please return the following items to RTI for this part of the proficiency test, making sure that your laboratory code is on each item:

   1) A hard copy of one of the ten spectra with the particle number

   2) Form 1, completed

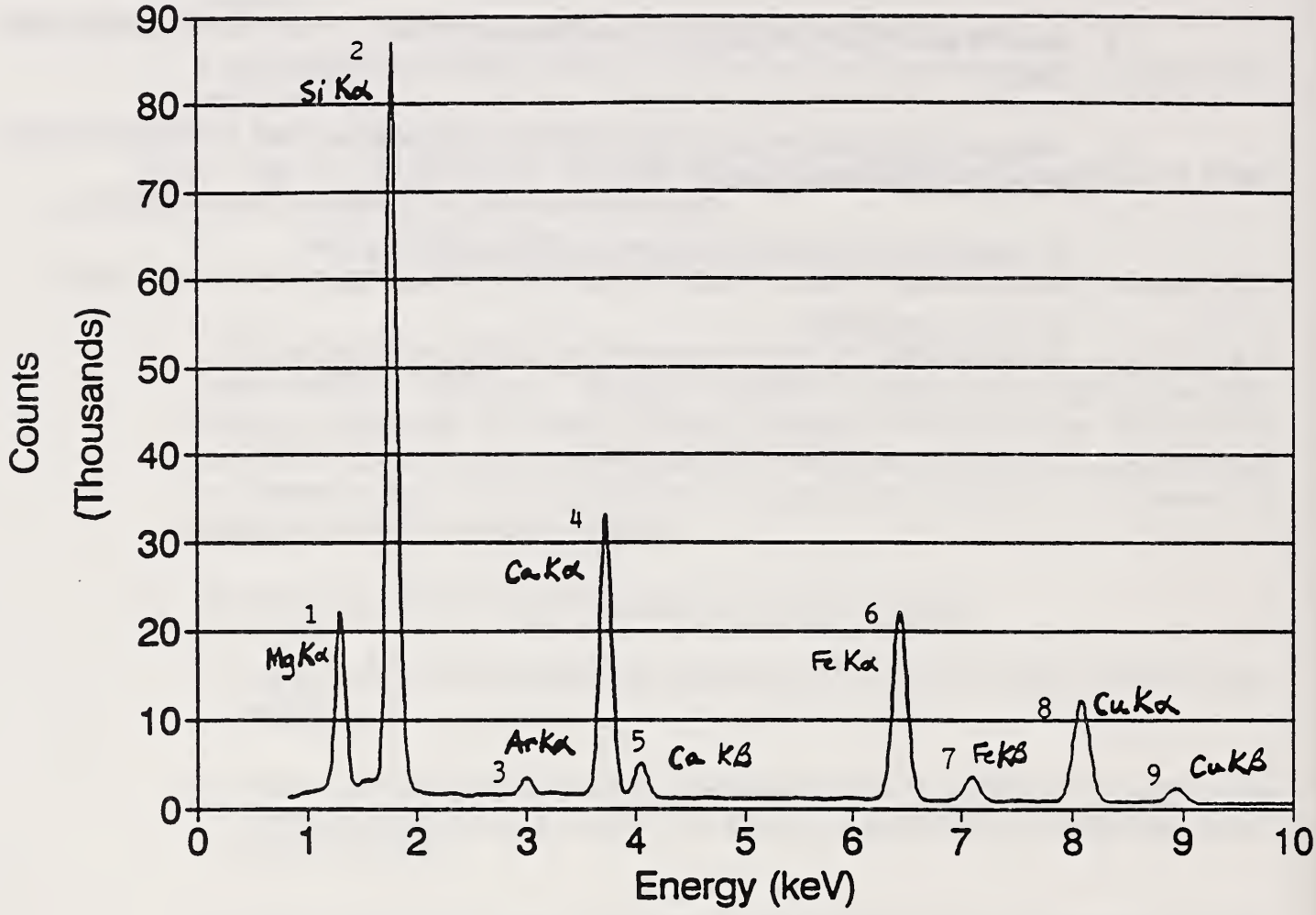


Figure 1a. Example spectrum from Standard Reference Material (SRM) 2063 on which the peaks have been numbered and identified.

NIST AIR PROGRAM PROFICIENCY TEST 92-1 Form 1: EDS Analysis Lab Code 0003

	Particle	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9	Peak 10	Peak 11	Peak 12	Peak 13	Peak 14	Peak 15
1	Element/line	Mg Kα	Si Kα	Ar Kα	Ca Kα	Ca Kβ	Fe Kα	Fe Kβ	Cu Kα	Cu Kβ						
	Gross counts	301096	1057111	81774	489331	102715	397562	87025	252694	76655						
	Bkgd. sub. counts	213242	980256	24078	434036	51779	341445	47234	200347	31256						
2	Element/line															
	Gross counts															
	Bkgd. sub. counts															
3	Element/line															
	Gross counts															
	Bkgd. sub. counts															
4	Element/line															
	Gross counts															
	Bkgd. sub. counts															
5	Element/line															
	Gross counts															
	Bkgd. sub. counts															

Figure 1b. Sample Form 1 Completed for Analysis of Standard Reference Material (SRM) 2063

NIST AIR PROGRAM PROFICIENCY TEST 92-1 Form 1: EDS Analysis Lab Code _____

Particle		Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9	Peak 10	Peak 11	Peak 12	Peak 13	Peak 14	Peak 15
1	Element/line															
	Gross counts															
	Bkgd. sub. counts															
2	Element/line															
	Gross counts															
	Bkgd. sub. counts															
3	Element/line															
	Gross counts															
	Bkgd. sub. counts															
4	Element/line															
	Gross counts															
	Bkgd. sub. counts															
5	Element/line															
	Gross counts															
	Bkgd. sub. counts															

Form 1: EDS Analysis (Cont'd)

Lab Code _____

Particle		Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9	Peak 10	Peak 11	Peak 12	Peak 13	Peak 14	Peak 15
6	Element/line															
	Gross counts															
	Bkgd. sub. counts															
7	Element/line															
	Gross counts															
	Bkgd. sub. counts															
8	Element/line															
	Gross counts															
	Bkgd. sub. counts															
9	Element/line															
	Gross counts															
	Bkgd. sub. counts															
10	Element/line															
	Gross counts															
	Bkgd. sub. counts															
Element/line																
Average Bkgd. sub. counts																
Standard Deviation																

The objective of this portion of the 92-1 proficiency test is to identify an unknown mineral as either chrysotile, a regulated amphibole or "other". Please read the instructions carefully and examine the reporting form before beginning.

Note: For the purposes of this test, aspect ratio should not be used to distinguish asbestos from nonasbestos.

1. For this portion of the test use grid box labelled: NIST 92-1 II. This grid box contains a particle mount prepared from an aqueous solution. For archival purposes, the laboratory may consider very carefully applying a light carbon coating. The location of the grid is circled on the grid box cover. If the grid has been damaged during shipment, or is otherwise not conducive to analysis, please contact RTI.

2. Analyze several grains on the grid to determine the dominant mineral species (ignore minor or trace constituents).

3. Identify the dominant mineral species as either chrysotile, a regulated amphibole mineral or as "other". Record identification and supporting evidence on Form 2.

