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(2,9-Dimethyl-1,10-phenanthroline-κ^2N,N')bis(3-hydroxybenzoato-κ^2O,O')-lead(II)–2,9-dimethyl-1,10-phenanthroline–water (1/1/3)

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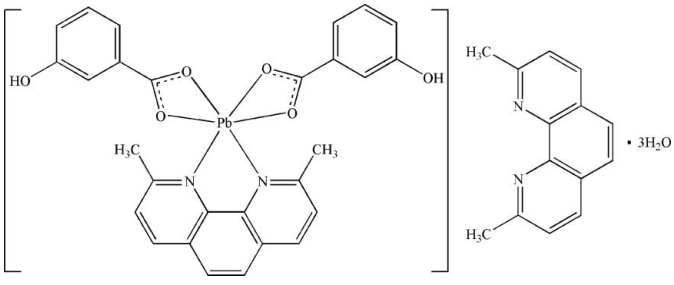
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.020; wR factor = 0.047; data-to-parameter ratio = 14.3.

In the title complex, $[Pb(C_{14}H_{12}N_2)(C_7H_5O_3)_2]\cdot C_{14}H_{12}N_2\cdot 3H_2O$, the Pb^{II} ion is coordinated by two N atoms from one 2,9-dimethyl-1,10-phenanthroline (dmphen) ligand and four O atoms from two 3-hydroxybenzoate anions in a distorted pseudo-square-pyramidal environment, considering the dmphen molecule as an apical ligand. Molecules are linked into a three-dimensional framework through O—H···O and O—H···N hydrogen bonds. The crystal structure is further stabilized by π–π interactions between the dmphen rings of neighboring molecules, with a distance between the ring planes of 3.493 Å.

Related literature

For related literature, see: Shimoni-Livny *et al.* (1998); Xuan & Zhao (2007).



Experimental

Crystal data

$[Pb(C_{14}H_{12}N_2)(C_7H_5O_3)_2]\cdot C_{14}H_{12}N_2\cdot 3H_2O$
M_r = 951.97
Monoclinic, $C2/c$
a = 31.662 (3) Å
b = 10.8291 (10) Å
c = 27.381 (4) Å
β = 123.530 (1)°
V = 7825.9 (15) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 4.37 mm^{-1}
T = 291 (2) K
0.41 × 0.35 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.217, T_{max} = 0.334
28443 measured reflections
7287 independent reflections
6143 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.047
S = 1.03
7287 reflections
511 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pb1—N1	2.493 (2)	Pb1—O2	2.637 (2)
Pb1—N2	2.477 (2)	Pb1—O4	2.569 (2)
Pb1—O1	2.639 (2)	Pb1—O5	2.6296 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O8^{i}	0.82	1.86	2.669 (4)	169
O6—H6···N4	0.82	2.15	2.916 (4)	155
O7—H1W···O1	0.83	1.88	2.702 (3)	172
O7—H2W···O6^{ii}	0.84	2.15	2.977 (3)	169
O8—H3W···O4^{iii}	0.85	1.94	2.784 (3)	180
O8—H4W···O9^{iv}	0.83	2.02	2.828 (4)	166

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: BH2147).

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0), *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shimoni-Livny, L., Glusker, J. P. & Bock, C. W. (1998). *Inorg. Chem.* **37**, 1853–1867.
Xuan, X.-P. & Zhao, P.-Z. (2007). *Acta Cryst.* E**63**, m2678.

supplementary materials

Acta Cryst. (2007). E**63**, m3179 [**doi:10.1107/S1600536807061065**]

(2,9-Dimethyl-1,10-phenanthroline-κ^2N,N')bis(3-hydroxybenzoato-κ^2O,O')lead(II)-2,9-dimethyl-1,10-phenanthroline-water (1/1/3)

P.-Z. Zhao and X.-P. Xuan

Comment

The coordination chemistry of lead(II) with N and O donor ligands has been investigated in the past decade and frequently discussed, as lead is an environmental pollutant with severe toxic effects (Shimoni-Livny *et al.*, 1998; Xuan & Zhao, 2007). The title complex, (I), was recently obtained from the reaction of lead acetate, sodium *m*-hydroxybenzoate and 2,9-dimethyl-1,10-phenanthroline (dmphen) in an ethanol/water mixture (see Experimental), and its crystal structure is reported here.

