

Crystal-structure refinement of a rubidian cesian phlogopite

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ABSTRACT

The crystal structure of a rubidian cesian phlogopite-1M from pegmatite exocontacts at Red Cross Lake, Manitoba, monoclinic, $a = 5.343(1)$, $b = 9.247(2)$, $c = 10.397(3)$ Å, $\beta = 100.04(2)^\circ$, $V = 505.8(2)$ Å³, has been refined to an R index of 4.5% based on 519 observed reflections measured with graphite-monochromated MoK α X-radiation on an automated four-circle diffractometer. The crystal used in the collection of the X-ray intensity data was also analyzed by electron microprobe, giving the unit formula $(K_{0.46}Rb_{0.28}Cs_{0.23})(Mg_{1.20}Fe_{1.00}Al_{0.38}Li_{0.34}Mn_{0.04}Ti_{0.04})(Si_{2.91}Al_{1.09})O_{10}[(OH)_{1.55}F_{0.45}]$. The interlayer site, X, contains large amounts of Rb and Cs, and cell dimensions and the $\langle X-O \rangle$ distance are in accord with data from synthetic Rb and Cs phlogopites and plutonic phlogopites. The interlayer coordination is much more regular in rubidian cesian phlogopite than in other trioctahedral micas, defining a trend of increasing regularity with increasing interlayer-cation size.

INTRODUCTION

Ferromagnesian micas are a common product of contact reactions between granitic pegmatites and basic wallrocks such as amphibolites, metabasalts, dolomitic marbles or serpentinites (Černý and Burt 1984). Ever since the classic description by Hess and Stevens (1937), these micas are known to carry substantial Li, Rb, and Cs, particularly when associated with highly fractionated pegmatites (e.g., Morgan and London 1987). Ginsburg et al. (1972), Zagorskiy et al. (1974), and Černý et al. (1981) reported chemical-composition data for exomorphic micas from several localities, but the crystal chemistry and crystal structures of these micas have never been investigated. Here we present a crystal-structure refinement of a rubidian cesian phlogopite from the swarm of lepidolite-subtype pegmatites at Red Cross Lake, Manitoba.

This paper is dedicated to Gene Foord, a colleague for many years, and an inspiration to all involved in working on any aspect of pegmatities and pegmatite minerals.

EXPERIMENTAL METHODS

Sample provenance

The Archean pollucite-bearing pegmatites at Red Cross Lake, in the Sachigo Subprovince of the Superior Province of the Canadian Shield, are the most fractionated granitic pegmatites known to date (Černý et al. 1994). Internal and exocontact micas of these pegmatites are strongly zoned and show extreme enrichment in rare alkalis in their outer zones. The ferromagnesian micas from contacts with meta-andesitic to metabasaltic country rocks show K/Rb and K/Cs (wt%) ratios as low as 0.18 and 0.06, respectively. However, the outer zones are invariably thin (Fig. 1) and difficult to physically extract for single-crystal investigation. Nevertheless, a thin

plate of a strongly Rb- and Cs-enriched phlogopite was successfully extracted for X-ray diffraction studies.

Single-crystal diffraction and structure refinement

A thin plate was mounted on a Nicolet P4 automated four-circle diffractometer; the crystal appeared slightly curved, as are all crystals from this particular locality, but gave good-quality diffractions. Cell dimensions (Table 1) were refined from the setting angles of 25 automatically aligned intense reflections, and conform to those of the 1M polytype of phlogopite. Intensity data were collected from $2\theta = 3\text{--}60^\circ$ according to the procedure of Hawthorne and Groat (1985). Absorption corrections were done, modeling the crystal shape as a polyhedron. Intensities were corrected for background, absorption, Lorentz and polarization effects, and reduced to structure factors. Details concerning these procedures are given in Table 1.

