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ASBESTOS CHARACTERIZATION USING
SCANNING ELECTRON MICROSCOPY/LIGHT
ELEMENT X-RAY SPECTROMETRY

G.C. Fisher - R.M. Morchat

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**ASBESTOS CHARACTERIZATION USING
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Approved by R.T. Schmitke
Director / Technology Division

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**Defence
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ABSTRACT

The Defence Research Establishment Atlantic (DREA) has traditionally used scanning electron microscopy (SEM) coupled with energy dispersive x-ray (EDX) spectrometry to identify asbestos fibres in solid insulating materials. This analysis typically utilizes fibre morphology to determine the presence of asbestiform fibers and EDX analysis to characterize asbestos type. The characterization is accomplished by comparison of the relative amounts of magnesium, silicon and iron present in the fibres. The EDX detector traditionally used in asbestos characterization employs a protective beryllium shield that effectively blocks the passage of low energy x-rays. Thus characteristic x-rays from "light elements" (those below sodium in atomic weight) are not detected.

Recently, DREA acquired a commercial EDX detector that employs a polymer shield that allows for detection of x-rays from elements as low as boron in atomic weight. This report summarizes results of a study on the effects of using a "light element" detector on characterization of both asbestos standards and commercial asbestos-containing insulating material. The study showed that "light element" SEM/EDX can be used to characterize asbestos fibers in bulk insulations.

RÉSUMÉ

Le laboratoire du chantier naval du CRDA a habituellement utilisé la microscopie électronique à balayage (SEM) et la spectrométrie des rayons X à dispersion d'énergie (EDX) pour reconnaître les fibres d'amiante dans les matériaux isolants solides. Cette analyse utilise généralement la morphologie des fibres pour déterminer la présence de fibres et l'analyse EDX pour caractériser le type d'amiante. La caractérisation est réalisée par la comparaison des quantités relatives de magnésium, de silicium et de fer présentes dans les fibres. Le détecteur EDX qui était utilisé habituellement pour la caractérisation des fibres comprend un écran de protection en béryllium qui bloque efficacement les rayons X de faible énergie. Par conséquent, les rayons X caractéristiques des "éléments légers" (poids atomique inférieur à celui du sodium) ne sont pas détectés.

Récemment, le laboratoire de l'arsenal maritime du CRDA a fait l'acquisition d'un détecteur EDX commercial comprenant un écran de polymère qui permet la détection des rayons X émis par des éléments de poids atomique aussi léger que celui du bore. Le présent rapport résume les résultats d'une étude relative aux effets de l'utilisation d'un détecteur d' "éléments légers" sur la caractérisation d'étalons d'amiante et de matériau isolant commercial contenant de l'amiante.

EXECUTIVE SUMMARY

There have been many analytical techniques employed for the identification of asbestos fibres in materials, including polarized light microscopy, x-ray diffraction, and infrared spectrometry. The Defence Research Establishment Atlantic (DREA) has traditionally used scanning electron microscopy (SEM) coupled with energy dispersive x-ray (EDX) spectrometry to identify asbestos fibres in solid insulating materials. This analysis typically utilizes fibre morphology to determine the presence of asbestiform fibers and EDX analysis to characterize asbestos type. The characterization is accomplished by comparison of the relative amounts of magnesium, silicon and iron present in the fibres. The EDX detector traditionally used in asbestos characterization employs a protective beryllium shield that effectively blocks the passage of low energy x-rays. Thus characteristic x-rays from "light elements" (those below sodium in atomic weight) are not detected.

Recently, DREA acquired a commercial EDX detector that employs a polymer shield that allows for detection of x-rays from elements as low as boron in atomic weight. As the use of this "light element" may alter the appearance of the asbestos fibre x-ray spectra, DREA investigated whether the use of such detectors would pose significant difficulties in asbestos fibre identification. Thus, the EDX spectra of the four most common asbestos minerals, chrysotile, amosite, crocidolite and anthophyllite, were collected with a "light element" detector and compared to spectra collected with a traditional detector. As well, any potential effects that binders present in commercial insulating materials may have on asbestos characterization using a "light element" detector were investigated. The study showed that "light element" SEM/EDX can be used to characterize asbestos fibers in bulk insulations.

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NOTATIONS

α	Alpha
Al	Aluminum
Ca	Calcium
CF	Canadian Forces
cps	Counts Per Second
dc	Direct Current
DND	Department of National Defence
DREA	Defence Research Establishment Atlantic
EDX	Energy Dispersive X-ray
eV	Electron Volt
Fe	Iron
H	Hydrogen
KV	Kilovolt
Mg	Magnesium
mm	Millimeter
Na	Sodium
SEM	Scanning Electron Microscopy
Si	Silicon
UICC	Union Internationale Contre Le Cancer

1. INTRODUCTION

Asbestos is a generic term used to describe two families of minerals, namely the serpentines and the amphiboles, which occur naturally as fibre bundles, have a fibrous texture and are composed of hydrated inorganic silicates with complex crystal structures. Of the serpentines, chrysotile is the only flexible fibrous member, and this mineral accounts for 90% of the world's asbestos production. Chrysotile is hydrated magnesium silicate having the composition $Mg_3(Si_2O_5)(OH)_4$ and is commonly referred to as white asbestos. The other group of fibrous minerals, the amphiboles, include amosite, anthophyllite, crocidolite, actinolite and tremolite, with the following chemical compositions:

amosite (brown asbestos)	$(FeMg)_7(Si_8O_{22})(OH)_2$
anthophyllite	$7MgO \cdot 8SiO_2 \cdot H_2O$
crocidolite (blue asbestos)	$Na_2Fe_5(Si_8O_{22})(OH)_2$
actinolite	$Ca_2(MgFe)_5(Si_8O_{22})(OH)_2$
tremolite	$Ca_2Mg_5(Si_8O_{22})(OH)_2$

These asbestos minerals have a unique combination of chemical and physical properties that make them virtually indestructible. Thus, they have found widespread use in the production of chemical, fire and heat resistant materials including flooring and roofing products, electrical and thermal insulation products and various other textiles and coatings. The properties of asbestos that control its stability in the environment and its biological behaviour include fibre length and diameter, surface area and the stability of the mineral in the biological host.

