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NH_4PbI_3

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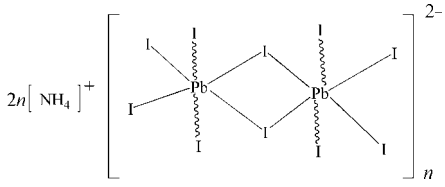
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(b–I) = 0.001 Å; R factor = 0.033; wR factor = 0.083; data-to-parameter ratio = 27.7.

Single crystals of ammonium lead iodide, NH_4PbI_3, have been obtained by solid-state reactions. Each Pb^{II} ion is coordinated by six I^- ions in a distorted octahedral environment. PbI_6 octahedra are connected by common edges, forming a one-dimensional anion chain. All atoms except one H atom in a general position have site symmetry *m*.

Related literature

For related literature, see: Fan, Chen & Wu (2006); Fan, Wu & Chen (2006); Guloy *et al.* (2001); Krautscheid *et al.* (2001); Mitzi *et al.* (1995).



Experimental

Crystal data

NH_4PbI_3
M_r = 605.94
Orthorhombic, *Pnma*
a = 10.3029 (14) Å
b = 4.7411 (5) Å
c = 17.288 (2) Å
V = 844.48 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 30.84 mm^{-1}
T = 293 (2) K
0.10 × 0.08 × 0.05 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.064, T_{max} = 0.214
6247 measured reflections
1080 independent reflections
1012 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.083
S = 1.02
1080 reflections
39 parameters
9 restraints
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 1.11 e Å^{-3}
$\Delta\rho_{min}$ = −1.78 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); 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: PK2055).

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supplementary materials

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NH_4PbI_3

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Comment

Considerable current interests focus on fundamental as well as more applied studies of iodoplumbates related to their significant structural, electrical, non-linear optical, and other physical properties (Mitzi *et al.*, 1995; Guloy *et al.*, 2001; Fan *et al.*, 2006). Lead(II) iodide and its low-dimensional derivatives represent a potential class of functional materials. We report here the crystal structure of the title lead(II) iodide complex, which has a one-dimensional anion chain.

There is one crystallographically independent Pb^{II} ion in the asymmetric unit (Fig. 1). Pb^{II} ion is six-coordinated in a distorted octahedral environment by six I^- ions with Pb—I distances ranging from 3.0595 (9) to 3.3679 (9) Å and *cis* I—Pb—I angles from 84.40 (2) to 94.78 (2)° (Table 1). Adjacent octahedra are joined by a common edge (I1/I3) to form a chain; two neighboring chains are connected through a common edge (I1/I1) to form a one-dimensional anion chain $[PbI_3]_n^{n-}$ along the *b* axis (Fig. 2). As a consequence of the connectivity of PbI_6 octahedra, the I^- ions are acting as μ_3 (I1) and μ bridge (I3) and as a terminal ligand (I2). The longest Pb—I bond length is observed for the triply bridging ligand I1 due to its higher connectivity and the *trans* influence of the terminal ligand I2 in *trans* position to I2 (Krautscheid, *et al.*, 2001). At 473 K, the C—N bonds of $[Bu_4N]^+$ are broken, so the cation of $[NH_4]^+$ is formed. The anion chain has no significant hydrogen-bonding interactions with the cations.

Experimental

A mixture of PbI_2 (92 mg, 0.2 mmol) and Bu_4NI (74 mg, 0.2 mmol) was pressed into a pellet, which was then sealed into an evacuated quartz tube. The quartz tube was heated at 473 K for 2 days, and then cooled slowly to room temperature. Prism-shaped yellow crystals of suitable for X-ray analysis were obtained.

Refinement

The N—H distances were restrained to 0.80 (2) Å. The displacement parameters of H atoms were set at 1.5 times U_{eq} of the N atom. The highest peak is located 1.17 Å from I1 and the deepest hole is located 0.74 Å from Pb1.

Figures

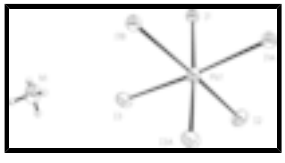

Fig. 1. The asymmetric uni of (I), together with additional I atoms to complete the coordination of Pb atom. Displacement ellipsoids are plotted at the 50% probability level. [Symmetry code: (A) x, $1+y$, z; (B) $1-x$, $1-y$, $1-z$, (C) x, $-0.5-y$, z]
Fig. 2. Packing Diagram of (I) looking down *b* axis.

ammonium lead iodide

Crystal data

$NH_4Pb_1I_3$	F_{000} = 1008
M_r = 605.94	D_x = 4.766 Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 2002 reflections
a = 10.3029 (14) Å	θ = 3.1–27.5º
b = 4.7411 (5) Å	μ = 30.84 mm^{-1}
c = 17.288 (2) Å	*T* = 293 (2) K
V = 844.48 (18) Å^3	Prism, yellow
Z = 4	0.10 × 0.08 × 0.05 mm

