

1-Aminopyridinium triiodidoplumbate(II)

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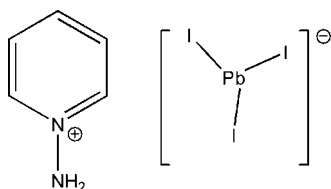
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.013$ Å; disorder in main residue; R factor = 0.025; wR factor = 0.056; data-to-parameter ratio = 26.3.

The title complex, $(\text{C}_5\text{H}_7\text{N}_2)[\text{PbI}_3]$, consists of a 1-aminopyridinium cation, disordered about a mirror plane, and a $[\text{PbI}_3]^-$ anion. The Pb^{2+} ion (site symmetry $\bar{1}$) is surrounded by six I atoms in a slightly distorted octahedral coordination. The PbI_6 octahedra share faces, building up ${}^1[\text{PbI}_{6/2}]$ chains running along [010]. The cations are situated between the chains. Coulombic attractions and van der Waals interactions between the inorganic and organic components are mainly responsible for the cohesion of the structure.

Related literature

For background to hybrid materials, see: Rogow *et al.* (2010); Thirumurugan & Rao (2008). For structures with lead halide building blocks, see: Li *et al.* (2008); Zhang *et al.* (2008).



Experimental

Crystal data

$(\text{C}_5\text{H}_7\text{N}_2)[\text{PbI}_3]$
 $M_r = 683.03$
 Orthorhombic, $Pnma$
 $a = 15.0417$ (19) Å
 $b = 8.1316$ (10) Å
 $c = 10.5625$ (14) Å

$V = 1291.9$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 20.18$ mm⁻¹
 $T = 296$ K
 $0.6 \times 0.2 \times 0.1$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{\min} = 0.011$, $T_{\max} = 0.133$

10792 measured reflections
 1607 independent reflections
 1263 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.056$
 $S = 1.11$
 1607 reflections
 61 parameters

1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.86$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.05$ e Å⁻³

Table 1

Selected bond lengths (Å).

Pb1—I3	3.2301 (5)	Pb1—I2	3.2412 (5)
Pb1—I1	3.2303 (5)		

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2395).

References

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supplementary materials

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Comment

Inorganic metal-halide building-blocks have received special attention with respect to the construction of inorganic-organic hybrid materials (Rogow *et al.*, 2010; Thirumurugan *et al.*, 2008). Among these materials octahedral building blocks of lead halides are frequently found and numerous crystal structures, from one-dimensional chains to three-dimensional frameworks (Li *et al.*, 2008; Zhang *et al.*, 2008), were observed. Herein we report the crystal structure of the title compound, (C₅H₇N₂)[PbI₃] (I).

Compound (I) crystallizes with one [PbI₃]⁻ anion and one 1-aminopyridinium cation in the asymmetric unit (Fig. 1). The cation is disordered about a mirror plane. The Pb²⁺ cation is coordinated by six iodide anions in a slightly distorted octahedral coordination geometry. The PbI₆ octahedra share faces, resulting in anionic chains running along [010]. As shown in Fig. 2., the straight inorganic chains are embedded in cationic stacks. Besides Coulomb attractions, only weak van der Waals interactions between the inorganic and organic components exist.

Experimental

A mixture of PbI₂ (922 mg, 2.0 mmol) and 1-aminopyridinium iodide (190 mg, 2.0 mmol) in a molar ratio of 1:1 in DMF was slowly evaporated to produce orange needle-shaped crystals.

Refinement

The H atoms were placed in geometrically idealized positions and refined as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The cation is disordered about a mirror plane. Atoms C3 and N1 occupy the same site with an occupation factor of 50%. The respective —NH₂ group and the H atom show likewise half-occupation.

Figures

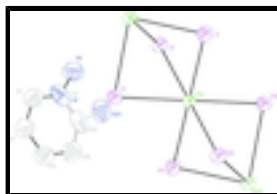


Fig. 1. Part of the structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. Atom C3 and N1 are positionally disordered. H atoms have been omitted for clarity. [Symmetry code: A $x, 0.5 - y, z$.]

