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catena-Poly[[(2,9-dimethyl-1,10-phenanthroline-κ^2N,N')lead(II)]-di-μ-bromido]

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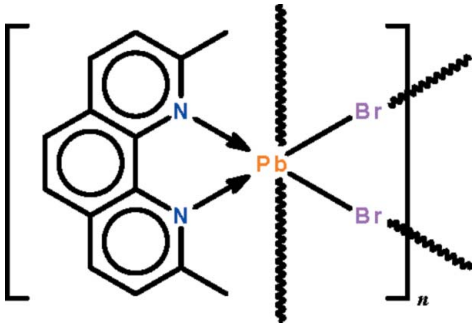
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.026; wR factor = 0.058; data-to-parameter ratio = 19.7.

In the title compound, $[PbBr_2(C_{14}H_{12}N_2)]_n$, the Pb^{II} atom lies on a twofold rotation axis. The *N*-heterocycle-chelated Pb^{II} atom exists in a distorted octahedral geometry owing to two long Pb···Br interactions [2.9562 (5) and 3.2594 (5) Å]. These result in a zigzag chain running along the *c* axis. The lone pair is stereochemically inactive.

Related literature

For the lead(II) bromide–1,10-phenanthroline homolog, see: Bowmaker *et al.* (1996).



Experimental

Crystal data

$[PbBr_2(C_{14}H_{12}N_2)]$
M_r = 575.27
Monoclinic, $C2/c$
a = 18.3852 (13) Å
b = 11.8312 (5) Å
c = 7.4609 (5) Å
β = 112.346 (8)°
V = 1501.02 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 16.55 mm^{-1}
T = 100 K
0.15 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.190, T_{max} = 0.492
4947 measured reflections
1734 independent reflections
1620 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.058
S = 1.01
1734 reflections
88 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.43 e Å^{-3}
$\Delta\rho_{min}$ = −1.45 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5905).

References

Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bowmaker, G. A., Harrowfield, J. M., Miyamae, H., Shand, T. M., Skelton, B. W. & White, A. H. (1996). *Aust. J. Chem.* **49**, 1089–1097.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2012). E**68**, m729 [doi:10.1107/S160053681201940X]

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Comment

The *N*-heterocycle chelated Pb^{II} atom in $PbBr_2(C_{14}H_{12}N_2)$ exists in a slightly distorted octahedral geometry with Pb···Br distances of 2.9562 (5)Å and 3.2594 (5)Å. The result are zigzag chains running along the *c*-axis of the monoclinic unit cell. The Pb centre lies on a twofold rotation axis. The lack of stereochemical activity can be attributed to crowding from the methyl substituents of the *N*-heterocycle (Bowmaker *et al.*, 1996).

Experimental

Lead(II) bromide (0.37 g, 1 mmol) and 2,9-dimethyl-1,10-phenanthroline (1/5, 1 mmol) were loaded in a convection tube; the tube was filled with methanol and kept at 333 K. Colorless crystals were collected from the side arm after several days.

Refinement

H-atoms were placed in calculated positions [C–H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

The final difference Fourier map had a peak at 0.82 Å and a hole at 1.01 Å from Pb1.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

