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DETERMINATION OF THE HEAVY ATOM CONTENT IN CHLORITE BY MEANS OF THE *X*-RAY DIFFRACTOMETER[1]

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ABSTRACT

A relationship between the approximate iron content in chlorite and intensity data obtained by means of the *x*-ray diffractometer has been established. Twenty-nine chemically analysed chlorites whose compositions range from those of the iron-rich varieties to those of the magnesium-rich varieties were studied. The results show that the intensities of *x*-rays diffracted from even-ordered basal planes of chlorite are proportional to the number of heavy atoms (Fe+Mn+Cr) in the octahedral layers of the mineral. A ratio of these intensities, expressed by the term $(I_{002}+I_{004})/I_{003}$, was selected as a variable factor representing the intensity, and its relationship to the number of atoms in the octahedral layers of chlorite was derived.

INTRODUCTION

Chlorites are a group of hydrous silicate minerals that have exceedingly varied chemical compositions. A number of schemes for subdividing them into species have been suggested (Winchell, 1936; Hey, 1954; Foster, 1962), and some relationships between their optical properties and chemical compositions have been established. Recent work has shown that it is possible to relate the approximate composition of chlorites to certain *x*-ray diffraction data (Brown, 1955; Brindley and Gillery, 1956; Shirozu, 1958; Eckstrand, 1962; Schoen, 1962). The approximate Si content can be determined by measuring the cell dimension *c*. The approximate Fe+Mn content can be determined either by measuring the cell dimension *b* from the 060 reflection or by comparing intensities of *x*-rays diffracted from basal planes. The former has been investigated by Shirozu (1958) and Eckstrand (1962) who showed that it can be used satisfactorily if there is no interference of the 060 reflection. The method based on intensity measurements proposed by Brindley and Gillery (1956), and tested to a limited extent by Schoen (1962), involves converting intensities to structure factors. The present investigation was undertaken to simplify the procedure and to provide an empirical relationship between intensity data and the Fe+Mn content in chlorite.

STRUCTURAL CHEMISTRY OF CHLORITES

Identification of a chlorite species by *x*-ray methods involves a consideration of the finer details of *x*-ray diffractometer data that are dependent on both the chlorite structure and the isomorphous substitution of ions.

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The chlorite structure consists of alternating layers of Si-O tetrahedra and layers composed of a variety of ions in octahedral co-ordination (Fig. 1). One octahedral layer is bound between two tetrahedral layers that, together, form a mica-like unit. Another lies between two mica-like units and forms a brucite-like layer. The chlorite structure is, therefore, interpreted as alternating mica-like units and brucite-like layers (Pauling, 1930; McMurchy, 1934; Brindley, 1951; Steinfink, 1958).

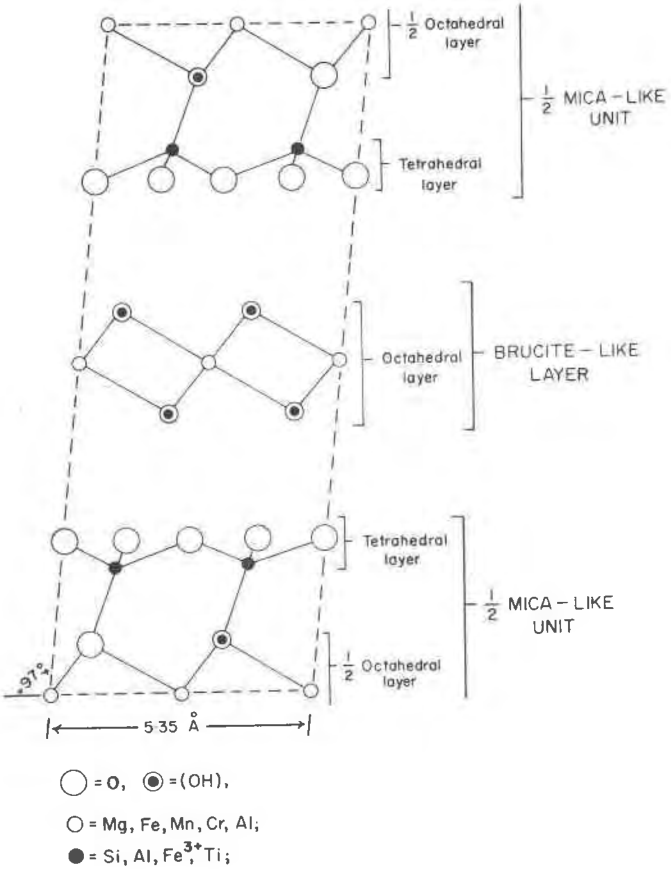

FIG. 1. Unit cell of chlorite 010 plane.