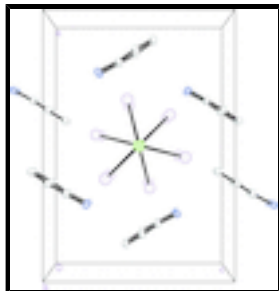


Fig. 2. The alignment of inorganic and organic components in the crystals of I along [010].

1-Aminopyridinium triiodidoplumbate(II)

Crystal data

(C₅H₇N₂)[PbI₃]

M_r = 683.03

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

a = 15.0417 (19) Å

b = 8.1316 (10) Å

c = 10.5625 (14) Å

V = 1291.9 (3) Å³

Z = 4

F(000) = 1168

D_x = 3.512 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

μ = 20.18 mm⁻¹

T = 296 K

Needle, orange

0.6 × 0.2 × 0.1 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

phi and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2000)

T_{min} = 0.011, *T_{max}* = 0.133

10792 measured reflections

1607 independent reflections

1263 reflections with *I* > 2σ(*I*)

R_{int} = 0.036

θ_{max} = 27.6°, θ_{min} = 2.4°

h = -19→19

k = -10→10

l = -13→13

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.025

wR(*F*²) = 0.056

S = 1.11

1607 reflections

61 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.016*P*)² + 2.8133*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.86 e Å⁻³

Δρ_{min} = -1.05 e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Pb1	0.5000	0.5000	0.5000	0.05028 (11)	
I1	0.66188 (4)	0.2500	0.44218 (7)	0.06995 (19)	
I2	0.45861 (4)	0.2500	0.73162 (5)	0.05961 (16)	
I3	0.38187 (5)	0.2500	0.33215 (6)	0.06837 (19)	
C1	0.0996 (6)	0.1682 (10)	0.4247 (8)	0.100 (3)	
H1	0.0708	0.1100	0.3609	0.120*	
C2	0.1419 (7)	0.0883 (12)	0.5186 (9)	0.103 (3)	
H2	0.1424	-0.0261	0.5197	0.123*	
C3	0.1824 (5)	0.1708 (11)	0.6087 (7)	0.100 (3)	0.50
H3	0.2114	0.1142	0.6731	0.120*	0.50
N1	0.1824 (5)	0.1708 (11)	0.6087 (7)	0.100 (3)	0.50
N2	0.2230 (10)	0.132 (2)	0.7177 (16)	0.121 (5)	0.50
H2A	0.2415	0.2091	0.7672	0.146*	0.50
H2B	0.2307	0.0310	0.7382	0.146*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0585 (2)	0.03787 (16)	0.05448 (19)	0.00017 (13)	0.00130 (15)	-0.00097 (14)
I1	0.0549 (3)	0.0611 (4)	0.0938 (5)	0.000	0.0189 (3)	0.000
I2	0.0727 (4)	0.0575 (3)	0.0486 (3)	0.000	0.0023 (3)	0.000
I3	0.0753 (4)	0.0603 (4)	0.0696 (4)	0.000	-0.0268 (3)	0.000
C1	0.120 (7)	0.100 (6)	0.081 (5)	-0.001 (5)	-0.015 (5)	-0.010 (5)
C2	0.104 (7)	0.089 (6)	0.114 (8)	-0.005 (5)	0.008 (6)	0.004 (6)
C3	0.064 (4)	0.162 (9)	0.074 (5)	0.010 (4)	0.010 (3)	0.026 (5)
N1	0.064 (4)	0.162 (9)	0.074 (5)	0.010 (4)	0.010 (3)	0.026 (5)
N2	0.111 (11)	0.121 (12)	0.133 (13)	0.004 (10)	-0.004 (11)	0.029 (11)

Geometric parameters (\AA , $^\circ$)

Pb1—I3 ⁱ	3.2301 (5)	C1—C1 ⁱⁱⁱ	1.331 (16)
Pb1—I3	3.2301 (5)	C1—C2	1.345 (12)

