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catena-Poly[lead(II)-[μ-2,4-diamino-6-(piperidin-1-yl)pyrimidine *N*-oxide-κ^2O:*O*]di-μ-iodido]

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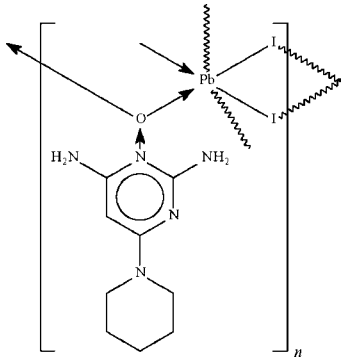
Key indicators: single-crystal X-ray study; T = 140 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.018; wR factor = 0.043; data-to-parameter ratio = 20.2.

The *N*-oxide O atom of the minoxidil unit in the 1/1 adduct with lead(II) iodide, $[PbI_2(C_9H_{15}N_5O)]_n$, bridges two Pb^{II} atoms, as do each of the I atoms. The bridging interactions give rise to a linear chain motif that propagates along the *a* axis of the orthorhombic unit cell. The coordination sphere around the six-coordinate Pb^{II} atom is a distorted ψ-monocapped octahedron in which the stereochemically active lone pair caps one of the faces defined by the O and I atoms forming the longer Pb—O or Pb—I bonds. The Pb^{II} atom lies on a mirror plane; the mirror plane is perpendicular to the pyrimidine ring and it bisects the piperidine ring. The aromatic ring is disordered about the mirror plane with respect to the 1-nitrogen and 5-carbon atoms.

Related literature

For the crystal structure of minoxidil, see: Akama *et al.* (2004); Martín-Islán *et al.* (2008).



Experimental

Crystal data

$[PbI_2(C_9H_{15}N_5O)]$
M_r = 670.25
Orthorhombic, *Pnma*
a = 8.1010 (1) Å
b = 13.5126 (2) Å
c = 14.0140 (2) Å
V = 1534.05 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 15.02 mm^{-1}
T = 140 K
0.20 × 0.10 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.153, T_{max} = 0.521
(expected range = 0.139–0.472)
10101 measured reflections
1837 independent reflections
1752 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.043
S = 1.05
1837 reflections
91 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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, 2009).

We thank IROST and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2471).

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supporting information

Acta Cryst. (2009). E**65**, m749 [doi:10.1107/S1600536809020972]

catena-Poly[lead(II)-[μ-2,4-diamino-6-(piperidin-1-yl)pyrimidine *N*-oxide-κ^2O:*O*]di-μ-iodido]

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S1. Experimental

Minoxidil [6-(1-piperidinyl)-2,4-pyrimidinediamide 3-oxide) (0.10 g, 0.5 mmol), lead(II) acetate (0.17 g, 0.5 mmol) and potassium iodide (0.16 g, 1 mmol) were placed one arm of a two-arm glass tube. Methanol was added to fill both arms. The tube was sealed and the arm containing the reactants immersed in an oil bath at 333 K while the other arm was kept at ambient temperature. After 10 days, light-brown crystals deposited in the cooler arm. These were collected, washed with acetone and ether, and finally air dried.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C). The amino H-atoms were similarly treated (N–H 0.88 Å).

The minoxidil molecule is disordered about a mirror plane that is perpendicular to the pyrimidinyl ring; the mirror plane also bisects piperidinyl ring. In the aromatic ring, the nitrogen atom at the 1-position shares the same site as the carbon atom at the 5-position; this site was refined as half a nitrogen atom and half a C-H group. The short H2···H4a distance of 1.74 Å is an artifact of the disorder about a mirror plane.

