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catena-Poly[1,1′-dimethyl-4,4′-(ethane-1,2-diyl)dipyridinium [lead(II)-tri-μ-iodido-lead(II)-tri-μ-iodido]]

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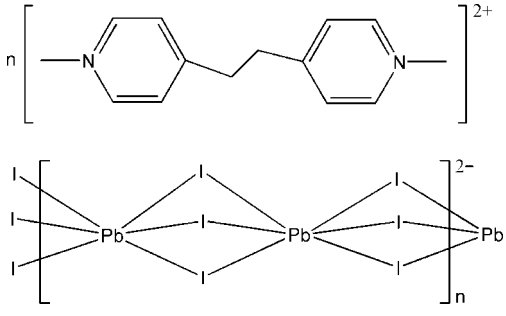
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; R factor = 0.022; wR factor = 0.046; data-to-parameter ratio = 31.4.

The title compound, $\{(C_{14}H_{18}N_2)[Pb_2I_6]\}_n$, consists of discrete 1,1′-dimethyl-4,4′-(ethane-1,2-diyl)dipyridinium cations and one-dimensional $[Pb_2I_6]_n$ anions. The organic cation has an inversion center at the mid-point of the ethane C—C bond. In the anion, the Pb^{II} atom is coordinated by six I atoms in a distorted octahedral geometry. The I atoms bridge the Pb^{II} atoms into a polymeric chain running along [001]. These inorganic chains are separated by the isolated organic cations.

Related literature

For general background to the applications of metal halides, see: Jin *et al.* (2011); Manjunatha *et al.* (2011). For bond-length data, see: Lemmerer & Billing (2006).



Experimental

Crystal data

$(C_{14}H_{18}N_2)[Pb_2I_6]$
M_r = 1390.08
Monoclinic, $P2_1/c$
a = 10.120 (6) Å
b = 17.575 (10) Å
c = 8.025 (4) Å
β = 101.239 (10)°
V = 1399.9 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 18.63 mm^{-1}
T = 296 K
0.25 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.016, T_{max} = 0.030
22768 measured reflections
3425 independent reflections
2838 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.046
S = 1.02
3425 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −1.16 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pb1—I1	3.2296 (13)	Pb1—I2^{ii}	3.1724 (11)
Pb1—I1^{i}	3.2214 (13)	Pb1—I3	3.2435 (12)
Pb1—I2	3.2311 (11)	Pb1—I3^{ii}	3.2073 (12)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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: HY2435).

References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jin, J., Jia, M.-J., Peng, Y., Yu, J.-H. & Xu, J.-Q. (2011). *CrystEngComm*, **13**, 2942–2947.
Lemmerer, A. & Billing, D. G. (2006). *Acta Cryst.* E**62**, m904–m906.
Manjunatha, M. N., Ziaulla, M., Sankolli, R., Begum, N. S. & Nagasundara, K. R. (2011). *Acta Cryst.* E**67**, m578.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, m847 [doi:10.1107/S160053681102006X]

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Guohai Xu and Xiyun He

S1. Comment

Organic–inorganic hybrid materials offer an important opportunity to combine useful properties of inorganic and organic systems within a single molecular-scale composite (Manjunatha *et al.*, 2011). As a member of this family, there continues to be interest in the study on the design and synthesis of novel metal halides due to their potential applications in the fields of optics and electrical conductivity, as well as their structural diversity (Jin *et al.*, 2011). In this contribution, we use 1,1′-dimethyl-4,4′-(ethane-1,2-diyl)dipyridinium dichloride as an organic ligand, generating an organic-inorganic hybrid, which is reported here.

The title compound consists of discrete 1,1′-dimethyl-4,4′-(ethane-1,2-diyl)dipyridinium cations and one-dimensional ploymeric anions, $[Pb_2I_6]_n$. The organic cation has an inversion center at the middle of the ethane C—C bond. In the anion, the Pb^{II} atom is coordinated by six iodide atoms in a distorted octahedral geometry (Fig. 1). The Pb—I bond lengths lie in a normal range (Table 1) (Lemmerer & Billing, 2006). The iodide atoms bridge the Pb^{II} atoms, forming polymeric chains running along [0 0 1]. These inorganic chains are separated by the isolated organic cations.

