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Poly[diaquabis(μ_2-5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-$\kappa^3N^3,O^4:O^5$)-lead(II)]

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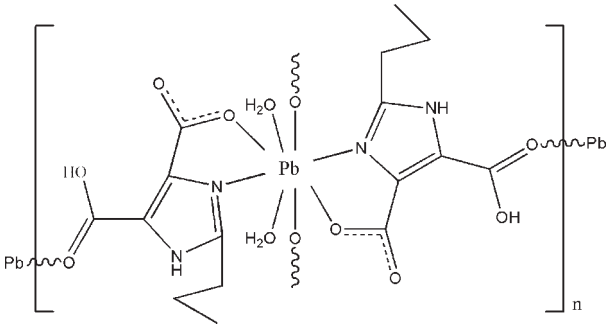
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.019; wR factor = 0.039; data-to-parameter ratio = 12.2.

In the title complex, $[Pb(C_8H_9N_2O_4)_2(H_2O)_2]_n$, the eight-coordinate Pb^{II} atom lies on a twofold rotation axis and adopts a slightly distorted square-antiprismatic N_2O_6 coordination geometry. The ligand donor atoms are the tertiary N atoms of the imidazole rings and the carboxylate O atoms of two chelating 5-carboxy-2-propyl-1*H*-imidazole-4-carboxylate ligands, the carboxy O atoms of two additional imidazole ligands and two water O atoms. The carboxy O and the *N*,*O*-chelate systems also link adjacent Pb^{II} atoms, forming a two-dimensional layer structure, with four individual Pb^{II} atoms located at the corners of a square. These layers are further interconnected by an extensive array of O—H···O and N—H···O hydrogen bonds into a three-dimensional network.

Related literature

For the properties and uses of imidazoledicarboxylate complexes, see: Cao *et al.* (2002); Rajendiran *et al.* (2003).



Experimental

Crystal data

$[Pb(C_8H_9N_2O_4)_2(H_2O)_2]$
M_r = 637.57
Monoclinic, $C2/c$
a = 13.1201 (15) Å
b = 13.2929 (16) Å
c = 11.5910 (13) Å
β = 98.531 (2)°
V = 1999.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.50 mm^{-1}
T = 293 K
0.45 × 0.17 × 0.13 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.114, T_{max} = 0.404
4884 measured reflections
1751 independent reflections
1640 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.039
S = 1.02
1751 reflections
143 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5D···O4^{i}	0.85	2.58	3.182 (4)	129
O5—H5D···O3^{i}	0.85	2.16	3.004 (3)	176
O5—H5C···O1^{ii}	0.85	2.19	3.035 (3)	176
O3—H3···O2	0.82	1.64	2.459 (3)	178
N2—H2···O1^{iii}	0.86	2.05	2.909 (4)	172

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x, -y+1, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2771).

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Rajendiran, T. M., Kirk, M. L., Setyawati, I. A., Caudle, M. T., Kampf, J. W. & Pecoraro, V. L. (2003). *Chem. Commun.* **7**, 824–825.
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supporting information

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Poly[diaquabis(μ_2-5-carboxy-2-propyl-1*H*-imidazole-4-carboxylato-$\kappa^3N^3,O^4{:}O^5$)lead(II)]

Xiang Chen and Hai-Cheng Liu

S1. Comment

It is well known that 4,5-imidazoledicarboxylic acid, a rigid N-heterocyclic dicarboxylic acid has great potential as a ligand in coordination complexes and for hydrogen bond formation. Imidazoledicarboxylate complexes have been found to exhibit useful properties, such as magnetism and porosity (Cao *et al.*, 2002; Rajendiran *et al.*, 2003). We have therefore reacted the 2-propyl-1*H*-imidazole-4,5 dicarboxylic acid ligand with $Pb(NO_3)_2$ under hydrothermal conditions to obtain a new Pb^{II} complex and its structure is reported here.

In the title complex, $[Pb(C_8H_9N_2O_4)_2(H_2O)_2]_n$, the eight coordinate Pb^{II} atom lies on a two-fold rotation axis and adopts a slightly distorted square-antiprismatic N_2O_6 coordination geometry. The ligand donor atoms are the N1 atoms of the imidazole rings and the carboxylate O1 atoms of two chelating 5-carboxy-2-propyl-1*H*-imidazole-4- carboxylato ligands, the O4 carboxy oxygen atoms of two additional imidazole ligands and two O5 water molecules. The O1 and N1 atoms of the chelate systems also link adjacent Pb^{II} centres forming a two-dimensional layer structure, with four individual Pb^{II} atoms located at the corners of a square. These layers are further interconnected by an extensive array of O—H···O and N—H···O hydrogen bonds into a three-dimensional network.