Each Pb^{II} ion is six-coordinated by two N atoms from a dmphen ligand, and four O atoms from two *m*-hydroxybenzoate ligands (Fig.1). The four Pb—O bond lengths are different, Pb1—O1 = 2.639 (2), Pb1—O2 = 2.637 (2), Pb1—O4 = 2.569 (2) and Pb1—O5 = 2.6296 (19) Å, respectively. The two Pb—N bond lengths are also different, Pb1—N1 = 2.493 (2) Å and Pb1—N2= 2.477 (2) Å. The Pb^{II} ion is located in the center of the basal coordinationplane, and PbO_4N_2 unit forms a distorted pseudo square-pyramidal environment, considering the dmphen molecule as an apical ligand.

In the crystal structure, molecules are linked into a three dimensional framework by intermolecular O—H···O and O—H···N hydrogen bonds (Fig. 2 and 3). Molecules are also linked into a one dimensional network by π–π interactions between the dmphen ring systems (Fig. 4). These intermolecular interactions occur between the rings within offset face-to-face packing. The distance of neighboring molecules parallel ring planes is 3.493 Å. A three dimensional architecture is thus formed through hydrogen bonds and π–π stacking interactions (Fig. 4).

Experimental

A mixture of *m*-hydroxybenzoic acid (0.070 g, 0.5 mmol) and NaOH (0.018 g, 0.5 mmol) in distilled water (10 ml) was stirred until dissolution, and then $Pb(CH_3COOH)_2{\cdot}3H_2O$ (0.190 g, 0.5 mmol) was added. A solution of 2,9-dimethyl-1,10-phenanthroline hemihydrate ($C_{14}H_{12}N_2{\cdot}0.5H_2O$, 0.109 g, 0.5 mmol) in ethanol (10 ml) was then added. The mixture was refluxed for 4 h. The hot solution was cooled to room temperature and then filtered. Bright colorless single crystals of (I) appeared over a period of five days by slow evaporation at room temperature.

Refinement

C-bonded H atoms were placed in calculated positions, with C—H = 0.93 Å (aromatic CH) or C—H = 0.96 Å (methyl CH_3), and refined in the riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic CH and $U_{iso}(H) = 1.5U_{eq}(C)$ otherwise. Hydroxyl and water H atoms were found in a difference map and their coordinates fixed after regularization of geometry. Isotropic displacement parameters were fixed to $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

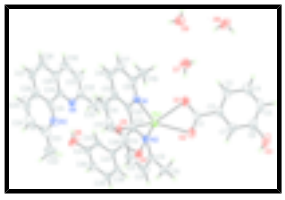

Fig. 1. The molecular structure of the title complex, (I), with atom labels and 30% probability displacement ellipsoids.

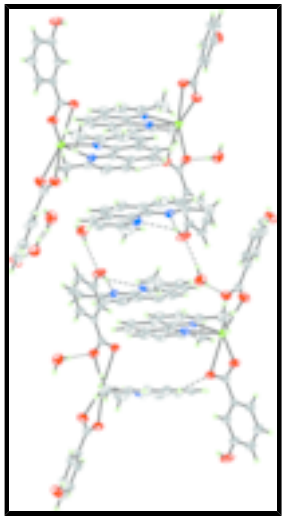

Fig. 2. The hydrogen-bonding motifs in the crystal structure of (I). Dashed lines indicate the hydrogen bonds.

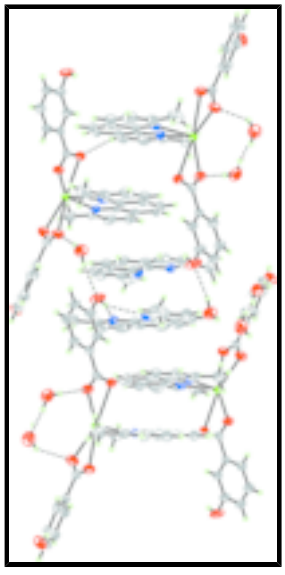

Fig. 3. The hydrogen-bonding motifs in the crystal structure of (I), including all molecules. Dashed lines indicate the hydrogen bonds.

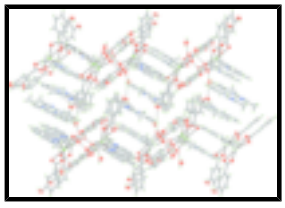

Fig. 4. The π–π stacking and hydrogen bonds in the crystal structure of (I).

