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THE CRYSTAL CHEMISTRY OF As- AND Sb-BEARING DUMORTIERITE

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ABSTRACT

Dumortierite samples from two pegmatite localities in Antarctica and one each in Germany and Russia show a range of As and Sb compositions (As + Sb + minor Bi = 0.001–0.212 *apfu*) and low Ta + Nb + Ti (0.001–0.079 *apfu*). Single-crystal diffraction data obtained from crystals from each sample refined to $R1$ = 0.0161–0.0285, the latter value for a twinned crystal. Initial refinements of three of the four crystals showed considerable electron density at the Sb1 and Sb2 sites; however, the atoms at these sites are also highly anisotropic, and consequently the sites were split into distinct As1, Sb1, As2, and Sb2 positions. Such distinct As and Sb sites are not seen in the isostructural mineral holtite, which contains considerably more As and Sb (and Ta, Nb, and Ti). Initial refinements also showed that in all four crystals, the atom at the Al1 site, with occupancies of 0.81–0.88, is highly anisotropic with most of the positional displacement in the **a** direction. The Al1 site was then split into Al1a, Al1, and Al1b positions, whose occupancies refined to Al1 > Al1a > Al1b. The unequal occupancy of Al1a, Al1, and Al1b suggests that the hexagonal channel contains a disordered mix of face-sharing octahedron dimers, trimers and longer units separated by vacancies. A plot of Si + P *apfu versus* As + Sb + Bi *apfu* for 340 dumortierite and 627 holtite compositions shows no gap between the two minerals. Although there is a pronounced gap in terms of As and Sb occupancy, it separates dumortierite and Sb-poor holtite from Sb-bearing holtite. The continuum of compositions between dumortierite and holtite, and the discovery of very (As, Sb)-rich, (Ta, Nb)-poor compositions, suggest that the distinction between what has been called dumortierite and what has been called holtite should be reconsidered.

Keywords: dumortierite, holtite, borosilicate, electron microprobe, X-ray diffraction, disorder, twinning.

INTRODUCTION

Dumortierite [*ca*. $(Al,\square)Al_6(BO_3)Si_3O_{13}(O,OH)_2$] is the most widespread of the three minerals in the dumortierite group and is second only to tourmaline as the most abundant borosilicate in aluminous metamorphic and metasomatic rocks (Grew 2002). Although of relatively restricted occurrence in granitic pegmatites compared to tourmaline, dumortierite is a typical mineral of the abyssal class, AB–BBe subclass of pegmatites, which Černý & Ercit (2005) recognized in their revision of the classification of pegmatites. Magnesiodumortierite [*ca*. $(Mg,Ti,\square)Al_4(Al,Mg)_2(BO_3)Si_3O_{12}(OH,O)_3$] is a rare mineral found in ultrahigh-pressure rocks of the western Alps (Chopin *et al*. 1995). Holtite [*ca*. $(Al,Ta,Nb,\square)Al_6(BO_3)(Si,Sb,As)_3O_{12}(O,OH,\square)_3$], has been described from pegmatites in Greenbushes, Western Australia (type locality), Voron'i Tundry, Kola Peninsula, in Russia, Szklary, Lower Silesia, in Poland (Pryce 1971, Voloshin *et al*. 1977, Pieczka & Marszałek 1996, Groat *et al*. 2009), and from Virorco, San Luis range, in Argentina (Galliski *et al*. 2012).

We undertook this study to investigate the crystal chemistry of dumortierite samples containing appreciable As + Sb, but less than reported for holtite, and low Ta + Nb + Ti contents, in order to better understand the crystallographic role played by As + Sb in dumortierite and how it might differ from the role played by these two constituents in holtite.

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Background Information

The crystal structure of dumortierite was described by Golovastikov (1965) and Moore & Araki (1978) as a design on the semiregular planar net {6•4•3•4}. Moore & Araki (1978) showed that the net can be broken down into four regions: (1) [AlO_3] chains of face-sharing octahedra (the Al1 sites) with circumjacent "pinwheels" of six SiO_4 tetrahedra, two Si1 and four Si2 sites, (2) [Al_4O_{12}] cubic close-packed chains, containing the Al2 and Al3 octahedral sites, that are joined to equivalent chains by reflection at the O1 corners of the Al2 octahedra to form [Al_4O_{11}] sheets oriented parallel to (010), (3) [Al_4O_{12}] double chains containing the Al4 octahedral sites, and (4) BO_3 triangles (Fig. 1). The Al1–Al1 distance is ~2.35 Å, which is unusually short for face-sharing octahedra, and the Al1 site is on average between 75% and 90% occupied (Moore & Araki 1978, Alexander *et al.* 1986, Fuchs *et al.* 2005, Evans *et al.* 2012). The chains of Al1 face-sharing octahedra are disordered, which results in an average chain length that can be adjusted to fit the repeat distance of the remaining octahedra in the framework in the structure (Moore & Araki 1978).

Hoskins *et al.* (1989) determined that the crystal structure of holtite is closely related to that of dumortierite, but it differs in several important respects, all of which lie within the first region of Moore & Araki (1978), *i.e.*, within six-sided tunnels bounded by the two regions composed of [Al_4O_{12}] chains. Both SiO_4 tetrahedra are partially replaced by $Sb^{3+}O_3$ triangular pyramids with no evidence of a preference of Sb for one of the Si sites, and Ta replaces Al at the Al1 position (Fig. 2). As a result, there are vacancies at the coordinating anion sites (O2 and O7) as well as at the Al1 site. Relative to the Si positions, the Sb^{3+} sites are shifted about 0.5 Å closer to the Al1 position to accommodate the longer Sb^{3+}–anion bonds (average ~1.9 Å). Where the Sb sites are occupied, the adjacent Si, O2 (for Si1), and O7 (for Si2) positions are vacant. Groat *et al.* (2009) refined the crystal structure of holtite samples with different amounts of (Sb,As) and (Ta,Nb) and obtained the general formula $Al_{7-[5x+y+z]/3}(Ta,Nb)_x\square_{[2x+y+z]/3}Si_{3-y}(Sb,As)_yBO_{18-y-z}(OH)_z$, where x is the total number of pentavalent cations, y is the total amount of Sb + As, and $z \leq y$ is the total amount of OH.

None of the four constituents that distinguish holtite from dumortierite is dominant at a specific crystallographic site, *i.e.*, Si is dominant over Sb^{3+} and As^{3+} at the two tetrahedral sites, and Al is dominant over Ta, Nb and a vacancy at the Al1 site in both minerals. Moreover, recent studies have narrowed the compo-

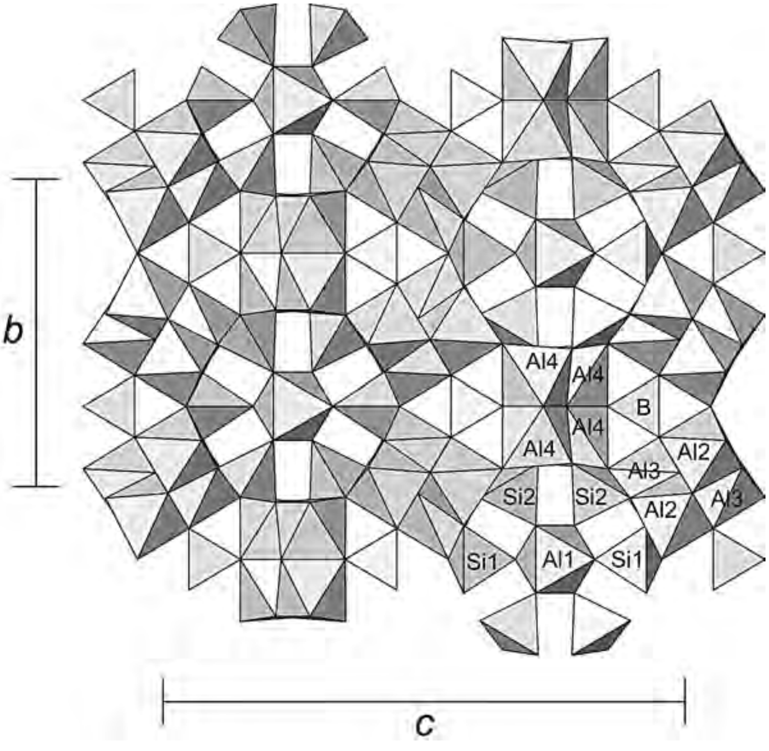

FIG. 1. The crystal structure of dumortierite observed along the **a** axis.

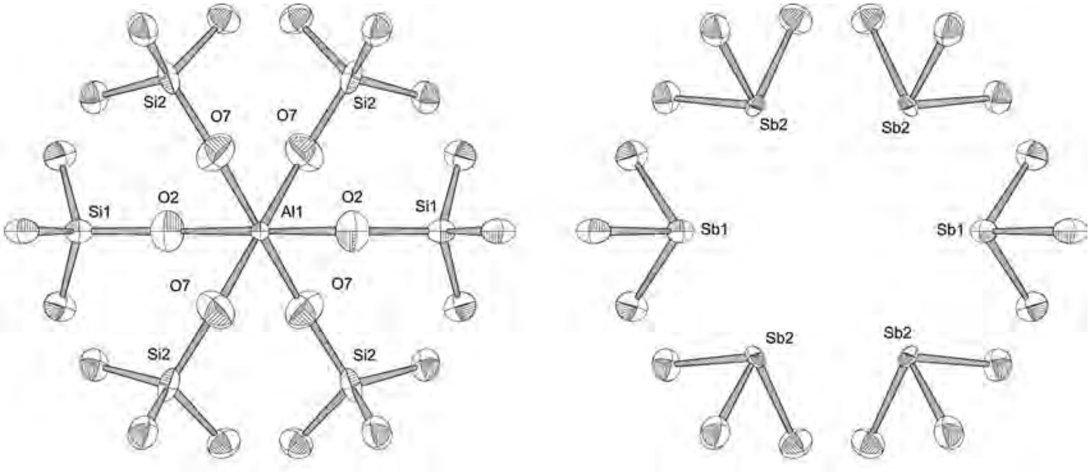

FIG. 2. Disposition of SiO_4 tetrahedra (left) and $(Sb,As)O_3$ groups (right) and the coordinated central Al1 site in holtite (after Hoskins *et al.* 1989).

sitional distinctions between dumortierite and holtite. Groat *et al.* (2001) reported up to 1.0 wt.% Sb_2O_3 in dumortierite from localities worldwide. Borghi *et al.* (2004) and Vaggelli *et al.* (2004) measured up to 4.50 wt.% Sb_2O_3 (0.19 Sb^{3+} per formula unit, *pfu*), varying inversely with Si in zoned dumortierite in quartzites from Mozambique. Cempírek & Novák (2004) found up to 3.77 wt.% As_2O_3 (0.22 As^{3+} *pfu*) in dumortierite from abyssal pegmatites at Vémyslice in the Czech Republic. Cempírek *et al.* (2010) reported up to 10.97 wt.% Sb_2O_3 (0.46 Sb^{3+} *pfu*) in zones in dumortierite crystals from the Bory Granulite Massif in the Czech Republic. On the basis of a gap in Ta + Nb + Ti contents evident in compositional data for holtite and dumortierite from the Szklary pegmatite in Poland, Pieczka *et al.* (2011) arbitrarily called a phase holtite if Ta + Nb + Ti exceeds 0.25 atoms per formula unit (*apfu*), and dumortierite if Ta + Nb + Ti is less than 0.1 *apfu*. They described a dumortierite sample with 6.50 wt.% Sb_2O_3 (0.27 Sb^{3+} *pfu*) and 5.56 wt.% As_2O_3 (0.34 As^{3+} *pfu*), and a dumortierite-like mineral with 10.23 wt.% Sb_2O_3 (0.43 Sb^{3+} *pfu*) and 16.81 wt.% As_2O_3 (1.04 As^{3+} *pfu*) and negligible Ta, Nb, and Ta, occurring as a few tiny inclusions ≤20 μm across in quartz. Galliski *et al.* (2012) described dumortierite with up to 11.79 wt.% As_2O_3 (0.701 As^{3+} *pfu*) and minor Sb from the Virorco pegmatite, San Luis, Argentina.

SAMPLES

As noted above, dumortierite is a typical mineral of the provisional AB–BBe subclass of the abyssal class of pegmatite as defined by Černý & Ercit (2005). The pegmatites of this subclass are generally strongly peraluminous and developed in complex environments during multistage events. The B and Be minerals characteristic of these pegmatites, dumortierite, grandidierite, prismatine, werdingite, chrysoberyl, beryllian sapphirine, khmaralite, and surinamite, are Al-rich phases that are better characterized as high-temperature than high-pressure phases, as Černý & Ercit (2005) wrote. Localities cited by Černý & Ercit (2005) as examples for such abyssal pegmatites are Rogaland, southwestern Norway, Andrahomana, southeast Madagascar, Kutná Hora, Czech Republic, Enderby Land, East Antarctica, South Kerala, India, and Kalanga Hill, northeastern Zambia. According to Černý & Ercit (2005), the lack of bulk-composition data for these pegmatites makes their degree of departure from truly granitic compositions unclear and deserves attention. The four samples analyzed in this study are from granitic pegmatites, including two from widely separated localities in the East Antarctic Precambrian shield, one from the Ural Mountains, Russia, and one from the Saxony granulite complex in Germany.

Sample D67 was collected in 2003 (ESG sample 121502M) from the Larsemann Hills, Prydz Bay, Princess Elizabeth Land, East Antarctica, where it occurs in a cross-cutting pegmatite at locality 121502 with tourmaline and boralsilite (Grew *et al.* 2008, Wadoski *et al.* 2011). This pegmatite is one of the later-generation anatectic pegmatites cutting B-rich granulite-facies metasedimentary rocks in the Larsemann Hills. According to Wadoski *et al.* (2011), microstructures in samples from this locality suggest the presence of at least two generations of dumortierite. Primary dumortierite occurs as coarse prisms overgrown and broken by a later, but presumably early-formed dumortierite; our crystal was selected from the primary dumortierite. A clearly secondary variety has in places partially replaced boralsilite. Wadoski *et al.* (2011) reported that Ti and As contents of dumortierite in pegmatite 121502 range from 0 to 5.45 wt.% TiO_2 and from 0 to 2.8 wt.% As_2O_3, respectively, in individual analyses; the maximum Nb_2O_5 content is 2.08 wt.%.

Sample D27 comprises small blue crystals, most likely corresponding to the deep-blue dumortierite reported as a largely microscopic accessory mineral in two pegmatite veins cutting gabbro and serpentinized peridotite on the west shore of Lake Uvil'dy in the Il'men Mountains, southern Urals, Russia (Kuznetsov 1923, Avdonin 1987). In addition to quartz, microcline and albite, the pegmatites contain tourmaline, which is mostly dark blue in thin section, also zoned with a colorless rim (one sample is pink in hand specimen), garnet, muscovite and cordierite, but none of these accessory minerals occur with dumortierite. Kuznetsov (1923) and Avdonin (1987) also described from another pegmatite a yellow dumortierite in aragonite-like triply twinned crystals, one of which Golovastikov (1965) used for a refinement of the crystal structure. Avdonin (1987) reported the chemical composition of the yellow variety, including qualitative spectral data giving a few tenths weight % As.

Sample D21 is from the Hartmannsdorf quarry 12 km northwest of Chemnitz in Saxony, Germany. The dumortierite occurs in pegmatites in the Saxony Granulite Complex (Neumann & Tischendorf 1986, Vollstädt & Weiss 1991, Anderson *et al.* 1998).

