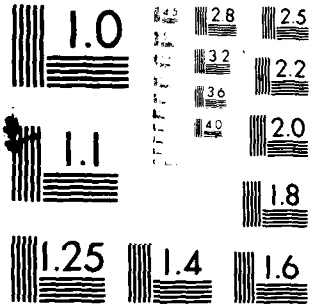
AD-A110 084　DEFENCE RESEARCH ESTABLISHMENT PACIFIC VICTORIA (BRIT--ETC　F/G 13/8
CHARACTERIZATION AND DETERMINATION OF ASBESTOS IN INDUSTRIAL PR--ETC(U)
DEC 80　K I MCRAE, C A WAGGONER
UNCLASSIFIED　DREP-TM-80-10　NL

1 of 1
AD
A110084

END
DATE
FILMED
3 82
DTIC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963 A

PACIFIC

Victoria, B.C.

Technical Memorandum 80-10

CHARACTERIZATION AND DETERMINATION OF ASBESTOS IN INDUSTRIAL PRODUCTS

[illegible]

[illegible]

This information is furnished with the express
understanding that proprietary and patent rights
will be protected.

DEFENCE RESEARCH ESTABLISHMENT PACIFIC
VICTORIA, B.C.

DREP -TM-

Technical Memorandum 80-10

CHARACTERIZATION AND DETERMINATION OF ASBESTOS IN INDUSTRIAL PRODUCTS

by K.I. McRae and C.A. Waggoner

December 1980

APPROVED BY:

Chief

RESEARCH AND DEVELOPMENT BRANCH
DEPARTMENT OF NATIONAL DEFENCE
CANADA

403 516

Abstract

The positive identification of asbestos in industrial products frequently requires the application of more than one method of analysis. Phase contrast optical microscopy, a standard method of asbestos analysis, is often inadequate, particularly if interfering fibres are present. Characterization and determination of asbestos is facilitated by the complementary application of X-ray diffractometry and scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEMEDS). Identification of asbestos type and semi-quantitative estimation of content is often possible in bulk specimens by X-ray diffractometry. In the event of ambiguous results, definitive qualitative identification may be achieved by SEMEDS analysis based on the determination of characteristic ratios of constituent elements, including magnesium, silicon, iron, sodium and calcium, relative to standard asbestos fibres. Details of analytical procedures are described.

A

INTRODUCTION

Asbestos is the name given to several fibrous materials belonging to the serpentine and the amphibole mineral groups. The asbestiform minerals in the amphibole group include amosite, anthophyllite, crocidolite, tremolite and actinolite, while chrysotile is the only asbestos mineral in the serpentine group. These minerals have attained considerable commercial importance because of their extraordinary thermal stability, resistance to strong chemicals, and electrical insulation properties. For many years it has been known that exposure to asbestos may induce cancers in the lungs, stomach and esophagus. With additional evidence of the toxicity and lethality of products containing asbestos, concern for those in the workplace has created the need for rapid and definitive methods of identifying and monitoring the various mineral forms. The techniques most commonly utilized for this purpose include X-ray diffraction (XRD)[1-4], scanning electron microscopy (SEM) in combination with energy dispersive spectrometry (EDS)[5-13], transmission electron microscopy (TEM) with selected area electron diffraction (SAED)[14-17] and polarized light microscopy[18]. Each of these techniques has some limitation which may not permit a definitive analysis, depending upon the type and quantity of specimen available. X-ray diffraction analysis of a bulk material requires a larger amount of material and often produces uncertain results if asbestos is present in low concentrations. The SEM-EDS and light microscopy techniques are not able to identify the smallest fibrils of chrysotile (300-400 Å). Selected area electron diffraction patterns are often of too low quality for very small fibrils. It should, however, be possible to gain enough information for the identification of the material by the judicious use of a combination of two or more of the above techniques. Chemical and morphological information obtained by the application of SEM-EDS, combined with the unique structural information provided by XRD or TEM-SAED should produce a positive identification of asbestos fibres in most instances.

DREP has been periodically requested to carry out asbestos determinations in various products used by the Canadian Armed Forces. It is usually necessary to at least identify the type of asbestos mineral present and, occasionally, to estimate the concentration of asbestos in an industrial product. The approach most often taken at DREP involves a preliminary analysis of the bulk material by an X-ray diffraction procedure, followed by SEM-EDS analysis similar to that proposed by Langer et al[7,9] and other workers. This combination provides sufficient morphological, chemical and structural information to qualitatively identify fibre type. The X-ray diffraction technique may be quantified by calibration with suitable standards, and estimates of asbestos concentration can be obtained by the SEM-EDS method by counting fibres which have a composition corresponding to a known asbestos type.

