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Crystal structure of a new hybrid compound based on an iodidoplumbate(II) anionic motif

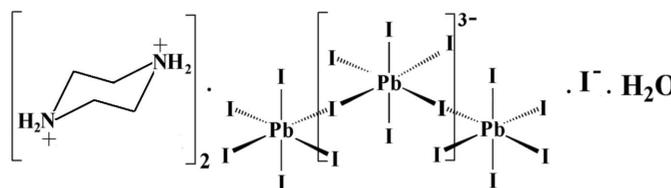
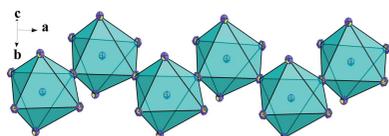
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Crystals of the one-dimensional organic–inorganic lead iodide-based compound *catena*-poly[bis(piperazine-1,4-dium) [[tetraiodidoplumbate(II)]- μ -iodido] iodide monohydrate], $(C_4N_2H_{12})_2[PbI_5]I \cdot H_2O$, were obtained by slow evaporation at room temperature of a solution containing lead iodide and piperazine in a 1:2 molar ratio. Inorganic lead iodide chains, organic $(C_4N_2H_{12})^{2+}$ cations, water molecules of crystallization and isolated I^- anions are connected through $N-H \cdots I$, $N-H \cdots OW$ and $OW-H \cdots I$ hydrogen-bond interactions. Zigzag chains of corner-sharing $[PbI_6]^{4-}$ octahedra with composition $[Pb_{4/1}I_{2/2}]^{3-}$ running parallel to the *a* axis are present in the structure packing.

1. Chemical context

Organic–inorganic hybrid materials offer the opportunity to combine the desirable properties of the organic moiety such as processability, toughness and impact strength with the typical properties of the inorganic part such as high temperature stability and durability. The opto-electronic characteristics of hybrid materials are closely related to the metal cluster size. In recent years, a significant number of organic–inorganic hybrid materials based on lead halide units have been prepared and studied (Billing & Lemmerer, 2006; Rayner & Billing, 2010), in particular with self-organized low-dimensional families of lead iodide-based crystals where the $[PbI_6]$ octahedra form one-, two- or three-dimensional networks (Elleuch *et al.*, 2007; Trigui *et al.*, 2011). In one-dimensional lead halide hybrid compounds, the inorganic chains may be formed by one, two or three bridging halides, referred to as corner-, edge- and face-sharing polyhedra, respectively. Thanks to their anticipated electroluminescence, photoluminescence and non-linear optical properties, these compounds are the most desired ones (Lemmerer & Billing, 2006). Lead iodide-based hybrid materials are studied extensively for their excitonic and magneto-optical properties. In this work we report the synthesis and crystal structure determination of a new lead iodide hybrid, $(C_4N_2H_{12})_2[PbI_5] \cdot H_2O$, (I).



2. Structural commentary

The structural units of (I) consist of one piperazine molecule, one water molecule, one isolated iodine and one $[PbI_6]$ unit

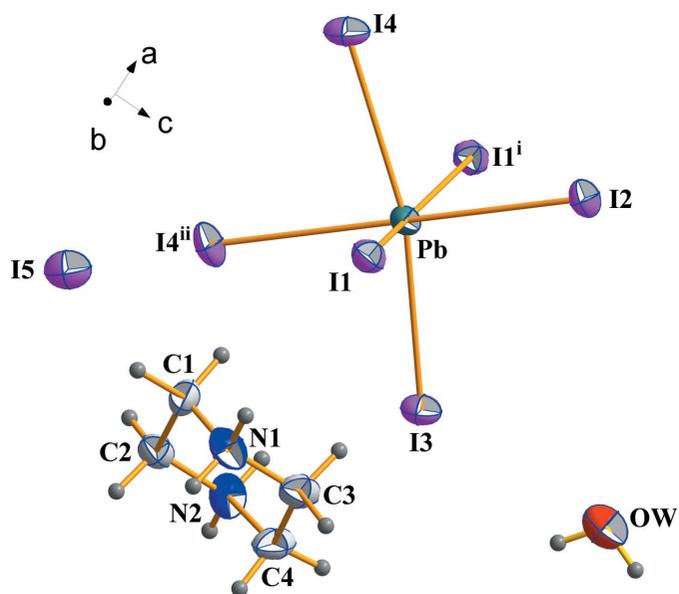


Figure 1
Structural units of the title compound, showing the atom-numbering scheme. Atomic displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius. [Symmetry codes: (i) $x, \frac{1}{2} - y, z$; (ii) $-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} - z$.]