Sample D31 was collected in 1998 (ESG sample EG98122603) from a Cambrian pegmatite (498 ± 1.7 Ma, ion microprobe U–Pb age from zircon, Carson *et al.* 2002) on Tonagh Island in Amundsen Bay, Enderby Land, East Antarctica. Beryl, tourmaline, apatite, garnet and muscovite are also found in these Cambrian pegmatites (E.S. Grew, unpubl. data). The pegmatites intruded granulite-facies rocks of the Archean Napier complex, resulting in retrogression of the host rocks in discrete zones (Carson *et al.* 2002, Carson & Ague 2008).

EXPERIMENTAL

Single-crystal X-ray diffraction measurements were made at C–HORSE (the Centre for Higher Order Structure Elucidation, in the Department of Chemistry at the University of British Columbia) using a Bruker X8 APEX II diffractometer with graphite-monochromated Mo$K\alpha$ radiation. Data were collected in a series of ϕ and ω scans in 0.50° oscillations with 20.0 second exposures. The crystal-to-detector distance was 40 mm. Data were collected at room temperature (except for the D27 sample, where data collection was done at 100 K) and integrated using the Bruker SAINT software package; they were corrected for absorption effects using the multi-scan technique (SADABS) and for Lorentz and polarization effects. Examination of the data for the D27 sample showed that the crystal is made up of three twin individuals related by 120° rotation about a threefold twin axis parallel to **a**. The twinning is by reticular pseudomerohedry (obliquity 0.56°) and is based on a pseudohexagonal lattice sublattice with $a \approx 23.376$, $c \approx 4.690$ Å, $\gamma \approx 119.44°$. This cell is obtained from the orthorhombic cell *via* the matrix $/0\ \bar{1}\ 1/\ \bar{1}\ 0\ \bar{1}/1\ 0\ 0/$; because the hexagonal cell has double the volume of the orthorhombic cell (index 2), only 50% of the diffraction spots overlap (G. Ferraris, pers. commun.). The data were resolved using CELL NOW and were corrected for absorption effects using TWINABS and then for Lorentz and polarization effects.

All refinements were performed using the SHELXTL crystallographic software package of Bruker AXS. The structures were refined using starting parameters from Groat *et al.* (2009). Scattering factors for neutral atoms were used for all cations, and scattering factors for O^{2-} for oxygen. The weighting scheme was based on counting statistics. Neutral-atom scattering factors were taken from Cromer & Waber (1974). Anomalous dispersion effects were included in F_{calc} (Ibers & Hamilton 1964); the values for $\Delta f'$ and $\Delta f''$ are those of Creagh & McAuley (1992). The values for the mass attenuation coefficients are those of Creagh & Hubbell (1992).

Initially, all atoms were refined anisotropically without any splitting of sites. The occupancies of all of the Al sites were refined, and they were not linked to the occupancies of any other sites. Moore & Araki (1978) and Alexander *et al.* (1986) suggested that the apparent partial occupancies of the Al2, Al3, and Al4 sites in dumortierite are due to a correlation between site occupancies and thermal motion. Alexander *et al.* (1986) therefore scaled the refined occupancies of the Al2–4 sites by dividing them by 0.95. However, Ferraris *et al.* (1995) suggested that for their refinement of the crystal structure of magnesiodumortierite, there is no valid reason to disregard the refined values. In our study, constraining these sites to be fully occupied with Al resulted in increases in the *R* values by at least 1%; on the other hand, attempts to refine for cations in addition to Al (such as Fe) were unsuccessful.

Initial refinements showed that in all four crystals, the atom at the Al1 site is highly anisotropic, with U_{eq} values for D21, D27, D31, and D67 of 0.0313, 0.0316, 0.0251, and 0.0204 Å^2, respectively. The high U_{11} factors (0.0787, 0.071, 0.0658, and 0.0480 Å^2 for D21, D27, D31 and D67, respectively) indicate that most of the positional displacement is along the **a** direction. The structures were then refined with Al1 isotropic and U_{iso} fixed at 0.0050, which revealed electron density both above and below the Al1 position; these new sites were labeled Al1a and Al1b, respectively. The occupancies of the three Al1 sites were then allowed to vary freely, with the Al atoms at the sites constrained to have equal isotropic displacements.

The Si1 and Si2 sites were assumed to contain only silicon atoms or to be vacant. Where occupied, the sites were assumed to be coordinated by four O atoms in tetrahedral coordination. The O2 and O7 sites were assumed to contain only oxygen atoms or be vacant. Where unconstrained, the occupancies of the O2 and O7 sites refined to slightly less than the occupancies of the Si1 and Si2 positions. As was done by Groat *et al*. (2009) for holtite, the occupancies of all four sites were constrained to be equivalent (taking into account the different multiplicities of the sites). Also following Groat *et al*. (2009), the Sb1 site was assumed to be coordinated by three O atoms in trigonal coordination, and to be occupied if the Si1 site is empty; the same assumptions were made for Sb2 and Si2. The occupancies of the Si1 and Sb1 positions were constrained to vary inversely within a combined total occupancy of 1.0, as were those of Si2 and Sb2.

In the initial refinement of data from sample D67, the occupancies of the Si1, Si2, O2 and O7 sites refined to 0.988, and difference-Fourier maps showed no electron density at the As1 and As2 sites. Consequently in subsequent refinements the Si1, Si2, O2 and O7 sites were constrained to be fully occupied.

The initial refinements of data from the other three crystals showed considerable electron density at the Sb1 and Sb2 sites; however, the atoms at these sites are highly anisotropic, with Sb1 U_{eq} values of 0.032, 0.12, and 0.024 Å^2, and Sb2 U_{eq} values of 0.021, 0.08, and 0.031 Å^2 for D21, D27, and D31, respectively (Fig. 3). Consequently, the Sb1 and Sb2 sites were split into As1, Sb1, As2, and Sb2 positions, and the occupancies of the Si1, As1, and Sb1 sites were refined within a total combined occupancy of 1.0, as were the occupancies of the Si2, As2, and Sb2 positions.

Refinement of data for the twinned D27 crystal also showed that the atoms at the O1 and O6 positions are highly anisotropic, likely owing to the twinning; thus these sites were split as well into the O1, O1a, O6, and O6a positions. The isotropic displacement parameters of the atoms at the O1a and O6a positions were fixed at 0.005 Å^2, and their occupancies were constrained to vary inversely with those of the O1 and O6 positions, with total combined occupancies of 1.0 in each case.

After collection of X-ray diffraction data, the single crystals were attached to Lucite disks with Petropoxy and polished for analysis with a fully automated CAMECA SX–50 electron microprobe. Two of the structure crystals, D27 and D31, were lost during polishing, and thus were not available for analysis. In addition, compositions of other crystals in samples D21, D27 and D31 were measured in order to assess the heterogeneity of the bulk sample. The Cameca was operated in the wavelength-dispersion mode with the following operating conditions: excitation voltage: 20 kV, beam current: 20 nA, peak count time: 20 s, background count time: 10 s, beam diameter: 10 μm. Data reduction was done with the "PAP" $\phi(\rho Z)$ method (Pouchou & Pichoir 1985). For the elements considered, the following standards and X-ray lines were used: kyanite, Al*K*α, Si*K*α; apatite, P*K*α; rutile, Ti*K*α; synthetic fayalite, Fe*K*α; tennantite, As*K*α; columbite, Nb*L*α; tetrahedrite, Sb*L*α; microlite, Ta*M*α; Bi metal, Bi*M*α. Formulae were calculated on the basis of 18 (O + F + As + Sb) per formula unit and assuming 1 B *pfu*; this takes into account the vacancies created at the O2 and O7 sites with substitution of As and Sb at the Sb sites (see Groat *et al*. 2009).

RESULTS

Electron-microprobe compositions

Average electron-microprobe compositions with standard deviations are given in Table 1. The maximum Ta + Nb + Ti value is 0.08 *apfu*, which is within the range 0.0–0.1 *apfu* reported by Pieczka *et al*. (2011) for dumortierite in the Szklary pegmatite. All of the dumortierite samples except D67 contain elevated As and Sb, with maximum values of 2.85 wt.% As_2O_3 (0.18 As^{3+} *pfu*) and 1.03 Sb_2O_3 (0.04 Sb^{3+} *pfu*) for sample D31. Dumortierite D27 also contains some Bi (1.07 wt.% Bi_2O_3, corresponding to 0.03 Bi^{3+} *pfu*). The dumortierite crystals in samples D21, D27 and D31 show a wide compositional range compared with the compositions of the structure crystals for D21 and D67. The compositions of crystals other than the structure crystal in sample D67 were not measured.

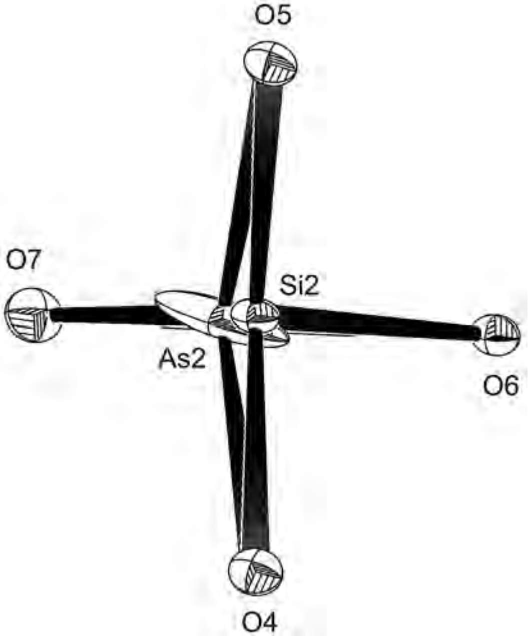

FIG. 3. The atomic displacement ellipsoid for As, Sb in sample D31 prior to splitting.

TABLE 1. AVERAGE COMPOSITIONS OF DUMORTIERITE, WITH STANDARD DEVIATIONS

	D67		D27		D21		D21		D31	
	Antarctica		Russia		Germany		Germany		Antarctica	
	Structure crystal		Bulk		Structure crystal		Bulk		Bulk	
	n = 15		*n* = 7		*n* = 5		*n* = 5		*n* = 7	
	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.
P_2O_5 wt.%	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.12	0.03
Nb_2O_5	0.01	0.01	0.17	0.07	0.03	0.04	0.01	0.01	0.38	0.15
Ta_2O_5	0.02	0.04	0.05	0.06	0.01	0.02	0.04	0.04	1.62	1.25
SiO_2	30.59	0.18	27.19	0.46	28.73	0.12	28.33	1.34	26.22	1.65
TiO_2	0.10	0.01	0.94	0.26	0.92	0.06	0.29	0.51	0.04	0.02
B_2O_3	6.03	0.02	5.80	0.02	5.86	0.01	5.82	0.04	5.72	0.09
Al_2O_3	62.09	0.17	59.14	0.27	57.65	0.12	57.77	0.71	57.39	1.38
Fe_2O_3	0.36	0.02	0.21	0.14	0.95	0.02	0.92	0.12	1.01	0.10
As_2O_3	0.06	0.02	1.69	0.50	1.82	0.10	2.32	1.48	2.85	1.09
Sb_2O_3	0.01	0.01	0.68	0.08	0.62	0.04	0.57	0.34	1.03	0.40
Bi_2O_3	0.02	0.02	1.07	0.24	0.02	0.02	0.01	0.01	0.17	0.09
MgO	0.01	0.00	0.08	0.19	0.84	0.01	0.82	0.19	0.21	0.06
F	0.09	0.08	0.07	0.05	0.07	0.05	0.06	0.06	0.04	0.04
O=F	-0.04	0.03	-0.03	0.02	-0.03	0.02	-0.02	0.02	-0.02	0.02
Total	99.41	0.34	97.14	0.25	97.58	0.14	97.05	0.46	96.89	0.35
P^{5+} *apfu*	0.002	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.011	0.003
Nb^{5+}	0.000	0.000	0.007	0.003	0.001	0.002	0.000	0.001	0.018	0.007
Ta^{5+}	0.000	0.001	0.001	0.002	0.000	0.000	0.001	0.001	0.045	0.035
Si^{4+}	2.936	0.010	2.717	0.043	2.843	0.010	2.818	0.117	2.654	0.124
Ti^{4+}	0.008	0.001	0.070	0.020	0.069	0.005	0.021	0.038	0.003	0.001
B^{3+}	1.000	0.000	1.000	0.000	1.000	0.000	1.000	0.000	1.000	0.000
Al^{3+}	7.025	0.011	6.966	0.027	6.722	0.007	6.774	0.061	6.851	0.057
Fe^{3+}	0.026	0.001	0.016	0.010	0.071	0.001	0.069	0.009	0.077	0.008
As^{3+}	0.003	0.001	0.103	0.030	0.109	0.006	0.141	0.090	0.176	0.068
Sb^{3+}	0.000	0.000	0.028	0.003	0.025	0.002	0.023	0.014	0.043	0.017
Bi^{3+}	0.001	0.001	0.028	0.006	0.001	0.001	0.000	0.000	0.004	0.002
Mg^{2+}	0.001	0.000	0.012	0.029	0.124	0.001	0.121	0.027	0.032	0.008
F^{-}	0.028	0.023	0.023	0.017	0.021	0.017	0.018	0.018	0.014	0.011
O^{2-}	17.967	0.023	17.819	0.039	17.843	0.016	17.818	0.107	17.762	0.080
As + Sb + Bi	0.004	0.001	0.172	0.026	0.135	0.006	0.164	0.103	0.212	0.101
Ta + Nb + Ti	0.008	0.001	0.079	0.024	0.070	0.006	0.023	0.038	0.066	0.051

The data were acquired with an electron microprobe. The formulae were calculated on the basis of 18 (O + F + As + Sb) per formula unit assuming 1 B *pfu*. We sought Na, K, Ca, Sc and Mn, but did not detect these above 0.010 *apfu*.

A graph of Si + P *versus* As + Sb + Bi for the crystals in the samples and for the structure crystals (Fig. 4) shows the same inverse relationship seen in holtite, which suggests that if the site hosting As + Sb + Bi is occupied, the adjacent Si position is vacant. The greater inhomogeneity of the bulk *versus* structure crystals is readily seen; points from the structure crystal of D21 (half-shaded squares in Fig. 4) fall in the middle of the range for the bulk (fully shaded squares), and follow the same trend. The graph also shows that most of the compositions, except perhaps those for sample D21, lie to the left of the 1:1 line. This suggests a presence of some Al at the Si sites; we note that Alexander *et al.* (1986) reported up to 0.15 *apfu* ^{IV}Al substituting for Si in some samples of Fe- and Ti-poor dumortierite, and Fuchs *et al.* (2005) reported a similar content of 0.15–0.17 *apfu* ^{IV}Al in two samples of dumortierite from Lower Austria.

Sample D27 also contains 0.94 wt.% TiO_2, or 0.07 Ti *pfu*, sample D21 (structure crystal) shows 0.82 wt.% MgO, or 0.12 Mg *pfu*, and samples D21 and D31 contain 0.92 and 1.10 wt.% Fe_2O_3, corresponding to 0.07 and 0.08 Fe *pfu*, respectively.