(2,9-Dimethyl-1,10-phenanthroline-κ^2*N*,*N*$^{\prime}$)bis(3- hydroxybenzoato-κ^2*O*,*O*$^{\prime}$)lead(II)–2,9-dimethyl-1,10-phenanthroline–water (1/1/3)

Crystal data

$[Pb(C_{14}H_{12}N_2)(C_7H_5O_3)_2]\cdot C_{14}H_{12}N_2\cdot 3H_2O$	F_{000} = 3792
M_r = 951.97	D_x = 1.616 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 6283 reflections
a = 31.662 (3) Å	θ = 2.4–27.0º
b = 10.8291 (10) Å	μ = 4.37 mm^{-1}
c = 27.381 (4) Å	*T* = 291 (2) K
β = 123.530 (1)º	Block, colourless
V = 7825.9 (15) Å^3	0.41 × 0.35 × 0.25 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	7287 independent reflections
Radiation source: fine-focus sealed tube	6143 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 291(2) K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	*h* = −37→38
T_{min} = 0.217, T_{max} = 0.334	*k* = −13→13
28443 measured reflections	*l* = −32→33

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.020	H-atom parameters constrained
$wR(F^2)$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.0222P)^2 + 6.8596P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.003
7287 reflections	$\Delta\rho_{max}$ = 0.45 e Å^{-3}
511 parameters	$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.224379 (4)	0.879582 (10)	0.373487 (4)	0.04096 (4)
O1	0.29906 (8)	0.7989 (2)	0.47679 (9)	0.0708 (7)
O2	0.26753 (9)	0.9783 (2)	0.47864 (9)	0.0709 (6)
O3	0.38864 (13)	1.1174 (3)	0.68961 (11)	0.0961 (9)
H3	0.3825	1.1750	0.6672	0.144*
O4	0.14958 (7)	1.02393 (19)	0.30704 (9)	0.0565 (5)
O5	0.15723 (7)	0.87141 (19)	0.25972 (8)	0.0532 (5)
O6	−0.02455 (8)	0.8054 (3)	0.08300 (11)	0.0905 (9)
H6	−0.0049	0.7516	0.0864	0.136*
O7	0.36781 (9)	0.6228 (2)	0.49854 (11)	0.0779 (7)
H1W	0.3477	0.6811	0.4901	0.117*
H2W	0.3970	0.6429	0.5259	0.117*
O8	0.37422 (9)	0.3244 (3)	0.62932 (12)	0.0858 (8)
H3W	0.3670	0.3704	0.6488	0.129*
H4W	0.3535	0.3454	0.5950	0.129*
O9	0.18511 (11)	0.0677 (3)	0.48323 (13)	0.1003 (9)
H5W	0.1638	0.0550	0.4478	0.150*
H6W	0.2104	0.0227	0.4956	0.150*
N1	0.27666 (8)	0.7787 (2)	0.34240 (9)	0.0445 (5)
N2	0.27281 (7)	1.0306 (2)	0.35509 (9)	0.0386 (5)
N3	0.05922 (9)	0.7416 (3)	0.05994 (11)	0.0587 (7)
N4	0.02519 (8)	0.5671 (2)	0.10211 (10)	0.0507 (6)
C1	0.29728 (11)	0.8896 (3)	0.50406 (13)	0.0522 (7)
C2	0.33241 (11)	0.8923 (3)	0.56993 (12)	0.0506 (7)
C3	0.35607 (12)	0.7845 (3)	0.60090 (13)	0.0601 (8)
H3A	0.3504	0.7099	0.5813	0.072*
C4	0.38851 (13)	0.7905 (4)	0.66183 (15)	0.0739 (11)
H4	0.4040	0.7190	0.6832	0.089*
C5	0.39779 (14)	0.9014 (4)	0.69050 (15)	0.0765 (12)
H5	0.4190	0.9039	0.7312	0.092*
C6	0.37589 (13)	1.0092 (4)	0.65958 (15)	0.0689 (10)
C7	0.34198 (11)	1.0042 (3)	0.59884 (13)	0.0571 (8)
H7	0.3259	1.0756	0.5778	0.069*
C8	0.24701 (13)	1.2019 (3)	0.39162 (16)	0.0654 (9)
H8A	0.2132	1.2276	0.3619	0.098*
H8B	0.2658	1.2713	0.4158	0.098*
H8C	0.2456	1.1388	0.4153	0.098*
C9	0.27266 (10)	1.1519 (2)	0.36334 (13)	0.0479 (7)
C10	0.29607 (12)	1.2343 (3)	0.34563 (15)	0.0656 (9)
H10	0.2957	1.3185	0.3521	0.079*
C11	0.31891 (13)	1.1934 (4)	0.31954 (15)	0.0699 (10)
H11	0.3338	1.2490	0.3075	0.084*
C12	0.32017 (11)	1.0657 (3)	0.31043 (12)	0.0557 (8)
C13	0.34404 (13)	1.0145 (5)	0.28337 (15)	0.0770 (12)
H13	0.3592	1.0670	0.2705	0.092*