(Fig. 1). The electrical neutrality is ensured by two organic molecules of doubly protonated piperazine.

The main part of the inorganic moiety is composed by the lead Pb^{2+} cation which adopts a distorted octahedral coordination. The angles between *cis*-related I^- ions range from $85.022(12)$ to $96.89(3)^\circ$ at most, whereas the *trans* angles deviate from 180° by $12.95(3)^\circ$ (Table 1). Two adjacent corners connect the $[\text{PbI}_6]$ octahedron to its neighbours, leading to zigzag chains running parallel to the *a* axis (Fig. 2). This one-dimensional anionic network leaves empty spaces in which the organic cations are located. The $[\text{PbI}_6]$ octahedra establish two strong hydrogen bonds (Table 2), $\text{N2}-\text{H4N}\cdots\text{I3}$ and $\text{N2}^i-\text{H4N}^i\cdots\text{I3}$, via the I3 corners [symmetry code: (i) $x, \frac{1}{2} - y, z$] as illustrated in Fig. 3.

The second part of the inorganic moiety contains a water molecule and the iodide anion I5 linked by a strong hydrogen-bond interaction (Table 2). Both are located in the same layers in which the $[\text{PbI}_6]$ octahedra are located. As shown in Fig. 4, the anion I5 is linked to one water molecule by $\text{I5}\cdots\text{HW1}^i$

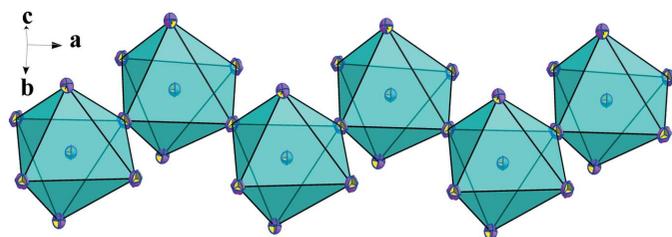


Figure 2
The $[\text{PbI}_4\text{I}_2]^{3-}$ chain of (I) running parallel to the *a*-axis direction and exhibiting a zigzag conformation.

Table 1
Selected geometric parameters (\AA , $^\circ$).

Pb—I2	3.0689 (9)	Pb—I4	3.2396 (9)
Pb—I3	3.1511 (9)	Pb—I4 ⁱⁱ	3.3535 (9)
Pb—I1	3.2173 (8)	I4—Pb ⁱⁱⁱ	3.3535 (9)
Pb—I1 ⁱ	3.2173 (8)	OW—HW2	0.86 (2)
I2—Pb—I3	96.06 (3)	I1—Pb—I4	87.185 (13)
I2—Pb—I1	85.021 (12)	I1 ⁱ —Pb—I4	87.185 (13)
I3—Pb—I1	93.943 (13)	I2—Pb—I4 ⁱⁱ	179.99 (3)
I2—Pb—I1 ⁱ	85.022 (12)	I3—Pb—I4 ⁱⁱ	83.95 (3)
I3—Pb—I1 ⁱ	93.944 (13)	I1—Pb—I4 ⁱⁱ	94.978 (12)
I1—Pb—I1 ⁱ	167.89 (2)	I1 ⁱ —Pb—I4 ⁱⁱ	94.977 (12)
I2—Pb—I4	96.89 (3)	I4—Pb—I4 ⁱⁱ	83.105 (14)
I3—Pb—I4	167.05 (3)	Pb—I4—Pb ⁱⁱⁱ	178.91 (3)

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

Table 2
Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots OW ^{iv}	0.90	2.05	2.874 (5)	155
N1—H2N \cdots I5 ^v	0.90	2.69	3.543 (4)	160
N2—H4N \cdots I3	0.90	2.85	3.656 (4)	151
OW—HW1 \cdots I5 ^{vi}	0.86	2.74	3.477 (5)	145