Asbestos has been used in thousands of applications. The greatest use of asbestos fibres occurs in the manufacture of cement drainage pipes, friction materials, insulation boards, papers and felts, reinforced plastics, vinyl tiles, woven yarn and textiles. In most of the asbestos-containing products used in industrial operations, the asbestos fibres are contained in a support matrix, organic or inorganic, which physically binds the fibres in place and are not expected to be released under normal conditions. However, fibres can be released from these materials as a result of manipulation, removal, vibration, abrasion or machining operations and thus will pose a potential health threat to personnel employed in the installation, maintenance and removal of such materials [1].

Due to the high incidence of asbestos-related health problems (asbestosis, mesothelioma and cancer) reported by medical authorities [2-11], asbestos has been

recognized as an industrial health hazard. Consequently, worker exposure to these materials must be minimized, if not prevented completely. In fact, most countries, including Canada, have banned or limited the use of asbestos fibres for industrial applications. However, as materials that were installed prior to these legislative acts may contain such fibres, health and safety regulations have been developed to permit removal of these materials without posing a significant health risk to workers or the public. Recent Canadian Forces policy [12] demands the use of appropriate non-asbestos substitute materials in all new construction and the replacement of asbestos in existing installations with non-asbestos materials wherever possible.

Many analytical techniques have been developed to identify and quantitate fibrous materials [13-18]. Some of these techniques, such as x-ray diffraction and infrared spectrometry, have inherent instrumental limitations that are dependent upon the origin, quantity, physical size and any environmental modifications of the fibres of interest. For example, x-ray diffraction techniques, require that sufficient sample, free from matrix material, be available for analysis; otherwise, a timely single crystal analysis is required. Infrared spectrometry requires that the fibres are relatively free of the non-asbestos matrix as its presence can provide interfering bands that will hamper the identification.

The technique that has been accepted by regulatory agencies as being capable of characterizing asbestos content of most industrial materials is polarized light microscopy. This technique employs morphological examination and determination of sample refractive indices for characterization. Some common natural and man-made fibres (such as wollastonite or polyethylene) have morphologies and refractive indices similar to asbestos fibres and can be misidentified as asbestos by inexperienced analysts. Thus, a technique, such as scanning electron microscopy (SEM) coupled with energy dispersive x-ray (EDX) spectrometry which identifies asbestos fibres by morphological and chemical analysis [19], can be useful as a complement to polarized light microscopy. The technique involves examination of fibre morphology by SEM and comparison of the EDX spectra of suspected asbestos fibres with spectra of known asbestos materials. Typically the morphological examination portion of this technique is sufficient to characterize a fibrous material as asbestiform and the EDX analysis serves to confirm this characterization and to identify the asbestiform mineral(s) present. Each asbestos mineral yields a characteristic EDX spectrum, and the types can be differentiated by comparing the relative amounts of several elements, specifically magnesium, silicon and iron, found in the fibres.

The EDX detectors normally employed for asbestos identification have utilized a beryllium window as a protective coating for the lithium-drifted silicon chip that forms the integral part of the detector. This window has the effect of blocking x-rays of energies below 800 eV; therefore, characteristic x-rays from elements below sodium in atomic weight are not detected. Recently, EDX detectors employing a polymer protective window in place of beryllium have become commercially available. These systems permit detection of x-rays with energies as low as 150 eV; therefore, elements down to boron in atomic weight are detectable. The use of such detectors for asbestos identification could potentially pose a problem by altering the spectra of asbestos minerals in one of several ways. First, since all asbestos minerals are essentially silicates, asbestos mineral spectra should contain an oxygen characteristic x-ray peak. Second, asbestos minerals are often vacuum coated with graphite prior to the SEM examination to prevent charge buildup from the electron beam and thus facilitate the morphological examination. Carbon x-rays are undetectable with an EDX detector having a beryllium window, and since the coated layer is sufficiently thin to not significantly hinder the transmission of high energy x-rays, the graphite coating does not interfere with the EDX analysis. With the new "light element" detectors, however, this graphite layer should be readily detected. Third, industrial insulations often contain binding agents, such as carbonates, which may cause additional interference with the "light element" detector.

In this paper we investigated whether the use of a "light element" EDX detector would pose significant difficulties in asbestos fibre identification. The EDX spectra obtained with a "light element" detector for the four most common asbestos minerals, chrysotile, amosite, crocidolite and anthophyllite, at two accelerating voltages, 20 and 10 KV, are examined. Also examined were the affects of carbon coating the sample to enhance image clarity, and the presence of binders in insulation samples on the appearance of the EDX spectrum.

