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Bis(μ-quinoline-2-carboxylato)-κ³N,O:O;κ³O:N,O-bis[(acetato-κ²O,O′)(methanol-κO)lead(II)]

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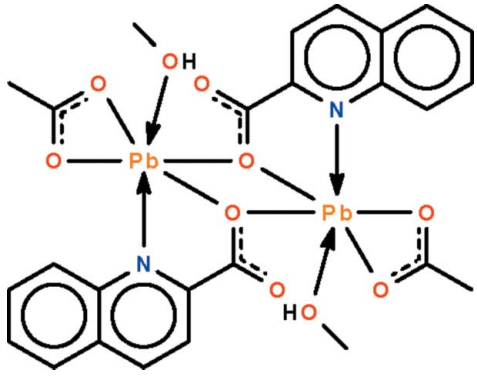
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.019; wR factor = 0.049; data-to-parameter ratio = 17.6.

The dinuclear title compound, $[Pb_2(C_{10}H_6NO_2)_2(CH_3COO)_2(CH_3OH)_2]$, lies across an inversion center. The methanol-coordinated Pb^{II} atom is chelated by the acetate anion as well as by the quinoline-2-carboxylate anion. One O atom of the quinoline-2-carboxylate anion bridges two symmetry-related Pb^{II} atoms, forming the dinuclear compound. Aside from the six atoms connected to the Pb^{II} atom by regular coordination bonds, the structure features a long Pb···O interaction [3.145 (3) Å] that gives rise to a distorted Ψ-square-antiprismatic geometry at the metal center. The H atom of the methanol is hydrogen bonded to an O atom of the acetate.

Related literature

For a related structure, see: Mohammadnezhad *et al.* (2010).



Experimental

Crystal data

$[Pb_2(C_{10}H_6NO_2)_2(C_2H_3O_2)_2(CH_4O)_2]$
M_r = 940.87
Monoclinic, $P2_1/c$
a = 7.3197 (3) Å
b = 8.3065 (4) Å
c = 23.3247 (10) Å
β = 90.397 (1)°
V = 1418.13 (11) Å³
Z = 2
Mo $K\alpha$ radiation
μ = 11.91 mm⁻¹
T = 100 K
0.25 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.155, T_{max} = 0.382
13298 measured reflections
3267 independent reflections
3055 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.049
S = 1.06
3267 reflections
186 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.65 e Å⁻³
$\Delta\rho_{min}$ = −1.43 e Å⁻³

Table 1
Selected bond lengths (Å).

Pb1—O1	2.377 (3)	Pb1—O4	2.761 (3)
Pb1—O1^{i}	2.490 (2)	Pb1—O5	2.696 (3)
Pb1—O2^{ii}	3.145 (3)	Pb1—N1	2.643 (3)
Pb1—O3	2.382 (3)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O3^{i}	0.84 (4)	1.89 (4)	2.685 (4)	158 (5)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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, 2010).

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: XU2798).

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supporting information

Acta Cryst. (2010). E**66**, m963 [https://doi.org/10.1107/S1600536810027777]

Bis(*μ*-quinoline-2-carboxylato)-$\kappa^3N,O:O;\kappa^3O:N,O$-bis[(acetato-$\kappa^2O,O'$)(methanol-$\kappa O$)lead(II)]

Gholamhossein Mohammadnezhad, Ali Reza Ghanbarpour, Mostafa M. Amini and Seik Weng Ng

S1. Comment

The lead(II) atom in $Pb(C_{10}H_6NO_2)_2$ is *N*,*O*-chelated by the carboxylate anions in a Ψ-trigonal bipyramidal environment; four atoms are connected to the lead atom by regular coordination bonds (Mohammadnezhad *et al.*, 2010). This compound was synthesized by the reaction of lead acetate and quinoline-2-carboxylic acid in the presence of potassium nitrite. With potassium nitrate in place of potassium nitrite, the synthesized yielded instead dinuclear $[Pb(CH_4O)_2(C_2H_3O_2)_2(C_{10}H_6NO_2)_2]_2$ (Scheme I, Fig. 1) in which only one of the acetate groups is replaced by the quinoline-2-carboxylate group. The methanol-coordinated lead(II) atom is chelated by the acetate anion as well as by the quinoline-2-carboxylate anion; the oxygen atom involved in chelation is also datively coordinated to the symmetry-related lead atom. When a long Pb···O2^{ii} (*ii* = *x* - 1, *y*, *z*) interaction of 3.145 (3) Å is considered to be a bond, the geometry is a Ψ-antiprism (Fig. 2).

