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NOTES

REFINEMENT OF A ONE-LAYER TRICLINIC CHLORITE

Key Words—Cation ordering, Chlorite, Crystal structure, X-ray diffraction.

Chlorite minerals commonly exhibit stacking disorder. Among those with ordered arrangements, the one-layer triclinic II*b*-4 structure is most abundant. The II*b*-4 structure was first determined by film methods (Steinfink, 1958) and subsequently refined from X-ray diffraction data by Phillips *et al.* (1980) and Zheng and Bailey (1989) and from neutron diffraction by Joswig *et al.* (1980).

The X-ray diffraction structure of the triclinic II*b*-4 chlorite presented here is the base for a comparison of the structural details of this polytype with a coexisting monoclinic II*b*-2 chlorite (Joswig *et al.*, 1989).

EXPERIMENTAL

A single crystal of II*b*-4 polytype was chosen from a sample from the Achmatow mine, Ural Mountains, U.S.S.R., which contained disordered and semi-ordered chlorites, several one-layer triclinic, two one-layer monoclinic, one two-layer triclinic, and one two-layer monoclinic polytypes of very poor quality. Microprobe analyses at eight positions in a single grain: average (in wt. %) SiO_2, 29.31; Al_2O_3, 18.50; MgO, 31.32; MnO, 0.13; K_2O, 0.04; Na_2O, 0.11; and TiO_2, Cr_2O_3, PO_4, and CaO, <0.05; and by wet-chemical analysis: FeO, 3.34 and Fe_2O_3, 2.52. The calculated chemical formula on the basis of 28 positive charges is: $(Mg_{4.54}Al_{0.97}Fe^{2+}_{0.28}Fe^{3+}_{0.18}Mn_{0.01})(Si_{2.85}Al_{1.15})O_{10}(OH)_8$ and indicates that these chlorites are clinochlore (Bayliss, 1975).

The crystal of the triclinic II*b*-4 polytype had dimensions of 0.4 × 0.18 × 0.05 mm and was of good quality (i.e., no streaks parallel to c*). A NONIUS CAD4 diffractometer was used for data collection. Unit-cell constants were refined from 25 reflections as follows: a = 5.325(2), b = 9.234(5), c = 14.358(6) Å, α = 90.33(4), β = 97.38(3), γ = 90.00(4)°. Altogether, 2459 reflections—to $\sin\theta/\lambda$ = 0.7027—were collected with MoKα radiation monochromatized by pyrolytic graphite. After an empirical psi-scan absorption correction, the data set was averaged to 1966 reflections. For the refinement, 1900 reflections ($F_0 > 5\sigma$) were used, and a weighting scheme $\omega(F_0) = [5\sigma + 0.0001 \cdot F_0^2 + 0.5]^{-1}$ was employed. All atomic coordinates, anisotropic temperature factors of the nonhydrogen atoms, and an isotropic extinction parameter were refined with 143 variables. A final reliability factor R = 0.047 and a weighted R_w = 0.057 was achieved in space group $C\bar{1}$.

The parameters are given in Table 1 and the bond-lengths in Table 2.

Table 1. Atomic coordinates and thermal parameters ($U_{ij} \times 10^{-2}$) of clinochlore, Achmatow mine, U.S.S.R.

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
$M_t(1)$	0.00	0.00	0.00	0.48(5)	0.47(5)	0.96(5)	0.05(4)	0.09(4)	0.04(4)
$M_t(2)$	0.00127(16)	0.33382(9)	0.00003(6)	0.44(4)	0.52(4)	0.98(4)	−0.02(3)	0.12(3)	0.06(3)
T(1)	0.23211(15)	0.16809(8)	0.19257(6)	0.59(4)	0.51(4)	0.93(4)	−0.02(3)	0.13(3)	0.05(3)
T(2)	0.73204(15)	0.00138(8)	0.19252(5)	0.59(4)	0.55(4)	0.88(4)	0.01(3)	0.14(3)	0.01(3)
O(1)	0.1925(4)	0.1679(2)	0.0770(1)	0.78(9)	0.82(9)	1.14(9)	−0.08(7)	0.20(7)	0.02(7)
O(2)	0.6927(4)	0.0008(2)	0.0769(1)	0.82(10)	0.82(9)	1.13(9)	0.02(7)	0.12(7)	0.05(7)
O(3)	0.2081(5)	0.3348(3)	0.2337(2)	1.92(12)	1.29(10)	1.54(10)	−0.04(9)	0.30(8)	0.01(8)
O(4)	0.5146(4)	0.1033(3)	0.2341(2)	1.01(10)	1.80(11)	1.59(10)	0.38(9)	0.19(9)	−0.07(8)
O(5)	0.0153(4)	0.0664(3)	0.2338(2)	1.24(11)	1.93(11)	1.62(10)	−0.37(9)	0.10(8)	0.12(8)
O(6)	0.6919(4)	0.3338(2)	0.0733(2)	1.06(10)	1.02(10)	1.12(9)	0.12(8)	0.25(7)	0.11(7)
H(1)	0.717(11)	0.340(6)	0.135(4)	2.00					
$M_b(1)$	−0.00044(17)	0.16666(9)	0.49994(6)	0.66(4)	0.69(4)	1.44(4)	−0.04(3)	0.18(3)	0.06(3)
$M_b(2)$	0.00	0.50	0.50	0.33(5)	0.38(5)	1.01(5)	−0.09(4)	0.12(4)	0.05(4)
O(7)	0.1528(5)	−0.0007(3)	0.4301(2)	1.69(11)	1.66(11)	1.20(10)	−0.30(9)	0.12(8)	0.12(8)
O(8)	0.1374(5)	0.3370(3)	0.4302(2)	1.95(12)	1.84(11)	0.96(10)	−0.14(9)	0.15(8)	0.01(8)
O(9)	0.6382(4)	0.1617(3)	0.4306(2)	1.20(10)	1.82(11)	1.12(10)	0.64(9)	0.08(8)	−0.13(8)
H(2)	0.128(11)	−0.001(7)	0.369(4)	2.00					
H(3)	0.125(12)	0.335(6)	0.375(5)	2.00					
H(4)	0.613(11)	0.151(7)	0.369(5)	2.00					