4. After completing the analysis of this sample, please save the grid for a reference specimen.

5. Return Form 2 and supporting evidence to RTI for this part of the 92-1 proficiency test.

Form 2: Identification of an Unknown

1. Put a check next to the identity of the dominant mineral species on the grid.

_____ chrysotile (serpentine)

_____ regulated amphibole (if checked, also check one of the following)

_____ amosite (grunerite, cummingtonite)

_____ crocidolite (riebeckite)

_____ anthophyllite

_____ actinolite-tremolite

_____ other

2. In two pages or less, summarize the data used to make the determination. Criteria can include:

1) morphology, excluding aspect ratio

2) crystallographic data

3) chemical data

Include at least the minimum data needed to make the identification or if the "other" category was checked, include the minimum information necessary to distinguish the compound from the regulated minerals.

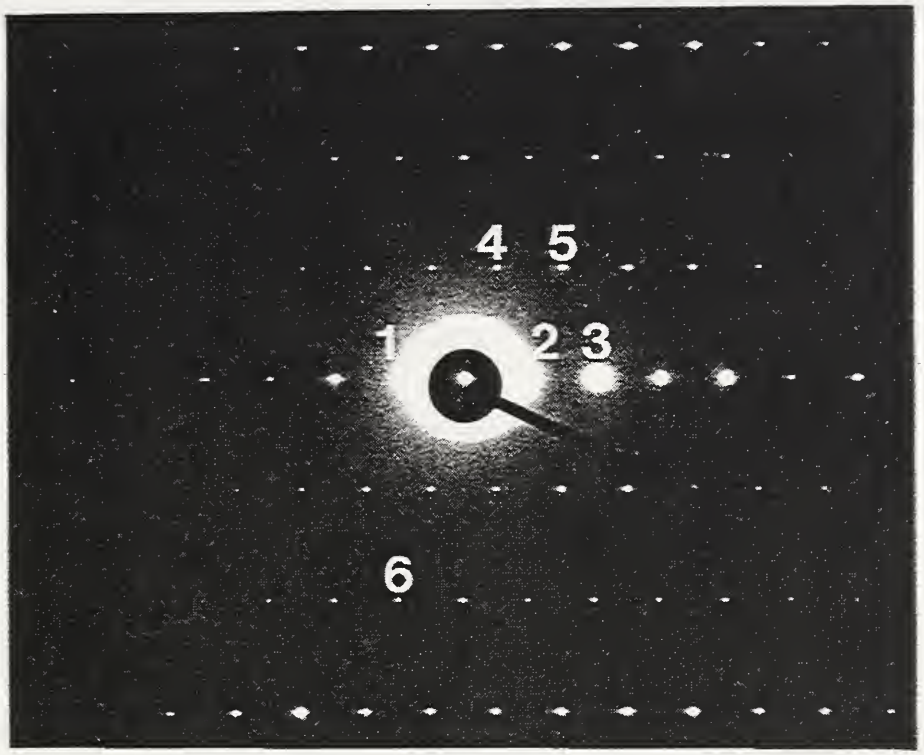
Instructions for evaluation of electron diffraction patterns

Carefully follow the instructions below for determining and recording values for each diffraction pattern. Any length measurements made from the diffraction patterns should be recorded in mm. D-spacings should be recorded in nm [Note: 1 nm = 10 Å]. Record positive (hkl) values for the labelled reflections lying in the positive x-y quadrant of the diffraction pattern (upper right-hand quadrant of diffraction pattern). If you run out of space for your answers on the forms provided, please feel free to use extra sheets.

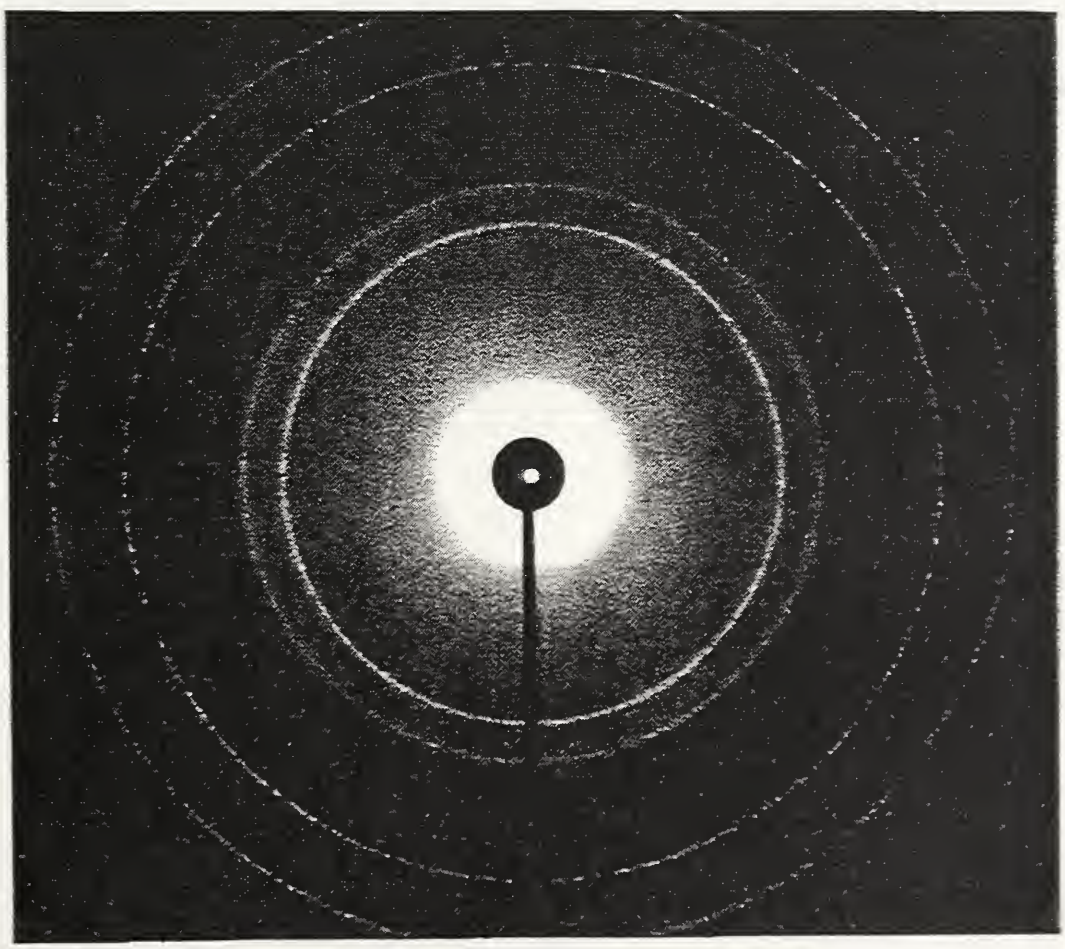
Figure 2: tremolite-actinolite and evaporated gold

Figure 2 consists of a diffraction pattern from an amphibole from the tremolite-actinolite series and a diffraction pattern from evaporated gold. The tremolite-actinolite pattern and the gold pattern have the same camera constant. From the gold pattern, determine the camera constant and the standard deviation of this value. From the tremolite-actinolite pattern, determine 1) the d-spacings for the reflections labelled 1-6, 2) the (hkl) values for the labelled reflections, and 3) the zone-axis orientation of the pattern. Show the work involved in calculating the camera constant and d-spacings on Form 3. Also on Form 3 describe the way in which the (hkl) values and the zone-axis orientation for the tremolite-actinolite pattern were determined. Record the camera constant, standard deviation, d-spacings, (hkl) values, and zone-axis orientation on Form 4.

