

**Bis(μ -2-methylquinolin-8-olato)-
 $\kappa^3 N,O;O;\kappa^3 O:N,O$ -bis[(methanol- κ O)-
(nitrato- $\kappa^2 O,O'$)lead(II)]**

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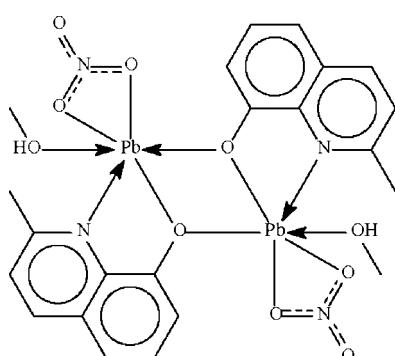
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.007$ Å; R factor = 0.021; wR factor = 0.058; data-to-parameter ratio = 16.5.

The molecule of the title compound, $[Pb_2(C_{10}H_8NO)_2](NO_3)_2(CH_3OH)_2$, lies about a centre of inversion. The Pb atom is chelated by nitrate and substituted quinolin-8-olate anions. The O atom of the quinolin-8-olate also bridges, to confer a six-coordinate status on the metal centre. When a longer Pb···O interaction is considered, the geometry approximates a Ψ -cube in which one of the sites is occupied by a stereochemically active lone pair.

Related literature

The 8-hydroxyquinolinate group engages in μ_3 -bridging in dinitratohexa(quinolin-8-olato)tetralead(II); see: Zhang *et al.* (2008). It also exhibits this feature in the chain compound, bis(methanol)dinitratodi(quinolin-8-olato)dilead(II); see Shahverdizadeh *et al.* (2008). Both reports comment on lone-pair stereochemistry in this class of lead(II) compounds.



Experimental

Crystal data

$[Pb_2(C_{10}H_8NO)_2](NO_3)_2(CH_3OH)_2$	$\gamma = 108.190$ (1) $^\circ$
$M_r = 918.83$	$V = 630.07$ (1) Å 3
Triclinic, $P\bar{1}$	$Z = 1$
$a = 8.2579$ (1) Å	Mo $K\alpha$ radiation
$b = 8.8052$ (1) Å	$\mu = 13.41$ mm $^{-1}$
$c = 9.6765$ (1) Å	$T = 100$ (2) K
$\alpha = 103.976$ (1) $^\circ$	$0.20 \times 0.15 \times 0.05$ mm
$\beta = 98.262$ (1) $^\circ$	

Data collection

Bruker SMART APEX	5958 measured reflections
diffractometer	2872 independent reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	2754 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.175$, $T_{\max} = 0.554$	$R_{\text{int}} = 0.019$
(expected range = 0.162–0.512)	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$	H atoms treated by a mixture of
$wR(F^2) = 0.058$	independent and constrained
$S = 1.08$	refinement
2872 reflections	$\Delta\rho_{\max} = 1.41$ e Å $^{-3}$
174 parameters	$\Delta\rho_{\min} = -2.12$ e Å $^{-3}$
1 restraint	

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2363).

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supplementary materials

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Bis(μ -2-methylquinolin-8-olato)- $\kappa^3N,O;O;\kappa^3O:N,O$ -bis[(methanol- κO)(nitrato- κ^2O,O')lead(II)]

G. Mohammadnezhad Sh., M. M. Amini and S. W. Ng

Experimental

Lead nitrate (0.33 g, 1 mmol) and 2-methyl-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 3 d.

Refinement

Carbon-bound H atoms were placed in calculated positions (C—H 0.93 to 0.98 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to 1.2 to 1.5 $U(C)$. The methanol H atom was located in a difference Fourier map, and was refined with a distance restraint of O—H 0.84 (1) Å; its temperature factor was freely refined.

The crystal diffracted strongly owing to the extremely heavy metal atom; however, its presence 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 Pb atom.

Figures

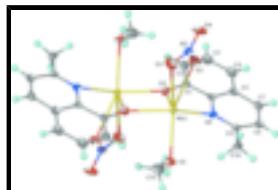


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $Pb_2(NO_3)_2(CH_4O)_2(C_{10}H_8NO)_2$; ellipsoids are drawn at the 70% probability level and H atoms of arbitrary radius. The unlabelled atoms are related by 1- x , 2- y , 1- z .