The crystal structure was refined using the SHELXTL-PC system of programs; R indices are of the form given in Table 1. The structure was refined in the space group $C2/m$ (1M polytype) using the structural parameters of Rayner (1974) as the starting model. The H position was taken from a difference-Fourier map calculated after some cycles of refinement, and the H position was refined subject to the soft constraint that $O4-H \sim 0.97$ Å. Full-matrix least-squares refinement of positional and anisotropic-displacement parameters and site-scattering factors converged to an R index of 4.5%. Atomic positions and displacement factors are listed in Table 2, interatomic distances are shown in Table 3, and observed and calculated structure factors are listed in Table 4¹.

¹For a copy of Table 4, document item AM-99-009, contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. Deposit items may also be available on the *American Mineralogist* web site at <http://www.minsocam.org>.

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TABLE 1. Single-crystal X-ray diffraction data-collection and refinement information for rubidian cesian phlogopite

| | | | |
|---------------------|-----------|---------------------------------|---------------------|
| a (Å) | 5.343(1) | Crystal size (mm ³) | 0.19 × 0.14 × 0.005 |
| b (Å) | 9.247(2) | Radiation/Mono. | MoK /Gr |
| c (Å) | 10.397(3) | Total no. F | 782 |
| b (°) | 100.04(2) | No. of F obs | 519 |
| V (Å ³) | 505.8(2) | R(azimuthal)% | 2.0 |
| | | R(observed)% | 4.5 |
| Space group | C2/m | wR(observed)% | 4.5 |
| Z | 4 | R(all data)% | 6.6 |

Notes: $R = (|F_o| - |F_c|) / |F_o|$; $wR = [\sum (w|F_o| - |F_c|)^2 / \sum w^2]^{1/2}$; $w = 1$.

Electron-microprobe analysis

Electron-microprobe analysis was done using a CAMECA SX-50 operating in wavelength-dispersion mode, with a beam diameter of 5 μm and an accelerating potential of 15 kV. A sample current of 20 nA measured on a Faraday cup and a counting time of 20 s were used for K, Cs, Rb, Mg, Mn, Al, Fe, Si, and F. The following standards were used for analysis: albite (NaKa), fayalite (FeK), diopside (CaK, SiK), kyanite (AlK), spessartine (MnK), orthoclase (KK), zinnwaldite (FK), olivine (MgK), titanite (TiK), pollucite (CsL), and Rb leucite (RbL); Na, Ca, Zn, Sc, P, Ba, Sr, and Cl were not detected. The data were reduced using the PAP routine of Pouchou and Pichoir (1985). The mean composition is given in Table 5. The value for Li was calculated using the expression $\text{Li} = 7\text{-Si-Al-Fe-Mn-Mg-Ti}$; the value for H₂O was calculated assuming $\text{OH} + \text{F} = 2$ apfu (atoms per formula unit). The total (Table 4) is somewhat low, due to slight sample charging that is difficult to avoid with small single crystals mounted in epoxy.

DISCUSSION

Chemical composition

If the unit formula is calculated from the chemical analysis of Table 5 on the basis of 12 anions pfu with $\text{OH} + \text{F} = 2$ apfu, there is significant vacancy content at the M site ($^{[6]}\text{Al} + \text{Mg} + \text{Fe} + \text{Mn} + \text{Ti} = 1.822$ apfu). This is the minimum value of such a "vacancy." As $\text{Fe}^{2+} \gg \text{Fe}^{3+}$ in most Fe-bearing trioctahedral micas, we assume $\text{Fe} = \text{Fe}^{2+}$ in the calculation of the unit-cell content, and any Fe^{3+} present will increase the apparent "vacancy" content. The intimate intergrowth of the rubidian cesian phlogopite with phlogopite (Fig. 1) precludes separation of the former to measure the $\text{Fe}^{3+}/\text{Fe}^{2+}$ ratio. Significant Li is expected at the M site in this mica due to its occurrence in a Li-rich environment. Hence the Li content was calculated from the relation $\text{Li}(\text{pfu}) = 7\text{-Si-Al-Mg-Fe-Mn-Ti}$, and the calculation of the unit formula was iterated to self-consistency. The resulting unit formula contains 0.343 Li apfu (equivalent to 1.02 wt% Li₂O in the chemical analysis). This amount of Li is in accord



FIGURE 1. Backscattered-electron image of subparallel platy crystals of phlogopite (dark grey) with outer zones enriched in Rb and Cs (white), in plagioclase and quartz (black). The scale bar is 50 μm long.

