Identification of Minerals Associated with Asbestos by X-Ray Diffraction Patterns

By M. S. BADOLLET and J. P. McGOURTY*

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Introduction

IT HAS LONG been customary for chemists to analyze samples of asbestos and their associated rocks by routine chemical procedures. Results of these investigations are usually reported in the conventional way, *i.e.*, the percentages of the various constituents existing in the sample under investigation. Often an examination of the data shows that re-grouping of the chemical analyses and re-calculation of the data in terms of minerals will shed more light on the true composition of the sample.

The late Dr. H. C. Cooke in his G.S.C. Memoir 211 (1) presented an exhaustive study of analyses of asbestos and rocks taken from the Thetford, Disraeli, and eastern half of Warwick map-areas in the Province of Quebec. Also he discussed the composition of asbestos and other fibres of the Thetford district in a paper he presented to the Royal Society of Canada in 1935 (2). Both papers include a series of analyses of fibres from the chemical point of view. The analyses are accompanied by a recast of the data in terms of minerals in which Dr. Cooke sought to deduce the mineral composition of each sample. This presentation of data was well done. It has been stimulating to the X-ray specialist who is interested in carrying the investigation further to confirm the predictions of the chemist, mineralogist, and geologist, and to make definite identification of the minerals which the chemical analyses indicate to be present.

Dr. Cooke was very co-operative in furnishing us with samples of material identical to those he used in his studies. We accepted his chemical analyses and his re-cast minerals data and have applied the X-ray technique for checking and identifying the minerals actually present in the samples. The dual purpose of this investigation was, first, to explore the limits of the X-ray diffraction method for identifying the minerals; and second, to develop a simplified technique for applying this information in sample identification. Such a technique would be of great value to company geologists in searching for new asbestos deposits, as well as in the examination of drill cores.

X-Ray Technique Employed

The X-ray pictures were taken by the Norelco X-ray diffraction unit with a Debye-Scherrer camera for a four-hour exposure, using copper radiation with a nickel filter. All samples were carefully powdered in an agate mortar before they were examined in the unit.

The X-ray pictures accompanying this paper carry the following identifications:

	A Line
S—Serpentine	2.49
C—Chrysotile	2.44
A—Antigorite	2.53
B—Brucite	2.35
F—Iron oxide	1.61
G—Grammatite (tremolite)	8.4
E—Epidote	2.9
Q—Quartz	3.34

When doubt existed in identifications, it was necessary to adopt spectrographic and petrographic techniques.

Discussion of Data

*Sample 1**

Sample 1 represented a "normal red-weathering serpentine from a chromite pit in the northwest corner of lot 19 N.W., Range X, Coleraine township". The chemical analysis (Table I) revealed that it contained SiO_2, MgO, and H_2O, with small amounts of other metals. Re-casting into minerals by Dr. Cooke indicated that the sample was mainly serpentine rock. The reddish colour probably was due to weathering of the small amount of iron present.

During recent years the X-ray diffraction patterns of serpentine have been fairly well indexed and identifications have been made easier. The pattern of Sample 1 showed clearly the typical ring structure of serpentine with a considerable amount of crystalline brucite. These two diffraction lines are identified in Figure 1.

The chemical analysis showed an excess of MgO over the amount required to combine with SiO_2 to form serpentine, $H_4Mg_3Si_2O_9$; this excess of MgO crystallized in the form of brucite, $Mg(OH)_2$, which is intimately associated mechanically with the serpentine. The asbence of evidence in the diagram of the presence of other minerals is due to the fact that any compounds formed by the minor constituents shown by the chemical analysis are not in sufficient quantity to yield definite X-ray diffraction patterns. The brucite is so closely associated with the serpentine that it could not be removed by mechanical means but it could be dissolved out chemically.

*Section Chief, †Research Engineer, Johns-Manville Research-Center, Manville, New Jersey, U.S.A.