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

OW^i [symmetry code: (i) $1 - x, \frac{1}{2} + y, 1 - z$] and two organic cations via $\text{I5}\cdots\text{H2N}^{ii}-\text{N1}^{ii}$ and $\text{I5}\cdots\text{H2N}^{iii}-\text{N1}^{iii}$ [symmetry codes: (ii) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} - z$; (iii) $\frac{1}{2} + x, y, \frac{1}{2} - z$]. On the other hand, the water molecule is associated to one iodine (I5) via $\text{OW}-\text{HW1}\cdots\text{I5}^{iii}$ [symmetry code: (iii) $1 - x, -\frac{1}{2} + y, 1 - z$] and to two piperazinium cations via $\text{OW}\cdots\text{H1N}^{ii}-\text{N1}^{ii}$ and $\text{OW}\cdots\text{H1N}^i-\text{N1}^i$ (Fig. 5). In this configuration, no acceptor was found for HW2 and H3N.

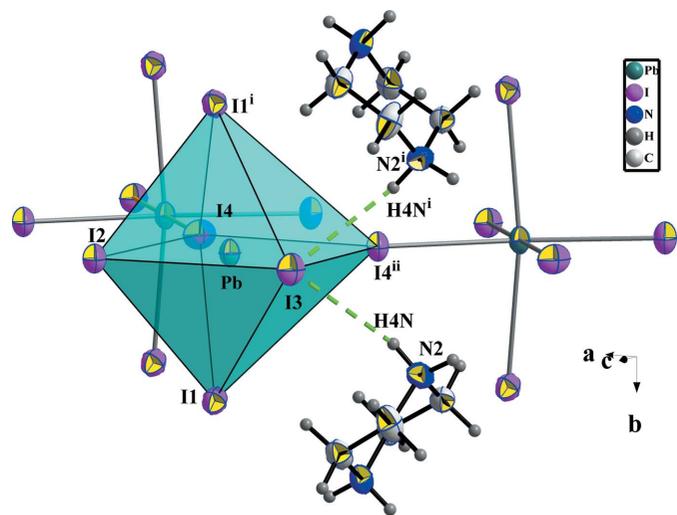


Figure 3
Linkage around one $[\text{PbI}_6]$ octahedron formed by two similar octahedra and two protonated piperazine cations. Hydrogen bonds are drawn as dashed green lines. [Symmetry codes: (i) $x, \frac{1}{2} - y, z$; (ii) $-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} - z$.]

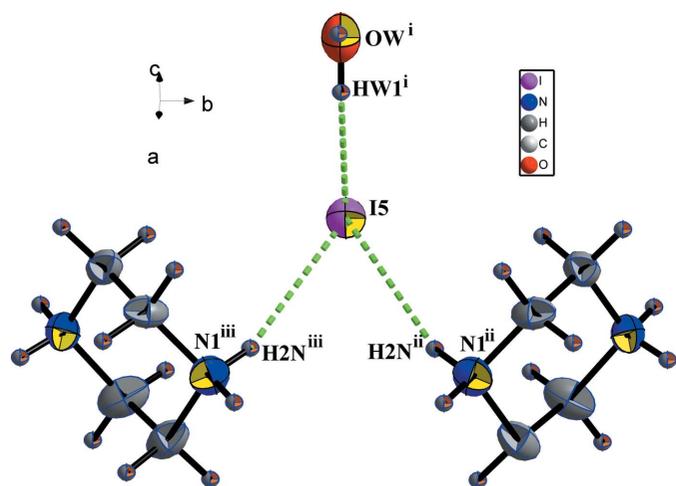


Figure 4
Hydrogen-bonding interactions with isolated iodide in (I). [Symmetry codes: (i) $1 - x, \frac{1}{2} + y, 1 - z$; (ii) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} - z$; (iii) $\frac{1}{2} + x, y, \frac{1}{2} - z$.]

The six-membered piperazinium cation ring adopts a chair conformation. It interacts with the inorganic chain *via* strong $N2-H4N \cdots I3$ hydrogen bonds with a 2.85 Å bond length (Table 2 and Fig. 6). In the crystal structure, the piperazinium cations are also linked to the water molecule by an $N1-H1N \cdots OW^{iii}$ hydrogen bond and to the iodide anion by $N1-H2N \cdots I5^{iii}$ hydrogen bonds.

