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Bis(μ-2-methylquinolin-8-olato)-κ³N,O:O;κ³O:N,O-bis[(methanol-κO)-(nitrato-κ²O,O′)lead(II)]

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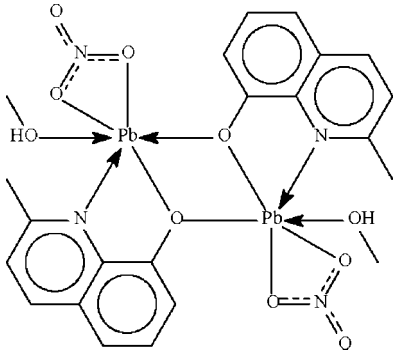
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.021; wR factor = 0.058; data-to-parameter ratio = 16.5.

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

$[Pb_2(C_{10}H_8NO)_2(NO_3)_2(CH_4O)_2]$
M_r = 918.83
Triclinic, $P\overline{1}$
a = 8.2579 (1) Å
b = 8.8052 (1) Å
c = 9.6765 (1) Å
α = 103.976 (1)°
β = 98.262 (1)°
γ = 108.190 (1)°
V = 630.07 (1) Å³
Z = 1
Mo $K\alpha$ radiation
μ = 13.41 mm⁻¹
T = 100 (2) K
0.20 × 0.15 × 0.05 mm

Data collection

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

Refinement

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

The authors 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: TK2363).

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supporting information

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

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

S1. 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.

S2. 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.

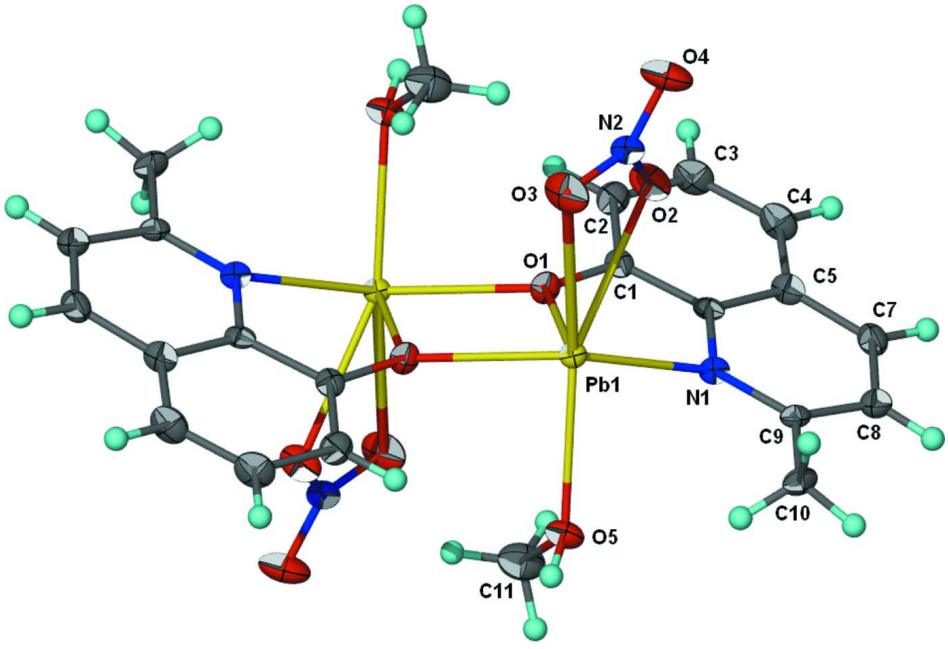

Figure 1

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*.

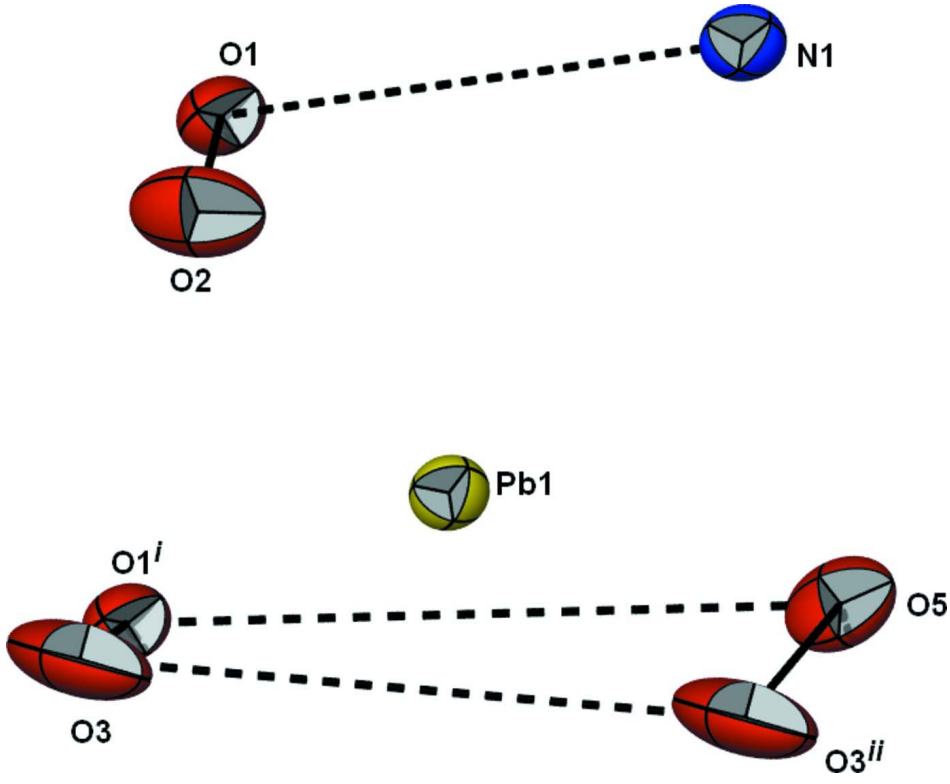

Figure 2

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-$\kappa O$)(nitrato-$\kappa^2O,O'$)lead(II)]

Crystal data

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.2579$ (1) Å
$b = 8.8052$ (1) Å
$c = 9.6765$ (1) Å
$\alpha = 103.976$ (1)°
$\beta = 98.262$ (1)°
$\gamma = 108.190$ (1)°
$V = 630.07$ (1) Å^3
$Z = 1$
$F(000) = 428$
$D_x = 2.422$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5067 reflections
$\theta = 2.2$–$28.3°$
$\mu = 13.41$ mm^{-1}
$T = 100$ K
Block, yellow
0.20 × 0.15 × 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)
$T_{min} = 0.175$, $T_{max} = 0.554$
5958 measured reflections
2872 independent reflections
2754 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.058$
$S = 1.08$
2872 reflections
174 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 1.3909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.41$ e Å^{-3}
$\Delta\rho_{min} = -2.12$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)

Geometric parameters (Å, º)

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)
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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O4^{iii}	0.84 (1)	2.06 (2)	2.869 (5)	161 (6)

Symmetry code: (iii) *x*−1, *y*, *z*.