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Bis(μ-5-chloroquinolin-8-olato)-κ³N,O:O;κ³O:N,O-bis[(acetato-κ²O,O′)lead(II)]

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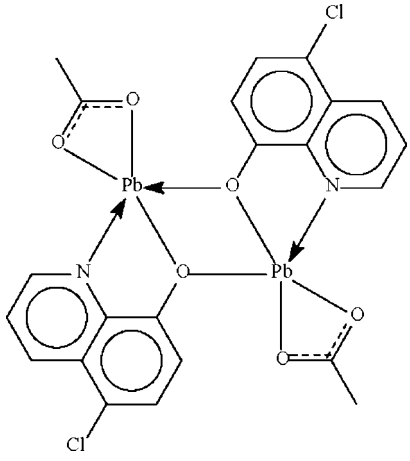
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.013 Å; R factor = 0.040; wR factor = 0.107; data-to-parameter ratio = 12.4.

The molecule of the title compound, $[Pb_2(C_9H_5ClNO)_2(C_2H_3O_2)_2]$, lies about a center of inversion. The Pb^{II} atom is chelated by acetate and substituted quinolin-8-olate anions; the O atoms of the quinolin-8-olates also bridge to confer a five-coordinate status to each metal center. The geometry approximates a distorted Ψ-*fac* octahedron in which one of the sites is occupied by a stereochemically active lone pair.

Related literature

The structural chemistry of lead(II) 8-hydroxyquinolinates has been reviewed, including bis(μ-acetato)diacetatotetrakis(μ-quinolin-8-olato)tetralead dihydrate (Shahverdizadeh *et al.*, 2008).



Experimental

Crystal data

$[Pb_2(C_9H_5ClNO)_2(C_2H_3O_2)_2]$
M_r = 889.65
Monoclinic, $P2_1/n$
a = 5.3049 (1) Å
b = 11.8200 (3) Å
c = 17.4928 (3) Å
β = 94.569 (1)°
V = 1093.38 (4) Å³
Z = 2
Mo $K\alpha$ radiation
μ = 15.67 mm⁻¹
T = 100 (2) K
0.10 × 0.03 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.303, T_{max} = 0.745
7713 measured reflections
1925 independent reflections
1655 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.00
1925 reflections
155 parameters
72 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 5.25 e Å⁻³
$\Delta\rho_{min}$ = −3.38 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 Shahid Beheshti University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2365).

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supporting information

Acta Cryst. (2009). E**65**, m261 [doi:10.1107/S1600536809003559]

Bis(*μ*-5-chloroquinolin-8-olato)-$\kappa^3N,O:O;\kappa^3O:N,O$-bis[(acetato-$\kappa^2O,O'$)lead(II)]

Gholamhossein Mohammadnezhad Sh., Mostafa M. Amini and Seik Weng Ng

S1. Experimental

Lead acetate (0.38 g, 1 mmol) and 5-chloro-8-hydroxyquinoline (0.32 g, 2 mmol) were loaded into a convection tube; the tube was filled with dry methanol and kept at 333 K. Crystals were collected from the side arm after 1 day.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C).

The crystal diffracted strongly owing to the presence of the heavy metal atom. However, this introduced severe absorption problems that could not be corrected analytically as the crystal did not have regular faces. Although a sphere of reflections was measured, multi-scan treatment only marginally improved the quality. The final difference Fourier map had large peaks/deep holes near the lead atom. The anisotropic displacement factors of the carbon atoms were restrained to be nearly isotropic.