Compared to its homologous hybrids, the structure of the title compound exhibits an original arrangement of the inorganic layers. It is composed by two parts: the first are the $[PbI_6]$ octahedra sharing adjacent corners and so assembling into chains running along the [100] direction. The second original feature is the structural cohesion by water molecules and isolated iodide anions. This structural arrangement will probably have an impact on the dielectric behavior of the material. Luminescence and UV-visible spectroscopy

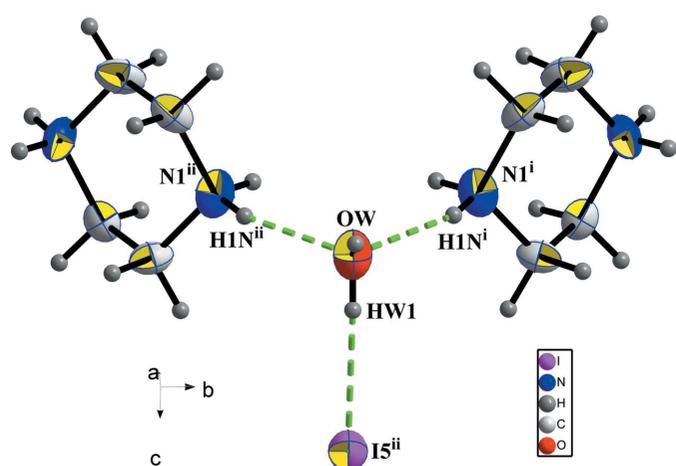


Figure 5
Water molecule hydrogen bonding interactions in (I). [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, -\frac{1}{2} + y, 1 - z$.]

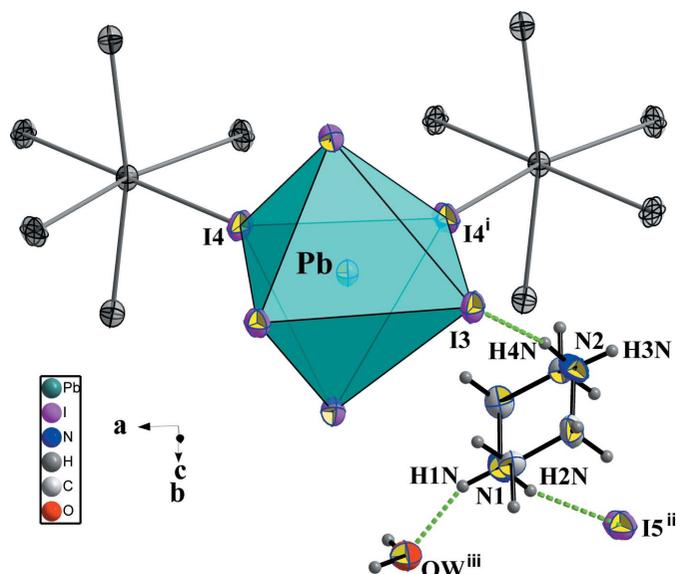


Figure 6
The hydrogen bonding environment of the cation of the title compound. [Symmetry codes: (i) $-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} - z$; (ii) $-\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} - z$; (iii) $1 - x, \frac{1}{2} + y, 1 - z$.]

measurements of this compound, coupled to theoretical calculation of the Highest Occupied Molecular Orbital (HOMO) and Lowest Unoccupied Molecular Orbital (LUMO) electronic transitions are in progress.

As shown in Fig. 7, the structure of (I) is self-assembled into alternating organic and inorganic layers parallel to the *ac* plane. The organic part is made up of $(C_4H_{12}N_2)^{2+}$ cations located in the voids around the corner-sharing $[PbI_6]^{4-}$ octahedra. The iodide anions and the water molecules connect the organic and inorganic sheets by strong hydrogen-bond interactions.

