Crystal structure refinement of aluminian lizardite-$2H_2$

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Abstract

Well-crystallized euhedral crystals of aluminian lizardite-$2H_2$ $(Mg_{2.35}Fe^{2+}_{0.06}Fe^{3+}_{0.07}Al_{0.52})$ $(Si_{1.41}Al_{0.59})O_{5.00}(OH)_{4.00}$ were found near Schio (Vicenza, Italy). To gain insight into the role of a high Al content lizardite, chemical analyses and single-crystal X-ray data collection were conducted. Structure refinement, completed in space group $P6_3$ (agreement factor $R = 0.034$), gives mean T-O values of 1.654 Å and 1.664 Å for T1 and T2 sites, respectively. The ditrigonal distortion of the six-membered tetrahedral ring is positive ($\alpha = +9.7°$), as expected for the $2H_2$ polytype. The octahedral site has a mean bond length similar to that of the Mg-rich octahedra of amesite and distortion parameters similar to those of Al-rich octahedra.

Introduction

Trioctahedral hydrous phyllosilicates of the 1:1 layer type show high variability both in adjacent layer positioning, which produces different polytypes, and in cation ordering, which produces a lower symmetry than the ideal polytype space group (Bailey 1988). In flat serpentine polymorphs the exchange vector $^{[4]}Si_{-1}^{[6]}Mg_{-1}^{[4]}Al^{[6]}Al$ links the end-members lizardite, ideal formula $Mg_3Si_2O_5(OH)_4$, and amesite, ideal formula $(Mg_2Al)(SiAl)O_5(OH)_4$.

In the lizardite-amesite series the few crystal-structure refinements of the two-layer polytypes ($c \cong 14$ Å) indicate that cation ordering is unlikely near end-member lizardite (space group $P6_3cm$) composition, whereas it does occur and lowers the symmetry in crystals near the end-member amesite composition (Mellini and Zanazzi 1987; Wicks and O'Hanley 1988). Amesite-$2H_2$ may display only tetrahedral Si-Al ordering, and this preserves the identity of the ideal hexagonal space group $P6_3$ (Bailey 1988). However, the presence of additional octahedral Mg-Al ordering or a minor distortion in the structure of amesite-$2H_2$ lowers the symmetry of the layer to triclinic $P1$ (Steinfink and Brunton 1956; Hall and Bailey 1979; Anderson and Bailey 1981; Wicks and O'Hanley 1988).

Detailed three-dimensional structural refinements are available for lizardite-$1T$ crystals, refined in space group $P31m$ (Mellini 1982; Mellini and Zanazzi 1987; Mellini and Viti 1994), for lizardite-$2H_1$, refined in space group $P6_3cm$ (Mellini and Zanazzi 1987), for amesite-$2H_2$, refined in space group $C1$ (Hall and Bailey 1979; Anderson and Bailey 1981; Zheng and Bailey 1995), and for an amesite non-standard polytype, refined in space group $C1$ (Wiewiora et al. 1991). Recently, the single-layer lizardite structure was refined in the monoclinic space group Cm (Krstanovic and Karanovic 1995). Structural information on chemically analyzed specimens within the lizardite-amesite series is scant; thus little information exists about tetrahedral and octahedral Al ordering. Wicks and O'Hanley (1988) emphasized a tendency for amesite with higher Al contents to order, whereas Anderson and Bailey (1981) hypothesized that, for similar crystallization conditions, the ordering pattern can be independent of composition and of polytypic sequence.

This paper reports the structure refinement of an aluminian lizardite-$2H_2$ polytype from Schio (Vicenza, Italy), which has an Al content intermediate between lizardite and amesite. The purposes of this study are to verify the ordering preference of Al, if any, and to determine how Al substitution affects the crystal-chemical features of the aluminian lizardite-$2H_2$ in comparison with the end-members lizardite and amesite.

Occurrence and specimen description

Well-crystallized euhedral crystals of aluminian lizardite-$2H_2$ were found near Schio (Vicenza, Italy). This area is mainly composed of sedimentary formations of dolomitic and gypsum-containing limestones, with subvolcanic masses, seams, and lavas (Boscardin et al. 1988).

The mineral occurs in the form of overturned hexagonal or triangular pyramids (greatest dimensions: 0.2 mm in diameter and 0.4 mm in length) from light gray to green-clear blue in color, in a 1.0×2.5 mm cavity (Fig. 1). Aluminian lizardite-$2H_2$ is associated with transparent lemon-yellow vesuvianite, hydrogarnet, bottle-green hexagonal plates of lizardite, and fibers of clear pink trioctahedral smectite.