Composition and Properties of the Asbestos Minerals

Six minerals are typically classed as asbestos: chrysotile in the serpentine group and five minerals from the amphibole group. By far the most commercially important and most commonly encountered form is chrysotile, which constitutes approximately ninety-five percent of the total world production of asbestos. The principal sources of chrysotile are Canada and Rhodesia and their respective varieties exhibit only minor differences in morphology and physical properties. The amphibole group contains the minerals crocidolite, amosite, anthophyllite, tremolite and actinolite. All of the asbestos minerals in this group contain a common $(Si_8O_{22}(OH)_2)^{-14}$ structure, to which variable amounts of Mg, Fe, Na and Ca are substituted to form the characteristic composition of each. Structural formulae and chemical compositions of the asbestos minerals are presented in Table 1. It should be noted that the chemical compositions of the individual forms are quite unique within the asbestos group and it is possible to distinguish each type on the basis of composition.

The morphology and physical properties of the five common forms of asbestos are summarized below:

(1) Chrysotile (Figure 1). The most prominent characteristic of this asbestos type is a tendency to form in bundles of smaller fibres or "fibrils" (usually 300-400 Å in diameter). This tends to give the fibres a rough surface or a rope-like appearance. Individual fibrils usually have a hollow central shaft, a characteristic which is only readily observable in the TEM. The fibres tend to be quite fine and long, with a length-to-width ratio commonly greater than 100.

Table 1

Chemical Compositions of Asbestos Minerals

Mineral	%Si	%Mg	%Fe	%Na	%Ca	%Mn
Chrysotile $Mg_6(Si_4O_{10}(OH)_8)$	18.2[a] (20)[b]	32 (26)	2.6 (1)	0.08 (-)	- (-)	0.05 (-)
Crocidolite $Na_2Fe_5(Si_8O_{22}(OH)_2)$	23.9 (24)	3.6 (1)	27 (30)	4.4 (2)	- (-)	0.09 (-)
Anthophyllite $(Mg,Fe)_7(Si_8O_{22}(OH)_2)$	27.3 (22)	24 (13)	4.4 (8)	- (-)	- (-)	0.12 (-)
Amosite $(Fe,Mg)_7(Si_8O_{22}(OH)_2)$	24.9 (23)	1.1 (2)	28 (34)	- (-)	- (-)	1.5 (1)
Tremolite $Ca_2Mg_5(Si_8O_{22}(OH)_2)$	(27)	(14)	(-)	- (-)	(9)	(2)
Actinolite $Ca_2(Mg_2Fe)_5(Si_8O_{22}(OH)_2)$	(26)	(13)	(5)	(-)	(9)	(1)

(a) Data compiled by neutron activation, gravimetric, flame emission and atomic absorption analyses in Reference 19.

(b) Data compiled by X-ray microanalysis with Philips EM 300 TEM with EDS in Reference 17.

(2) Amosite (Figure 2). Fibrous masses are grey to white in colour and exhibit a much straighter fibre in comparison to chrysotile. Fibres are coarse and may be very long (up to 10-11 inches), and usually exhibit a rectangular cross-section. A common morphological characteristic of all forms of amphibole asbestos is the formation of cleavage planes with angles of 56° and 124° between the planes. Cleavage, where visible, is an important morphological feature of all types of amphibole asbestos.

(3) Crocidolite. This mineral form is blue in colour. Fibres are long and coarse and circular in cross-section, and are stronger than chrysotile.

(4) Anthophyllite. This variety is grey in colour. Fibres are typically very short and brittle, and are usually milled to a length of ∿1/10" due to extreme inflexibility.

(5) Tremolite. A characteristic feature of this form is the occurrence of long lath-like crystals with a length-to-width ratio typically ranging from 10 to 20.

(6) Actinolite. This variety is chemically similar to tremolite, but fibres are smaller in width and are very brittle. The mineral is useful only as a cheap filler.

Specimen Preparation

The methods of specimen preparation necessary for the examination of asbestos fibres by the two techniques of principal interest, i.e. SEM-EDS and X-ray diffractometry, are to a large extent in conflict. The SEM technique requires a representative dispersion of both the fibres and the matrix material, with only a minimum disturbance in the fibre length and morphology, whereas the X-ray diffraction technique demands a very finely ground (<1 μm) and homogenous mixture of both the asbestos fibres and the matrix components. For this reason, after a preliminary examination of the sample in a light microscope to determine the major morphological characteristics, the sample is divided into two appropriate portions for analysis by the two techniques. The larger portions is then prepared for X-ray diffraction analysis by grinding in a tungsten carbide ball mill for about ten minutes. The resulting fibre size is sufficiently small for the identification of major amounts of asbestos by X-ray diffractometry. Sieving of the sample is usually avoided, since it may isolate the longer asbestos fibres and thus change their distribution in the specimen. It has been noted that preferred orientation effects are not usually seen for most asbestos forms and that the grinding procedure is adequate, as outlined, for qualitative identification purposes.

Preparation of a representative distribution of asbestos fibres and matrix components with minimal alteration of the fibre size is required for SEM analysis. Selective collection of asbestos fibres should be avoided. Initially, the sample may require light grinding to break the larger fibrous masses and to provide a more homogenous mixture of asbestos fibres and matrix material. The sample is then dispersed in a small volume of acetone and a few drops of this suspension are transferred to the surface of a polished, graphite planchet. The acetone is allowed to evaporate on the planchet surface, which is then coated by evaporation with approximately 100 Å of graphite to prevent charging effects in the SEM and to facilitate the subsequent X-ray microanalysis.

