X-RAY DIFFRACTION PATTERNS OF ASBESTOS

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ABSTRACT

X-ray diffraction patterns are given for 8 samples of chrysotile, 1 crocidolite and 1 anthophyllite. Samples were studied in powder and fiber form and compared to patterns obtained by other investigators. The salient features of the typical asbestos pattern needed for identification purposes are obtained from both fiber and powder samples. Patterns obtained of the various chrysotiles indicate variations in the number, sharpness, *d* values and hence crystallinity of the samples.

INTRODUCTION

The detection and identification of asbestos in commercial materials are of considerable interest in analytical laboratories. The principal purpose of this paper is thus to provide data for the identification of these minerals by means of *x*-ray diffraction. However, several points of interest to the mineralogist are also discussed. Special emphasis has been placed on the patterns for chrysotile since this is the principal variety used commercially and 8 samples are included in the study. One sample of crocidolite from South Africa is included since this variety is also widely used and 1 sample of anthophyllite from Georgia.

TABLE 1. DESCRIPTION OF BULK ASBESTOS SAMPLES

			Feel	
	Color	Luster	Bulk	Fibre
Chrysotile				
Zermatt	light brown	dull, earthy	slightly waxy	matted, flexible
Thetford	green	high, pearly	very smooth, waxy	silky, very flexible
Richmond	green	high, pearly	very smooth	silky, very flexible
Nevada	green	slight	smooth	soft to threadlike
Corsica	white	high, silky	very soft	soft, very flexible
Easton	white	high, silky	slightly waxy	soft, threadlike
Labrador	white	high, pearly	granular	very brittle
Anthophyllite				
Georgia	brownish-white	dull to pearly	waxy	short fuzz
Crocidolite				
South Africa	blue-gray	dull	soft	soft, flexible

Diffraction patterns were obtained of both the powders and fibers.

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TABLE 2. DIFFRACTION PATTERN—CHRYSOTILE—ZERMATT, SWITZERLAND

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
10.0	3			2.98	3		
7.2	41	7.3	st.	2.79	2		
5.0	2			2.53	7	2.55	wk.
4.77	4			2.43 w	2		
4.50	4	4.55	wk.	2.16 w	1.5		
4.31	3			2.10 w	1.5		
3.97 w	2			1.82 w	1.5	1.89	wk.
3.85	2			1.51 w	1.5		
3.62	38	3.65	st.	1.45 w	1.5		
3.35	4						

The powder patterns were obtained with the x-ray spectrometer using filtered Cu radiation. All these patterns were made under identical conditions so that the intensities of the lines can be directly compared from one sample to another. Patterns of the fibers were obtained with a standard diffraction unit using filtered Co radiation.

TABLE 3. DIFFRACTION PATTERN—CHRYSOTILE—THETFORD, CANADA

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
7.8	4			2.45	12	2.45	wk.
7.2	19	7.3	st.	2.14 w	2		
5.7 w	2			2.10 w	3		
4.92 w	1.5			1.82 w	2		
4.45	9	4.52	wk.	1.74 w	3	1.74	wk.
4.31 w	2			1.59 w	2		
4.13	3			1.55 w	2		
3.82	2			1.53 w	8	1.54	wk.
3.63	15	3.66	st.	1.51 w	2		
3.34	2			1.50 w	2		
3.28	2			1.31 w	3		
2.79	2				cf. Index cards	II 782	
2.58	6					II 140	
2.52	8						

TABLE 4. DIFFRACTION PATTERN—CHRYSOTILE—RICHMOND, CANADA

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
7.8	3			2.40	3		
7.4	10	7.4	st.	2.08	5		
6.1 w	2					1.84	wk.
5.2 w	2			1.61	2	1.69	wk.
4.5	5	4.5	st.	1.56 w	4		
4.2	2	4.0	st.	1.54 w	3	1.54	st.
3.68	10	3.66	st.	1.48	3	1.45	wk.
3.40 w	2					1.21	wk.
2.98 w	2					1.04	wk.
2.60	6					.986	wk.
2.54	10					.947	wk.
2.47	5	2.43	wk.			.912	wk.

DESCRIPTION OF SAMPLES

The bulk samples of chrysotile and anthophyllite were obtained through the courtesy of Mr. David Seaman of Harvard University, then curator of the Mineralogy Department of the Carnegie Museum of Pittsburgh. These include chrysotile from Zermatt, Switzerland; Thetford, Canada; Richmond, Canada; Nevada, California; Corsica; Easton, Pennsylvania, and Labrador. The anthophyllite and crocidolite were

TABLE 5. DIFFRACTION PATTERN—CHRYSOTILE—NEVADA, CALIF.