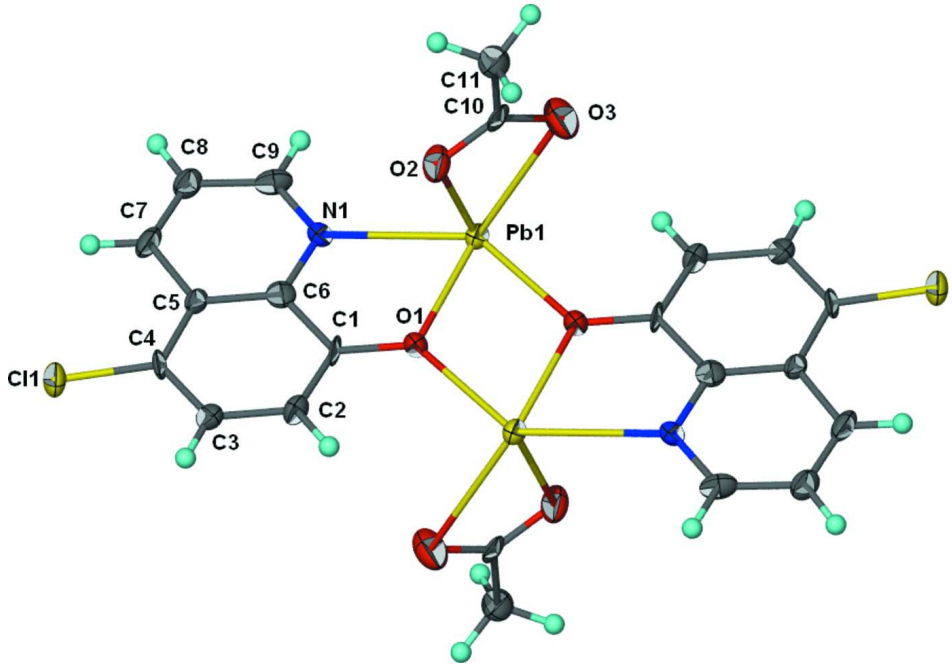

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $Pb_2(CH_3CO_2)_2(C_9H_5ClNO)_2$; ellipsoids are drawn at the 70% probability level and H atoms of arbitrary radius. Unlabelled atoms are related by 1-x, 1-y, 1-z.

Bis(μ-5-chloroquinolin-8-olato)- $\kappa^3N,O:O;\kappa^3O:N,O$- bis[(acetato-$\kappa^2O,O'$)lead(II)]

Crystal data

$[Pb_2(C_9H_5ClNO)_2(C_2H_3O_2)_2]$
$M_r = 889.65$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.3049$ (1) Å
$b = 11.8200$ (3) Å
$c = 17.4928$ (3) Å
$\beta = 94.569$ (1)°
$V = 1093.38$ (4) Å^3
$Z = 2$
$F(000) = 816$
$D_x = 2.702$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4123 reflections
$\theta = 2.9$–$28.3°$
$\mu = 15.67$ mm^{-1}
$T = 100$ K
Yellow, prism
0.10 × 0.03 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.303$, $T_{max} = 0.745$
7713 measured reflections
1925 independent reflections
1655 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
$h = -6 \rightarrow 6$
$k = -13 \rightarrow 14$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.00$
1925 reflections
155 parameters
72 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
$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 5.25$ e Å^{-3}
$\Delta\rho_{min} = -3.38$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.63668 (6)	0.60161 (3)	0.421032 (17)	0.01511 (18)
Cl1	−0.4232 (4)	0.30986 (19)	0.20373 (12)	0.0219 (5)
O1	0.3463 (11)	0.4603 (5)	0.4439 (3)	0.0175 (13)
O2	0.2902 (11)	0.7164 (6)	0.4314 (4)	0.0244 (14)
O3	0.6097 (12)	0.8054 (6)	0.4933 (4)	0.0318 (16)
N1	0.3482 (13)	0.5526 (6)	0.3014 (4)	0.0164 (15)
C1	0.1685 (16)	0.4300 (8)	0.3908 (5)	0.0149 (17)
C2	−0.0268 (17)	0.3547 (8)	0.4053 (5)	0.0186 (18)