Figure 2



Anthophyllite diffraction pattern



Evaporated gold diffraction pattern

Form 3: tremolite-actinolite and evaporated gold

1. In the space below, list any measured distances and show the work done to determine the camera constant for the gold diffraction pattern in Figure 2.

2. In the space below, show the measurements and the work done to calculate the d-spacings corresponding to the labelled reflections on the tremolite-actinolite pattern in Figure 2.

Form 3: tremolite-actinolite and evaporated gold (continued)

3. What method was used to assign (hkl) values to the labelled reflections?

4. How was the zone-axis orientation of the tremolite-actinolite pattern determined?

Form 4: Summary sheet for evaluation of diffraction patterns

Figure 2

Gold diffraction pattern	
Camera constant (mm•nm)	
Standard deviation	

Tremolite-actinolite diffraction pattern		
Reflection #	d-spacing (nm)	(hkl)
1		
2		
3		
4		
5		
6		

Zone axis orientation	

II. Summary Report for PT92-1

The information presented in this report is a summary of the analysis of materials and the performance of laboratories on the proficiency test sent to laboratories in the Airborne Asbestos Program in April of 1992 (designated as test 92-1). Discussion of results for the EDS analysis, identification of an unknown and diffraction pattern analysis is given in the main portion of the report. The results obtained by the laboratory receiving this report are given in Appendix A.

PART 1 - EDS ANALYSIS

Summary of test design

For this section of the proficiency test, the laboratories were sent a carbon film grid onto which glass particles had been deposited. The laboratories were asked to acquire spectra from each of ten particles that were approximately 0.1 μm in diameter and free of aluminum. The spectra were to be accumulated so that the largest peak reached a minimum of 10,000 background-subtracted counts. The element symbol, x-ray line type, gross counts, and background-subtracted counts for each peak in each spectrum were recorded.

This section of the proficiency test was the first part of a two-part test. The data collected in this part of the test will be used in a future proficiency test to calculate k-values for the elements.

Composition of glass particles

The glass, synthesized by the Ceramics Division of NIST, was designed to simulate the composition of crocidolite. A comparison of the spectrum for the glass to a spectrum obtained from crocidolite (Standard Reference Material 1866[1]) is given in Figure 1. The approximate composition of the glass is given in Table 1.

Table 1. Approximate weight percent for elements in proficiency test glass

Element	Approximate Weight Percent
Na	5-10%
Mg	5-10%
Si	> 10%
K	< 1%
Ca	< 1%
Ti	< 1%
Mn	< 1%
Fe	> 10%

More detailed results for the glass composition will be given in the proficiency test where the k-value calculation is required.

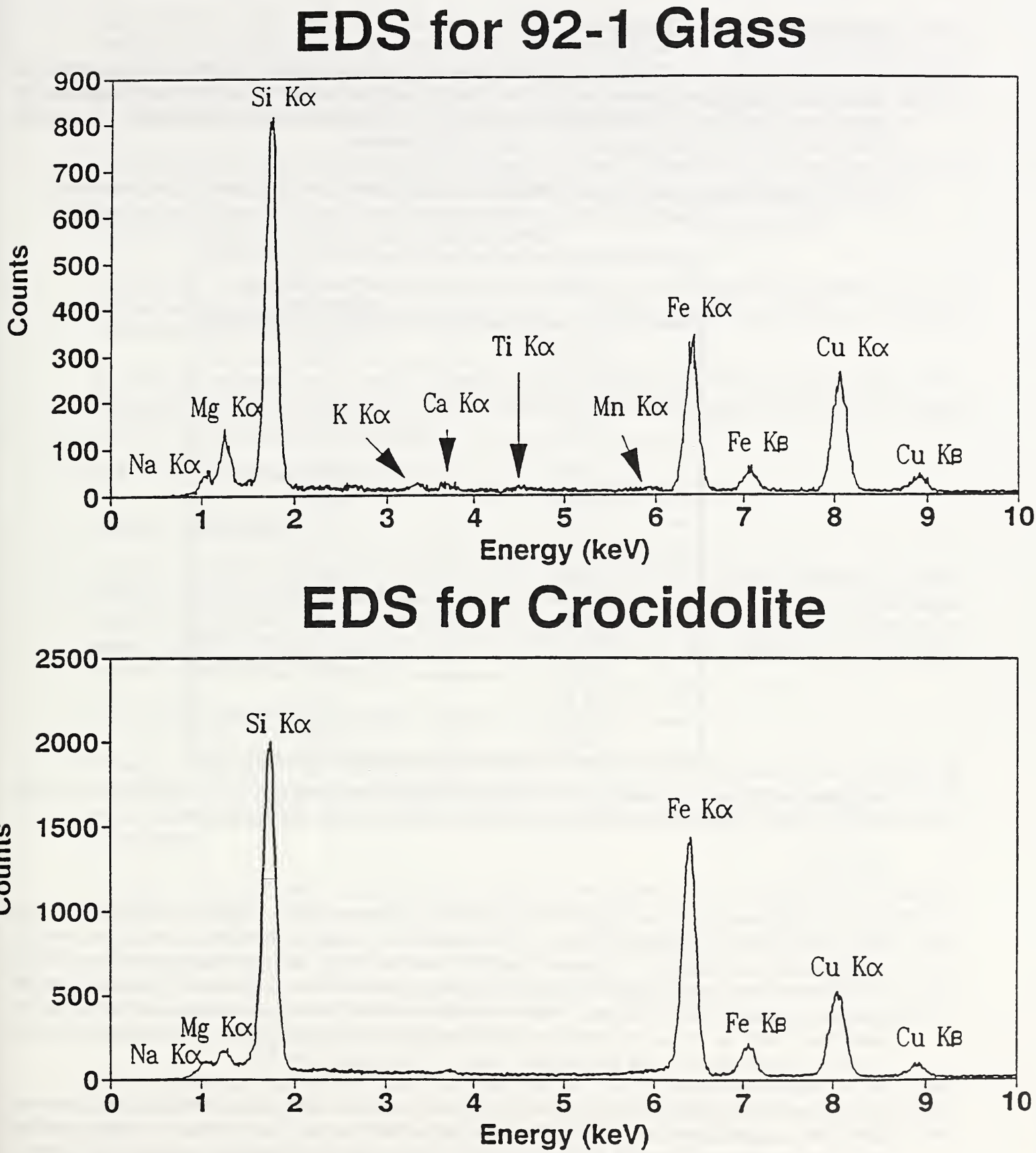


Figure 1. Spectra obtained from proficiency test glass (top) and crocidolite (SRM 1866)(bottom).