Al^{3+}, and probably minor amounts of Ti^{4+}, Fe^{3+} and Cr^{3+} replace silicon in the tetrahedral layers. Divalent and trivalent ions such as Mg^{2+}, Fe^{2+}, Mn^{2+}, Fe^{3+}, Cr^{3+}, and Al^{3+} replace one another in the octahedral layers.

X-RAY ANALYSIS OF CHLORITES BY MEANS OF INTENSITY MEASUREMENTS

Theoretical considerations The intensity of an *x*-ray beam diffracted from a particular plane in a mineral depends largely upon the diffracting powers of atoms in the mineral. Whenever isomorphous substitution occurs the diffracting powers of the new atoms may differ from those of the replaced atoms and the intensity of the diffracted *x*-ray beam is changed proportionately. The diffracting powers of heavy atoms that may be present in silicate minerals (*e.g.*, Fe, Mn, Cr) are about twice as large as the diffracting powers of the light atoms (*e.g.*, Mg, Si, Al). Isomorphous substitutions of light atoms by heavy atoms would, therefore, cause the intensity of particular reflections to increase.

The reflections that would be affected by such substitutions are determined by the sites at which replacement occurs. In chlorites the main replacement of light atoms by heavy atoms occurs in the octahedral layers and causes reflections from these layers to increase. Similar replacements in the tetrahedral layers are negligible.

X-rays diffracted from the basal planes of chlorite represent reflections from the octahedral and tetrahedral layers, and the intensity of a diffracted *x*-ray beam is a measure of the diffracting powers of atoms in these layers.

Intensities of x-rays diffracted from even-ordered basal planes When *x*-rays are diffracted from even-ordered basal planes of chlorite (002, 004, etc), reflections from the octahedral layers are in phase and reinforce each other. Thus, if the heavy atom content in the octahedral layers is increased, the intensity of an *x*-ray beam diffracted from an even-ordered basal plane will increase proportionately.

Intensities of x-rays diffracted from odd-ordered basal planes When *x*-rays are diffracted from odd-ordered basal planes of chlorite (001, 003, etc.), reflections from the octahedral layers are out of phase and cancel each other. If the two types of octahedral layers contain the same number of light and heavy atoms, reflections from the octahedral layers will be cancelled completely. Under these conditions the intensity of an *x*-ray beam diffracted from an odd-ordered basal plane represents only contributions from the tetrahedral layers.

If the two types of octahedral layers contain different numbers of light and heavy atoms, *x*-ray reflections from the octahedral layers will not cancel each other completely. Under these conditions a diffracted *x*-ray beam will represent contributions from the tetrahedral layers plus uncancelled contributions from the octahedral layers.

It is possible to determine whether the different octahedral layers contain unequal amounts of light and heavy atoms from the ratios of intensities of *x*-rays diffracted from the odd-ordered basal planes. The intensity of an *x*-ray beam diffracted from the 001 plane and the 005 plane increases, and that diffracted from the 003 plane decreases, when the heavy atom content in the octahedral layer of the mica-like unit increases over that in the brucite-like layer. The intensity of an *x*-ray beam diffracted by the 001 plane, however, is unreliable due to the low angle of diffraction and, therefore, only I_{003} and I_{005} can be used with confidence. The theoretical relationship between the ratio I_{003}/I_{005} and the number of heavy atoms in the octahedral layer of the mica-like unit minus the number of heavy atoms in the brucite-like layer was calculated and the results plotted in Fig. 2.

