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Potassium oxoaluminate antimonate(III), $K_2[Al_2Sb_2O_7]$

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Dipotassium dialuminium diantimonate, $K_2[Al_2Sb_2O_7]$, crystallizes in the trigonal space group $P\overline{3}m1$. The structure is isotypic with $K_2Pb_2Ge_2O_7$ and consists of $[Al_2Sb_2O_7]^{2-}$ layers containing Al^{3+} in a nearly regular tetrahedral and Sb^{3+} in a Ψ-tetrahedral environment of O ligands.

Comment

The title compound (Fig. 1) is isotypic with the thallium vanadate $Tl_4V_2O_7$ (= $Tl_2^{I}[Tl_2^{I}V_2^{V}O_7]$; Jouanneaux *et al.*, 1992), the structure of which was determined from powder diffraction data. $K_2Pb_2Ge_2O_7$ (Bassi & Lajzerowicz, 1965) is probably isotypic, but was first described as crystallizing in the subgroup $P\overline{3}$. A symmetry check (Le Page, 1987) and transformation to the standard setting with the help of the program *STRUCTURE TIDY* (Gelato & Parthé, 1987) shows the isotypic nature of $K_2Pb_2Ge_2O_7$ and the title compound.

The Al atoms in $K_2Al_2Sb_2O_7$ are located on the edges of the unit cell (Fig. 2) and are coordinated by four O atoms in an approximately regular tetrahedral environment, with Al—O distances of 1.702 (1) (Al—O2, ×1) and 1.762 (2) Å (Al—O1, ×3) and O—Al—O angles ranging from 108.1 (1) to 110.9 (1)° (shown as tetrahedra in Fig. 2). Two AlO_4 tetrahedra are connected by a common O2 atom to form linear $[Al_2O_7]$ dimers with a staggered conformation of the six O1 ligands (Fig. 1). These dimers are connected by Sb^{3+} ions to form layers perpendicular to the threefold axis. The Sb atoms are coordinated by three O ligands in a Ψ-tetrahedral coordination, with an Sb—O distance of 1.936 (2) Å and an O—Sb—O angle of 91.8 (1)°. The corresponding distances and angles in $CsSbO_2$ (Hirschle & Röhr, 1998), $Cs_4Sb_2O_5$ (Hirschle & Röhr, 1999) and Na_3SbO_3 (Stöver & Hoppe, 1980) are comparable to these values. They clearly indicate the stereochemical activity of the antimony(III) lone pair, which in $K_2Al_2Sb_2O_7$ points towards the centre of the layered $[Al_2Sb_2O_7]^{2-}$ anions running perpendicular to the (001) direction. The anions are bounded by oxygen kagome (3.6.3.6) nets stacked in the sequence *A*—*B*. The K^+ cations are intercalated between the $[Al_2Sb_2O_7]^{2-}$ layers, with a resulting coordination number of nine and K—O distances ranging from 2.900 (2) to 2.979 (1) Å.

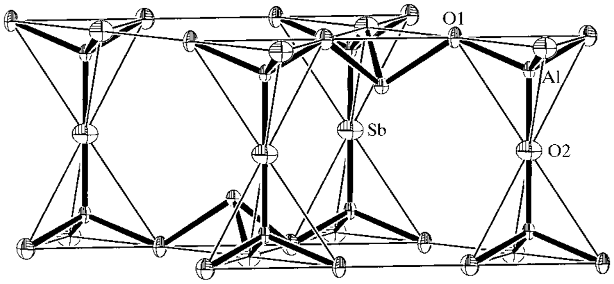

Figure 1
ORTEP (Johnson, 1968) view of the layered $[Al_2Sb_2O_7]^{2-}$ anions in the title compound. Displacement ellipsoids are shown at the 50% probability level.