2. MATERIALS, EQUIPMENT AND PROCEDURES

The four standard reference asbestos samples were the Union Internationale Contre Le Cancer (UICC) reference samples of asbestos that included:

Canadian Chrysotile
South African Amosite
Finnish Anthophyllite
South African Crocidolite

The physical and chemical properties of the UICC asbestos samples have been used extensively for international research purposes and are well documented [20, 21].

All x-ray spectra were recorded with a Princeton Gamma Tech Omega SLS Model OS14-I008 "light element" detector using a detector bias of -600V dc. In the "light element" mode a spectral low energy threshold of 200 eV was used. This detector was also used to mimic the results obtained with a "normal" EDX detector having a beryllium protective window. This was accomplished by raising the low energy threshold to 800 eV, thereby eliminating the "light element" peaks from the spectrum.

The detector was mounted on an International Scientific Instruments Model DS-130 scanning electron microscope. The spectra were collected using an accelerating voltage of either 10 or 20 KV. The specimens were mounted onto copper adhesive tape on aluminum stubs and oriented in the SEM chamber such that a working distance of 30 mm, a spectrometer distance of 60 mm, a sample tilt of +20° and a detector tilt of 6° were achieved. This corresponded to a takeoff angle of 36.5°. Prior to the collection of each spectrum the electron beam energy in the SEM was adjusted to yield an x-ray count rate in the 1500 - 2000 cps range.

Where appropriate, specimens were coated with graphite in an Edwards Model 306 vacuum coater.

3. RESULTS AND DISCUSSION

The energy dispersive x-ray spectra of graphite coated UICC chrysotile, amosite, anthophyllite and crocidolite asbestos fibres recorded at an accelerating voltage of 20 KV with a low energy threshold of 800 eV are shown as Figures 1a-d. The differences in chemical composition among the four asbestos minerals become apparent from an examination of the relative proportion of the elements magnesium (Mg), silicon (Si) and iron (Fe). For example, Table 1, which lists the relative percentages of various compounds in each of the asbestos minerals, indicates that the major elements for chrysotile asbestos are magnesium and silicon with a small amount of iron, and as expected the energy dispersive x-ray spectrum (Figure 1a) indicates the presence of Mg and Si in a Mg/Si ratio of approximately 0.9, with a small amount of iron. The ratio was determined by measuring the number of x-ray counts detected within a pre-defined energy range centered about the x-ray peak of interest. The major elements for amosite asbestos are Si and Fe with a small amount of Mg, and the energy dispersive x-ray spectrum for amosite asbestos (Figure 1b) indicates the presence of Si and Fe in a Si/Fe

ratio of approximately 1.5, with a small amount of Mg. Table 1 indicates that for anthophyllite asbestos one expects a small amount of Fe, a large amount of Si but a smaller amount of Mg than that found in chrysotile asbestos. A visual examination of the energy dispersive x-ray spectrum for anthophyllite asbestos (Figure 1c) indicates the presence of Mg and Si in a Mg/Si ratio of approximately 0.3 (Mg/Si~0.9 for chrysotile) with a small amount of iron. Finally, for crocidolite asbestos the energy dispersive x-ray spectrum (Figure 1d) indicates the presence of Si and Fe in a Si/Fe ratio of approximately 1.7 with a smaller amount of Mg than that found in amosite asbestos (as expected from the data in Table 1). As these spectra can be differentiated by asbestos type based on chemical compositions (Table 1), a comparison of spectra generated by the "light element" detector was required.

Figures 2a-d show the EDX spectra of graphite coated samples of the UICC asbestos fibres recorded with the "light element" detector at 20 KV. For all four asbestos samples little difference between the spectra in Figure 2 and the "normal" spectra shown in Figure 1 can be noticed, other than the presence of small oxygen and carbon peaks. Thus, the use of the "light element" detector in the identification of graphite coated asbestos samples seems to pose little problem.

Although the presence of the carbon peaks (from the graphite coating) does not seem to hinder the identification of the four asbestos samples, a study of the effect on the spectra obtained by analyzing non-graphite coated fibres using the "light element" detector was conducted. The coating process is time consuming, so if no observable deterioration of the EDX spectra occurs, this step in the sample preparation could be eliminated for bulk insulation samples where morphological identification of asbestos fibres is not difficult.

Figures 3a-d show the EDX spectra of uncoated samples of the same asbestos standards recorded at 20 KV with a low energy threshold of 200 eV. A visual comparison of the spectra for all four asbestos samples indicates little qualitative difference between the spectra in Figure 3 and the "normal" spectra shown in Figure 1, other than the presence of the small oxygen peak in the spectra recorded with the "light element" detector.

TABLE I
CHEMICAL COMPOSITION (%) OF ASBESTOS
MINERALS [20, 21]

Compound	Chrysotile	Amosite	Anthophyllite	Crocidolite
SiO ₂	39-42	49-53	56-58	49-53
MgO	38-43	1-7	28-34	0-3
FeO	0-2	34-44	3-12	13-20
Fe ₂ O ₃	0-2	-	-	17-20
Al ₂ O ₃	0-5	-	0.5-1.5	0-0.2
CaO	0.5-2	-	-	0.5-3
K ₂ O	-	0-0.5	-	0-0.5
Na ₂ O	0-0.1	-	-	4-9

To enhance the intensity of the "light element" peaks, spectra of the four asbestos samples (Figures 4a-d) were recorded with the "light element" detector at an accelerating voltage of 10 KV. Marked differences in the relative heights of the oxygen K and iron K α characteristic x-ray peaks can be detected from the spectra at 20 KV. All of the spectra show a diminished iron K α peak at 10 KV due to less efficient excitation of iron x-rays at 10 KV as compared to 20 KV. The oxygen K peak is increased in height in the 10 KV spectra due to the decreased depth of sample penetration achieved by the SEM electron beam with the relatively low energy beam electrons generated at 10 KV as compared to the higher energy electrons generated at 20 KV. The decreased depth of penetration means that x-ray signals are produced closer to the sample surface at 10 KV and thus the low energy oxygen characteristic x-rays are less likely to be absorbed before they are able to escape from the sample.