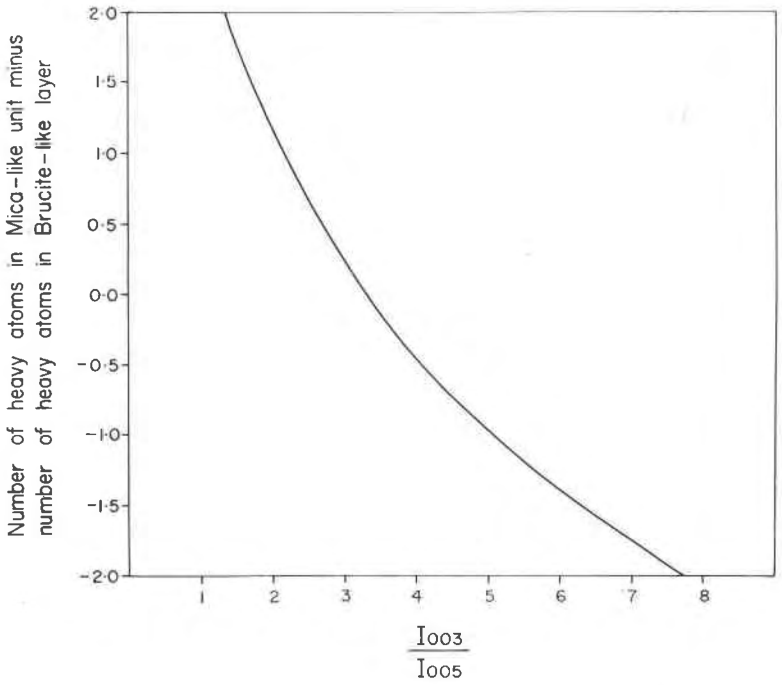

FIG. 2. Relationship between "degree of asymmetry" (number of heavy atoms in mica-like octahedral layer minus number of heavy atoms in brucite-like layer) and the ratio I_{003}/I_{005}.

The numerical expression for the number of heavy atoms in the octahedral layer of the mica-like unit minus the number of heavy atoms in the brucite-like layer is defined by the writer as the "degree of asymmetry" and will be used in this manner throughout the remainder of the paper.

Relationship between intensities and heavy atom content in chlorite

(1) Symmetrical distribution

The preceding sections show that if the two types of octahedral layers contain an equal number of heavy atoms (symmetrical distribution) the following relationships are valid: (a) the intensity of an *x*-ray beam diffracted from an even-ordered basal plane increases in proportion to the number of heavy atoms in the octahedral layers; (b) the intensity of an *x*-ray beam diffracted from an odd-ordered basal plane is independent of the heavy atom content in the two types of octahedral layers and is relatively constant. Under these conditions a ratio of the above intensities would be a measure of the heavy atom content in chlorite. The ratio $(I_{002}+I_{004})/I_{003}$ was selected as this measure because I_{002} and I_{004} are the strongest even-ordered basal reflections and the position of I_{003} is relatively near the 002 and 004 reflections.

(2) Asymmetrical distribution

When the two types of octahedral layers contain unequal numbers of heavy atoms ("asymmetrical distribution") the intensity of an *x*-ray beam diffracted from an even-ordered basal plane is still proportional to the number of heavy atoms in the octahedral layers, but that diffracted from an odd-ordered basal plane is no longer constant. Under these conditions the ratio $(I_{002}+I_{004})/I_{003}$ is not a reliable measure of the heavy atom content in the octahedral layers. The value of I_{003} can, however, be corrected to the value it should have with a "symmetrical distribution" of heavy atoms in the octahedral layers, and the ratio $(I_{002}+I_{004})/I_{003\ \text{corrected}}$ would then be a measure of the heavy atom content in the octahedral layers.

A correction factor for the odd-ordered planes can be developed from the equation:

$$I \alpha F^2 \text{ L.P.} \qquad 1)$$

where I is the intensity of an *x*-ray beam diffracted from a particular plane, F is the structure factor, and L.P. is the Lorentz polarization factor. From equation 1 the following relationship may be derived:

$$\frac{I_{\text{sym}}}{I_{\text{asym}}} = \frac{F^2_{\text{sym}}}{F^2_{\text{asym}}} \qquad 2)$$

where I_{sym} is the intensity diffracted from a basal plane when both octahedral layers contain equal numbers of light and heavy atoms ("symmetrical distribution"), and I_{asym} is the observed intensity, which is the intensity diffracted when the octahedral layers have an "asymmetrical distribution" of heavy atoms. In chlorite F_{sym} for the 003 plane is 55.5; with an asymmetrical distribution F_{asym} is 55.5 minus "degree of asymmetry"×12.3. 12.3 is the difference between the effective diffracting powers (atomic scattering factors) of Mg and Fe for the 003 plane of chlorite irradiated with either iron, copper or cobalt radiation.