C14	0.34495 (13)	0.8918 (5)	0.27618 (15)	0.0797 (13)
H14	0.3603	0.8609	0.2579	0.096*
C15	0.32265 (11)	0.8086 (4)	0.29622 (13)	0.0618 (9)
C16	0.32383 (15)	0.6795 (5)	0.29173 (16)	0.0848 (13)
H16	0.3392	0.6447	0.2742	0.102*
C17	0.30295 (17)	0.6053 (4)	0.31249 (18)	0.0862 (13)
H17	0.3042	0.5201	0.3094	0.103*
C18	0.27932 (13)	0.6565 (3)	0.33871 (14)	0.0622 (9)
C19	0.25614 (17)	0.5778 (3)	0.36306 (17)	0.0871 (13)
H19A	0.2816	0.5561	0.4028	0.131*
H19B	0.2425	0.5041	0.3400	0.131*
H19C	0.2295	0.6229	0.3619	0.131*
C20	0.29834 (10)	0.8550 (3)	0.32251 (11)	0.0440 (7)
C21	0.29668 (9)	0.9861 (3)	0.32931 (11)	0.0413 (6)
C22	0.06912 (18)	0.9596 (4)	0.0499 (2)	0.1226 (19)
H22A	0.0502	0.9636	0.0678	0.184*
H22B	0.0511	1.0029	0.0132	0.184*
H22C	0.1017	0.9970	0.0754	0.184*
C23	0.07584 (13)	0.8271 (4)	0.03950 (17)	0.0797 (11)
C24	0.10013 (16)	0.7940 (6)	0.0111 (2)	0.1032 (17)
H24	0.1111	0.8552	−0.0032	0.124*
C25	0.10757 (16)	0.6739 (7)	0.00463 (19)	0.1022 (17)
H25	0.1236	0.6527	−0.0142	0.123*
C26	0.09143 (12)	0.5823 (4)	0.02599 (14)	0.0699 (10)
C27	0.09865 (15)	0.4532 (6)	0.02211 (17)	0.0940 (16)
H27	0.1147	0.4275	0.0038	0.113*
C28	0.08299 (16)	0.3691 (5)	0.04407 (19)	0.0915 (15)
H28	0.0888	0.2860	0.0414	0.110*
C29	0.05769 (12)	0.4029 (3)	0.07143 (15)	0.0629 (9)
C30	0.04061 (15)	0.3174 (4)	0.09526 (19)	0.0881 (14)
H30	0.0453	0.2334	0.0930	0.106*
C31	0.01745 (16)	0.3562 (4)	0.1214 (2)	0.0929 (15)
H31	0.0065	0.2993	0.1376	0.112*
C32	0.00986 (12)	0.4827 (4)	0.12410 (15)	0.0694 (10)
C33	−0.01798 (15)	0.5277 (5)	0.15087 (18)	0.1048 (16)
H33A	−0.0053	0.6074	0.1682	0.157*
H33B	−0.0129	0.4706	0.1805	0.157*
H33C	−0.0535	0.5338	0.1210	0.157*
C34	0.04918 (10)	0.5289 (3)	0.07621 (12)	0.0483 (7)
C35	0.06661 (10)	0.6209 (3)	0.05336 (12)	0.0523 (7)
C36	0.13066 (10)	0.9517 (2)	0.26357 (11)	0.0392 (6)
C37	0.07541 (10)	0.9626 (2)	0.21688 (11)	0.0390 (6)
C38	0.04679 (11)	1.0572 (3)	0.21906 (13)	0.0519 (7)
H38	0.0622	1.1156	0.2489	0.062*
C39	−0.00465 (12)	1.0644 (3)	0.17683 (14)	0.0596 (8)
H39	−0.0238	1.1273	0.1786	0.071*
C40	−0.02758 (11)	0.9795 (3)	0.13249 (14)	0.0589 (8)
H40	−0.0622	0.9849	0.1044	0.071*
C41	0.00033 (11)	0.8858 (3)	0.12926 (14)	0.0573 (8)