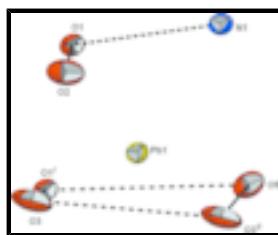


Fig. 2. Detail of the environment of the Pb atom. Symmetry codes: (i) 1 - x , 2 - y , 1 - z ; (ii) 1 - x , 1 - y , 1 - z .

Bis(μ -2-methylquinolin-8-olato)- $\kappa^3N,O;O;\kappa^3O:N,O$ -bis[(methanol- κO)(nitrato- κ^2O,O')lead(II)]

Crystal data

[$Pb_2(C_{10}H_8NO)_2(NO_3)_2(CH_4O)_2$]

$Z = 1$

$M_r = 918.83$

$F_{000} = 428$

Triclinic, $P\bar{1}$

$D_x = 2.422 \text{ Mg m}^{-3}$

supplementary materials

Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 8.2579 (1) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.8052 (1) \text{ \AA}$	Cell parameters from 5067 reflections
$c = 9.6765 (1) \text{ \AA}$	$\theta = 2.2\text{--}28.3^\circ$
$\alpha = 103.976 (1)^\circ$	$\mu = 13.41 \text{ mm}^{-1}$
$\beta = 98.262 (1)^\circ$	$T = 100 \text{ K}$
$\gamma = 108.190 (1)^\circ$	Block, yellow
$V = 630.07 (1) \text{ \AA}^3$	$0.20 \times 0.15 \times 0.05 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer	2872 independent reflections
Radiation source: fine-focus sealed tube	2754 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.019$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
ω scans	$\theta_{\text{min}} = 2.2^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.175, T_{\text{max}} = 0.554$	$k = -11 \rightarrow 11$
5958 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.021$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.058$	$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 1.3909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2872 reflections	$\Delta\rho_{\text{max}} = 1.41 \text{ e \AA}^{-3}$
174 parameters	$\Delta\rho_{\text{min}} = -2.12 \text{ e \AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.403585 (18)	0.747859 (17)	0.434423 (15)	0.01020 (6)
O1	0.5672 (4)	0.9839 (4)	0.3886 (3)	0.0139 (6)
O2	0.7047 (5)	0.7066 (4)	0.4111 (4)	0.0202 (7)
O3	0.6609 (5)	0.6459 (5)	0.6104 (4)	0.0263 (8)
O4	0.8652 (5)	0.5918 (5)	0.5137 (4)	0.0261 (8)
O5	0.0903 (4)	0.7403 (4)	0.3446 (4)	0.0175 (6)

