Crystal structures and compressibilities of synthetic $2M_1$ and $3T$ phengite micas

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Abstract: The crystal structures of co-existing monoclinic $2M_1$ and trigonal $3T$ polytypes of phengitic micas synthesized at 11 GPa and 900 °C have been refined at ambient conditions. The compositions of both crystals are approximately $K(Al_{1.21}Mg_{0.75}Fe_{0.04})(Al_{0.19}Si_{3.81})O_{22}(OH_{1.2}F_{0.8})$. The unit cell parameters for the $2M_1$ sample are $a = 5.2046(8)$ Å; $b = 9.0368(16)$ Å; $c = 19.886(4)$ Å, $\beta = 95.615(14)^\circ$; vol. = $930.8(2)$ Å³; and for the $3T$: $a = 5.2110(4)$ Å; $c = 29.689(5)$ Å; vol. = $698.08(13)$ Å³. The molar volumes of the two polytypes are identical within error (approximately one part in 4000). The structures show closely similar distortions consistent with the nearly pure silicate tetrahedral layer. The tetrahedral rotation angles, α , are both about 2.4° and thus the smallest yet reported for dioctahedral micas. There is no indication of tetrahedral ordering of Al and Si. The $3T$ polytype contains two distinct octahedral sites that appear to be distinctly different in size indicating possible ordering of Mg and Al. The unit cell parameters of the $2M_1$ sample have been measured at several pressures up to 7.5 GPa and those of the $3T$ sample to 4.0 GPa. Fitting compression data to a third-order Birch-Murnaghan equation of state gives a K_0 of 57 ± 3 GPa with K' of 9.2 ± 1.7 for the $2M_1$ and a K_0 of 62 ± 2 GPa with a fixed K' of 9 for the $3T$. These are statistically identical and represent the largest bulk modulus yet measured for any mica. As with other micas, compressional anisotropy is large with compression normal to the layers being about seven times that within the layers.

Keywords: mica, phengite, crystal structure, compression

Introduction

Relatively few hydrous silicate phases are stable in the pressure range of 8 to 12 GPa. Chlorite and phlogopite are believed to be abundant in hydrated oceanic crust and are stable to about 4 GPa and 8 GPa respectively (Kawamoto *et al.*, 1996). Phengite mica, however, may be the dominant host mineral phase for both K and water (as hydroxyl) in subducted crustal rocks at depths of 110 to 300 km (Schmidt, 1996). It occurs in rocks of a wide range of bulk compositions from mid-ocean ridge basalts (MORB) to pelitic sedimentary compositions. The breakdown of phengitic mica may control release of water and K-rich fluids on subduction, particularly at depths of 100 to 330 km.

Bailey (1984) gives the ideal formula of phengite as $KAl_{1.5}(Mg,Fe)_{0.5}Si_{3.5}Al_{0.5}O_{10}(OH)_2$. Phengitic substitution of Si for Al in the tetrahedral sheet enhances the pressure stability range of these micas, so that samples synthesized at the highest pressures have nearly pure Si in the tetrahedral layer (Domanik & Holloway, 1996). Swope *et al.* (1997) determined the polytype and unit cell parameters of seven crystals of phengite synthesized at 900 °C and 11 GPa by Domanik & Holloway (1996). They observed approximately equal proportions of $2M_1$, $3T$, and mixed polytypes in the same charge. The samples have a chemical formula of $K(Al_{1.21}Mg_{0.75}Fe_{0.04})(Al_{0.19}Si_{3.81})O_{10}((OH)_{1.2}F_{0.8})$, so there is ~5 % Al occupancy of the tetrahedral layer. This is the most silica-rich phengite composition yet reported and is consistent with the extreme pressure conditions of the synthesis.

Refinement of these structures at ambient conditions may add to our knowledge of phengite crystal chemistry. Bailey (1984) noted that tetrahedral rotation angles (α) for dioctahedral micas range from about 6 to 19°. The crystal structures of two natural 3T phengitic muscovites have recently been reported by Amisano-Canesi *et al.* (1994). One of these samples (from Dora Maira) contains 11 % Al on the T sites and shows a tetrahedral rotation angle of 5.4°. The synthetic samples extend the range of studied compositions to the more silica rich end-member and may clarify details of structure variation with composition.