S2. Experimental

A mixture of $Pb(NO_3)_2$ (0.5 mmol, 0.07 g) and 2-propyl-1*H*-imidazole-4,5-dicarboxylic acid(0.5 mmol, 0.99 g) in 15 ml of H_2O solution was sealed in an autoclave equipped with a Teflon liner (25 ml) and then heated at 373k for 3 days. Crystals of the title compound were obtained by slow evaporation of the solvent at room temperature.

S3. Refinement

Carbon and nitrogen bound H atoms were placed at calculated positions and were treated as riding on the parent C or N atoms with C—H = 0.93 Å, N—H = 0.86 Å, and with U_{iso}(H) = 1.2 U_{eq}(C, N). H atoms of the water molecule were located in a difference map and were allowed to ride on the parent atom, with U_{iso}(H)=1.2 U_{eq}.

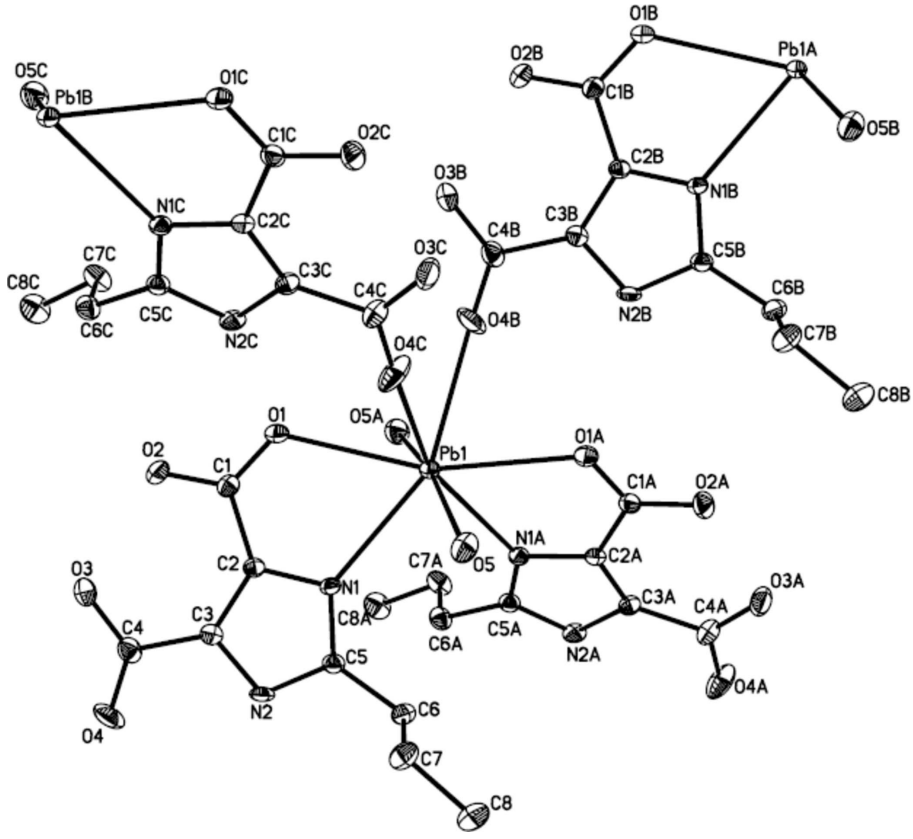

Figure 1

The structure of the title compound, showing the atomic numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids(H atoms are omitted for clarity). [Symmetry codes: (i) 1-x, y, 1.5-z; (ii) -0.5+x, 0.5+y, z; (iii) 0.5+x, 0.5+y, z; (iv) 0.5+x, -0.5+y, z; (v) 0.5-x, -0.5+y, 1.5-z]