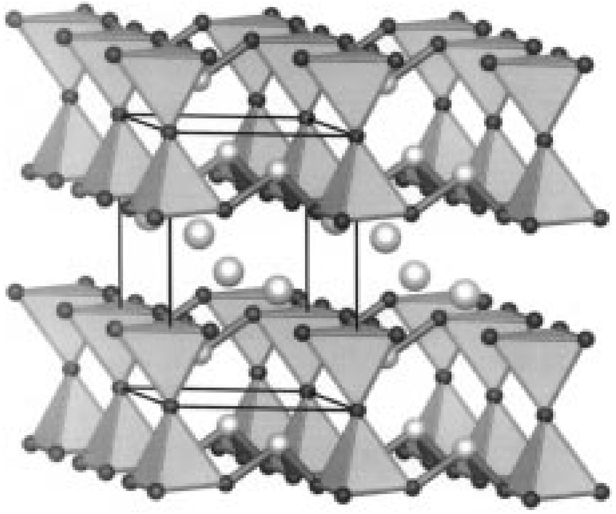

Figure 2
View of the unit cell of the title compound.

Experimental

Potassium (156 mg, 4.0 mmol; Merck, 99%) was reacted with a powdered mixture of Al_2O_3 (204 mg, 2.0 mmol; Merck, p.a.), Sb_2O_3 (292 mg, 1.0 mmol; Merck, p.a.) and Sb_2O_5 (323 mg, 1.0 mmol; ABCR, 99%) in a corundum crucible under an argon (99.99%) atmosphere. The mixture was heated to 1050 K at a rate of 100 K h^{-1} and then cooled to 590 K at 5 K h^{-1} and from 590 K to room temperature at 15 K h^{-1}. The title compound crystallizes as clear thin plates of hexagonal shape. The X-ray powder patterns of the samples can be indexed with the single-crystal data of the title compound and show only weak reflections of corundum, Sb_2O_3 and additional unknown compounds.

Crystal data

$K_2[Al_2Sb_2O_7]$
M_r = 487.66
Trigonal, $P\overline{3}m1$
a = 5.6325 (8) Å
c = 8.045 (2) Å
V = 221.04 (7) Å^3
Z = 1
D_x = 3.664 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 6.3–23.8°
μ = 7.250 mm^{-1}
T = 293 (2) K
Hexagonal plate, colourless
0.10 × 0.07 × 0.03 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{\min} = 0.589$, $T_{\max} = 0.805$
1691 measured reflections
342 independent reflections
$R_{\rm int} = 0.085$
$\theta_{\max} = 32.37°$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -12 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.018$
$wR(F^2) = 0.044$
$S = 1.140$
342 reflections
19 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0191P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.81$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.014 (3)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *HELENA* (Spek, 1993); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1968) and *DRAWxtl* (Finger & Kroeker, 1997); software used to prepare material for publication: *SHELXL*97.

Table 1
Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2).

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
K1	1/3	2/3	0.58969 (14)	0.01743 (18)
Sb1	1/3	2/3	0.15516 (3)	0.00913 (12)
Al1	0	0	0.21158 (14)	0.0081 (2)
O1	0.16874 (17)	0.83126 (17)	0.2895 (2)	0.0152 (3)
O2	0	0	0	0.0206 (9)

Table 2
Selected geometric parameters (Å, °).

K1—O1	2.900 (2)	Al1—O2	1.7022 (12)
K1—O1$^{\rm i}$	2.9792 (8)	Al1—O1$^{\rm iii}$	1.7616 (18)
Sb1—O1$^{\rm ii}$	1.9358 (17)		
O1$^{\rm ii}$—Sb1—O1$^{\rm iii}$	91.84 (7)	Al1$^{\rm v}$—O1—Sb1	125.19 (10)
O2—Al1—O1$^{\rm iii}$	110.86 (7)	Al1—O2—Al1$^{\rm vi}$	180.0
O1$^{\rm iii}$—Al1—O1$^{\rm iv}$	108.05 (7)		

Symmetry codes: (i) $1 + x - y, 1 + x, 1 - z$; (ii) $-x + y, 1 - x, z$; (iii) $1 - y, 1 + x - y, z$; (iv) $-1 - x + y, -x, z$; (v) $x, 1 + y, z$; (vi) $-x, -y, -z$.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1259). Services for accessing these data are described at the back of the journal.

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