Results submitted by laboratories

One-hundred and eleven laboratories submitted results. A summary of the peaks and the number of laboratories that missed the peaks for all ten particles is given in Table 2. The value in parentheses indicates the number of laboratories that did not report the peak for some of the ten particles analyzed. (Note: the Na, Mg and Si peaks include a minor Kβ component.)

Table 2. Peaks in glass and number of laboratories that missed the peaks (numbers in parentheses indicate the number of laboratories that missed the peak in at least one of the ten particles)

Peak	Number of labs that missed peak
Na Kα	18 (9)
Mg Kα	0 (2)
Si Kα	0 (0)
K Kα	58 (3)
Ca Kα	57 (5)
Ti Kα	57 (5)
Mn Kα	61 (5)
Fe Kα	7 (3)
Fe Kβ	5 (2)
Cu Kα	1 (1)
Cu Kβ	7 (3)

Numerous other elements were reported by the laboratories including C, O, F, Al, S, Cl, Ar, Ca (Kβ), Ti (Kβ), Cr (Kα, Kβ), Fe (Lα), Ni (Kα), Cu (Lα), Zn (Kα, Kβ), and Au (Lα, Mα).

Discussion

All of the laboratories found Mg, Si and Fe in the particles examined. Eighteen laboratories did not report Na for any of the ten particles. This is considered a major deficiency as the Handbook for Airborne Asbestos Analysis[2] states under item 39d that the energy dispersive unit should be calibrated so that there is "proven detection of Na peak in standard crocidolite or equivalent". Upon reviewing the spectra obtained by the laboratories, it was evident that several of the laboratories obtained a Na peak but identified it only as Cu Lα. Other laboratories identified Na on the spectra but did not determine the number of counts for the peak and did not report it on the appropriate form.

Roughly half of the laboratories did not report K, Ca, Ti or Mn. The weight percent of these elements is < 1%, so for this proficiency test, the laboratories were not marked in error for these omissions. However, detection of elements in these concentrations can be important. For instance, Mn occurs in a low concentration in amosite and is a good indicator for the presence of this mineral. Laboratories were not marked in error for additional peaks found in the spectra. Several peaks including C, O, Ca

Kβ, Ti Kβ, Fe Lα and Cu Lα are considered acceptable for the glass material given long count times or ultrathin or windowless detectors. The presence of elements such as Cr, Ni and Zn may indicate x rays from the specimen holder or microscope. Other elements such as F, S, Cl, Ar and Au do not occur in the glass material. Laboratories obtaining these peaks should investigate the source of these x-ray lines. However, laboratories obtaining these elements were not marked in error for this proficiency test.

Comments are given in the individual laboratory reporting form (Appendix A) for anomalies noted in the spectra. For example, two of the laboratories acquired spectra with an unusually large Ca peak.

PART II - IDENTIFICATION OF AN UNKNOWN

For this part of the proficiency test, laboratories were sent a grid with a material deposited on it. The laboratories were asked to analyze several particles and to identify the dominant material as chrysotile, a regulated amphibole or as "other."

The material deposited on the grids is amosite used for Standard Reference Material 1866[1]. Two laboratories incorrectly identified it as "other." Incorrect identification of this material indicates a serious deficiency in the laboratory's proficiency.

PART III - ELECTRON DIFFRACTION

Summary of test design

Laboratories were sent copies of a diffraction pattern of polycrystalline sputtered gold and a zone axis diffraction pattern from the tremolite-actinolite series. Laboratories were asked to determine the camera constant from the gold ring pattern in units of mm-nm, index the labeled reflections on the zone axis pattern, report d-spacings in nanometers and calculate the zone axis. One hundred eleven laboratories returned results in this proficiency test.

Polycrystalline gold diffraction pattern

Distances on the gold ring diffraction pattern were to be reported in millimeters and d-spacings in nanometers. Therefore, the units for the camera constant were mm-nm. Seventeen laboratories reported results in units other than mm-nm. Two laboratories reported camera constants based on diameter measurements.

The range of reported camera constants based on radial measurements was 7.28 mm-nm to 7.5726 mm-nm with a mean of 7.438 mm-nm and a standard deviation of 0.043 mm-nm. The range of reported camera constants based on diameter measurements was 14.7423 mm-nm to 14.896 mm-nm with a mean and standard deviation of 14.819 mm-nm and 0.109 mm-nm, respectively. For purposes of this proficiency test only, those laboratories reporting values in units other than mm-nm were marked correct if the converted values were within the acceptable ranges listed. Those laboratories reporting numerical values outside the ranges listed above were marked not acceptable.

Table 3 summarizes the reported camera constants and provides the acceptable ranges for the results. The acceptable range is within ± 5% of the mean reported value. Outliers are defined as being greater than ±10% of the population mean and are not used in calculating the sample mean or acceptable ranges. There were no outliers for this case.

Table 3. Reported Camera Constant Statistics

Statistic	Radius	Diameter
Mean	7.438 mm-nm	14.819 mm-nm
Standard Deviation	0.043 mm-nm	0.109 mm-nm
Acceptable Range	7.066-7.810 mm-nm	14.078-15.560 mm-nm
Labs Outside Range (> ± 5%)	0	0
Outliers (> ± 10%)	0	0

Tremolite-Actinolite zone axis pattern

The laboratories were asked to obtain d-spacings and Miller indices from the labeled reflections and to determine the correct zone axis for the tremolite-actinolite diffraction pattern. Due to some ambiguity in the instructions, five laboratories did not report results for reflections 1 and 6. One laboratory did not report d-spacings or indices for these reflections.

Table 4 summarizes the d-spacings reported for the diffraction pattern. As stated in the Summary Report for the December 1990 Round of the NIST Airborne Asbestos Proficiency Test, the acceptable range for the d-spacing measurements is taken as ± 5% of the mean reported value. Reported values that were significantly (> ± 10%) outside the population mean were considered outliers and were not included in the calculation of the mean reported value.

Table 4. Reported d-Spacing Statistics

Statistic	R1	R2	R3
Mean	0.908 nm	0.908 nm	0.452 nm
Standard Deviation	0.013 nm	0.012 nm	0.006 nm
Acceptable Range	0.863-0.953 nm	0.863-0.953 nm	0.429-0.475 nm
Labs Outside Range (> ± 5%)	3	3	9
Outliers (> ± 10%)	1	1	8

Table 4 (continued).

Statistic	R4	R5	R6
Mean	0.510 nm	0.444 nm	0.246 nm
Standard Deviation	0.005 nm	0.005 nm	0.003 nm
Acceptable Range	0.484-0.536 nm	0.422-0.467 nm	0.234-0.258 nm
Labs Outside Range (> ± 5%)	3	2	4
Outliers (> ± 10%)	3	2	3

Four possible combinations of Miller indices were considered correct for the zone axis diffraction pattern. Only the complete combination of indices were considered correct. If a laboratory mixed possible indices, only those which are internally consistent were considered correct. Table 5 summarizes the combinations and responses. It should be noted that the number of correct responses reflects only the individual reflection's Miller index, not the combination of indices. The incorrect responses reflect indices which were not possible for the indicated reflection.

