Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.598$, $T_{\max} = 0.999$
1985 measured reflections
983 independent reflections
905 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0394$
$\theta_{\max} = 29.85°$
$h = -9 \rightarrow 1$
$k = -13 \rightarrow 11$
$l = -14 \rightarrow 1$
3 standard reflections frequency: 60 min intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0477$
$wR(F^2) = 0.0860$
$S = 4.139$
983 reflections
35 parameters
H atoms refined with common $U_{\rm iso}$
$w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = -0.319$
$\Delta\rho_{\max} = 2.27$ e Å^{-3}
$\Delta\rho_{\min} = -1.52$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0038 (10)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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	$U_{\rm eq}$
Sn	0.02310 (8)	1/4	0.52960 (4)	0.0488 (2)
Cl1	0.2001 (3)	1/4	0.3434 (2)	0.0746 (6)
Cl2	0.1692 (3)	0.4354 (2)	0.63900 (14)	0.0781 (5)
C	−0.3004 (13)	1/4	0.5314 (9)	0.073 (2)

Table 2. *Selected geometric parameters* (Å, °)

Values in square brackets are calculated bond orders.

Sn—C	2.074 (8) [1.18]	Sn—Cl2	2.318 (1) [0.90]
Sn—Cl1	2.283 (2) [1.00]	Sn—Cl2$^{\rm i}$	3.714 (2) [0.02]
C—Sn—Cl1	120.3 (3)	Cl1—Sn—Cl2	103.58 (6)
C—Sn—Cl2	113.5 (1)		

Symmetry code: (i) $-x, 1-y, 1-z$.

Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C) Tables 4.2.6.8 and 4.2.4.2, respectively.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: HU1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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mer-Tris[2-(5-bromo-2-hydroxyphenyl)-2-oxazolinato]aluminium(III) Methanol Solvate

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Abstract

The title complex, (*OC*-6-21)-tris[4-bromo-2-(4,5-dihydro-1,3-oxazol-2-yl)phenolato-*N*,*O*]aluminium(III) methanol solvate, $[Al(C_9H_7BrNO_2)_3].CH_4O$, has a slightly distorted octahedral coordination geometry with a meridional arrangement of the ligands. Pertinent Al—O and Al—N bond distances are 1.839 (2)–1.876 (2) and 2.015 (3)–2.024 (3) Å, respectively. The methanol solvent is hydrogen bonded to one of the coordinated O atoms.

Comment

The synthesis of the title compound, (I), has been published elsewhere (Hoveyda, Karunaratne, Rettig & Orvig, 1992). The meridional hexacoordinate geometry

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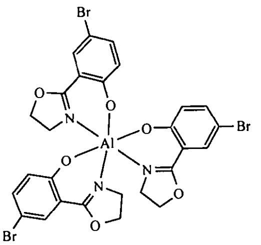
of this tris(ligand)aluminium(III) complex is very similar to that reported for five closely related Al, Ga and In derivatives (Hoveyda *et al.*, 1992), in particular that of the unsubstituted analog tris[2-(2-hydroxyphenyl)-2-oxazolinato]aluminium(III) which also crystallizes as a methanol solvate. The Al—N distances are equal within experimental error and average 2.019 (4) Å. The Al—O(2) bond [1.876 (2) Å], atom O(2) being involved in a hydrogen bond to the methanol solvent, is significantly longer than the other two Al—O bonds [both 1.839 (2) Å].



(I)

The most notable difference between the present structure and those reported earlier (Hoveyda *et al.*, 1992) is the magnitude of the displacements of the metal atom from the nearly planar —N═C—C═C—O— portions of the six-membered chelate rings [0.84, 0.41 and 0.02 Å, respectively for the rings containing atoms N(1), N(2) and N(3)]. Variations in the degree of planarity of the three chelate rings have been observed before, but the largest displacement noted was one of 0.18 Å in the structure of tris[2-(2-hydroxy-3-methylphenyl)-2-oxazolinato]aluminium(III).