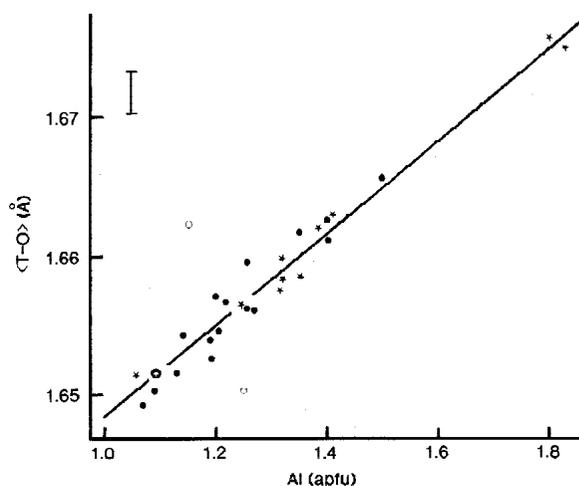


FIGURE 2. Variation in $\langle T\text{-O} \rangle$ distance as a function of $^{[4]}\text{Al}$ content in some recent refinements of mica crystals. Filled circles = biotite-1M and phlogopite-1M (data from Brigatti and Davoli 1990; Brigatti et al. 1991; Bigi and Brigatti 1994; Alietti et al. 1995). Stars = Ba-rich 1M trioctahedral micas (Brigatti and Poppi 1993). Two outliers are shown as unshaded circles. The starred circle = the rubidian cesian phlogopite of this study; solid line = a linear least-squares fit to the data, omitting the outliers and rubidian cesian phlogopite. The average standard deviation of the data points is shown by the () bar.

TABLE 2. Final atomic parameters for rubidian cesian phlogopite

| | x | y | z | U_{eq} | U_{11} | U_{22} | U_{33} | U_{23} | U_{13} | U_{12} |
|------|------------|-----------|-----------|----------|----------|----------|----------|----------|----------|----------|
| K,Cs | 0 | 1/2 | 0 | 248(6) | 185(8) | 268(9) | 297(10) | 0 | 56(7) | 0 |
| M1 | 0 | 0 | 1/2 | 92(9) | 21(13) | 74(14) | 192(17) | 0 | 49(11) | 0 |
| M2 | 0 | 0.3331(2) | 1/2 | 109(6) | 23(9) | 117(10) | 192(12) | 0 | 35(7) | 0 |
| T | 0.0785(3) | 0.1668(2) | 0.2328(2) | 112(5) | 37(7) | 112(8) | 188(9) | 3(7) | 23(6) | 1(6) |
| O1 | 0.0419(12) | 0 | 0.1765(7) | 220(20) | 204(32) | 185(34) | 267(35) | 0 | 31(27) | 0 |
| O2 | 0.3171(8) | 0.2411(6) | 0.1748(5) | 250(15) | 167(21) | 324(27) | 267(25) | 18(22) | 65(18) | -70(20) |
| O3 | 0.1335(8) | 0.1684(5) | 0.3939(4) | 183(12) | 137(19) | 192(21) | 219(22) | -8(20) | 25(16) | 5(18) |
| O4 | 0.1288(11) | 1/2 | 0.4009(7) | 207(19) | 79(27) | 308(37) | 225(33) | 0 | 7(25) | |
| H | 0.117(18) | 1/2 | 0.306(2) | 100(-) | - | - | - | - | - | - |