It has been suggested that the 3T polytype is more common in Mg-rich phengites, whereas the 2M₁ may be more common in Mg-poor compositions (Stöckert, 1985). Sassi *et al.* (1994) suggested that pressure may favor the 3T over the 2M₁ polytype, because the 3T polytype has two distinct octahedral M sites which would allow for ordering of Al and Mg. Indeed, Pavese *et al.* (1997) report significant ordering of Mg into the M3 octahedral site. If cation ordering significantly reduces molar volume, pressure could well stabilize the ordered polytype. If so, structure determination of co-existing high silica phengite polytypes may help to elucidate how polytype differences may affect stability with pressure and or accommodation of divalent cations. Further, measurement of relative compression of the two polytypes as a function of pressure may allow us to estimate the pressure effect on relative stabilities of the two polytypes.

Pavese *et al.* (1997 and 1999a) report the results of crystal structure refinements of 2M₁ and 3T phengites from neutron data at elevated temperatures to 600 °C. They report volume thermal expansion coefficients of $34.3 \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ for 2M₁ and $33.1 \pm 0.5 \times 10^{-6} \text{ K}^{-1}$ for 3T. Curiously, they report rather different anisotropies of expansion between the two polytypes with expansion normal to the layer being about four times that within the layers for 3T and about twice that within the layers for 2M₁.

All micas exhibit a strong anisotropy of compression with the direction normal to the sheets being approximately five times more compressible than those within the sheets. Phlogopite has an approximate bulk modulus of 58 GPa (Hazen & Finger, 1978). Muscovites have been studied at elevated pressures (Catti *et al.*, 1994; Comodi & Zanazzi, 1995). Isothermal bulk moduli range from 49 to about 54 GPa based on an assumed K' ($\partial K/\partial P$ isothermal) of 4.0. Vaughan & Guggenheim (1986) measured a full set of elastic constants for a 2M₁ muscovite by Brillouin scattering methods. Pavese *et al.* (1999b) report the pressure-temperature-volume equation of state of phengite-3T from Dora Maira based on synchrotron X-ray powder diffraction. They report a K_0 of 55.8 GPa with a refined K' of 8.9 at 300 K, whereas earlier studies assumed a K' of 4.0. They report only the unit cell volumes rather than the actual unit cell parameters derived from their study so the degree of anisotropy of compression is not available.

We anticipate that the bulk moduli of the phengites synthesized at pressures in excess of 10 GPa may be somewhat higher than micas previously studied mainly because these samples have higher tetrahedral silica contents (3.81 Si per formula unit). Phengites are possible significant phases in subduction zones and are likely to show extreme elastic anisotropy. Further, alignment of micas in shear stresses is a common feature of metamorphic rocks that could give rise to zones of significant seismic anisotropy. We have therefore investigated the compression of the two polytypes in order to clarify the relative effects of pressure on the structures.

Experimental

Synthesis

The synthesis experiments are described in detail by Domanik & Holloway (1996). Briefly, the samples were grown in a Walker-type multi-anvil press at Arizona State University. Run conditions were 900 °C and 11 GPa, and run duration was 50.8 hours. Run products consisted of phengite, garnet, topaz, kyanite, stishovite, and jadeite. Chemical analysis was performed by electron microprobe and gives the formula of $\text{K}_{0.75}(\text{Al}_{1.21}\text{Mg}_{0.75}\text{Fe}_{0.04})(\text{Al}_{0.19}\text{Si}_{3.81})\text{O}_{10}((\text{OH})_{1.2}\text{F}_{0.8})$. Domanik & Holloway (1996) state that the probe analysis methods caused electron beam damage of the phengite that resulted in a systematic underestimation of the K contents.

In total, seven euhedral crystals were separated from the experimental capsule and mounted for X-ray examination. Crystals were first examined by X-ray precession photography for crystal quality, diffuse scattering, and polytype identification. Crystals of both 2M₁ and 3T, as well as composites of both polytypes were observed. Of seven crystals, two crystals were 2M₁ and two were 3T, and three were composites of both polytypes. Although the reflections were relatively broad, especially for the 3T sample, the crystals were relatively free of streaking parallel to c^* .