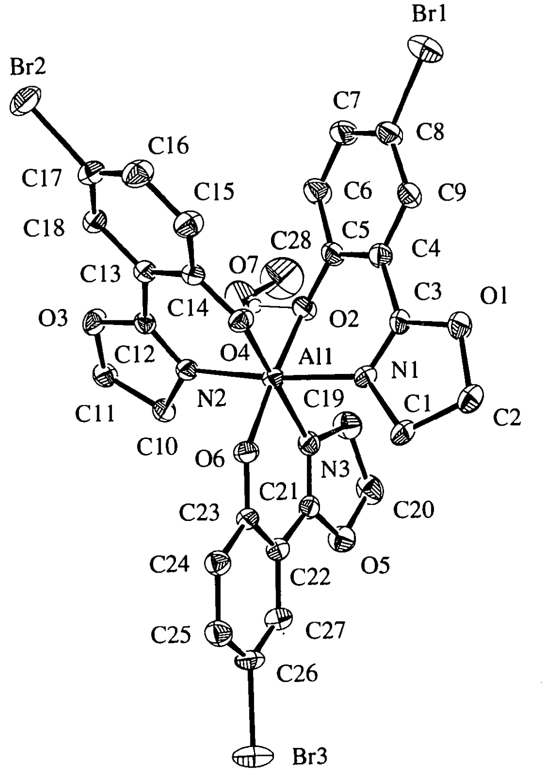


Fig. 1. View of the molecule (33% probability ellipsoids) with the fine line representing a hydrogen bond.

The methanol solvent molecule is hydrogen bonded to the aluminium complex *via* a coordinated O atom [O(7)—H(22)···O(2); O—H 0.74 (6), H···O 2.05 (6), O···O 2.791 (4) Å and O—H···O 170 (7)°]. There are three weak intermolecular C—H···O interactions, the most significant being C(2)—H(4)···O(4^{i}) [H···O 2.34, C···O 3.247 (4) Å and C—H···O 155°; symmetry code: (i) $-x$, $1-y$, $-z$]. Full details of the remaining interactions are included in the supplementary material.

Experimental

Crystal data

$[Al(C_9H_7BrNO_2)_3].CH_4O$
$M_r = 782.21$
Triclinic
$P\bar{1}$
$a = 11.978\ (1)$ Å
$b = 13.2165\ (8)$ Å
$c = 10.375\ (2)$ Å
$\alpha = 106.387\ (8)°$
$\beta = 106.95\ (1)°$
$\gamma = 75.253\ (6)°$
$V = 1480.4\ (3)$ Å^3
$Z = 2$
$D_x = 1.755$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 54.8$–$57.0°$
$\mu = 5.787$ mm^{-1}
$T = 294.0$ K
Plate
0.40 × 0.40 × 0.20 mm
Pink

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical, based on ψ scans for 3 reflections
$T_{\min} = 0.452$, $T_{\max} = 1.000$
6400 measured reflections
6400 independent reflections
5266 observed reflections [$I \geq 3\sigma(I)$]

$\theta_{\max} = 77.50°$
$h = 0 \rightarrow 15$
$k = -15 \rightarrow 16$
$l = -12 \rightarrow 11$
3 standard reflections monitored every 200 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.039$
$wR = 0.056$
$S = 3.460$
5266 reflections
384 parameters
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.66$ e Å^{-3}
$\Delta\rho_{\min} = -0.78$ e Å^{-3}

