Refined muscovite structure

THE structure of the $2M_1$ polymorph of muscovite has been refined by a least-squares analysis of the data of Radoslovich (1960). The purpose of this communication is to present in one report both the refined atomic co-ordinates and temperature factors and the pertinent bond distances and angles. Previously, Gatineau (1963) has presented refined atomic co-ordinates based on the original data of Radoslovich, and Burnham and Radoslovich (1963–64) have presented interatomic distances and bond angles for two additional $2M_1$ micas, one of which was potash rich (K = 0·66) and muscovite-like and the other soda-rich (K = 0·15) and paragonite-like.

The refinement was carried out using a modified version of the least-squares and function and error programmes of Busing, Martin, and Levy (1962 and 1964) translated into Fortran IV. The final atomic co-ordinates and isotropic temperature factors of Radoslovich were used as initial parameters. Atomic scattering factors for half-ionized atoms were used except for potassium which was assumed to be fully ionized. These data were taken from the International Tables for X-ray Crystallography. The scattering factor curve for octahedral Al was modified to include the amount of iron shown in the chemical analysis of Radoslovich (Al = 0·94). The unit cell dimensions reported by Radoslovich were b = 8·996±0·006 Å, c = 20·096±0·02 Å, and β was assumed to be 95° 11′ ($b = \sqrt{3}\ a$). A three-cycle refinement with no weighting scheme was employed after which the shift in all parameters was less than the errors. The refinement was carried out in the space group $C2/c$ and the final unweighted R-factor for all measured and estimated reflections was 12 % compared to 17 % for the original data.

An attempt to refine the temperature factors on an anisotropic basis was unsuccessful because of insufficient data. A further attempt was made to reduce the R-factor by employing a scheme whereby all visually estimated reflections were assumed to be 100 % in error. No significant change in the parameters was noted and the co-ordinates from the unweighted refinement (table I) were utilized to compute the bond angles and distances (table II).

Several observations from these refined data, some of which have been noted in the more recent structure analyses, may be made. The co-ordination polyhedra are more regular than originally presumed (table II). Individual layers of atoms are very nearly planar with the exception of the depression of O_D in the basal oxygen layer. The temperature

TABLE I. Atomic parameters for muscovite (as fractions of unit cell dimensions), together with final temperature factors and their errors

	Radoslovich			Final co-ordinates			Error		
	$x \times 10^4$	$y \times 10^4$	$z \times 10^4$	$x \times 10^4$	$y \times 10^4$	$z \times 10^4$	$\Delta x \times 10^4$	$\Delta y \times 10^4$	$\Delta z \times 10^4$
Al	2484	871	16	2514	848	4	13	7	3
O_A	4650	9450	527	4607	9440	528	27	15	7
O_B	4250	2600	542	3794	2530	534	24	16	6
OH	4530	5580	520	4585	5608	509	24	14	6
$SiAl_1$	4625	9242	1372	4659	9286	1352	11	6	3
$SiAl_2$	4593	2550	1365	4515	2576	1351	10	6	3
O_C	4080	960	1680	4138	0951	1680	26	17	6
O_D	2450	8020	1620	2508	8082	1567	27	16	7
O_E	2629	3713	1674	2552	3710	1685	25	15	7
K	0	1016	2500	0	991	2500	—	9	—

Final temperature factors

	Al	O_A	O_B	OH	$SiAl_1$	$SiAl_2$	O_C	O_D	O_E	K
B	0.74	0.80	0.88	0.40	0.71	0.49	1.17	1.18	0.88	1.64
ΔB	0.08	0.22	0.21	0.20	0.09	0.08	0.22	0.25	0.22	0.12

TABLE II. Bond lengths, interatomic distances, and bond angles. R, Radoslovich's values; BT, new refined values

Tetrahedral groups

			Around SiAl$_1$		Around SiAl$_2$	
	R	BT		R	BT		R	BT
SiAl$_1$–O$_C$	1·69$_5$ Å	1·67$_2$ Å					
SiAl$_1$–O$_D$	1·68$_2$	1·64$_2$	O$_C$–O$_D$	2·77$_5$ Å	2·72$_2$ Å	O$_C$–O$_D$	2·58$_3$ Å	2·62$_2$ Å
SiAl$_1$–O$_E$	1·68$_9$	1·67$_1$	O$_C$–O$_E$	2·73$_3$	2·68$_2$	O$_C$–O$_E$	2·58$_8$	2·62$_2$
SiAl$_1$–O$_A$*	1·71$_0$	1·66$_1$	O$_D$–O$_E$	2·74$_9$	2·67$_2$	O$_D$–O$_E$	2·59$_1$	2·67$_2$
SiAl$_2$–O$_C$	1·59$_6$	1·62$_2$	O$_A$*–O$_C$	2·74$_3$	2·71$_2$	O$_B$*–O$_C$	2·74$_3$	2·70$_2$
SiAl$_2$–O$_D$	1·58$_1$	1·64$_2$	O$_A$*–O$_D$	2·87$_0$	2·73$_2$	O$_B$*–O$_D$	2·80$_6$	2·75$_2$
SiAl$_2$–O$_E$	1·62$_3$	1·63$_1$	O$_A$*–O$_E$	2·74$_1$	2·75$_2$	O$_B$*–O$_E$	2·73$_2$	2·68$_2$
SiAl$_2$–O$_B$*	1·64$_8$	1·65$_1$					