One hundred and five laboratories reported either the correct zone axis or a crystallographically equivalent zone axis. Of these laboratories, five did not reduce the zone axis [uvw] to the lowest order. For this proficiency test, reducible, higher order zone axis designations, were not marked as incorrect if they were equivalent. Six laboratories reported incorrect zone axis designations.

Table 5. Miller Indices and Zone Axes

Reflection	Case 1	Case 2	Case 3	Case 4	Correct	Incorrect
4	(0-20)	(020)	(0-20)	(020)	108	3
2	(020)	(0-20)	(020)	(0-20)	108	3
4	(040)	(0-40)	(040)	(0-40)	106	5
4	(001)	(001)	(00-1)	(00-1)	108	5
5	(021)	(0-21)	(02-1)	(0-2-1)	100	11
5	(0-2-2)	(02-2)	(02-2)	(022)	99	12
Zone Axis	[100]	[100]	[100]	[100]	105	6

Discussion

The most common error observed in the electron diffraction part of the test was the incorrect reporting of the d-spacing for the (040) reflection, diffraction spot 3 on the pattern. Seven laboratories reported values equivalent to the (020) reflections, diffraction spots 1 and 2. By definition the (040) d-spacing is one half the (020) or, for this test, approximately 0.452 nm. The incorrect assignment of Miller indices was the next most common error. Some combination of a) inconsistent indexing, b) application of indices from forbidden reflections or, c) incorrect vector addition was observed from twenty-one of the laboratories returning results. Internally inconsistent indexing is assigning the same Miller index to two or more different reflections or failure to maintain the proper sign convention throughout the pattern.

Only six of the laboratories reported an incorrect zone axis. In checking their results, the zone axis designations were consistent with the reported Miller indices. This indicates that no difficulty exists in calculating zone axes. It is suggested, however, that the laboratories consistently check each combination of Miller indices in order to assure that the correct axis is determined.

DISCUSSION OF GRADING OF THE PROFICIENCY TEST

The results for the laboratory receiving this report are given in Appendix A. A laboratory passed this proficiency test if it accumulated fewer than two errors. Omission of Na on the EDS analysis and misidentification of amosite were considered serious deficiencies and were counted as two errors each. An incorrect zone axis or d-spacing counted as one error, and incorrect assignment of Miller indices to reflections counted as 0.5 errors. Twenty-nine of one hundred eleven laboratories did not pass this proficiency test.

References

1. NIST Certificate of Analysis SRM 1866; Office of Standard Reference Materials, NIST, Gaithersburg, MD.

2. E.B. Steel, S. Turner, H.W. Berger, NVLAP Program Handbook for Airborne Asbestos Analysis, National Institute of Standards and Technology, NISTIR 89-4137, 1989.

III. Instructions for PT92-2

NIST AIR PROGRAM PROFICIENCY TEST 92-2

Part I - EDS Analysis

Instructions:

Part I of the 92-2 proficiency test is a continuation of the EDS Analysis portion of the 92-1 test. For the previous test a carbon film grid containing glass particles was sent to each laboratory. The laboratories were asked to identify the elements present in the glass by energy dispersive spectroscopy (EDS), and to determine the x-ray line types. The laboratories were also asked to determine the gross and background-subtracted counts for each peak. The purpose of Part I of the 92-2 proficiency test is to determine each laboratory's ability to calculate k-values for each of the major elements in the glass sample.

Please read the following instructions carefully before beginning.

Calculate the k-values relative to Si for the Na, Mg, Si and Fe in the proficiency test glass from round 92-1 using the chemical composition information in Table 1 and peak counts which can be obtained in any of the following ways:

1. Use the peak counts obtained and reported for the 92-1 proficiency test if the counts are satisfactory as indicated by your laboratory's results from the previous test.

OR

2. If Na was not reported on the previous proficiency test, Na counts must be derived either from the previously collected spectra or by acquiring new spectra from the archived test sample.

OR

3. If your laboratory was not enrolled in NVLAP for the 92-1 TEM proficiency test, please see the Special Note included in your test packet.

If new count information is obtained, please collect the counts using the following instructions:

- Acquire spectra from 10 particles that do not contain aluminum and that have diameters ≤ 0.1 μm.
- Allow acquisition to continue until the largest peak (not Cu) reaches a minimum of approximately 10,000 background-subtracted counts.
- Save the spectra and make a hard copy of one of the spectra. Label the peaks on the hard copy with element type and x-ray line type.
- Record the element, x-ray line type and newly acquired counts on Form 3.

Show the work done to determine the k-values on Form 1. Record the k-values, the number of spectra used to determine the k-values, and the standard deviations on Form 2. If the previous spectra are reanalyzed, or new spectra are acquired, complete Form 3.

Note: The glass sample along with the compositional information in Table 1 can be used as a reference for routine monitoring of k-values.

Table 1. Listing of the chemical composition of the proficiency test glass

Element	Weight % oxide	Weight % element	Atomic % element
O	---	42.4	59.5
Na	9.3	6.9	6.7
Mg	9.9	6.0	5.5
Si	54.5	25.5	20.4
K	0.5	0.4	0.2
Ca	0.5	0.4	0.2
Ti	0.7	0.4	0.2
Mn	0.6	0.5	0.2
Fe	24.0	17.5	7.0

Part I - EDS Analysis

Form 1

Show the work done to calculate the k-values relative to Si (use extra pages if necessary):

Part I - EDS Analysis

Form 2

Record the mean k-values relative to Si, the number of spectra used to determine the k-values, and the standard deviation in the appropriate spaces.

Element	k-value	# Spectra	Standard dev.
Na			
Mg			
Si			
Fe			

NIST AIR PROGRAM PROFICIENCY TEST 92-2

Form 3: EDS Analysis

Lab Code _____

Check one choice below:

___ reanalysis of septra collected from proficiency test 92-1

___ new spectra acquired

Particle		Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9	Peak 10	Peak 11	Peak 10	Peak 13	Peak 14	Peak 15
1	Element/line															
	Gross counts															
	Bkgd. sub. counts															
2	Element/line															
	Gross counts															
	Bkgd. sub. counts															
3	Element/line															
	Gross counts															
	Bkgd. sub. counts															
4	Element/line															
	Gross counts															
	Bkgd. sub. counts															
5	Element/line															
	Gross counts															
	Bkgd. sub. counts															

Form 3: EDS Analysis (Cont'd)

Lab Code _____

Particle		Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8	Peak 9	Peak 10	Peak 11	Peak 12	Peak 13	Peak 14	Peak 15
6	Element/line															
	Gross counts															
	Bkgd. sub. counts															
7	Element/line															
	Gross counts															
	Bkgd. sub. counts															
8	Element/line															
	Gross counts															
	Bkgd. sub. counts															
9	Element/line															
	Gross counts															
	Bkgd. sub. counts															
10	Element/line															
	Gross counts															
	Bkgd. sub. counts															

Part II - Identification of an Unknown

Instructions:

The objective of this portion of the 92-2 proficiency test is to identify an unknown mineral as either chrysotile, a regulated amphibole or "other". Please read the instructions carefully and examine the reporting form before beginning.