For the 003 plane of chlorite irradiated with either FeKα, CoKα or CuKα, equation 2 gives:

$$I_{\mathrm{sym}} = I_{\mathrm{asym(observed)}} \left(\frac{(55.5)^2}{(55.5 - \text{“degree of asymmetry”} \times 12.3)^2} \right) \qquad 3)$$

$I_{003\ \mathrm{corrected}}$ is equal to I_{sym}.

Intensity calculations Theoretical structure factors were calculated for the basal planes of chlorites irradiated with Cu, Fe, and CoKα radiation. The calculations were made according to the method outlined by Brindley and Gillery (1956) and Petruk (1959) by assuming that the atom positions in chlorite are those reported by Brindley (1951) and that the composition varies between $(MgAl)_6(SiAl)_4O_{10}(OH)_8$ and $R_6(AlSi)_4O_{10}(OH)_8$, where R is Fe+Mn+Cr. In these calculations the atomic scatter-

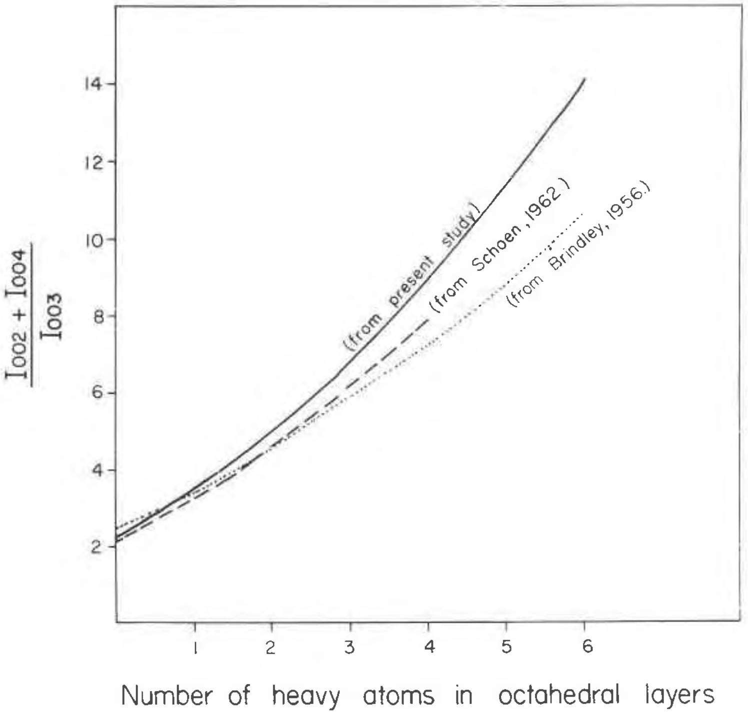

FIG. 3. Theoretical relation between number of heavy atoms in octahedral layers of chlorite and $(I_{002}+I_{004})/I_{003}$ for FeKα, CuKα and CoKα radiation.

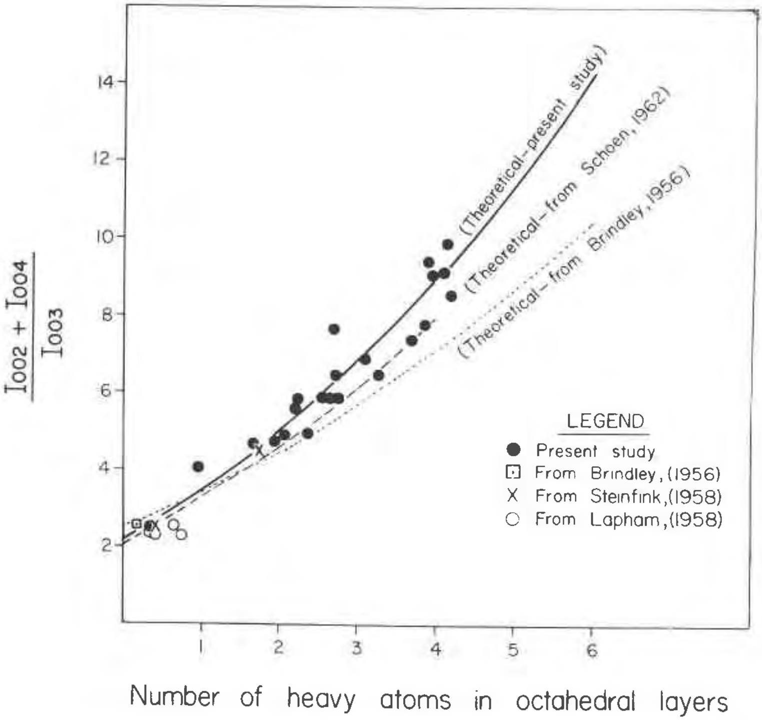

FIG. 4. Observed relationship between $(I_{002}+I_{004})/I_{003}$ and number of heavy atoms in octahedral layers in chlorite (FeKα, CuKα and CoKα radiation).