H5	0.006 (6)	0.691 (7)	0.376 (7)	0.028 (17)*
N1	0.3858 (5)	0.7002 (4)	0.1665 (4)	0.0114 (7)
N2	0.7441 (5)	0.6484 (5)	0.5124 (4)	0.0146 (7)
C1	0.6165 (6)	0.9696 (5)	0.2615 (5)	0.0123 (8)
C2	0.7547 (6)	1.0900 (6)	0.2411 (5)	0.0169 (9)
H2	0.8219	1.1885	0.3203	0.020*
C3	0.7985 (6)	1.0691 (6)	0.1028 (5)	0.0205 (9)
H3	0.8963	1.1529	0.0913	0.025*
C4	0.7024 (6)	0.9308 (6)	-0.0140 (5)	0.0199 (9)
H4	0.7315	0.9203	-0.1065	0.024*
C5	0.5592 (6)	0.8028 (5)	0.0032 (5)	0.0141 (8)
C6	0.5180 (5)	0.8209 (5)	0.1415 (4)	0.0115 (8)
C7	0.4520 (6)	0.6577 (6)	-0.1116 (5)	0.0167 (9)
H7	0.4731	0.6417	-0.2070	0.020*
C8	0.3161 (6)	0.5389 (6)	-0.0852 (5)	0.0165 (9)
H8	0.2419	0.4414	-0.1626	0.020*
C9	0.2879 (5)	0.5628 (5)	0.0573 (5)	0.0121 (8)
C10	0.1480 (5)	0.4306 (4)	0.0890 (5)	0.0160 (9)
H10A	0.2007	0.3646	0.1358	0.024*
H10B	0.0632	0.3567	-0.0028	0.024*
H10C	0.0879	0.4834	0.1549	0.024*
C11	0.0687 (6)	0.8890 (4)	0.3246 (5)	0.0257 (11)
H11A	0.0928	0.9717	0.4205	0.039*
H11B	-0.0520	0.8616	0.2712	0.039*
H11C	0.1504	0.9358	0.2684	0.039*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.01050 (9)	0.00980 (9)	0.01087 (9)	0.00331 (6)	0.00258 (6)	0.00458 (6)
O1	0.0147 (15)	0.0116 (13)	0.0134 (14)	0.0013 (12)	0.0045 (12)	0.0043 (11)
O2	0.0235 (18)	0.0268 (17)	0.0212 (16)	0.0148 (14)	0.0101 (14)	0.0161 (14)
O3	0.0270 (19)	0.046 (2)	0.0163 (16)	0.0181 (17)	0.0125 (14)	0.0177 (16)
O4	0.0162 (17)	0.0333 (19)	0.039 (2)	0.0136 (15)	0.0108 (15)	0.0205 (17)
O5	0.0129 (16)	0.0205 (16)	0.0227 (16)	0.0073 (13)	0.0072 (13)	0.0098 (13)
N1	0.0117 (17)	0.0121 (16)	0.0120 (16)	0.0041 (14)	0.0028 (13)	0.0067 (13)
N2	0.0126 (18)	0.0164 (17)	0.0159 (17)	0.0051 (14)	0.0036 (14)	0.0068 (14)
C1	0.014 (2)	0.0129 (18)	0.0128 (19)	0.0072 (16)	0.0030 (16)	0.0055 (16)
C2	0.017 (2)	0.015 (2)	0.017 (2)	0.0048 (17)	0.0033 (17)	0.0049 (17)
C3	0.018 (2)	0.019 (2)	0.027 (2)	0.0039 (18)	0.0113 (19)	0.0115 (19)
C4	0.021 (2)	0.022 (2)	0.022 (2)	0.0080 (19)	0.0124 (19)	0.0110 (19)
C5	0.015 (2)	0.017 (2)	0.0138 (19)	0.0064 (17)	0.0063 (16)	0.0096 (17)
C6	0.0095 (19)	0.0148 (19)	0.0134 (19)	0.0071 (16)	0.0022 (15)	0.0067 (16)
C7	0.018 (2)	0.021 (2)	0.014 (2)	0.0101 (18)	0.0053 (17)	0.0049 (17)
C8	0.014 (2)	0.017 (2)	0.017 (2)	0.0061 (17)	0.0020 (16)	0.0030 (17)
C9	0.0088 (19)	0.0137 (19)	0.0140 (19)	0.0047 (16)	0.0011 (15)	0.0045 (16)
C10	0.011 (2)	0.0125 (19)	0.020 (2)	-0.0014 (16)	0.0022 (16)	0.0038 (16)
C11	0.022 (3)	0.024 (2)	0.036 (3)	0.010 (2)	0.008 (2)	0.014 (2)

supplementary materials

Geometric parameters (\AA , $^\circ$)