H2	−0.0351	0.3259	0.4557	0.022*
C3	−0.2105 (16)	0.3205 (7)	0.3474 (5)	0.0186 (19)
H3	−0.3392	0.2691	0.3594	0.022*
C4	−0.2057 (16)	0.3610 (8)	0.2735 (5)	0.0154 (17)
C5	−0.0195 (16)	0.4416 (7)	0.2573 (5)	0.0143 (18)
C6	0.1645 (16)	0.4747 (7)	0.3134 (5)	0.0179 (18)
C7	−0.0074 (17)	0.4913 (8)	0.1825 (5)	0.0203 (19)
H7	−0.1270	0.4707	0.1416	0.024*
C8	0.1763 (17)	0.5679 (8)	0.1710 (5)	0.0193 (19)
H8	0.1863	0.6018	0.1221	0.023*
C9	0.3506 (19)	0.5960 (7)	0.2321 (6)	0.021 (2)
H9	0.4784	0.6497	0.2232	0.025*
C10	0.3841 (15)	0.7984 (8)	0.4697 (5)	0.0172 (18)
C11	0.202 (2)	0.8912 (7)	0.4903 (6)	0.025 (2)
H11A	0.1228	0.8698	0.5369	0.037*
H11B	0.0713	0.9013	0.4481	0.037*
H11C	0.2953	0.9622	0.4992	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0176 (2)	0.0145 (2)	0.0126 (2)	−0.00021 (12)	−0.00237 (15)	0.00053 (12)
Cl1	0.0203 (10)	0.0240 (11)	0.0198 (11)	−0.0012 (9)	−0.0085 (9)	−0.0023 (9)
O1	0.021 (3)	0.014 (3)	0.017 (3)	−0.005 (2)	−0.005 (3)	0.001 (3)
O2	0.022 (3)	0.027 (4)	0.023 (3)	0.006 (3)	−0.007 (3)	−0.005 (3)
O3	0.026 (4)	0.027 (4)	0.041 (4)	0.000 (3)	−0.004 (3)	−0.012 (3)
N1	0.022 (4)	0.012 (4)	0.015 (4)	−0.003 (3)	−0.002 (3)	0.000 (3)
C1	0.017 (3)	0.017 (3)	0.010 (3)	0.002 (3)	−0.005 (3)	−0.002 (3)
C2	0.021 (4)	0.021 (4)	0.013 (4)	−0.001 (4)	−0.003 (4)	0.003 (4)
C3	0.019 (4)	0.016 (4)	0.020 (4)	−0.001 (3)	−0.004 (3)	0.004 (3)
C4	0.016 (3)	0.015 (3)	0.013 (3)	−0.001 (3)	−0.008 (3)	−0.006 (3)
C5	0.020 (4)	0.013 (4)	0.009 (4)	0.003 (3)	−0.002 (3)	−0.001 (3)
C6	0.018 (4)	0.014 (4)	0.021 (4)	0.003 (3)	0.000 (3)	−0.001 (3)
C7	0.026 (4)	0.021 (4)	0.013 (4)	0.007 (4)	−0.006 (3)	0.001 (4)
C8	0.024 (4)	0.021 (4)	0.012 (4)	0.001 (4)	0.000 (4)	0.002 (4)
C9	0.025 (5)	0.017 (4)	0.022 (5)	0.000 (3)	0.008 (4)	0.006 (4)
C10	0.018 (4)	0.023 (4)	0.010 (4)	−0.005 (4)	−0.005 (3)	0.010 (4)
C11	0.031 (5)	0.020 (5)	0.022 (5)	0.003 (4)	−0.003 (4)	−0.004 (4)

Geometric parameters (Å, º)

Pb1—O2	2.303 (6)	C2—H2	0.9500
Pb1—O1	2.328 (6)	C3—C4	1.380 (13)
Pb1—O1^{i}	2.469 (6)	C3—H3	0.9500
Pb1—N1	2.559 (7)	C4—C5	1.417 (13)
Pb1—O3	2.729 (7)	C5—C6	1.384 (13)
Pb1—C10	2.848 (9)	C5—C7	1.441 (12)
Cl1—C4	1.722 (9)	C7—C8	1.356 (13)