Extinction correction: Zachariasen type 2, Gaussian isotropic
Extinction coefficient: 4.0 (3) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br(1)	0.53439 (4)	0.34713 (4)	−0.21242 (5)	0.0606 (1)
Br(2)	0.20419 (5)	−0.14550 (4)	−0.53171 (5)	0.0687 (1)
Br(3)	−0.35934 (4)	0.42750 (4)	0.48365 (5)	0.0688 (1)
Al(1)	0.13485 (7)	0.24979 (6)	0.11986 (9)	0.0307 (3)
O(1)	0.2165 (2)	0.5330 (2)	0.1050 (3)	0.0466 (6)
O(2)	0.3003 (2)	0.2178 (2)	0.1421 (2)	0.0384 (5)
O(3)	0.1674 (2)	−0.0825 (2)	−0.0104 (2)	0.0440 (6)
O(4)	0.1062 (2)	0.2296 (2)	−0.0688 (2)	0.0360 (5)
O(5)	0.1091 (2)	0.3428 (2)	0.5322 (2)	0.0462 (6)
O(6)	−0.0264 (2)	0.2871 (2)	0.1047 (2)	0.0345 (5)
O(7)	0.4085 (3)	0.0319 (3)	0.2408 (5)	0.084 (1)
N(1)	0.1472 (2)	0.4037 (2)	0.1400 (3)	0.0339 (6)
N(2)	0.1400 (2)	0.0920 (2)	0.0949 (3)	0.0317 (6)
N(3)	0.1572 (2)	0.2796 (2)	0.3268 (3)	0.0351 (6)
C(1)	0.0668 (3)	0.5035 (2)	0.1862 (3)	0.0406 (8)
C(2)	0.1201 (3)	0.5919 (2)	0.1723 (4)	0.0431 (9)
C(3)	0.2246 (3)	0.4289 (2)	0.0979 (3)	0.0344 (8)
C(4)	0.3206 (3)	0.3529 (2)	0.0423 (3)	0.0370 (8)
C(5)	0.3542 (3)	0.2494 (2)	0.0693 (4)	0.0379 (8)
C(6)	0.4481 (3)	0.1798 (3)	0.0162 (5)	0.052 (1)
C(7)	0.5007 (3)	0.2093 (3)	−0.0658 (5)	0.053 (1)
C(8)	0.4632 (3)	0.3102 (3)	−0.0946 (4)	0.0444 (9)
C(9)	0.3755 (3)	0.3831 (3)	−0.0392 (3)	0.0391 (8)
C(10)	0.1363 (3)	0.0362 (2)	0.1982 (3)	0.0377 (8)
C(11)	0.1625 (3)	−0.0819 (3)	0.1283 (3)	0.0399 (8)
C(12)	0.1558 (2)	0.0203 (2)	−0.0150 (3)	0.0317 (7)
C(13)	0.1573 (3)	0.0390 (2)	−0.1459 (3)	0.0334 (8)
C(14)	0.1284 (3)	0.1448 (2)	−0.1648 (3)	0.0329 (8)
C(15)	0.1201 (3)	0.1594 (3)	−0.2972 (4)	0.0454 (9)
C(16)	0.1406 (4)	0.0738 (3)	−0.4032 (4)	0.052 (1)
C(17)	0.1700 (3)	−0.0295 (3)	−0.3825 (4)	0.0456 (9)
C(18)	0.1780 (3)	−0.0483 (2)	−0.2561 (3)	0.0412 (8)
C(19)	0.2726 (3)	0.2812 (3)	0.4300 (4)	0.052 (1)
C(20)	0.2383 (3)	0.3263 (3)	0.5674 (4)	0.052 (1)
C(21)	0.0746 (3)	0.3150 (2)	0.3933 (3)	0.0335 (8)
C(22)	−0.0528 (3)	0.3306 (2)	0.3348 (3)	0.0336 (7)
C(23)	−0.0962 (3)	0.3159 (2)	0.1900 (3)	0.0321 (7)
C(24)	−0.2200 (3)	0.3354 (3)	0.1379 (3)	0.0384 (8)
C(25)	−0.2966 (3)	0.3665 (3)	0.2236 (4)	0.0441 (9)
C(26)	−0.2521 (3)	0.3803 (3)	0.3655 (4)	0.0438 (9)
C(27)	−0.1319 (3)	0.3626 (3)	0.4218 (3)	0.0417 (9)
C(28)	0.5219 (6)	0.0412 (7)	0.3220 (8)	0.119 (3)

Table 2. *Selected geometric parameters* (Å, °)