The best crystal of each polytype was mounted on a Siemens P4 automated X-ray diffractometer on a rotating-anode generator operated at 50 kV and 250 mA. The 2M₁ crystal was a nearly euhedral hexagonal plate approximately 180 μm in diameter and 140 μm thick. The 3T crystal was similar in shape and approximately 130 μm in diameter and 80 μm thick. Unit cell parameters were refined from centering parameters of 20 reflections between 2θ values of 10 and 25°. Before and after each cell refinement, the effective wavelength of the $\alpha_1 - \alpha_2$ mix from our horizontal graphite monochromator was determined from the unit cell refinement of a standard ruby sphere. Unit cell parameters determined by this method are generally reproducible to within one standard deviation. Unit cell parameters are reported in Table 1. The experimentally determined molar volumes of the two structures are identical within error.

Table 1. Unit cell parameters and X-ray intensity data parameters for $2M_1$ and $3T$ phengites.

Parameter	Phengite- $2M_1$	Phengite- $3T$
Space Group	$C2/c$	$P3_112$
$a(\text{\AA})$	5.2046(8)	5.2110(4)
$b(\text{\AA})$	9.0368(16)	
$c(\text{\AA})$	19.886(4)	29.689(5)
$\beta(^{\circ})$		95.615(14)
Vol($\text{\AA}^3$)	930.8(2)	698.1(1)
Z	8	6
Molar Volume (cm^3)	70.06(2)	70.06(1)
Scan Type	ω	ω
Scan Width ($^{\circ}\omega$)	4	6
2θ Range	2 – 60	2 – 60
$\langle I/\sigma \rangle$	21.3	15.1
Generator (kV/mA)	50/250	50/250
No. Reflections	3069	3246
No. unique	1371	1370
No. observed ($>2\sigma$)	1246	1230
R_{merg}	0.013	0.009
R	0.075	0.098
R_w	0.056	0.076

$$R_{\text{merg}} = \frac{\sum |F_{\text{hkl}}^2 - \bar{F}_{\text{hkl}}^2|}{\sum F_{\text{hkl}}^2} \quad R = \frac{\sum |F_{\text{obs}} - F_{\text{calc}}|}{\sum F_{\text{obs}}}$$

$$R_w = \frac{\sum w |F_{\text{obs}} - F_{\text{calc}}|}{\sum w F_{\text{obs}}}, \text{ where } w = \frac{1}{\sigma^2(F) + (0.00003)F^2}$$

Data collection

X-ray intensity data were measured for each crystal using variable speed omega scans. Scan speeds ranged from 4 to 20° ω per minute. A similar methodology was used for each crystal except that a slightly larger scan angle was used for the $3T$ sample as was necessitated by the broader diffraction peaks. Data collection parameters are outlined in Table 1. *Structure refinement*

All absorption correction and structure refinements were carried out using the program SHELXTL (Sheldrick, 1990). Scattering factors for neutral atoms were used throughout. The data were corrected for absorption using an analytical absorption correction based on the measured shape of the crystals. After correction for absorption, equivalent reflections were averaged with an R of merging

of 0.013 for $2M_1$ and 0.008 for the $3T$ sample. The initial atom positions were those from Comodi & Zanazzi (1995) for $2M_1$ and Amisano-Canesi *et al.* (1994) for $3T$. The $2M_1$ structure was refined in space group $C2/c$ and the $3T$ in $P3_112$. Refining on F, the final weighted R -value for observed ($>2\sigma$) reflections for the $2M_1$ structure was 0.056 and for the $3T$ structure, 0.076 (Table 1). Final atom position and atomic displacement parameters are reported in Tables 2 and 3. Nearest neighbor distances and coordination distortion parameters are reported in Table 4. Mica structure distortion parameters are reported in Table 5.

Table 5. Structural parameters for $2M_1$ and $3T$ phengites.