TABLE 3. Interatomic distances (Å) and angles (°) in rubidian cesian phlogopite

| | | | | |
|-----------|-------------|-----------|----------|----|
| T1-O1 | .649(3) | M2BO3 | 2.079(5) | ×2 |
| T1-O2 | 1.652(5) | M2BO3b | 2.073(4) | ×2 |
| T1-O2a | 1.657(5) | M2BO4 | 2.041(5) | ×2 |
| T1-O3 | 1.649(5) | <M2-O> | 2.064 | |
| <T1-O> | 1.652 | | | |
| M1-O3 | 2.104(5) ×4 | X-O1c | 3.306(7) | ×2 |
| M1-O4a | 2.069(6) ×2 | X-O1d | 3.146(5) | ×2 |
| <M1-O> | 2.092 | X-O2 | 3.291(5) | ×4 |
| | | X-O2c | 3.138(5) | ×4 |
| O4-H | 0.97(3) | <X-O> | 3.218 | |
| O1-T1-O2 | 108.1(3) | O3-M2-O3b | 84.1(2) | ×2 |
| O1-T1-O3 | 110.9(3) | O3BM2-O3e | 85.8(3) | |
| O1-T1-O2a | 108.1(3) | O3-M2-O3f | 95.3(2) | ×2 |
| O2-T1-O2a | 108.0(2) | O3-M2-O4 | 96.2(2) | ×2 |
| O2-T1-O3 | 110.8(2) | O3b-M2-O4 | 84.5(2) | ×2 |
| O2a-T1-O3 | 110.8(2) | O3f-M2-O4 | 96.1(2) | ×2 |
| <O-T1-O> | 109.5 | O4BM2-O4e | 81.7(3) | |
| | | <M2-O> | 90.0 | |

| | | |
|-----------|---------|----|
| O3-M1-O3e | 84.5(3) | ×2 |
| O3-M1-O3d | 95.5(3) | ×2 |
| O3-M1BO4a | 97.0(2) | ×4 |
| O3-M1-O4b | 83.0(2) | ×4 |
| <O-M1-O> | 90.0 | |

Notes: Coordinates for a are: B2+x, B2+y, z; Coordinates for b are: 2Bx, 2By, 1Bz; Coordinates for c are: B2+x, 2+y, z; Coordinates for d are: 2+x, 2+y, z; Coordinates for e are: Bx, y, 1Bz; Coordinates for f are: B2+x, 2By, z; Coordinates for g are: Bx, By, 1Bz; Coordinates for h are: x, By, z.

with the refined site-scattering at the M sites (Table 6). Including the calculated Li content at the M sites gives a calculated site-scattering value of 48.2 epfu (electrons per formula unit) as compared to the refined site-scattering value of 47.6 epfu. Omitting the Li content and normalizing the site populations to complete occupancy gives a calculated M site-scattering value of 51.9 epfu, in significant disagreement with the refined value of 47.6 epfu. This indicates that Li occupies the M sites in the quantity calculated by the procedure described above.

Site populations

The unit formula calculated from the chemical analysis (Table 5) shows 1.089 ⁴¹Al apfu, with a <T-O> distance of 1.652 Å. Figure 2 shows the variation in <T-O> as a function of ⁴¹Al content in some recent refinements of micas (Brigatti and Davoli 1990; Brigatti et al. 1991; Brigatti and Poppi 1993; Bigi and Brigatti 1994; Alietti et al. 1995). Ignoring a couple of outliers, the data show a well-developed trend that can be defined by the linear regression equation <T-O> = 1.612 + 0.036 ⁴¹Al ($R^2 = 0.964$). The data from the present study fall almost exactly on the line defined by this equation, indicating that the structure refinement and electron-microprobe results are compatible.