ing factors listed in International Tables (1935) were used. Since Fe, Mn and Cr have similar atomic scattering factors, only those for Fe were used. Theoretical ratios of $(I_{002}+I_{004})/I_{003}$ were then calculated from the structure factors and the results plotted in Fig. 3.

Brindley and Gillery (1956) and Schoen (1962) reported the calculated structure factors for the basal planes of chlorite. These structure factors were recalculated to $(I_{002}+I_{004})/I_{003}$ and plotted in Fig. 3. It is noted that the curve, obtained from values calculated by the writer, does not superimpose upon either the one recalculated from Brindley and Gillery's structure factors or the one recalculated from the structure factors reported by Schoen. The reason for the discrepancy between the curves is not known.

Actual relationship between the heavy atom content in chlorite and the ratio $(I_{002}+I_{004})/I_{003}$ The validity of the theoretical relationship between the number of heavy atoms in the octahedral layers in chlorite and the ratio $(I_{002}+I_{004})/I_{003\text{ corrected}}$ was tested by using twenty-nine chemically ana-

TABLE 1. ANALYSES AND STRUCTURAL FORMULAE OF CHLORITES USED IN THE PRESENT STUDY

Sample No.	Chemical analysis											Octahedral positions occupied						Tetrahedral position occupied by			Total heavy* atoms in octahedral layers
	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	Cr_2O_3	FeO	MgO	MnO	CaO	H_2O^-	H_2O^+	Al^{3+}	Fe^{3+}	Cr^{3+}	Fe^{2+}	Mg^{2+}	Mn^{2+}	Si^{4+}	Ti^{4+}	Al^{3+}	
Brindley #2[1]	31.85	—	21.82	1.91	—	—	30.00	—	0.94	—	11.79	1.39	0.14	—	—	4.22	—	2.97	—	1.03	0.14
Clark L. A.[8]	31.62	0.31	16.10	2.00	0.15	2.69	33.93	0.10	—	—	13.16	0.83	0.15	—	0.21	4.80	—	3.00	0.03	0.97	0.36
Lapham #2[3]	31.18	tr	17.68	1.88	1.14	1.72	32.58	0.01	0.10	0.48	12.93	0.89	0.14	0.09	0.14	4.72	—	2.92	—	1.08	0.37
Steinfink[3]	27.40	—	18.90	2.40	2.30	0.80	34.00	—	—	—	—	0.80	0.17	0.17	0.06	4.90	—	2.65	—	1.31	0.40
Lapham #10[2]	32.08	tr	13.98	0.42	3.40	1.36	34.66	0.02	0.72	0.32	12.93	0.51	0.03	0.27	0.11	5.16	—	3.03	—	0.97	0.41
Lapham #24[2]	32.22	tr	8.66	0.46	6.47	1.38	35.06	tr	1.04	0.58	13.82	0.07	0.04	0.52	0.11	5.35	—	3.18	—	0.82	0.67
Lapham #11[2]	32.12	—	9.50	—	7.88	1.98	35.36	—	1.24	—	10.52	0.19	—	0.60	0.15	5.09	—	3.11	—	0.89	0.75
63(4)	33.00	0.30	24.00	—	—	12.00	23.00	0.50	—			1.53	—	—	0.93	3.20	0.04	3.06	0.02	0.92	0.97
64(4)‡	32.20	0.40	11.73	—	—	19.85	26.95	0.53	—			0.54	—	—	1.63	3.92	0.05	3.15	0.03	0.82	1.68
Steinfink[3]	25.40	—	22.80	2.86	—	17.77	19.09	—	—		12.21	1.32	0.22	—	1.51	2.89	—	2.59	—	1.41	1.73
61[4]	29.45	0.60	13.32	—	—	22.18	23.18	0.81	—			0.67	—	—	1.89	3.53	0.07	3.01	0.05	0.94	1.96