Pb1—O1	2.281 (3)	C2—C3	1.421 (6)
Pb1—O1 ⁱ	2.478 (3)	C2—H2	0.9500
Pb1—N1	2.499 (3)	C3—C4	1.365 (7)
Pb1—O5	2.583 (3)	C3—H3	0.9500
Pb1—O2	2.655 (3)	C4—C5	1.418 (6)
Pb1—O3	3.019 (4)	C4—H4	0.9500
Pb1—O3 ⁱⁱ	3.248 (4)	C5—C7	1.406 (6)
Pb1—O4 ⁱⁱ	3.320 (4)	C5—C6	1.412 (6)
O1—C1	1.341 (5)	C7—C8	1.378 (6)
O1—Pb1 ⁱ	2.478 (3)	C7—H7	0.9500
O2—N2	1.259 (5)	C8—C9	1.408 (6)
O3—N2	1.248 (5)	C8—H8	0.9500
O4—N2	1.248 (5)	C9—C10	1.490 (5)
O5—C11	1.430 (5)	C10—H10A	0.9800
O5—H5	0.838 (10)	C10—H10B	0.9800
N1—C9	1.326 (5)	C10—H10C	0.9800
N1—C6	1.362 (5)	C11—H11A	0.9800
C1—C2	1.369 (6)	C11—H11B	0.9800
C1—C6	1.433 (6)	C11—H11C	0.9800
O1—Pb1—O1 ⁱ	64.88 (12)	O4—N2—O2	119.8 (4)
O1—Pb1—N1	68.38 (11)	O1—C1—C2	123.2 (4)
O1 ⁱ —Pb1—N1	124.94 (11)	O1—C1—C6	117.8 (4)
O1—Pb1—O5	100.85 (11)	C2—C1—C6	119.0 (4)
O1 ⁱ —Pb1—O5	82.53 (10)	C1—C2—C3	120.6 (4)
N1—Pb1—O5	79.21 (11)	C1—C2—H2	119.7
O1—Pb1—O2	75.46 (11)	C3—C2—H2	119.7
O1 ⁱ —Pb1—O2	114.95 (11)	C4—C3—C2	121.2 (4)
N1—Pb1—O2	78.25 (11)	C4—C3—H3	119.4
O5—Pb1—O2	156.87 (11)	C2—C3—H3	119.4
O1—Pb1—O3	105.98 (11)	C3—C4—C5	119.8 (4)
O1 ⁱ —Pb1—O3	100.58 (10)	C3—C4—H4	120.1
N1—Pb1—O3	119.16 (10)	C5—C4—H4	120.1
O5—Pb1—O3	151.65 (10)	C7—C5—C6	117.2 (4)
O2—Pb1—O3	44.42 (9)	C7—C5—C4	123.5 (4)
O1—Pb1—O3 ⁱⁱ	144.45 (10)	C6—C5—C4	119.3 (4)
O1 ⁱ —Pb1—O3 ⁱⁱ	144.84 (9)	N1—C6—C5	122.2 (4)
N1—Pb1—O3 ⁱⁱ	89.96 (10)	N1—C6—C1	117.7 (4)
O5—Pb1—O3 ⁱⁱ	102.30 (10)	C5—C6—C1	120.2 (4)
O2—Pb1—O3 ⁱⁱ	72.63 (10)	C8—C7—C5	119.8 (4)
O3—Pb1—O3 ⁱⁱ	59.42 (12)	C8—C7—H7	120.1
O1—Pb1—O4 ⁱⁱ	175.03 (10)	C5—C7—H7	120.1
O1 ⁱ —Pb1—O4 ⁱⁱ	114.40 (10)	C7—C8—C9	119.6 (4)