(1) Cooke, H. C., Mem. 211, Geol. Survey, Dept. of Mines and Resources, Ottawa, 1937.

(2) Cooke, H. C., Roy Soc. Can., Trans., Sec. IV, 3rd Series, Vol. XXXIX, 1935.

*The descriptions and chemical analyses of samples 1 to 21 are reproduced from *G.S.C. Memoir* 211, by Dr. H. C. Cooke. Numbering of the samples is the same as in that report.

Sample 2

Sample 2 represented the same material as in Sample 1, but it exhibited "material blackened over a zone a foot wide and flanking a dyke about a foot wide". Chemical analysis (Table 1) showed the presence of Al_2O_3, Fe_2O_3, and FeO in quantities that indicated other minerals than serpentine must be present as impurities.

The X-ray diffraction patterns did not reveal the presence of brucite, thus confirming the chemical analysis, which showed sufficient SiO_2 to combine with all the MgO to form serpentine; hence, no excess MgO remained to form brucite or any other magnesia mineral. Although the chemical analysis showed the presence of aluminum and iron compounds, they were not identified in the X-ray pattern because they are present only in very small amount.

Sample 3

Sample 3, which represented "chrysotile asbestos taken from the King mine", is typical of the fibre from that area. The chemical analysis (Table 1) disclosed a composition equivalent to that of chrysotile asbestos, with small traces of impurities. Dr. Cooke, in his re-calculation of the analysis, deduced the possible presence of minerals such as chlorite, talc, and periclase as impurities associated with the chrysotile asbestos.

X-ray diffraction lines did not indicate the presence of brucite, but they did show a trace of quartz. Although the chemical analysis, when re-cast in terms of minerals, indicated the presence of chlorite, talc, and periclase, lines for these minerals were not visible in the X-ray pattern.

This sample was also analyzed quantitatively by emission spectroscopy* with known oxide mixes as standards; the data obtained, on an ignited basis, showed the presence of Al_2O_3, 0.10%; Fe_2O_3, 1.3%; and CaO, 0.11%. If the iron in the original sample was in the form of Fe_3O_4, it would have been oxidized to Fe_2O_3 by the treatment preceding to the spectroscopic examination.

The total amounts of Al_2O_3, Fe_2O_3, and CaO by spectroscopic

*Emission spectroscopy is not a means of identifying compounds but can be used as a supplement to ordinary wet chemical analysis.

TABLE I.—ANALYSES, SAMPLES 1 TO 4

	1	2	3	4
SiO_2	34.40	40.42	42.05	43.48
Al_2O_3 ...	0.50	2.24	Nil	1.41
Fe_2O_3 ...	0.82	2.11	0.96	0.60
FeO	3.79	4.26	0.39	2.36
CaO	0.02	0.29	0.05	0.12
MgO	42.36	39.44	43.30	39.50
H_2O+ ...	15.17	11.11	12.52	12.69
H_2O- ...	0.47	0.10	0.75	0.45
TiO_2 ...	Nil			
MnO	0.94			
CO_2	0.38			
Cr_2O_3 ...	0.48			
NiO	0.35			
Total	99.68	99.97	100.02	100.61

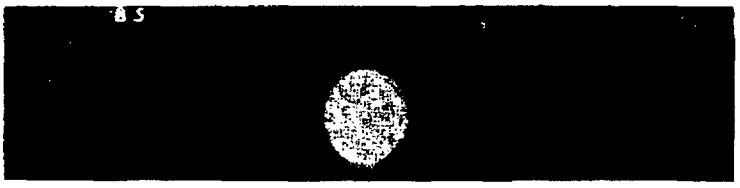

Figure 1.
S—serpentine line; B—brucite line.

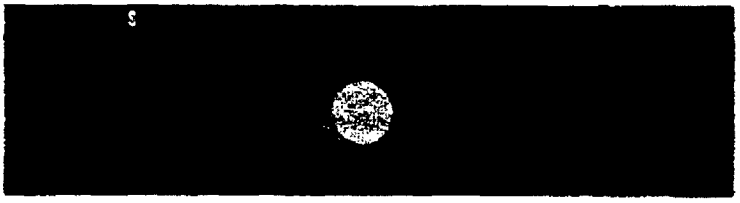

Figure 2.
S—serpentine line.

