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catena-Poly[[[3,7-bis(dimethylamino)phenothiazonium] lead(II)-tri-μ-iodo] *N*,*N*′-dimethylformamide]

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In the title compound, $\{(C_{16}H_{18}N_3OS)[PbI_3]\cdot C_3H_7NO\}_n$, each Pb^{II} atom is bonded to six I atoms to form chains of *trans* face-shared PbI_6 octahedra. In the crystal structure, the cations are stacked along the *c* axis, with significant π–π interactions.

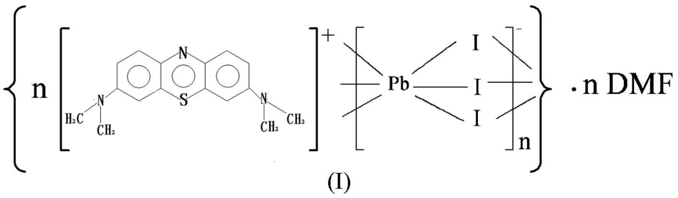
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.014 Å
R factor = 0.037
wR factor = 0.072
Data-to-parameter ratio = 22.7

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

Comment

Organic–inorganic hybrid coordination polymers containing iodoplumbate have been studied extensively due to their variety of applications (Krautscheid *et al.*, 2001; Tang *et al.*, 2001). We report here the crystal structure of such a compound, (MB)[PbI_3]·DMF (MB is methyl blue and DMF is *N*,*N*′-dimethylformamide), (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The anion consists of chains of *trans* face-shared PbI_6 octahedra. The Pb—I distances range from 3.1234 (9) to 3.3827 (10) Å, with an average value of 3.2624 Å, which is in agreement with that reported for [Me_4N][PbI_3] (3.22 Å; Contreras *et al.*, 1983). However, the Pb—I—Pb angles in (I) (Table 1) are narrower than those observed in [$Na_4(DMF)_{14}$][PbI_3]$_4$, in which the angles lie in the range 81.98 (3)–91.13 (3)° (Krautscheid *et al.*, 2001).

In the crystal structure of (I), the methyl blue cations, which counter-balance the charge on the inorganic anion, are

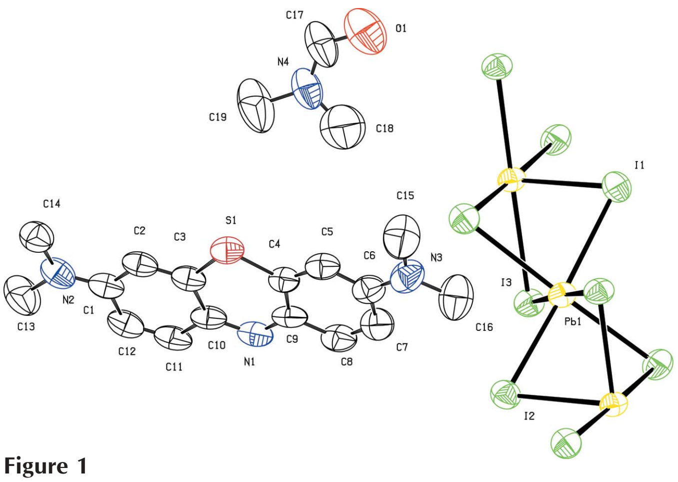


Figure 1
View of (I), showing the atom-labelling and 50% probability displacement ellipsoids. H atoms have been omitted.

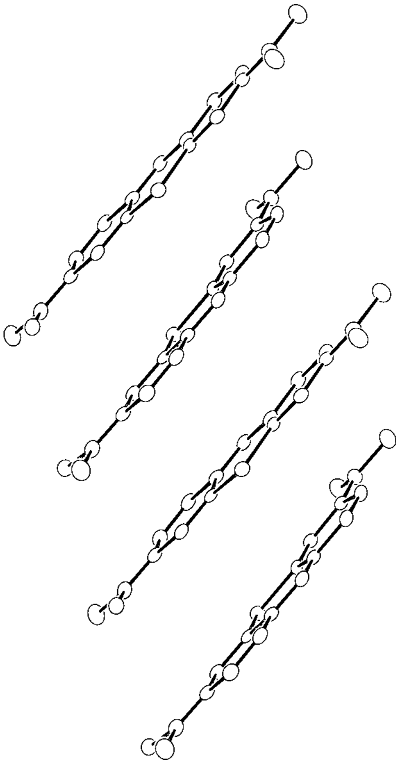


Figure 2
The crystal packing of the cations of (I), viewed along the c axis. H atoms have been omitted.