In some instances it may be desirable to eliminate the matrix components. This must be done in such a way that little or no change in the asbestos fibre composition or morphology is observable. The technique chosen will be largely dependent upon the nature of the matrix material. An organic matrix may be eliminated by low-temperature ashing or by dissolution in a suitable solvent. It should be noted, however, that chemical modification of the fibre may occur in strong acid or alkaline solutions, and that the stability of the minerals in such chemical environments follows the approximate order:

Anthophyllite $\geq$ crocidolite >> amosite >>> chrysotile[7].

In many asbestos-containing products, refractory components such as silica or diatomaceous earth compose the bulk of the matrix. In such cases, it is virtually impossible to selectively remove the matrix components without alteration of the asbestos fibres. For samples such as these, all matrix components must be known since interference by the matrix in the analysis of the asbestos fibre will be unavoidable and must be accounted for.

Several alternative specimen preparation techniques have also been proposed which produce a distribution of fibres such that they are essentially sandwiched between two layers of evaporated carbon on a copper grid[6,10,11]. This procedure is far superior for quantitative estimation of asbestos fibre content by the SEM-EDS technique, since the original collected distribution is not altered and the X-ray continuum emission is greatly diminished by the removal of the graphite substrate. For the types of sample considered here, where accurate fibre counts are not necessary and fibres are not usually broken down to their smallest dimension (300 Å), this more time-consuming approach is not considered necessary for simple qualitative identification of the fibre type.

X-Ray Diffraction Analysis

If the asbestos component is present in sufficient amount, X-ray diffraction analysis alone may prove adequate to identify the asbestos mineral in the product to be examined. The major lines of the X-ray diffraction patterns and their interplanar spacings - d(Å) - and relative intensities (I/I_1) have been measured previously[19] and are listed in Table 2. Since it is

Table 2

Principal Diffraction Lines of UICC Asbestos Standards

Mineral	d(Å)	I/I_1
Amosite	8.26	100
	3.27	36
	3.07	52
	2.77	43
Anthophyllite	9.50	100
	8.40	26
	4.58	55
	3.25	47
	3.13	58
	5.06	64
Crocidolite	8.43	100
	4.51	26
	3.43	26
	3.11	41
	2.72	50
	2.61	20
	2.54	36

Table 2 (continued)

Mineral	d(Å)	I/I$_1$
Chrysotile	7.38	100
	4.55	27
	3.66	57
	2.46	24
	1.54	21

not always possible to separate or to concentrate the asbestos fibres in the matrix, it is usually necessary to produce a homogenous mixture of the entire product. In these cases, the reliability of this method to identify positively the mineralogical forms is inhibited by two factors; diffraction lines of the asbestos component and of the matrix may fortuitously overlap and the concentration of asbestos in the specimen may not be sufficient to produce a diffraction pattern which has an adequate number of lines for qualitative identification. Figure 3 shows a typical X-ray diffractogram of chrysotile, obtained from a product which until recently was in local service (asbestos fibre rope). The concentration of chrysotile in the sample is well in excess of 50% by weight and does not contain a matrix component which may complicate the diffraction pattern. The resulting identification of chrysotile as a major component in this sample would be definite, without resorting to a further analysis by a complementary technique such as SEM-EDS. A less conclusive example may be seen in the diffractogram of the abrasive component in a non-slip epoxy deck coating (Figure 4). It is immediately noted, after reference to Table 2, that the majority of the specimen is some component other than an asbestos mineral (subsequently identified as α-quartz, SiO_2). Since the asbestos forms tend to have rather simple diffraction patterns which contain only one or two strong lines, the small peaks present in the diffractogram are not adequate to unambiguously identify chrysotile in this specimen. Furthermore, it is usually necessary to identify all phases present in the matrix in order to eliminate the possibility of a fortuitous occurrence of a matrix diffraction line at the same position as the major line of an asbestos mineral. Although the presence of chrysotile is strongly suspected, this example illustrates that X-ray diffraction analysis of the bulk specimen often does not supply enough information for the positive identification of asbestos. The required information may be obtained by further investigation with other techniques, such as SEM-EDS.

Scanning Electron Microscopic Analysis

Valuable information may be obtained from individual fibres of asbestos by combining two unique capabilities of the scanning electron microscope (SEM) coupled with energy dispersive spectrometry: high resolution, high magnification imaging and the ability to perform X-ray microanalysis. Asbestos fibres with diameters as small as 0.1 μm can be easily located and imaged if the substrate is sufficiently flat and featureless. The SEM has the unique ability of obtaining high magnification with a large depth of fields, thus decreasing the possibility of overlooking many of the smaller fibres. Still higher magnification may be obtained in the transmission electron microscope (TEM), but the ability to view the smallest chrysotile fibrils (300-400 Å) is seldom required for the identification of this mineral in products which, by nature, contain relatively large fibres (0.1-1 μm diameter). The resolution of the SEM is adequate for the elucidation of many of the finer morphological characteristics peculiar to the particular asbestos fibres that are not otherwise visible by light microscopy.