Experimental procedures

Electron microprobe analysis

The preliminary qualitative chemical investigation, carried out by energy-dispersive analysis (EDS) during scanning electron microscope observations (Philips SEM-XL 40), revealed that some crystals contain abundant Mg, Al, and Si, with a low Fe content. The quantitative chemical data (Table 1), for the same crystal used for the structure refinement, were obtained using an ARL-SEMQ wavelength-dispersive (WDS) electron micropro-

FIGURE 1. Microphotograph of aluminian lizardite-$2H_2$ from Schio (Vicenza, Italy). Field of view is 0.4 mm.

TABLE 1. Chemical data for aluminian lizardite-$2H_2$ from Schio (Vicenza, Italy)

Oxide	Wt%	Atom	Unit-cell content*
SiO$_2$	30.35	[4]Si	1.41
Al$_2$O$_3$	21.11	[4]Al	0.59
Fe$_2$O$_3$†	1.99	Sum	2.00
FeO†	1.55		
MgO	33.90	[6]Al	0.52
H$_2$O‡	12.10	[6]Fe^{3+}	0.07
Sum	100.00	[6]Fe^{2+}	0.06
		[6]Mg	2.35
		Sum	3.00

* Unit-cell content calculated on the basis of O$_5$(OH)$_4$.
† Total Fe was shared between the two oxidation states to ensure full occupancy of cation sites and charge balance.
‡ Calculated by difference.

be operating at 15 kV accelerating voltage, 15 nA sample current, and 8 μm electron beam diameter and using the Φ(ρZ) correction procedures (Pouchou and Pichoir 1985). Fifteen point analyses were performed; the intergranular compositional variation is <1% relative for the oxide of the more abundant elements and >3% relative of the oxide for the less abundant ones. The chemical formulas were calculated on the basis of O$_5$(OH)$_4$ and the Fe^{2+} and Fe^{3+} contents were calculated assuming full occupancy of the cation sites by Si, Mg, Fe, and Al (five cations per unit cell) and charge balance (Mellini 1982).

The H$_2$O content was calculated by difference from 100 wt% as 12.10 wt%, i.e., lower than the expected value of 12.96 wt%. This difference may be due to unknown analytical errors as well as to partial dehydration of aluminian lizardite-$2H_2$ under the electron beam. Unfortunately, the shortage of material prevented an independent H$_2$O determination.

Single-crystal X-ray data collection and structure refinement

In our preliminary investigation, thin slices of some lizardite crystals were analyzed under crossed nicols to verify homogeneous extinction and the lack of twinning. The best crystal (a truncated pyramid 0.09 mm across the minor hexagonal base, 0.13 mm across the major hexagonal base, and 0.19 mm down the edge) was used for X-ray diffraction measurements.

$P6_3$ symmetry. Single-crystal X-ray intensity data were obtained using a four-circle, automated Enraf Nonius CAD4 diffractometer, operating at 52 kV, 40 mA, and using graphite-monochromatized MoKα radiation (λ = 0.71073 Å). The cell dimensions [a = 5.334(1), c = 14.370(4) Å, V = 354.1 Å^3] were refined using the 2θ values for 25 centered reflections with $15° \leq \theta \leq 25°$. Reflections with $-1 \leq h \leq +8$, $-1 \leq k \leq +14$, $-16 \leq l \leq +16$ were measured in the θ

range 1.5–35.0° using ω scans (scan width = 2°). The intensities were corrected for Lorentz and polarization effects and for absorption using a complete Ψ scan (0–360° at 10° intervals in Ψ) with more than five selected reflections. The values of the equivalent reflections were then averaged. The calculated discrepancy value R_{sym} ($R_{sym} = \Sigma |F^2_{hkl} - F^2_{hkl}$ mean$| / \Sigma F^2_{hkl}$) between equivalent reflections in the hexagonal Laue symmetries $6/mmm$ and $6/m$ was 0.036 ($6/mmm$ Laue symmetry) and 0.016 ($6/m$ Laue symmetry). The structure refinement was therefore performed in space group $P6_3$ using 316 averaged reflections with $I \geq 3\sigma_I$ by full-matrix least-squares techniques (SHELX76, Sheldrick 1976), starting from the idealized structural model for the $2H_2$ polytype. Fully ionized scattering factors were used for octahedral cations, whereas both neutral and ionized scattering factors were used for occupants of tetrahedral and anion sites, and a neutral scattering factor was used for H atoms. The atomic scattering curves were taken from *International table for X-ray crystallography* (Ibers and Hamilton 1974) and from Tokonami (1965). The starting structural model was refined with isotropic displacement factors to R = 0.073. Final refinements, performed using atomic scattering factors consistent with chemical analysis, anisotropic displacement factors for octahedral, tetrahedral, and anion sites, and isotropic displacement factors for two independent H atoms (located by difference-Fourier maps), produced final agreement factors R = 0.034 and R_w = 0.029. The final difference-Fourier maps were featureless.