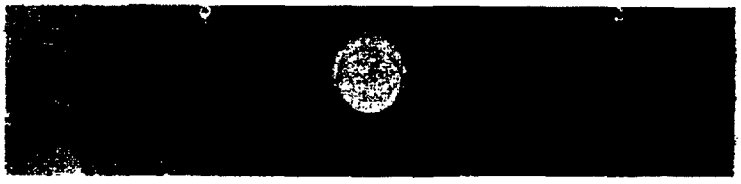

Figure 3.
C—Chrysotile line; Q—quartz line.

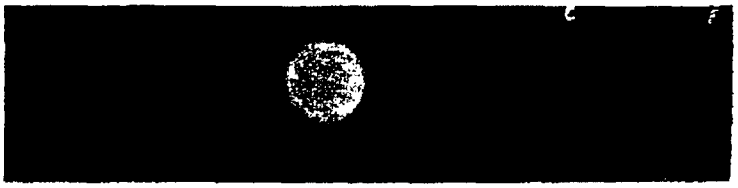

Figure 4.
C—chrysotile line; F—iron line.

Figure [illegible]
C—chrysotile lines; F—iron line.

Sample [illegible] chrysotile fibre [illegible] which was [illegible] and made brittle apparently by the blackened zone close to the acid dyke". The chemical analysis (Table I) indicated chrysotile asbestos, with Al_2O_3 and FeO and an excess of SiO_2 present. The data, when recast into minerals, showed the possible presence of chlorite, brucite, and silica.

The X-ray diagram did not show brucite, silica as quartz, or chlorite. The chemical analysis, however, indicated an excess of SiO_2 over the amount required by the MgO to form asbestos. This SiO_2 may have been in the form of amorphous SiO_2, which would not show in the X-ray diagram; or it may have been in combination with the minor constituents shown by the chemical analysis, and the compounds thus formed are not in sufficient quantity to produce an X-ray diagram. The X-ray film did indicate iron as Fe_3O_4 or Fe_2O_3.

Emission spectroscopy of this sample showed the presence of Al_2O_3, 0.20%; Fe_2O_3, 4.2%; and CaO, 0.08%. These spectroscopic data did not agree with the chemical analysis, again pointing to the difficulty of identifying the true minerals present.

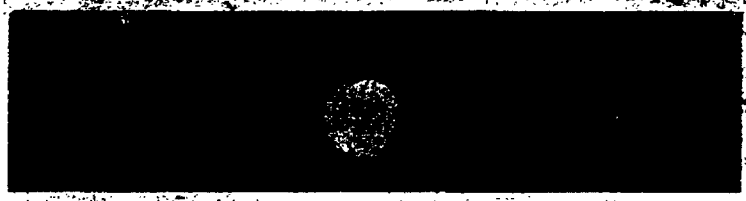

Figure 6.
C—chrysotile line.

Sample 7

Sample 7 represented a so-called "high-grade chrysotile [illegible] taken from the [illegible] chemical analysis [illegible] minor amounts [illegible] minum, possibly [illegible] presence of [illegible] re-cast data [illegible] cite, and perhaps [illegible]

Figure 7.
C—chrysotile line; F—iron line.

The X-ray diagram did not show lines for brucite, chlorite, or periclase; although the chemical analysis indicated a slight excess of MgO it was not identified in the X-ray diagram. Iron as Fe_3O_4 did appear in the X-ray diagram. Emission spectroscopy showed the presence of Al_2O_3, 0.12%; Fe_2O_3, 1.70%; and CaO, 0.08%.

Figure 8.
C—chrysotile line; F—iron line.