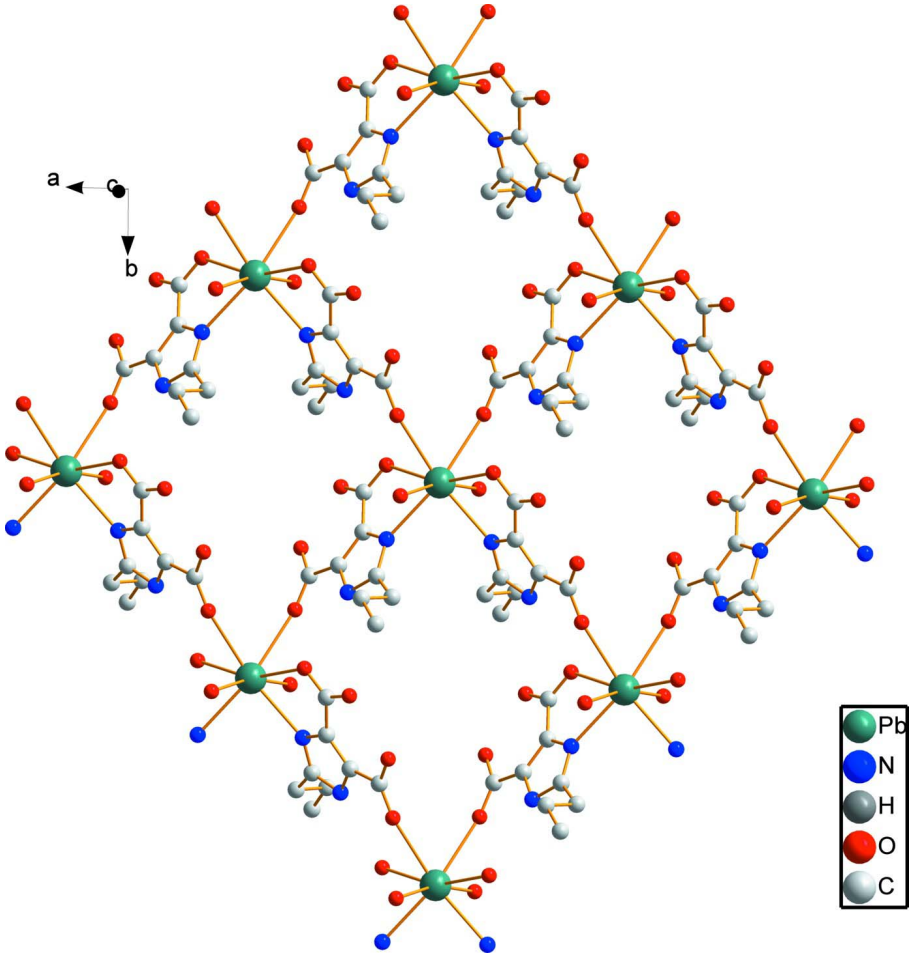

Figure 2

A two-dimensional layer constructed of Pb_4 squares

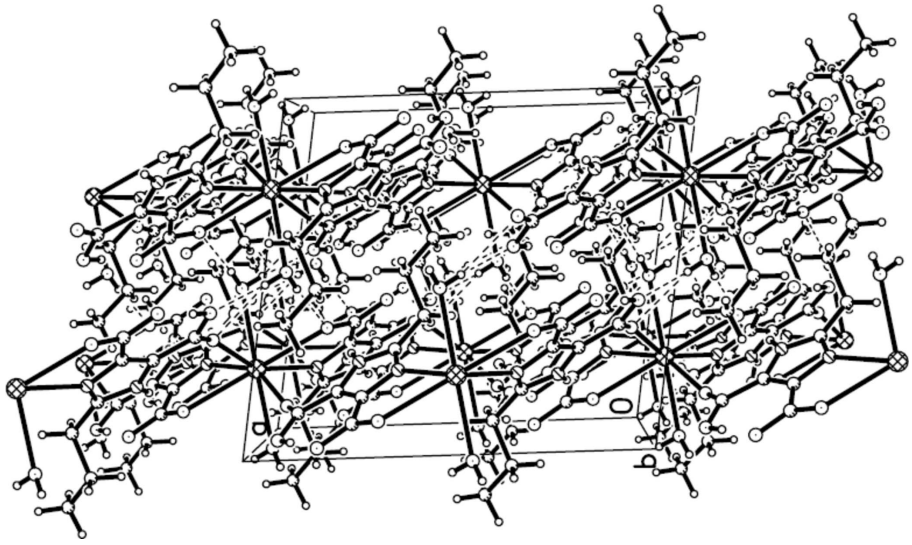

Figure 3

Crystal packing of the title compound

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Crystal data

$[Pb(C_8H_9N_2O_4)_2(H_2O)_2]$
$M_r = 637.57$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 13.1201\ (15)$ Å
$b = 13.2929\ (16)$ Å
$c = 11.5910\ (13)$ Å
$\beta = 98.531\ (2)°$
$V = 1999.2\ (4)$ Å^3
$Z = 4$
$F(000) = 1232$
$D_x = 2.118$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2051 reflections
$\theta = 2.5–23.9°$
$\mu = 8.50$ mm^{-1}
$T = 293$ K
Block, colorless
0.45 × 0.17 × 0.13 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.114$, $T_{max} = 0.404$
4884 measured reflections
1751 independent reflections
1640 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -13\rightarrow15$
$k = -15\rightarrow15$
$l = -10\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.039$
$S = 1.02$
1751 reflections
143 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
$w = 1/[\sigma^2(F_o^2) + 0.170P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.69$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00501 (14)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor wR and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) etc. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.5000	0.585420 (12)	0.7500	0.02187 (10)
N1	0.36101 (17)	0.72953 (19)	0.7416 (2)	0.0206 (6)
N2	0.26176 (19)	0.8636 (2)	0.7255 (2)	0.0245 (6)
H2	0.2380	0.9221	0.7040	0.029*
O1	0.33533 (16)	0.55121 (18)	0.8607 (2)	0.0288 (6)
O2	0.20043 (17)	0.61233 (19)	0.9317 (2)	0.0346 (6)
O3	0.09662 (17)	0.7669 (2)	0.9230 (2)	0.0400 (7)
H3	0.1304	0.7149	0.9245	0.060*
O4	0.0980 (2)	0.9168 (2)	0.8428 (3)	0.0521 (9)