S2. Experimental

A mixture of 1,1′-dimethyl-4,4′-(ethane-1,2-diyl)dipyridinium dichloride (0.1 mmol, 0.030 g), KI (0.6 mmol, 0.10 g) and $Pb(NO_3)_2$ (0.2 mmol, 0.0662 g) in distilled water (12 ml) was placed in a Teflon-lined stainless steel vessel, heated to 423 K for 4 d and then cooled to room temperature over 12 h. Black block crystals were obtained after five months by slow evaporation of the solvent (yield: 62% based on Pb).

S3. Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (CH), 0.97 (CH_2) and 0.96 (CH_3) Å and U_{iso}(H) = 1.2(1.5 for methyl)U_{eq}(C).

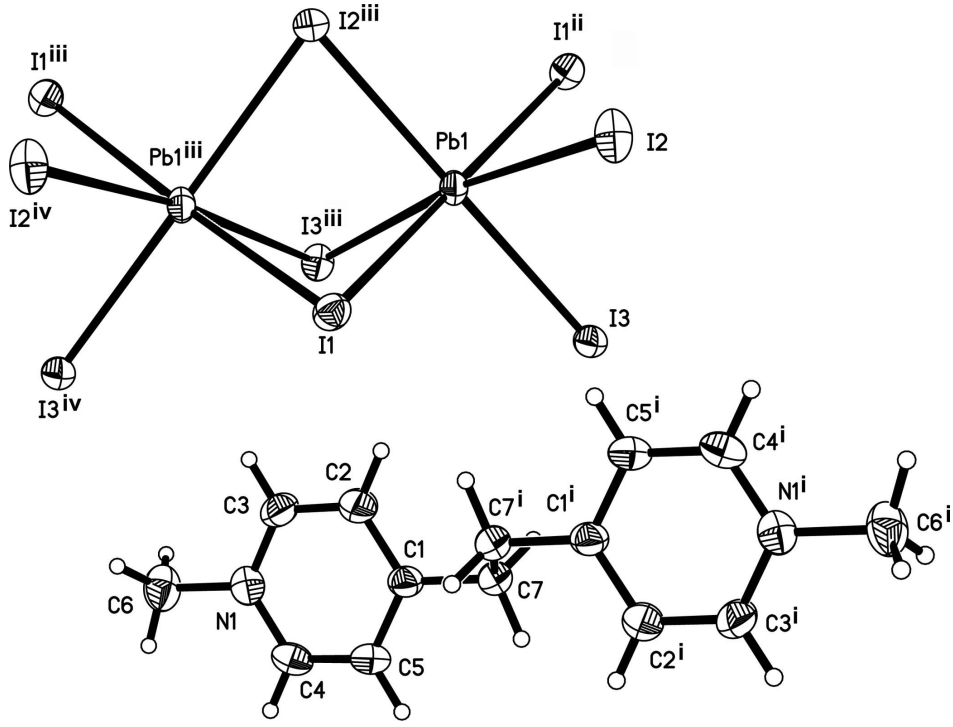

Figure 1

The structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) 1-*x*, -*y*, 1-*z*; (ii) *x*, 1/2-*y*, -1/2+*z*; (iii) *x*, 1/2-*y*, 1/2+*z*; (iv) *x*, *y*, 1+*z*.]