Note: For the purposes of this test, length and width should <u>not</u> be used to distinguish asbestos from nonasbestos.

1. The sample for this portion of the test is located in the side of the enclosed grid box labelled: NIST 92-2 II. The sample is a particle mount prepared from an aqueous solution. For archival purposes, the laboratory may consider very carefully applying a light carbon coating. The location of the grid is circled on the grid box cover. If the grid has been damaged during shipment, or is otherwise not conducive to analysis, please contact RTI.

2. Analyze several grains on the grid to determine the dominant mineral species (ignore minor or trace constituents).

3. Identify the dominant mineral species as either chrysotile, a regulated amphibole or as "other". Record identification and supporting evidence on Form 4.

4. After completing the analysis of this sample, please save the grid for a reference sample.

5. Return Form 4 and supporting evidence to RTI for this part of the 92-2 proficiency test.

Part II - Identification of an Unknown

Form 4

1. Put a check next to the identity of the dominant mineral species on the grid.

_____chrysotile (serpentine)

_____regulated amphibole (if checked, also check one of the following)

_____amosite (grunerite, cummingtonite)

_____crocidolite (riebeckite)

_____anthophyllite

_____actinolite-tremolite

_____other

2. In two pages or less, summarize the data used to make the determination. Criteria can include:

1) morphology, excluding aspect ratio

2) crystallographic data

3) chemical data

Include at least the minimum data needed to make the identification or if the "other" category was checked, include the minimum information necessary to distinguish the compound from the regulated minerals.

Part III - Grid Opening Measurement

Instructions:

Grid opening measurements which have an accuracy within $\pm 5\%$ of the true value are a requirement of NVLAP accredited laboratories performing airborne asbestos analysis by transmission electron microscopy as documented in Appendix F of the "NVLAP Program Handbook for Airborne Asbestos Analysis", NISTIR 89-4137 August, 1989. The purpose of this portion of the 92-2 round is to test grid opening measurement.

Note: The grids are to be kept by the laboratory for use in the next proficiency test for the preparation of filters.

Please read the instructions carefully and examine the reporting forms before beginning.

Twenty 200-mesh indexed copper EM grids have been sent to each laboratory. The grids are located in a plastic vial taped to a pressboard sheet. Please place the grids in slots A1 - D5 in the grid box attached to the pressboard sheet. Determine the areas of 20 grid squares on each of 20 grids using one of the procedures listed in the AHERA method[1] or in the "bronze" book[2]. On Form 5, describe the method used to measure the grid openings. On Form 6, record the mean grid square area (μm^2) and standard deviation for each grid. Calculate the grand mean and grand standard deviation of the ≥ 400 grid square measurements (do not use the grid means to obtain the grand mean and grand standard deviation).

1. Code of Federal Regulations, Asbestos-containing materials in schools; final rule and notice, 40 CFR Part 763, 41826-41905, 1987.

2. Chesson, J., Chatfield, E. (1989) Transmission Electron Microscopy Asbestos Laboratories Quality Assurance Guidelines, Environmental Protection Agency, EPA 560/5-90-002.

Part III - Grid Opening Measurement

Form 5

Describe the method used to measure the grid openings:

Part III - Grid Opening Measurement

Form 6

Grid designation	Mean area (μm^2)	# Grid squares measured (≥ 20)	Standard deviation
Grid A1			
Grid A2			
Grid A3			
Grid A4			
Grid A5			
Grid B1			
Grid B2			
Grid B3			
Grid B4			
Grid B5			
Grid C1			
Grid C2			
Grid C3			
Grid C4			
Grid C5			
Grid D1			
Grid D2			
Grid D3			
Grid D4			
Grid D5			

Grand mean_____ Grand st. dev._____ Grand # grid squares_____

IV. Summary Report for PT92-2

The information presented in this report is a summary of the analysis of materials and the performance of laboratories on the proficiency test sent to laboratories in the Airborne Asbestos Program in September of 1992 (designated as test 92-2). Discussion of results for the determination of k-values, identification of an unknown, and grid opening measurement is given in the main portion of the report. Individual laboratory results are given in a separate enclosure as an attachment to the cover letter sent with this summary report.

PART I - EDS ANALYSIS

Summary of test design

This section of the proficiency test is a continuation of the energy dispersive spectrometry (EDS) analysis portion of the 92-1 test. For the previous test the laboratories were sent a carbon film grid onto which glass particles had been deposited. The laboratories were asked to identify the elements present in the glass by EDS, and to determine the gross and background-subtracted counts for each peak. For this part of the 92-2 test, the laboratories were given the composition of the glass particles and were asked to determine the k-values for each of the major elements (Na, Mg, Si, Fe).

Composition of glass particles

The composition of the glass particles was given in the instructions for this test. Details of the analyses and the standard deviations for the composition values are given in this section. The appropriate form of the composition to use for calculation of k-values is the element weight percent.

Portions of the glass samples were analyzed in a JEOL JXA-8600 Superprobe[1] equipped with a ZMAX 30 Tracor Northern EDS detector. Spectra were acquired at 15 kV and with a beam current of 1.5 nA. The NIST/NIH Desktop Spectrum Analyzer Program (DTSA) was used to perform the quantitative analysis of the collected x-ray data (1). The weight percent for oxygen was determined by stoichiometry. Table 1 gives the weight percent and standard deviation derived from averaging eight analyses. The composition reported in Table 1 is in good agreement with the weight percent of elements used to form the glass.

[1]Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedures. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for the purpose.

Table 1. Weight percent for elements in glass sample distributed in proficiency test 92-1.

Element	Weight percent (st. dev.)
O	42.4
Na	6.9 (0.1)
Mg	6.0 (0.1)
Si	25.5 (0.3)
K	0.4 (0.04)
Ca	0.4 (0.04)
Mn	0.7 (0.05)
Fe	17.5 (0.1)

Results submitted by laboratories

The range and median value for the k-values calculated from the data submitted by the laboratories are given in Table 2.

Table 2. Range and median value for k-values (calculated from laboratory counts).

Element	Range	Median
Na	1.56 - 240.19	5.00
Mg	1.07 - 26.09	1.85
Si	1	1
Fe	0.9 - 6.32	1.44

For this proficiency test, the laboratories were evaluated on their ability to calculate correctly k-values from their background-subtracted counts and were not evaluated on the absolute value obtained for k-factors. Values determined by the laboratory and by RTI/NIST analysts were compared and the percentage difference determined. A summary of the number of laboratories obtaining differences less than 5% and greater than or equal to 5% is given in Table 3.

Table 3. Percentage difference for average k-values calculated by laboratory and by RTI/NIST.

Element	Number of labs < 5% difference	Number of labs ≥ 5% difference
Na	94	7
Mg	95	6
Si	101	0
Fe	94	7

Those laboratories obtaining a greater than or equal to 5% difference were found to have some error in their calculation of k-values. Examples of errors include: 1) use of the average counts to determine the k-value rather than the average of the k-values obtained for each particle, 2) use of the average number of Si counts rather than the Si counts obtained for each particle, 3) incorrect use of the formula for k-value determination and 4) use of background counts rather than peak counts.