Octahedral groups

			Around Al				
	R	BT		R	BT		R	BT
Al–O$_A$	1·93$_5$ Å	1·93$_2$ Å	O$_A$–O$_A$	2·39$_6$ Å*†	2·42$_3$ Å†	O$_A$–OH	2·73$_1$	2·79$_2$
Al–O$_A$	1·94$_4$	1·92$_1$	O$_A$–O$_B$	2·90$_9$	2·81$_2$	O$_B$–OH	2·80$_7$	2·80$_2$
Al–O$_B$	1·93$_2$	1·90$_1$	O$_A$–O$_B$	2·92$_6$	2·95$_2$	O$_B$–OH	3·04$_6$	2·87$_2$
Al–O$_B$	2·04$_8$	1·93$_1$	O$_A$–O$_B$	2·84$_1$	2·77$_2$	O$_B$–OH	2·68$_4$	2·78$_2$
Al–OH	1·93$_9$	1·94$_1$	O$_A$–OH	2·73$_4$	2·81$_2$	OH–OH	2·51$_1$*†	2·39$_2$*†
Al–OH	1·93$_0$	1·92$_1$	O$_A$–OH	2·88$_1$	2·88$_1$	O$_B$–O$_B$	2·76$_8$*†	2·43$_2$*†

Interlayer cation

	R	BT		R	BT	R		BT
K–O$_C$	2·79$_9$ Å	2·82$_1$ Å	K–O$_E$	2·86$_2$	2·85$_1$	K–O$_D$	3·51$_1$	3·53$_2$
K–O$_D$	2·77$_5$	2·88$_2$	K–O$_C$	3·35$_7$	3·33$_1$	K–O$_E$	3·30$_3$	3·29$_1$

Bond angles

	R	BT	Δ		R	BT	Δ
O$_C$–SiAl$_1$–O$_D$	110° 24′	110° 28′	45′	O$_D$–SiAl$_2$–O$_E$	107° 49′	109° 17′	43′
O$_C$–SiAl$_1$–O$_E$	108° 15′	106° 59′	44′	O$_B$–SiAl$_2$–O$_C$	114° 35′	110° 58′	46′
O$_D$–SiAl$_1$–O$_E$	111° 52′	107° 22′	46′	O$_B$–SiAl$_2$–O$_D$	109° 8′	113° 14′	42′
O$_A$–SiAl$_1$–O$_C$	106° 16′	109° 19′	44′	O$_B$–SiAl$_2$–O$_E$	109° 32′	109° 20′	43′
O$_A$–SiAl$_1$–O$_D$	115° 33′	111° 34′	46′	SiAl$_1$–O$_C$–SiAl$_2$	129° 22′	128° 9′	49′
O$_A$–SiAl$_1$–O$_E$	107° 22′	111° 1′	44′	SiAl$_1$–O$_D$–SiAl$_2$	135° 24′	139° 53′	58′
O$_C$–SiAl$_2$–O$_D$	107° 14′	106° 47′	47′	SiAl$_1$–O$_E$–SiAl$_2$	128° 42′	127° 4′	51′
O$_C$–SiAl$_2$–O$_E$	107° 3′	107° 4′	44′				

* Apical oxygens.

† These oxygen–oxygen distances correspond to shared edges of neighbouring octahedra.

factor for the $SiAl_1$ tetrahedron and the $SiAl_1$–O and O–O distances are larger than those of the second tetrahedron although the differences are not sufficiently large to indicate ordering of tetrahedral aluminium (Smith and Bailey, 1963). The basal oxygen triads are rotated 13°.

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Two staining tests for brucite in marble

IN a white or light grey marble brucite can be distinguished from the matrix minerals by staining. The traditional test for brucite in a marble is the Lemburg test, which involves dehydrating the brucite by heating the rock sample at 400° C for 30 minutes then immersing the sample in a solution of silver nitrate. The brucite is thus stained brown. Any method that involves heating is impracticable in the field or on mounted thin sections.

Two methods that can be used without heating have been developed; they use readily prepared reagents and are very easy to perform.

Alkali-hypoiodite test. Brucite is stained brown by adsorbed iodine in this technique; calcite and dolomite remain unstained. This brown is virtually permanent, fading only after a period of days. The reagents are:

Solution A. N potassium hydroxide, i.e. 56 g KOH per litre of distilled water.

Solution B. N iodine in 20 % potassium iodide solution, i.e. 127 g I_2 per litre of 200 g KI/litre aqueous solution.