Perhaps of even greater utility is the ability of the SEM to obtain easily and rapidly qualitative or semi-quantitative chemical information from individual fibres by the use of either energy dispersive (EDS) or wavelength dispersive (WDS) X-ray spectrometry. Several methods for the identification of the various asbestos minerals have already been established[6-12]. Usually full quantitative analysis of an individual fibre is impractical and some form of elemental intensity ratio is used to characterize the chemical composition of the asbestos fibre. Pattnaik and Meakin[7,11,12] consider the five element intensity ratio ("A-ratio") for the elements Si, Mg, Fe, Na and Ca as defined by:

$$Ai = I_i/I_i^o \Big/ \sum_i I_i/I_i^o \qquad (1)$$

where I_i is the intensity of element i in the sample and I_i is the intensity obtained for pure element i, under the same analytical conditions. If an EDS system is used for the acquisition of X-ray data, comparison to the pure element intensity should not be necessary since relative elemental intensities under a given set of working conditions are essentially invariant for long periods of time. Therefore, a simple intensity extraction with background subtraction may be used to calculate net elemental intensities and a modified A-ratio may be defined as follows:

$$A_i^* = I_i \sum_i I_i = \text{peak intensity }\% \qquad (2)$$

Although the operating conditions of the electron beam and the specimen-detector geometry must be selected and reproduced for each analysis, the normalization of the X-ray intensities obviates the need for close monitoring of the electron beam current. Values of A_i^* were measured for the asbestos minerals chrysotile, crocidolite, amosite and anthophyllite and these data are presented in Table 3. Extraction of net intensities from the recorded spectrum was carried out by two methods:

(1) simple background subtraction by spectrum stripping, integration of peaks in a region of width 2σ and a simple iterative overlap correction (DREP program - "Asbestos").

Table 3

Measured A-Ratios for Asbestos Minerals

Mineral		Si	Fe	Mg	Na
Chrysotile	(1)	0.547 ± 1.4%	0.024 ± 56%	0.427 ± 4.1%	--
	(2)	0.573 ± 1.1%	0.021 ± 40%	0.404 ± 3.4%	--
	(3)	0.583	0.023	0.393	--
Crocidolite	(1)	0.559 ± 3.9%	0.371 ± 6.8%	0.040 ± 37%	0.024 ± 31%
	(2)	0.584 ± 5.0%	0.379 ± 10.4%	0.011 ± 47.2%	0.021 ± 66%
	(3)	0.540	0.388	0.032	0.039
Amosite	(1)	0.520 ± 6.3%	0.425 ± 11.8%	0.047 ± 38%	--
	(2)	0.533 ± 4.9%	0.425 ± 8.4%	0.038 ± 26%	--
	(3)	0.532	0.423	0.045	--
Anthophyllite	(1)	0.665 ± 2.7%	0.073 ± 39%	0.256 ± 11.4%	--
	(2)	0.689 ± 4.2%	0.065 ± 13.6%	0.241 ± 6.3%	--
	(3)	0.688	0.071	0.241	--

(1) A-ratios calculated by DREP program "Asbestos".

(2) A-ratios calculated by least squares fitting to stored spectral data ("Super ML").

(3) A-ratios estimated from relative elemental sensitivities and stoichiometric concentrations.

(4) All uncertainties are ±2σ relative, based on 12 determinations on separate fibres.

(2) multiple least squares fitting of the digitally filtered spectrum against standard pure element spectra (Tracor Northern program - "Super ML").
Although the first program is a less accurate means of intensity extraction, it can execute much faster than the multiple least squares fitting procedure. The increased speed of execution is of great benefit if a large number of fibres are to be identified. A comparison of the uncertainties in the A-ratio produced by each procedure indicates that large relative intensity variations may occur, most likely as a result of fibre inhomogeneity and variable fibre diameter, and not as a result of the intensity extraction procedure.

If sufficient asbestos minerals are not available, it is possible to at least estimate suitable values of A_i^* from the pure element intensities of the elements concerned, i.e. Si, Mg, Fe, Na and Ca. Table 3 also includes values of A_i^* which have been calculated using the stoichiometric concentration for these elements and estimated values of I_i, assuming no ZAF correction is necessary, i.e. $I_i = C_i \times I_i^o$.

It is noted that these estimated values compare quite well with the measured values of A_i^*, especially those generated by multiple least squares fitting. From this it may be inferred that most asbestos fibres are not sufficiently large to warrant corrections necessary for bulk specimens, i.e. ZAF correction. Acceptable results may be obtained by assuming a direct proportionality of concentration with relative intensity.

Conclusion

The utilization of two analytical techniques, X-ray diffractometry and X-ray microanalysis with a scanning electron microscope, produces structural, morphological and chemical information on common varieties of asbestos. This information suffices to discriminate the asbestos minerals from other mineralogical forms and to characterize asbestos minerals. X-ray diffractometry is capable of rapid identification of asbestos components in bulk materials, provided that sufficient sample is available. In some instances, the quantity of specimen may be limited or the concentration of asbestos in the sample is not sufficient to produce adequate diffraction information for the positive identification of asbestos. In these cases, individual fibres may be viewed and semi-quantitatively analyzed in the SEM in order to identify them by their morphological and chemical characteristics.