S2. Experimental

Lead(II) acetate (1 mmol, 0.38 g), quinoline-2-carboxylic acid (1 mmol, 0.17 g) and potassium nitrate (1 mmol, 0.10 g) were loaded into one arm of a convection tube and both of the arms were filled slowly by methanol. The chemical-bearing arm was immersed in an oil bath kept at 333 K. Crystals were formed on the inside surface of the arm kept at ambient temperature after a weekfew days.

S3. Refinement

Hydrogen atoms were placed in calculated positions (C–H 0.95–0.98 Å) and included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5U_{eq}(C).

The hydroxy H-atom was located in a difference Fourier map, and was refined with a distance restraint of O–H 0.84±0.01 Å; its temperature factor was refined.

The final difference Fourier map had a peak/hole in the vicinity of Pb1.

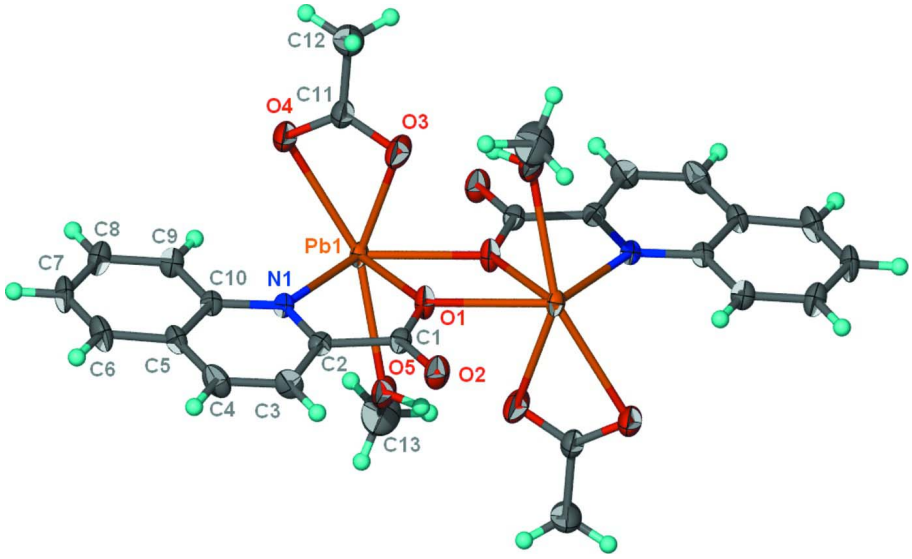

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[Pb(CH_4O)_2(C_2H_3O_2)_2(C_{10}H_6NO_2)_2]_2$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The dinuclear molecule lies on a center-of-inversion.