Sample 8

Sample 8 represented a "high-grade chrysotile asbestos taken

Figure 9.
C—chrysotile lines; F—iron line.

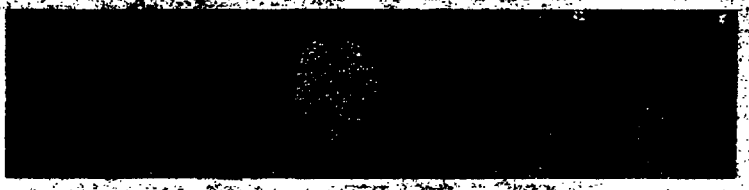

Figure 10.
C—chrysotile line; F—iron line.

from Deloro township, Province of Ontario". The chemical analysis (Table II) showed the presence of impurities. The re-cast data indicated the possible presence of chlorite, talc, and periclase.

The X-ray diagram did not show chlorite, talc, brucite, or periclase. If these minerals were present, the quantities were insufficient to produce an X-ray diffraction pattern. Emission spectroscopy demonstrated the presence of Al_2O_3, 0.07%; Fe_2O_3, 1.0%; and CaO, 0.02%; amounts which differ from the chemical analysis.

Sample 9

Sample 9 is a chrysotile fibre from the "British Canadian mine at Black Lake". The fibre is further identified as a semi-harsh type, *i.e.*, not actually brittle, but not soft and silky. The chemical analysis (Table II) showed small amounts of Al_2O_3, Fe_2O_3, and CaO, and almost 2 per cent FeO. Calculated in terms of minerals, the presence of chlorite, talc, and periclase was indicated.

The X-ray diagram did not reveal the presence of brucite, chlorite, or periclase, but it did show small amounts of Fe_3O_4.

Emission spectroscopy showed the presence of Al_2O_3, 0.16%; Fe_2O_3, 4.0%; and CaO, 0.02%. If Fe_3O_4 were present originally it was probably oxidized to Fe_2O_3 by the ignition process in preparing the fibre for this test.

Sample 10

Sample 10 is an extrardinary chrysotile asbestos fibre of harsh texture bordering upon a brittle nature. It is from the "Vimy Ridge mine in Coleraine township". The chemical analysis (Table II) indicated a low MgO-SiO_2 ratio, a low combined water content, as well as a high percentage of Fe_2O_3. Recast in terms of minerals, the presence of chlorite, talc, and periclase was indicated.

The X-ray pattern did not show lines for brucite, talc, periclase, or chlorite. If these minerals were present they must have been in very small quantities, not detectable by the X-ray technique.

Emission spectroscopy showed Al_2O_3, 0.52%; Fe_2O_3, 3.0%; and CaO, 0.03%; amounts which do not agree with the chemical data.

TABLE II.—ANALYSES, SAMPLES 7 TO 10

	7	8	9	10
SiO_2	41.13	42.40	41.80	42.95
Al_2O_3 ...	0.80	0.14	0.64	Nil
Fe_2O_3 ...	1.01	0.97	0.80	2.08
FeO	0.58	0.19	1.89	0.84
CaO	0.03	0.14	0.14	0.19
MgO	43.24	43.09	42.18	41.60
H_2O+ ...	12.74	12.60	12.07	10.26
H_2O- ...	0.45	0.45	0.20	1.61
Totals.	99.98	99.98	99.72	99.53

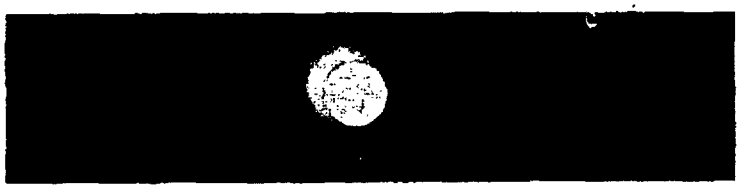

Figure 11.
C—chrysotile line.