Parameter	Phengite- $2M_1$	Phengite- $3T$
$\alpha_{\text{tet}}(^{\circ})$	2.4(1)	2.5(5)
$\tau_{T1}(^{\circ})$	112.6(2)	112.8(5)
$\tau_{T2}(^{\circ})$	112.6(2)	112.8(5)
$\psi_{\text{oct}}(^{\circ})$	56.80	56.63
		56.96
Δz ($\text{\AA}$)	0.131(4)	0.11(1)
D.I. (K) ($\text{\AA}$)	0.017(7)	0.009(8)
Thickness		
Tetrahedral($\text{\AA}$)	2.219(4)	2.231(10)
Octahedral($\text{\AA}$)	2.164(4)	2.159(10)
Interlayer($\text{\AA}$)	3.288(4)	3.276(10)

High pressure X-ray diffraction

The crystals were loaded separately in a diamond anvil cell along with a crystal of quartz to act as an internal pressure standard. The diffractometer was a Huber four-circle goniometer at Bayerisches Geoinstitut as described by Angel *et al.* (1997). The diamond anvil cell (DAC) was that described by Allan *et al.* (1996) fitted with diamonds having 0.8 mm culet faces. The pressure medium was 4:1 ethanol-methanol mixture that should remain hydrostatic to pressures in excess of 10 GPa (Piermarini *et al.*, 1975). The gasket material was stainless steel. The unit cell of the $3T$ crystal was measured in the cell at one atmosphere and at seven pressures up to 4.0 GPa. Pressure was initially estimated using the ruby fluorescence method (Piermarini *et al.*, 1975). Pressures were determined by measuring the unit-cell parameters of a standard quartz crystal at each pressure and using the equation of state determined by Angel *et al.* (1997). Using the quartz-standard method, we are able to measure the pressure with an experimental uncertainty of approximately 5 MPa over the pressure range studied. to the volumetric data.

After measurement at 4.0 GPa, the 3T crystal was lost during a re-load of the cell. The original $2M_1$ crystal was too thick for measurement in the diamond anvil cell and so was cleaved to reduce the thickness. The cleaved crystal was approximately 80 μm thick. The unit cell parameters of the $2M_1$ crystal were measured at one atmosphere in the cell and at 8 pressures up to 7.4 GPa. The data for both crystals are presented in Table 6 along with the quartz unit cell volume determined at each pressure.

Fig. 1. Relative axial and volumetric compressions (X/X_0) of phengite- $2M_1$ to 7.4 GPa. The curve is the fit equation of state to the volumetric data.

The volume-pressure data were fit to a third-order Birch-Murnaghan equation of state (Birch, 1978). Due to the broad diffraction maxima of both phengite crystals their cell parameters were not determined as precisely as those of the quartz standard crystal. Nevertheless, the data are adequate to define an isothermal bulk modulus (K_0) and to estimate K' for a 3rd-order Birch-Murnaghan EoS (Birch, 1978). Assuming an initial K' of 4.0, we obtained an isothermal bulk modulus of 70 ± 8 GPa for the $2M_1$ crystal and 72 ± 2 GPa for the 3T crystal. However, the volume compression data for both crystals indicated considerably more curvature than would be consistent with the estimated K' of 4. Refining K' for the current crystals gave values larger than 4 in both cases and considerably better fits to the data. For the $2M_1$ we obtain values of 57 ± 3 GPa and 9.2 ± 1.7 for K_0 and K' respectively. For the 3T crystal we obtain values of K_0 of 63 ± 11 GPa and K' of 9 ± 7 . Pavese *et al.* (1999b) determined a K' for phengite 3T of 8.9 ± 4 . Given the higher precision of the K' estimate for the $2M_1$ sample and the good agreement with the study by Pavese (1999b), we re-fit the

equation of state for the 3T sample with an assumed (fixed) K' of 9 and obtained a K_0 of 62 ± 2 GPa. The relative compression data are plotted in Figs. 1 and 2.

Fig. 2. Relative axial and volumetric compressions (X/X_0) of phengite-3T to 4.0 GPa. The curve is the fit equation of state

Discussion

Composition

Domanik & Holloway (1996) report a microprobe chemical analysis that indicates a significant K deficiency of 0.75 K per formula unit, but state that K loss due to beam damage may have been as much as 20 % of the amount present. We refined the K occupancy of both structures and observed 96.3 ± 0.7 % occupancy in the $2M_1$ and 100.1 ± 1.5 % in the 3T sample. We therefore conclude that there is little if any K deficiency in either sample. This is also consistent with the rest of the chemical analysis, which requires no significant K deficiency for charge balance.