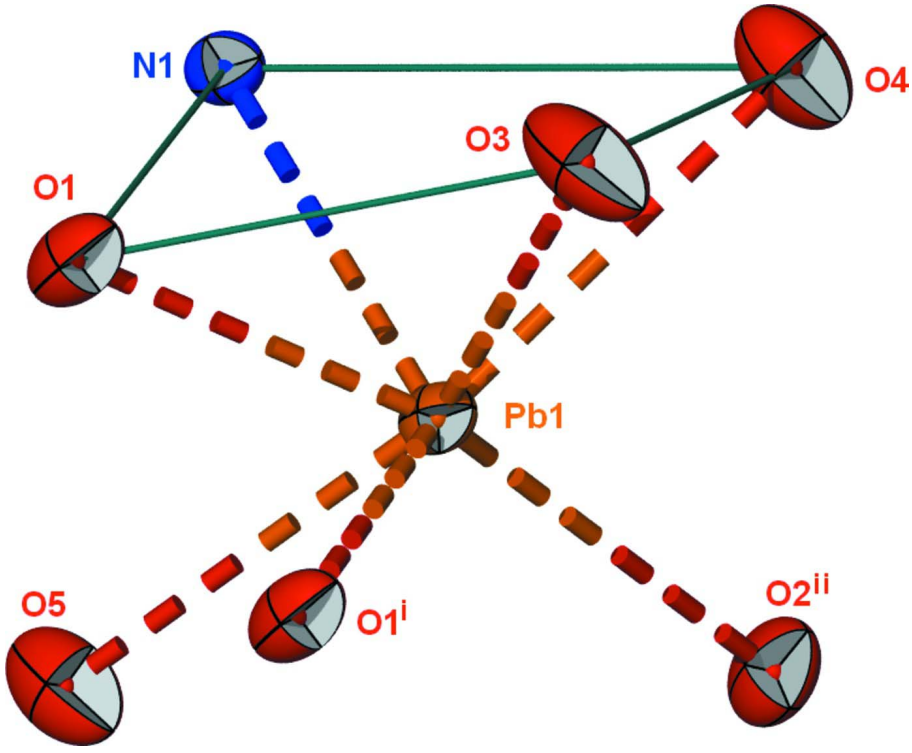

Figure 2

Ψ-Square antiprismatic geometry of lead. Symmetry codes are given in Table 1.

Bis(μ-quinoline-2-carboxylato)-$\kappa^3N,O:O;\kappa^3O:N,O$-bis[(acetato-$\kappa^2O,O'$)(methanol-$\kappa O$)lead(II)]

Crystal data

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$M_r = 940.87$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.3197$ (3) Å
$b = 8.3065$ (4) Å
$c = 23.3247$ (10) Å
$\beta = 90.397$ (1)°
$V = 1418.13$ (11) Å^3
$Z = 2$
$F(000) = 880$
$D_x = 2.203$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8885 reflections
$\theta = 2.6$–$28.3°$
$\mu = 11.91$ mm^{-1}
$T = 100$ K
Block, colorless
0.25 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.155$, $T_{max} = 0.382$
13298 measured reflections
3267 independent reflections
3055 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.8°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -30 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.049$
$S = 1.06$
3267 reflections
186 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
$w = 1/[\sigma^2(F_o^2) + (0.0252P)^2 + 2.8416P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.65$ e Å^{-3}
$\Delta\rho_{min} = -1.43$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.271223 (16)	0.534286 (15)	0.449130 (5)	0.01353 (5)
O1	0.5946 (3)	0.5505 (3)	0.45723 (11)	0.0199 (5)
O2	0.8654 (3)	0.6518 (3)	0.43362 (11)	0.0240 (6)
O3	0.3648 (4)	0.2628 (4)	0.43453 (12)	0.0313 (7)
O4	0.1736 (4)	0.3174 (3)	0.36464 (12)	0.0294 (6)
O5	0.3561 (4)	0.8211 (4)	0.49639 (12)	0.0284 (6)
H5	0.454 (4)	0.818 (7)	0.515 (2)	0.043*
N1	0.4435 (4)	0.6512 (3)	0.35907 (12)	0.0142 (5)
C1	0.7022 (4)	0.6278 (4)	0.42410 (14)	0.0158 (7)
C2	0.6171 (4)	0.6881 (4)	0.36843 (14)	0.0154 (6)
C3	0.7256 (5)	0.7731 (5)	0.32939 (16)	0.0212 (7)
H3	0.8477	0.8020	0.3389	0.025*