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.120 (6) Å
b = 17.575 (10) Å
c = 8.025 (4) Å
β = 101.239 (10)°
V = 1399.9 (13) Å^3
Z = 2
$F(000)$ = 1196
D_x = 3.298 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3425 reflections
θ = 1.0–28.2°
μ = 18.63 mm^{-1}
T = 296 K
Block, black
0.25 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.016, T_{max} = 0.030
22768 measured reflections
3425 independent reflections
2838 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 28.2°, θ_{min} = 2.6°
h = −13→13
k = −23→23
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.046$
$S = 1.02$
3425 reflections
109 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
$w = 1/[\sigma^2(F_o^2) + (0.0121P)^2 + 2.6844P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -1.16$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3721 (5)	0.0444 (3)	0.6192 (6)	0.0475 (11)
C2	0.4292 (6)	0.1058 (4)	0.7117 (8)	0.0714 (17)
H2	0.5030	0.1300	0.6817	0.086*
C3	0.3793 (6)	0.1318 (4)	0.8467 (8)	0.0678 (16)
H3	0.4190	0.1734	0.9084	0.081*
C4	0.2127 (5)	0.0402 (3)	0.7986 (7)	0.0542 (13)
H4	0.1365	0.0184	0.8274	0.065*
C5	0.2599 (5)	0.0132 (3)	0.6641 (7)	0.0495 (12)
H5	0.2158	−0.0269	0.6011	0.059*
C6	0.2247 (6)	0.1251 (4)	1.0451 (8)	0.0715 (17)
H6A	0.1488	0.0950	1.0604	0.107*
H6B	0.1983	0.1775	1.0303	0.107*
H6C	0.2955	0.1202	1.1433	0.107*
C7	0.4287 (5)	0.0148 (3)	0.4740 (6)	0.0538 (12)
H7A	0.4275	0.0552	0.3913	0.065*
H7B	0.3715	−0.0259	0.4194	0.065*
N1	0.2741 (4)	0.0979 (2)	0.8906 (5)	0.0521 (10)
I1	0.84206 (3)	0.106359 (17)	0.85470 (4)	0.04934 (8)
I2	1.04264 (4)	0.16980 (2)	0.40344 (5)	0.06218 (11)
I3	0.60383 (3)	0.190241 (18)	0.29726 (4)	0.04830 (8)
Pb1	0.835213 (18)	0.251424 (10)	0.60612 (2)	0.04107 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.043 (3)	0.050 (3)	0.047 (3)	−0.010 (2)	0.003 (2)	0.003 (2)
C2	0.060 (3)	0.092 (4)	0.068 (4)	−0.031 (3)	0.027 (3)	−0.018 (3)
C3	0.059 (3)	0.084 (4)	0.064 (4)	−0.027 (3)	0.019 (3)	−0.027 (3)
C4	0.041 (3)	0.052 (3)	0.072 (4)	−0.003 (2)	0.016 (3)	0.009 (3)
C5	0.043 (3)	0.040 (2)	0.067 (3)	−0.0040 (19)	0.012 (2)	0.000 (2)
C6	0.054 (3)	0.102 (5)	0.061 (4)	0.011 (3)	0.019 (3)	−0.008 (3)
C7	0.053 (3)	0.064 (3)	0.044 (3)	−0.017 (2)	0.008 (2)	−0.004 (2)
N1	0.045 (2)	0.063 (3)	0.048 (3)	0.0076 (19)	0.0089 (19)	0.003 (2)
I1	0.05674 (19)	0.04184 (15)	0.04717 (19)	−0.00310 (13)	0.00453 (14)	−0.00125 (13)

I2	0.0597 (2)	0.0799 (2)	0.0471 (2)	0.02966 (18)	0.01069 (16)	0.00598 (17)
I3	0.04488 (17)	0.05507 (18)	0.04462 (18)	−0.00848 (13)	0.00792 (13)	0.00162 (14)
Pb1	0.04505 (10)	0.04862 (10)	0.02984 (9)	−0.00104 (7)	0.00803 (7)	−0.00048 (7)

Geometric parameters (Å, º)