O5	0.43915 (18)	0.6096 (2)	0.5232 (2)	0.0374 (6)
H5C	0.4120	0.5656	0.4750	0.045*
H5D	0.4830	0.6427	0.4915	0.045*
C1	0.2736 (2)	0.6209 (3)	0.8716 (3)	0.0240 (7)
C2	0.2842 (2)	0.7169 (2)	0.8095 (3)	0.0204 (7)
C3	0.2221 (2)	0.8003 (3)	0.7997 (3)	0.0228 (8)
C4	0.1323 (3)	0.8316 (3)	0.8572 (3)	0.0314 (9)
C5	0.3450 (2)	0.8188 (2)	0.6912 (3)	0.0230 (8)
C6	0.4039 (2)	0.8661 (3)	0.6046 (3)	0.0285 (8)
H6A	0.4688	0.8309	0.6062	0.034*
H6B	0.4193	0.9353	0.6274	0.034*
C7	0.3461 (3)	0.8646 (3)	0.4806 (3)	0.0355 (9)
H7A	0.3380	0.7954	0.4541	0.043*
H7B	0.2779	0.8928	0.4803	0.043*
C8	0.4016 (3)	0.9236 (3)	0.3967 (4)	0.0408 (10)
H8A	0.4129	0.9913	0.4246	0.061*
H8B	0.3604	0.9245	0.3210	0.061*
H8C	0.4667	0.8923	0.3914	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.02470 (12)	0.01520 (13)	0.02690 (14)	0.000	0.00777 (7)	0.000
N1	0.0251 (13)	0.0146 (16)	0.0229 (15)	0.0002 (11)	0.0059 (11)	0.0026 (13)
N2	0.0337 (15)	0.0126 (15)	0.0273 (16)	0.0049 (12)	0.0047 (12)	0.0059 (14)
O1	0.0343 (13)	0.0162 (13)	0.0375 (15)	0.0015 (10)	0.0110 (10)	0.0029 (12)
O2	0.0376 (14)	0.0306 (14)	0.0396 (16)	−0.0018 (11)	0.0191 (11)	0.0072 (13)
O3	0.0354 (13)	0.0414 (18)	0.0474 (17)	0.0086 (12)	0.0203 (11)	0.0008 (15)
O4	0.0632 (18)	0.046 (2)	0.051 (2)	0.0373 (14)	0.0236 (15)	0.0114 (15)
O5	0.0407 (14)	0.0395 (16)	0.0338 (16)	−0.0070 (12)	0.0115 (11)	−0.0025 (13)
C1	0.0266 (17)	0.0222 (19)	0.0236 (19)	−0.0044 (15)	0.0049 (14)	−0.0031 (17)
C2	0.0235 (15)	0.0163 (18)	0.0212 (18)	−0.0013 (13)	0.0026 (13)	−0.0018 (15)
C3	0.0264 (16)	0.022 (2)	0.0198 (18)	0.0019 (14)	0.0022 (13)	−0.0012 (16)
C4	0.0335 (19)	0.035 (2)	0.027 (2)	0.0074 (17)	0.0083 (15)	−0.0017 (19)
C5	0.0244 (16)	0.0178 (19)	0.0266 (19)	−0.0001 (14)	0.0030 (13)	0.0008 (16)
C6	0.0286 (18)	0.025 (2)	0.033 (2)	−0.0009 (15)	0.0050 (14)	0.0066 (18)