Finally, an evaluation of the effect the presence of impurities or binders in "real" samples have on the appearance of the EDX spectrum was conducted. Figure 5 shows the EDX spectrum of an uncoated sample of a commercial insulation containing amosite asbestos fibres recorded with a "light element" detector at 20 KV. The calcium, carbon and sulfur peaks detected in this spectrum result from the presence of binders (possibly calcium carbonates and sulfates) in the insulation. Figure 6 is a scanning electron micrograph that shows the presence of the binder particles on the asbestos fibres. Even though the spectrum shown in Figure 5 contains x-ray peaks that did not originate from

the fibre, the relative amounts of magnesium, silicon and iron still permitted easy identification of the fibres as amosite asbestos. The extraneous calcium and carbon peaks can be reduced (or removed) from the spectrum by recording a spectrum from an area relatively free of binder particles (Figure 7) or by first washing the sample with dilute acid (Figure 8).

4. CONCLUSIONS

The use of a "light element" EDX detector for asbestos fibre characterization does not pose any significant problems or complications over the use of a "normal" EDX detector. Observed differences between the spectra of standard asbestos fibres recorded with either detector are minimal. Graphite coating the sample also does not interfere with the analysis as the carbon layer does not interfere significantly with the transmission of x-rays from the sample and the carbon peak is easily attributed to the coating with no overlap of the important magnesium, silicon and iron peaks used to characterize asbestos. Extraneous x-ray peaks from binders in commercial insulating products can be avoided by careful selection of analysis area or by washing the sample with dilute hydrochloric acid prior to examination. However, even if binder peaks are present, asbestos identification by morphological examination and type characterization by the EDX spectrum is possible provided the stray peaks do not alter the magnesium/silicon/iron ratios.

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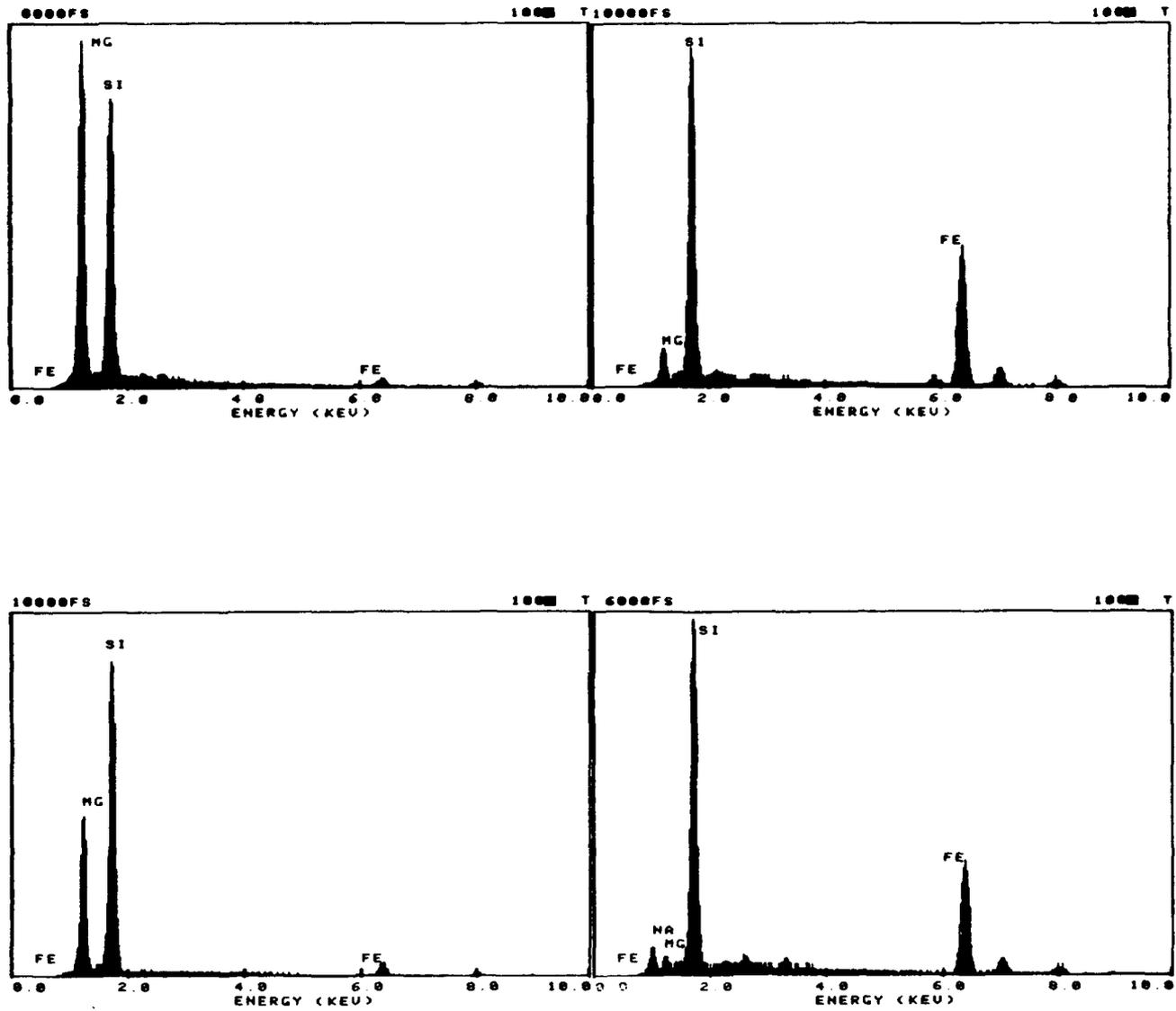


Figure 1. UICC Fibers recorded at 20KV with low energy threshold of 800-eV (graphite coated); a) chrysotile, b) amosite, c) anthophyllite and d) crocidolite.