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
7.4	6	7.4	v. st.	2.70	2		
5.3	2			2.60	3		
4.6	4	4.5	st.	2.53	4	2.45	wk.
4.1	2			2.35	1		
4.0	2					1.84	v. wk.
3.95	3			1.91	1		
3.68	5	3.67	st.	1.54	3	1.54	st.
3.37	2					1.47	
3.06	3.5			1.31	2		
2.96	4					1.221	wk.
2.76	2					1.044	wk.
						.914	wk.

TABLE 6. DIFFRACTION PATTERN—CHRYSOTILE, CORSICA

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
8.8	6	9.1	st.	1.750	2		
8.4	14	8.4	v. st.	1.714	2		
7.4	2			1.691	3	1.70	v. wk.
5.54	2			1.653	7	1.64	wk.
5.24	1.5			1.623	3	1.62	st.
5.09	2	5.10	wk.	1.583	3	1.59	wk.
4.92	7			1.558	1	1.56	v. wk.
4.77	5	4.75	wk.	1.537	2	1.530	v. wk.
4.55	7	4.55	v. st.	1.512	5	1.507	v. st.
4.21	3	4.23	wk.	1.473	1		
3.90	4					1.461	wk.
3.68	1.5			1.453	2		
3.39	11			1.442	8	1.434	wk.
3.28	7	3.30	v. st.	1.366	3	1.357	wk.
3.13	22	3.14	v. st.	1.339	3.5	1.328	v. wk.
2.94	7			1.310	4		
2.81	5	2.83	wk.	1.299	3	1.298	v. wk.
2.71	19			1.278	2		
2.69	1.5					1.261	v. wk.
2.60	7			1.201	1.5		
2.54	8					1.192	st.
2.41	3	2.40	wk.			1.133	wk.
2.34	9					1.047	v. st.
2.30	3					1.028	v. wk.
2.28	3					1.018	v. wk.
2.169	8					.993	v. wk.
2.047	3					.979	wk.
2.021	7	2.01	wk.			.972	v. wk.
1.970	1.5	1.97	wk.			.952	st.
1.897	2	1.90	wk.			.934	wk.
1.871	2					.903	v. wk.
1.842	1					.902	wk.
1.819	2	1.81	wk.			.900	v. wk.

obtained in loose fibers and the chrysotile from Chester, Pennsylvania, in powder form.

A general description of the samples is given in Table 1. The order of appearance of the chrysotiles was decided from a cursory visual inspection, the first sample appearing the least crystalline. All the chrysotile powders are white excepting that of Zermatt which remains brownish. The crocidolite and anthophyllite powders are the same color as the bulk.

TABLE 7. DIFFRACTION PATTERN—CHRYSOTILE—EASTON, PA.

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
8.9	6	9.1	st.	1.747	2		
8.4	10	8.4	v. st.	1.685	3		
5.04	3	5.01	wk.	1.651	5		
4.84	5	4.77	wk.	1.631	2	1.631	st.
4.51	4	4.51	st.			1.613	st.
4.33	2			1.590	4	1.582	wk.
4.21	2	4.20	wk.	1.546	3	1.559	wk.
3.87	5			1.512	4	1.499	st.
3.69	4					1.451	wk.
3.37	11			1.439	5	1.432	wk.
3.26	8	3.28	v. st.	1.363	3	1.374	v. wk.
3.12	15	3.12	v. st.			1.353	wk.
3.02	7			1.336	2		
2.94	8					1.321	v. wk.
2.81	3	2.80	wk.	1.296	2.5	1.295	wk.
2.70	18	2.69	v. wk.			1.265	v. wk.
2.59	6	2.58	v. wk.			1.237	v. wk.
2.54	8			1.200	3		
2.40	2	2.38	wk.			1.188	wk.
2.34	6					1.122	st.
2.29	4					1.045	v. st.
2.28	4	2.22	v. wk.			1.028	v. wk.
2.169	5	2.14	v. wk.			1.017	wk.
2.043	2					.9776	st.
2.017	4	2.070	v. wk.			.9716	wk.
		1.998	wk.			.9512	wk.
		1.960	wk.			.9326	wk.
		1.889	wk.			.9095	wk.
1.864	3					.9022	wk.
1.812	6	1.807	wk.			.9005	wk.