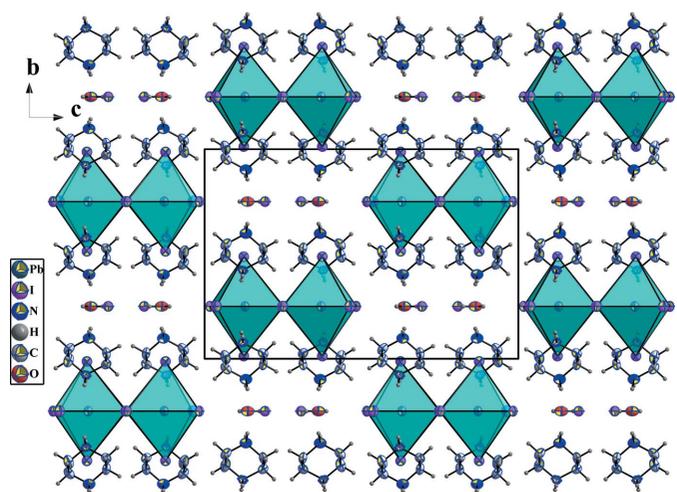


Figure 7
A packing diagram of (I), viewed along the *a* axis showing the alternating organic and inorganic layers. Hydrogen bonds are omitted for clarity.

Table 3
Experimental details.

Crystal data	
Chemical formula	(C ₄ H ₁₂ N ₂) ₂ [PbI ₅]I·H ₂ O
<i>M_r</i>	1162.92
Crystal system, space group	Orthorhombic, <i>Pnma</i>
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.7477 (10), 13.488 (2), 20.336 (3)
<i>V</i> (Å ³)	2399.4 (6)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm ⁻¹)	14.75
Crystal size (mm)	0.45 × 0.14 × 0.10
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Absorption correction	<i>ψ</i> scan (North <i>et al.</i> , 1968)
<i>T_{min}</i> , <i>T_{max}</i>	0.622, 0.999
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	3601, 2729, 1941
<i>R_{int}</i>	0.034
(sin <i>θ</i> /λ) _{max} (Å ⁻¹)	0.638
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.037, 0.086, 1.05
No. of reflections	2729
No. of parameters	105
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	2.00, -1.28

Computer programs: *CAD-4 EXPRESS* (Enraf–Nonius, 1994), *XCAD4* (Harms & Wocadlo, 1995), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2006) and *pubCIF* (Westrip, 2010).

3. Database survey

Using the piperazine-1,4-dium cation scheme in the similarity option of the WEBCSD interface (Groom & Allen, 2014), more than 90 records are found in the CCDC database. Only 24 are inorganic–organic hybrid compounds with several metals Cu, Zn, Co, Bi, Cd, Sb, Au *etc.* The closest chemical composition found is a bismuth-based compound (II): (C₄N₂H₁₂)₂[BiCl₆]·Cl·H₂O (Gao *et al.*, 2011). In spite of the chemical formula similarity, it seems that the orthorhombic (*Pnma*) title structure is much more regular than the monoclinic (*P2₁/c*) compound (II) with approximately the same cell volume, where the small difference is probably due to the chlorine/iodine substitution. In contrast to the structure of (I), the anionic network in the structure of (II) is 0-D, built up by

isolated [BiCl₆] octahedra. The water molecule and the isolated halogen play, in both cases, the same crucial role in the structural cohesion, linking the anionic part to the organic moieties.

4. Synthesis and crystallization

Crystals of the title compound were prepared by slow evaporation at room temperature by mixing 1,4-diazacyclohexane (C₄H₁₀N₂) (2 mol) with a solution of lead iodide PbI₂ (1 mol) in an equimolar mixture of ethanol and DMF. After several weeks, the obtained crystals were isolated and dried.

5. Refinement

Data collection and structure refinement details are summarized in Table 3. Hydrogen atoms were placed using geometrical constraints using adequate HFIX instructions (SHELXL) and refined with AFIX instructions. Water hydrogen atoms were found in Fourier difference maps and O–H distances were restrained using DFIX (0.86 Å) and DANG instructions.