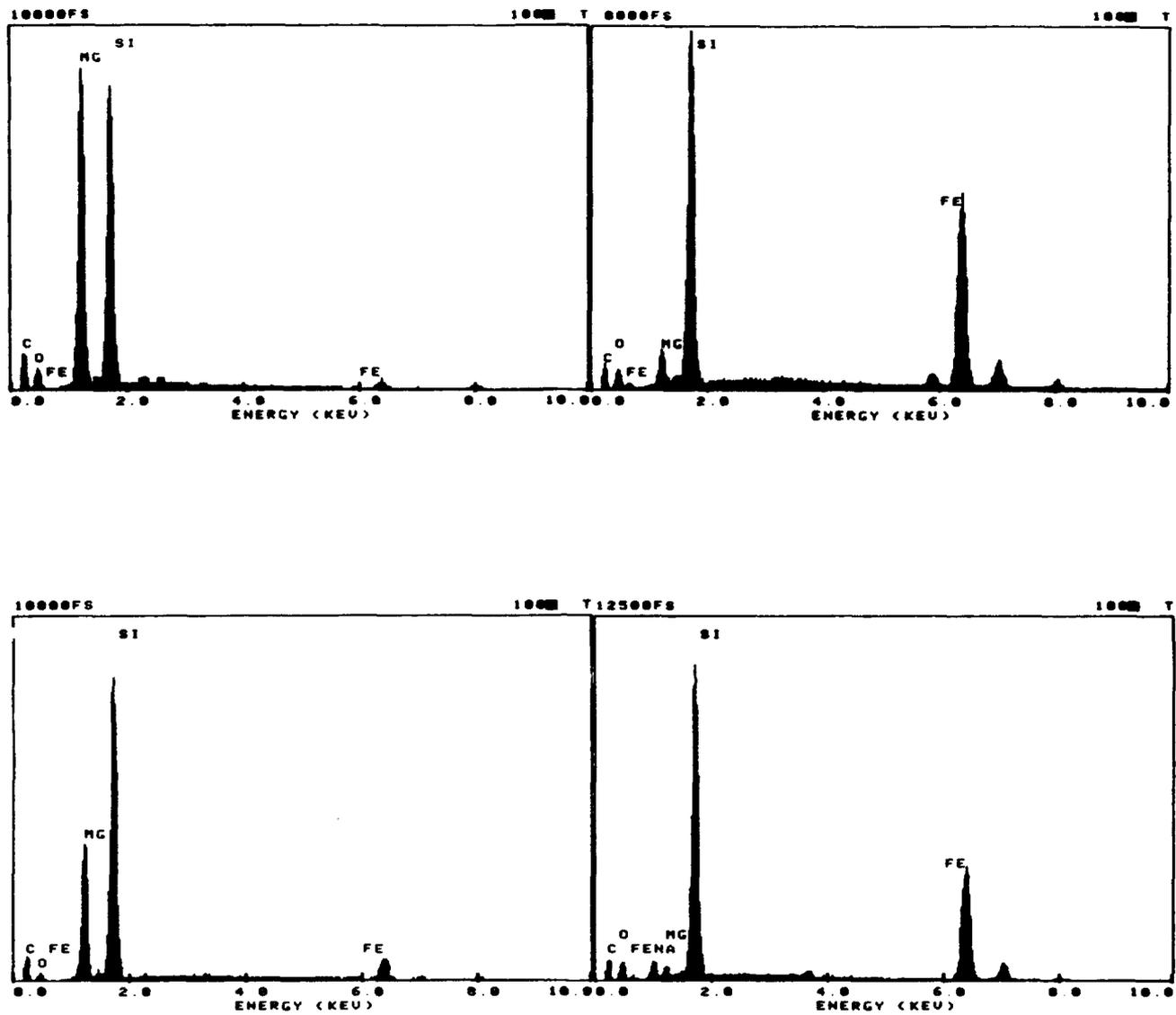


Figure 2. UICC Fibers recorded at 20 KV with low energy threshold of 200-eV (graphite coated); a) chrysotile, b) amosite, c) anthophyllite and d) crocidolite.