C7	0.0365 (19)	0.037 (2)	0.033 (2)	−0.0111 (17)	0.0026 (15)	0.003 (2)
C8	0.049 (2)	0.042 (3)	0.031 (2)	−0.0065 (18)	0.0058 (18)	0.0088 (19)

Geometric parameters (Å, º)

Pb1—N1	2.637 (2)	O4—C4	1.221 (4)
Pb1—N1^{i}	2.637 (2)	O4—Pb1^{iv}	2.725 (3)
Pb1—O5^{i}	2.651 (3)	O5—H5C	0.8500
Pb1—O5	2.652 (3)	O5—H5D	0.8500
Pb1—O1	2.710 (2)	C1—C2	1.481 (5)
Pb1—O1^{i}	2.710 (2)	C2—C3	1.370 (4)
Pb1—O4^{ii}	2.725 (3)	C3—C4	1.496 (5)
Pb1—O4^{iii}	2.725 (3)	C5—C6	1.494 (5)
N1—C5	1.326 (4)	C6—C7	1.522 (4)
N1—C2	1.377 (4)	C6—H6A	0.9700
N2—C5	1.354 (4)	C6—H6B	0.9700
N2—C3	1.361 (5)	C7—C8	1.517 (5)
N2—H2	0.8600	C7—H7A	0.9700
O1—C1	1.249 (4)	C7—H7B	0.9700
O2—C1	1.273 (4)	C8—H8A	0.9600
O3—C4	1.284 (4)	C8—H8B	0.9600
O3—H3	0.8200	C8—H8C	0.9600
N1—Pb1—N1^{i}	86.81 (11)	Pb1—O5—H5C	127.1
N1—Pb1—O5^{i}	93.06 (8)	Pb1—O5—H5D	111.6
N1^{i}—Pb1—O5^{i}	76.75 (8)	H5C—O5—H5D	108.5
N1—Pb1—O5	76.75 (8)	O1—C1—O2	122.8 (3)
N1^{i}—Pb1—O5	93.06 (8)	O1—C1—C2	118.7 (3)
O5^{i}—Pb1—O5	166.09 (11)	O2—C1—C2	118.4 (3)
N1—Pb1—O1	62.85 (7)	C3—C2—N1	109.3 (3)
N1^{i}—Pb1—O1	134.44 (7)	C3—C2—C1	129.8 (3)
O5^{i}—Pb1—O1	72.23 (7)	N1—C2—C1	120.8 (3)
O5—Pb1—O1	110.24 (7)	N2—C3—C2	106.0 (3)
N1—Pb1—O1^{i}	134.44 (7)	N2—C3—C4	120.6 (3)
N1^{i}—Pb1—O1^{i}	62.85 (7)	C2—C3—C4	133.3 (3)
O5^{i}—Pb1—O1^{i}	110.23 (7)	O4—C4—O3	122.8 (3)
O5—Pb1—O1^{i}	72.23 (7)	O4—C4—C3	119.8 (4)
O1—Pb1—O1^{i}	160.68 (10)	O3—C4—C3	117.3 (3)
N1—Pb1—O4^{ii}	153.57 (9)	N1—C5—N2	110.3 (3)
N1^{i}—Pb1—O4^{ii}	107.45 (9)	N1—C5—C6	127.6 (3)
O5^{i}—Pb1—O4^{ii}	69.71 (9)	N2—C5—C6	122.1 (3)
O5—Pb1—O4^{ii}	123.10 (9)	C5—C6—C7	113.2 (3)
O1—Pb1—O4^{ii}	92.16 (8)	C5—C6—H6A	108.9
O1^{i}—Pb1—O4^{ii}	71.71 (8)	C7—C6—H6A	108.9
N1—Pb1—O4^{iii}	107.46 (9)	C5—C6—H6B	108.9
N1^{i}—Pb1—O4^{iii}	153.56 (9)	C7—C6—H6B	108.9
O5^{i}—Pb1—O4^{iii}	123.10 (9)	H6A—C6—H6B	107.8
O5—Pb1—O4^{iii}	69.70 (9)	C8—C7—C6	112.3 (3)

