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catena-Poly[1-[(2-fluorobenzylidene)amino]quinolinium [plumbate(II)-tri-μ-iodido]]

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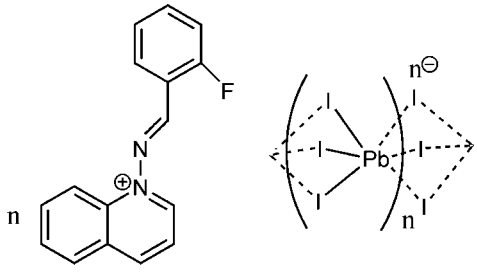
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.020 Å; R factor = 0.047; wR factor = 0.082; data-to-parameter ratio = 19.7.

The title complex, $\{(C_{16}H_{12}FN_2)[PbI_3]\}_n$, consists of 1-[(2-fluorobenzylidene)amino]quinolinium cations and a polymeric PbI_3^- anion formed by face-sharing PbI_6 octahedra. These octahedra form straight and regular infinite chains along the b axis. In the asymmetric unit, one cation and one anionic $[PbI_3]^-$ fragment are observed in general positions. Polymeric chains are produced by the glide plane perpendicular to the a axis.

Related literature

For second-order non-linear optical (NLO) properties, pyroelectricity, ferroelectricity and triboluminescence of inorganic-organic hybrid materials, see: Guloy *et al.* (2001); Horiuchi *et al.* (2010); Chen *et al.* (2001). For related structures, see: Bi *et al.* (2008); Zhang *et al.* (2006); Duan *et al.* (2011); Zhao *et al.* (2010).



Experimental

Crystal data

$(C_{16}H_{12}FN_2)[PbI_3]$
M_r = 839.17
Orthorhombic, *Pbca*
a = 20.888 (4) Å
b = 7.9112 (15) Å
c = 25.197 (5) Å
V = 4163.8 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 12.56 mm^{-1}
T = 296 K
0.04 × 0.02 × 0.01 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.747, T_{max} = 0.882
30847 measured reflections
4090 independent reflections
1770 reflections with $I > 2\sigma(I)$
R_{int} = 0.156

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.082
S = 0.96
4090 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.96 e Å^{-3}
$\Delta\rho_{min}$ = −0.95 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: IM2337).

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catena-Poly[1-[(2-fluorobenzylidene)amino]quinolinium [plumbate(II)-tri-μ-iodido]]

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S1. Comment

Inorganic-organic hybrid materials have attracted intense interest in recent years, owing to their technologically important physical properties from optics to electronics, such as second-order nonlinear optical (NLO) properties, (Guloy *et al.*, 2001) pyroelectricity, ferroelectricity (Horiuchi *et al.*, 2010) and triboluminescence (Chen *et al.*, 2001).

Inorganic metal-halide building blocks exhibiting $[MX_6]^{4-/3-}$ fragments (M = Sn^{2+}, Pb^{2+}, Bi^{3+}, Sb^{3+}; X = F^-, Cl^-, Br^-, I^-) have received special attention in the construction of inorganic-organic hybrid materials (Zhang *et al.*, 2006; Bi *et al.*, 2008). Herein we report the crystal structure of the title compound (I) (Figure 1).

The title compound crystallizes in the orthorhombic space group P*bca* with an asymmetric unit containing one anionic PbI_3 fragment together with one Schiff base cation. The polymeric anion $[PbI_3]_n^{n-}$ possesses slightly distorted PbI_6 octahedra which are linked to polymeric chains by symmetry related atoms (symmetry code 1/2 - x, 1/2 + y, z). Bond lengths and angles are in good agreement with the other structurally characterized compounds with the same anion (Zhao *et al.*, 2010; Duan *et al.*, 2011)

S2. Experimental

A mixture of PbI_2 (461.3 mg, 1.0 mmol) and 1-(2-fluorobenzylideneamino)-quinolinium iodide (377.9 mg, 1.0 mmol) in a 1:1 molar ratio in DMF was slowly evaporated to produce orange-red needle-shaped crystals. The yield of the compound (1) was 67%.

S3. Refinement

H atoms were placed to the bonded parent atoms in geometrically idealized positions and refined as riding atoms, with U_{iso}(H) = 1.2U_{eq}(C).