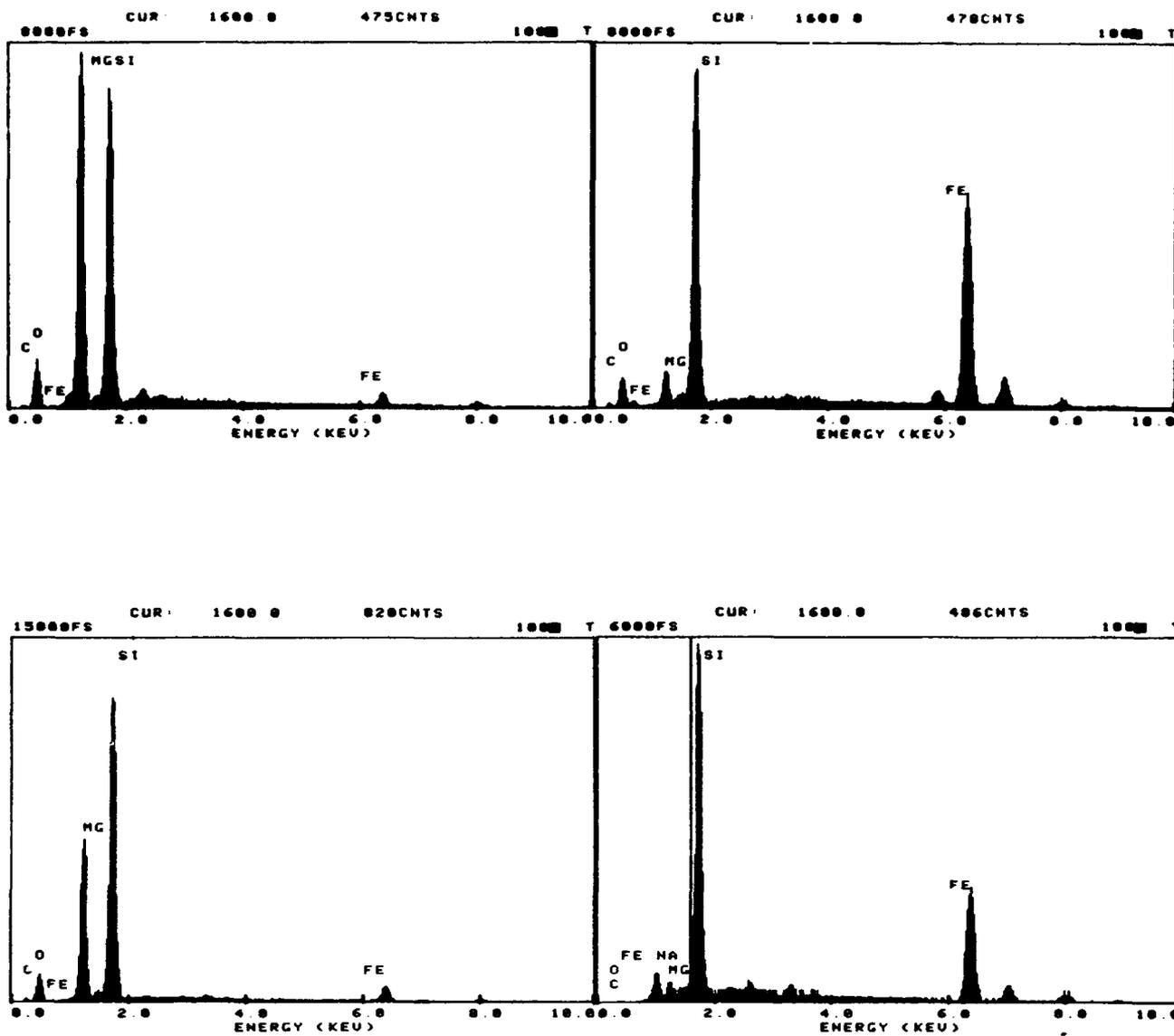


Figure 3. UICC Fibers recorded at 20 KV with low energy threshold of 200-eV (not coated);
 a) chrysotile, b) amosite, c) anthophyllite and d) crocidolite.