Normalization of the analytical results based on 18 (O + F + As + Sb) per formula unit, although it accounts for oxygen vacancies due to (As, Sb) substitution for Si, can lead to excess octahedrally coordinated cations and low Al/Si values compared with the single-crystal structure refinement. In some cases, this is due to the presence of OH in the structure, which cannot be estimated consistently in every case because of the many cation substitutions possible. For dumortierite, normalization based on the combined total occupancy of octahedrally and tetrahedrally coordinated cations from the crystal-structure refinement, or equal to 9.75 *apfu* for samples where structural data are unavailable

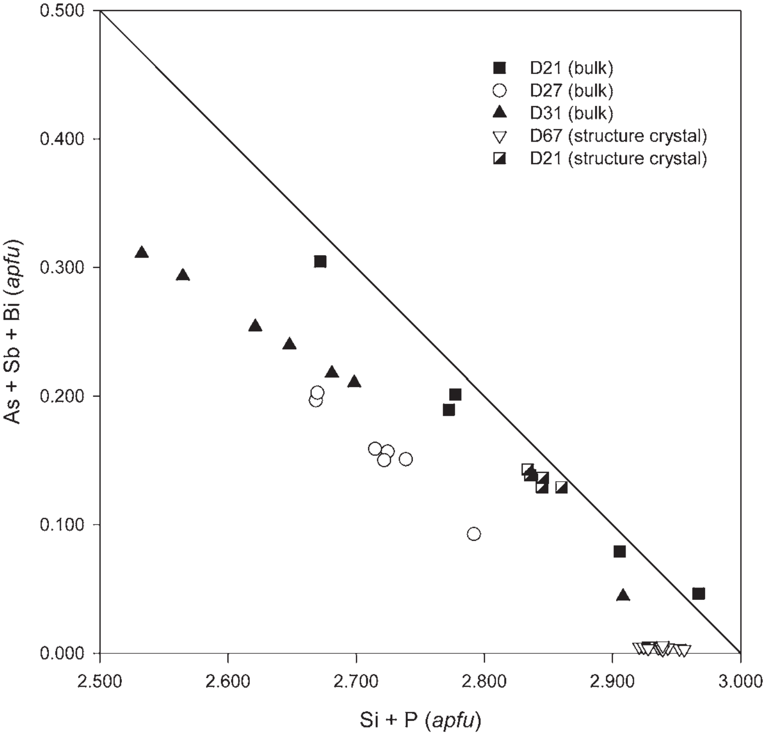

FIG. 4. Si + P *versus* As + Sb + Bi (*apfu*) for samples D21, D27, D31, and D67.

(assuming a total Al1 occupancy of 0.75), can result in better agreement if the deficit in tetrahedrally coordinated cations [= 3 – (Si + P + As + Sb)] is assumed to be ^{IV}Al. However, we have found that this scheme does not work well for holtite, as it overestimates the anion totals. As the demarcation between (As, Sb)- or (Ta, Nb, Ti)-rich dumortierite and holtite is unclear, the current scheme is preferred.

Crystal-structure refinements

Data measurement and refinement information are listed in Table 2, atomic parameters in Table 3, atomic displacement parameters in Table 4, and interatomic distances in Table 5. A table of structure factors and a cif file for each crystal refined are available from the Depository of Unpublished Data on the Mineralogical Association of Canada website [document Dumortierite CM50_855].

The final R_1 values were 0.0176, 0.0285, 0.0185, and 0.0161 for samples D67, D27, D21, and D31, respectively. The occupancies of the Al1a, Al1, and Al1b positions were 0.30, 0.42, and 0.12 for sample D67, 0.186, 0.44, and 0.186 for sample D27, 0.264, 0.424, and 0.178 for sample D21, and 0.26, 0.482, and 0.14 for sample D31. The Al1a, Al1 and Al1b occupancies were allowed to vary independently for all samples except D27, where Al1a and Al1b were constrained to be equal. The U_{eq} values range from 0.0064 to 0.0078 Å^2. Although the distance from an Al1a, Al1, and Al1b position to an individual O site differs greatly, the <Al1a–O>, <Al1–O>, and <Al1b–O> distances, with ranges over the four samples of 1.907–1.939 Å, 1.90–1.923 Å, and 1.91–1.94 Å, respectively, are much more uniform. The Al1–Al1a distances vary from 0.30 to 0.36 Å, and the Al1–Al1b distances from 0.24 to 0.37 Å.

The occupancies of the Al2 sites range from 0.977 to 0.990, those of the Al3 positions from 0.981 to 0.990, and those of the Al4 sites from 0.974 to 0.992. The <Al2–O> distances vary from 1.9000 to 1.9034 Å, the <Al3–O> values are 1.8995–1.9025 Å, and the <Al4–O> distances range from 1.8948 to 1.9005 Å.

Occupancy of the Si1 site varies from 1.0 (D67) to 0.9592 (D31), and <Si1–O> varies in the range 1.643–1.645 Å. The occupancy of the As1 position is 0.014, 0.0155, and 0.0163 in samples D27, D21, and D31, respectively, and <As1–O> is 1.82, 1.72, and 1.70 Å for the same samples. The Sb1 site occupancy is 0.0034, 0.0039, and 0.0041, and the <Sb1–O> distances

TABLE 2. DUMORTIERITE: DATA MEASUREMENT AND REFINEMENT INFORMATION

	D67 Larsemann Hills, Antarctica	D27 Il'men Mountains, Russia	D21 Hartmannsdorf quarry, Saxony, Germany	D31 Tonagh Island, Antarctica
a (Å)	4.6995(1)	4.6901(3)	4.6971(1)	4.6871(2)
b (Å)	11.7790(4)	11.7874(6)	11.8149(3)	11.7901(5)
c (Å)	20.1671(7)	20.187(1)	20.2267(5)	20.1825(8)
V (Å^3)	1116.36(6)	1116.0(1)	1122.50(5)	1115.31(8)
Space group	*Pnma*	*Pnma*	*Pnma*	*Pnma*
Z	4	4	4	4
Crystal size (mm)	0.13 × 0.13 × 0.13	0.15 × 0.15 × 0.15	0.13 × 0.17 × 0.15	0.15 × 0.15 × 0.15
Radiation	Mo*K*α	Mo*K*α	Mo*K*α	Mo*K*α
Monochromator	Graphite	Graphite	Graphite	Graphite
T (K)	293	100	293	293
Total F_o	19985	19522	16322	7059
Unique F_o	2209	1453	2245	1448
$F_o > 4\sigma F_o$	1978	1281	2036	1342
R_{int}	0.03(1)	0.03(2)	0.03(2)	0.02(1)
L.s. parameters	151	169	164	164
Range of *h*	–7 → 7	–6 → 6	–7 → 7	–5 → 6
Range of *k*	–18 → 13	–15 → 15	–18 → 18	–15 → 15
Range of *l*	–31 → 31	–26 → 26	–18 → 31	–26 → 26
R_1 for $F_o > 4\sigma F_o$	0.0176	0.0285	0.0185	0.0161
R_1 for all unique F_o	0.0222	0.0371	0.0214	0.0186
wR_2	0.0419	0.0733	0.0477	0.0415
a	0.0214	0.0375	0.0290	0.0243
b	0.37	2.47	0.18	0.38
GooF (= *S*)	1.052	1.106	1.044	1.067
$\Delta\rho_{max}$ (e Å^{-3})	0.43	1.21	0.42	0.51
$\Delta\rho_{min}$ (e Å^{-3})	–0.37	–0.36	–0.44	–0.28

$w = 1/[s^2(F_o^2) + (a \times P)^2 + b \times P]$ where $P = [\mathrm{Max}\,(F_o^2, 0) + 2 \times F_c^2)]/3$.

are 1.95, 1.93, and 1.90 Å for samples D27, D21, and D31, respectively.

Occupancy of the Si2 position ranges from 1.0 (D67) to 0.958 (D31). The <Si2–O> distance for D67, D21, and D31 is virtually the same at 1.6408, 1.6402, and 1.640 Å, but slightly larger (1.643 Å) for D27. The occupancy of the As2 site is 0.030, 0.0311, and 0.033 for samples D27, D21, and D31, respectively, but <As2–O> is considerably longer (1.91 Å) for D27 than for D21 and D31 (1.77 and 1.76 Å). The occupancy of the Sb2 site is 0.0075, 0.0078, and 0.0083, and <Sb2–O> is 2.00, 1.96, and 1.97 Å for samples D27, D21, and D31, respectively. The <B–O> distance ranges from 1.359 to 1.362 Å in the four samples.

The occupancy of the O2 and O7 positions is 0.966, 0.961, and 0.958 for D27, D21, and D31, respectively. For sample D27, the occupancies of the O1 and O1a sites are 0.962 and 0.038, respectively, and those for the O6 and O6a positions are 0.969 and 0.031.

DISCUSSION

Compositions

A graph of Si + P *apfu versus* As + Sb + Bi *apfu* (Fig. 5) for 340 dumortierite and 627 holtite compositions (representing approximately 36 world localities for dumortierite and four for holtite) shows that most of the compositions lie to the left of the 1:1 line, which suggests that some Al for Si substitution at the Si sites is common in dumortierite and holtite. The graph also shows no significant gap between the compositional fields for dumortierite and holtite.

A graph of Sb *versus* As (Fig. 6) for the same number of compositions and localities as in Figure 5 shows that for the majority of dumortierite compositions, As is greater than Sb. We note that the three dumortierite compositions with Sb > 0.250 Sb *pfu* are from Cempírek *et al.* (2010), and two of these compositions plot in the holtite field in Figure 5. All dumortierite compositions with > 0.027 Sb *pfu* and no As are from Borghi *et al.* (2004) and Vaggelli *et al.* (2004), who did not measure As. All other dumortierite compositions where Sb exceeds As by more than 0.002 *apfu* were obtained from quartzite in sample D51, donated by G. Vaggelli, thus presumably from the same area, if not the same locality, as the samples from Mozambique measured by Borghi *et al.* (2004) and Vaggelli *et al.* (2004).

Figure 6 also shows that Sb exceeds As for the majority of holtite compositions; the exceptions are those from Virorco, San Luis, Argentina (Galliski *et al.* 2012), which have elevated As and low Sb contents.

TABLE 3. DUMORTIERITE: ATOMIC PARAMETERS

		D67	D27	D21	D31
Al1a	*x*	0.348(2)	0.332(4)	0.333(2)	0.335(3)
	y	¾	¾	¾	¾
	z	0.2493(1)	0.2494(4)	0.25050(9)	0.2503(1)
	U_{eq}	0.0064(2)	0.0074(5)	0.0078(2)	0.0071(3)
	n	0.15(1)	0.093(7)	0.132(6)	0.13(1)
Al1	*x*	0.413(6)	0.3954(8)	0.410(3)	0.412(4)
	y	¾	¾	¾	¾
	z	0.2501(1)	0.2498(2)	0.24960(8)	0.24963(9)
	U_{eq}	0.0064(2)	0.0074(5)	0.0078(2)	0.0071(3)
	n	0.42(3)	0.43(3)	0.423(7)	0.48(1)
Al1b	*x*	0.465(9)	0.475(4)	0.480(3)	0.487(6)
	y	¾	¾	¾	¾
	z	0.2506(3)	0.2503(4)	0.2493(1)	0.2495(2)
	U_{eq}	0.0064(2)	0.0074(5)	0.0078(2)	0.0071(3)
	n	0.06(2)	0.093(7)	0.089(8)	0.07(1)
Al2	*x*	0.55801(6)	0.5580(2)	0.55788(5)	0.55802(7)
	y	0.61028(2)	0.61038(6)	0.61057(2)	0.61051(3)
	z	0.47236(1)	0.47240(3)	0.47255(1)	0.47248(2)
	U_{eq}	0.00453(8)	0.0049(2)	0.00549(8)	0.0051(1)
	n	0.983(2)	0.977(4)	0.989(2)	0.990(2)
Al3	*x*	0.05978(5)	0.0598(2)	0.05966(5)	0.05971(7)
	y	0.49104(2)	0.49106(6)	0.49113(2)	0.49106(3)
	z	0.43084(1)	0.43092(3)	0.43113(1)	0.43101(2)
	U_{eq}	0.00445(8)	0.0050(2)	0.00559(8)	0.0052(1)
	n	0.990(2)	0.981(4)	0.989(2)	0.990(2)
Al4	*x*	0.05696(6)	0.0580(2)	0.05850(6)	0.05802(8)
	y	0.35809(2)	0.35854(6)	0.35910(2)	0.35872(3)
	z	0.28856(1)	0.28907(3)	0.28931(1)	0.28907(2)
	U_{eq}	0.00589(8)	0.0065(2)	0.00748(8)	0.0066(1)
	n	0.992(2)	0.974(4)	0.989(2)	0.984(2)
Si1	*x*	0.08747(7)	0.0879(3)	0.0871(4)	0.087(1)
	y	¾	¾	¾	¾
	z	0.40497(2)	0.40474(8)	0.40487(7)	0.4045(4)
	U_{eq}	0.00563(7)	0.0056(3)	0.0073(2)	0.0068(7)
	n	1.0	0.966(3)	0.961(1)	0.959(2)
As1	*x*	-	0.118(6)	0.108(5)	0.10(1)
	y	-	¾	¾	¾
	z	-	0.388(1)	0.397(1)	0.399(5)
	U_{eq}	-	0.008(5)	0.007(2)	0.002(4)
	n	-	0.027(3)	0.031(1)	0.033(1)
Sb1	*x*	-	0.057(8)	0.117(3)	0.113(5)
	y	-	¾	¾	¾
	z	-	0.358(3)	0.3789(6)	0.3802(9)
	U_{eq}	-	0.008(5)	0.007(2)	0.002(4)
	n	-	0.0069(7)	0.0077(2)	0.0083(3)
Si2	*x*	0.58740(5)	0.5883(2)	0.5874(1)	0.5875(3)
	y	0.52501(2)	0.5257(1)	0.52488(5)	0.52556(8)
	z	0.32792(1)	0.32776(5)	0.32822(3)	0.32794(6)
	U_{eq}	0.00561(6)	0.0057(2)	0.0071(1)	0.0063(2)
	n	1.0	0.962(2)	0.961(1)	0.958(1)
As2	*x*	-	0.616(2)	0.607(2)	0.608(4)
	y	-	0.563(2)	0.5443(7)	0.542(1)
	z	-	0.3158(6)	0.3218(5)	0.3214(9)
	U_{eq}	-	0.0065	0.006(1)	0.006(2)
	n	-	0.030(2)	0.0311(8)	0.033(1)
Sb2	*x*	-	0.555(6)	0.610(2)	0.607(3)
	y	-	0.597(2)	0.5700(8)	0.573(1)
	z	-	0.306(1)	0.3143(5)	0.3153(7)
	U_{eq}	-	0.0065	0.006(1)	0.006(2)
	n	-	0.0075(4)	0.0078(2)	0.0083(3)
B	*x*	0.2254(3)	0.2267(8)	0.2254(3)	0.2261(4)
	y	¼	¼	¼	¼
	z	0.41616(7)	0.4162(2)	0.41611(6)	0.41615(9)
	U_{eq}	0.0056(2)	0.0073(7)	0.0067(2)	0.0067(3)
O1	*x*	0.3769(2)	0.3775(5)	0.3767(2)	0.3768(3)
	y	¾	¾	¾	¾
	z	0.45354(5)	0.4541(1)	0.45439(4)	0.45417(6)
	U_{eq}	0.0058(2)	0.0050(5)	0.0078(2)	0.0072(2)
O1a	*x*	-	0.41(1)	-	-
	y	-	¾	-	-
	z	-	0.398(3)	-	-
	U_{eq}	-	0.005	-	-
	n		0.038(5)		
O2	*x*	0.1477(2)	0.1498(6)	0.1510(2)	0.1500(3)
	y	¾	¾	¾	¾
	z	0.32501(5)	0.3254(1)	0.32637(5)	0.32577(6)
	U_{eq}	0.0107(2)	0.0123(6)	0.0130(2)	0.0128(3)
	n	1.0	0.966(3)	0.961(1)	0.959(2)
O3	*x*	0.8959(1)	0.8960(4)	0.8957(1)	0.8957(2)
	y	0.63937(5)	0.6392(1)	0.63925(5)	0.63919(7)
	z	0.42397(3)	0.42401(7)	0.42440(3)	0.42411(4)
	U_{eq}	0.0055(1)	0.0064(3)	0.0067(1)	0.0061(2)
O4	*x*	0.3991(1)	0.4000(3)	0.4016(1)	0.4005(2)
	y	0.43670(6)	0.4361(1)	0.43581(5)	0.43611(7)
	z	0.28250(3)	0.28232(7)	0.28267(3)	0.28251(4)
	U_{eq}	0.0058(1)	0.0063(3)	0.0072(1)	0.0063(2)
O5	*x*	0.3953(1)	0.3953(4)	0.3952(1)	0.3951(2)
	y	0.55070(5)	0.5506(1)	0.55015(5)	0.55028(7)
	z	0.39328(3)	0.39335(7)	0.39349(3)	0.39341(4)
	U_{eq}	0.0055(1)	0.0064(3)	0.0066(1)	0.0060(2)
O6	*x*	0.8809(1)	0.8813(4)	0.8807(1)	0.8808(2)
	y	0.45479(6)	0.4537(1)	0.45381(5)	0.45396(7)
	z	0.34985(3)	0.35027(8)	0.35051(3)	0.35023(4)
	U_{eq}	0.0067(1)	0.0054(4)	0.0080(1)	0.0071(2)
O6a	*x*	-	0.90(1)	-	-
	y	-	0.510(5)	-	-
	z	-	0.323(3)	-	-
	U_{eq}	-	0.005	-	-
	n		0.031(4)		
O7	*x*	0.6464(2)	0.6478(4)	0.6489(2)	0.6481(2)
	y	0.64080(6)	0.6401(2)	0.63881(6)	0.63972(8)
	z	0.28635(3)	0.28647(9)	0.28694(3)	0.28660(4)
	U_{eq}	0.0108(1)	0.0118(4)	0.0134(2)	0.0128(2)
	n	1.0	0.966(3)	0.961(1)	0.958(1)
O8	*x*	0.1605(2)	0.1674(6)	0.1639(2)	0.1641(3)
	y	¼	¼	¼	¼
	z	0.35081(4)	0.3505(1)	0.35054(4)	0.35051(6)
	U_{eq}	0.0072(2)	0.0089(5)	0.0090(2)	0.0087(3)
O9	*x*	0.2552(1)	0.2545(4)	0.2545(1)	0.2547(2)
	y	0.35121(5)	0.3511(1)	0.35110(5)	0.35117(7)
	z	0.44821(3)	0.44822(7)	0.44802(3)	0.44805(4)
	U_{eq}	0.0062(1)	0.0072(3)	0.0073(1)	0.0070(2)
O10	*x*	0.7607(2)	0.7598(5)	0.7608(2)	0.7611(3)
	y	¼	¼	¼	¼
	z	0.27190(5)	0.2723(1)	0.27239(4)	0.27224(6)
	U_{eq}	0.0070(2)	0.0074(5)	0.0092(2)	0.0081(2)
O11	*x*	0.7504(1)	0.7503(3)	0.7503(1)	0.7501(2)
	y	0.46621(5)	0.4663(1)	0.46648(5)	0.46640(6)
	z	0.48795(3)	0.48804(7)	0.48806(3)	0.48808(4)
	U_{eq}	0.0046(1)	0.0048(3)	0.0057(1)	0.0049(2)