stacked along the c axis, with $Cg1\cdots Cg2^{\rm i}$ and $Cg1\cdots Cg3^{\rm ii}$ distances of 3.494 (5) and 3.579 (5) Å, respectively [$Cg1$, $Cg2$ and $Cg3$ are the S1/N1/C3/C4/C9/C10, C1–C3/C10–C12 and C4–C9 ring centroids; symmetry codes: (i) $x, 2-y, z+\frac{1}{2}$; (ii) $x, 2-y, z-\frac{1}{2}$], indicating significant π–π interaction (Fig. 2). The anionic chain has no significant hydrogen-bonding interactions with the organic cations. The iodoplumbate chains exist in the cavities formed by the methyl blue cations and the DMF molecules.

Experimental

PbI_2 (230 mg, 0.5 mmol) and NaI (188 mg, 1.25 mmol) were dissolved in acetone (50 ml) and the mixture was stirred for 2 h at room temperature. The resulting yellow solution was filtered off and added to a DMF solution of $C_{16}H_{18}N_3OS^+\cdot Cl^-\cdot 3H_2O$ (320 mg, 0.5 mmol) and the mixture was stirred at 343 K for 5 h. Propan-2-ol vapour was allowed to diffuse into the blue solution. Over the course of two weeks, dark-blue crystals of (I) formed.

Crystal data

$(C_{16}H_{18}N_3OS)[PbI_3]\cdot C_3H_7NO$
$M_r = 945.38$
Monoclinic, Cc
$a = 23.458$ (7) Å
$b = 13.923$ (4) Å
$c = 8.228$ (2) Å
$\beta = 101.163$ (5)°
$V = 2636.5$ (13) Å^3
$Z = 4$
$D_x = 2.382$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4580 reflections
$\theta = 2.9$–24.8°
$\mu = 10.01$ mm^{-1}
$T = 292$ (2) K
Block, dark blue
0.20 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{\rm min} = 0.203$, $T_{\rm max} = 0.368$
14424 measured reflections
6078 independent reflections
4912 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\rm max} = 28.0°$
$h = -30 \rightarrow 30$
$k = -18 \rightarrow 18$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.072$
$S = 0.93$
6078 reflections
268 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.010$
$\Delta\rho_{\rm max} = 1.47$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.72$ e Å^{-3}
Absolute structure: Flack (1983); 2937 Friedel pairs
Flack parameter: 0.013 (6)

Table 1
Selected geometric parameters (Å, °).

Pb1—I3	3.1234 (9)	Pb1—I2$^{\rm ii}$	3.3391 (11)
Pb1—I1$^{\rm i}$	3.1862 (10)	Pb1—I1	3.3510 (11)
Pb1—I2	3.1920 (11)	Pb1—I3$^{\rm i}$	3.3827 (10)
I3—Pb1—I1$^{\rm i}$	93.83 (3)	I2$^{\rm ii}$—Pb1—I1	81.02 (3)
I3—Pb1—I2	94.50 (3)	I3—Pb1—I3$^{\rm i}$	177.76 (2)
I1$^{\rm i}$—Pb1—I2	85.90 (3)	I1$^{\rm i}$—Pb1—I3$^{\rm i}$	84.51 (3)
I3—Pb1—I2$^{\rm ii}$	85.68 (3)	I2—Pb1—I3$^{\rm i}$	83.89 (3)
I1$^{\rm i}$—Pb1—I2$^{\rm ii}$	177.98 (3)	I2$^{\rm ii}$—Pb1—I3$^{\rm i}$	96.02 (3)
I2—Pb1—I2$^{\rm ii}$	96.10 (3)	I1—Pb1—I3$^{\rm i}$	95.66 (3)
I3—Pb1—I1	86.03 (3)	Pb1$^{\rm ii}$—I1—Pb1	77.99 (3)
I1$^{\rm i}$—Pb1—I1	96.99 (4)	Pb1—I2—Pb1$^{\rm i}$	78.09 (3)
I2—Pb1—I1	177.03 (3)	Pb1—I3—Pb1$^{\rm ii}$	78.37 (2)

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

The H atoms were placed in idealized positions, with C—H distances of 0.93–0.96 Å, and constrained to ride on their parent atoms, with $U_{\rm iso}(H) = 1.5U_{\rm eq}(C)$ for methyl H atoms and $1.2U_{\rm eq}(C)$ for the other H atoms. The highest peak in the final difference map is located 0.95 Å from Pb1.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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