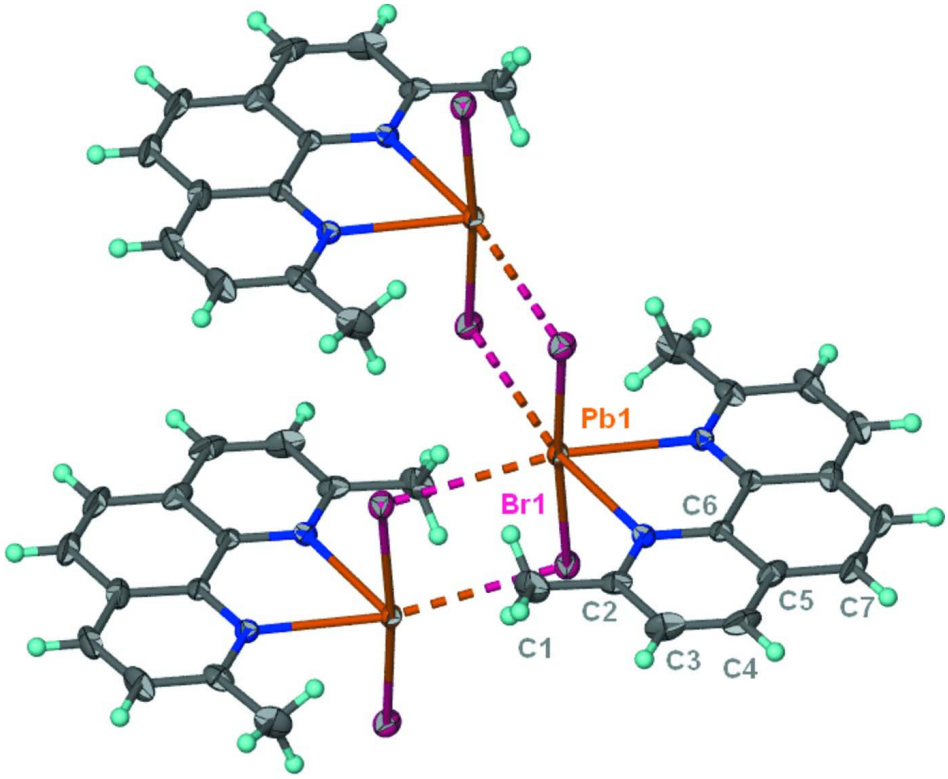

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $PbBr_2(C_{14}H_{12}N_2)$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius; symmetry-related atoms are not labeled. Adjacent molecules are linked by a weak Pb···Br bond, which is denoted as a dashed line.

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Crystal data

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$M_r = 575.27$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 18.3852\ (13)$ Å
$b = 11.8312\ (5)$ Å
$c = 7.4609\ (5)$ Å
$\beta = 112.346\ (8)°$
$V = 1501.02\ (16)$ Å^3
$Z = 4$
$F(000) = 1048$
$D_x = 2.546$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2739 reflections
$\theta = 2.4–27.5°$
$\mu = 16.55$ mm^{-1}
$T = 100$ K
Prism, colorless
0.15 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.190$, $T_{max} = 0.492$
4947 measured reflections
1734 independent reflections
1620 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.4°$
$h = -22\rightarrow23$
$k = -15\rightarrow11$
$l = -8\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.058$
$S = 1.01$
1734 reflections
88 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
$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.43$ e Å^{-3}
$\Delta\rho_{min} = -1.45$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.5000	0.399831 (16)	0.2500	0.01318 (9)
Br1	0.40287 (3)	0.38883 (3)	0.48715 (6)	0.01909 (12)
N1	0.5735 (2)	0.2162 (3)	0.3998 (5)	0.0138 (7)
C1	0.6822 (3)	0.3265 (4)	0.6184 (6)	0.0261 (11)
H1A	0.6768	0.3765	0.5092	0.039*
H1B	0.7381	0.3151	0.6972	0.039*
H1C	0.6563	0.3610	0.6980	0.039*
C2	0.6450 (3)	0.2162 (4)	0.5439 (6)	0.0183 (9)
C3	0.6839 (3)	0.1139 (4)	0.6216 (7)	0.0241 (11)
H3	0.7345	0.1152	0.7230	0.029*
C4	0.6485 (3)	0.0134 (4)	0.5503 (6)	0.0250 (11)
H4	0.6743	−0.0554	0.6030	0.030*
C5	0.5748 (3)	0.0111 (3)	0.4008 (6)	0.0206 (10)
C6	0.5374 (3)	0.1159 (3)	0.3270 (6)	0.0141 (9)
C7	0.5352 (4)	−0.0932 (3)	0.3218 (7)	0.0257 (12)
H7	0.5598	−0.1631	0.3730	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.01465 (15)	0.01085 (12)	0.01436 (13)	0.000	0.00588 (10)	0.000
Br1	0.0217 (3)	0.0169 (2)	0.0212 (2)	−0.00258 (17)	0.0109 (2)	−0.00127 (15)
N1	0.014 (2)	0.0143 (16)	0.0136 (16)	0.0002 (15)	0.0052 (15)	−0.0005 (13)
C1	0.016 (3)	0.035 (3)	0.021 (2)	−0.004 (2)	0.001 (2)	−0.0017 (19)
C2	0.016 (3)	0.026 (2)	0.0156 (19)	−0.0006 (19)	0.0089 (19)	0.0041 (17)
C3	0.014 (3)	0.034 (3)	0.023 (2)	0.009 (2)	0.005 (2)	0.0105 (18)
C4	0.032 (3)	0.024 (2)	0.024 (2)	0.013 (2)	0.017 (2)	0.0115 (19)
C5	0.028 (3)	0.017 (2)	0.024 (2)	0.0061 (19)	0.018 (2)	0.0055 (17)
C6	0.020 (3)	0.0131 (19)	0.0137 (19)	0.0009 (16)	0.011 (2)	0.0008 (14)
C7	0.045 (4)	0.011 (2)	0.032 (3)	0.0033 (19)	0.028 (3)	0.0029 (16)