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Computing details

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

catena-Poly[bis(piperazine-1,4-dium) [[tetraiodidoplumbate(II)]- μ -iodido] iodide monohydrate]

Crystal data

(C₄H₁₂N₂)₂[PbI₅]I·H₂O

$M_r = 1162.92$

Orthorhombic, *Pnma*

$a = 8.7477$ (10) Å

$b = 13.488$ (2) Å

$c = 20.336$ (3) Å

$V = 2399.4$ (6) Å³

$Z = 4$

$F(000) = 2040$

$D_x = 3.219$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 13.7$ – 14.7°

$\mu = 14.75$ mm⁻¹

$T = 298$ K

Prism, yellow

$0.45 \times 0.14 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

$\omega/2\tau$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.622$, $T_{\max} = 0.999$

3601 measured reflections

2729 independent reflections

1941 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -11 \rightarrow 2$

$k = -1 \rightarrow 17$

$l = -1 \rightarrow 25$

2 standard reflections every 120 min

intensity decay: -1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.086$

$S = 1.05$

2729 reflections

105 parameters

3 restraints

Primary atom site location: heavy-atom method

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 1.6589P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 2.00 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.28 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. Number of psi-scan sets used was 4 Theta correction was applied. Averaged transmission function was used. No Fourier smoothing was applied.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb	0.70782 (5)	0.2500	0.37128 (2)	0.02909 (13)
I1	0.74558 (6)	0.48720 (5)	0.37511 (3)	0.03537 (16)
I2	0.94306 (9)	0.2500	0.48324 (4)	0.0367 (2)
I3	0.41658 (9)	0.2500	0.46247 (4)	0.0410 (2)
I4	0.95081 (10)	0.2500	0.25107 (4)	0.0421 (2)
I5	0.54190 (11)	0.7500	0.19255 (5)	0.0489 (3)
N1	0.3401 (9)	0.5979 (6)	0.3685 (3)	0.0430 (19)
H1N	0.2673	0.6433	0.3635	0.052*
H2N	0.4301	0.6287	0.3680	0.052*
N2	0.1611 (9)	0.4232 (6)	0.3784 (3)	0.0407 (19)
H3N	0.0691	0.3951	0.3792	0.049*
H4N	0.2305	0.3754	0.3828	0.049*
C1	0.3329 (10)	0.5257 (8)	0.3131 (4)	0.039 (2)
H1A	0.3455	0.5604	0.2716	0.046*
H1B	0.4151	0.4779	0.3172	0.046*
C2	0.1836 (9)	0.4737 (7)	0.3138 (4)	0.035 (2)
H2A	0.1805	0.4252	0.2787	0.042*
H2B	0.1019	0.5210	0.3066	0.042*
C3	0.3199 (10)	0.5469 (8)	0.4320 (4)	0.041 (2)
H3A	0.4040	0.5012	0.4389	0.050*
H3B	0.3214	0.5953	0.4672	0.050*
C4	0.1744 (10)	0.4919 (8)	0.4337 (4)	0.046 (3)
H4A	0.1677	0.4552	0.4746	0.055*
H4B	0.0900	0.5386	0.4325	0.055*
OW	0.4301 (11)	0.2500	0.6369 (5)	0.051 (3)
HW1	0.393 (13)	0.2500	0.676 (2)	0.080*
HW2	0.352 (9)	0.2500	0.612 (4)	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb	0.0224 (2)	0.0364 (3)	0.0284 (2)	0.000	0.00002 (19)	0.000

I1	0.0287 (3)	0.0386 (4)	0.0388 (3)	0.0019 (2)	-0.0011 (2)	0.0008 (3)
I2	0.0322 (4)	0.0408 (6)	0.0372 (4)	0.000	-0.0079 (4)	0.000
I3	0.0296 (4)	0.0498 (6)	0.0435 (4)	0.000	0.0088 (4)	0.000
I4	0.0353 (4)	0.0420 (5)	0.0491 (5)	0.000	0.0180 (4)	0.000
I5	0.0525 (5)	0.0392 (6)	0.0550 (5)	0.000	0.0114 (5)	0.000
N1	0.030 (4)	0.042 (5)	0.058 (5)	-0.007 (4)	-0.009 (4)	0.002 (4)
N2	0.044 (4)	0.032 (4)	0.046 (4)	-0.009 (4)	-0.008 (4)	0.006 (4)
C1	0.039 (5)	0.051 (6)	0.025 (4)	-0.002 (5)	-0.001 (4)	0.005 (4)
C2	0.031 (4)	0.035 (5)	0.040 (5)	0.002 (4)	-0.004 (4)	-0.005 (4)
C3	0.035 (5)	0.046 (6)	0.043 (5)	0.000 (5)	0.001 (4)	-0.015 (5)
C4	0.029 (5)	0.067 (8)	0.041 (5)	-0.009 (5)	0.008 (4)	-0.012 (5)
OW	0.046 (6)	0.038 (6)	0.068 (6)	0.000	-0.003 (5)	0.000