Tetrahedral sites

The $2M_1$ structure contains two distinct T-sites that alternate around the six-membered rings of the tetrahedral sheets. The mean T-O distances and volumes of the two sites are consistent with 5 % Al and 95 % Si occupancy. They are statistically identical in size and volume indicating little if any ordering of Si and Al in the tetrahedral sheet. There are also two distinct T sites in the 3T structure. Similarly, we see no significant difference in mean T – O distance or volume of the two sites. They are also nearly identical to those of the $2M_1$ structure. These results are consistent with those of Flux *et al.* (1984) who conclude that there is no evidence for pressure-induced ordering of Al and Si in synthetic muscovite and paragonite up to 1.8 GPa.

The rotation angles, α , (Table 5) of the tetrahedral sheet in the two structures is a measure of the distortion of the sheet relative to an undistorted hexagonal ring, and is known to decrease systematically with the Al content of the tetrahedra. The values observed for these two structures, $2.4 \pm 0.1^\circ$ and $2.5 \pm 0.5^\circ$ are statistically identical and the lowest yet reported for a dioctahedral mica, consistent with the low Al contents of the T sites. However, in contrast to the overall regular geometry of the tetrahedral sheets, the tetrahedra themselves are quite distorted.

The angle, τ , of the tetrahedra (Table 5) is an average of the central bond angles involving the apical oxygen and is a measure of the distortion of the tetrahedra by elongation or compression normal to the sheet. Angles greater than the ideal tetrahedral angle of 109° indicate elongation, and angles less than 109° indicate compression. All tetrahedra are similar, and τ values are among the largest reported for any mica. This is consistent with the relatively large tetrahedral layer thickness ($2.231 \text{ \AA}$) despite low Al occupancies, shown by both structures.

Octahedral sites

Perhaps the greatest difference between the $2M_1$ and $3T$ structures is that the $2M_1$ structure contains a single M site, whereas the $3T$ structure contains two distinct M sites. The $3T$ octahedral layer is heterogeneous in that each layer contains both types of octahedra. The mean M-O distance in the $2M_1$ octahedral site is consistent with the occupancy inferred from the analysis of 38 % Mg, 2 % Fe and 60 % Al. In the $3T$ structure, the two sites are different in mean M-O distance with M3 being larger by about $0.017 \text{ \AA}$ (2σ). It is thus possible that a small amount of ordering of Mg and Al is occurring in this structure, with Mg showing a slight preference for M3. Refinement of occupancies using scattering factors for neutral Al only gave values $98 \pm 2 \%$ for M2 and $100 \pm 2 \%$ for M3. These values are not significantly different but do not rule out significant ordering of Al and Mg on these sites.

K-sites

The interlayer sites show an unusually high degree of regularity consistent with the small rotation angle of the tetrahedral layer. Thus there is relatively little difference between the inner and outer K-O bonds (Table 4). This allows the K cations to intrude further into the tetrahedral layers and gives a relatively short interlayer spacing (Table 5) of $3.288 \text{ \AA}$ for a pure K mica which are typically greater than $3.35 \text{ \AA}$ (Bailey, 1984).

Compression

First, although the errors in cell volume determinations are large relative to other common silicates, the volume compressions of both crystals are statistically identical within 2σ which is consistent with their identical molar volumes at room pressure. Further, the axial compressions in the plane of the layers and normal to the layers are identical. Thus the molar volume and volumetric and axial

compressions of the two polytypes are identical within error. This is consistent with there being no significant pressure effect on polytype distribution in rocks, contrary to the suggestion of Sassi *et al.* (1994) that pressure may favor the $3T$ polytype.