The asbestos minerals of interest have sufficiently unique compositions to permit the unambiguous identification of single fibres. Certain non-asbestos minerals may interfere with the analysis of the asbestos minerals; for example, the mineral diopside is similar in composition to chrysotile. However, the occurrence of similar minerals in the type of proprietary products commonly examined is thought to be rare and possible confusion with an asbestos mineral is unlikely. If, however, the analysis is limited to fibres which have morphological features characteristic of the asbestos minerals, all possible ambiguities in the identification are eliminated.

Bibliography

1. C. Plowman, "Determination of Minimum Detectable Amounts of Atmospheric Particulates by X-Ray Diffractometry", Analyst, 103, 982-985, 1978.
2. B.A. Lange and J.C. Haartz, "Determination of Microgram Quantities of Asbestos by X-Ray Diffraction: Chrysotile in Thin Dust Layers of Matrix Material", Anal. Chem., 51(4), 520-525, 1979.
3. A.L. Rickards, "Estimation of Trace Amounts of Chrysotile Asbestos by X-Ray Diffraction", Anal. Chem., 44(11), 1872-1873, 1972.
4. J.V. Crable, "Quantitative Determination of Chrysotile, Amosite and Crocidolite by X-Ray Diffraction", Am. Ind. Hyg. Assoc. J., 27, 293, 1966.
5. J.R. Millette and E.F. McFarren, "EDS of Waterborne Fibres in TEM, SEM and STEM", Scanning Electron Microscopy/1976, O. Johari (ed.), IIT Research Institute, Chicago, Il., 452-460, 1976.
6. A. Pattnaik and J.D. Meakin, "Development of Scanning Electron Microscopy for Measurement of Airborne Asbestos Concentrations", Scanning Electron Microscopy/1977, O. Johari (ed.), IIT Research Institute, Chicago, Il., 442-449, 1977.
7. A.M. Langer, I.B. Rubin, I.J. Selikoff and F.D. Pooley, "Chemical Characterization of Uncoated Asbestos Fibres from the Lungs of Asbestos Workers by Electron Microprobe Analysis", J. Histochem. and Cytochem., 20(9), 735-740, 1972.
8. R.E. Ferrell, G.G. Paulson and C.W. Walker, "Evaluation of an SEM-EDS Method for Identification of Chrysotile", Scanning Electron Microscopy/1975, O. Johari (ed.), IIT Research Institute, Chicago, Il., 538-548, 1975.
9. A.M. Langer, I.B. Rubin,and I.J. Selikoff, "Chemical Characterization of Asbestos Body Cores by Electron Microprobe Analysis", J. Histochem. and Cytochem., 20(8), 723-734, 1972.
10. A. Pattnaik and J.D. Meakin, "Development of Scanning Electron Microscopy for Measurement of Airborne Asbestos Concentrations", U.S. Environmental Protection Agency Report EPA-650/2-75-029, 1975.

Bibliography (continued)

11. A. Pattnaik and J.D. Meakin, "Development of an Instrumental Monitoring Method for Measurement of Asbestos Concentrations In or Near Sources", U.S. Environmental Protection Agency Report EPA-650/2-73-016, 1973.

12. A.L. Rickards, "Estimation of Submirogram Quantities of Chrysotile Asbestos by Electron Microscopy", Anal. Chem. 45(4), 809-811, 1973.

13. C.J. Maggiore and I.B. Rubin, "Optimization of an SEM X-Ray Spectrometer System for the Identification and Characterization of Ultramicroscopic Particles", Scanning Electron Microscopy/1973, O. Johari (ed.), IIT Research Institute, Chicago, Il., 130-136, 1973.

14. P.K. Mueller, A.E. Alcocer, R.L. Stanley and G.R. Smith, "Asbestos Fibre Atlas", U.S. Environmental Protection Agency Report EPA-650/2-75-036, 1975.

15. A.V. Samudra, "Optimum Procedure for Asbestos Fibres Identification from Selected Area Electron Diffraction Patterns in a Modern Analytical Electron Microscope Using Tilted Specimens", Scanning Electron Microscopy/1977, O. Johari (ed.), IIT Research Institute, Chicago, Il., 385-391, 1977.

16. C.O. Ruud, C.S. Barrett, P.A. Russell and R.A. Clark, "Selected Area Electron Diffraction and Energy Dispersive X-Ray Analysis for the Identification of Asbestos Fibres, a Comparison", Micron, 7, 115-132, 1976.

17. D.R. Beaman and D.M. File, "Quantitative Determination of Asbestos Fibre Concentrations", Anal. Chem., 48(1), 101-110, 1976.

18. W.C. McCrone, "Identification of Asbestos by Polarized Light Microscopy", The Microscope, 25, 251-264, 1977.

19. V. Timbrell, "Characteristics of the UICC Standard Reference Samples of Asbestos", Pneumoconiosis Proceedings International Conference, Johannesburg, 1969.