Final atomic displacement and positional parameters are reported in Table 2. Selected interatomic distances are reported in Table 3, selected octahedral and tetrahedral distortion parameters are listed in Table 4, and the observed and calculated structure factors are given in Table 5[1].

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[1] For a copy of Table 5, order Document AM-97-645 from the Business Office, Mineralogical Society of America, 1015 Eighteenth St., NW, Suite 601, Washington, DC 20036, U.S.A. Please remit $5.00 in advance. Deposit items may also be available on the *American Mineralogist* web site, refer to the inside back cover of a current issue for the web address.

TABLE 2. Final atomic fractional coordinates, equivalent isotropic and anisotropic (Å²) displacement factors of aluminian lizardite-2H_2 from Schio (Vicenza, Italy)

Atom	x/a	y/b	z/c	B_{eq}	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
T1	0	0	0.1303(3)	1.19(7)	0.99(8)	0.99(8)	1.6(1)	0.50(4)	0	0
T2	⅔	⅓	0.1285(3)	0.88(6)	0.76(7)	0.76(7)	1.1(1)	0.38(4)	0	0
M	0.6695(8)	0.0020(4)	0.3215(2)	1.11(1)	0.94(1)	1.15(1)	1.57(1)	0.77(1)	−0.01(1)	−0.19(1)
O1	0	0	0.2412(6)	2.2(2)	1.3(2)	1.3(2)	3.9(4)	0.66(9)	0	0
O2	⅔	⅓	0.2471(5)	2.4(2)	2.3(2)	2.3(2)	2.6(3)	1.2(1)	0	0
O3	0.7829(7)	0.1179(7)	0.0899(3)	2.4(2)	2.3(2)	2.8(2)	2.6(2)	1.5(2)	−0.3(2)	−0.8(2)
O4	0.3356(8)	0.0038(8)	0.3916(2)	2.0(2)	1.5(2)	2.1(2)	2.0(2)	0.5(2)	0.1(2)	−0.9
O5	⅓	⅔	0.2469(4)	1.1(1)	1.2(1)	1.2(1)	0.8(2)	0.6(7)	0	0
H4	0.30(1)	0.04(1)	0.459(4)	3.2(1)						
H5	⅓	⅔	0.179(3)	3.8(1)						

Note: The form of the anisotropic displacement parameter is exp[−¼($\beta_{11}h^2a^{*2}$ + $\beta_{22}k^2b^{*2}$ + $\beta_{33}lc^*$ + $2\beta_{12}hka^*b^*$ + $2\beta_{13}hla^*c^*$ + $2\beta_{23}klb^*c^*$)].

C1 symmetry. We considered the possibility of the loss of the ideal symmetry of the structure from space group $P6_3$ to the triclinic space group $P1$, which can be produced, as in amesite, by cation ordering. To assess this possibility, a complete data set of intensities was collected on the same crystal, using a triclinic symmetry (space group $C1$), a rotating-anode, single-crystal, four-circle automated Siemens P4P diffractometer (MoKα graphite-monochromatized radiation, at 52 kV and 140 mA operating conditions) and X-SCANS software (Siemens 1993). The same data collection parameters and intensity corrections were used as in the collection of the earlier data set in $6/m$ Laue symmetry. The structure was refined using the orthohexagonal cell in space group $C1$, after Anderson and Bailey (1981) (cell parameters $a_{triclinic}$ = $a_{hexagonal}$, $b_{triclinic}$ = $\sqrt{3}\ b_{hexagonal}$, $c_{triclinic}$ = $c_{hexagonal}$, α = 90°, β = 90°, γ = 90°). The starting atomic coordinates were derived from equivalent positions of the $P6_3$ space group for the same setting used by Anderson and Bailey (1981). During the least-squares refinement the position of the

tetrahedral cation T1 was fixed to establish an origin in the non-centrosymmetric structure. With respect to the $P6_3$ refinement, the refinement in $C1$ gave an R index definitely higher (R = 0.055). Moreover, the refinement of this aluminian lizardite-2H_2 crystal in space group $C1$ cannot be considered reliable for the following reasons: (1) After every cycle of least-squares refinement the isotropic displacement parameters of some O atoms became negative; (2) Numerous significant correlation effects (interaction parameters larger than 0.97) for the subgroup symmetry refinement were indicated; (3) There was no evidence of ordering with respect to the ideal 2H_2 polytype in the final values of cation-oxygen distances, the polyhedral distortion parameters, the thermal displacement factors, or the occupancies. In particular, the mean bond distances and the distortion parameters of the six octahedra derived in $C1$ symmetry are comparable within