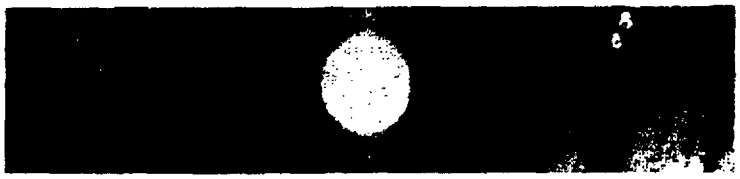

Figure 12.
B—brucite line; C—chrysotile line (barely visible).

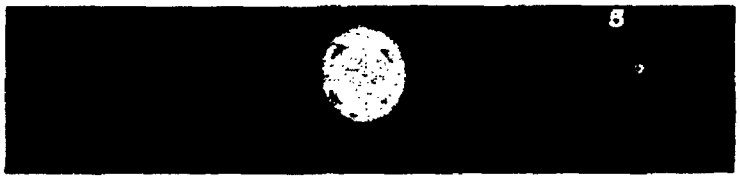

Figure 13.
B—brucite line.

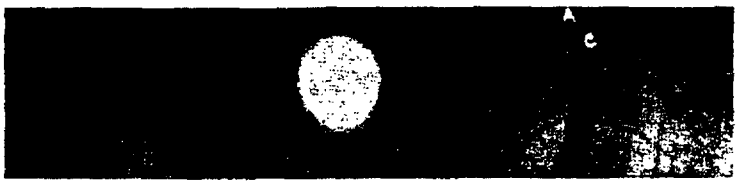

Figure 14.
C—chrysotile line; A—antigorite line.

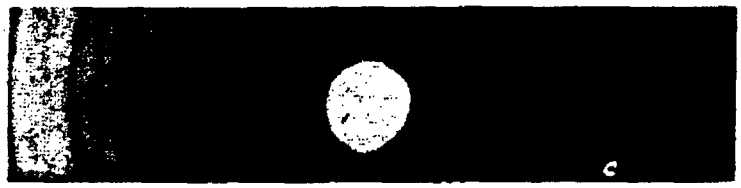

Figure 15.
C—chrysotile line.

[illegible]

Sample 15 represented a type of "slip serpentine" taken from a broad fault-zone, [illegible] King mine, Thetford". [illegible] (Table III) showed [illegible] such as Fe_2O_3 and [illegible] in terms of minerals, [illegible], brucite, and periclase were indicated.

The X-ray diagram showed no trace of brucite, periclase, or chlorite; however, the diffraction lines for chrysotile and iron are clearly defined.

Sample 16

Sample 16 is another specimen of "slip serpentine taken from a fault-zone in drift 504X of the King mine, Thetford". The analysis (Table III) showed that the SiO_2 was slightly low, and the MgO rather high, for serpentine. Minor constituents were Al_2O_3, Fe_2O_3, FeO, and CO_2. The data, re-cast as minerals, suggest the presence of chlorite, brucite, and hydromagnesite.

The X-ray diagram showed no trace of brucite, chlorite, or hydromagnesite. It did show, however, chrysotile, and iron as Fe_3O_4.

Sample 17

Sample 17 represented a "tough, stringy fibre from a fault, drift C502E, near D 505X, King mine, Thetford". Chemical analysis (Table III) showed substantial amounts of Fe_2O_3 and FeO. Re-cast of the analysis in terms of minerals indicated the presence of chlorite, brucite, and periclase.

The X-ray diagram showed no trace of chlorite, brucite, or periclase, but the chrysotile line is well defined.

Sample 18

Sample 18 was [illegible] asbestos fibre [illegible] King mine, [illegible] further identified [illegible] as a fault fibre. The chemical analysis (Table III) showed relatively large amounts of Al_2O_3, Fe_2O_3, and FeO, indicating considerable contamination. SiO_2 was extremely low, H_2O very high, and MgO unusually high, indicating a peculiar fibre composition. Re-calculated data indicated that the minerals present might be brucite, chlorite, and periclase, with brucite comprising 57.61 per cent of the sample. This is an interesting sample for further investigation.