Comodi & Zanazzi (1995) measured compression and refined atom position coordinates for $2M_1$ polytypes of Na- and K-rich muscovites to about 3.5 GPa. Hazen & Finger (1978) measured compression of chlorite and phlogopite- $1M$ to 4.0 and 4.7 GPa respectively. These studies of micas have indicated total volume compressions of 7 to 8 percent at 4 GPa, whereas the current data indicate approximately 4.8 % volume compression at this pressure. So these synthetic phengites are both significantly less compressible than previously studied layer-silicates. The lower compressibility of the phengites is consistent with the already short interlayer spacing of these samples. Looking at the axial compressions, we observe that the current samples are less compressible in both the a and c directions. Similar to previous studies, compression is highly anisotropic with compression normal to the layers is four to five times greater than compression in the plane of the layers.

Previous single crystal studies of mica compression have gone to a maximum pressure of 4.7 GPa and assumed a K' value of 4. Pavese *et al.* (1999b) used synchrotron X-ray powder diffraction to measure the unit cell volume of a natural $3T$ phengite and functions of both temperature and pressure to approximately 1000 K and 5 GPa. Their sample contained about 10 percent Al in the tetrahedral sites, or about twice the amount in the current samples. They used several models to fit equation of state parameters to their volume data and report a K_0 of $55.8 \pm 5.7 \text{ GPa}$ with a refined K' of 8.9 ± 4.0 at 300 K.

Indeed, the data presented here for the $3T$ crystal give a refined K' value that is not statistically different from this value. However, compression measurements to higher pressure for the $2M_1$ crystal allow us to refine $\partial K/\partial P$ (K'). Fitting the current data to a third order Birch-Murnaghan equation of state gives an apparent value of 9.2 ± 1.7 for K' of the $2M_1$ crystal. The derived value of K' thus differs significantly from 4. Using these higher K' values we obtain a value for K_{0T} that is similar to, but slightly higher than, values obtained for $3T$ phengite by Pavese *et al.* (1999b) and significantly higher than values obtained for other micas. The values of K' are also in good agreement with those obtained by Pavese *et al.* (1999b) and are probably characteristic of the mica structure. In examining the compression curves (Fig. 1 and 2) it appears that most of the curvature is due to non-linear compression normal to the layers.

In summary, we have refined the crystal structures and measured the compressibilities of $2M_1$ and $3T$ polytypes of phengite synthesized at 900°C and 11 GPa with less than 5 % tetrahedral Al. At ambient conditions, the molar volumes are identical within error and the structures show similar polyhedral volumes, rotation angles and distortions. The

very low rotation angles of the tetrahedral sheet indicate that this composition may be at or near the compositional limit of silica substitution in the tetrahedral layer. Compression of the 3T sample has been measured to 4.0 GPa and the 2M₁ to 7.5 GPa. The samples show statistically identical volume compressions and are significantly less compressible than previously studied micas. Further, there appears to be an equal distribution of the two polytypes in the synthesis run at 11 GPa. This indicates that there is likely to be little if any pressure effect on polytype distribution. Compression is highly anisotropic with compression normal to the layer being about 4.5 times greater than compression in the plane. Phengite micas are believed to be significant host phases for K and H in subducting hydrated oceanic basalt. Because micas are known to show near perfect alignment under shear deformation, their elastic anisotropy could give rise to significant seismic velocity anisotropy in the crustal portion of the slab if mica were present even in minor amounts.

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Table 4. Cation site parameters in phengite-2*M*₁ and 3*T*.