SAMPLE PREPARATION AND PROCEDURE

Powder patterns were obtained for the front reflection region using the spectrometer with slit settings combining to give maximum resolution and intensity, i.e. 4 mm. high and .5 mm. wide. The samples were ground to pass 325 mesh and packed into a holder having a circular opening of 1.4 cm. in the manner described by the author in a previous paper.[1] Four or five runs were made for each sample and the mean values of the interplanar spacings, *d*, and the intensities tabulated. The intensity

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[1] Beatty, S., Van Dijke, *Am. Mineral.*, **34,** 74–82 (1949).

TABLE 8. DIFFRACTION PATTERN—CHRYSOTILE—LABRADOR

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
9.0	6	9.1	st.	1.871	3		
8.4	19	8.3	v. st.	1.850	1.5		
7.2	2			1.815	3	1.80	wk.
6.50	2			1.779	1.5	1.78	v. wk.
5.98	2			1.747	2	1.75	v. wk.
5.60	2			1.715	2		
5.24	2			1.688	4		
5.06	3	5.03	wk.	1.653	8		
4.87	4			1.639	2	1.63	st.
4.77	4			1.618	4	1.62	st.
4.51	4	4.49	st.	1.600	1.5		
4.39	1.5			1.585	5.5	1.58	wk.
4.21	3	4.17	v. wk.	1.558	3		
4.00	2			1.535	3.5		
3.88	3			1.514	4		
3.63	2	3.60	v. wk.	1.505	4	1.50	st.
3.56	2			1.473	2		
3.44	2	3.42	wk.	1.440	10	1.434	v. wk.
3.37	8			1.402	1.5		
3.27	10	3.26	st.	1.366	3	1.352	v. wk.
3.12	32	3.10	v. st.	1.339	2		
3.04	2			1.324	1.5	1.321	v. wk.
2.94	9			1.313	3		
2.80	4	2.78	wk.	1.295	3	1.299	v. wk.
2.71	16			1.272	1.5	1.269	v. wk.
2.67	7					1.243	v. wk.
2.60	6			1.201	2		
2.54	8			1.192	3	1.187	wk.
2.453	2			1.162	1.5	1.155	v. wk.
2.389	3	2.37	wk.			1.124	v. wk.
2.336	10					1.079	v. wk.
2.301	3					1.046	st.
2.279	4					1.019	wk.
2.209	2					.978	v. wk.
2.169	9	2.16	v. wk.			.952	v. wk.
2.136	2					.934	v. wk.
2.043	5	2.08	v. wk.			.9107	wk.
2.017	8	1.99	v. wk.			.9030	wk.
1.966	3	1.96	v. wk.			.9014	wk.
1.943	1.5					.8991	v. wk.
1.897	5	1.89	wk.				

scale is comparable to that obtained in a study of mica powders.[2] In the present case, the quartz line at $d=4.25$ has an intensity of 59.

Fine pencils of fibers were then separated from the bulk samples and inserted into a 114.9 mm. camera; the fiber axis being perpendicular to the beam. The purpose in obtaining this type of diffraction pattern in addition to the spectrometer powder pattern is threefold. A long film exposure may bring out weak lines which disappear into the background noise of an instantaneous electronic recording. It is further interesting to see how similar the film and powder patterns will be for identification purposes since they represent extremes in sample preparation. Last of all, a comparison of these pairs of patterns should give some indication of the randomness in the structure at right angles to the fibers axis. If the orientation about the fiber axis were random, the two patterns would agree closely. Strictly speaking, of course, this is not the conventional "fiber pattern."

Table 9. Diffraction Pattern—Chrysotile—Chester, Pa.

Spectrometer		Spectrometer		Spectrometer		Spectrometer	
d	Meas. Int.	d	Meas. Int.	d	Meas. Int.	d	Meas. Int.
8.6	7	2.54	9	3.29	5	1.653	3
5.12	1.5	2.40	1.5	3.13	9	1.583	3
4.85	3	2.34	3	2.95	4	1.537	2
4.55	4	2.17	3.5	2.81	2	1.518	4
4.48	4	2.04	2	2.76	4	1.467	1
4.23	2	2.02	3	2.70	10	1.440	4
3.91	1.5	1.867	2	2.60	5	1.340	2
3.39	5	1.688	2			1.302	3

On the whole the patterns are quite diffuse and weak, and in most cases the measurements are accurate at best to 0.1° in 2θ. Lines marked "w" are particularly wide or diffuse and in some cases the assigned value of 2θ is merely the center of a band or hump. All intensities of the spectrometer patterns were measured above the local background. The visual intensity scale for the film patterns is very rough and serves merely for comparison with corresponding spectrometer patterns.