N1—Pb1—O4 ⁱⁱ	109.62 (11)	C7—C8—H8	120.2
O5—Pb1—O4 ⁱⁱ	74.21 (9)	C9—C8—H8	120.2
O2—Pb1—O4 ⁱⁱ	108.83 (9)	N1—C9—C8	121.5 (4)
O3—Pb1—O4 ⁱⁱ	78.99 (10)	N1—C9—C10	118.5 (4)
O3 ⁱⁱ —Pb1—O4 ⁱⁱ	38.47 (9)	C8—C9—C10	120.0 (4)
C1—O1—Pb1	119.2 (2)	C9—C10—H10A	109.5
C1—O1—Pb1 ⁱ	124.9 (2)	C9—C10—H10B	109.5
Pb1—O1—Pb1 ⁱ	115.12 (12)	H10A—C10—H10B	109.5
N2—O2—Pb1	106.1 (2)	C9—C10—H10C	109.5
N2—O3—Pb1	88.6 (2)	H10A—C10—H10C	109.5
C11—O5—Pb1	118.7 (2)	H10B—C10—H10C	109.5
C11—O5—H5	108 (4)	O5—C11—H11A	109.5
Pb1—O5—H5	122 (4)	O5—C11—H11B	109.5
C9—N1—C6	119.7 (4)	H11A—C11—H11B	109.5
C9—N1—Pb1	127.4 (3)	O5—C11—H11C	109.5
C6—N1—Pb1	111.6 (3)	H11A—C11—H11C	109.5
O3—N2—O4	120.2 (4)	H11B—C11—H11C	109.5
O3—N2—O2	119.9 (4)		
O1 ⁱ —Pb1—O1—C1	−170.3 (4)	O1—Pb1—N1—C6	17.3 (3)
N1—Pb1—O1—C1	−20.3 (3)	O1 ⁱ —Pb1—N1—C6	50.8 (3)
O5—Pb1—O1—C1	−93.9 (3)	O5—Pb1—N1—C6	123.7 (3)
O2—Pb1—O1—C1	62.6 (3)	O2—Pb1—N1—C6	−61.5 (3)
O3—Pb1—O1—C1	95.3 (3)	O3—Pb1—N1—C6	−79.4 (3)
O3 ⁱⁱ —Pb1—O1—C1	36.0 (4)	O3 ⁱⁱ —Pb1—N1—C6	−133.8 (3)
O4 ⁱⁱ —Pb1—O1—C1	−87.4 (12)	O4 ⁱⁱ —Pb1—N1—C6	−167.5 (3)
O1 ⁱ —Pb1—O1—Pb1 ⁱ	0.0	Pb1—O3—N2—O4	−170.3 (4)
N1—Pb1—O1—Pb1 ⁱ	150.03 (17)	Pb1—O3—N2—O2	9.0 (4)
O5—Pb1—O1—Pb1 ⁱ	76.34 (14)	Pb1—O2—N2—O3	−10.7 (5)
O2—Pb1—O1—Pb1 ⁱ	−127.07 (15)	Pb1—O2—N2—O4	168.6 (3)
O3—Pb1—O1—Pb1 ⁱ	−94.40 (14)	Pb1—O1—C1—C2	−159.2 (3)
O3 ⁱⁱ —Pb1—O1—Pb1 ⁱ	−153.76 (12)	Pb1 ⁱ —O1—C1—C2	31.5 (6)
O4 ⁱⁱ —Pb1—O1—Pb1 ⁱ	82.9 (12)	Pb1—O1—C1—C6	21.2 (5)
O1—Pb1—O2—N2	137.6 (3)	Pb1 ⁱ —O1—C1—C6	−148.1 (3)
O1 ⁱ —Pb1—O2—N2	84.7 (3)	O1—C1—C2—C3	−178.8 (4)
N1—Pb1—O2—N2	−152.0 (3)	C6—C1—C2—C3	0.8 (7)
O5—Pb1—O2—N2	−138.9 (3)	C1—C2—C3—C4	1.4 (7)
O3—Pb1—O2—N2	5.4 (2)	C2—C3—C4—C5	−1.8 (7)
O3 ⁱⁱ —Pb1—O2—N2	−58.3 (3)	C3—C4—C5—C7	179.0 (5)
O4 ⁱⁱ —Pb1—O2—N2	−45.1 (3)	C3—C4—C5—C6	0.1 (7)
O1—Pb1—O3—N2	−53.6 (3)	C9—N1—C6—C5	−2.1 (6)
O1 ⁱ —Pb1—O3—N2	−120.3 (3)	Pb1—N1—C6—C5	166.0 (3)
N1—Pb1—O3—N2	20.2 (3)	C9—N1—C6—C1	178.0 (4)
O5—Pb1—O3—N2	145.9 (3)	Pb1—N1—C6—C1	−13.9 (4)
O2—Pb1—O3—N2	−5.3 (2)	C7—C5—C6—N1	3.2 (6)