Figure 1. Typical Chrysotile Asbestos Fibres. (900X/2600X)

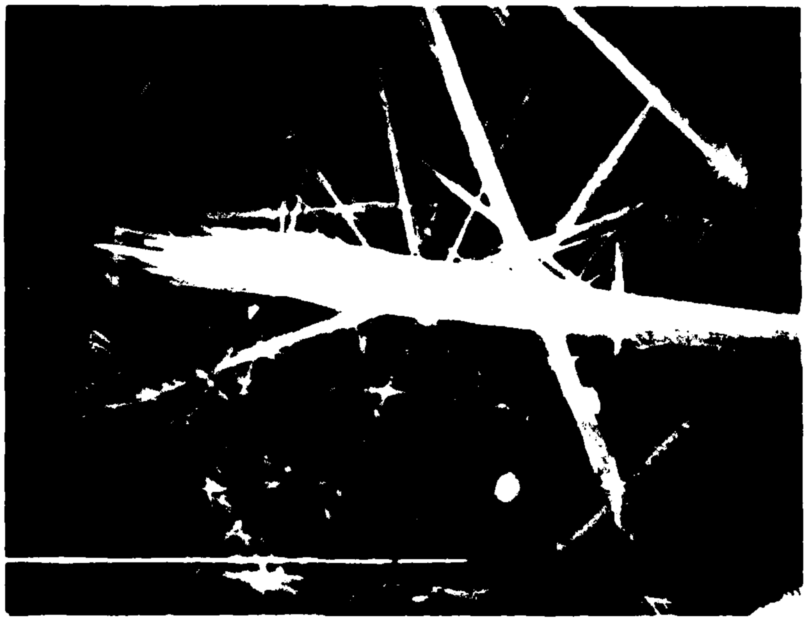
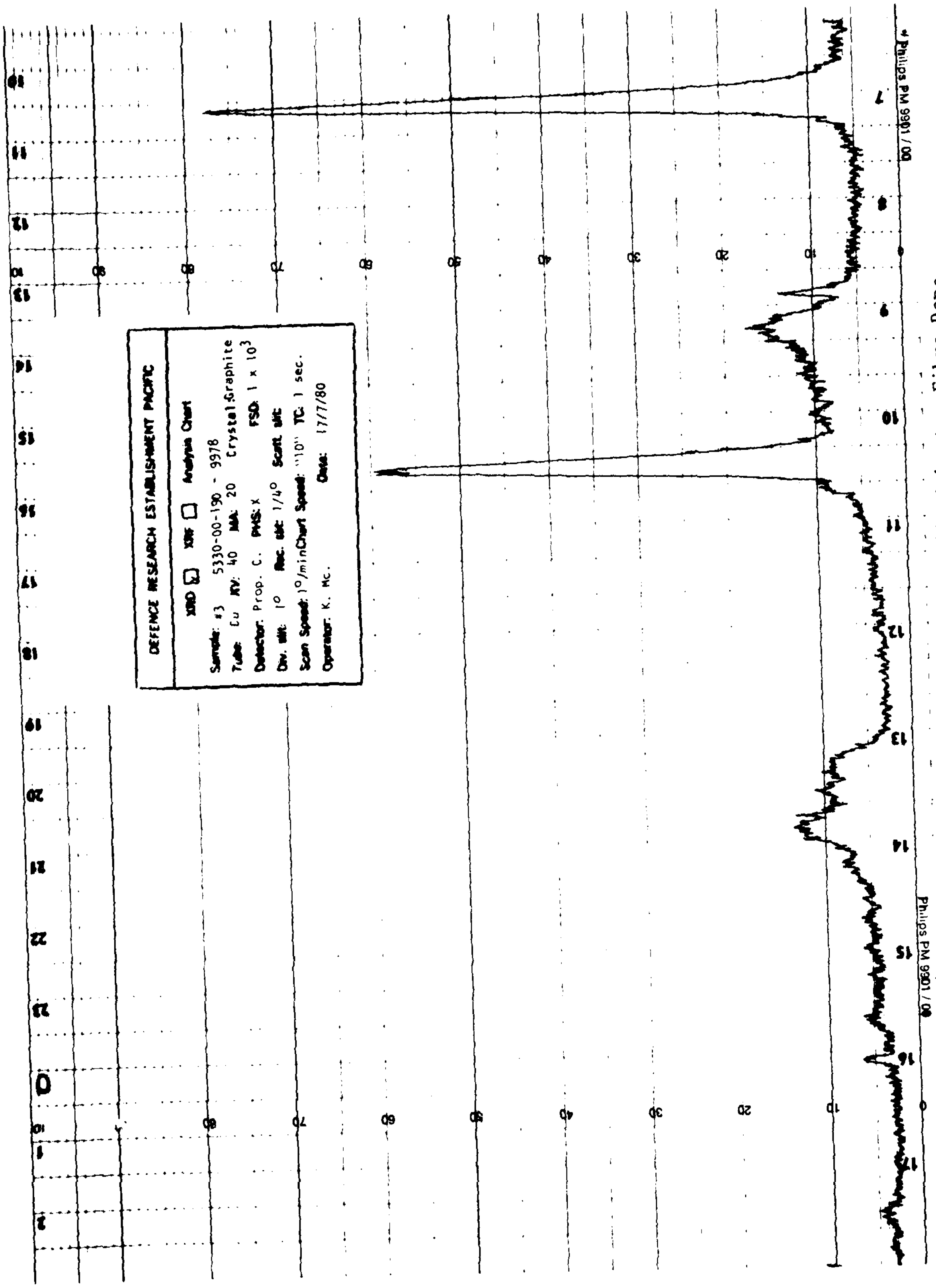