C4	0.6514 (5)	0.8138 (5)	0.27705 (17)	0.0247 (8)
H4	0.7230	0.8689	0.2495	0.030*
C5	0.4697 (5)	0.7735 (4)	0.26467 (15)	0.0185 (7)
C6	0.3834 (5)	0.8108 (5)	0.21153 (16)	0.0252 (8)
H6	0.4512	0.8628	0.1823	0.030*
C7	0.2051 (6)	0.7731 (5)	0.20205 (15)	0.0251 (8)
H7	0.1489	0.8001	0.1665	0.030*
C8	0.1033 (5)	0.6945 (5)	0.24449 (15)	0.0238 (8)
H8	−0.0207	0.6672	0.2371	0.029*
C9	0.1807 (5)	0.6563 (5)	0.29670 (15)	0.0200 (7)
H9	0.1099	0.6046	0.3253	0.024*
C10	0.3661 (5)	0.6944 (4)	0.30758 (14)	0.0152 (6)
C11	0.2681 (5)	0.2208 (5)	0.39130 (15)	0.0213 (7)
C12	0.2797 (8)	0.0481 (5)	0.3721 (2)	0.0366 (11)
H12A	0.1787	0.0246	0.3456	0.055*
H12B	0.2717	−0.0229	0.4056	0.055*
H12C	0.3962	0.0302	0.3527	0.055*
C13	0.2189 (7)	0.8980 (7)	0.5289 (2)	0.0478 (13)
H13A	0.0984	0.8608	0.5160	0.072*
H13B	0.2276	1.0148	0.5237	0.072*
H13C	0.2356	0.8717	0.5696	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.00951 (7)	0.01915 (8)	0.01191 (7)	0.00240 (5)	−0.00103 (4)	0.00037 (5)
O1	0.0138 (12)	0.0310 (15)	0.0148 (12)	0.0017 (10)	−0.0010 (9)	0.0060 (10)
O2	0.0141 (12)	0.0336 (15)	0.0244 (13)	−0.0003 (11)	−0.0035 (10)	0.0041 (11)
O3	0.0334 (16)	0.0291 (15)	0.0313 (15)	0.0141 (13)	−0.0169 (12)	−0.0117 (12)
O4	0.0368 (16)	0.0248 (14)	0.0264 (14)	0.0051 (13)	−0.0145 (12)	−0.0018 (12)
O5	0.0276 (15)	0.0275 (14)	0.0300 (15)	0.0107 (12)	−0.0137 (12)	−0.0051 (12)
N1	0.0136 (13)	0.0167 (14)	0.0123 (13)	0.0012 (11)	−0.0002 (10)	0.0004 (11)
C1	0.0140 (15)	0.0187 (17)	0.0147 (15)	0.0024 (13)	−0.0004 (12)	−0.0010 (13)
C2	0.0140 (15)	0.0158 (16)	0.0164 (16)	0.0025 (13)	0.0001 (12)	−0.0024 (13)
C3	0.0126 (16)	0.0240 (18)	0.0270 (19)	−0.0014 (14)	0.0025 (14)	0.0049 (15)
C4	0.0204 (18)	0.0270 (19)	0.0269 (19)	0.0012 (16)	0.0050 (15)	0.0112 (16)
C5	0.0208 (17)	0.0197 (17)	0.0151 (16)	0.0040 (14)	0.0011 (13)	0.0045 (13)
C6	0.029 (2)	0.030 (2)	0.0169 (17)	0.0070 (17)	0.0022 (15)	0.0094 (15)
C7	0.028 (2)	0.034 (2)	0.0130 (17)	0.0087 (17)	−0.0044 (14)	0.0041 (15)
C8	0.0205 (18)	0.033 (2)	0.0176 (17)	0.0043 (16)	−0.0045 (14)	−0.0012 (15)
C9	0.0181 (17)	0.0249 (18)	0.0171 (16)	0.0017 (14)	−0.0004 (13)	0.0005 (14)
C10	0.0163 (16)	0.0159 (16)	0.0135 (15)	0.0049 (13)	0.0007 (12)	−0.0016 (12)
C11	0.0220 (18)	0.0237 (19)	0.0182 (17)	0.0009 (15)	−0.0047 (14)	0.0001 (14)
C12	0.055 (3)	0.023 (2)	0.032 (2)	0.0088 (19)	−0.012 (2)	−0.0055 (17)
C13	0.047 (3)	0.047 (3)	0.049 (3)	0.022 (3)	−0.006 (2)	−0.013 (3)

Geometric parameters (Å, º)