C42	0.05214 (10)	0.8775 (3)	0.17154 (12)	0.0497 (7)
H42	0.0711	0.8148	0.1693	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.03378 (6)	0.05033 (7)	0.03576 (6)	−0.00360 (5)	0.01731 (5)	0.00157 (5)
O1	0.0534 (13)	0.0931 (18)	0.0474 (13)	0.0185 (12)	0.0163 (11)	−0.0101 (12)
O2	0.0758 (16)	0.0763 (16)	0.0482 (13)	0.0180 (14)	0.0264 (12)	0.0043 (12)
O3	0.105 (2)	0.125 (3)	0.0602 (16)	−0.017 (2)	0.0464 (17)	−0.0352 (16)
O4	0.0442 (11)	0.0573 (13)	0.0520 (12)	−0.0007 (10)	0.0165 (10)	−0.0189 (10)
O5	0.0401 (10)	0.0676 (14)	0.0426 (11)	0.0102 (10)	0.0170 (9)	−0.0114 (10)
O6	0.0435 (13)	0.101 (2)	0.0773 (17)	0.0139 (13)	0.0020 (12)	−0.0476 (15)
O7	0.0653 (15)	0.0707 (16)	0.0834 (17)	0.0128 (12)	0.0319 (14)	0.0016 (13)
O8	0.0697 (16)	0.0994 (19)	0.0879 (19)	−0.0021 (15)	0.0433 (15)	−0.0342 (16)
O9	0.097 (2)	0.108 (2)	0.105 (2)	0.0183 (18)	0.0613 (19)	0.0024 (18)
N1	0.0465 (13)	0.0411 (13)	0.0351 (12)	0.0026 (11)	0.0157 (11)	−0.0009 (10)
N2	0.0328 (11)	0.0392 (12)	0.0395 (12)	−0.0002 (9)	0.0173 (10)	0.0029 (10)
N3	0.0425 (14)	0.0665 (18)	0.0574 (16)	−0.0041 (13)	0.0214 (13)	0.0051 (14)
N4	0.0359 (13)	0.0639 (16)	0.0456 (14)	−0.0023 (12)	0.0182 (12)	−0.0017 (12)
C1	0.0418 (16)	0.071 (2)	0.0460 (17)	−0.0045 (16)	0.0254 (14)	−0.0047 (16)
C2	0.0410 (15)	0.075 (2)	0.0394 (15)	−0.0063 (15)	0.0244 (13)	0.0013 (15)
C3	0.059 (2)	0.076 (2)	0.0487 (18)	−0.0096 (17)	0.0317 (16)	0.0072 (16)
C4	0.062 (2)	0.105 (3)	0.054 (2)	−0.006 (2)	0.0318 (18)	0.022 (2)
C5	0.059 (2)	0.128 (4)	0.0382 (18)	−0.016 (2)	0.0243 (17)	0.002 (2)
C6	0.061 (2)	0.108 (3)	0.050 (2)	−0.013 (2)	0.0379 (18)	−0.013 (2)
C7	0.0515 (18)	0.084 (2)	0.0435 (17)	−0.0019 (17)	0.0309 (15)	−0.0026 (16)
C8	0.064 (2)	0.0451 (18)	0.083 (2)	−0.0014 (16)	0.0382 (19)	−0.0140 (17)
C9	0.0381 (15)	0.0383 (16)	0.0536 (17)	−0.0010 (12)	0.0167 (14)	0.0022 (13)
C10	0.056 (2)	0.0467 (19)	0.080 (2)	−0.0059 (15)	0.0290 (19)	0.0172 (17)
C11	0.053 (2)	0.074 (3)	0.070 (2)	−0.0105 (17)	0.0257 (18)	0.0306 (19)
C12	0.0381 (16)	0.085 (2)	0.0373 (16)	−0.0023 (15)	0.0166 (14)	0.0156 (16)
C13	0.0469 (19)	0.142 (4)	0.046 (2)	−0.004 (2)	0.0281 (17)	0.018 (2)
C14	0.049 (2)	0.151 (4)	0.0439 (19)	0.015 (2)	0.0286 (17)	−0.002 (2)
C15	0.0424 (17)	0.099 (3)	0.0357 (16)	0.0159 (17)	0.0165 (14)	−0.0070 (17)
C16	0.068 (2)	0.112 (4)	0.057 (2)	0.034 (2)	0.023 (2)	−0.024 (2)
C17	0.089 (3)	0.064 (3)	0.074 (3)	0.027 (2)	0.025 (2)	−0.015 (2)
C18	0.063 (2)	0.0459 (18)	0.0468 (18)	0.0084 (15)	0.0113 (16)	−0.0071 (14)
C19	0.113 (3)	0.0406 (18)	0.081 (3)	−0.010 (2)	0.037 (3)	0.0009 (18)
C20	0.0314 (13)	0.0614 (19)	0.0289 (13)	0.0072 (13)	0.0102 (11)	−0.0007 (12)
C21	0.0323 (14)	0.0541 (17)	0.0315 (14)	0.0021 (12)	0.0138 (12)	0.0055 (12)
C22	0.097 (3)	0.086 (3)	0.147 (5)	−0.016 (3)	0.043 (3)	0.031 (3)
C23	0.048 (2)	0.091 (3)	0.074 (3)	−0.0106 (19)	0.0175 (19)	0.020 (2)
C24	0.065 (3)	0.160 (5)	0.079 (3)	−0.024 (3)	0.036 (2)	0.034 (3)
C25	0.058 (2)	0.191 (6)	0.064 (3)	−0.009 (3)	0.038 (2)	0.007 (3)
C26	0.0425 (18)	0.117 (3)	0.0456 (19)	0.0036 (19)	0.0214 (16)	−0.0049 (19)
C27	0.054 (2)	0.157 (5)	0.058 (2)	0.027 (3)	0.022 (2)	−0.030 (3)
C28	0.065 (3)	0.101 (3)	0.071 (3)	0.026 (2)	0.014 (2)	−0.031 (3)