Geometric parameters (Å, º)

Pb1—N1^{i}	2.578 (3)	C2—C3	1.413 (6)
Pb1—N1	2.578 (3)	C3—C4	1.362 (7)
Pb1—Br1^{i}	2.9562 (5)	C3—H3	0.9500

Pb1—Br1	2.9562 (5)	C4—C5	1.390 (7)
Pb1—Br1^{ii}	3.2594 (5)	C4—H4	0.9500
N1—C2	1.345 (6)	C5—C6	1.423 (5)
N1—C6	1.368 (5)	C5—C7	1.440 (6)
C1—C2	1.481 (6)	C6—C6^{i}	1.417 (9)
C1—H1A	0.9800	C7—C7^{i}	1.330 (12)
C1—H1B	0.9800	C7—H7	0.9500
C1—H1C	0.9800		

N1^{i}—Pb1—N1	65.15 (16)	N1—C2—C3	121.1 (4)
N1^{i}—Pb1—Br1^{i}	92.27 (7)	N1—C2—C1	118.1 (4)
N1—Pb1—Br1^{i}	83.46 (7)	C3—C2—C1	120.8 (4)
N1^{i}—Pb1—Br1	83.46 (7)	C4—C3—C2	119.6 (5)
N1—Pb1—Br1	92.27 (7)	C4—C3—H3	120.2
Br1^{i}—Pb1—Br1	174.955 (16)	C2—C3—H3	120.2
N1^{i}—Pb1—Br1^{ii}	169.82 (7)	C3—C4—C5	120.4 (4)
N1—Pb1—Br1^{ii}	107.98 (8)	C3—C4—H4	119.8
Br1^{i}—Pb1—Br1^{ii}	94.383 (13)	C5—C4—H4	119.8
Br1—Pb1—Br1^{ii}	89.490 (13)	C4—C5—C6	118.3 (4)
C2—N1—C6	119.7 (4)	C4—C5—C7	122.1 (4)
C2—N1—Pb1	122.6 (3)	C6—C5—C7	119.6 (5)
C6—N1—Pb1	117.6 (3)	N1—C6—C6^{i}	119.8 (2)
C2—C1—H1A	109.5	N1—C6—C5	120.8 (4)
C2—C1—H1B	109.5	C6^{i}—C6—C5	119.4 (3)
H1A—C1—H1B	109.5	C7^{i}—C7—C5	121.0 (3)
C2—C1—H1C	109.5	C7^{i}—C7—H7	119.5
H1A—C1—H1C	109.5	C5—C7—H7	119.5
H1B—C1—H1C	109.5		

N1^{i}—Pb1—N1—C2	179.5 (4)	C2—C3—C4—C5	0.5 (7)
Br1^{i}—Pb1—N1—C2	83.9 (3)	C3—C4—C5—C6	−0.8 (7)
Br1—Pb1—N1—C2	−98.8 (3)	C3—C4—C5—C7	−179.8 (4)
Br1^{ii}—Pb1—N1—C2	−8.6 (3)	C2—N1—C6—C6^{i}	−179.0 (4)
N1^{i}—Pb1—N1—C6	−0.2 (2)	Pb1—N1—C6—C6^{i}	0.7 (6)
Br1^{i}—Pb1—N1—C6	−95.8 (3)	C2—N1—C6—C5	−0.5 (6)
Br1—Pb1—N1—C6	81.5 (3)	Pb1—N1—C6—C5	179.2 (3)
Br1^{ii}—Pb1—N1—C6	171.7 (3)	C4—C5—C6—N1	0.8 (6)
C6—N1—C2—C3	0.2 (6)	C7—C5—C6—N1	179.8 (4)
Pb1—N1—C2—C3	−179.5 (3)	C4—C5—C6—C6^{i}	179.2 (5)
C6—N1—C2—C1	179.8 (4)	C7—C5—C6—C6^{i}	−1.7 (7)
Pb1—N1—C2—C1	0.1 (5)	C4—C5—C7—C7^{i}	−179.7 (5)
N1—C2—C3—C4	−0.3 (7)	C6—C5—C7—C7^{i}	1.3 (8)
C1—C2—C3—C4	−179.8 (4)		

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