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A one-dimensional organic–inorganic hybrid compound $[(CH_3)_2C{=}NHCH_2CH_2CH_3][PbI_3]$

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.027 Å
R factor = 0.055
wR factor = 0.152
Data-to-parameter ratio = 31.1

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

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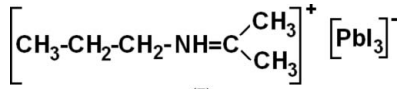
Crystals of the one-dimensional organic–inorganic lead iodide-based compound *N*-isopropylidenepropanaminium triiodoplumbate(II), $(C_6H_{14}N)[PbI_3]$, were prepared by slow evaporation at room temperature. In the inorganic sublattice, the PbI_6 octahedra form infinite one-dimensional chains sharing triangular faces. The inorganic PbI_3 chain and the organic cations are connected through N—H···I hydrogen-bonding interactions.

Comment

Recently there has been considerable interest in lead(II) halide organic–inorganic hybrid compounds due to their diverse electrical, magnetic and optical properties, as well as their excellent film processability (Era *et al.*, 1998; Ishihara *et al.*, 1990; Mitzi *et al.*, 2001). In particular, the family of lead iodide-based crystals are self-organized low-dimensional nanostructures in which PbI_6 octahedra form one-, two- or three-dimensional networks. Among them, the layered organic–inorganic two-dimensional structure of $(R{-}NH_3)_2[PbI_4]$ still attracts a lot of interest because of its application in electroluminescent devices, opto-electronic materials and thin-film field-effect transistors (Mitzi *et al.*, 2001). More recently, extensive studies have been reported on the structures and physical properties of one-dimensional lead iodide-based structures (Zhu *et al.*, 2004; Maxcy *et al.*, 2003). To our knowledge, there are two types of lead iodide-based one-dimensional structures, *viz*. (i) $C_5H_{10}NH_2[PbI_3]$, in which the PbI_6 octahedra form an infinite one-dimensional chain sharing triangular faces (Gridnuva *et al.*, 1984) and (ii) $[NH_2C(I){=}NH_2]_3[PbI_5]$ (Wang *et al.*, 1995; Tanaka *et al.*, 2005) or $[NH_2SC(=NH_2)NH_2]_3[PbI_5]$ (Mousdis *et al.*, 1998), in which the $[PbI_6]$ octahedra form an infinite one-dimensional chain sharing corner atoms. In this paper, we report the structural properties of the organic–inorganic one-dimensional hybrid compound (I) $[(CH_3)_2C{=}NHCH_2CH_2CH_3][PbI_3]$.



(I)

The asymmetric unit of (I) consists of a $[PbI_3]^-$ ion (a part of the face-sharing octahedral chain) and one $(C_6H_{14}N)^+$ cation (Fig. 1). The inorganic part forms one-dimensional chains of triangular face-sharing PbI_6 octahedra aligned along the *a* axis. The organic cations are linked to the inorganic chain by N—H···I hydrogen bonds (Fig. 2). A close exam-

ination of the structure of the title compound, especially the hydrogen bonds, shows it to compare well with related lead iodide-based organic–inorganic compounds (Zhu *et al.*, 2004; Maxcy *et al.*, 2003).

Experimental

The precursor $C_6H_{14}N^+I^-$ was prepared by mixing hydroiodic acid HI (aq. 57%) with a solution of imine in acetone in equimolar amounts. The reaction solution was evaporated to remove the water. $C_6H_{14}N[PbI_3]$ crystals were grown by slow evaporation, at ambient temperature, of an *N*,*N*-dimethylformamide (DMF) solution in which stoichiometric amounts of $C_6H_{14}N^+I^-$ and PbI_2 had been dissolved. This mixture was stirred and remained clear without any precipitate. Three days later, yellow crystals were obtained. Crystals suitable for X-ray analysis were obtained from a second recrystallization also from DMF.