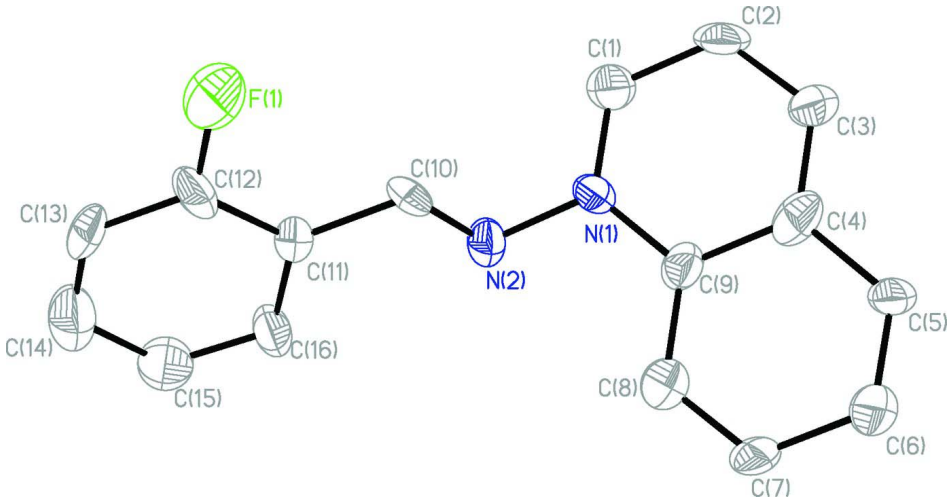

Figure 1

Molecular structure of the cation showing displacement ellipsoids at the 30% probability level.

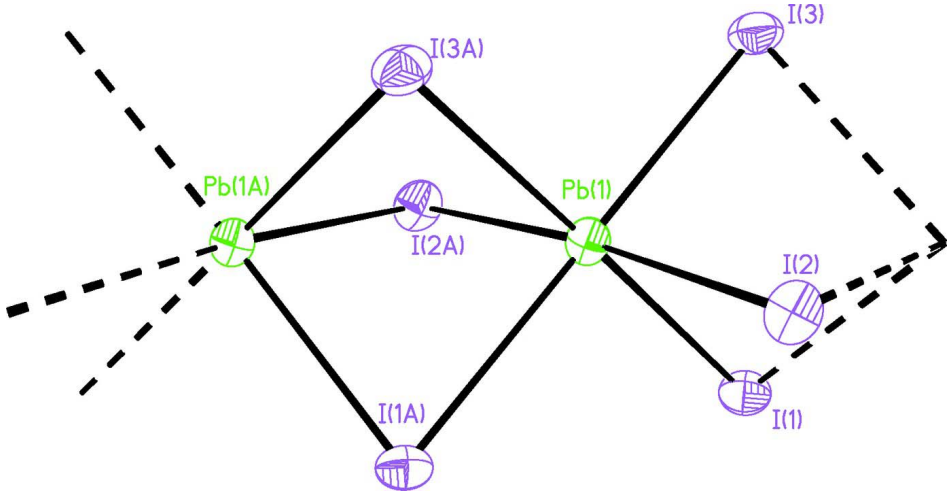

Figure 2

Cut-out of the polymeric polyanion consisting of face-sharing PbI_6 octahedra showing displacement ellipsoids at the 30% probability level.

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Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.747, T_{max} = 0.882
30847 measured reflections
4090 independent reflections
1770 reflections with $I > 2\sigma(I)$
R_{int} = 0.156
θ_{max} = 26.0°, θ_{min} = 1.6°
h = −25→25
k = −9→9
l = −31→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.082
S = 0.96
4090 reflections
208 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0158P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.96 e Å^{-3}
$\Delta\rho_{min}$ = −0.95 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C12	−0.1124 (8)	0.409 (2)	0.2967 (6)	0.073 (5)
Pb1	0.24868 (3)	0.30676 (6)	0.394853 (19)	0.04686 (15)
I1	0.31600 (4)	0.05236 (13)	0.48094 (3)	0.0538 (3)
I2	0.12891 (4)	0.05643 (12)	0.40030 (4)	0.0583 (3)
I3	0.31533 (4)	0.05436 (13)	0.30998 (3)	0.0596 (3)
F1	−0.1508 (4)	0.4781 (12)	0.2589 (4)	0.111 (4)
N1	0.0224 (5)	0.2690 (14)	0.1530 (4)	0.049 (3)
N2	0.0119 (5)	0.2585 (14)	0.2088 (4)	0.058 (3)
C1	−0.0187 (7)	0.2012 (17)	0.1191 (5)	0.060 (4)
H1	−0.0562	0.1525	0.1318	0.073*
C2	−0.0062 (7)	0.2018 (18)	0.0630 (6)	0.071 (5)
H2	−0.0359	0.1579	0.0392	0.086*
C3	0.0503 (7)	0.2683 (18)	0.0453 (6)	0.064 (4)
H3	0.0597	0.2675	0.0093	0.077*
C4	0.0927 (7)	0.336 (2)	0.0803 (7)	0.070 (5)
C5	0.1517 (7)	0.408 (2)	0.0643 (6)	0.090 (6)