DISCUSSION OF RESULTS

The pattern for Zermatt chrysotile given in Table 2 is sparse and quite

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[2] *Ibid.*

TABLE 10. DIFFRACTION PATTERN—CROCIDOLITE, SOUTH AFRICA

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
		9.2	st.			1.613	st.
8.4	13	8.4	v. st.			1.572	wk.
7.19	2			1.52	2	1.533	wk.
4.92	3	4.93	v. wk.			1.499	st.
4.52	4.5	4.51	st.	1.44	3	1.449	wk.
		4.20	v. wk.			1.420	st.
		3.89	v. wk.			1.369	wk.
		3.61	v. wk.			1.345	st.
3.42	2	3.41	wk.			1.310	v. wk.
3.36	4					1.291	st.
3.29	2	3.26	wk.			1.265	st.
3.11	8	3.11	v. st.			1.249	v. wk.
2.98	1.5	2.98	v. wk.			1.186	wk.
2.79	3.5	2.79	wk.			1.119	wk.
2.71	12	2.71	st.			1.100	wk.
2.61	3	2.60	wk.			1.080	wk.
2.54	6	2.53	v. wk.			1.056	wk.
2.48	1.5	2.44	wk.			1.041	st.
2.34	2	2.31	st.			1.027	st.
2.27	2					1.014	st.
2.18	2	2.18	st.			1.000	wk.
2.03	3	2.02	wk.			.9881	wk.
		1.990	v. wk.			.9787	wk.
		1.860	st.			.9704	wk.
		1.799	st.			.9460	wk.
		1.714	v. wk.			.9247	wk.
		1.684	v. wk.			.9167	wk.
		1.649	st.			.9097	wk.

diffuse although the reflections at $d=7.2$, and 3.62 are quite well defined, the very weak spectrometer lines did not appear on the film, and this is undoubtedly due to their very low intensity and diffuseness.

The pattern for Thetford chrysotile given in Table 3 is quite similar to that obtained from the Zermatt sample. Additional reflections appear but the pattern as a whole is still very diffuse.

Tables 4 and 5 give the diffraction data for the chrysotiles from Richmond, Canada, and Nevada, California. These are both extremely diffuse, particularly the latter. It is noted that the values of the interplanar spacings have shifted to larger values compared to the two previous samples.

The diffraction patterns of the chrysotiles from Corsica; Easton,

TABLE 11. DIFFRACTION PATTERNS—ANTHOPHYLLITE, GEORGIA

Spectrometer		Film		Spectrometer		Film	
d	Meas. Int.	d	Est. Int.	d	Meas. Int.	d	Est. Int.
9.3	6	9.4	st.			2.026	v. wk.
8.9	8	9.1	st.	1.991	4	1.982	v. wk.
8.26	14	8.25	v. st.			1.951	v. kw.
7.48	1.5			1.875	2.5		
5.04	3	5.05	v. wk.	1.839	5	1.838	st.
4.90	2			1.734	7	1.768	wk.
4.62	3			1.693	3		
4.50	6	4.51	v. wk.	1.639	2		
4.13	5	4.13	st.	1.618	7	1.610	st.
3.90	3			1.583	5		
3.65	9			1.549	4	1.542	st.
3.36	7	3.36	st.	1.530	2		
3.24	15	3.23	v. st.	1.514	5		
		3.12	wk.	1.503	7	1.498	wk.
3.05	26	3.04	v. st.	1.451	2		
2.87	5					1.443	wk.
2.84	10			1.419	6		
2.74	5	2.75	st.	1.364	3		
2.688	8			1.332	5		
2.590	7					1.319	wk.
2.540	10	2.52	v. wk.	1.308	2		
2.434	3			1.294	2.5		
2.318	5	2.350	v. wk.	1.260	1.5		
2.290	5					1.221	wk.
2.252	3	2.252	v. wk.			1.156	wk.
2.174	2			cf. Index Card		1.107	wk.
2.142	7			II 1320		1.100	wk.
2.074	2					1.028	st.
2.060	2					.926	st.

Pennsylvania; and Labrador given in Tables 6, 7, and 8 are all rich with fairly sharp lines. The values of the interplanar spacings have again shifted to even larger values. Furthermore, several strong lines appear in the powder sample which are absent or very weak in the fiber type pattern e.g. $d=2.71$, 3.37, 2.34. This would seem to indicate that the structures here are not as simple as that of the Thetford chrysotile where the distribution about the c axis is random.

The pattern of the chrysotile sample from Chester, Pennsylvania, given in Table 9 seems to fit in this latter group since it has a rich, fairly sharp pattern and the d values correspond closely. The reflection at $d=8.6$ is probably unresolved since the pattern as a whole is somewhat more diffuse.