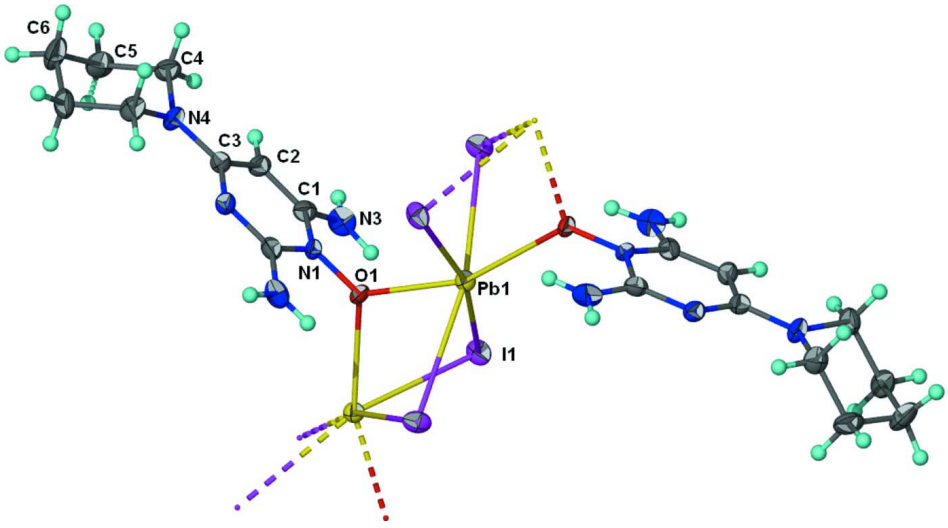

Figure 1

Thermal ellisoid plot (Barbour, 2001) of the lead diiodide–minoxidil adduct extended to show the coordination geometries of two lead atoms of the linear chain formed in the crystal structure. Atoms comprising the asymmetric unit are labelled, probability levels are set at 70% and H-atoms are drawn as spheres of arbitrary radius. Only one component of the disorder is shown.

***catena*-Poly[lead(II)-[μ-2,4-diamino-6-(piperidin-1-yl)pyrimidine *N*-oxide-κ^2O:*O*]di-μ-iodido]**

Crystal data

$[PbI_2(C_9H_{15}N_5O)]$
M_r = 670.25
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
a = 8.1010 (1) Å
b = 13.5126 (2) Å
c = 14.0140 (2) Å
V = 1534.05 (4) Å^3
Z = 4
$F(000)$ = 1200
D_x = 2.902 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5726 reflections
θ = 2.9–28.3°
μ = 15.02 mm^{-1}
T = 140 K
Prism, yellow
0.20 × 0.10 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.153, T_{max} = 0.521
10101 measured reflections
1837 independent reflections
1752 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −10→10
k = −17→17
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.043
S = 1.05
1837 reflections
91 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

$w = 1/[\sigma^2(F_o^2) + (0.0233P)^2 + 1.8105P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Pb1	0.55277 (2)	0.7500	0.769395 (12)	0.01565 (6)	
I1	0.26714 (3)	0.588041 (17)	0.831700 (16)	0.02101 (7)	
O1	0.3482 (4)	0.7500	0.6436 (2)	0.0171 (7)	
N1	0.3822 (5)	0.7500	0.5481 (3)	0.0137 (8)	
N2	0.4236 (4)	0.6615 (2)	0.4053 (2)	0.0184 (6)	0.50
N3	0.3560 (4)	0.5806 (2)	0.5498 (2)	0.0237 (7)	
H31	0.3565	0.5226	0.5213	0.028*	
H32	0.3340	0.5847	0.6112	0.028*	
N4	0.4716 (6)	0.7500	0.2638 (3)	0.0187 (9)	
C1	0.3887 (4)	0.6625 (3)	0.5000 (2)	0.0183 (7)	
C2	0.4236 (4)	0.6615 (2)	0.4053 (2)	0.0184 (6)	0.50
H2	0.4352	0.6007	0.3718	0.022*	0.50
C3	0.4417 (6)	0.7500	0.3590 (3)	0.0173 (9)	
C4	0.5041 (5)	0.6591 (3)	0.2101 (3)	0.0224 (7)	
H4A	0.4732	0.6010	0.2493	0.027*	
H4B	0.6234	0.6544	0.1956	0.027*	
C5	0.4058 (5)	0.6583 (3)	0.1176 (3)	0.0280 (9)	
H5A	0.4367	0.5992	0.0798	0.034*	
H5B	0.2867	0.6535	0.1326	0.034*	
C6	0.4363 (8)	0.7500	0.0587 (4)	0.0350 (14)	
H6A	0.5517	0.7500	0.0354	0.042*	
H6B	0.3621	0.7500	0.0026	0.042*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.01521 (10)	0.01598 (10)	0.01577 (10)	0.000	−0.00072 (6)	0.000
I1	0.02066 (14)	0.01606 (12)	0.02631 (12)	−0.00058 (9)	0.00187 (9)	0.00768 (8)
O1	0.0150 (17)	0.0263 (18)	0.0099 (13)	0.000	0.0015 (12)	0.000
N1	0.0143 (19)	0.0163 (19)	0.0105 (16)	0.000	−0.0019 (14)	0.000
N2	0.0168 (16)	0.0230 (17)	0.0155 (14)	−0.0007 (13)	0.0001 (12)	−0.0035 (12)
N3	0.0270 (17)	0.0148 (15)	0.0294 (15)	−0.0038 (13)	−0.0040 (13)	−0.0053 (12)
N4	0.026 (2)	0.018 (2)	0.0118 (18)	0.000	0.0012 (16)	0.000
C1	0.0145 (16)	0.0208 (18)	0.0197 (16)	−0.0011 (14)	−0.0007 (13)	−0.0043 (13)
C2	0.0168 (16)	0.0230 (17)	0.0155 (14)	−0.0007 (13)	0.0001 (12)	−0.0035 (12)
C3	0.014 (2)	0.022 (2)	0.016 (2)	0.000	−0.0009 (18)	0.000
C4	0.0249 (19)	0.0211 (18)	0.0211 (16)	0.0032 (16)	0.0036 (15)	−0.0038 (14)
C5	0.025 (2)	0.036 (2)	0.0226 (18)	−0.0012 (17)	−0.0035 (15)	−0.0130 (16)
C6	0.039 (4)	0.051 (4)	0.014 (2)	0.000	−0.006 (2)	0.000