H5	0.1622	0.4115	0.0284	0.108*
C6	0.1922 (7)	0.471 (2)	0.0994 (6)	0.112 (7)
H6	0.2299	0.5216	0.0877	0.134*
C7	0.1793 (7)	0.464 (2)	0.1555 (6)	0.097 (6)
H7	0.2087	0.5072	0.1796	0.117*
C8	0.1249 (7)	0.394 (2)	0.1730 (6)	0.080 (5)
H8	0.1170	0.3871	0.2092	0.096*
C9	0.0800 (7)	0.3326 (18)	0.1367 (6)	0.055 (4)
C10	−0.0397 (6)	0.3288 (15)	0.2223 (5)	0.043 (3)
H10	−0.0665	0.3773	0.1971	0.052*
C11	−0.0565 (7)	0.3323 (17)	0.2785 (5)	0.051 (4)
C13	−0.1289 (7)	0.421 (2)	0.3469 (6)	0.080 (5)
H13	−0.1660	0.4788	0.3563	0.096*
C14	−0.0916 (9)	0.350 (2)	0.3848 (7)	0.096 (6)
H14	−0.1040	0.3526	0.4202	0.115*
C15	−0.0353 (8)	0.274 (2)	0.3700 (6)	0.083 (6)
H15	−0.0097	0.2252	0.3959	0.099*
C16	−0.0158 (7)	0.2680 (18)	0.3182 (6)	0.065 (4)
H16	0.0238	0.2220	0.3093	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C12	0.076 (12)	0.102 (14)	0.042 (10)	0.015 (11)	−0.003 (9)	−0.037 (10)
Pb1	0.0469 (3)	0.0437 (3)	0.0499 (3)	−0.0005 (2)	−0.0006 (4)	−0.0024 (3)
I1	0.0494 (6)	0.0675 (6)	0.0444 (5)	−0.0033 (6)	−0.0076 (4)	−0.0005 (5)
I2	0.0422 (5)	0.0616 (6)	0.0710 (6)	−0.0023 (5)	−0.0070 (5)	−0.0082 (6)
I3	0.0676 (6)	0.0681 (6)	0.0432 (5)	−0.0080 (6)	0.0125 (5)	−0.0060 (6)
F1	0.067 (7)	0.161 (10)	0.104 (8)	0.055 (6)	0.000 (6)	−0.021 (7)
N1	0.046 (8)	0.065 (9)	0.035 (8)	0.004 (7)	−0.006 (6)	−0.009 (6)
N2	0.050 (8)	0.080 (9)	0.043 (8)	0.009 (6)	0.010 (6)	−0.005 (6)
C1	0.052 (10)	0.071 (11)	0.059 (11)	0.007 (8)	−0.001 (8)	−0.019 (8)
C2	0.037 (10)	0.117 (15)	0.060 (11)	0.014 (10)	−0.023 (8)	−0.021 (10)
C3	0.044 (10)	0.088 (12)	0.061 (11)	−0.013 (9)	−0.002 (8)	−0.015 (9)
C4	0.039 (10)	0.085 (13)	0.087 (13)	0.008 (9)	0.016 (9)	−0.004 (10)
C5	0.034 (9)	0.190 (19)	0.046 (9)	−0.021 (11)	0.002 (7)	−0.037 (11)
C6	0.059 (11)	0.21 (2)	0.062 (11)	−0.040 (12)	0.019 (10)	−0.037 (13)
C7	0.023 (8)	0.20 (2)	0.066 (11)	−0.017 (11)	0.003 (7)	−0.045 (12)
C8	0.052 (11)	0.124 (16)	0.064 (11)	−0.005 (10)	0.008 (9)	−0.012 (10)
C9	0.043 (10)	0.067 (11)	0.057 (11)	−0.004 (8)	0.013 (8)	−0.013 (9)
C10	0.057 (10)	0.046 (9)	0.027 (8)	0.006 (7)	−0.014 (7)	0.000 (6)
C11	0.044 (9)	0.065 (10)	0.042 (9)	0.001 (8)	0.007 (7)	−0.005 (7)
C13	0.035 (9)	0.134 (15)	0.072 (12)	0.023 (11)	0.016 (8)	−0.017 (12)
C14	0.089 (15)	0.123 (16)	0.077 (15)	0.019 (12)	0.020 (12)	−0.018 (12)
C15	0.071 (14)	0.109 (15)	0.068 (13)	0.014 (11)	−0.011 (10)	0.010 (11)
C16	0.065 (12)	0.095 (13)	0.036 (9)	0.017 (9)	0.005 (9)	−0.004 (9)