Discussion

In the present version of the Handbook for Airborne Asbestos Analysis (2), there is a requirement for laboratories to determine and monitor the k-values for Na, Mg, Al, Si, Ca and Fe. The next handbook revision will contain upper limits for each of the k-values. The determination of tentative upper limits for these k-values was done by calculating k-values from spectra generated from DTSA. By using this program, spectra can be calculated for given compositions, sample thicknesses, electron microscope parameters, and detector characteristics. Spectra were calculated for five materials, for both 50 nm and 100 nm thicknesses, for both 80 and 120 keV accelerating voltage and for a Si detector with a 7.6 μm Be window with a 0.1 μm layer of ice on the window surface. The range of k-values calculated for Na, Mg and Fe is given in Table 4.

Table 4. Range of k-values determined for Na, Mg, Si and Fe from spectra generated using DTSA.

Element	Range in k-values	Number of materials
Na	2.06 - 2.19	3
Mg	1.28 - 1.46	4
Si	1	
Fe	1.47 - 1.69	5

The revision to the Handbook for Airborne Asbestos Analysis will specify allowable upper limits for k-values for these elements. The proposed upper limits are: 4.0 for Na, and 3.0 for Mg and Fe. If the laboratory obtained values above these upper limits in the analysis of the proficiency test glass, they should redetermine the values with a second test material. If elevated values are confirmed, the cause should be investigated. Examples of problems that can cause incorrect k-values include: excessive icing or contamination of the detector window; improper TEM alignment; instrumentation

not designed for EDS; improper specimen and/or detector geometry; and excessive specimen contamination.

PART II - IDENTIFICATION OF AN UNKNOWN

Summary of test design

For this part of the proficiency test, laboratories were sent a grid with a material deposited on it. The laboratories were asked to analyze several particles and to identify the dominant material as chrysotile, a regulated amphibole or as "other".

Identity of unknown

The material deposited on the grids is a magnesian hedenbergite with minor quartz. An x-ray diffraction pattern of the material is shown in Figure 1. The diffraction pattern is compared to the d-spacings in the JCPDS file for hedenbergite, quartz and actinolite. An EDS spectrum obtained from the sample is shown in Figure 2. The presence of major Mg, Si, Ca and Fe is evident. These elements are consistent with identification of the material as a magnesian hedenbergite. The material should have been identified as "other" by the laboratories.

Results submitted by laboratories

Of the one hundred and one laboratories submitting results, seventy-six correctly identified the material as "other" and twenty-five incorrectly identified it as tremolite-actinolite.

Discussion

In the Handbook for Airborne Asbestos Analysis (item 5k, p. F3) (2) there is a requirement that laboratories have criteria for differentiating asbestos minerals from the pyroxenes. Laboratories that incorrectly identified this material as actinolite-tremolite should determine the chemical and electron diffraction criteria that can be used to differentiate hedenbergite from actinolite. These criteria should include: 1) determination of peak ratios from EDS spectra (the Ca/Si ratio for hedenbergite is approximately twice that for actinolite) and 2) identification of distinguishing features of diffraction patterns (hedenbergite and actinolite have approximately the same a and c unit cell parameters, but the b unit cell parameter for actinolite is twice that for hedenbergite). Additionally, it is recommended that laboratories accumulate a file of diffraction patterns from reference amphibole specimens so that laboratory personnel are familiar with the d-spacings and symmetries found on commonly obtained amphibole patterns. Laboratories should also determine or review criteria for distinguishing other amphibole and pyroxene pairs that qualitatively have the same composition (e.g., tremolite and diopside).

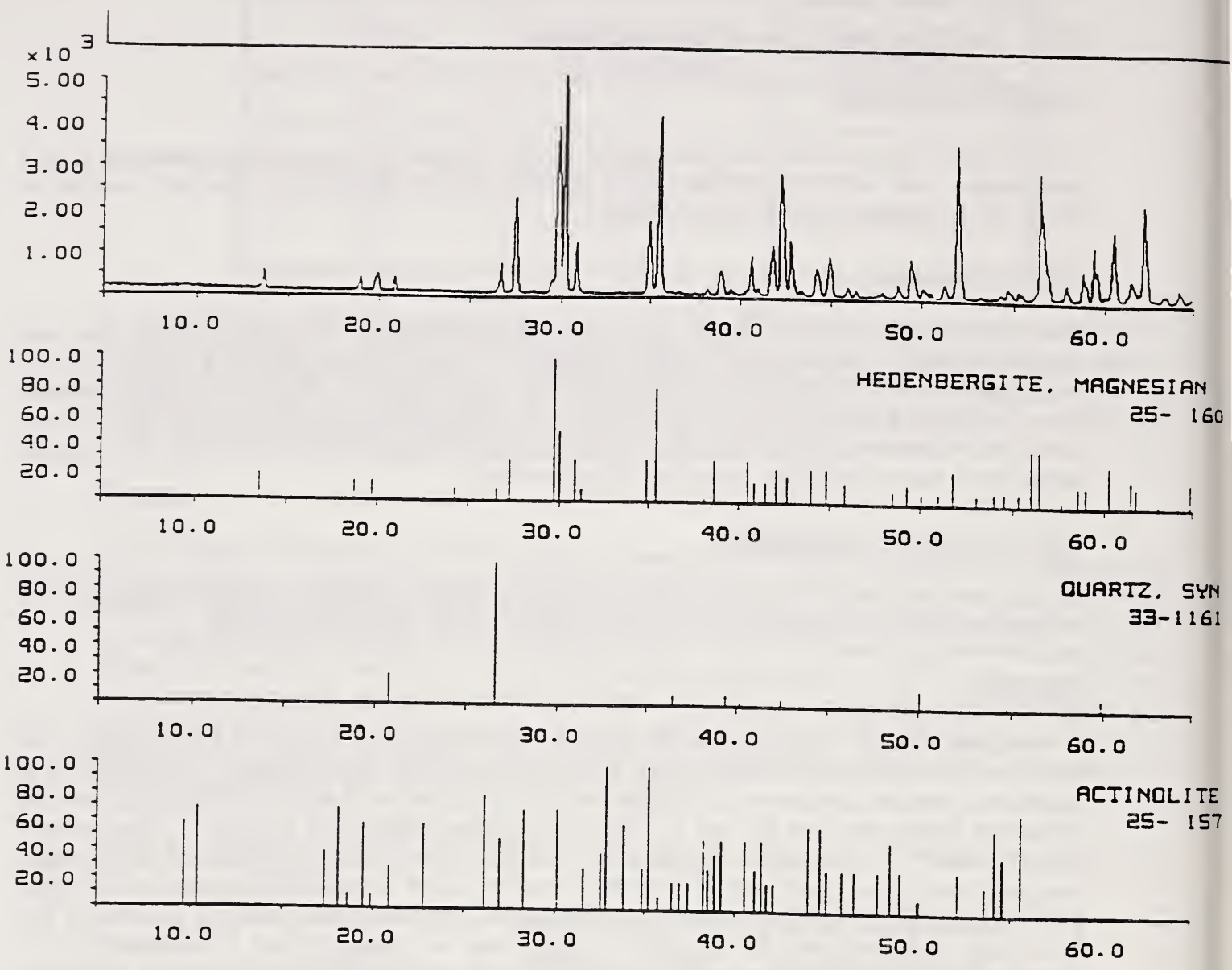


Figure 1. X-ray diffraction pattern obtained from unknown (top) and comparison to d-spacings for hedenbergite, quartz and actinolite.