O1—C1	1.319 (11)	C7—H7	0.9500
O1—Pb1^{i}	2.469 (6)	C8—C9	1.397 (15)
O2—C10	1.257 (11)	C8—H8	0.9500
O3—C10	1.237 (10)	C9—H9	0.9500
N1—C9	1.317 (12)	C10—C11	1.524 (13)
N1—C6	1.370 (11)	C11—H11A	0.9800
C1—C2	1.404 (13)	C11—H11B	0.9800
C1—C6	1.451 (13)	C11—H11C	0.9800
C2—C3	1.407 (13)		

O2—Pb1—O1	82.3 (2)	C2—C3—H3	119.6
O2—Pb1—O1^{i}	93.9 (2)	C3—C4—C5	119.1 (8)
O1—Pb1—O1^{i}	66.3 (2)	C3—C4—Cl1	118.8 (7)
O2—Pb1—N1	76.6 (2)	C5—C4—Cl1	122.2 (7)
O1—Pb1—N1	67.5 (2)	C6—C5—C4	120.7 (8)
O1^{i}—Pb1—N1	133.6 (2)	C6—C5—C7	116.7 (8)
O2—Pb1—O3	51.1 (2)	C4—C5—C7	122.6 (8)
O1—Pb1—O3	119.6 (2)	N1—C6—C5	123.4 (8)
O1^{i}—Pb1—O3	79.5 (2)	N1—C6—C1	115.5 (8)
N1—Pb1—O3	121.9 (2)	C5—C6—C1	121.0 (8)
O2—Pb1—C10	25.6 (2)	C8—C7—C5	119.4 (9)
O1—Pb1—C10	101.6 (2)	C8—C7—H7	120.3
O1^{i}—Pb1—C10	86.5 (2)	C5—C7—H7	120.3
N1—Pb1—C10	99.5 (2)	C7—C8—C9	119.0 (8)
O3—Pb1—C10	25.5 (2)	C7—C8—H8	120.5
C1—O1—Pb1	121.3 (5)	C9—C8—H8	120.5
C1—O1—Pb1^{i}	124.2 (5)	N1—C9—C8	123.9 (8)
Pb1—O1—Pb1^{i}	113.7 (2)	N1—C9—H9	118.1
C10—O2—Pb1	102.2 (5)	C8—C9—H9	118.1
C10—O3—Pb1	82.6 (5)	O3—C10—O2	124.1 (9)
C9—N1—C6	117.6 (8)	O3—C10—C11	119.2 (8)
C9—N1—Pb1	128.0 (6)	O2—C10—C11	116.7 (8)
C6—N1—Pb1	114.4 (6)	O3—C10—Pb1	71.9 (5)
O1—C1—C2	122.8 (8)	O2—C10—Pb1	52.2 (4)
O1—C1—C6	120.9 (8)	C11—C10—Pb1	168.8 (6)
C2—C1—C6	116.3 (8)	C10—C11—H11A	109.5
C1—C2—C3	122.1 (8)	C10—C11—H11B	109.5
C1—C2—H2	119.0	H11A—C11—H11B	109.5
C3—C2—H2	119.0	C10—C11—H11C	109.5
C4—C3—C2	120.7 (8)	H11A—C11—H11C	109.5
C4—C3—H3	119.6	H11B—C11—H11C	109.5

O2—Pb1—O1—C1	73.2 (6)	Cl1—C4—C5—C6	−175.1 (6)
O1^{i}—Pb1—O1—C1	170.9 (8)	C3—C4—C5—C7	−177.6 (8)
N1—Pb1—O1—C1	−5.5 (6)	Cl1—C4—C5—C7	3.6 (12)
O3—Pb1—O1—C1	109.5 (6)	C9—N1—C6—C5	−1.4 (12)
C10—Pb1—O1—C1	90.1 (6)	Pb1—N1—C6—C5	176.2 (6)
O2—Pb1—O1—Pb1^{i}	−97.7 (3)	C9—N1—C6—C1	−179.6 (8)