The X-ray diagram showed a very high percentage of brucite, with some chrysotile present. No evidence of chlorite or periclase was indicated by the X-ray diagram.

Sample 19

Sample 19 was a fault fibre taken from the [illegible] at Thetford. [illegible] analysis (Table III) indicated only small amounts of SiO_2, Al_2O_3, Fe_2O_3, and CaO, but a relatively high percentage of FeO. The MgO content (67 per cent) and the combined H_2O (28.76 per cent) indicated the presence of some magnesium mineral other than asbestos or serpentine. The re-calculated data likewise showed about 9[illegible] per cent brucite together with small amounts of chlorite, periclase, and magnetite.

The X-ray diagram showed that the specimen was practically all brucite. The diagrams displayed no evidence of chlorite, periclase, or magnetite.

Sample 20

Sample 20 was identified as a "green, fibrous picrolite taken from a fault plane at the 800-foot level in the King mine in Thetford". Chemical analysis showed ((Table III) SiO_2 and MgO, the chief constituents, were in about the right proportion to form serpentine, but the presence of relatively large amounts of Fe_2O_3 and FeO suggested an admixture of other minerals.

Re-calculated data indicated the presence of chlorite, talc, periclase, and hydromagnesite.

The X-ray diagram showed the material to be chrysotile and antigorite. No evidence of chlorite, talc, periclase, or hydromagnesite was observed.

Sample 21

Sample 21 was described as a "semi-fibrous picrolite filling vein at the 300-foot level in the King mine at Thetford". Chemical analysis showed (Table III) that it contained appreciable amounts of Al_2O_3, Fe_2O_3, and FeO denoting the presence of other minerals than picrolite. Re-cast of the analysis indicated that there might be chlorite (19.12%), talc (9.22%), and periclase (0.99%).

The X-ray diagram did not show lines for brucite, chlorite, talc, or periclase, but it did show the diffraction line for chrysotile.

TABLE III.—ANALYSES, SAMPLES 15 TO 21

	15	16	17	18	19	20	21
[illegible]	[illegible]	39.26	40.02	14.97	[illegible]	[illegible]	[illegible]
[illegible]	nil	0.09	0.49	1.2[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	1.13	1.69	1.[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	0.49	1.09	1.4[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	0.09	trace	0.[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	42.97	42.31	56.[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	12.98	15.20	13.27	27.[illegible]	[illegible]	12.[illegible]	[illegible]
[illegible]	0.39	0.34	1.36	0.7[illegible]	0.16	[illegible]	[illegible]
[illegible]					0.34		
[illegible]							
[illegible]							
[illegible]		1.69				0.47	
Total	109.38	100.00	100.60	100.75	99.40	99.29	100.39

Figure 16.
C—chrysotile line; B—brucite line.

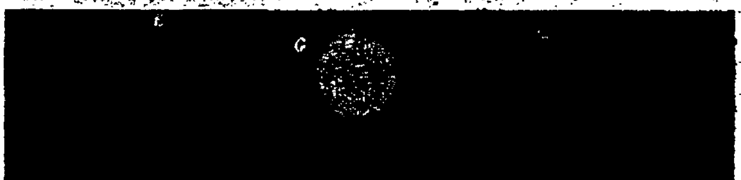

Figure 17.
D—grammatite line; E—epidote line.

UCC 004640

*Sample 50**

Sample 50 was described as a "tough stringy fibre forming cross-fibre veins at drift 505X in the King mine, Thetford". It was more specifically identified as a soft-chrysotile fibre. Chemical analysis (Table IV) showed the presence of Al_2O_3, Fe_2O_3, FeO, and CaO, as well as a small amount of Na_2O and K_2O. Re-cast by Dr. Cooke in terms of mineral composition the analysis indicates that chlorite 8.01%, and brucite 9.19%, may be associated with the chrysotile in this sample.

