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Bis[*N*-methylpiperidinium(2+)] di-μ-chloro-bis[tetrachlorobismuthate(III)] dihydrate

Yun-Long Fu,[a] Zhi-Wei Xu,[a] Jia-Lin Ren[a] and Seik Weng Ng[b]*

[a]School of Chemistry and Materials Science, Shanxi Normal University, Linfen 041004, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

In the title compound, $(C_5H_{14}N_2)_2[Bi_2Cl_{10}]\cdot 2H_2O$, each Bi^{III} atom of the centrosymmetric anion is six-coordinate in a distorted octahedral geometry. The salt adopts a three-dimensional network arising from the hydrogen bonds between the cations, anions and water molecules.

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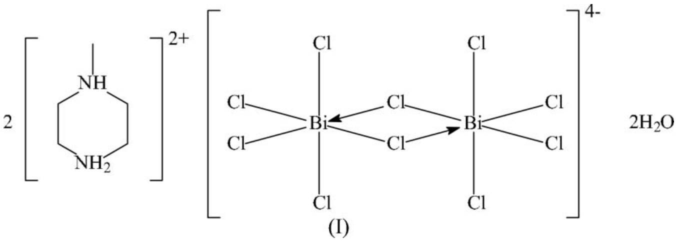
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
R factor = 0.027
wR factor = 0.064
Data-to-parameter ratio = 25.1

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

Comment

The preceding report (Fu *et al.*, 2005) describes the isolation of diethylenetriammonium hexachlorobismuthate from the reaction of diethylenetriamine and bismuth trichloride. The use of the diamine *N*-methylpiperazine in place of the triamine yielded bis(*N*-methylpiperidinium) decachlorodibismuthate dihydrate, (I) (Fig. 1), the dinuclear dianion of which lies on a special position of $\overline{1}$ site symmetry in the crystal structure.



(I)

Unlike the large number of hexachlorobismuthates that have been crystallographically verified, there are only a few examples of decachlorobismuthates, as exemplified by the inorganic tetrahydrated potassium (Volkova *et al.*, 1983) and the trihydrated bis(piperazinium) (Wu *et al.*, 2005) salts. In these structures, the dianion consists of two edge-sharing

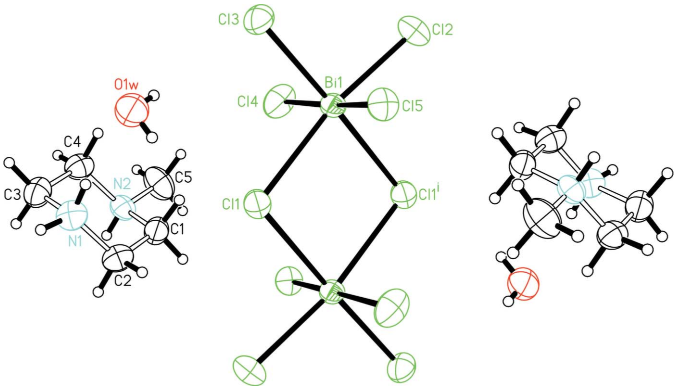


Figure 1
An *ORTEPII* plot (Johnson, 1976) of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code (i) as in Table 1; this symmetry code also generates the unlabelled atoms.]

$BiCl_6$ octahedra. In the present compound, hydrogen bonds link the cation, anion and water molecules into a three-dimensional network.

Experimental

Bismuth trichloride (3.15 g, 10 mmol) was dissolved in 4 *M* hydrochloric acid (30 ml). *N*-Methylpiperazine (1.0 ml, 10.0 mmol) was added to the solution. Colourless crystals separated from the solution in about 70% yield after 4 h.

Crystal data

$(C_5H_{14}N_2)_2[Bi_2Cl_{10}]\cdot 2H_2O$
$M_r = 1012.86$
Monoclinic, $P2_1/n$
$a = 7.7443$ (6) Å
$b = 18.550$ (2) Å
$c = 9.9726$ (8) Å
$\beta = 92.925$ (1)°
$V = 1430.8$ (2) Å^3
$Z = 2$
$D_x = 2.351$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3612 reflections
$\theta = 2.2$–28.2°
$\mu = 13.23$ mm^{-1}
$T = 295$ (2) K
Needle, colourless
0.31 × 0.09 × 0.06 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.183$, $T_{max} = 0.504$
9046 measured reflections
3219 independent reflections
2724 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 7$
$k = -21 \rightarrow 23$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.064$
$S = 1.01$
3219 reflections
128 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.78$ e Å^{-3}
$\Delta\rho_{min} = -0.93$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Bi1—Cl1	2.857 (1)	Bi1—Cl3	2.608 (1)
Bi1—Cl1^i	2.885 (1)	Bi1—Cl4	2.615 (1)
Bi1—Cl2	2.590 (1)	Bi1—Cl5	2.755 (1)
Cl1—Bi1—Cl1^i	85.28 (4)	Cl1^i—Bi1—Cl5	100.13 (4)
Cl1—Bi1—Cl2	170.19 (4)	Cl2—Bi1—Cl3	93.64 (5)
Cl1—Bi1—Cl3	94.49 (4)	Cl2—Bi1—Cl4	94.60 (5)
Cl1—Bi1—Cl4	91.17 (5)	Cl2—Bi1—Cl5	89.05 (4)
Cl1—Bi1—Cl5	86.12 (4)	Cl3—Bi1—Cl4	87.97 (4)
Cl1^i—Bi1—Cl2	87.17 (4)	Cl3—Bi1—Cl5	85.46 (4)
Cl1^i—Bi1—Cl3	174.37 (4)	Cl4—Bi1—Cl5	172.67 (4)
Cl1^i—Bi1—Cl4	86.42 (4)		

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*2···Cl2^{ii}	0.87	2.72	3.360 (4)	131
N1—H1*n*1···O1*w*	0.90	1.90	2.781 (6)	167
N1—H1*n*2···Cl5^{iii}	0.90	2.47	3.248 (5)	145
N2—H2*n*···Cl3^{iv}	0.90	2.54	3.281 (4)	140

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

H atoms were positioned geometrically [C—H = 0.97 (CH_2) or 0.96 Å (CH_3), O—H = 0.85 or 0.87 Å (OH_2) and N—H = 0.90 Å (NH)] and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2$ (1.5 for methyl) times $U_{eq}(C,N,O)$. The highest residual electron density is located about 1 Å from Bi1. The components of the anisotropic displacement parameters of Bi1 and Cl1 appear to be somewhat unequal, as noted from the Hirshfeld (1976) test. However, there is no indication of disorder of atom Cl1.

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*.

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References

Bruker (2002). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fu, Y. -L., Xu, Z. -W., Ren, J. -L. & Ng, S. W. (2005). *Acta Cryst.* E**61**, m1717–m1718.
Hirshfeld, F. L. (1976). *Acta Cryst.* A**32**, 239–244.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Volkova, L. M., Udovenko, A. A., Levin, A. N. & Shevchenko, V. Ya. (1983). *Koord. Khim.* **9**, 356–360. (In Russian.)
Wu, P.-F., Tan, X.-F., Meng, X.-G., Li, D.-S., Zhu, Y.-L. & Wei, Y.-G. (2005). *Acta Cryst.* E**61**, m1506–m1508.

m1720 Fu *et al.* • $(C_5H_{14}N_2)_2[Bi_2Cl_{10}]\cdot 2H_2O$ *Acta Cryst.* (2005). E**61**, m1719–m1720