TABLE 3. Selected bond lengths (Å) of aluminian lizardite-2H_2 from Schio (Vicenza, Italy)

T1-O1	1.594(9)	O1-O3 × 3	2.682(8)
T1-O3 × 3	1.674(5)	O3-O3 × 3	2.719(8)
⟨T1-O⟩	1.654	⟨O-O⟩$_{T1}$	2.701
T2-O2	1.704(8)	O2-O3 × 3	2.742(7)
T2-O3 × 3	1.651(5)	O3-O3 × 3	2.693(8)
⟨T2-O⟩	1.664	⟨O-O⟩$_{T2}$	2.717
⟨T-O⟩	1.659	⟨O-O⟩	2.709
M-O1	2.111(6)	O4-H4	1.02(6)
M-O2	2.072(6)	O5-H5	0.98(4)
M-O4	2.020(5)		
M-O4′	2.050(6)		
M-O4″	2.061(5)		
M-O5	2.087(4)		
⟨M-O⟩	2.067		
O1-O2	3.081(1)	O1-O4	2.800(7)
O1-O5	3.081(1)	O1-O4″	2.800(7)
O2-O5	3.079(1)	O2-O4	2.723(6)
O4-O4′	3.051(5)	O2-O4′	2.723(6)
O4-O4″	3.083(8)	O4′-O5	2.744(6)
O4′-O4″	3.104(8)	O4″-O5	2.744(6)
e_u	3.080	e_s	2.756
⟨O-O⟩$_M$	2.918		
Tetrahedral thickness	2.217 Å		
Octahedral thickness	2.106 Å		
Interlayer separation	2.863 Å		

TABLE 4. Selected distortion parameters of aluminian lizardite-2H_2 from Schio (Vicenza, Italy)

α (°)	+9.7	BLD$_{T2}$ (%)	1.199
Dimensional misfit (Å)	0.605	ELD$_{T2}$ (%)	0.905
TAV$_{T1}$ (°)²	0.81	ψ (°)	59.37
τ$_{T1}$ (°)	110.3	OAV (°)²	45.98
BLD$_{T1}$ (%)	1.805	e_u/e_s	1.118
ELD$_{T1}$ (%)	0.691	BLD$_M$ (%)	1.121
TAV$_{T2}$ (°)²	0.03	ELD$_M$ (%)	5.554
τ$_{T2}$ (°)	109.6		

Notes: α (tetrahedral rotation angle) = $\sum_{i=1}^{6} \alpha_i/6$ where $\alpha_i = |120° - \phi_i|/2$ and where ϕ_i is the angle between basal edges of neighboring tetrahedra articulated in the ring. τ (tetrahedral flattening angle) = $\sum_{i=1}^{3}$ (O$_{basal}$ − T̂ − O$_{apical}$)/3. TAV (tetrahedral angle variance) = $\sum_{i=1}^{6}$ (θ_i − 109.47)²/5 (Robinson et al. 1971). ψ (octahedral flattening angle) = cos⁻¹[octahedral thickness/(2⟨M-O⟩)] (Donnay et al. 1964). e_u, e_s = mean lengths of unshared and shared edges, respectively (Toraya 1981). OAV (octahedral angle variance) = $\sum_{i=1}^{12}$ (θ_i − 90°)²/ll (Robinson et al. 1971). BLD (bond length distortion) = $\frac{100}{n}\sum_{i=1}^{n}\frac{|(X - O)_i - \langle X - O\rangle|}{\langle X - O\rangle}$% where n is the number of bonds (Renner and Lehmann 1986). ELD (edge length distortion) = $\frac{100}{n}\sum_{i=1}^{n}\frac{|(O - O)_i - \langle O - O\rangle|}{\langle O - O\rangle}$% where n is the number of bonds (Renner and Lehmann 1986). Δ$_{TM}$ (dimensional misfit) = $2\sqrt{3}$ ⟨O-O⟩$_{basal}$ − $3\sqrt{2}$ ⟨M-O⟩ (Toraya 1981).

the standard deviation (e.g., 2.067 ≤ <M-O> ≤ 2.075 Å, σ = 0.008 Å; 59.1° ≤ Ψ ≤ 59.4°, σ = 0.3°).