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Table 2. Interatomic distances (Å) of clinochlore, Achmatow mine, U.S.S.R.

Tetrahedra			
T(1)–O(1)	1.646(2)	T(2)–O(2)	1.647(2)
–O(3)	1.657(2)	–O(4)	1.659(3)
–O(4)	1.659(2)	–O(5)	1.659(2)
–O(5)	1.657(3)	–O(3)	1.662(3)
Mean:	1.655	Mean:	1.657
Octahedra			
$M_t(1)$–O(1)	2.089(2) ×2	$M_t(2)$–O(1)	2.082(2)
–O(2)	2.088(2) ×2		2.085(2)
–O(6)	2.061(2) ×2	–O(2)	2.080(2)
Mean:	2.079		2.084(2)
		–O(6)	2.066(3)
			2.066(2)
		Mean:	2.077
$M_b(1)$–O(7)	2.062(3)	$M_b(2)$–O(8)	1.994(3) ×2
	2.060(3)	–O(7)	1.987(2) ×2
–O(8)	2.055(3)	–O(9)	1.992(2) ×2
	2.061(3)	Mean:	1.991
–O(9)	2.050(2)		
	2.052(3)		
Mean:	2.057		

RESULTS AND DISCUSSION

Joswig *et al.* (1989) reported the neutron diffraction refinement of a coexisting monoclinic II*b*-2 polytype. Both the II*b*-2 and the II*b*-4 polytypes showed a similar ordering pattern in the 2:1 layer and the interlayer sheets; the octahedral cations in the 2:1 layer were disordered ($M_t(1)$–O = 2.079 Å; $M_t(2)$–O = 2.077 Å), whereas Al^{VI} predominantly occupies the $M_b(2)$ site ($M_b(1)$–O = 2.057 Å; $M_b(2)$–O = 1.991 Å) compared with $M_b(1)$–O = 2.055 Å and $M_b(2)$–O = 1.997 Å of the coexisting monoclinic polytype, where the subscript t and b represent the 2:1 layer and brucite-like sheet, respectively. Thus, as a consequence no significant difference within experimental error was found between the coexisting monoclinic II*b*-2 and the triclinic II*b*-4 polytypes, in contrast with the finding of Zheng and Bailey (1989) for intergrown monoclinic and triclinic polytypes. The two independent T sites were found to be disordered: T(1)–O = 1.655, T(2)–O = 1.657 Å. This result is identical to the neutron diffraction refinement of a triclinic II*b*-4 penninite (Joswig *et al.*, 1980).

The refined hydrogen positions in the triclinic polytype were nearly the same as determined by neutron diffraction of a penninite sample (Joswig *et al.*, 1980). Therefore, the same hydrogen bonding pattern must be present. The high estimated standard deviations, however, prevent a detailed discussion. As noted by Phillips *et al.* (1980), the ordering of a trivalent cation in the M(4) (= $M_b(2)$) site increased the angle α of the lattice constants. Table 3 gives the dependence of the angle α from the degree of ordering in the two independent octahedral positions of the interlayer sheet.

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Table 3. Angle α in triclinic II*b* chlorite samples as an indicator of cation ordering in the interlayer sheet.

	α (°)	$M_b(1)$ (Å)	$M_b(2)$ (Å)	Δ (Å)
Penninite (Joswig *et al.*, 1980)	89.95(1)	2.045	2.023	0.022
Clinochlore (this work)	90.33(4)	2.057	1.991	0.066
Chromian clinochlores (Phillips *et al.*, 1980)	90.45(3)	2.069	1.963	0.106
	90.53(6)	2.075	1.960	0.115
Clinochlore (Zheng and Bailey, 1989)	90.48(2)	2.078	1.956	0.122

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