Data collection

Rigaku Mercury CCD diffractometer	1080 independent reflections
Radiation source: Sealed Tube	1012 reflections with $I > 2\sigma(I)$
Monochromator: Graphite Monochromator	R_{int} = 0.045
T = 293(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.1º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2000)	*h* = −12→13
T_{min} = 0.064, T_{max} = 0.214	*k* = −6→6
6247 measured reflections	*l* = −22→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.033	Only H-atom coordinates refined
$wR(F^2)$ = 0.083	$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 2.6931P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
1080 reflections	$\Delta\rho_{max}$ = 1.11 e Å^{-3}
39 parameters	$\Delta\rho_{min}$ = −1.78 e Å^{-3}
9 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.66816 (4)	0.7500	0.44155 (2)	0.02949 (16)
I1	0.47613 (7)	0.2500	0.38364 (4)	0.0303 (2)
I2	0.80933 (8)	0.7500	0.28586 (5)	0.0401 (2)
I3	0.84262 (7)	0.2500	0.51297 (5)	0.0374 (2)
N1	0.9132 (13)	−0.2500	0.6757 (6)	0.053 (4)
H1	0.952 (12)	−0.2500	0.715 (5)	0.080*
H2	0.963 (11)	−0.2500	0.641 (6)	0.080*
H3	0.871 (5)	−0.115 (12)	0.674 (5)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0328 (3)	0.0274 (3)	0.0283 (3)	0.000	0.00360 (17)	0.000
I1	0.0298 (4)	0.0360 (4)	0.0250 (4)	0.000	−0.0016 (3)	0.000
I2	0.0453 (5)	0.0413 (5)	0.0336 (4)	0.000	0.0133 (4)	0.000
I3	0.0326 (4)	0.0293 (4)	0.0503 (5)	0.000	−0.0092 (3)	0.000
N1	0.071 (9)	0.053 (8)	0.036 (6)	0.000	−0.001 (6)	0.000

Geometric parameters (Å, °)

Pb1—I2	3.0595 (9)	I1—Pb1^{iii}	3.2459 (6)
Pb1—I3^{i}	3.2210 (6)	I1—Pb1^{ii}	3.3679 (9)
Pb1—I3	3.2210 (6)	I3—Pb1^{iii}	3.2210 (6)
Pb1—I1	3.2459 (6)	N1—H1	0.79 (7)
Pb1—I1^{i}	3.2459 (6)	N1—H2	0.78 (7)
Pb1—I1^{ii}	3.3679 (9)	N1—H3	0.78 (6)
I2—Pb1—I3^{i}	94.12 (2)	I3^{i}—Pb1—I1^{ii}	84.40 (2)
I2—Pb1—I3	94.12 (2)	I3—Pb1—I1^{ii}	84.40 (2)
I3^{i}—Pb1—I3	94.78 (2)	I1—Pb1—I1^{ii}	90.443 (18)
I2—Pb1—I1	91.05 (2)	I1^{i}—Pb1—I1^{ii}	90.443 (18)

$I3^{i}$—Pb1—I1	174.78 (2)	Pb1—I1—$Pb1^{iii}$	93.83 (2)
I3—Pb1—I1	85.461 (16)	Pb1—I1—$Pb1^{ii}$	89.557 (18)
I2—Pb1—$I1^{i}$	91.05 (2)	$Pb1^{iii}$—I1—$Pb1^{ii}$	89.557 (18)
$I3^{i}$—Pb1—$I1^{i}$	85.461 (16)	$Pb1^{iii}$—I3—Pb1	94.78 (2)
I3—Pb1—$I1^{i}$	174.78 (2)	H1—N1—H2	108 (5)
I1—Pb1—$I1^{i}$	93.83 (2)	H1—N1—H3	109 (3)
I2—Pb1—$I1^{ii}$	177.81 (3)	H2—N1—H3	110 (3)
I2—Pb1—I1—$Pb1^{iii}$	88.87 (2)	$I1^{i}$—Pb1—I1—$Pb1^{ii}$	−90.474 (19)
I3—Pb1—I1—$Pb1^{iii}$	−5.18 (2)	$I1^{ii}$—Pb1—I1—$Pb1^{ii}$	0.0
$I1^{i}$—Pb1—I1—$Pb1^{iii}$	180.0	I2—Pb1—I3—$Pb1^{iii}$	−85.50 (2)
$I1^{ii}$—Pb1—I1—$Pb1^{iii}$	−89.526 (19)	$I3^{i}$—Pb1—I3—$Pb1^{iii}$	180.0
I2—Pb1—I1—$Pb1^{ii}$	178.399 (19)	I1—Pb1—I3—$Pb1^{iii}$	5.23 (2)
I3—Pb1—I1—$Pb1^{ii}$	84.34 (2)	$I1^{ii}$—Pb1—I3—$Pb1^{iii}$	96.12 (2)

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

Fig. 1