3058[5]‡	27.80	1.01	19.55	4.62	—	20.73	19.40	0.87	—			1.02	0.33	—	1.69	2.81	0.07	2.71	0.07	1.22	2.09
7[5]	27.10	0.20	21.40	3.70	—	22.20	15.90	0.60	0.10			1.29	0.28	—	1.87	2.39	0.05	2.74	0.01	1.25	2.20
1[5]	26.60	0.30	21.80	2.80	—	23.30	15.60	0.60	0.20			1.33	0.21	—	1.97	2.34	0.05	2.69	0.02	1.29	2.23
3[5]	24.80	0.90	21.00	3.90	—	22.90	15.60	0.60	0.40			1.21	0.31	—	1.99	2.41	0.05	2.57	0.07	1.36	2.35
NH-6[6]	Analysis not available											1.29	—	—	2.54	2.16	0.01	2.71	—	1.29	2.55
42[5]	24.00	0.10	21.40	7.10	—	23.30	13.60	0.50	0.10			1.14	0.56	—	2.03	2.11	0.04	2.50	0.01	1.49	2.63
212-2A[7]	25.92	0.13	23.70	1.70	—	27.67	8.66	0.30	0.36			1.66	0.14	—	2.53	1.41	0.03	2.70	0.01	1.29	2.70
66[4]	Analysis not available											1.40	—	—	2.71	1.72	—	2.95	—	1.05	2.71
Ch65[11]†	29.61	0.35	22.85	2.86	—	31.50	10.30	0.36	—			1.53	0.20	—	2.51	1.46	0.03	2.82	0.03	1.15	2.74
197-2[7]	23.62	0.04	23.04	2.20	—	31.18	8.64	0.15	—	0.20	11.60	1.44	0.18	—	2.91	1.39	0.01	2.51	—	1.49	3.10
33[8]‡	26.10	2.00	11.75	6.90	—	28.45	13.10	0.70	0.28	0.28	11.00	0.54	0.57	—	2.61	2.14	0.06	2.86	0.16	0.98	3.24
F-87[9]	22.81	—	20.35	4.84	—	34.87	6.11	0.56	0.11	0.23	10.80	1.21	0.40	—	3.24	1.01	0.05	2.54	—	1.46	3.69
F-147[9]	23.43	—	18.21	4.49	—	36.30	5.41	tr	—	tr	12.15	1.13	0.39	—	3.47	0.92	—	2.68	—	1.32	3.86
F-145[9]	23.38	—	19.32	2.51	—	38.38	5.39	0.58	—	0.48	10.62	1.20	0.21	—	3.61	0.90	0.06	2.63		1.37	3.88
15-4[3 10]	24.50	—	16.32	7.45	—	31.46	4.59	3.33	—		11.36	1.03	0.62	—	3.03	0.75	0.33	2.82	—	1.18	3.98
F-137[9]	21.17	0.07	20.02	9.88	—	33.48	3.10	0.45	0.10	0.57	10.63	1.14	0.85	—	3.21	0.53	0.04	2.43	—	1.57	4.10
F-149[9]	23.32	0.03	17.45	4.09	—	38.90	4.54	0.01	0.24	0.80	10.89	1.06	0.36	—	3.75	0.78	—	2.69	—	1.31	4.11
F-88[9]	22.24	—	17.05	13.38	—	26.26	4.10	5.42	tr	0.98	10.05	0.84	1.15		2.51	0.70	0.53	2.54	—	1.46	4.19

* Heavy atoms = Fe + Mn + Cr.

† Analysis corrected for muscovite.

‡ Analysis corrected for muscovite plus quartz.

[1] Chemical analysis of Sample reported by Pask and Warner (1954).

[2] Chemical analyses of Sample Nos. 2, 10, 24, and 11 reported by Lapham (1958).

[3] Chemical analyses of samples reported by Steinfink (1958).

[4] Samples analyzed spectrochemically by Oiva Joensuu.

[5] Samples analyzed chemically by Warnock and Hersey, Montreal.

[6] Chemical analysis of Sample No. NH-6 reported by Morse (1958).

[7] Chemical analyses of sample Nos. 212-2A and 197-2 reported by E-an Zen (1960).

[8] Chemical analysis reported by Analytical Chemistry Subdivision, Mineral Sciences Division, Mines Branch, Ottawa, Internal Reports MS-AC-62-421 and MS-AC-62-1417.

[9] Chemical analyses reported by Foster (1962).

[10] Chemical analysis supplied by Dr. J. M. Moore, Carleton University, Ottawa.