Figure 2. Typical Amosite Asbestos Fibres. (1200X)



DEFENCE RESEARCH ESTABLISHMENT PACIFIC

XRD ☒ XRF ☐ Analysis Chart

Sample: #3 5330-00-190 - 9978

Tube: Cu KV: 40 MA: 20 Crystal:Graphite

Detector: Prop. C. PHS: X FSD: 1 x 10^3

Div. slit: 1° Rec. slit: 1/4° Scatt. slit:

Scan Speed: 1°/min Chart Speed: "10" TC: 1 sec.

Operator: K. Mc. Date: 17/7/80

Figure 3. X-Ray Diffractogram of Chrysotile Asbestos Fibre Rope.

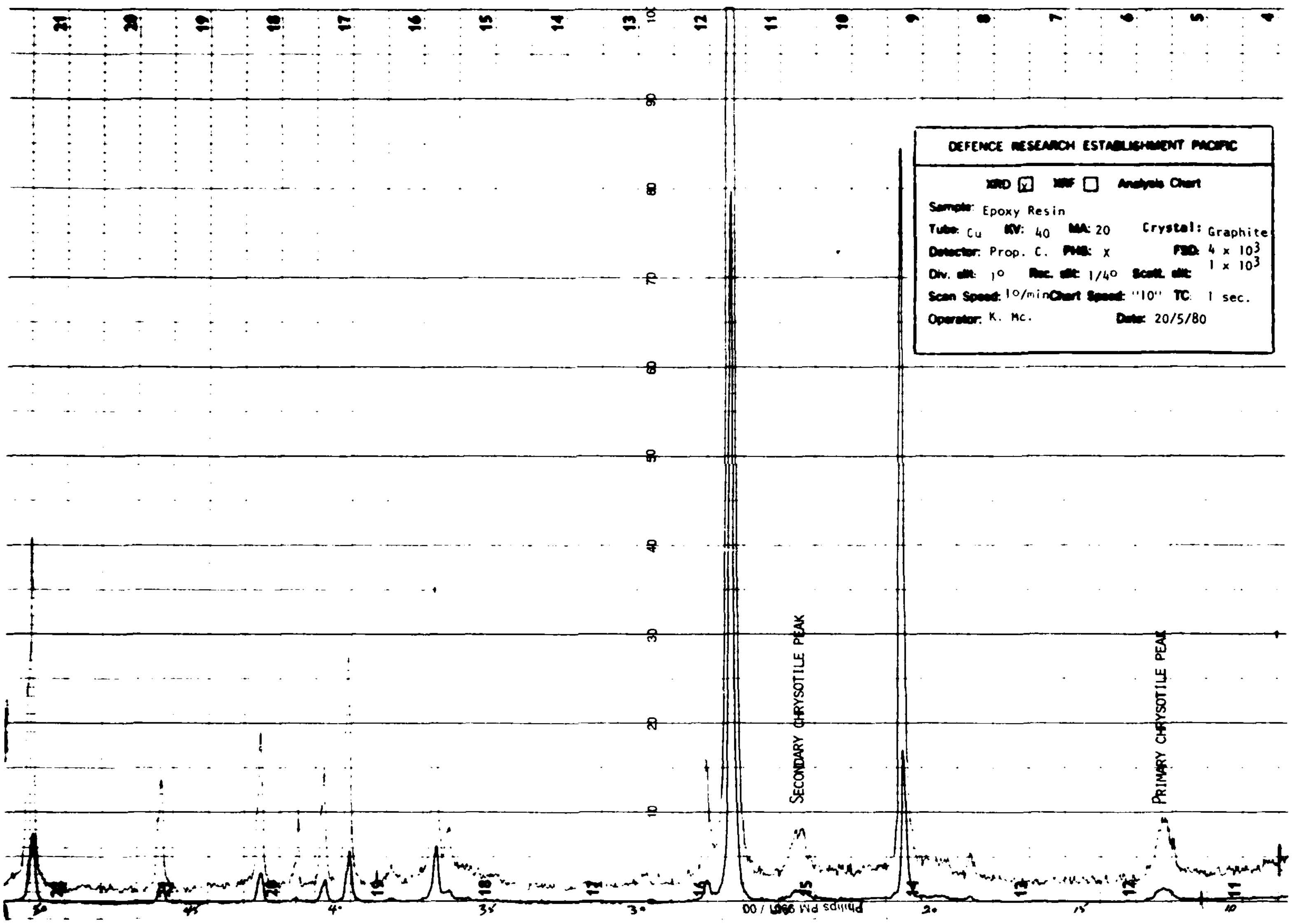


Figure 4. X-Ray Diffractogram Illustrating Small Chrysotile Asbestos Content in Non-Slip Epoxy Deck Coating with Quartz Matrix.