Values of U_{eq} are quoted in Å^2.

Al1 sites

Evans *et al.* (2012) obtained occupancies of 0.23, 0.28, and 0.23 for Al1, Al1a, and Al1b, respectively, for a sample of metamorphic dumortierite (D34) from Madagascar. They suggested that the splitting of Al1 into three sites is likely due to a local order of vacancies in the structural channels. As the occupancies of their three sites were approximately equal at ~0.25 and total approximately 0.75, they suggested that in each structural channel, the Al sites are ordered as

...–☐–Al1a–Al1–Al1b–☐–Al1a–Al1–Al1b–☐–...

moving along the **a** direction. This allows an increase of the minimum Al–Al distance in the channel from ~2.35 Å in adjacent undistorted Al1 sites to ~2.55 Å. Evans *et al.* (2012) explained that whereas this order of Al1 sites occurs in individual channels, the structure from channel to channel remains disordered, as there are no indications of a duplicated unit-cell along the **a** direction. For samples D67, D27, D21, D27, the totals of the occupancies of the Al1 sites are 0.84, 0.81, 0.866, and

TABLE 4. DUMORTIERITE: ATOMIC DISPLACEMENT PARAMETERS

		D67	D27	D21	D31
Al2	U_{11}	0.0045(1)	0.0050(4)	0.0042(1)	0.0046(2)
	U_{22}	0.0042(1)	0.0046(4)	0.0057(1)	0.0053(2)
	U_{33}	0.0049(1)	0.0051(3)	0.0066(1)	0.0053(2)
	U_{12}	-0.00018(9)	0.0001(2)	-0.00013(8)	-0.0002(1)
	U_{13}	-0.00027(8)	-0.0002(2)	-0.00032(8)	-0.0003(1)
	U_{23}	-0.00015(9)	-0.0003(2)	-0.00009(8)	-0.0001(1)
Al3	U_{11}	0.0042(1)	0.0049(4)	0.0040(1)	0.0046(2)
	U_{22}	0.0047(1)	0.0048(4)	0.0064(1)	0.0060(2)
	U_{33}	0.0045(1)	0.0052(3)	0.0064(1)	0.0049(2)
	U_{12}	0.00005(8)	0.0000(3)	0.00010(8)	0.0001(1)
	U_{13}	-0.00011(8)	0.0001(2)	-0.00006(8)	0.0001(1)
	U_{23}	-0.00012(9)	0.0000(2)	-0.00015(8)	-0.0001(1)
Al4	U_{11}	0.0063(1)	0.0074(4)	0.0069(1)	0.0071(2)
	U_{22}	0.0056(1)	0.0059(3)	0.0076(1)	0.0067(12)
	U_{33}	0.0057(1)	0.0061(4)	0.0079(1)	0.0061(2)
	U_{12}	-0.00009(9)	0.0004(3)	0.00006(8)	-0.0001(1)
	U_{13}	-0.00049(9)	-0.0006(3)	-0.00076(9)	-0.0008(1)
	U_{23}	0.00120(9)	0.0009(2)	0.00137(8)	0.0011(1)
Si1	U_{11}	0.0045(1)	0.0036(5)	0.0035(3)	0.004(1)
	U_{22}	0.0041(2)	0.0035(5)	0.0051(2)	0.0054(5)
	U_{33}	0.0083(2)	0.0096(6)	0.0133(5)	0.011(2)
	U_{12}	0	0	0	0
	U_{13}	-0.0003(1)	-0.0001(5)	-0.0001(3)	-0.0003(8)
	U_{23}	0	0	0	0
Si2	U_{11}	0.0050(1)	0.0047(3)	0.0043(2)	0.0046(3)
	U_{22}	0.0066(1)	0.0071(4)	0.0100(2)	0.0091(5)
	U_{33}	0.0052(1)	0.0052(3)	0.0069(2)	0.0051(3)
	U_{12}	0.00005(8)	0.0002(3)	-0.0001(2)	-0.0002(3)
	U_{13}	0.00006(8)	0.0001(2)	0.0001(1)	0.0003(2)
	U_{23}	-0.00083(8)	-0.0017(3)	-0.0023(1)	-0.0019(3)
B	U_{11}	0.0053(5)	0.009(2)	0.0063(5)	0.0066(8)
	U_{22}	0.0059(6)	0.005(2)	0.0071(5)	0.0067(8)
	U_{33}	0.0057(5)	0.009(2)	0.0067(4)	0.0067(8)
	U_{12}	0	0	0	0
	U_{13}	-0.0001(4)	0.001(1)	-0.0001(4)	0.0000(6)
	U_{23}	0	0	0	0
O1	U_{11}	0.0051(4)	0.004(1)	0.0055(3)	0.0058(6)
	U_{22}	0.0042(4)	0.003(1)	0.0061(3)	0.0059(5)
	U_{33}	0.0082(4)	0.008(1)	0.0117(4)	0.0099(5)
	U_{12}	0	0	0	0
	U_{13}	-0.0013(3)	-0.0005(9)	-0.0013(3)	-0.0011(4)
	U_{23}	0	0	0	0
O2	U_{11}	0.0111(4)	0.013(1)	0.0125(4)	0.0135(7)
	U_{22}	0.0129(5)	0.013(1)	0.0126(4)	0.0129(6)
	U_{33}	0.0080(4)	0.011(1)	0.0138(4)	0.0118(6)
	U_{12}	0	0	0	0
	U_{13}	0.0015(3)	0.003(1)	0.0045(4)	0.0042(5)
	U_{23}	0	0	0	0
O3	U_{11}	0.0047(3)	0.0064(8)	0.0046(2)	0.0055(4)
	U_{22}	0.0044(3)	0.0059(7)	0.0065(2)	0.0057(4)
	U_{33}	0.0074(3)	0.0068(7)	0.0091(2)	0.0070(4)
	U_{12}	-0.0003(2)	0.0000(6)	-0.0001(2)	0.0001(3)
	U_{13}	0.0003(2)	0.0006(6)	0.0006(2)	0.0005(3)
	U_{23}	-0.0002(2)	-0.0007(6)	0.0004(2)	-0.0004(3)
O4	U_{11}	0.0051(3)	0.0065(7)	0.0052(3)	0.0051(4)
	U_{22}	0.0067(3)	0.0056(7)	0.0087(3)	0.0078(4)
	U_{33}	0.0056(3)	0.0068(7)	0.0078(2)	0.0060(4)
	U_{12}	0.0000(2)	0.0008(6)	-0.0005(2)	0.0001(3)
	U_{13}	0.0002(2)	0.0003(6)	0.0003(2)	0.0006(3)
	U_{23}	-0.0009(2)	-0.0004(6)	-0.0013(2)	-0.0009(3)
O5	U_{11}	0.0052(3)	0.0068(8)	0.0049(2)	0.0053(4)
	U_{22}	0.0064(3)	0.0060(7)	0.0082(2)	0.0076(4)
	U_{33}	0.0050(3)	0.0063(7)	0.0068(2)	0.0052(3)
	U_{12}	-0.0008(2)	0.0001(6)	-0.0006(2)	-0.0006(3)
	U_{13}	0.0000(2)	-0.0002(6)	0.0003(2)	-0.0001(3)
	U_{23}	-0.0009(2)	-0.0001(6)	-0.0007(2)	-0.0008(3)
O6	U_{11}	0.0057(3)	0.0047(8)	0.0057(3)	0.0056(4)
	U_{22}	0.0075(3)	0.0061(8)	0.0103(3)	0.0093(4)
	U_{33}	0.0057(3)	0.0052(7)	0.0079(2)	0.0064(4)
	U_{12}	0.0009(2)	-0.0001(6)	0.0008(2)	0.0009(3)
	U_{13}	-0.0005(2)	0.0000(6)	-0.0010(2)	-0.0006(3)
	U_{23}	-0.0016(2)	-0.0011(6)	-0.0019(2)	-0.0017(3)
O7	U_{11}	0.0122(3)	0.0136(9)	0.0137(3)	0.0157(5)
	U_{22}	0.0085(3)	0.0104(9)	0.0130(3)	0.0116(5)
	U_{33}	0.0117(3)	0.0114(8)	0.0134(3)	0.0110(4)
	U_{12}	-0.0012(2)	-0.0019(8)	-0.0039(2)	-0.0025(4)
	U_{13}	0.0011(2)	0.0006(8)	0.0021(3)	0.0014(4)
	U_{23}	0.0020(2)	0.0010(7)	0.0006(2)	0.0012(4)
O8	U_{11}	0.0128(4)	0.016(1)	0.0137(4)	0.0144(6)
	U_{22}	0.0044(4)	0.005(1)	0.0069(4)	0.0062(5)
	U_{33}	0.0044(4)	0.006(1)	0.0063(3)	0.0055(5)
	U_{12}	0	0	0	0
	U_{13}	-0.0024(3)	-0.0014(9)	-0.0019(3)	-0.0018(4)
	U_{23}	0	0	0	0
O9	U_{11}	0.0083(3)	0.0104(8)	0.0081(3)	0.0088(4)
	U_{22}	0.0044(3)	0.0052(7)	0.0062(2)	0.0059(4)
	U_{33}	0.0059(3)	0.0059(7)	0.0077(2)	0.0062(4)
	U_{12}	0.0006(2)	-0.0003(6)	0.0004(2)	0.0004(3)
	U_{13}	-0.0018(2)	-0.0008(6)	-0.0016(2)	-0.0017(3)
	U_{23}	-0.0007(2)	-0.0012(6)	-0.0007(2)	-0.0010(3)
O10	U_{11}	0.0059(4)	0.005(1)	0.0063(4)	0.0064(6)
	U_{22}	0.0057(4)	0.006(1)	0.0084(4)	0.0072(6)
	U_{33}	0.0093(4)	0.011(1)	0.0128(4)	0.0109(5)
	U_{12}	0	0	0	0
	U_{13}	-0.0006(3)	0.0003(9)	-0.0012(3)	-0.0003(4)
	U_{23}	0	0	0	0
O11	U_{11}	0.0044(3)	0.0036(7)	0.0042(3)	0.0040(4)
	U_{22}	0.0044(3)	0.0055(7)	0.0063(2)	0.0058(4)
	U_{33}	0.0050(3)	0.0052(7)	0.0066(2)	0.0048(4)
	U_{12}	-0.0001(2)	0.0000(6)	-0.0003(2)	-0.0002(3)
	U_{13}	-0.0001(2)	-0.0001(5)	0.0001(2)	0.0001(3)
	U_{23}	-0.0001(2)	-0.0002(5)	0.0001(2)	0.0000(3)

Values of the displacement parameters are quoted in Å^2.

0.88, respectively, which suggests a more disordered situation, with fewer vacancies and at least some longer repeat-units, than in sample D34 of Evans *et al.* (2012). In addition, in samples D67, 21, and 31 the occupancies of the Al1 sites are very different, with Al1 > Al1a > Al1b (in sample D27, Al1 > Al1a = Al1b).

The unequal Al1a and Al1b occupancies are puzzling. One possibility is that Al1a is preferentially occupied by stronger scatterers, although there are only minor amounts in these samples. Another possibility is that the channels contain a disordered mix of dimers, trimers and longer sequences that are arranged in such a way that they have a slightly polar character. The Al1a site is one with a vacancy above and an occupied site below, whereas the Al1b site has an occupied site above and a vacancy below. The undistorted Al1 site corresponds to a site where the site above and the site below are equivalent, either both occupied or both vacant. Thus a structure with either a large number of isolated occupied Al1 sites, or many long (> 3) chains of occupied sites, would have occupancies of Al1 > A1a = Al1b, resulting in chains like

...–□–Al1a–Al1…Al1–Al1b–□–...

The meaning of Al1a > Al1b occupancies is not clear. One possibility is the existence of dimers where the upper cation is shifted disproportionately more than the lower cation, resulting in pairs that resemble Al1a–Al1 more than Al1a–Al1b.