C1—C5	1.371 (6)	C6—H6A	0.9600
C1—C2	1.372 (7)	C6—H6B	0.9600
C1—C7	1.489 (7)	C6—H6C	0.9600
C2—C3	1.361 (8)	C7—C7^{i}	1.514 (10)
C2—H2	0.9300	C7—H7A	0.9700
C3—N1	1.326 (7)	C7—H7B	0.9700
C3—H3	0.9300	Pb1—I1	3.2296 (13)
C4—N1	1.334 (6)	Pb1—I1^{ii}	3.2214 (13)
C4—C5	1.349 (7)	Pb1—I2	3.2311 (11)
C4—H4	0.9300	Pb1—I2^{iii}	3.1724 (11)
C5—H5	0.9300	Pb1—I3	3.2435 (12)
C6—N1	1.502 (7)	Pb1—I3^{iii}	3.2073 (12)
C5—C1—C2	117.1 (5)	C1—C7—H7B	108.9
C5—C1—C7	122.1 (4)	C7^{i}—C7—H7B	108.9
C2—C1—C7	120.8 (4)	H7A—C7—H7B	107.7
C3—C2—C1	120.8 (5)	C3—N1—C4	120.4 (5)
C3—C2—H2	119.6	C3—N1—C6	119.3 (5)
C1—C2—H2	119.6	C4—N1—C6	120.3 (4)
N1—C3—C2	120.2 (5)	Pb1^{iii}—I1—Pb1	76.93 (4)
N1—C3—H3	119.9	Pb1^{ii}—I2—Pb1	77.60 (4)
C2—C3—H3	119.9	Pb1^{ii}—I3—Pb1	76.93 (4)
N1—C4—C5	120.7 (5)	I2^{iii}—Pb1—I3^{iii}	86.47 (4)
N1—C4—H4	119.6	I2^{iii}—Pb1—I1^{ii}	92.33 (4)
C5—C4—H4	119.6	I3^{iii}—Pb1—I1^{ii}	99.11 (3)
C4—C5—C1	120.7 (5)	I2^{iii}—Pb1—I1	87.05 (4)
C4—C5—H5	119.6	I3^{iii}—Pb1—I1	83.49 (3)
C1—C5—H5	119.6	I1^{ii}—Pb1—I1	177.283 (13)
N1—C6—H6A	109.5	I2^{iii}—Pb1—I2	99.95 (4)
N1—C6—H6B	109.5	I3^{iii}—Pb1—I2	171.539 (12)
H6A—C6—H6B	109.5	I1^{ii}—Pb1—I2	86.20 (3)
N1—C6—H6C	109.5	I1—Pb1—I2	91.30 (1)
H6A—C6—H6C	109.5	I2^{iii}—Pb1—I3	173.097 (12)
H6B—C6—H6C	109.5	I3^{iii}—Pb1—I3	89.19 (4)
C1—C7—C7^{i}	113.3 (5)	I1^{ii}—Pb1—I3	83.05 (3)
C1—C7—H7A	108.9	I1—Pb1—I3	97.80 (1)
C7^{i}—C7—H7A	108.9	I2—Pb1—I3	84.91 (1)
C5—C1—C2—C3	3.0 (9)	Pb1^{iii}—I1—Pb1—I2^{iii}	41.42 (2)
C7—C1—C2—C3	−178.5 (6)	Pb1^{iii}—I1—Pb1—I3^{iii}	−45.363 (18)
C1—C2—C3—N1	−0.1 (10)	Pb1^{iii}—I1—Pb1—I2	141.321 (18)
N1—C4—C5—C1	0.1 (8)	Pb1^{iii}—I1—Pb1—I3	−133.64 (2)

C2—C1—C5—C4	−3.0 (8)	Pb1^{ii}—I2—Pb1—I2^{iii}	−133.01 (3)
C7—C1—C5—C4	178.6 (5)	Pb1^{ii}—I2—Pb1—I1^{ii}	−41.307 (16)
C5—C1—C7—C7^{i}	−118.8 (6)	Pb1^{ii}—I2—Pb1—I1	139.759 (16)
C2—C1—C7—C7^{i}	62.9 (8)	Pb1^{ii}—I2—Pb1—I3	42.04 (2)
C2—C3—N1—C4	−3.0 (9)	Pb1^{ii}—I3—Pb1—I3^{iii}	144.45 (2)
C2—C3—N1—C6	177.1 (6)	Pb1^{ii}—I3—Pb1—I1^{ii}	45.168 (14)
C5—C4—N1—C3	3.0 (8)	Pb1^{ii}—I3—Pb1—I1	−132.231 (15)
C5—C4—N1—C6	−177.1 (5)	Pb1^{ii}—I3—Pb1—I2	−41.62 (2)

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