O1^{i}—Pb1—O1—Pb1^{i}	0.0	Pb1—N1—C6—C1	−2.0 (9)
N1—Pb1—O1—Pb1^{i}	−176.4 (3)	C4—C5—C6—N1	−179.6 (8)
O3—Pb1—O1—Pb1^{i}	−61.4 (3)	C7—C5—C6—N1	1.7 (12)
C10—Pb1—O1—Pb1^{i}	−80.8 (3)	C4—C5—C6—C1	−1.5 (12)
O1—Pb1—O2—C10	138.8 (5)	C7—C5—C6—C1	179.8 (8)
O1^{i}—Pb1—O2—C10	73.4 (5)	O1—C1—C6—N1	−2.8 (12)
N1—Pb1—O2—C10	−152.6 (6)	C2—C1—C6—N1	176.6 (8)
O3—Pb1—O2—C10	0.3 (5)	O1—C1—C6—C5	179.0 (8)
O2—Pb1—O3—C10	−0.3 (5)	C2—C1—C6—C5	−1.6 (12)
O1—Pb1—O3—C10	−49.2 (6)	C6—C5—C7—C8	−1.2 (12)
O1^{i}—Pb1—O3—C10	−104.0 (5)	C4—C5—C7—C8	−179.9 (8)
N1—Pb1—O3—C10	31.3 (6)	C5—C7—C8—C9	0.4 (13)
O2—Pb1—N1—C9	93.9 (7)	C6—N1—C9—C8	0.6 (13)
O1—Pb1—N1—C9	−179.0 (8)	Pb1—N1—C9—C8	−176.6 (6)
O1^{i}—Pb1—N1—C9	176.4 (6)	C7—C8—C9—N1	−0.1 (14)
O3—Pb1—N1—C9	69.1 (8)	Pb1—O3—C10—O2	0.4 (8)
C10—Pb1—N1—C9	82.3 (7)	Pb1—O3—C10—C11	177.7 (7)
O2—Pb1—N1—C6	−83.4 (6)	Pb1—O2—C10—O3	−0.5 (10)
O1—Pb1—N1—C6	3.7 (5)	Pb1—O2—C10—C11	−177.9 (6)
O1^{i}—Pb1—N1—C6	−0.9 (7)	O2—Pb1—C10—O3	179.5 (8)
O3—Pb1—N1—C6	−108.1 (6)	O1—Pb1—C10—O3	137.8 (5)
C10—Pb1—N1—C6	−95.0 (6)	O1^{i}—Pb1—C10—O3	72.9 (5)
Pb1—O1—C1—C2	−172.5 (7)	N1—Pb1—C10—O3	−153.5 (5)
Pb1^{i}—O1—C1—C2	−2.6 (11)	O1—Pb1—C10—O2	−41.8 (6)
Pb1—O1—C1—C6	6.8 (10)	O1^{i}—Pb1—C10—O2	−106.7 (5)
Pb1^{i}—O1—C1—C6	176.8 (6)	N1—Pb1—C10—O2	27.0 (6)
O1—C1—C2—C3	−178.1 (8)	O3—Pb1—C10—O2	−179.5 (8)
C6—C1—C2—C3	2.6 (13)	O2—Pb1—C10—C11	10 (3)
C1—C2—C3—C4	−0.4 (14)	O1—Pb1—C10—C11	−32 (3)
C2—C3—C4—C5	−2.8 (13)	O1^{i}—Pb1—C10—C11	−97 (3)
C2—C3—C4—Cl1	176.0 (7)	N1—Pb1—C10—C11	37 (3)
C3—C4—C5—C6	3.7 (13)	O3—Pb1—C10—C11	−170 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.