Pb1—O1	2.377 (3)	C4—C5	1.400 (5)
Pb1—O1^{i}	2.490 (2)	C4—H4	0.9500
Pb1—O2^{ii}	3.145 (3)	C5—C10	1.421 (5)
Pb1—O3	2.382 (3)	C5—C6	1.422 (5)
Pb1—O4	2.761 (3)	C6—C7	1.359 (6)
Pb1—O5	2.696 (3)	C6—H6	0.9500
Pb1—N1	2.643 (3)	C7—C8	1.404 (5)
O1—C1	1.280 (4)	C7—H7	0.9500
O2—C1	1.230 (4)	C8—C9	1.376 (5)
O3—C11	1.276 (4)	C8—H8	0.9500
O4—C11	1.226 (4)	C9—C10	1.415 (5)
O5—C13	1.416 (6)	C9—H9	0.9500
O5—H5	0.84 (4)	C11—C12	1.505 (5)
N1—C2	1.324 (4)	C12—H12A	0.9800
N1—C10	1.372 (4)	C12—H12B	0.9800
C1—C2	1.521 (5)	C12—H12C	0.9800
C2—C3	1.403 (5)	C13—H13A	0.9800
C3—C4	1.375 (5)	C13—H13B	0.9800
C3—H3	0.9500	C13—H13C	0.9800
O1—Pb1—O3	77.17 (10)	C4—C3—H3	120.7
O1—Pb1—O1^{i}	63.88 (10)	C2—C3—H3	120.7
O3—Pb1—O1^{i}	75.27 (9)	C3—C4—C5	119.6 (3)
O1—Pb1—N1	64.02 (8)	C3—C4—H4	120.2
O3—Pb1—N1	95.46 (10)	C5—C4—H4	120.2
O1^{i}—Pb1—N1	127.85 (8)	C4—C5—C10	118.4 (3)
O1—Pb1—O5	71.99 (9)	C4—C5—C6	122.9 (3)
O3—Pb1—O5	146.04 (8)	C10—C5—C6	118.6 (3)
O1^{i}—Pb1—O5	78.63 (8)	C7—C6—C5	120.8 (4)
N1—Pb1—O5	83.70 (9)	C7—C6—H6	119.6
O1—Pb1—O4	110.27 (9)	C5—C6—H6	119.6
O3—Pb1—O4	49.71 (8)	C6—C7—C8	120.4 (3)
O1^{i}—Pb1—O4	122.70 (8)	C6—C7—H7	119.8
N1—Pb1—O4	78.17 (9)	C8—C7—H7	119.8
O5—Pb1—O4	157.89 (8)	C9—C8—C7	120.9 (4)
O1—Pb1—O2^{i}	105.53 (7)	C9—C8—H8	119.5
O3—Pb1—O2^{i}	76.37 (9)	C7—C8—H8	119.5
O1^{i}—Pb1—O2^{i}	42.37 (7)	C8—C9—C10	119.7 (3)
N1—Pb1—O2^{i}	168.31 (8)	C8—C9—H9	120.2
O5—Pb1—O2^{i}	98.35 (8)	C10—C9—H9	120.2
O4—Pb1—O2^{i}	101.97 (8)	N1—C10—C9	119.2 (3)
C1—O1—Pb1	126.7 (2)	N1—C10—C5	121.2 (3)
C1—O1—Pb1^{i}	115.5 (2)	C9—C10—C5	119.5 (3)
Pb1—O1—Pb1^{i}	116.12 (10)	O4—C11—O3	122.0 (4)
C11—O3—Pb1	102.3 (2)	O4—C11—C12	120.4 (3)
C11—O4—Pb1	85.6 (2)	O3—C11—C12	117.6 (3)

C13—O5—Pb1	117.1 (3)	C11—C12—H12A	109.5
C13—O5—H5	110 (4)	C11—C12—H12B	109.5
Pb1—O5—H5	112 (4)	H12A—C12—H12B	109.5
C2—N1—C10	118.3 (3)	C11—C12—H12C	109.5
C2—N1—Pb1	114.6 (2)	H12A—C12—H12C	109.5
C10—N1—Pb1	126.5 (2)	H12B—C12—H12C	109.5
O2—C1—O1	125.0 (3)	O5—C13—H13A	109.5
O2—C1—C2	119.5 (3)	O5—C13—H13B	109.5
O1—C1—C2	115.5 (3)	H13A—C13—H13B	109.5
N1—C2—C3	123.8 (3)	O5—C13—H13C	109.5
N1—C2—C1	116.9 (3)	H13A—C13—H13C	109.5
C3—C2—C1	119.3 (3)	H13B—C13—H13C	109.5
C4—C3—C2	118.6 (3)		