Br(1)—C(8)	1.901 (3)	C(1)—C(2)	1.523 (5)
Br(2)—C(17)	1.905 (3)	C(3)—C(4)	1.461 (4)
Br(3)—C(26)	1.903 (3)	C(4)—C(5)	1.408 (4)
Al(1)—O(2)	1.876 (2)	C(4)—C(9)	1.397 (4)
Al(1)—O(4)	1.839 (2)	C(5)—C(6)	1.401 (4)
Al(1)—O(6)	1.839 (2)	C(6)—C(7)	1.379 (5)
Al(1)—N(1)	2.024 (3)	C(7)—C(8)	1.382 (5)
Al(1)—N(2)	2.015 (3)	C(8)—C(9)	1.374 (5)
Al(1)—N(3)	2.019 (3)	C(10)—C(11)	1.518 (4)
O(1)—C(2)	1.461 (4)	C(12)—C(13)	1.453 (4)
O(1)—C(3)	1.336 (3)	C(13)—C(14)	1.408 (4)
O(2)—C(5)	1.327 (4)	C(13)—C(18)	1.405 (4)
O(3)—C(11)	1.455 (4)	C(14)—C(15)	1.412 (4)
O(3)—C(12)	1.343 (3)	C(15)—C(16)	1.365 (5)
O(4)—C(14)	1.301 (3)	C(16)—C(17)	1.383 (5)
O(5)—C(20)	1.456 (4)	C(17)—C(18)	1.375 (5)
O(5)—C(21)	1.347 (4)	C(19)—C(20)	1.514 (5)
O(6)—C(23)	1.307 (3)	C(21)—C(22)	1.453 (4)
O(7)—C(28)	1.388 (7)	C(22)—C(23)	1.413 (4)
N(1)—C(1)	1.470 (4)	C(22)—C(27)	1.403 (4)
N(1)—C(3)	1.280 (4)	C(23)—C(24)	1.405 (4)
N(2)—C(10)	1.477 (4)	C(24)—C(25)	1.371 (5)
N(2)—C(12)	1.288 (4)	C(25)—C(26)	1.388 (5)
N(3)—C(19)	1.482 (4)	C(26)—C(27)	1.373 (5)
N(3)—C(21)	1.286 (4)		
O(2)—Al(1)—O(4)	93.0 (1)	C(5)—C(6)—C(7)	121.3 (3)
O(2)—Al(1)—O(6)	177.2 (1)	C(6)—C(7)—C(8)	120.1 (3)
O(2)—Al(1)—N(1)	86.19 (10)	Br(1)—C(8)—C(7)	119.2 (3)
O(2)—Al(1)—N(2)	87.93 (10)	Br(1)—C(8)—C(9)	120.2 (3)
O(2)—Al(1)—N(3)	90.0 (1)	C(7)—C(8)—C(9)	120.6 (3)
O(4)—Al(1)—O(6)	88.82 (10)	C(4)—C(9)—C(8)	119.4 (3)
O(4)—Al(1)—N(1)	89.2 (1)	N(2)—C(10)—C(11)	103.7 (2)
O(4)—Al(1)—N(2)	89.1 (1)	O(3)—C(11)—C(10)	104.7 (2)
O(4)—Al(1)—N(3)	176.4 (1)	O(3)—C(12)—N(2)	116.8 (3)
O(6)—Al(1)—N(1)	91.68 (10)	O(3)—C(12)—C(13)	116.3 (3)
O(6)—Al(1)—N(2)	94.2 (1)	N(2)—C(12)—C(13)	126.9 (3)
O(6)—Al(1)—N(3)	88.17 (10)	C(12)—C(13)—C(14)	119.4 (3)
N(1)—Al(1)—N(2)	173.8 (1)	C(12)—C(13)—C(18)	120.1 (3)
N(1)—Al(1)—N(3)	88.9 (1)	C(14)—C(13)—C(18)	120.3 (3)
N(2)—Al(1)—N(3)	93.1 (1)	O(4)—C(14)—C(13)	124.0 (3)
C(2)—O(1)—C(3)	107.0 (2)	O(4)—C(14)—C(15)	118.1 (3)
Al(1)—O(2)—C(5)	122.7 (2)	C(13)—C(14)—C(15)	117.9 (3)
C(11)—O(3)—C(12)	106.8 (2)	C(14)—C(15)—C(16)	121.1 (3)
Al(1)—O(4)—C(14)	131.9 (2)	C(15)—C(16)—C(17)	120.2 (3)
C(20)—O(5)—C(21)	106.7 (3)	Br(2)—C(17)—C(16)	118.4 (3)
Al(1)—O(6)—C(23)	135.9 (2)	Br(2)—C(17)—C(18)	120.5 (3)
Al(1)—N(1)—C(1)	129.1 (2)	C(16)—C(17)—C(18)	121.1 (3)
Al(1)—N(1)—C(3)	123.0 (2)	C(13)—C(18)—C(17)	119.4 (3)
C(1)—N(1)—C(3)	107.5 (2)	N(3)—C(19)—C(20)	104.0 (3)
Al(1)—N(2)—C(10)	127.8 (2)	O(5)—C(20)—C(19)	104.7 (3)
Al(1)—N(2)—C(12)	124.3 (2)	O(5)—C(21)—N(3)	116.9 (3)
C(10)—N(2)—C(12)	107.8 (2)	O(5)—C(21)—C(22)	116.0 (3)
Al(1)—N(3)—C(19)	125.5 (2)	N(3)—C(21)—C(22)	127.1 (3)
Al(1)—N(3)—C(21)	126.3 (2)	C(21)—C(22)—C(23)	119.4 (3)
C(19)—N(3)—C(21)	107.5 (3)	C(21)—C(22)—C(27)	120.2 (3)
N(1)—C(1)—C(2)	104.3 (3)	C(23)—C(22)—C(27)	120.4 (3)
O(1)—C(2)—C(1)	103.5 (2)	O(6)—C(23)—C(22)	122.8 (3)
O(1)—C(3)—N(1)	117.2 (3)	O(6)—C(23)—C(24)	119.6 (3)
O(1)—C(3)—C(4)	117.6 (3)	C(22)—C(23)—C(24)	117.6 (3)
N(1)—C(3)—C(4)	125.2 (3)	C(23)—C(24)—C(25)	121.6 (3)
C(3)—C(4)—C(5)	119.1 (3)	C(24)—C(25)—C(26)	119.9 (3)
C(3)—C(4)—C(9)	119.7 (3)	Br(3)—C(26)—C(25)	119.4 (2)
C(5)—C(4)—C(9)	121.2 (3)	Br(3)—C(26)—C(27)	119.7 (3)
O(2)—C(5)—C(4)	122.2 (3)	C(25)—C(26)—C(27)	120.9 (3)
O(2)—C(5)—C(6)	120.6 (3)	C(22)—C(27)—C(26)	119.6 (3)
C(4)—C(5)—C(6)	117.2 (3)		