Geometric parameters (Å, º)

C12—C13	1.313 (17)	C3—H3	0.9300
C12—F1	1.359 (16)	C4—C5	1.418 (18)
C12—C11	1.397 (17)	C4—C9	1.446 (18)
Pb1—I3^{i}	3.1935 (12)	C5—C6	1.323 (17)
Pb1—I2	3.1938 (11)	C5—H5	0.9300
Pb1—I1^{i}	3.2102 (11)	C6—C7	1.440 (18)
Pb1—I2^{i}	3.2339 (11)	C6—H6	0.9300
Pb1—I3	3.2402 (11)	C7—C8	1.335 (18)
Pb1—I1	3.2761 (11)	C7—H7	0.9300
I1—Pb1^{ii}	3.2102 (11)	C8—C9	1.399 (18)
I2—Pb1^{ii}	3.2339 (11)	C8—H8	0.9300
I3—Pb1^{ii}	3.1935 (11)	C10—C11	1.457 (16)
N1—C1	1.324 (14)	C10—H10	0.9300
N1—C9	1.366 (15)	C11—C16	1.407 (17)
N1—N2	1.427 (13)	C13—C14	1.355 (19)
N2—C10	1.260 (14)	C13—H13	0.9300
C1—C2	1.437 (17)	C14—C15	1.373 (19)
C1—H1	0.9300	C14—H14	0.9300
C2—C3	1.366 (17)	C15—C16	1.368 (17)
C2—H2	0.9300	C15—H15	0.9300
C3—C4	1.358 (18)	C16—H16	0.9300
C13—C12—F1	119.5 (15)	C3—C4—C9	120.7 (15)
C13—C12—C11	124.6 (16)	C5—C4—C9	116.5 (15)
F1—C12—C11	115.9 (13)	C6—C5—C4	121.3 (15)
I3^{i}—Pb1—I2	94.65 (3)	C6—C5—H5	119.4
I3^{i}—Pb1—I1^{i}	84.55 (3)	C4—C5—H5	119.4
I2—Pb1—I1^{i}	90.95 (3)	C5—C6—C7	121.4 (15)
I3^{i}—Pb1—I2^{i}	89.13 (3)	C5—C6—H6	119.3
I2—Pb1—I2^{i}	175.06 (4)	C7—C6—H6	119.3
I1^{i}—Pb1—I2^{i}	86.24 (3)	C8—C7—C6	120.0 (14)
I3^{i}—Pb1—I3	96.66 (3)	C8—C7—H7	120.0
I2—Pb1—I3	89.01 (3)	C6—C7—H7	120.0
I1^{i}—Pb1—I3	178.79 (3)	C7—C8—C9	120.0 (14)
I2^{i}—Pb1—I3	93.71 (3)	C7—C8—H8	120.0
I3^{i}—Pb1—I1	179.26 (3)	C9—C8—H8	120.0
I2—Pb1—I1	85.80 (3)	N1—C9—C8	121.5 (14)
I1^{i}—Pb1—I1	96.03 (3)	N1—C9—C4	117.6 (14)
I2^{i}—Pb1—I1	90.45 (3)	C8—C9—C4	120.8 (15)
I3—Pb1—I1	82.76 (3)	N2—C10—C11	118.4 (12)
Pb1^{ii}—I1—Pb1	75.16 (2)	N2—C10—H10	120.8
Pb1—I2—Pb1^{ii}	75.97 (2)	C11—C10—H10	120.8
Pb1^{ii}—I3—Pb1	75.88 (2)	C16—C11—C12	115.4 (13)
C1—N1—C9	121.7 (13)	C16—C11—C10	122.6 (13)
C1—N1—N2	120.8 (12)	C12—C11—C10	121.9 (13)
C9—N1—N2	117.0 (12)	C12—C13—C14	119.9 (16)

C10—N2—N1	111.8 (11)	C12—C13—H13	120.1
N1—C1—C2	121.0 (14)	C14—C13—H13	120.1
N1—C1—H1	119.5	C13—C14—C15	118.9 (17)
C2—C1—H1	119.5	C13—C14—H14	120.6
C3—C2—C1	118.6 (13)	C15—C14—H14	120.6
C3—C2—H2	120.7	C16—C15—C14	122.0 (16)
C1—C2—H2	120.7	C16—C15—H15	119.0
C4—C3—C2	120.2 (15)	C14—C15—H15	119.0
C4—C3—H3	119.9	C15—C16—C11	119.1 (14)
C2—C3—H3	119.9	C15—C16—H16	120.5
C3—C4—C5	122.9 (16)	C11—C16—H16	120.5

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