TABLE 12. DIFFRACTION PATTERN OF THETFORD CHRYSOTILE OBTAINED BY WARREN AND BRAGG

Sin θ	d	Obs. Int.	*hk0*	Sin θ	d	Obs. Int.	*hk0*
.0484	7.33	v.s.	200	.0310	12.0	weak and too diffuse to be identified	110
.0968	3.67	v.s.	400	.0625	5.68		130
.1452	2.443	w.	600	.0990	3.58		150
.1936	1.832	w.	800	.0620	5.74		220
.2420	1.466	w.	1000	.0908	3.91		240
.2904	1.221	w.	1200	.1240	2.843		260
.3388	1.047	w.	1400	.0752	4.75		310
.3872	.916	w.	1600	.0925	3.84		330
.0772	4.60	w.	040	.1205	2.94		350
.2316	1.531	v.s.	0120	.1042	3.40		420
				.0386			020
				.1158	3.06	not observed	060
				.1544	2.29		080
				.1930	1.838		0100
				.2702	1.313		0140

TABLE 13. DIFFRACTION PATTERN OF ANTHOPHYLLITE BY WARREN AND MODELL

Sin θ	d	Obs. Int.	*hkl*	Sin θ	d	Obs. Int.	*hkl*
.039	9.1	w.	200	.110	3.23	s.	440
.078	4.58	w.m.	400	.115	3.09	v.s.	610
.117	3.03	w.	600	.130	2.74	s.	630
.156	2.27	w.m.	800	.195	1.82	m.s.	1010
.234	1.51	s.	1200				
				.140	2.53	v.s.	202
.040	9.4	w.m.	020	.150	2.36	m.s.	302
.080	4.42	m.s.	040	.160	2.22	w.	402
.120	2.95	w.	060	.170	2.08	s.	502
.160	2.21	w.	080				
.201	1.76	w.	0100	.070	5.1	w.	011
.241	1.47	v.s.	0120	.091	3.91	w.	031
.281	1.26	w.	0140	.210	1.69	m.s.	033
				.225	1.58	s.	053
.045	7.9	m.s.	210	.245	1.45	m.s.	073
.070	5.1	w.	230	.270	1.32	w.	024
.090	3.95	w.	420	.280	1.27	w.	044

The very rich pattern of crocidolite from South Africa is given in Table 10. Powder patterns were obtained using Cu and Fe radiation and although the background was reduced using the latter radiation no additional reflections appeared. Here the film gave a far richer pattern which was undoubtedly due to a high degree to the greater efficiency of the Co radiation.

The pattern of Georgia anthophyllite given in Table 11 is comparable to the Chester chrysotile in sharpness. The pattern is rich and shows the lack of correspondence between the powder and film intensities expected.

The diffraction data for the Thetford chrysotile obtained by Warren and Bragg[3] is given in Table 12 for reference. The indices of the reflections, the observed intensities and sin θ are listed. The corresponding d values are here added. Values for the strongest lines for anthophyllite are given in Table 13 from the work of Warren and Modell[4]. It is evident from a comparison of Tables 3 and 12 for Thetford chrysotile that the agreement is close for the two sets of data. The absence of weak lines in the present film patterns can be readily attributed to the lower efficiency of the Co radiation compared to both Cu and Mo. It is obvious, however, that the effect of different radiations will be one of damping or enhancing for the pattern as a whole such that the relative intensities remain the same.

It is interesting to note that all powder patterns were also obtained using an alcohol smear of the powder on a glass plate. On the whole, the same relative intensities were maintained although the background increased. In the case of the very diffuse patterns of Zermatt and Nevada, the stronger lines were considerably enhanced. In the latter case particularly, the only self respecting powder pattern was obtained in this fashion; when the holder was employed and random orientation was nearly obtained, the lines were sufficiently weak to be lost in the background noise. Thus, in powdered mixtures, it may be necessary to prepare the specimen in this manner before any asbestos lines can be expected. In most cases, only the prominent lines will appear so that the data of the weaker lines become primarily of academic interest.

CONCLUSIONS

X-ray diffraction patterns are given for 10 different asbestos samples including chrysotile, crocidolite, and anthophyllite.

Patterns obtained for various chrysotile samples show considerable variation in the number, sharpness and d values of the reflections.

The salient features of the typical asbestos patterns for identification purposes are maintained whether the pattern is obtained from a powder or fiber sample. In some cases, it may be advisable to enhance the weak lines of a powdered sample by using an alcohol smear.

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[3] *Zeit. Krist.*, **76,** 201–210 (1930).

[4] *Zeit. Krist.*, **75,** 161–178 (1930).