Geometric parameters (Å, º)

Pb1—O1	2.419 (3)	N4—C3	1.356 (6)
Pb1—O1^{i}	2.686 (3)	N4—C4	1.464 (4)
Pb1—I1	3.3024 (3)	N4—C4^{iii}	1.464 (4)
Pb1—I1^{ii}	3.1325 (3)	C3—C2^{iii}	1.368 (4)
Pb1—I1^{i}	3.1325 (3)	C3—N2^{iii}	1.368 (4)
Pb1—I1^{iii}	3.3024 (3)	C4—C5	1.522 (5)
O1—N1	1.366 (5)	C4—H4A	0.9900
N1—C1	1.363 (4)	C4—H4B	0.9900
N1—C1^{iii}	1.363 (4)	C5—C6	1.509 (5)
N2—C1	1.358 (4)	C5—H5A	0.9900
N2—C3	1.368 (4)	C5—H5B	0.9900
N3—C1	1.335 (4)	C6—C5^{iii}	1.509 (5)
N3—H31	0.8800	C6—H6A	0.9900
N3—H32	0.8800	C6—H6B	0.9900
O1—Pb1—O1^{i}	160.22 (9)	C4—N4—C4^{iii}	114.1 (4)
O1—Pb1—I1^{i}	92.88 (5)	N3—C1—N2	123.0 (3)
O1^{i}—Pb1—I1^{i}	73.21 (5)	N3—C1—N1	116.9 (3)
O1—Pb1—I1^{ii}	92.88 (5)	N2—C1—N1	120.1 (3)
O1^{i}—Pb1—I1^{ii}	73.21 (5)	N4—C3—N2	119.0 (2)
I1^{i}—Pb1—I1^{ii}	88.636 (10)	N4—C3—C2^{iii}	119.0 (2)
O1—Pb1—I1	73.30 (5)	N2—C3—C2^{iii}	121.9 (4)
O1^{i}—Pb1—I1	120.29 (4)	N4—C3—N2^{iii}	119.0 (2)
I1^{i}—Pb1—I1	92.581 (7)	N2—C3—N2^{iii}	121.9 (4)
I1^{ii}—Pb1—I1	166.169 (8)	C2^{iii}—C3—N2^{iii}	0.0 (3)
O1—Pb1—I1^{iii}	73.30 (5)	N4—C4—C5	110.5 (3)
O1^{i}—Pb1—I1^{iii}	120.29 (4)	N4—C4—H4A	109.6
I1^{i}—Pb1—I1^{iii}	166.169 (8)	C5—C4—H4A	109.6
I1^{ii}—Pb1—I1^{iii}	92.581 (7)	N4—C4—H4B	109.6
I1—Pb1—I1^{iii}	83.011 (9)	C5—C4—H4B	109.6
Pb1^{iv}—I1—Pb1	78.806 (5)	H4A—C4—H4B	108.1
N1—O1—Pb1	125.2 (3)	C6—C5—C4	112.0 (4)
N1—O1—Pb1^{iv}	128.6 (3)	C6—C5—H5A	109.2
Pb1—O1—Pb1^{iv}	106.25 (11)	C4—C5—H5A	109.2
O1—N1—C1	119.5 (2)	C6—C5—H5B	109.2
O1—N1—C1^{iii}	119.5 (2)	C4—C5—H5B	109.2
C1—N1—C1^{iii}	120.5 (4)	H5A—C5—H5B	107.9
C1—N2—C3	118.5 (3)	C5^{iii}—C6—C5	110.4 (4)
C1—N3—H31	120.0	C5^{iii}—C6—H6A	109.6
C1—N3—H32	120.0	C5—C6—H6A	109.6
H31—N3—H32	120.0	C5^{iii}—C6—H6B	109.6
C3—N4—C4	122.51 (19)	C5—C6—H6B	109.6
C3—N4—C4^{iii}	122.51 (19)	H6A—C6—H6B	108.1
O1—Pb1—I1—Pb1^{iv}	−35.49 (5)	C3—N2—C1—N3	174.6 (4)
O1^{i}—Pb1—I1—Pb1^{iv}	160.24 (6)	C3—N2—C1—N1	−3.9 (5)