Similar schemes of order–disorder have been proposed for the Al1 chain of face-sharing octahedra previously. Platonov *et al.* (2000) attributed the different energies of the adsorption bands responsible for the colors of red and blue dumortierite to different

TABLE 5. DUMORTIERITE: INTERATOMIC DISTANCES (Å)

		D67	D27	D21	D31
Al1a-O2		1.793(6)	1.76(1)	1.757(5)	1.752(8)
Al1a-O2A		2.057(7)	2.12(1)	2.155(6)	2.13(1)
Al1a-O7B,C	× 2	1.752(6)	1.72(1)	1.747(4)	1.736(7)
Al1a-O7,D	× 2	2.044(8)	2.10(1)	2.113(6)	2.093(9)
<Al1a-O>		1.907	1.92	1.939	1.92
Al1-O2		1.96(2)	1.912(5)	1.972(9)	1.97(1)
Al1-O2A		1.88(2)	1.930(5)	1.909(9)	1.89(1)
Al1-O7B,C	× 2	1.94(2)	1.888(4)	1.943(9)	1.94(1)
Al1-O7,D	× 2	1.84(2)	1.905(3)	1.886(8)	1.86(1)
<Al1-O>		1.90	1.905	1.923	1.91
Al1b-O2		2.11(3)	2.15(1)	2.19(1)	2.20(2)
Al1b-O2A		1.75(2)	1.74(1)	1.731(8)	1.70(1)
Al1b-O7B,C	× 2	2.11(3)	2.14(1)	2.16(1)	2.18(2)
Al1b-O7,D	× 2	1.70(2)	1.69(1)	1.714(7)	1.68(1)
<Al1b-O>		1.91	1.93	1.94	1.94
Al1-Al1a		0.30(2)	0.30(2)	0.360(7)	0.36(1)
Al1-Al1b		0.24(2)	0.37(2)	0.326(5)	0.35(1)
Al2-O1		1.8914(5)	1.887(1)	1.8902(5)	1.8876(7)
Al2-O3		1.8949(7)	1.892(2)	1.8925(7)	1.8901(9)
Al2-O5		1.9029(7)	1.904(2)	1.9107(6)	1.9064(9)
Al2-O9E		1.8821(7)	1.884(2)	1.8876(6)	1.8834(9)
Al2-O11E		1.8849(7)	1.883(2)	1.8864(6)	1.8819(9)
Al2-O11		1.9483(7)	1.949(2)	1.9527(6)	1.9485(9)
<Al2-O>		1.9008	1.900	1.9034	1.9000
Al3-O3F		1.9144(7)	1.913(1)	1.9168(6)	1.9132(9)
Al3-O5		1.8850(7)	1.882(2)	1.8843(7)	1.8801(9)
Al3-O6F		1.8860(7)	1.883(2)	1.8868(6)	1.8847(9)
Al3-O9		1.9181(7)	1.918(2)	1.9214(6)	1.9166(9)
Al3-O11F		1.8778(7)	1.877(2)	1.8769(6)	1.8754(9)
Al3-O11E		1.9317(7)	1.929(2)	1.9287(6)	1.9268(8)
<Al3-O>		1.9022	1.900	1.9025	1.8995
Al4-O4		1.8595(7)	1.851(2)	1.8536(7)	1.8512(9)
Al4-O4B		1.8605(7)	1.860(2)	1.8666(6)	1.8614(9)
Al4-O6F		1.8736(7)	1.863(2)	1.8661(7)	1.8640(9)
Al4-O8		1.8530(7)	1.854(2)	1.8543(6)	1.8514(9)
Al4-O10F		1.9162(7)	1.926(2)	1.9323(7)	1.923(1)
Al4-O10B		2.0062(8)	2.017(2)	2.0302(7)	2.020(1)
<Al4-O>		1.8948	1.895	1.9005	1.895
Si1-O1		1.676(1)	1.685(3)	1.690(2)	1.686(8)
Si1-O2		1.637(1)	1.628(3)	1.616(3)	1.617(8)
Si1-O3F,G	× 2	1.6296(7)	1.634(2)	1.636(1)	1.634(4)
<Si1-O>		1.643	1.645	1.645	1.643
As1-O1		-	1.81(3)	1.71(3)	1.69(8)
As1-O3F,G		-	1.83(2)	1.73(1)	1.71(4)
<As1-O>		-	1.82	1.72	1.70
Sb1-O1		-	2.45(5)	1.96(1)	1.94(2)
Sb1-O1a		-	1.82(7)	-	-
Sb1-O3F,G	× 2	-	2.01(4)	1.91(1)	1.88(1)
<Sb1-O1a,3F,G>		-	1.95	1.93	1.90
Si1-As1		-	0.38(3)	0.18(2)	0.14(9)
Si1-Sb1		-	0.95(5)	0.54(1)	0.51(2)
As1-Sb1		-	0.66(4)	0.38(3)	0.38(9)
As1-O2		-	1.26(3)	1.45(2)	1.50(9)
Sb1-O2		-	0.79(5)	1.07(1)	1.11(2)
Si2-O4		1.6444(7)	1.654(2)	1.6487(9)	1.650(2)
Si2-O5		1.6261(7)	1.630(2)	1.6267(9)	1.626(2)
Si2-O6		1.6678(7)	1.678(2)	1.6752(9)	1.675(2)
Si2-O7		1.6248(7)	1.610(3)	1.6101(9)	1.609(1)
<Si2-O>		1.6408	1.643	1.6402	1.640
As2-O4		-	1.93(1)	1.79(1)	1.76(2)
As2-O5		-	1.88(1)	1.76(1)	1.77(2)
As2-O6		-	1.92(2)	1.77(1)	1.75(2)
<As2-O>		-	1.91	1.77	1.76
Sb2-O4		-	2.08(3)	1.97(1)	2.00(1)
Sb2-O5		-	1.99(3)	1.91(1)	1.88(2)
Sb2-O6		-	2.44(3)	2.01(1)	2.03(1)
Sb2-O6a		-	1.94(6)	-	-
<Sb2-O4,5,6a>		-	2.00	1.96	1.97
Si2-As2		-	0.52(2)	0.279(8)	0.25(1)
Si2-Sb2		-	0.96(3)	0.611(9)	0.63(1)
As2-Sb2		-	0.52(3)	0.34(1)	0.39(2)
As2-O7		-	1.09(2)	1.335(8)	1.37(1)
Sb2-O7		-	1.09(2)	1.001(9)	0.99(1)
B-O8		1.353(2)	1.355(4)	1.357(1)	1.356(2)
B-O9,H	× 2	1.3633(9)	1.362(2)	1.3646(8)	1.362(1)
<B-O>		1.360	1.359	1.362	1.360

A: $x + \frac{1}{2}, y, -z + \frac{1}{2}$; B: $x - \frac{1}{2}, y, -z + \frac{1}{2}$; C: $x - \frac{1}{2}, -y + {}^3/_2, -z + \frac{1}{2}$; D: $x, -y + {}^3/_2, z$; E: $-x + 1, -y + 1, -z + 1$; F: $x - 1, y, z$; G: $x - 1, -y + {}^3/_2, z$; H: $x, -y + \frac{1}{2}, z$.

Fe^{2+}–Ti^{4+} distances in dimers of the form ...–□–Fe^{2+}–Ti^{4+}–□–... *versus* ($n \geq 3$)-mers of the form ...–□–[(n – 2) × Al]–Fe^{2+}–Ti^{4+}–□–..., with Ti^{4+} shifted more strongly off-center than Fe^{2+} in either case. Violet dumortierite, which shows both bands in its adsorption spectra, were considered to contain a mixture of dimers and longer sequences.

Si, As and Sb sites

The results show that in samples D27, D21 and D31, both SiO$_4$ tetrahedra are partially replaced by As^{3+}O$_3$ and Sb^{3+}O$_3$ triangular pyramids, with distinct sites for As^{3+} and Sb^{3+}. The <As–O> and <Sb–O> distances are typical of As^{3+} and Sb^{3+} in this coordination. In sample D27, the O1–Sb1 distance is much greater (2.45 Å) than in D21 (1.96 Å) and D31 (1.94 Å), and is effectively replaced in the coordination by the atom at the O1a position, at a distance of 1.82 Å. Similarly, O6–Sb2 in sample D27 is much greater than for samples D21 (2.01 Å) and D31 (2.03 Å), but O6a–Sb2 is 1.94 Å. We suspect that the disorder at the O1 and O6 positions is an artifact of the twinning encountered in sample D27, especially since it is not seen in the other samples.

The role of Bi^{3+} in the structure is not clear. It may occupy sites similar to the other group-V chalcophile elements, As and Sb. The fact that <As2–O> is significantly larger (1.91 Å) in sample D27 than in D21 and D31 (1.77 and 1.76 Å, respectively) may suggest the presence of Bi^{3+} (0.03 Bi *pfu* in the electron-microprobe-derived compositions) at the As2 position. However, with a large cation radius, greater than 1 Å (Shannon 1976), Bi^{3+} is likely too large for tetrahedrally coordinated or (As, Sb)-like sites, and may instead substitute for Al^{3+} at the Al1 site.

What about pentavalent As, Sb, or Bi at the As and Sb sites? This seems unlikely, given that pentavalent cations would undoubtedly require a tetrahedral coordination of O atoms. The tetrahedral As^{5+}–O distances in Ni and Co arsenate compounds with dumortierite-like structures (Marcos *et al.* 1995, Hughes *et al.* 2003) are 1.69–1.70 Å, significantly longer than the observed Si1–O2 and Si2–O7 bond lengths; hence if As^{5+} was present in dumortierite, split O2 and O7 sites should

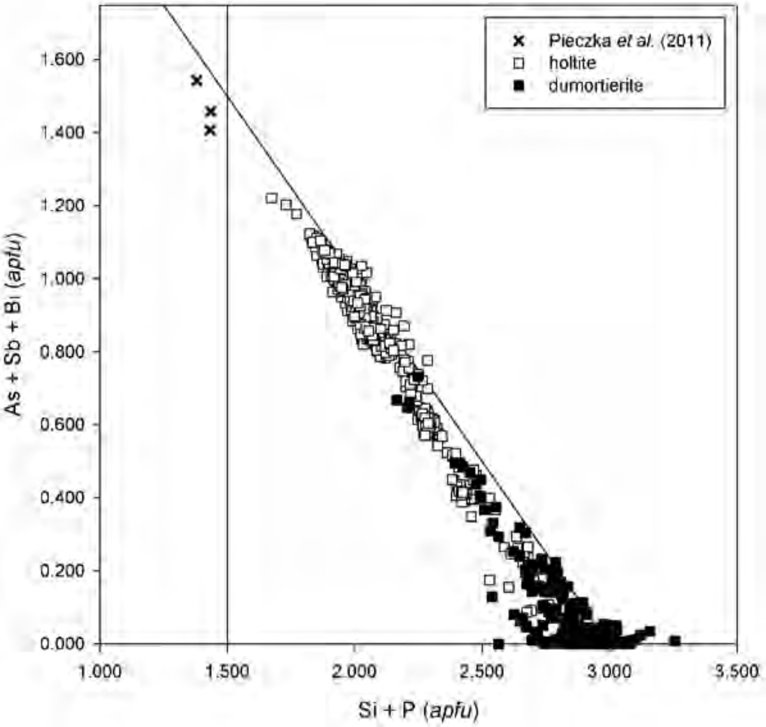

FIG. 5. Si + P *versus* As + Sb + Bi (*apfu*). The black squares represent 340 dumortierite compositions [272 unpublished data from LAG, 40 from Galliski *et al.* (2012), 12 from Borghi *et al.* (2004), seven from Cempírek & Novak (2004), five from Cempírek *et al.* (2010), and four from Vaggelli *et al.* (2004)]. The samples are from approximately 36 world localities. The white squares represent 627 holtite compositions [392 from Szklary, Lower Silesia, Poland (Pieczka *et al.* 2011), 199 from Greenbushes, Western Australia, Voron'i Tundry, Kola Peninsula, Russia and from Szklary, Lower Silesia, Poland (L.A. Groat, unpubl. data), and 36 from Virorco, San Luis, Argentina (Galliski *et al.* 2012)]. The "X" symbol represents the (Ti, Nb, Ta)-free, (As, Sb)-rich dumortierite-like phase described by Pieczka *et al.* (2011).

be observed. The lack of split O2 and O7 sites suggests that there is no As^{5+} in the structure.

Evidence from the O2 and O7 sites

The samples studied here show both the dominant vacancy substitution seen in dumortierite, $Al^{3+} + 3O^{2-} \rightarrow \square + 3OH^{-}$, and that seen in holtite, $Al^{3+} + 3(SiO)^{2+} \rightarrow \square + 3(As, Sb)^{3+}$, which could explain why there is so much disorder. Is the disorder at the Al1 and Si, As and Sb sites linked in some way? If so, we would expect to see disorder at the O2 and O7 sites, which host O atoms that coordinate Si at the Si1 and Si2 sites and cations at the Al1 site, or OH ions, which coordinate Si at the Si sites and vacancies at the Al1 position. The results show that U_{eq} for O2 is 0.0107, 0.0123, 0.0130, and 0.0128 Å^2, and U_{eq} for O7 is 0.0108, 0.0118, 0.0134, and 0.0128 Å^2 for samples D67, D27, D21, and D31, respectively. These are larger than for the other O positions, which is normal for dumortierite and holtite (because of the vacancies in the channels); the values for the As, Sb and Bi-bearing samples are higher than for sample D67, which may indicate some degree of linkage in the disorder at the Al1 and Si, As and Sb positions.

Formulae

The formula from the average electron-microprobe-derived composition of sample D67 is $(Al_{7.03}Fe_{0.03})_{\Sigma7.06}Si_{2.94}BO_{17.97}$. The formula from the crystal-structure refinement is $(Al_{6.77}\square_{0.23})_{\Sigma3}Si_3BO_{18}$, which for charge-balance considerations must include OH and becomes $(Al_{6.77}\square_{0.23})_{\Sigma3}Si_3B(O_{17.31}OH_{0.69})_{\Sigma18}$. In the dumortierite structure, OH is considered to occur in the hexagonal channel at the O2 and O7 positions (Moore & Araki 1978, Alexander *et al.* 1986, Werding & Schreyer 1990, Ferraris *et al.* 1995, Cempírek & Novák 2005, Fuchs *et al.* 2005), and at the four-coordinate O10 site (Chopin *et al.* 1995, Ferraris *et al.* 1995, Farges *et al.* 2004). The formula from the average electron-microprobe-derived composition of sample D27 is $(Al_{6.97}Ti_{0.07}Fe_{0.02}Mg_{0.01})_{\Sigma 7.07}(Si_{2.72}As_{0.10}Sb_{0.03}Bi_{0.03})_{\Sigma 2.88}B(O_{17.82}F_{0.02})_{\Sigma 17.84}$, and that from the crystal-structure refinement is $(Al_{6.68}\square_{0.32})_{\Sigma 7}(Si_{2.89}As_{0.09}Sb_{0.02})_{\Sigma 3}B(O_{17.03}OH_{0.87})_{\Sigma 17.90}$.

For sample D21, the formula from the average electron-microprobe-derived composition for the bulk is

$$(Al_{6.77}Mg_{0.12}Fe_{0.07}Ti_{0.02})_{\Sigma 6.98}(Si_{2.82}As_{0.14}Sb_{0.02})_{\Sigma 2.98}B(O_{17.82}F_{0.02})_{\Sigma 17.84},$$

that from the average electron-microprobe-derived composition for the single crystal is

$$(Al_{6.72}Mg_{0.12}Fe_{0.07}Ti_{0.07})_{\Sigma 6.98}(Si_{2.84}As_{0.11}Sb_{0.02})_{\Sigma 2.97}B(O_{17.84}F_{0.02})_{\Sigma 17.86}.$$

and that from the structure refinement is

$$(Al_{6.80}\square_{0.20})_{\Sigma 7}(Si_{2.90}As_{0.09}Sb_{0.02})_{\Sigma 3.01}B(O_{17.46}OH_{0.42})_{\Sigma 17.88}.$$

For sample D31 the formula from the average electron-microprobe-derived composition is

$$(Al_{6.85}Fe_{0.08}Ta_{0.05}Mg_{0.03}Nb_{0.02})_{\Sigma 7.03}(Si_{2.65}As_{0.18}Sb_{0.04})_{\Sigma 2.87}B(O_{17.76}F_{0.01})_{\Sigma 17.77}$$

and that from the structure refinement is

$$(Al_{6.81}\square_{0.19})_{\Sigma 7}(Si_{2.88}As_{0.10}Sb_{0.02})_{\Sigma 3}B(O_{17.43}OH_{0.45})_{\Sigma 17.88}.$$

These formulae illustrate one of the difficulties in working with members of the dumortierite group: electron-microprobe-derived compositions commonly indicate more Al than do the crystal-structure refinements. This might indicate that it is difficult to obtain proper electron-microprobe standards for Al in members of the dumortierite group; however, it may also reflect the difficulty in modeling substituents for Al and vacancies at the Al1 site. There is also a possibility that the Al2, Al3, and Al4 sites are fully occupied and that the apparent partial occupancies are incorrect.