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall document is classified)

1 ORIGINATING ACTIVITY

DEFENCE RESEARCH ESTABLISHMENT PACIFIC

2a. DOCUMENT SECURITY CLASSIFICATION

Unclassified

2b GROUP

3. DOCUMENT TITLE

CHARACTERIZATION AND DETERMINATION OF ASBESTOS IN INDUSTRIAL PRODUCTS

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Technical Memorandum

5. AUTHOR(S) (Last name, first name, middle initial)

K.I. McRae and C.A. Waggoner

6 DOCUMENT DATE

December 1980

7a. TOTAL NO. OF PAGES

21

7b. NO. OF REFS

8a. PROJECT OR GRANT NO

26C

9a ORIGINATOR'S DOCUMENT NUMBER(S)

Technical Memorandum 80-10

8b. CONTRACT NO

9b. OTHER DOCUMENT NO.(S) (Any other numbers that may be assigned this document)

10. DISTRIBUTION STATEMENT

Unlimited

11. SUPPLEMENTARY NOTES

12. SPONSORING ACTIVITY

13. ABSTRACT

The positive identification of asbestos in industrial products frequently requires the application of more than one method of analysis. Phase contrast optical microscopy, a standard method of asbestos analysis, is often inadequate, particularly if interfering fibres are present. Characterization and determination of asbestos is facilitated by the complementary application of X-ray diffractometry and scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEMEDS). Identification of asbestos type and semi-quantitative estimation of content is often possible in bulk specimens by X-ray diffractometry. In the event of ambiguous results, definitive qualitative identification may be achieved by SEMEDS analysis based on the determination of characteristic ratios of constituent elements, including magnesium, silicon, iron, sodium and calcium, relative to standard asbestos fibres. Details of analytical procedures are described.

DSIS
70-070

KEY WORDS

Asbestos
Characterization
Determination
Industrial Products
X-Ray Diffractometry
Scanning Electron Microscopy
Energy-Dispersive X-Ray Spectrometry

INSTRUCTIONS

1. ORIGINATING ACTIVITY: Enter the name and address of the organization issuing the document.

2a. DOCUMENT SECURITY CLASSIFICATION Enter the overall security classification of the document including special warning terms whenever applicable.

2b. GROUP Enter security reclassification group number. The three groups are defined in Appendix 'M' of the DRB Security Regulations.

3. DOCUMENT TITLE: Enter the complete document title in all capital letters. Titles in all cases should be unclassified. If a sufficiently descriptive title cannot be selected without classification, show title classification with the usual one-capital-letter abbreviation in parentheses immediately following the title.

4. DESCRIPTIVE NOTES: Enter the category of document, e.g. technical report, technical note or technical letter. If appropriate, enter the type of document, e.g. interim, progress, summary, annual or final. Give the inclusive dates when a specific reporting period is covered.

5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the document. Enter last name, first name, middle initial. If military, show rank. The name of the principal author is an absolute minimum requirement.

6. DOCUMENT DATE: Enter the date (month, year) of Establishment approval for publication of the document.

7a. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7b. NUMBER OF REFERENCES: Enter the total number of references cited in the document.

8a. PROJECT OR GRANT NUMBER: If appropriate, enter the applicable research and development project or grant number under which the document was written.

8b. CONTRACT NUMBER: If appropriate, enter the applicable number under which the document was written.

9a. ORIGINATOR'S DOCUMENT NUMBER(S) Enter the official document number by which the document will be identified and controlled by the originating activity. This number must be unique to this document

9b. OTHER DOCUMENT NUMBER(S) If the document has been assigned any other document numbers (either by the originator or by the sponsor), also enter this number(s).

10. DISTRIBUTION STATEMENT Enter any limitations on further dissemination of the document, other than those imposed by security classification, using standard statements such as

    (1) "Qualified requesters may obtain copies of this document from their defence documentation center."

    (2) "Announcement and dissemination of this document is not authorized without prior approval from originating activity."

11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.

12. SPONSORING ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring the research and development. Include address.

13. ABSTRACT Enter an abstract giving a brief and factual summary of the document, even though it may also appear elsewhere in the body of the document itself. It is highly desirable that the abstract of classified documents be unclassified Each paragraph of the abstract shall end with an indication of the security classification of the information in the paragraph (unless the document itself is unclassified) represented as (TS), (S), (C), (R), or (U).

    The length of the abstract should be limited to 20 single-spaced standard typewritten lines, 7½ inches long.

14. KEY WORDS. Key words are technically meaningful terms or short phrases that characterize a document and could be helpful in cataloging the document. Key words should be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context.

DA
FILM