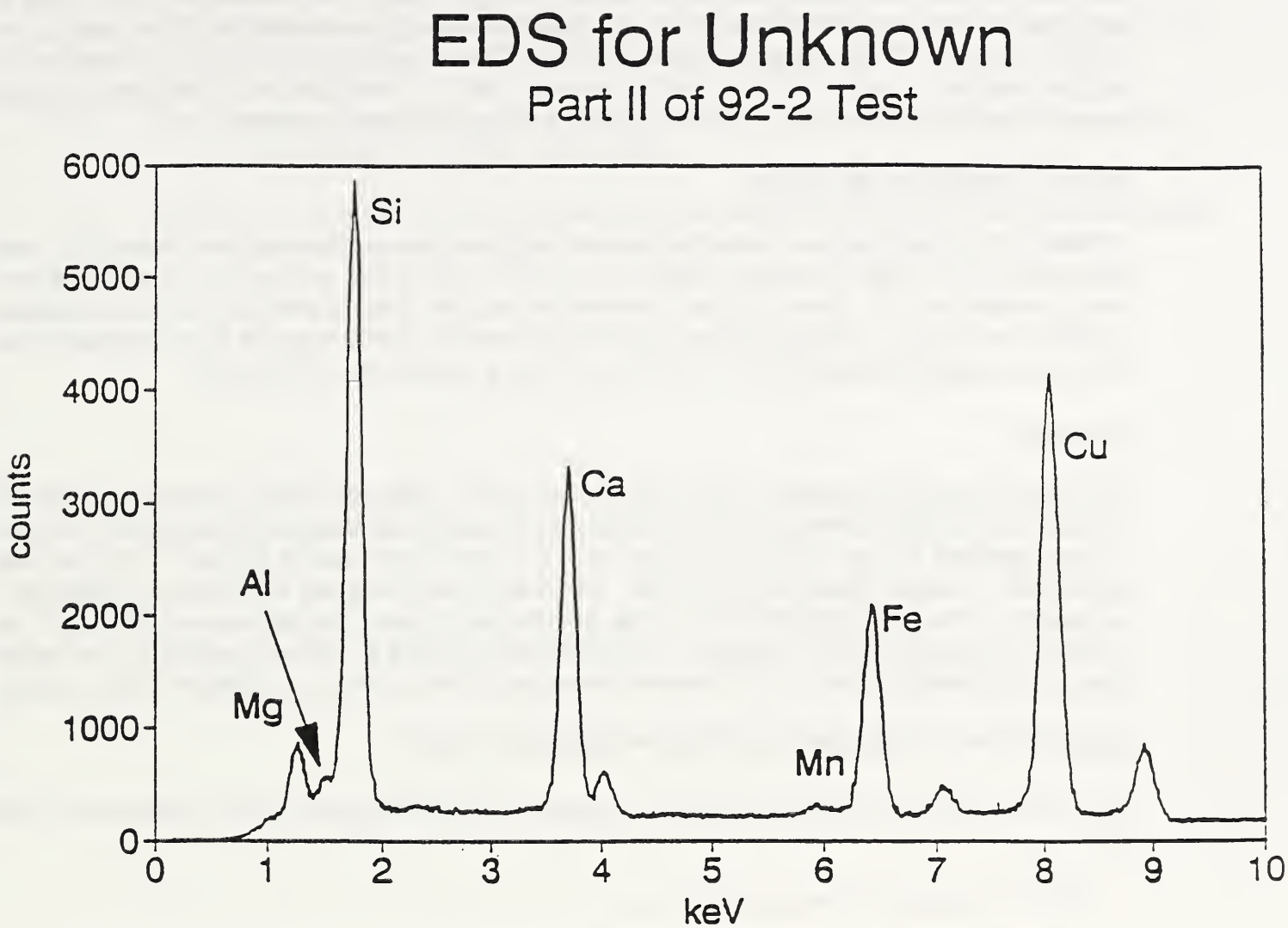


Figure 2. EDS spectrum obtained from the unknown. The elements are consistent with a magnesian hedenbergite.

PART III - GRID OPENING MEASUREMENT

Summary of test design

Laboratories were sent twenty 200-mesh, indexed, copper grids. The laboratories were asked to determine the areas of 20 grid squares on each of 20 grids using one of the procedures listed in the AHERA method (3) or in the "bronze" book (4). The laboratories were asked to describe the method used and to record the mean grid square area (μm^2) for each grid and to calculate the grand mean and grand standard deviation for all of the grid opening measurements.

Results submitted by laboratories

Of the one hundred and one laboratories submitting results, seventeen reported values that were apparently based on incorrect units (mm^2 and not μm^2). The values reported for these laboratories were corrected to μm^2. Four additional laboratories reported data in units that did not correspond to either mm^2 or μm^2. Excluding these four outlier cases, the grand mean of areas reported by the laboratories ranged from 8423 μm^2 to 10710 μm^2 with a mean value of 9652 μm^2.

Discussion

The grand mean of grid square areas for grids from twenty randomly chosen vials were determined by NIST using a light microscope, scanning electron microscope and image analysis system. The mean value determined for the vials was 9310 μm^2 with a standard deviation of 320 μm^2. The lower and upper 95% tolerance limits for 95% of the vial grand mean values are 8420 μm^2 and 10200 μm^2, respectively. For this proficiency test, values by the laboratories that fell between 8000 μm^2 and 11000 μm^2 were considered acceptable. Partial credit was given for values reported in mm^2 rather than the requested μm^2 units. As previously mentioned, four laboratories obtained outlier values.

DISCUSSION OF GRADING OF THE PROFICIENCY TEST

The number of errors assigned for incorrect responses to the proficiency test are summarized in Table 5.

Table 5. Summary of error assignments.

Section	# errors assigned	Description of error
Part I	1	≥ 5% difference between k-value reported by lab and calculated by RTI/NIST
Part II	1	incorrect identification
Part III	0.5	use of mm^2 instead of μm^2
	1	numeric value outside acceptable range

The results of the laboratory receiving this report are given in the attachment to the cover letter. A laboratory passed this proficiency test if it accumulated less than two errors. Eight laboratories did not pass this proficiency test.

REFERENCES

1. E.B. Steel, S. Turner, H.W. Berger, NVLAP Program Handbook for Airborne Asbestos Analysis, National Institute of Standards and Technology, NISTIR 89-4137, 1989.

2. NIST/NIH Desktop Spectrum Analyzer Program, NIST Standard Reference Database 36, Standard Reference Data, NIST, Gaithersburg, MD 20899.

3. Code of Federal Regulations, Asbestos-containing materials in schools; final rule and notice, 40 CFR Part 763, 41826-41905, 1987.

4. Chesson, J., Chatfield, E., Transmission Electron Microscopy Asbestos Laboratories Quality Assurance Guidelines, Environmental Protection Agency, EPA 560/5-90-002.