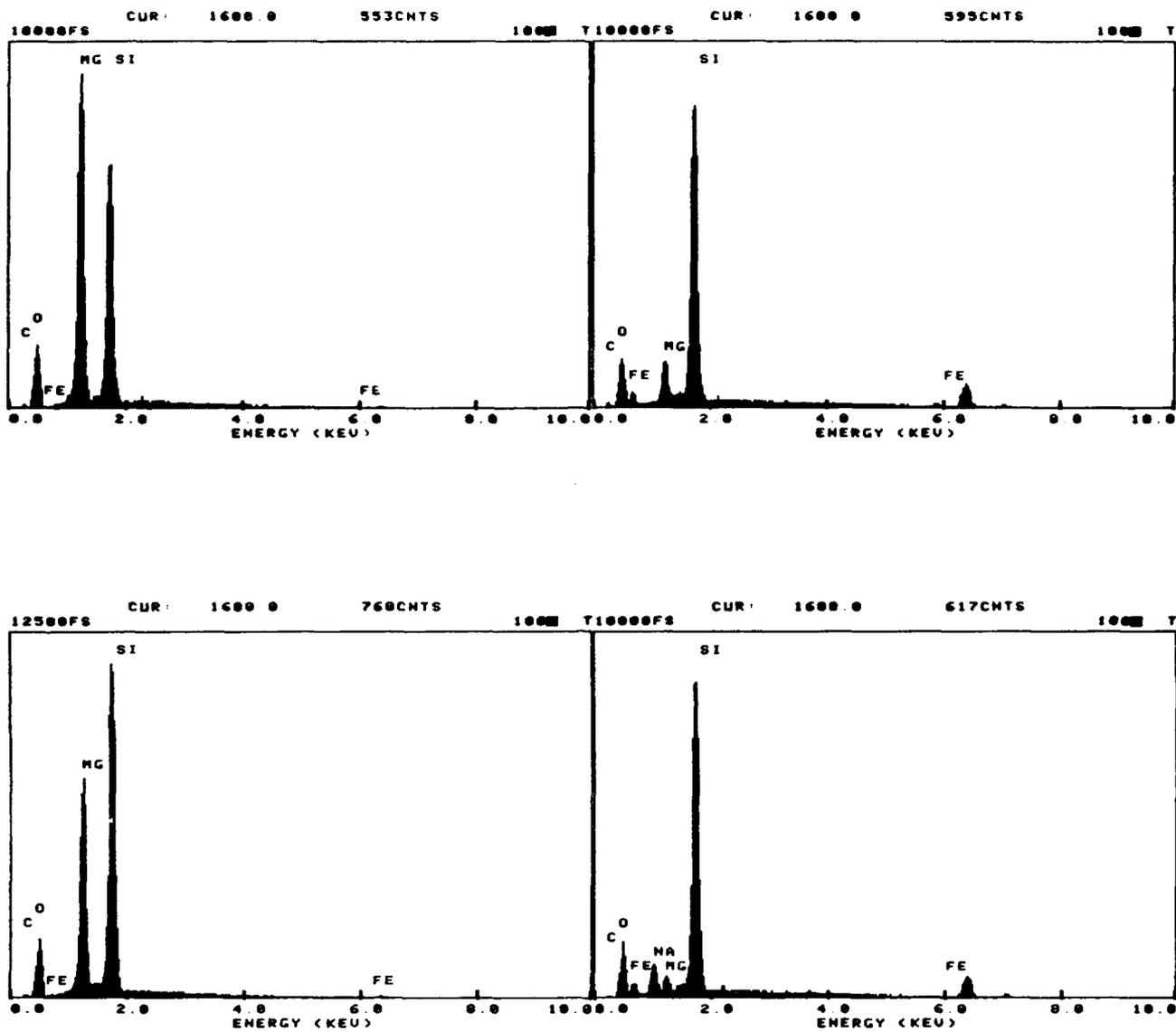


Figure 4. UICC Fibers recorded at 10 KV with low energy threshold of 200-eV (not coated);
 a) chrysotile, b) amosite, c) anthophyllite and d) crocidolite.

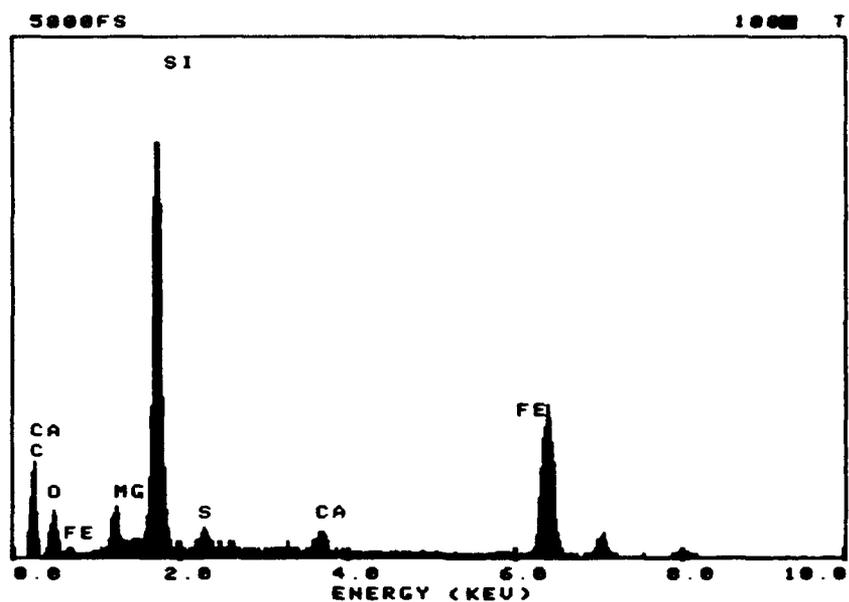


Figure 5. EDX spectrum of an amosite-containing insulating material recorded at 20 KV with low energy threshold of 200 eV (not coated).

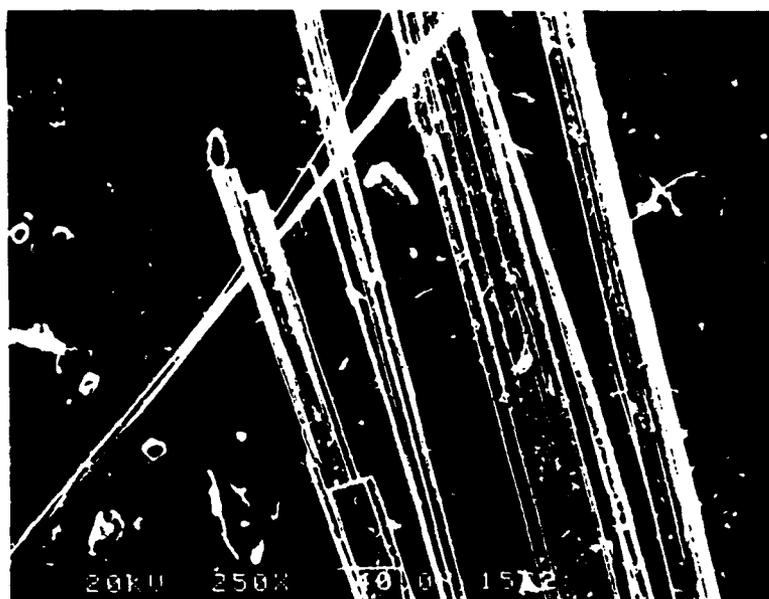


Figure 6. A scanning electron micrograph of binder particles and amosite asbestos fibres in a commercial insulating material (250X).