Crystal data

$(C_6H_{14}N)[PbI_3]$
M_r = 688.07
Monoclinic, $P2_1/c$
a = 11.533 (4) Å
b = 16.291 (5) Å
c = 8.251 (3) Å
β = 107.66 (3)°
V = 1477.2 (9) Å^3
Z = 4
D_x = 3.094 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 17.65 mm^{-1}
T = 293 (2) K
Parallelipiped, yellow
0.24 × 0.16 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.045, T_{max} = 0.126
5585 measured reflections
3207 independent reflections
2058 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 27.0°
2 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.152
S = 1.04
3207 reflections
103 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 3.92 e Å^{-3}
$\Delta\rho_{min}$ = −2.37 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—H1···I2	0.86	3.09	3.882 (17)	155
N—H1···I3	0.86	3.25	3.702 (14)	116

All H atoms were positioned geometrically and treated as riding with C—H = 0.96 Å (methyl) or 0.97 Å (methylene) and N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N) or U_{iso}(H) = 1.5U_{eq}(methyl C).

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

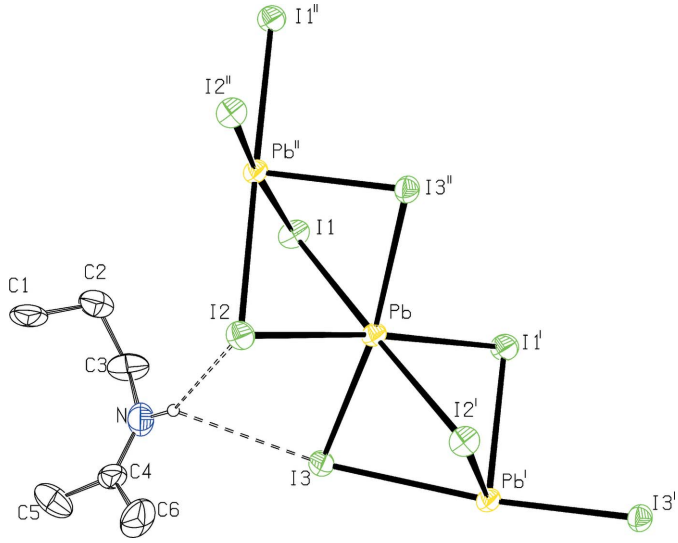


Figure 1
Partial view of the inorganic PbI_3 chains and the hydrogen-bonding interactions with the cation, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. N—H···I hydrogen intercations are shown as dashed lines. The H atom is represented as a small sphere of arbitrary radius. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) x, $\frac{3}{2} - y$, $z - \frac{1}{2}$; (ii) x, $\frac{3}{2} - y$, $\frac{1}{2} + z$]

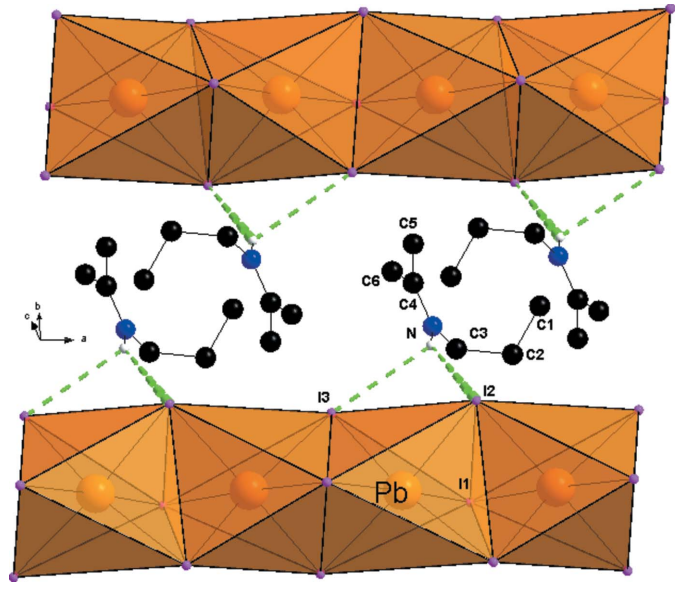


Figure 2
The arrangement of anionic chains along the *a*-axis direction. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonding have been omitted for clarity.

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