The scan width was (1.10 + 0.20tanθ)° with a θ scan rate of 32° min^{-1} (up to nine scans). Stationary background counts were taken at each end of the scan; scan time/background time ratio 2:1. The hydroxy H atom of the methanol solvent molecule was refined with an isotropic displacement parameter and the remaining H atoms were fixed in calculated positions (C—H = 0.97 Å, $U = 1.2 \times U$ of the bonded atom).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1985). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the Natural Sciences and Engineering Research Council of Canada and the US Public Health Service (CA 48964) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, least-squares-planes data and dihedral angles, torsion angles, non-bonded contacts and hydrogen-bond geometry have been deposited with the IUCr (Reference: BK1052). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Guanidinium Pentafluoroantimonate

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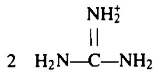
Abstract

The structure of diguanidinium pentafluoroantimonate, $(CH_6N_3)_2[SbF_5]$, has been determined and refined to $R = 2.3\%$. The SbF_5^{2-} anion is a distorted rectangular pyramid with F atoms at the vertices and a mirror plane C_s through atoms F(1) and Sb. The two inequivalent guanidinium ions are planar and display only mirror symmetry, not C_3, as expected. The crystal structure consists of two sets of guanidinium sheets linked by N—H···F hydrogen bonds to pentafluoroantimonate ions.