Table 2. Intensity Ratios of *X*-Rays Diffracted from Basal Planes of Chlorite

Sample	No. of heavy atoms in octahedral layers	I_{003}/I_{005}	"Degree of asymmetry" (Fig. 2)	$(I_{002}+I_{004})/I_{003}$	$(I_{002}+I_{004})/I_{003\text{ corrected}}$
Brindley #2[1]	0.14	3.39	−0.05	2.52	2.58
Clark, L. A.	0.36	3.80	−0.36	2.03	2.47
Lapham #2[2]	0.37	3.54	−0.15	2.20	2.35
Steinfink[3]	0.40	3.44	−0.10	2.41	2.52
Lapham #10[2]	0.41	3.72	−0.27	2.03	2.30
Lapham #24[2]	0.67	4.12	−0.52	2.07	2.57
Lapham #11[2]	0.75	4.34	−0.62	1.80	2.31
63	0.97	3.52	−0.15	3.76	4.04
64	1.68	3.50	−0.14	4.13	4.40
Steinfink[3]	1.73	2.53	+0.60	6.11	4.58
61	1.96	3.42	−0.06	4.65	4.79
3058	2.09	3.31	0.00	4.95	4.95
7	2.20	3.31	0.00	5.63	5.63
1	2.23	3.31	0.00	5.81	5.81
3	2.35	3.31	0.00	4.97	4.97
NH-6	2.55	3.31	0.00	5.87	5.87
42	2.63	3.31	0.00	5.87	5.87
212-2A	2.70	3.67	−0.23	7.00	7.75
66	2.71	3.20	+0.10	6.60	6.47
Ch 65	2.74	2.86	+0.35	6.65	5.90
197-2	3.10	3.23	+0.07	7.10	6.85
33	3.24	3.23	+0.07	6.65	6.45
F-87	3.69	2.63	+0.54	9.55	7.40
F-147	3.86	2.75	+0.40	9.45	7.85
F-145	3.88	2.60	+0.54	12.15	9.43
15-4(3)	3.98	3.04	+0.20	9.86	9.05
F-137	4.10	3.29	+0.03	9.24	9.14
F-149	4.11	3.57	−0.16	9.22	9.94
F-88	4.19	3.33	−0.01	8.50	8.55

[1] Ratios recalculated from those reported by Brindley (1956).

[2] Ratios calculated from intensities reported by Lapham (1958).

[3] Ratios calculated from observed structure factors reported by Steinfink (1958).

All other ratios determined from measurements made by the writer.

lyzed chlorites whose value of $(I_{002}+I_{004})/I_{003}$ could be obtained. The chemical analyses, calculated structure formulae and the heavy atom content of the chlorites are given in Table 1. The structural formulae and the heavy atom contents were calculated from the chemical analyses according to the method outlined by Foster (1962).

The values of $(I_{002}+I_{004})/I_{003}$ for twenty-two of the chlorites were obtained from intensity measurements made by the writer, and the values of $(I_{002}+I_{004})/I_{003}$ for the remaining seven chlorites were calculated from observed intensities or structure factors reported in the literature (Table 2). Intensity measurements for the twenty-two chlorites studied by the writer were made on finely powdered samples mounted in either standard aluminum sample holders, or in copper sample holders recessed 0.002 inches. The samples were scanned in duplicate using FeKα and in some cases, CoKα and CuKα radiation on both General Electric and Philips *x*-ray diffractometers. The average ratios of I_{003}/I_{005} and $(I_{002}+I_{004})/I_{003}$ were determined from the intensities (peak height minus background) and the value of I_{003} was corrected by use of equation 3 and Fig. 2. The values of $(I_{002}+I_{004})/I_{003\ \text{corrected}}$ were then determined (Table 2) and plotted in Fig. 4. The plotted points fall close to the calculated curve, and an empirical curve obtained from these points superimposes upon the calculated one.

CONCLUSIONS

The approximate number of heavy atoms in the octahedral layers of chlorite can be determined directly from intensities of *x*-rays diffracted from the basal planes of this mineral. Since Fe is generally the major heavy atom in chlorite this approach provides an approximation of the Fe content in chlorite. The advantages of this method are its greater simplicity in that it does not involve conversion of intensities to structure factors, and that determinations are possible even when the chlorite has not been separated from the other minerals in the sample. Results obtained for the chlorites used in this investigation are in close agreement with theoretical values.

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