Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# *catena*-Poly[1,1′-dimethyl-4,4′-(ethane-1,2-diyl)dipyridinium [lead(II)-tri-$\mu$-iodido-lead(II)-tri-$\mu$-iodido]]

**Guohai Xu* and Xiyun He**

Key Laboratory of Jiangxi University for Functional Materials Chemistry, Department of Chemistry and Life Science, Gannan Normal University, Ganzhou, Jiangxi 341000, People's Republic of China
Correspondence e-mail: xugh308@126.com

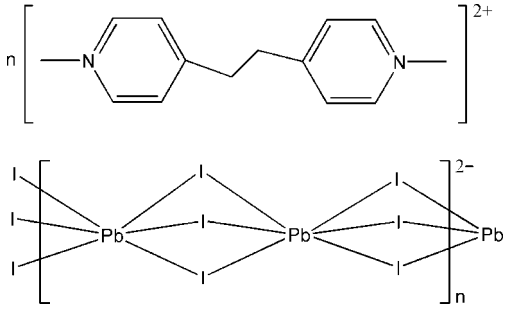
Received 20 May 2011; accepted 26 May 2011

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(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
$\beta$ = 101.239 (10)°
$V$ = 1399.9 (13) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 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*.

This work was supported by the NSF of Jiangxi Province (grant No. 2010GQH0022) and the Development Program of Science and Technology of the Education Department of Jiangxi Province (grant No. GJJ10716).

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.

# supplementary materials

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

# *catena*-Poly[1,1'-dimethyl-4,4'-(ethane-1,2-diyl)dipyridinium [lead(II)-tri-$\mu$-iodido-lead(II)-tri-$\mu$-iodido]]

**G. Xu and X. He**

## 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.

## 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).

## 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).

## Figures

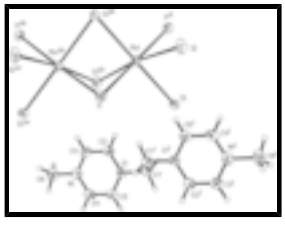


Fig. 1. The structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) 1-*x*, -*y*, 1-*z*; (ii) *x*, 1/2-*y*, -1/2+*z*; (iii) *x*, 1/2-*y*, 1/2+*z*; (iv) *x*, *y*, 1+*z*.]

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

*Crystal data*

| | |
|---|---|
| $(C_{14}H_{18}N_2)[Pb_2I_6]$ | $F(000) = 1196$ |
| $M_r = 1390.08$ | $D_x = 3.298$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 3425 reflections |
| $a = 10.120$ (6) Å | $\theta = 1.0$–$28.2°$ |
| $b = 17.575$ (10) Å | $\mu = 18.63$ mm$^{-1}$ |
| $c = 8.025$ (4) Å | $T = 296$ K |
| $\beta = 101.239$ (10)° | Block, black |
| $V = 1399.9$ (13) Å$^3$ | 0.25 × 0.20 × 0.19 mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 3425 independent reflections |
| Radiation source: fine-focus sealed tube | 2838 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.038$ |
| φ and ω scans | $\theta_{max} = 28.2°$, $\theta_{min} = 2.6°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2001) | $h = -13\rightarrow13$ |
| $T_{min} = 0.016$, $T_{max} = 0.030$ | $k = -23\rightarrow23$ |
| 22768 measured reflections | $l = -10\rightarrow10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.022$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.046$ | H-atom parameters constrained |
| $S = 1.02$ | $w = 1/[\sigma^2(F_o^2) + (0.0121P)^2 + 2.6844P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 3425 reflections | $(\Delta/\sigma)_{max} = 0.002$ |
| 109 parameters | $\Delta\rho_{max} = 0.79$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -1.16$ e Å$^{-3}$ |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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) |

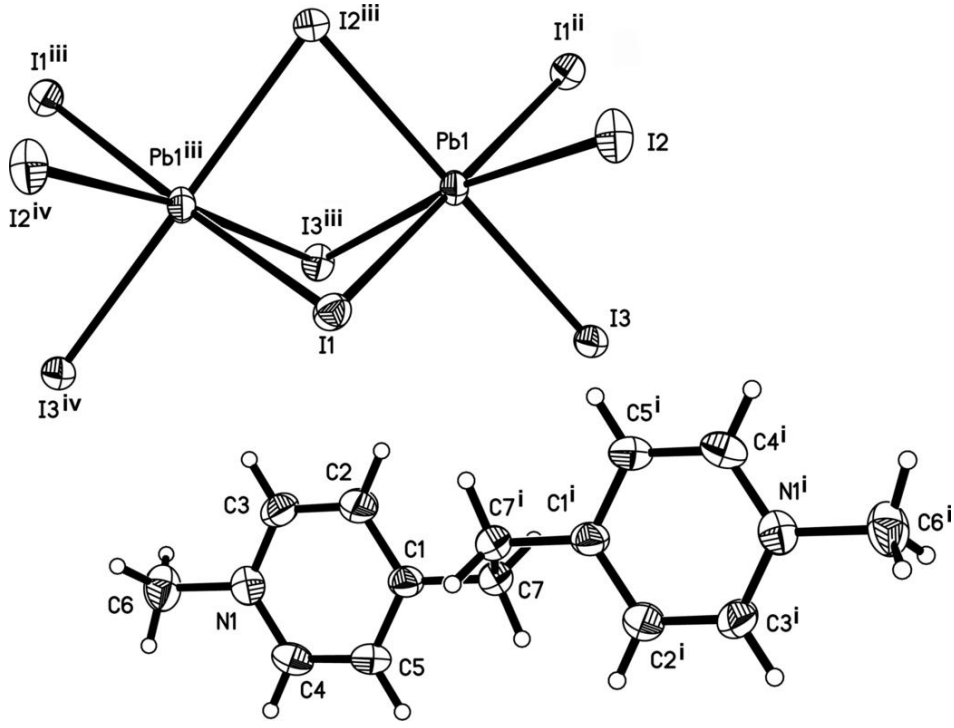
*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| 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.

**Fig. 1**


I2iii
I1ii
I1iii
Pb1
I2
Pb1iii
I2iv
I3iii
I3
I1
I3iv
C5i
C4i
C2
C3
C7i
C1i
N1i
C6i
C1
C7
C6
N1
C3i
C5
C2i
C4