Site	Phengite-2 <i>M</i> ₁ Parameter	Value	Phengite-3 <i>T</i> Parameter	Value
K	K – O1(2x)	3.100(4) Å	K – O3(2x)	2.998(8) Å
	K – O1(2x)	3.015(4) Å	K – O3(2x)	3.115(8) Å
	K – O2(2x)	3.203(4) Å	K – O4(2x)	3.177(8) Å
	K – O2(2x)	3.055(3) Å	K – O4(2x)	3.052(8) Å
	K – O3(2x)	3.100(3) Å	K – O5(2x)	3.107(8) Å
	K – O3(2x)	3.018(3) Å	K – O5(2x)	3.021(8) Å
Mean K-O	3.082 Å		Mean K – O	3.078 Å
Volume	59.95 Å ³		Volume (Å ³)	59.59 Å ³
M2	M2 – O4	1.968(3) Å	M2 – O1(2x)	1.942(9) Å
	M2 – O4	2.001(3) Å	M2 – O2(2x)	1.985(9) Å
	M2 – O5	2.003(3) Å	M2 – O6(2x)	1.963(9) Å
	M2 – O5	1.973(3) Å		
	M2 – O6	1.951(3) Å	Mean M2- O	1.963 Å
	M2 – O6	1.960(3) Å	Volume	9.955 Å ³
			A.V.	33.63
	Mean M2- O	1.976 Å	Q.E.	1.0093
	Volume	10.15 Å		
	A.V.	32.45	M3 – O1 (2x)	2.000(7) Å
	Q.E.	1.0090	M3 – O2 (2x)	2.009(9) Å
			M3 – O6 (2x)	1.931(10) Å
T1				
	T1 – O1	1.636(3) Å	T1 – O1	1.619(9) Å
	T1 – O2	1.629(4) Å	T1 – O3	1.610(9) Å
	T1 – O3	1.639(4) Å	T1 – O4	1.624(9) Å
	T1 – O4	1.594(3) Å	T1 – O5	1.625(9) Å
	Mean T1- O	1.625 Å	Mean T1- O	1.620 Å
	Volume	2.190 Å ³	Volume	2.167 Å ³
	A.V.	13.60	A.V.	16.90
T2				
	T2 – O1	1.634(3) Å	T2 – O2	1.587(10) Å
	T2 – O2	1.626(3) Å	T2 – O3	1.658(9) Å
	T2 – O3	1.632(3) Å	T2 – O4	1.656(10) Å
	T2 – O5	1.600(3) Å	T2 – O5	1.633(9) Å
	Mean T2- O	1.623 Å	Mean T2- O	1.623 Å
	Volume	2.184 Å ³	Volume	2.227 Å ³
	A.V.	13.61	A.V.	16.56
	Q.E.	1.0030	Q.E.	1.0037

* Angle variance (A.V.) and quadratic elongation (Q.E.) as defined by Robinson *et al.* (1971).

Table 6. Unit cell compression data for $2M_1$ and $3T$ phengites.

Pressure (GPa) ($\text{\AA}^3$)	Qtz. Vol. ($\text{\AA}$)	a ($\text{\AA}$)	b ($\text{\AA}$)	c ($^\circ$)	β ($\text{\AA}^3$)	Vol
Phengite $2M_1$						
0.0001*113.009(7)**	5.212(6)	9.056(6)	19.981(14)	95.54(16)	938.7(14)	
0.987(4)110.224(12)	5.204(7)	9.024(6)	19.731(15)	95.7(2)	922.0(16)	
2.024(3)107.584(7)	5.201(11)	8.996(9)	19.560(27)	95.8(5)	910.5(25)	
3.020(3)105.563(6)	5.180(10)	8.978(11)	19.407(25)	95.5(4)	898.4(25)	
4.041(3)103.818(5)	5.167(8)	8.953(9)	19.298(24)	95.6(4)	888.5(20)	
5.164(3)101.975(5)	5.163(16)	8.941(19)	19.147(44)	95.5(6)	879.8(40)	
6.096(5)100.589(7)	5.158(14)	8.929(13)	18.994(33)	95.4(5)	870.9(32)	
6.826(4)99.580(5)	5.149(9)	8.908(9)	18.923(23)	95.5(4)	864.0(20)	
7.443(5)98.773(6)	5.138(11)	8.888(11)	18.862(26)	95.4(4)	857.5(25)	
Phengite $3T$						
0.0001*112.993(12)	5.218(6)		29.696(26)		700.2(17)	
0.694(8)110.995(16)	5.216(4)		29.482(32)		694.6(18)	
0.823(2)110.652(5)	5.212(4)		29.393(17)		691.5(13)	
1.260(10)109.54(2)	5.208(3)		29.324(18)		688.8(13)	
1.813(5)108.231(11)	5.203(3)		29.150(17)		683.4(10)	
2.157(6)107.466(6)	5.201(4)		29.054(17)		680.6(12)	
3.152(4)105.438(8)	5.188(4)		28.803(14)		671.4(10)	
3.970(5)103.941(9)	5.182(4)		28.697(15)		667.4(10)	

* Measured in cell, no fluid

** Number in parentheses are one standard deviation (1σ) in last significant digit given.