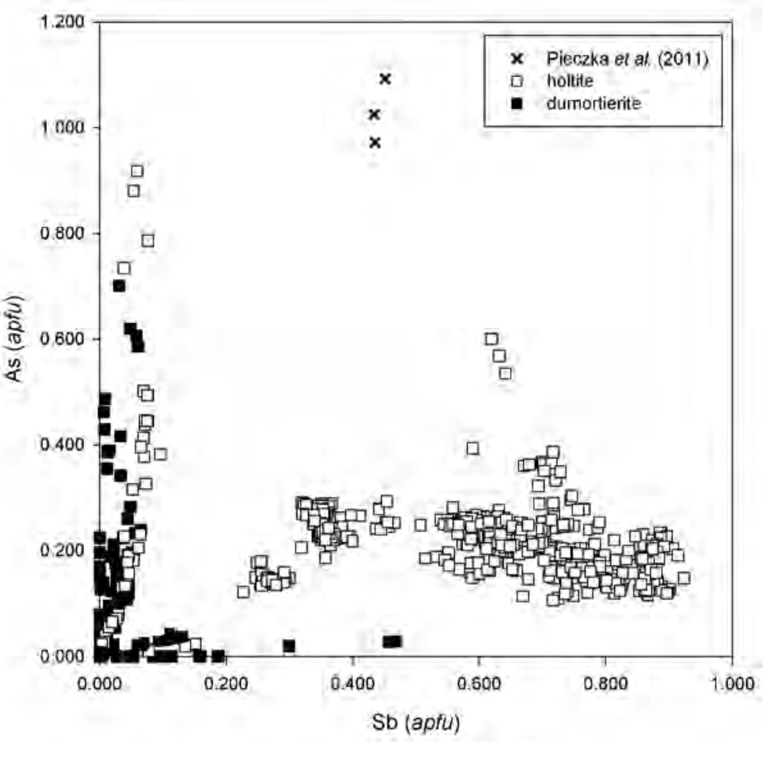

FIG. 6. Sb *versus* As (*apfu*) for the same compositions plotted in Figure 5.

Comparison with holtite

Groat *et al.* (2009) studied four holtite crystals from the three known localities and obtained compositions with an electron microprobe of 0.209 to 0.408 Nb + Ta *pfu* and As + Sb 0.381–1.026 *pfu*. In their crystal-structure refinements, a cation was assumed to occupy the Al1 site if there were oxygen atoms at the coordinating O2 and O7 positions. Therefore, the atomic occupancy of the Al1 site was fixed at the value obtained through refinement for the O2 and O7 (and hence Si1 and Si2) sites. The ratio Al:Ta was refined within this overall fixed value, which for the four crystals studied by Groat *et al.* (2009) was 0.719 to 0.883, similar to the crystals studied here. The resulting U_{eq} values for the atoms at the Al1 site were 0.0050, 0.01530, 0.01537 and 0.030 Å^2. Except for the last number, which is from a twinned crystal, these values are considerably lower than those obtained here for the Al1 site prior to splitting. Given the similar occupancies, why are the atoms at the Al1 site in the (As, Sb)-bearing dumortierite so much more strongly anisotropic than those in holtite? The answer may lie with the pentavalent Ta and Nb ions (and the Ti^{4+} ions in holtite samples from Szklary in Poland; Pieczka *et al.* 2011). Given their high charge and the short distance between Al1 sites, these ions might be expected to occupy the centers of the coordination octahedra, and be preceded and followed along the chain of Al1 positions by vacancies. The pentavalent cations are so highly charged that they require buffers of vacant sites above and below to maximize their distance from adjacent cations. These buffering vacancies, and the octahedral vacancies required by substitution of rings of (As, Sb) for Si, force the hexagonal channel in holtite to be occupied predominantly by Ta, Nb and Al in centered Al1-like sites.

The holtite samples studied by Groat *et al.* (2009) also show little disorder at the Sb1 and Sb2 positions; the U_{eq} values were 0.0067, 0.0103, 0.0107, and 0.0162 Å^2 for Sb1, and 0.0060, 0.0089, 0.0093, and 0.015 Å^2 for Sb2. The U_{eq} values for O2 and O7 were similarly low: 0.0086, 0.0.0104, 0.0106, 0.022 Å^2 for O2, and 0.0086, 0.0108, 0.0116, and 0.022 Å^2 for O7.

There is much more As + Sb in holtite than in As and Sb-bearing dumortierite, so why do they not occupy separate atomic positions? It may be that the relative lack of disorder at the Al1, O2 and O7 positions does not permit this to happen, and that <Sb1–O> and <Sb2–O> in holtite (1.864–1.908 and 1.903–1.94 Å, respectively) are sufficiently close to the average between As and Sb to satisfy both.

COMPARISON WITH NATURAL AND SYNTHETIC DUMORTIERITE-LIKE MATERIALS

Several materials with structures similar to dumortierite, both natural and synthetic, have been found to show a disorder in the hexagonal channels similar to that described here.

A material that gives rose quartz its color is closely related to dumortierite: it forms fibrous nano-inclusions, ranging in width from 0.1 to 0.5 μm (Goreva *et al.* 2001). Ma *et al.* (2002) reported that selected-area electron-diffraction (SAED) patterns and high-resolution transmission electron microscope (HRTEM) images show that the fibers have a superstructure with a doubled periodicity along the **b** and **c** axes of dumortierite, giving cell parameters $a = a_{\mathrm{dum}} = 4.7$ Å, $b = 2b_{\mathrm{dum}} = 23.6$ Å, $c = 2c_{\mathrm{dum}} = 40.5$ Å. Computer simulations suggested that periodic arrangements of two different Al1 site-occupancies in the chains of face-sharing octahedra give rise to the superstructure; one type of Al1 site is occupied mainly by Al, whereas the other type is dominated by Ti and Fe. Analytical electron microscopy (AEM) analysis showed that the fibers have a composition similar to dumortierite, but with a greater amount of Fe substituting for Al at the Al1 sites.

Among synthetic compounds, Ni-bearing hydroxyarsenates with general formula $Ni_{12+x}H_{6-2x}(AsO_4)_8(OH)_6$ have been reported to show disorder similar to that described here. Marcos *et al.* (1995) refined the crystal structures of the extreme compositions ($x = 1.16$ and 1.33) from X-ray powder-diffraction data. The results show that the materials crystallize in the space group $P6_3mc$ with a structure pseudomorphic with respect to that of dumortierite. The Ni atoms occur both inside the hexagonal channels at the 2*a* special position and in $[M_4O_{12}]_n$ double chains of octahedra running along the [001] direction. In both refinements, the Ni and As atoms are highly anisotropic, with U_{33} much greater than U_{11} and U_{22}. For the composition with $x = 1.33$ [unit cell *a* 12.6953(2) and *c* 5.031 l(1) Å], the refined occupancy of the channel Ni site is 0.67, which implies face-sharing of some of the NiO_6 octahedra, and results in local displacement of the Ni atoms along the *z* axis. In order to model the disorder, the authors devised a model allowing for disorder between two different positions along the *z* axis for all the heavy atoms; the resulting channel Ni positions were found to be Ni2 (0, 0, 0.61) and Ni2a (0, 0, 0.315), with Ni2–Ni2a = 0.63 Å. To obtain a proper geometry for the coordination polyhedra of the heavy atoms, disorder of the oxygen atoms was also introduced in the model. Marcos *et al.* (1995) noted that the large displacements affecting the Ni atoms are cushioned by the flexible AsO_4 groups, which undergo less severe disorder. The disorder propagates throughout the entire structure, but it becomes mitigated away from the hexagonal channels in which the main strain occurs. The authors noted that although they were able to model the disorder of the Ni atoms, a possible superstructure could not be ruled out, and that transmission electron microscopy experiments were underway. Disorder in other varieties of synthetic materials with dumortierite-like structures has not been reported.

Other investigators have reported order leading to a reduction in symmetry. Smit *et al.* (2006) described the lyonsite-type oxides [named after the mineral lyonsite, α-$Cu_3Fe_4(VO_4)_6$], with a general formula $M_{16}(TO_4)_{12}$. and crystal structures similar to dumortierite, with three unique MO_6 polyhedra (edge-sharing octahedra, edge-sharing trigonal prisms, and face-sharing octahedra along hexagonal channels) and two tetrahedrally coordinated *T* sites. Although the majority of lyonsite-type oxides crystallize in space group *Pnma*, several structures with lyonsite-type connectivity and lower symmetry have been described. In general, the orthorhombic crystal class is preserved, and subclasses of the *Pnma* space group are observed. For example, $Li_2Zr(MoO_4)_3$ and $Li_{3.35}Ta_{0.53}(MoO_4)_3$ crystallize in space group $P2_1mn$ ($Pmn2_1$). In $Li_2Zr(MoO_4)_3$, the face-shared octahedron position is ordered, with Zr^{4+} ions alternating with cation vacancies, which results in the doubling of one of the lattice constants and the loss of a mirror plane (Klevtsova *et al.* 1979, Smit *et al.* 2006). Similarly, Ta^{5+} ions alternate with Li^+ ions in $Li_{3.35}Ta_{0.53}(MoO_4)_3$ (Smit *et al.* 2006).

CONCLUSIONS

Among the four dumortierite samples from granitic pegmatites studied here, one (D67) is a classic boroaluminosilicate, dumortierite, with octahedral sites dominated by Al (and a small amount of Fe), and tetrahedral sites dominated by Si. In contrast, compositions of the other three samples lie on a continuum between dumortierite and holtite, with significant amounts of As^{3+} and Sb^{3+} (and in the case of D27, possibly Bi^{3+}) replacing Si, but without significant replacement of Al^{3+} by Ta^{5+} or Nb^{5+}, the other distinguishing characteristic of holtite. This compositional continuum is illustrated in a plot of As + Sb + Bi *versus* Si + P for a range of holtite and dumortierite samples (Fig. 5). Although the point (Si + P = 2.50 *apfu*, As + Sb + Bi = 0.30 *apfu*) lies in the transition between what has been considered holtite and what has been considered dumortierite, there is no gap, and there are compositions of minerals referred to as holtite plotting at Si + P > 2.50 *apfu* within the dumortierite field (Fig. 7; Cempírek *et al.* 2010, Borghi *et al.* 2004, Vaggelli *et al.* 2004), and compositions of minerals referred to as dumortierite plotting at Si + P < 2.50 *apfu* in the holtite field (Galliski *et al.* 2012). There is a pronounced gap in terms of As and Sb occupancy (Fig. 6), but the gap separates dumortierite and Sb-poor holtite from Sb-bearing holtite, further blurring the distinction between these two minerals. Moreover, this gap may well be filled as more holtite and dumortierite compositions become available. The continuum of compositions between dumortierite and holtite, and the discovery of very (As, Sb)-rich, (Ta, Nb)-poor compositions (Cempírek *et al.* 2010, Pieczka *et al.* 2011, Galliski

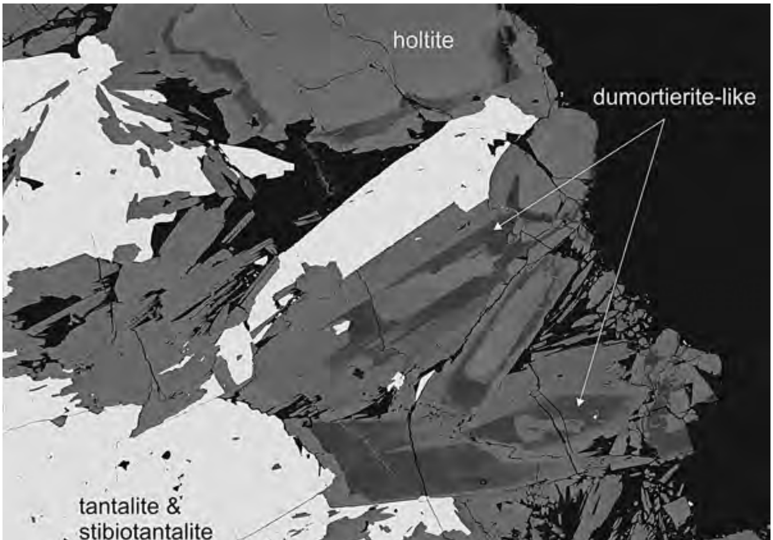

FIG. 7. Back-scattered electron (BSE) image of holtite crystals (medium gray) from the type locality, Greenbushes, Western Australia, showing darker gray areas with a dumortierite-like composition. The white crystals are tantalite and stibiotantalite. The area shown is approximately 750 μm by 570 μm.

et al. 2012) suggest that the distinction between what has been called dumortierite and what has been called holtite should be reconsidered. These minerals are solid solutions in Si–As–Sb–Al–Ta–Nb–Ti–O–H compositional space. A new set of end members in this space is needed to properly define these minerals, but this is a task beyond the scope of the present paper.

With compositions intermediate between dumortierite and holtite comes crystallographic disorder in the structure's hexagonal channel, which contains the Si, As, Sb and Al1 sites. Samples D21, D27 and D31 all have separate As and Sb triangular pyramidal sites, which has not been in observed in holtite. The Al1 site is also considerably more strongly anisotropic in all four samples than in holtite (Groat *et al.* 2009). In samples D67, D21, D27 and D31, the Al1 site is split into Al1a, Al1 and Al1b sites, with Al1a and Al1b shifted above and below Al1, respectively. The unequal occupancy of Al1a, Al1 and Al1b suggests that the hexagonal channel contains a disordered mixture of face-sharing octahedron dimers, trimers and longer units separated by vacancies, as opposed to the ordered trimer configuration found by Evans *et al.* (2012). This kind of disorder also is not observed in holtite, possibly in part due to a lack in dumortierite of (Ta^{5+}, Nb^{5+}) substitution at the Al1 site, which in holtite creates extra vacancies between occupied sites.

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_exptl_absorpt_correction_T_min   ?
_exptl_absorpt_correction_T_max   ?
_exptl_absorpt_process_details    ?