DISCUSSION AND CONCLUSIONS

The chemical analyses yield the following formula, calculated on the basis of charge balance and five cations per unit cell and assuming that all Fe is octahedrally co-ordinated and that there are the ideal two H_2O molecules per formula unit: $(Mg_{2.35}Fe^{2+}_{0.06}Fe^{3+}_{0.07}Al_{0.52})(Si_{1.41}Al_{0.59})O_{5.00}$ $(OH)_{4.00}$. The Al content, intermediate between lizardite and amesite, and the crystal symmetry allow us to define the analyzed crystal as aluminian lizardite-2H_2.

Tetrahedral sheet

The mean T-O values of 1.654 Å and 1.664 Å for T1 and T2 sites, respectively, are slightly different but well within one standard deviation of each other. Greater differences between the two polyhedra emerge from the analysis of their distortions, i.e., ELD (edge length distortion), BLD (bond length distortion), TAV (tetrahedral angle variance), and τ (tetrahedron flattening). The definitions of the different distortion modes, as well as the individual values, are reported in Table 4. The ELD parameter, which shows the degree of deviation from a regular tetrahedron, is less for the T1 than for the T2 site, whereas the BLD parameter, which measures the tetrahedral cation off-center shift, is greater for the T1 than for the T2 site. In addition, the T1 site displays greater angular distortions (TAV$_{T1}$ = 0.81°²; TAV$_{T2}$ = 0.03°²), and the tetrahedral flattening angle τ indicates that T2 is more flattened than T1. According to the conclusions of Renner and Lehmann (1986), who observed that a decrease in size of the central ions increases the off-center shift of tetrahedral cations, these features could indicate a preference of Al for the T2 site. Nevertheless, differences between T1 and T2 sites are mostly produced by short T1-O1 and long T2-O2 apical bond distances. In agreement with Mellini and Zanazzi (1987), it is uncertain whether this is due to cation ordering or is an artifact derived from the strongly anisotropic vibrational ellipsoids that are elongated parallel to [001] (Table 2).

The Al content of the T sheet was calculated using the linear relationship proposed by Anderson and Bailey (1981), <T-O> = 1.619 Å + 0.14 [$X_{Al}/(X_{Al} + X_{Si})$], which gives [4]Al% = 29%, where [4]Al% = [$X_{Al}/(X_{Al} + X_{Si})$] × 100. This result is in good agreement with the [4]Al% value determined by chemical analysis ([4]Al% = 29.5%). Also the [4]Al content seems to be responsible for the distortion of the tetrahedral ring, the α values increasing in the transition from lizardite to amesite. In lizardite, where the [4]Al content is < 9% of the site occupancy, |α| ranges between 1.7° and 6.4°, whereas in amesite, where the [4]Al content is >46%, the |α| value varies from 13.6° to 14.7°. In aluminian lizardite-2H_2 the |α| angle (|α| = 9.7°) is in good agreement with the tetrahedral Al for Si substitution of 29.5%. The direction of the tetrahedral ring rotation is positive, as in lizardite-2H_1 and amesite-2H_2.

Octahedral sheet

Unlike for amesite, the crystal structure refinement of aluminian lizardite-2H_2 in space group $C1$ did not make structural ordering evident; its structure is therefore characterized by only one symmetrically independent octahedral site.

When compared with the amesite of Anderson and Bailey (1981), aluminian lizardite-2H_2 has a <M-O> bond length (<M-O> = 2.067 Å) that is intermediate between those in amesite with four Mg-rich sites (<M1-O> = 2.056 Å; <M11-O> = 2.085 Å; <M3-O> = 2.056 Å; <M33-O> = 2.088 Å), whereas its edge distortion (e$_u$/e$_s$ parameter, where e$_u$ and e$_s$ are the mean values of unshared and shared octahedral edges, respectively) is comparable to those calculated for Al-rich octahedra (aluminian lizardite-2H_2: e$_u$/e$_s$ = 1.118; amesite: e$_u$/e$_{sM2}$ = 1.127, e$_u$/e$_{sM22}$ = 1.102).

The octahedral cation is shifted toward the O4 plane, as was observed in lizardite (Mellini and Viti 1994), with the result that the M-O4 bond distances are shorter than those between the octahedral cation and the O1, O2, and O5 O plane. The BLD parameter (BLD = 1.12%) of aluminian lizardite-2H_2 is similar to those of amesite Alrich octahedra (BLD$_{M2}$ = 1.09%, BLD$_{M22}$ = 1.20%), whereas it is lower than those of lizardite (1.91 ≤ BLD ≤ 2.21%).

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