The X-ray diagram disclosed the presence of chrysotile and brucite, but no chlorite.

*Sample 63**

Sample 63, described as "pseudo-asbestos", represented fibrous veins in the footwall of the Federal pit near Robertsonville, Quebec. The analysis is given in Table IV. This specimen was an interesting sample for X-ray analysis since it showed a high percentage of Al_2O_3, Fe_2O_3, FeO, and CaO, with MgO exceedingly low.

The X-ray diagram showed lines corresponding to grammatite (tremolite) and epidote.

TABLE IV.—ANALYSES, SAMPLES 50 AND 63*

	SAMPLE 50	SAMPLE 63
SiO_2	37.44	46.36
Al_2O_3	0.58	11.39
Fe_2O_3	1.23	9.30
FeO	0.54	3.18
CaO	0.20	18.47
MgO	43.49	8.94
H_2O+	14.60	1.87
H_2O-	1.71	0.12
TiO_2	—	0.05
MnO	—	0.61
NiO	—	0.15
Na_2O	0.28	—
K_2O	0.05	—
	100.12	100.44

CONCLUSIONS

Chemical analyses of chrysotile, picrolite, and other varieties of serpentine invariably, or almost invariably, show that, in addition to the MgO, SiO_2, and H_2O necessary to form chrysotile of theoretical composition $H_4Mg_3Si_2O_9$, they contain also a small, or in some specimens a very appreciable, percentage of Al_2O_3, Fe_2O_3, FeO, etc. By re-casting the analyses in terms of minerals these may be assigned to various minerals such as brucite, chlorite, periclase, and hydromagnesite, which may or may not actually be present admixed with the chrysotile. Except when in very small amount, definite verification of the presence of one or more of these minerals can be made by application of X-ray diffraction, emission spectroscopy, or petrographical methods of examination. These methods, and in particular the X-ray diffraction method, have been used in the present investigation of specimens of serpentine from a number of asbestos mines in the Eastern Townships of Quebec, and of one from Deloro township, Ontario. A critical study of chemical, and re-cast mineral, analyses of the specimens had been made earlier by Dr. H. C. Cooke, who very kindly supplied the writers with the material used in their investigation.

For some specimens, the X-ray diffraction patterns confirmed the mineral composition as derived by Dr. Cooke in his mineral re-cast of the chemical analysis; for some others, in which the extraneous mineral or minerals were in very small amount, diffraction lines for these did not appear. Clearly, with a sample containing two or more minerals, only those present in sufficient quantity to give diffraction lines can be definitely identified as being present.

Samples of the latter type were analyzed by emission spectroscopy and in a general way, though not quantitatively, the results were in agreement with those derived by Dr. Cooke from his study of the chemical analyses. Petrographic examinations under the microscope were also helpful in cases where doubt existed.

This investigation has indicated that no one method is adequate in itself for complete and positive identification of all the minerals present in a sample. For this it is necessary to resort to a combination of several methods. For complete identification of all the minerals present in samples obtained in prospecting or in drill cores of asbestos bodies, not only chemical analyses but also the results of X-ray diffraction, spectrographic, and petrographic studies are necessary.

ACKNOWLEDGMENTS

Chemical data used in this paper were taken from G.S.C. *Memoir* 211, by the late Dr. H. C. Cooke, and from a paper by him in the *Transactions* of the Royal Society of Canada, Volume XXIX, 1935.

This investigation was initiated six years ago after a discussion with Dr. Cooke, in Ottawa, on the use of X-ray technique for identification of minerals. Dr. Cooke supplied all samples used in the investigation.

The assistance of Mr. Charles Whinfrey, formerly of the Johns-Manville Research Centre, who made the preliminary X-ray studies, is appreciated.

*The descriptions and chemical analyses of samples 50 and 63 are reproduced from the paper by Dr. H. C. Cooke in Volume XXIX, 1935, of the *Transactions* of the Royal Society of Canada. Numbering of the samples is the same as in that paper.