Comment

Various guanidinium salts such as guanidinium hexafluorogallate (Małuszyńska, 1993; Kozak, Grottel & Pająk, 1993) and guanidinium nitrate (Katrusiak & Szafrański, 1993; Wąsicki, Grottel & Pająk, 1994) have been studied by X-ray and NMR methods. In the crystal structures of these salts, different types of ion motions and phase transitions in a large temperature range have been observed. The guanidinium ion usually undergoes a reorientation around the C_3 symmetry axis. The motions of the anions depend greatly on their symmetry, shape and environment.

In the title compound, (I), the SbF_5^{2-} ion is a distorted rectangular pyramid and its geometry agrees with that reported by Ryan & Cromer (1972). It has only C_s crystallographic symmetry with the Sb atom 0.395 Å below the plane of the basal F(2) and F(3) atoms. The axial Sb—F(1) distance is significantly shorter than the basal Sb—F(2) and Sb—F(3) distances, as observed in ions and molecules isoelectronic with SbF_5^{2-} (Ryan & Cromer, 1972, and references therein). The pyramids are related by a center of symmetry with an Sb···Sb distance of 4.191 Å.



$$2\ \overset{NH_2^+}{\overset{\|}{H_2N\text{—}C\text{—}NH_2}}\quad [SbF_5]^{2-}$$

(I)

The two inequivalent guanidinium cations are planar and display only crystallographic mirror symmetry perpendicular to the guanidinium plane. All H atoms are involved in rather strong N—H···F hydrogen bonds (see Table 3). The axial F(1) atom is the weakest hydrogen-bond acceptor. The N···F distances of the basal F atoms range from 2.827 to 2.984 Å, while those of the axial F(1) atom range from 3.209 to 3.226 Å.

The packing in the crystal structure, shown in Fig. 2, consists of two sets of guanidinium sheets interconnected through the SbF_5^{2-} ions by hydrogen bonds.

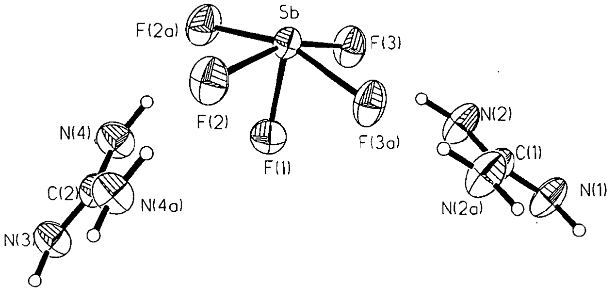


Fig. 1. *ORTEP* (Johnson, 1965) drawing of guanidinium pentafluoroantimonate with displacement ellipsoids at the 50% probability level.

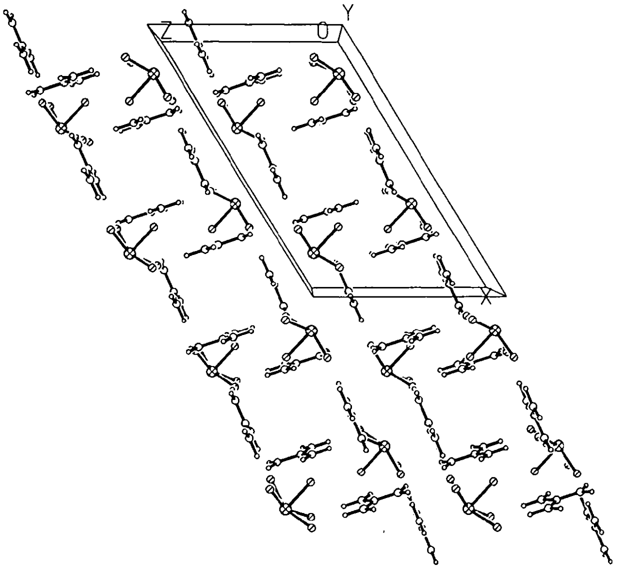


Fig. 2. Molecular packing diagram of the title compound viewed down the *y* axis.

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