supplementary materials

O3 ⁱⁱ —Pb1—O3—N2	90.9 (3)	C4—C5—C6—N1	-177.8 (4)
O4 ⁱⁱ —Pb1—O3—N2	126.7 (3)	C7—C5—C6—C1	-176.9 (4)
O1—Pb1—O5—C11	-16.8 (3)	C4—C5—C6—C1	2.0 (6)
O1 ⁱ —Pb1—O5—C11	45.7 (3)	O1—C1—C6—N1	-3.0 (6)
N1—Pb1—O5—C11	-82.1 (3)	C2—C1—C6—N1	177.4 (4)
O2—Pb1—O5—C11	-95.2 (4)	O1—C1—C6—C5	177.1 (4)
O3—Pb1—O5—C11	144.2 (3)	C2—C1—C6—C5	-2.5 (6)
O3 ⁱⁱ —Pb1—O5—C11	-169.7 (3)	C6—C5—C7—C8	-1.5 (6)
O4 ⁱⁱ —Pb1—O5—C11	163.8 (3)	C4—C5—C7—C8	179.6 (4)
O1—Pb1—N1—C9	-175.7 (4)	C5—C7—C8—C9	-1.0 (7)
O1 ⁱ —Pb1—N1—C9	-142.2 (3)	C6—N1—C9—C8	-0.7 (6)
O5—Pb1—N1—C9	-69.4 (3)	Pb1—N1—C9—C8	-166.7 (3)
O2—Pb1—N1—C9	105.4 (4)	C6—N1—C9—C10	178.1 (4)
O3—Pb1—N1—C9	87.5 (4)	Pb1—N1—C9—C10	12.1 (5)
O3 ⁱⁱ —Pb1—N1—C9	33.2 (4)	C7—C8—C9—N1	2.3 (7)
O4 ⁱⁱ —Pb1—N1—C9	-0.6 (4)	C7—C8—C9—C10	-176.5 (4)

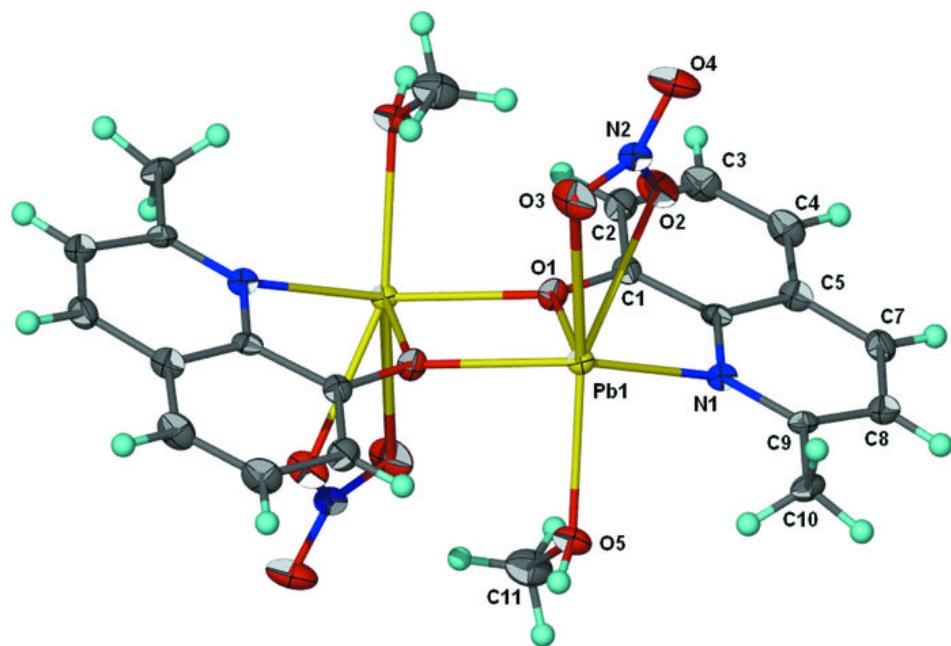
Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D\text{—H}\cdots A$	$D\text{—H}$	$H\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
O5—H5 ⁱⁱⁱ —O4 ⁱⁱⁱ	0.84 (1)	2.06 (2)	2.869 (5)	161 (6)

Symmetry codes: (iii) $x-1, y, z$.

Fig. 1



supplementary materials

Fig. 2