O1—Pb1—O4^{iii}	71.72 (8)	C8—C7—H7A	109.1
O1^{i}—Pb1—O4^{iii}	92.16 (8)	C6—C7—H7A	109.1
O4^{ii}—Pb1—O4^{iii}	69.29 (14)	C8—C7—H7B	109.1
C5—N1—C2	106.2 (3)	C6—C7—H7B	109.1
C5—N1—Pb1	136.9 (2)	H7A—C7—H7B	107.9
C2—N1—Pb1	116.79 (19)	C7—C8—H8A	109.5
C5—N2—C3	108.3 (3)	C7—C8—H8B	109.5
C5—N2—H2	125.9	H8A—C8—H8B	109.5
C3—N2—H2	125.9	C7—C8—H8C	109.5
C1—O1—Pb1	119.8 (2)	H8A—C8—H8C	109.5
C4—O3—H3	109.5	H8B—C8—H8C	109.5
C4—O4—Pb1^{iv}	163.7 (3)		

N1^{i}—Pb1—N1—C5	39.1 (3)	Pb1—N1—C2—C1	−7.6 (3)
O5^{i}—Pb1—N1—C5	115.6 (3)	O1—C1—C2—C3	174.0 (3)
O5—Pb1—N1—C5	−54.8 (3)	O2—C1—C2—C3	−4.4 (5)
O1—Pb1—N1—C5	−176.2 (3)	O1—C1—C2—N1	−0.5 (4)
O1^{i}—Pb1—N1—C5	−6.9 (3)	O2—C1—C2—N1	−178.9 (3)
O4^{ii}—Pb1—N1—C5	163.3 (3)	C5—N2—C3—C2	0.4 (4)
O4^{iii}—Pb1—N1—C5	−118.2 (3)	C5—N2—C3—C4	176.8 (3)
N1^{i}—Pb1—N1—C2	−137.1 (2)	N1—C2—C3—N2	0.0 (3)
O5^{i}—Pb1—N1—C2	−60.6 (2)	C1—C2—C3—N2	−175.0 (3)
O5—Pb1—N1—C2	129.0 (2)	N1—C2—C3—C4	−175.8 (3)
O1—Pb1—N1—C2	7.63 (19)	C1—C2—C3—C4	9.3 (6)
O1^{i}—Pb1—N1—C2	176.89 (18)	Pb1^{iv}—O4—C4—O3	−108.0 (9)
O4^{ii}—Pb1—N1—C2	−12.9 (3)	Pb1^{iv}—O4—C4—C3	74.0 (10)
O4^{iii}—Pb1—N1—C2	65.6 (2)	N2—C3—C4—O4	−4.3 (5)
N1—Pb1—O1—C1	−8.6 (2)	C2—C3—C4—O4	171.0 (4)
N1^{i}—Pb1—O1—C1	45.2 (3)	N2—C3—C4—O3	177.7 (3)
O5^{i}—Pb1—O1—C1	94.6 (2)	C2—C3—C4—O3	−7.1 (6)
O5—Pb1—O1—C1	−70.9 (2)	C2—N1—C5—N2	0.7 (3)
O1^{i}—Pb1—O1—C1	−164.9 (2)	Pb1—N1—C5—N2	−175.8 (2)
O4^{ii}—Pb1—O1—C1	162.5 (2)	C2—N1—C5—C6	−176.9 (3)
O4^{iii}—Pb1—O1—C1	−130.2 (2)	Pb1—N1—C5—C6	6.7 (5)
Pb1—O1—C1—O2	−173.4 (2)	C3—N2—C5—N1	−0.7 (4)
Pb1—O1—C1—C2	8.3 (4)	C3—N2—C5—C6	177.0 (3)
C5—N1—C2—C3	−0.4 (3)	N1—C5—C6—C7	105.0 (4)
Pb1—N1—C2—C3	176.90 (19)	N2—C5—C6—C7	−72.3 (4)
C5—N1—C2—C1	175.1 (3)	C5—C6—C7—C8	173.3 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*D*···O4^{v}	0.85	2.58	3.182 (4)	129
O5—H5*D*···O3^{v}	0.85	2.16	3.004 (3)	176
O5—H5*C*···O1^{vi}	0.85	2.19	3.035 (3)	176

O3—H3···O2	0.82	1.64	2.459 (3)	178
N2—H2···O1^{vii}	0.86	2.05	2.909 (4)	172

Symmetry codes: (v) $x+1/2, -y+3/2, z-1/2$; (vi) $x, -y+1, z-1/2$; (vii) $-x+1/2, y+1/2, -z+3/2$.