The M sites. There are two octahedrally coordinated M sites, M1 and M2. The M1 site lies on the mirror plane and has two (OH,F) ligands in a trans arrangement; the M2 site lies off the mirror plane and has two (OH,F) ligands in a cis arrangement. There is potential for cation ordering over these two sites, but ordering of Mg and Fe²⁺ is stated to be "not common" by Bailey (1984). As noted above, the collective scattering from the M sites is in accord with the contents of these sites as indicated by the unit formula (Table 5). The refined scattering value at the sites are similar, allowing for the difference in site multiplicity (Table 6). However, the mean bond lengths at the two

TABLE 5. Chemical composition and unit formula* of rubidian cesian phlogopite

| | | wt% | | |
|--------------------------------|--------|-----|------------------|-------|
| SiO ₂ | 35.15 | | Si | 2.911 |
| Al ₂ O ₃ | 15.02 | | ⁴¹ Al | 1.089 |
| TiO ₂ | 0.65 | | T | 4.000 |
| FeO | 14.41 | | | |
| MnO | 0.62 | | Al | 0.377 |
| MgO | 9.71 | | Fe | 0.998 |
| Li ₂ O† | (1.02) | | Ti | 0.040 |
| K ₂ O | 4.37 | | Mn | 0.043 |
| Rb ₂ O | 5.17 | | Mg | 1.199 |
| Cs ₂ O | 6.60 | | Li | 0.343 |
| F | 1.72 | | M | 3.000 |
| H ₂ O† | (2.80) | | | |
| O = F | -0.72 | | K | 0.462 |
| Total | 96.53 | | Cs | 0.233 |
| | | | Rb | 0.275 |
| | | | X | 0.970 |
| | | | F | 0.451 |
| | | | OH | 1.549 |

*Based on 12(O,OH,F) with OH + F = 2.0.

†Estimated by stoichiometry.

sites are significantly different: 2.092 vs. 2.064 Å, indicating that there is significant cation-ordering over these two sites. Assuming that the Fe is dominantly in the divalent state, the small <M2-O> distance must be caused primarily by the ordering of small highly charged cations at this M2 site. Note that this is in accord with the occurrence of Al at M2 in dioctahedral micas.

In most Li-bearing micas, Li tends to be partly ordered at the M1 site. However, we have no way of assigning the three significant occupants (Mg, Fe²⁺, and Li) to the M1 and M2 sites, particularly as we have no estimate of the Fe³⁺ content of this crystal.

The interlayer site. The unit formula indicates that the interlayer site contains significant amounts of K, Rb, and Cs. Moreover, the refined site-scattering value of 31.2 epfu is in excellent accord with the effective scattering value of 31.8 epfu calculated from the unit formula (Table 5).

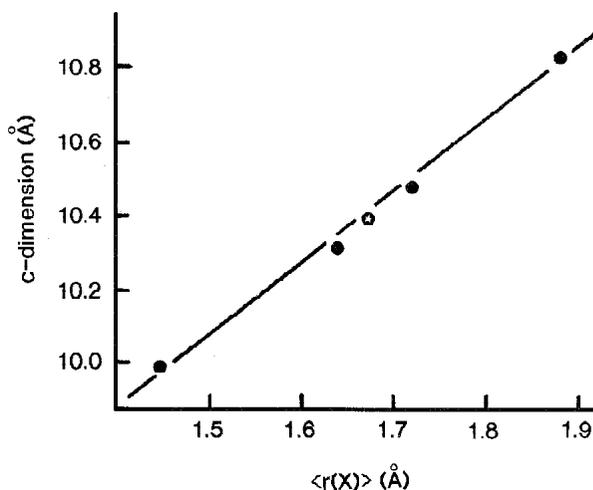


FIGURE 3. Variation in the *c* unit-cell dimension as a function of interlayer-cation radius for 12-fold coordination for synthetic phlogopite with X = Na, K, Rb, Cs (shaded circles, Hazen and Wones 1972), and rubidian cesian phlogopite (starred circle, this study); standard deviations are smaller than the data symbols.