I1^{i}—Pb1—I1—Pb1^{iv}	−127.726 (10)	O1—N1—C1—N3	1.9 (5)
I1^{ii}—Pb1—I1—Pb1^{iv}	−32.94 (2)	C1^{iii}—N1—C1—N3	−169.9 (3)
I1^{iii}—Pb1—I1—Pb1^{iv}	39.115 (7)	O1—N1—C1—N2	−179.5 (3)
O1^{i}—Pb1—O1—N1	0.000 (2)	C1^{iii}—N1—C1—N2	8.7 (7)
I1^{i}—Pb1—O1—N1	−44.389 (6)	C4—N4—C3—N2	−7.1 (7)
I1^{ii}—Pb1—O1—N1	44.389 (6)	C4^{iii}—N4—C3—N2	−175.6 (4)
I1—Pb1—O1—N1	−136.222 (16)	C4—N4—C3—C2^{iii}	175.6 (4)
I1^{iii}—Pb1—O1—N1	136.222 (17)	C4^{iii}—N4—C3—C2^{iii}	7.1 (7)
O1^{i}—Pb1—O1—Pb1^{iv}	180.0	C4—N4—C3—N2^{iii}	175.6 (4)
I1^{i}—Pb1—O1—Pb1^{iv}	135.611 (6)	C4^{iii}—N4—C3—N2^{iii}	7.1 (7)
I1^{ii}—Pb1—O1—Pb1^{iv}	−135.611 (6)	C1—N2—C3—N4	−178.0 (4)
I1—Pb1—O1—Pb1^{iv}	43.778 (16)	C1—N2—C3—C2^{iii}	−0.8 (7)
I1^{iii}—Pb1—O1—Pb1^{iv}	−43.778 (16)	C1—N2—C3—N2^{iii}	−0.8 (7)
Pb1—O1—N1—C1	94.1 (3)	C3—N4—C4—C5	134.8 (5)
Pb1^{iv}—O1—N1—C1	−85.9 (3)	C4^{iii}—N4—C4—C5	−55.8 (6)
Pb1—O1—N1—C1^{iii}	−94.1 (3)	N4—C4—C5—C6	54.1 (5)
Pb1^{iv}—O1—N1—C1^{iii}	85.9 (3)	C4—C5—C6—C5^{iii}	−53.5 (6)

Symmetry codes: (i) $x+1/2, y, -z+3/2$; (ii) $x+1/2, -y+3/2, -z+3/2$; (iii) $x, -y+3/2, z$; (iv) $x-1/2, y, -z+3/2$.