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Pb1—I1	3.2303 (5)	C1—H1	0.9300
Pb1—I1 ⁱ	3.2303 (5)	C2—C3	1.315 (11)
Pb1—I2	3.2412 (5)	C2—H2	0.9300
Pb1—I2 ⁱ	3.2412 (5)	C3—C3 ⁱⁱⁱ	1.288 (18)
I1—Pb1 ⁱⁱ	3.2303 (5)	C3—H3	0.9300
I2—Pb1 ⁱⁱ	3.2412 (5)	N2—H2A	0.8600
I3—Pb1 ⁱⁱ	3.2301 (5)	N2—H2B	0.8600
I3 ⁱ —Pb1—I3	180.0	I2—Pb1—I2 ⁱ	180.0
I3 ⁱ —Pb1—I1	94.886 (17)	Pb1 ⁱⁱ —I1—Pb1	78.000 (17)
I3—Pb1—I1	85.114 (17)	Pb1 ⁱⁱ —I2—Pb1	77.688 (16)
I3 ⁱ —Pb1—I1 ⁱ	85.114 (17)	Pb1—I3—Pb1 ⁱⁱ	78.007 (16)
I3—Pb1—I1 ⁱ	94.886 (17)	C1 ⁱⁱⁱ —C1—C2	118.9 (6)
I1—Pb1—I1 ⁱ	180.0	C1 ⁱⁱⁱ —C1—H1	120.6
I3 ⁱ —Pb1—I2	94.940 (16)	C2—C1—H1	120.6
I3—Pb1—I2	85.060 (16)	C3—C2—C1	120.4 (9)
I1—Pb1—I2	83.844 (15)	C3—C2—H2	119.8
I1 ⁱ —Pb1—I2	96.156 (15)	C1—C2—H2	119.8
I3 ⁱ —Pb1—I2 ⁱ	85.060 (16)	C3 ⁱⁱⁱ —C3—C2	120.7 (6)
I3—Pb1—I2 ⁱ	94.940 (16)	C3 ⁱⁱⁱ —C3—H3	119.7
I1—Pb1—I2 ⁱ	96.156 (15)	C2—C3—H3	119.7
I1 ⁱ —Pb1—I2 ⁱ	83.844 (15)	H2A—N2—H2B	120.0

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, y-1/2, -z+1$; (iii) $x, -y+1/2, z$.

Fig. 1

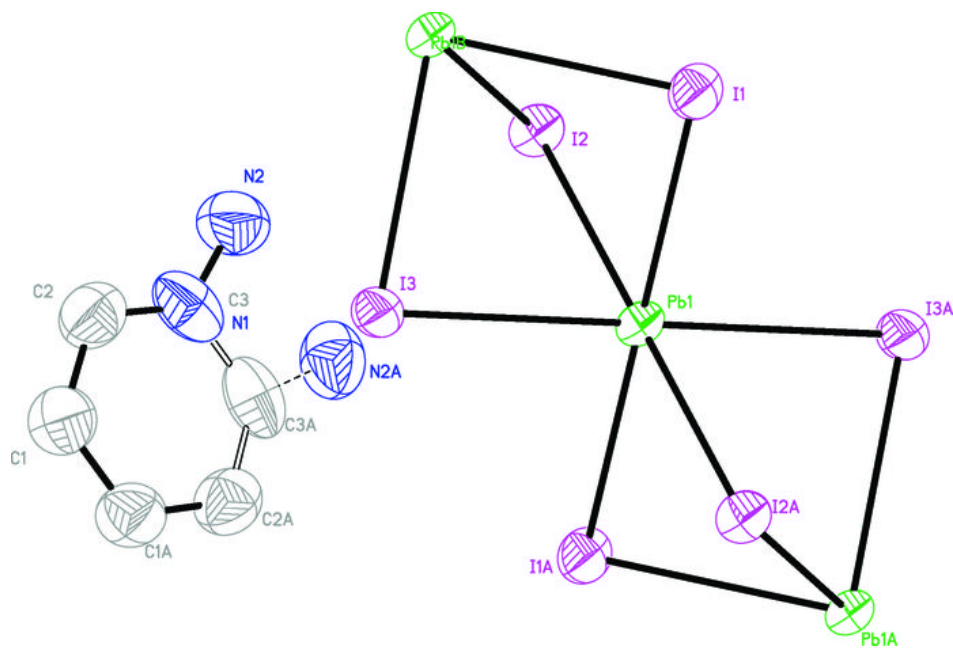


Fig. 2

