Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Diethylenetriaminium(3+) hexachlorobismuthate(III)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.010 Å
R factor = 0.031
wR factor = 0.081
Data-to-parameter ratio = 25.6

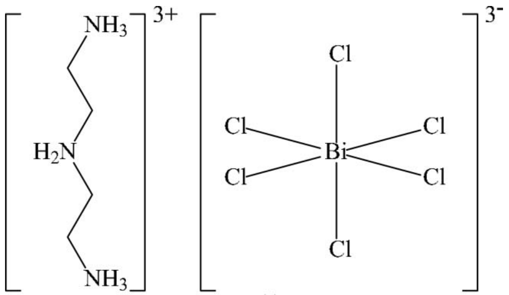
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $(C_4H_{16}N_3)[BiCl_6]$, the Bi^{III} atom is six-coordinate in a distorted octahedral geometry. The salt adopts a three-dimensional network arising from the hydrogen bonds between the cations and anions.

Received 18 July 2005
Accepted 29 July 2005
Online 6 August 2005

Comment

Ammonium chlorobismuthates(III) were first reported in 1982 (Remy & Pellens, 1928); such compounds are still of current interest owing to their ability to undergo phase transformations (Jakubas *et al.*, 1997; Zarychta *et al.*, 2004). Among the ammonium hexachlorobismuthates that have been crystallographically authenticated are the tris(dimethylammonium) hexachlorobismuthate–dimethylammonium chloride co-crystal (Herdtweck & Kreusel, 1993), tris(diethylammonium) hexachlorobismuthate (Lazarini, 1987; Jarraya *et al.*, 1993), sesqui(ethylenediammonium)hexachlorobismuthate dihydrate (Davidovich *et al.*, 1996) and the tri(pyridinium) hexachlorobismuthate–potassium hexachlorobismuthate co-crystal (Zhang & Fang, 2004).



(I)

The diethylenetriaminium analog, (I) (Fig. 1) is another example. The geometry, like those of the reported ammonium salts, is distorted octahedral; hydrogen bonds (Table 2) link the cations and anions into a three-dimensional network structure. The distortion of geometry can probably be attributed to packing as the cation is not able to occupy a special position in the crystal structure owing to its irregular shape. The packing contrasts with that of the inorganic salt, cesium hexachlorobismuthate, which is cubic (Von der Mühll *et al.*, 1982).

Experimental

Bismuth trichloride (3.15 g, 10.0 mmol) was dissolved in 4 *M* hydrochloric acid (30 ml). Diethylenetriamine (0.8 ml, 7.0 mmol) was added to the solution. Colorless crystals separated from the solution in about 80% yield after a day.

Crystal data

$(C_4H_{16}N_3)[BiCl_6]$
$M_r = 527.88$
Orthorhombic, $P2_12_12_1$
$a = 6.8466$ (3) Å
$b = 15.6680$ (8) Å
$c = 13.6328$ (7) Å
$V = 1462.4$ (1) Å^3
$Z = 4$
$D_x = 2.398$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 6004 reflections
$\theta = 3.0$–$28.7°$
$\mu = 13.12$ mm^{-1}
$T = 295$ (2) K
Block, colorless
0.26 × 0.15 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.138$, $T_{\max} = 0.420$
12004 measured reflections

3302 independent reflections
3119 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 8$
$k = -19 \rightarrow 20$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.02$
3302 reflections
129 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.89$ e Å^{-3}
Absolute structure: Flack (1983), 1379 Friedel pairs
Flack parameter: −0.034 (7)

Table 1
Selected geometric parameters (Å, °).

Bi1—Cl1	2.705 (2)	Bi1—Cl4	2.826 (2)
Bi1—Cl2	2.636 (2)	Bi1—Cl5	2.769 (2)
Bi1—Cl3	2.729 (2)	Bi1—Cl6	2.625 (2)
Cl1—Bi1—Cl2	91.33 (5)	Cl2—Bi1—Cl6	87.90 (6)
Cl1—Bi1—Cl3	176.08 (6)	Cl3—Bi1—Cl4	89.04 (5)
Cl1—Bi1—Cl4	90.12 (5)	Cl3—Bi1—Cl5	98.05 (6)
Cl1—Bi1—Cl5	85.87 (5)	Cl3—Bi1—Cl6	90.32 (7)
Cl1—Bi1—Cl6	85.85 (6)	Cl4—Bi1—Cl6	90.03 (6)
Cl2—Bi1—Cl3	89.36 (5)	Cl4—Bi1—Cl5	98.19 (5)
Cl2—Bi1—Cl4	177.37 (5)	Cl5—Bi1—Cl6	168.33 (7)
Cl2—Bi1—Cl5	84.10 (5)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$a\cdots$Cl3$^{\rm i}$	0.90	2.34	3.188 (6)	157
N1—H1$b\cdots$Cl6$^{\rm ii}$	0.90	2.63	3.182 (6)	121
N1—H1$c\cdots$Cl3$^{\rm iii}$	0.90	2.48	3.191 (6)	137
N2—H2$a\cdots$Cl4	0.90	2.45	3.259 (6)	150
N2—H2$b\cdots$Cl1$^{\rm iv}$	0.90	2.57	3.224 (6)	130
N3—H3$a\cdots$Cl4$^{\rm iv}$	0.90	2.52	3.288 (6)	143
N3—H3$a\cdots$Cl2$^{\rm v}$	0.90	2.66	3.250 (6)	124
N3—H3$b\cdots$Cl1$^{\rm vi}$	0.90	2.59	3.305 (6)	137
N3—H3$c\cdots$Cl5$^{\rm v}$	0.90	2.39	3.254 (5)	161

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x-\frac{1}{2}, -y+2, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, -y+2, z-\frac{1}{2}$; (iv) $x+1, y, z$; (v) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (vi) $-x+\frac{1}{2}, -y+2, z+\frac{1}{2}$.

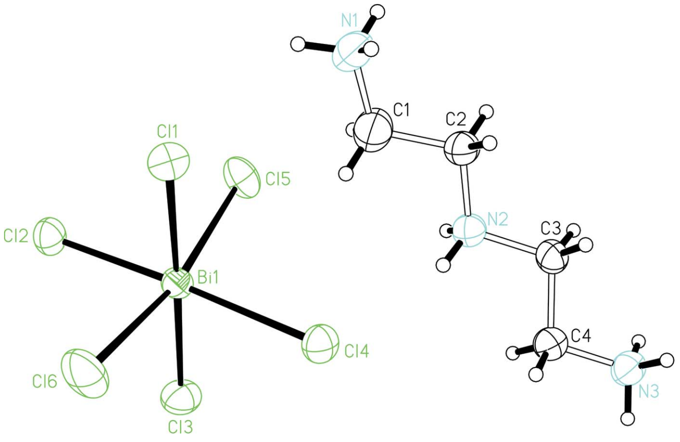


Figure 1
An *ORTEPII* plot (Johnson, 1976) of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

H atoms were positioned geometrically [C—H = 0.97 (for CH) and N—H = 0.90 Å] and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}(C,N)$. The ammonium group was rotated to fit the electron density. The highest residual electron density is located about 1 Å from Bi1. The components of the anisotropic displacement parameters of Bi1 and Cl5 appear to be somewhat unequal along the bond, as noted from the Hirshfeld test (Hirshfeld, 1976). They are also somewhat unequal for the Bi1—Cl4 bond. However, there is no indication of disorder of atoms Cl4 and Cl5.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors thank the Natural Scientific Foundation Committee of Shanxi Province (No. 20041031) and the University of Malaya for generously supporting this study.

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