_exptl_special_details
;
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;

_diffrn_ambient_temperature       293(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   ?
_diffrn_measurement_method        ?
_diffrn_detector_area_resol_mean  ?
_diffrn_reflns_number             14952
_diffrn_reflns_av_R_equivalents   0.0291
_diffrn_reflns_av_sigmaI/netI     0.0177
_diffrn_reflns_limit_h_min        -7
_diffrn_reflns_limit_h_max        7
_diffrn_reflns_limit_k_min        -18
_diffrn_reflns_limit_k_max        18
_diffrn_reflns_limit_l_min        -18
_diffrn_reflns_limit_l_max        31
_diffrn_reflns_theta_min          2.00
_diffrn_reflns_theta_max          33.20
_reflns_number_total              2245
_reflns_number_gt                 2036
_reflns_threshold_expression      >2sigma(I)
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_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0290P)^2^+0.1819P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      SHELXL
_refine_ls_extinction_coef        0.0017(5)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          2245
_refine_ls_number_parameters      164
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0214
_refine_ls_R_factor_gt            0.0185
_refine_ls_wR_factor_ref          0.0477
_refine_ls_wR_factor_gt           0.0463
_refine_ls_goodness_of_fit_ref    1.044
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_refine_ls_shift/su_max           0.001
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Al1 Al 0.410(3) 0.7500 0.24960(8) 0.00775(16) Uiso 0.423(7) 2 d SP . .
Al1B Al 0.479(3) 0.7500 0.24934(14) 0.00775(16) Uiso 0.177(17) 2 d SP . .
Al2 Al 0.55788(5) 0.61057(2) 0.472550(12) 0.00549(8) Uani 0.9923(15) 1 d P . .
Al3 Al 0.05966(5) 0.49113(2) 0.431125(12) 0.00559(8) Uani 0.9922(16) 1 d P . .
Al4 Al 0.05850(6) 0.35910(2) 0.289310(12) 0.00748(8) Uani 0.9885(17) 1 d P . .
Si1 Si 0.0871(4) 0.7500 0.40487(7) 0.0073(2) Uani 0.9614(12) 2 d SP . .
As1 As 0.108(5) 0.7500 0.3973(10) 0.0065(19) Uiso 0.0309(10) 2 d SP . .
Sb1 Sb 0.117(3) 0.7500 0.3789(6) 0.0065(19) Uiso 0.0077(2) 2 d SP . .
Si2 Si 0.58738(14) 0.52488(5) 0.32822(3) 0.00708(10) Uani 0.9612(10) 1 d P . .
As2 As 0.607(2) 0.5443(7) 0.3218(5) 0.0064(10) Uiso 0.0311(8) 1 d P . .
Sb2 Sb 0.610(2) 0.5699(8) 0.3143(5) 0.0064(10) Uiso 0.0078(2) 1 d P . .
B B 0.2254(3) 0.2500 0.41611(6) 0.0067(2) Uani 1 2 d S . .
O1 O2- 0.37674(19) 0.7500 0.45439(4) 0.00776(15) Uani 1 2 d S . .
O2 O2- 0.1510(2) 0.7500 0.32637(5) 0.01296(19) Uani 0.9614(12) 2 d SP . .
O3 O2- 0.89572(13) 0.63925(5) 0.42440(3) 0.00674(11) Uani 1 1 d . . .
O4 O2- 0.40157(13) 0.43581(5) 0.28267(3) 0.00724(11) Uani 1 1 d . . .
O5 O2- 0.39524(13) 0.55015(5) 0.39349(3) 0.00663(11) Uani 1 1 d . . .
O6 O2- 0.88067(14) 0.45381(5) 0.35051(3) 0.00795(12) Uani 1 1 d . . .
O7 O2- 0.64890(17) 0.63881(6) 0.28694(3) 0.01339(15) Uani 0.9612(10) 1 d P . .
O8 O2- 0.1639(2) 0.2500 0.35054(4) 0.00900(16) Uani 1 2 d S . .
O9 O2- 0.25449(13) 0.35110(5) 0.44802(3) 0.00733(11) Uani 1 1 d . . .
O10 O2- 0.76083(19) 0.2500 0.27239(4) 0.00918(16) Uani 1 2 d S . .
O11 O2- 0.75026(12) 0.46648(5) 0.48806(3) 0.00568(11) Uani 1 1 d . . .