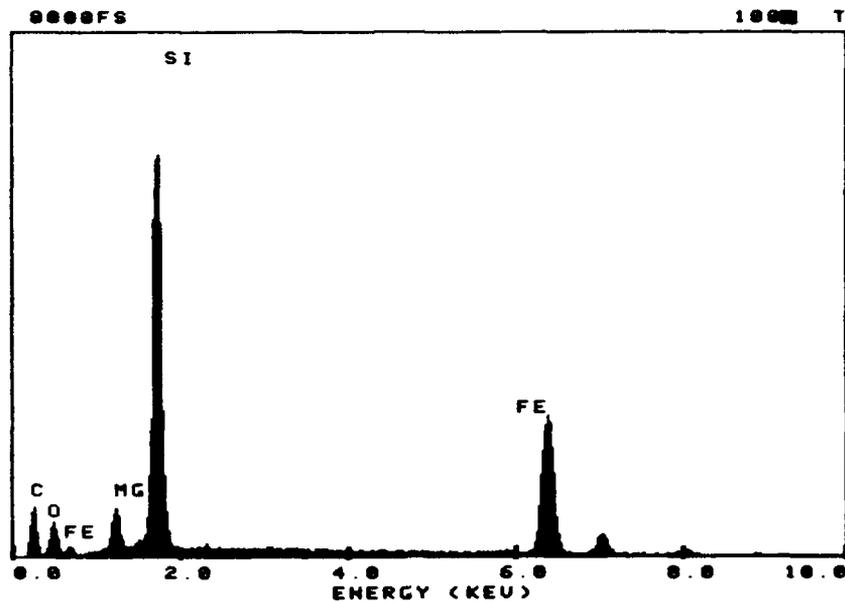


Figure 7. EDX spectrum of an area free of binder particles in an amosite-containing insulating material recorded at 20 KV with low energy threshold of 200 eV (not coated).

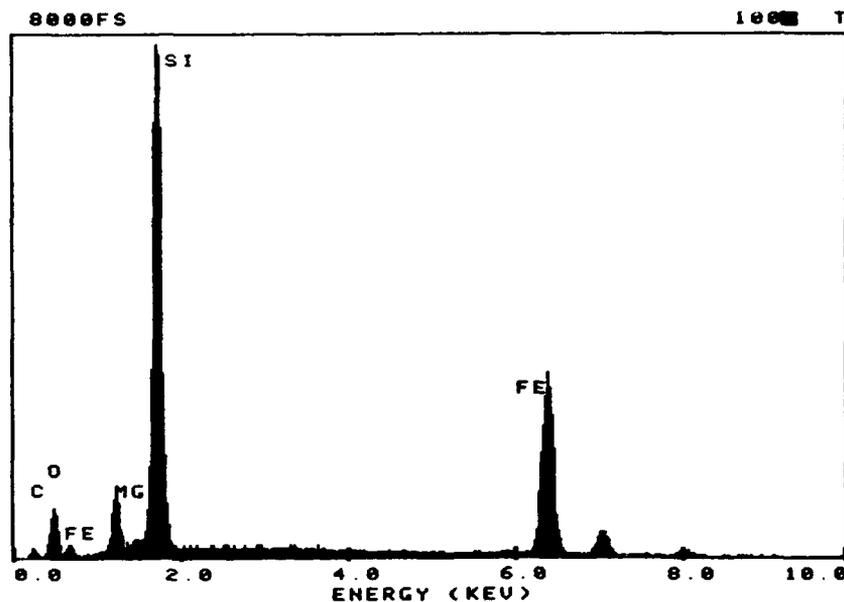


Figure 8. EDX spectrum of an amosite-containing insulating material washed in IN HCl and recorded at 20 KV with low energy threshold of 200 eV (not coated).

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4. AUTHORS (Last name, first name, middle initial. If military, show rank, e.g. Doe, Maj. John E.) Fisher, G.C. and Morchat, R.M.		
5. DATE OF PUBLICATION (Month and year of publication of document.) September 1993	6a. NO. OF PAGES (Total containing information. Include Annexes, Appendices, etc.) <p style="text-align: center;">22</p>	6b. NO. OF REFS. (Total cited in document.) <p style="text-align: center;">21</p>
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The Defence Research Establishment Atlantic (DREA) has traditionally used scanning electron microscopy (SEM) coupled with energy dispersive x-ray (EDX) spectrometry to identify asbestos fibers in solid insulating materials. This analysis typically utilizes fiber morphology to determine the presence of asbestiform fibers and EDX analysis to characterize asbestos type. The characterization is accomplished by comparison of the relative amounts of magnesium, silicon and iron present in the fibers. The EDX detector traditionally used in asbestos characterization employs a protective beryllium shield that effectively blocks the passage of low energy x-rays. Thus characteristic x-rays from "light elements" (those below sodium in atomic weight) are not detected.

Recently, DREA acquired a commercial EDX detector that employs a polymer shield that allows for detection of x-rays from elements as low as boron in atomic weight. This report summarizes results of a study on the effects of using a "light element" detector on characterization of both asbestos standards and commercial asbestos-containing insulating material. The study showed that "light element" SEM/EDX can be used to characterize asbestos fibers in bulk insulations.

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Asbestos
Energy Dispersive X-ray
Scanning Electron Microscopy
Chrysotile
Amosite
Anthophyllite
Crocidolite