Geometric parameters (Å, °)

Pb—I2	3.0689 (9)	N2—H4N	0.8900
Pb—I3	3.1511 (9)	C1—C2	1.483 (11)
Pb—I1	3.2173 (8)	C1—H1A	0.9700
Pb—I1 ⁱ	3.2173 (8)	C1—H1B	0.9700
Pb—I4	3.2396 (9)	C2—H2A	0.9700
Pb—I4 ⁱⁱ	3.3535 (9)	C2—H2B	0.9700
I4—Pb ⁱⁱⁱ	3.3535 (9)	C3—C4	1.473 (12)
N1—C1	1.490 (11)	C3—H3A	0.9700
N1—C3	1.474 (11)	C3—H3B	0.9700
N1—H1N	0.8900	C4—H4A	0.9700
N1—H2N	0.8900	C4—H4B	0.9700
N2—C4	1.462 (11)	OW—HW1	0.86 (2)
N2—C2	1.493 (10)	OW—HW2	0.86 (2)
N2—H3N	0.8900		
I2—Pb—I3	96.06 (3)	H3N—N2—H4N	107.9
I2—Pb—I1	85.021 (12)	C2—C1—N1	109.8 (7)
I3—Pb—I1	93.943 (13)	C2—C1—H1A	109.7
I2—Pb—I1 ⁱ	85.022 (12)	N1—C1—H1A	109.7
I3—Pb—I1 ⁱ	93.944 (13)	C2—C1—H1B	109.7
I1—Pb—I1 ⁱ	167.89 (2)	N1—C1—H1B	109.7
I2—Pb—I4	96.89 (3)	H1A—C1—H1B	108.2
I3—Pb—I4	167.05 (3)	C1—C2—N2	110.0 (7)
I1—Pb—I4	87.185 (13)	C1—C2—H2A	109.7
I1 ⁱ —Pb—I4	87.185 (13)	N2—C2—H2A	109.7
I2—Pb—I4 ⁱⁱ	179.99 (3)	C1—C2—H2B	109.7
I3—Pb—I4 ⁱⁱ	83.95 (3)	N2—C2—H2B	109.7
I1—Pb—I4 ⁱⁱ	94.978 (12)	H2A—C2—H2B	108.2
I1 ⁱ —Pb—I4 ⁱⁱ	94.977 (12)	C4—C3—N1	111.1 (7)
I4—Pb—I4 ⁱⁱ	83.105 (14)	C4—C3—H3A	109.4
Pb—I4—Pb ⁱⁱⁱ	178.91 (3)	N1—C3—H3A	109.4
C1—N1—C3	110.7 (7)	C4—C3—H3B	109.4
C1—N1—H1N	109.5	N1—C3—H3B	109.4

C3—N1—H1N	109.5	H3A—C3—H3B	108.0
C1—N1—H2N	109.5	N2—C4—C3	111.6 (7)
C3—N1—H2N	109.5	N2—C4—H4A	109.3
H1N—N1—H2N	108.1	C3—C4—H4A	109.3
C4—N2—C2	112.1 (7)	N2—C4—H4B	109.3
C4—N2—H3N	109.2	C3—C4—H4B	109.3
C2—N2—H3N	109.2	H4A—C4—H4B	108.0
C4—N2—H4N	109.2	HW1—OW—HW2	104 (3)
C2—N2—H4N	109.2		

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1N...OW ^{iv}	0.90	2.05	2.874 (5)	155
N1—H2N...I5 ^v	0.90	2.69	3.543 (4)	160
N2—H4N...I3	0.90	2.85	3.656 (4)	151
OW—HW1...I5 ^{vi}	0.86	2.74	3.477 (5)	145

Symmetry codes: (iv) $-x+1, y+1/2, -z+1$; (v) $x-1/2, -y+3/2, -z+1/2$; (vi) $-x+1, y-1/2, -z+1$.