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Al3 0.00402(12) 0.00640(12) 0.00635(12) -0.00015(8) -0.00006(8) 0.00010(8)
Al4 0.00691(13) 0.00759(13) 0.00794(12) 0.00137(8) -0.00076(9) 0.00006(8)
Si1 0.0035(3) 0.0051(2) 0.0133(5) 0.000 -0.0001(3) 0.000
Si2 0.00427(16) 0.0100(2) 0.00693(18) -0.00227(13) 0.00011(12) -0.00005(15)
B 0.0063(5) 0.0071(5) 0.0067(4) 0.000 -0.0001(4) 0.000
O1 0.0055(3) 0.0061(3) 0.0117(4) 0.000 -0.0013(3) 0.000
O2 0.0125(4) 0.0126(4) 0.0138(4) 0.000 0.0045(4) 0.000
O3 0.0046(2) 0.0065(2) 0.0091(2) 0.00041(19) 0.0006(2) -0.00013(19)
O4 0.0052(3) 0.0087(3) 0.0078(2) -0.00132(19) 0.00030(19) -0.0005(2)
O5 0.0049(2) 0.0082(2) 0.0068(2) -0.00067(19) 0.00028(19) -0.00057(19)
O6 0.0057(3) 0.0103(3) 0.0079(2) -0.00187(19) -0.0010(2) 0.0008(2)
O7 0.0137(3) 0.0130(3) 0.0134(3) 0.0006(2) 0.0021(3) -0.0039(2)
O8 0.0137(4) 0.0069(4) 0.0063(3) 0.000 -0.0019(3) 0.000
O9 0.0081(3) 0.0062(2) 0.0077(2) -0.00071(19) -0.0016(2) 0.0004(2)
O10 0.0063(4) 0.0084(4) 0.0128(4) 0.000 -0.0012(3) 0.000
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_geom_special_details
```

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;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Al1A O7 1.746(4) 6_556 ?
Al1A O7 1.746(4) 4_465 ?
Al1A O2 1.757(5) . ?
Al1A Al1 1.989(7) 6_556 ?
Al1A O7 2.113(6) . ?
Al1A O7 2.113(6) 7_575 ?
Al1A O2 2.155(6) 6_656 ?
Al1A Al1A 2.34862(11) 6_556 ?
Al1A Al1A 2.3487 6_656 ?
Al1 Al1B 0.326(5) . ?
Al1 O7 1.886(8) . ?
Al1 O7 1.886(8) 7_575 ?
Al1 O2 1.909(9) 6_656 ?
Al1 O7 1.943(9) 6_556 ?
Al1 O7 1.943(9) 4_465 ?
Al1 O2 1.972(9) . ?
Al1 Al1A 1.989(7) 6_656 ?
Al1 Al1B 2.022(5) 6_556 ?
Al1 Al1 2.34861(16) 6_656 ?
Al1 Al1 2.34861(15) 6_556 ?
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Al1B O7 1.714(7) . ?
Al1B O7 1.714(7) 7_575 ?
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Al1B Al1 2.022(5) 6_656 ?
Al1B O7 2.162(11) 6_556 ?
Al1B O7 2.162(11) 4_465 ?
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Al1B Al1B 2.34872(17) 6_556 ?
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Al2 O9 1.8876(6) 5_666 ?
Al2 O1 1.8902(5) . ?
Al2 O3 1.8925(7) . ?
Al2 O5 1.9107(6) . ?
Al2 O11 1.9527(6) . ?
Al2 Al3 2.8583(4) . ?
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Al3 O9 1.9214(6) . ?
Al3 O11 1.9287(6) 5_666 ?
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Al3 Al2 2.8720(4) 1_455 ?
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Al4 O8 1.8548(6) . ?
Al4 O6 1.8661(7) 1_455 ?
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Si1 O3 1.6358(12) 1_455 ?
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Si1 O1 1.690(2) . ?
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As1 O2 1.45(2) . ?
As1 O1 1.71(3) . ?
As1 O3 1.733(15) 1_455 ?
As1 O3 1.733(15) 7_475 ?
Sb1 O2 1.074(13) . ?
Sb1 O3 1.907(10) 7_475 ?
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Sb1 O1 1.955(13) . ?
Sb1 Al1B 2.673(13) 6_556 ?
Sb1 Al1 2.774(14) 6_556 ?
Sb1 Al1A 2.936(14) 6_556 ?
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Si2 Sb2 0.611(9) . ?
Si2 O7 1.6101(9) . ?
Si2 O5 1.6267(9) . ?
Si2 O4 1.6487(9) . ?
Si2 O6 1.6752(9) . ?
As2 Sb2 0.339(13) . ?
As2 O7 1.335(8) . ?
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As2 O6 1.770(10) . ?
As2 O4 1.789(10) . ?
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Sb2 O4 1.968(10) . ?
Sb2 O6 2.009(10) . ?
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O6 Al4 1.8661(7) 1_655 ?
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O7 Al1 1.943(9) 6_656 ?
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O9 Al2 1.8876(6) 5_666 ?
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Al1B Al1A O7 118.8(3) . 6_556 ?
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Al1 Al1A O7 118.3(4) . 4_465 ?
Al1B Al1A O7 118.8(3) . 4_465 ?
Al1B Al1A O7 60.3(2) 6_556 4_465 ?
O7 Al1A O7 97.6(3) 6_556 4_465 ?
Al1 Al1A O2 122.1(5) . . ?
Al1B Al1A O2 121.1(3) . . ?
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O7 Al1A O2 97.9(3) 6_556 . ?
O7 Al1A O2 97.9(3) 4_465 . ?
Al1 Al1A Al1 177.0(5) . 6_556 ?
Al1B Al1A Al1 178.0(3) . 6_556 ?
Al1B Al1A Al1 0.17(13) 6_556 6_556 ?
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O7 Al1A Al1 60.2(2) 6_556 6_556 ?
O7 Al1A Al1 60.2(2) 4_465 6_556 ?
O2 Al1A Al1 60.9(3) . 6_556 ?
Al1 Al1A O7 46.9(3) . . ?
Al1B Al1A O7 46.45(17) . . ?
Al1B Al1A O7 134.48(19) 6_556 . ?
O7 Al1A O7 91.81(9) 6_556 . ?
O7 Al1A O7 165.2(4) 4_465 . ?
O2 Al1A O7 92.13(10) . . ?
Al1 Al1A O7 134.56(18) 6_556 . ?
Al1 Al1A O7 46.9(3) . 7_575 ?
Al1B Al1A O7 46.45(17) . 7_575 ?
Al1B Al1A O7 134.48(19) 6_556 7_575 ?
O7 Al1A O7 165.2(4) 6_556 7_575 ?
O7 Al1A O7 91.81(9) 4_465 7_575 ?
O2 Al1A O7 92.13(10) . 7_575 ?
Al1 Al1A O7 134.56(18) 6_556 7_575 ?
O7 Al1A O7 76.9(3) . 7_575 ?
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Al1B Al1A O2 44.2(3) . 6_656 ?
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O7 Al1A O2 91.75(12) 6_556 6_656 ?
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O2 Al1A O2 165.3(4) . 6_656 ?
Al1 Al1A O2 133.76(14) 6_556 6_656 ?
O7 Al1A O2 76.5(3) . 6_656 ?
O7 Al1A O2 76.5(3) 7_575 6_656 ?
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Al1 Al1A Al1A 0.44(7) 6_556 6_556 ?
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O2 Al1A Al1A 133.32(18) 6_656 6_556 ?
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Al1B Al1A Al1A 1.5(2) . 6_656 ?
Al1B Al1A Al1A 179.6(2) 6_556 6_656 ?
O7 Al1A Al1A 119.5(3) 6_556 6_656 ?
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O2 Al1A Al1A 119.7(2) . 6_656 ?
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O7 Al1A Al1A 45.71(16) . 6_656 ?
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O2 Al1A Al1A 45.68(19) 6_656 6_656 ?
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Al1B Al1 Al1A 178.0(11) . . ?
Al1B Al1 O7 53.9(5) . . ?
Al1A Al1 O7 125.0(4) . . ?
Al1B Al1 O7 53.9(5) . 7_575 ?
Al1A Al1 O7 125.0(4) . 7_575 ?
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Al1B Al1 O2 52.7(8) . 6_656 ?
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O7 Al1 O2 88.2(5) . 6_656 ?
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Al1A Al1 O7 52.3(4) . 6_556 ?
O7 Al1 O7 93.26(4) . 6_556 ?
O7 Al1 O7 177.4(7) 7_575 6_556 ?
O2 Al1 O7 93.91(6) 6_656 6_556 ?
Al1B Al1 O7 128.7(5) . 4_465 ?
Al1A Al1 O7 52.3(4) . 4_465 ?
O7 Al1 O7 177.4(7) . 4_465 ?
O7 Al1 O7 93.26(4) 7_575 4_465 ?
O2 Al1 O7 93.91(6) 6_656 4_465 ?
O7 Al1 O7 85.1(5) 6_556 4_465 ?
Al1B Al1 O2 129.0(7) . . ?
Al1A Al1 O2 49.0(5) . . ?
O7 Al1 O2 92.96(7) . . ?
O7 Al1 O2 92.96(7) 7_575 . ?
O2 Al1 O2 178.3(7) 6_656 . ?
O7 Al1 O2 84.9(5) 6_556 . ?
O7 Al1 O2 84.9(5) 4_465 . ?
Al1B Al1 Al1A 0.8(7) . 6_656 ?
Al1A Al1 Al1A 177.2(5) . 6_656 ?
O7 Al1 Al1A 53.5(3) . 6_656 ?
O7 Al1 Al1A 53.5(3) 7_575 6_656 ?
O2 Al1 Al1A 53.6(4) 6_656 6_656 ?
O7 Al1 Al1A 129.1(3) 6_556 6_656 ?
O7 Al1 Al1A 129.1(4) 4_465 6_656 ?
O2 Al1 Al1A 128.1(3) . 6_656 ?
Al1B Al1 Al1B 179.7(6) . 6_556 ?
Al1A Al1 Al1B 2.3(5) . 6_556 ?
O7 Al1 Al1B 126.2(4) . 6_556 ?
O7 Al1 Al1B 126.2(4) 7_575 6_556 ?
O2 Al1 Al1B 127.0(3) 6_656 6_556 ?
O7 Al1 Al1B 51.2(3) 6_556 6_556 ?
O7 Al1 Al1B 51.2(3) 4_465 6_556 ?
O2 Al1 Al1B 51.3(3) . 6_556 ?
Al1A Al1 Al1B 179.45(8) 6_656 6_556 ?
Al1B Al1 Al1 1.3(6) . 6_656 ?
Al1A Al1 Al1 176.7(5) . 6_656 ?
O7 Al1 Al1 53.3(3) . 6_656 ?
O7 Al1 Al1 53.3(3) 7_575 6_656 ?
O2 Al1 Al1 54.0(4) 6_656 6_656 ?
O7 Al1 Al1 129.3(3) 6_556 6_656 ?
O7 Al1 Al1 129.3(3) 4_465 6_656 ?
O2 Al1 Al1 127.7(3) . 6_656 ?
Al1A Al1 Al1 0.46(7) 6_656 6_656 ?
Al1B Al1 Al1 179.00(9) 6_556 6_656 ?
Al1B Al1 Al1 179.5(8) . 6_556 ?
Al1A Al1 Al1 2.5(4) . 6_556 ?
O7 Al1 Al1 126.3(4) . 6_556 ?
O7 Al1 Al1 126.3(4) 7_575 6_556 ?
O2 Al1 Al1 126.8(3) 6_656 6_556 ?
O7 Al1 Al1 51.1(3) 6_556 6_556 ?
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Al1 Al1B Al1A 179.0(8) . 6_656 ?
Al1A Al1B Al1A 177.9(3) . 6_656 ?
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Al1A Al1B O7 116.7(4) . . ?
Al1A Al1B O7 62.3(4) 6_656 . ?
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Al1A Al1B O7 62.3(4) 6_656 7_575 ?
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Al1A Al1B O2 119.7(6) . 6_656 ?
Al1A Al1B O2 62.3(5) 6_656 6_656 ?
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O7 Al1B O2 100.1(6) 7_575 6_656 ?
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Al1A Al1B O7 135.9(3) 6_656 6_556 ?
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O8 Al4 O4 164.48(3) . 6_556 ?
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Sb1 As1 O1 126(6) . . ?
O2 As1 O1 124.3(15) . . ?
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Sb1 As1 O3 112(3) . 1_455 ?
O2 As1 O3 113.2(11) . 1_455 ?
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Si1 As1 O3 55(3) . 7_475 ?
Sb1 As1 O3 112(3) . 7_475 ?
O2 As1 O3 113.2(11) . 7_475 ?
O1 As1 O3 102.2(9) . 7_475 ?
O3 As1 O3 98.1(11) 1_455 7_475 ?
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As1 Sb1 O2 178(6) . . ?
Si1 Sb1 O2 174(2) . . ?
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Si1 Sb1 O3 52.7(7) . 7_475 ?
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Si1 Sb1 O3 52.7(7) . 1_455 ?
O2 Sb1 O3 124.0(6) . 1_455 ?
O3 Sb1 O3 86.6(6) 7_475 1_455 ?
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Si1 Sb1 O1 53.5(12) . . ?
O2 Sb1 O1 132.8(11) . . ?
O3 Sb1 O1 87.9(5) 7_475 . ?
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O3 Sb1 Al1B 109.7(5) 7_475 6_556 ?
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O3 Sb1 Al1A 100.6(5) 1_455 6_556 ?
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O2 Sb1 Al1 19.2(6) . . ?
O3 Sb1 Al1 132.9(3) 7_475 . ?
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O6 As2 Al1B 145.3(5) . . ?
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As1 O1 Al2 117.61(13) . 7_575 ?
Al2 O1 Al2 121.28(5) . 7_575 ?
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As1 O1 Sb1 9.0(10) . . ?
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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Al4 Al4 Al4 90.0 7_565 6_556 ?
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O8 Al4 Al4 98.26(8) . 6_656 ?
O4 Al4 Al4 84.15(6) 6_556 6_656 ?
O6 Al4 Al4 137.66(6) 1_455 6_656 ?
O10 Al4 Al4 120.31(7) 1_455 6_656 ?
O10 Al4 Al4 42.93(5) 6_556 6_656 ?
O6SB Al4 Al4 119.3(15) 1_455 6_656 ?
Al4 Al4 Al4 90.0 7_565 6_656 ?
Al4 Al4 Al4 112.15(4) 6_556 6_656 ?
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Sb1 Si1 O1SB 93(3) . . ?
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Sb1 Si1 O2 19(2) . . ?
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Sb1 Si1 O3 98.7(14) . 1_455 ?
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O3 Si1 O1 107.70(9) 7_475 . ?
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As2 Si2 O6SB 80(3) . . ?
Sb2 Si2 O6SB 104(3) . . ?
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Sb2 Si2 O7 19.9(16) . . ?
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Sb1 As1 O1SB 124(6) . . ?
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Si1 As1 O1 64(5) . . ?
Sb1 As1 O1 164(5) . . ?
O2 As1 O1 131.1(18) . . ?
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Sb1 As1 O3 97(4) . 1_455 ?
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O1SB As1 Al1B 112(3) . 6_556 ?
O1 As1 Al1B 151.5(13) . 6_556 ?
O3 As1 Al1B 104.9(11) 1_455 6_556 ?
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Sb2 As2 O5 94(4) . . ?
O7 As2 O5 126.3(10) . . ?
O6SB As2 O5 112(2) . . ?
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Sb2 As2 O6 173(4) . . ?
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O6SB As2 O6 25(2) . . ?
O5 As2 O6 90.1(8) . . ?
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Sb2 As2 O4 99(3) . . ?
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O6 As2 Al1A 118.1(7) . 6_656 ?
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Al1A As2 Al1 43.5(5) 6_656 . ?
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O6 As2 Al1 112.7(5) . 6_656 ?
O4 As2 Al1 132.1(6) . 6_656 ?
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Al1A As2 Al1 5.4(4) 6_656 6_656 ?
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O6 As2 Al1A 166.8(8) . . ?
O4 As2 Al1A 100.7(6) . . ?
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Si1 Sb1 O1SB 55(3) . . ?
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O1SB Sb1 Al1 135(3) . 6_556 ?
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O3 Sb1 Al1 120.0(15) 1_455 6_556 ?
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Si1 Sb1 Al1A 163(3) . 6_556 ?
O1SB Sb1 Al1A 142(3) . 6_556 ?
O3 Sb1 Al1A 115.5(15) 7_475 6_556 ?
O3 Sb1 Al1A 115.5(15) 1_455 6_556 ?
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Al1 Sb1 Al1A 6.7(5) 6_556 6_556 ?
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O2 Sb1 O1 109(3) . . ?
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Al1A Sb1 O1 168(2) 6_556 . ?
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O3 Sb1 Al1 139.1(9) 1_455 . ?
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Al1A Sb1 Al1 5.5(5) . . ?
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O7 Sb2 O5 147(2) . . ?
Si2 Sb2 O5 54.6(13) . . ?
O6SB Sb2 O5 91(2) . . ?
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O7 Sb2 O4 132(2) . . ?
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Si2 Sb2 Al1 169(2) . . ?
O6SB Sb2 Al1 142(2) . . ?
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O4 Sb2 Al1 119.6(11) . . ?
Al1B Sb2 Al1 9.3(5) . . ?
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Si2 Sb2 Al1A 139(2) . 6_656 ?
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O5 Sb2 Al1 141.9(10) . 6_656 ?
O4 Sb2 Al1 136.6(10) . 6_656 ?
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Al1A Sb2 Al1 5.4(4) 6_656 6_656 ?
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O8 B O9 119.00(15) . 7_565 ?
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As2 O7 Si2 2.7(6) . . ?
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As2 O7 Al1B 143.4(9) . . ?
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As2 O7 Al1A 157.2(8) . 6_656 ?
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As2 O7 Al1 149.6(6) . 6_656 ?
Si2 O7 Al1 151.94(15) . 6_656 ?
Al1B O7 Al1 66.6(5) . 6_656 ?
Al1A O7 Al1 7.7(6) 6_656 6_656 ?
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Al1A O7 Al1 68.7(6) 6_656 . ?
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Al1 O7 Al1B 7.8(3) 6_656 6_656 ?
Al1 O7 Al1B 84.2(3) . 6_656 ?
Al1A O7 Al1B 90.4(7) . 6_656 ?
B O8 Al4 135.34(7) . 7_565 ?
B O8 Al4 135.34(7) . . ?
Al4 O8 Al4 87.27(11) 7_565 . ?
B O9 Al2 131.30(18) . 5_666 ?
B O9 Al3 128.34(18) . . ?
Al2 O9 Al3 99.77(8) 5_666 . ?
Al4 O10 Al4 83.29(10) 1_655 7_665 ?
Al4 O10 Al4 150.48(13) 1_655 4 ?
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Al3 O11 Al2 158.34(9) 1_655 5_666 ?
Al3 O11 Al3 97.09(7) 1_655 5_666 ?
Al2 O11 Al3 96.89(7) 5_666 5_666 ?
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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_atom_sites_solution_secondary    difmap
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 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
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Al1B Al 0.487(6) 0.7500 0.2495(2) 0.0071(2) Uiso 0.14(2) 2 d SP . .
Al2 Al 0.55802(7) 0.61051(3) 0.472482(16) 0.00506(13) Uani 0.990(2) 1 d P . .
Al3 Al 0.05971(7) 0.49106(3) 0.431009(16) 0.00517(13) Uani 0.990(2) 1 d P . .
Al4 Al 0.05802(8) 0.35872(3) 0.289074(16) 0.00662(14) Uani 0.984(2) 1 d P . .
Si1 Si 0.0874(13) 0.7500 0.4045(4) 0.0068(7) Uani 0.9592(15) 2 d SP . .
As1 As 0.105(14) 0.7500 0.399(5) 0.002(4) Uiso 0.0326(12) 2 d SP . .
Sb1 Sb 0.113(5) 0.7500 0.3802(9) 0.002(4) Uiso 0.0082(3) 2 d SP . .
Si2 Si 0.5875(3) 0.52556(8) 0.32794(6) 0.0063(2) Uani 0.9583(13) 1 d P . .
As2 As 0.608(4) 0.5417(13) 0.3214(9) 0.0055(18) Uiso 0.0333(10) 1 d P . .
Sb2 Sb 0.607(3) 0.5735(12) 0.3153(7) 0.0055(18) Uiso 0.0083(3) 1 d P . .
B B 0.2261(4) 0.2500 0.41615(9) 0.0067(3) Uani 1 2 d S . .
O1 O2- 0.3767(3) 0.7500 0.45417(6) 0.0072(2) Uani 1 2 d S . .
O2 O2- 0.1500(3) 0.7500 0.32577(6) 0.0128(3) Uani 0.9592(15) 2 d SP . .
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O4 O2- 0.40051(17) 0.43611(7) 0.28251(4) 0.00632(18) Uani 1 1 d . . .
O5 O2- 0.39510(18) 0.55028(7) 0.39341(4) 0.00600(17) Uani 1 1 d . . .
O6 O2- 0.88080(18) 0.45396(7) 0.35023(4) 0.00709(18) Uani 1 1 d . . .
O7 O2- 0.6481(2) 0.63972(8) 0.28660(4) 0.0128(2) Uani 0.9583(13) 1 d P . .
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O9 O2- 0.25468(18) 0.35117(7) 0.44805(4) 0.00697(18) Uani 1 1 d . . .
O10 O2- 0.7611(3) 0.2500 0.27224(6) 0.0081(2) Uani 1 2 d S . .
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Si1 0.0044(10) 0.0054(5) 0.0106(17) 0.000 -0.0003(8) 0.000
Si2 0.0046(3) 0.0091(5) 0.0051(3) -0.0019(3) 0.0003(2) -0.0002(3)
B 0.0066(8) 0.0067(8) 0.0067(8) 0.000 0.0000(6) 0.000
O1 0.0058(6) 0.0059(5) 0.0099(5) 0.000 -0.0011(4) 0.000
O2 0.0135(7) 0.0129(6) 0.0118(6) 0.000 0.0042(5) 0.000
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O4 0.0051(4) 0.0078(4) 0.0060(4) -0.0009(3) 0.0006(3) 0.0001(3)
O5 0.0053(4) 0.0076(4) 0.0052(3) -0.0008(3) -0.0001(3) -0.0006(3)
O6 0.0056(4) 0.0093(4) 0.0064(4) -0.0017(3) -0.0006(3) 0.0009(3)
O7 0.0157(5) 0.0116(5) 0.0110(4) 0.0012(4) 0.0014(4) -0.0025(4)
O8 0.0144(6) 0.0062(5) 0.0055(5) 0.000 -0.0018(4) 0.000
O9 0.0088(4) 0.0059(4) 0.0062(4) -0.0010(3) -0.0017(3) 0.0004(3)
O10 0.0064(6) 0.0072(6) 0.0109(5) 0.000 -0.0003(4) 0.000
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;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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Al1A O7 1.736(7) 4_465 ?
Al1A O2 1.752(8) . ?
Al1A Al1 1.981(10) 6_556 ?
Al1A O7 2.092(9) 7_575 ?
Al1A O7 2.092(9) . ?
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Al1 O7 1.862(12) . ?
Al1 O2 1.886(12) 6_656 ?
Al1 O7 1.939(13) 6_556 ?
Al1 O7 1.939(13) 4_465 ?
Al1 O2 1.968(13) . ?
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Al1 Al1B 1.995(12) 6_556 ?
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Al3 O6 1.8847(9) 1_455 ?
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Al3 O9 1.9166(9) . ?
Al3 O11 1.9268(8) 5_666 ?
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Al3 Al2 2.8659(5) 1_455 ?
Al3 Al2 2.9049(5) 5_666 ?
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Al4 O8 1.8514(9) . ?
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Al4 O6 1.8640(9) 1_455 ?
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Al4 O10 2.0199(10) 6_556 ?
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As1 O2 1.50(9) . ?
As1 O1 1.69(8) . ?
As1 O3 1.71(4) 1_455 ?
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Sb1 O3 1.878(13) 1_455 ?
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Sb1 Al1B 2.68(2) 6_556 ?
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Si2 Sb2 0.626(14) . ?
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Si2 O4 1.6495(15) . ?
Si2 O6 1.6748(15) . ?
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B O9 1.3621(12) . ?
O1 Al2 1.8876(7) 7_575 ?
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O2 Al1 1.886(12) 6_556 ?
O2 Al1A 2.130(10) 6_556 ?
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O3 As1 1.71(4) 1_655 ?
O3 Sb1 1.878(13) 1_655 ?
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O7 Al1 1.939(13) 6_656 ?
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O8 Al4 1.8514(9) 7_565 ?
O9 Al2 1.8833(9) 5_666 ?
O10 Al4 1.9225(10) 1_655 ?
O10 Al4 1.9225(10) 7_665 ?
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Al1B Al1A O7 119.7(5) . 6_556 ?
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Al1 Al1A O7 119.2(6) . 4_465 ?
Al1B Al1A O7 119.7(5) . 4_465 ?
Al1B Al1A O7 59.7(4) 6_556 4_465 ?
O7 Al1A O7 97.0(5) 6_556 4_465 ?
Al1 Al1A O2 121.9(7) . . ?
Al1B Al1A O2 121.0(5) . . ?
Al1B Al1A O2 60.2(4) 6_556 . ?
O7 Al1A O2 97.1(5) 6_556 . ?
O7 Al1A O2 97.1(5) 4_465 . ?
Al1 Al1A Al1 177.8(7) . 6_556 ?
Al1B Al1A Al1 178.7(4) . 6_556 ?
Al1B Al1A Al1 0.1(2) 6_556 6_556 ?
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O2 Al1A Al1 60.3(4) . 6_556 ?
Al1 Al1A O7 46.6(4) . 7_575 ?
Al1B Al1A O7 46.1(3) . 7_575 ?
Al1B Al1A O7 134.5(3) 6_556 7_575 ?
O7 Al1A O7 165.8(7) 6_556 7_575 ?
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O2 Al1A O7 92.48(15) . 7_575 ?
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Al1B Al1A O7 46.1(3) . . ?
Al1B Al1A O7 134.5(3) 6_556 . ?
O7 Al1A O7 92.22(13) 6_556 . ?
O7 Al1A O7 165.8(7) 4_465 . ?
O2 Al1A O7 92.48(15) . . ?
Al1 Al1A O7 134.5(3) 6_556 . ?
O7 Al1A O7 76.8(4) 7_575 . ?
Al1 Al1A O2 43.9(7) . 6_656 ?
Al1B Al1A O2 44.8(5) . 6_656 ?
Al1B Al1A O2 134.0(4) 6_556 6_656 ?
O7 Al1A O2 92.31(18) 6_556 6_656 ?
O7 Al1A O2 92.31(18) 4_465 6_656 ?
O2 Al1A O2 165.8(6) . 6_656 ?
Al1 Al1A O2 133.9(2) 6_556 6_656 ?
O7 Al1A O2 76.5(4) 7_575 6_656 ?
O7 Al1A O2 76.5(4) . 6_656 ?
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Al1B Al1A Al1A 178.3(4) . 6_556 ?
Al1B Al1A Al1A 0.44(16) 6_556 6_556 ?
O7 Al1A Al1A 59.5(4) 6_556 6_556 ?
O7 Al1A Al1A 59.5(4) 4_465 6_556 ?
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Al1 Al1A Al1A 0.35(10) 6_556 6_556 ?
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Al1B Al1A Al1A 1.0(4) . 6_656 ?
Al1B Al1A Al1A 179.8(3) 6_556 6_656 ?
O7 Al1A Al1A 120.2(4) 6_556 6_656 ?
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O2 Al1A Al1A 45.8(3) 6_656 6_656 ?
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Al1A Al1 O7 125.2(6) . 7_575 ?
Al1B Al1 O7 53.8(7) . . ?
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Al1B Al1 O2 53.4(12) . 6_656 ?
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Al1A Al1 O7 51.4(6) . 4_465 ?
O7 Al1 O7 93.55(7) 7_575 4_465 ?
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O3 Sb1 Al1 132.2(5) 1_455 . ?
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Sb2 As2 O4 122(5) . . ?
O7 As2 O4 116.4(11) . . ?
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
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 and torsion angles; correlations between esds in cell parameters are only
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Si1 O2 Al1 133.9(7) . 6_556 ?
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AlA O2 AlA 74.88(8) . 6_556 ?
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Si1 O2 AlB 145.2(9) . . ?
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Si2 O5 Al3 136.12(4) . . ?
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Si2 O6 Al4 119.46(4) . 1_655 ?
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Si2 O7 AlB 140.0(12) . . ?
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O8 B O9 119.02(6) . . ?
O9 B O9 121.95(11) 7_565 . ?

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_refine_diff_density_rms    0.076
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