O3—Pb1—O1—C1	116.0 (3)	O5—Pb1—N1—C10	−110.9 (3)
O1^{i}—Pb1—O1—C1	−164.3 (3)	O4—Pb1—N1—C10	56.4 (3)
N1—Pb1—O1—C1	13.3 (3)	O2^{i}—Pb1—N1—C10	148.3 (3)
O5—Pb1—O1—C1	−78.4 (3)	Pb1—O1—C1—O2	169.7 (3)
O4—Pb1—O1—C1	78.3 (3)	Pb1^{i}—O1—C1—O2	5.3 (4)
O2^{i}—Pb1—O1—C1	−172.3 (3)	Pb1—O1—C1—C2	−12.1 (4)
O3—Pb1—O1—Pb1^{i}	−79.70 (12)	Pb1^{i}—O1—C1—C2	−176.6 (2)
O1^{i}—Pb1—O1—Pb1^{i}	0.0	C10—N1—C2—C3	2.0 (5)
N1—Pb1—O1—Pb1^{i}	177.62 (15)	Pb1—N1—C2—C3	−170.2 (3)
O5—Pb1—O1—Pb1^{i}	85.90 (12)	C10—N1—C2—C1	−175.7 (3)
O4—Pb1—O1—Pb1^{i}	−117.41 (11)	Pb1—N1—C2—C1	12.1 (4)
O2^{i}—Pb1—O1—Pb1^{i}	−8.01 (13)	O2—C1—C2—N1	176.4 (3)
O1—Pb1—O3—C11	−134.9 (3)	O1—C1—C2—N1	−1.8 (5)
O1^{i}—Pb1—O3—C11	159.2 (3)	O2—C1—C2—C3	−1.3 (5)
N1—Pb1—O3—C11	−73.1 (3)	O1—C1—C2—C3	−179.6 (3)
O5—Pb1—O3—C11	−159.9 (2)	N1—C2—C3—C4	−3.5 (6)
O4—Pb1—O3—C11	−3.6 (2)	C1—C2—C3—C4	174.1 (3)
O2^{i}—Pb1—O3—C11	115.4 (3)	C2—C3—C4—C5	1.7 (6)
O1—Pb1—O4—C11	55.1 (2)	C3—C4—C5—C10	1.4 (6)
O3—Pb1—O4—C11	3.7 (2)	C3—C4—C5—C6	−179.8 (4)
O1^{i}—Pb1—O4—C11	−16.2 (3)	C4—C5—C6—C7	−178.2 (4)
N1—Pb1—O4—C11	111.5 (2)	C10—C5—C6—C7	0.6 (6)
O5—Pb1—O4—C11	147.1 (3)	C5—C6—C7—C8	−0.8 (6)
O2^{i}—Pb1—O4—C11	−56.6 (2)	C6—C7—C8—C9	1.0 (6)
O1—Pb1—O5—C13	−157.6 (3)	C7—C8—C9—C10	−1.0 (6)
O3—Pb1—O5—C13	−131.9 (3)	C2—N1—C10—C9	−180.0 (3)
O1^{i}—Pb1—O5—C13	−91.6 (3)	Pb1—N1—C10—C9	−8.8 (4)
N1—Pb1—O5—C13	137.7 (3)	C2—N1—C10—C5	1.4 (5)
O4—Pb1—O5—C13	102.7 (4)	Pb1—N1—C10—C5	172.5 (2)
O2^{i}—Pb1—O5—C13	−53.9 (3)	C8—C9—C10—N1	−177.9 (3)
O1—Pb1—N1—C2	−12.5 (2)	C8—C9—C10—C5	0.8 (5)
O3—Pb1—N1—C2	−85.3 (2)	C4—C5—C10—N1	−3.0 (5)
O1^{i}—Pb1—N1—C2	−9.8 (3)	C6—C5—C10—N1	178.1 (3)
O5—Pb1—N1—C2	60.5 (2)	C4—C5—C10—C9	178.3 (3)

O4—Pb1—N1—C2	−132.2 (2)	C6—C5—C10—C9	−0.6 (5)
O2^{i}—Pb1—N1—C2	−40.3 (5)	Pb1—O4—C11—O3	−6.2 (4)
O1—Pb1—N1—C10	176.1 (3)	Pb1—O4—C11—C12	176.4 (4)
O3—Pb1—N1—C10	103.2 (3)	Pb1—O3—C11—O4	7.4 (5)
O1^{i}—Pb1—N1—C10	178.8 (2)	Pb1—O3—C11—C12	−175.2 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O3^{i}	0.84 (4)	1.89 (4)	2.685 (4)	158 (5)

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