TABLE 6. Refined site-scattering parameters (epfu) and assigned site-populations (apfu)

| | Site scattering | Site population |
|----|-----------------|---|
| M1 | 16.0 | 1.0 (Mg + Fe + Li) |
| M2 | 31.6 | 0.377 Al + 0.04 Ti + 1.583 (Mg + Fe + Li) |
| X | 31.2 | 0.462 K + 0.275 Rb + 0.233 Cs |

TABLE 7. Coordination of the interlayer site (Å) in 1M trioctahedral micas

| | | Biotite* | (Rb,Cs) phlogopite | Ba-rich mica† |
|------------------------|----|----------|--------------------|---------------|
| X-O1c | ×2 | 3.327(5) | 3.306(7) | 3.396(3) |
| X-O1d | ×2 | 3.029(5) | 3.146(2) | 2.866(3) |
| X-O2 | ×4 | 3.289(3) | 3.291(5) | 3.392(2) |
| X-O2c | ×4 | 3.028(3) | 3.138(5) | 2.864(2) |
| <X-O> | | 3.165 | 3.218 | 3.129 |
| <X-O> _{inner} | | 3.029 | 3.142 | 2.865 |
| <X-O> _{outer} | | 3.308 | 3.299 | 3.393 |

*Sample M13 from Brigatti and Davoli (1990).

†Sample 26 from Brigatti and Poppi (1993).

The interlayer-cation site occurs between the T-O-T (tetrahedral-octahedral-tetrahedral) layers that stack along the *z* direction. Thus variation in the size of the interlayer cation will strongly affect the *c*-dimension of the unit cell. Figure 3 shows the relation for the synthetic phlogopites (Hazen and Wones 1972) and the rubidian cesian phlogopite of this study. The smooth monotonic relation suggests that complete solid-solution in the K-Rb-Cs system is possible, in accord with the results for the phlogopite of this study, and in accord with our extensive electron-microprobe work on micas from Red Cross Lake.

Coordination of the interlayer site

The coordination of the interlayer site in rubidian cesian phlogopite is compared with analogous coordinations in biotite and barium-bearing mica (Table 7). It is immediately apparent that the <X-O> bond length has expanded to incorporate the larger Rb and Cs cations. The few data available show a linear relation (Fig. 4) between mean bond length and constituent-cation radius with a slope close to unity, the ideal value

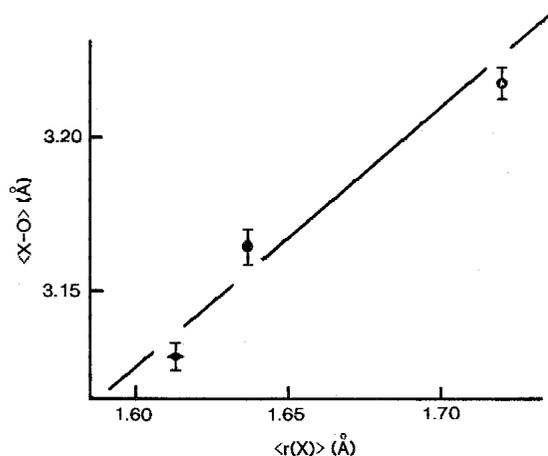


FIGURE 4. Variation in <X-O> distance as a function of constituent interlayer-cation radius (shaded circle = M13 of Brigatti and Davoli 1990. Shaded diamond = 26 of Brigatti and Poppi 1993. Starred circle = rubidian cesian phlogopite, this study).

for a hard-sphere model. The interlayer coordination polyhedron is also a lot more regular in rubidian cesian phlogopite than in the other trioctahedral micas of Table 7. Brigatti and Poppi (1993) have shown that there is an inverse linear relation between <X-O>_{inner} and θ , the tetrahedral ring-rotation angle. The rubidian cesian phlogopite refined here has a <X-O>_{inner} value of 3.142 Å and an θ value of 3.9°, and lies well off the trend of Brigatti and Poppi (1993).

Ni and Hughes (1996) report the crystal structure of nanpingite-2M₂, ideally CsAl₂(AlSi₃)O₁₀(OH)₂, the Cs equivalent of muscovite-2M₂. In nanpingite-2M₂, the interlayer-site occupancy is close to complete occupancy by Cs, and hence the <X-O> distance of 3.295 Å is considerably larger than that in rubidian cesian phlogopite, but the former values show reasonably close agreement with the trend of Figure 4.

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