C29	0.0454 (18)	0.059 (2)	0.0537 (19)	0.0055 (15)	0.0082 (16)	−0.0132 (16)
C30	0.065 (3)	0.054 (2)	0.087 (3)	−0.005 (2)	0.006 (2)	0.001 (2)
C31	0.062 (3)	0.090 (3)	0.090 (3)	−0.015 (2)	0.019 (2)	0.028 (3)
C32	0.0415 (18)	0.096 (3)	0.058 (2)	−0.0078 (18)	0.0194 (16)	0.012 (2)
C33	0.065 (3)	0.177 (5)	0.083 (3)	−0.007 (3)	0.048 (2)	0.020 (3)
C34	0.0336 (15)	0.0576 (18)	0.0386 (15)	0.0002 (13)	0.0105 (13)	−0.0070 (13)
C35	0.0313 (14)	0.076 (2)	0.0391 (15)	0.0004 (15)	0.0128 (12)	−0.0060 (15)
C36	0.0386 (14)	0.0427 (16)	0.0359 (14)	−0.0011 (12)	0.0203 (12)	0.0009 (12)
C37	0.0381 (14)	0.0429 (15)	0.0357 (14)	0.0037 (12)	0.0203 (12)	0.0017 (12)
C38	0.0492 (17)	0.0523 (18)	0.0458 (17)	0.0066 (14)	0.0208 (15)	−0.0102 (14)
C39	0.0502 (18)	0.063 (2)	0.060 (2)	0.0179 (16)	0.0267 (16)	−0.0034 (17)
C40	0.0373 (16)	0.074 (2)	0.0525 (19)	0.0135 (15)	0.0164 (14)	−0.0033 (16)
C41	0.0411 (16)	0.067 (2)	0.0488 (17)	0.0041 (15)	0.0152 (14)	−0.0138 (16)
C42	0.0391 (15)	0.0549 (18)	0.0475 (16)	0.0095 (13)	0.0192 (13)	−0.0084 (14)

Geometric parameters (Å, °)

Pb1—N1	2.493 (2)	C13—H13	0.9300
Pb1—N2	2.477 (2)	C14—C15	1.428 (5)
Pb1—O1	2.639 (2)	C14—H14	0.9300
Pb1—O2	2.637 (2)	C15—C20	1.405 (4)
Pb1—O4	2.569 (2)	C15—C16	1.406 (6)
Pb1—O5	2.6296 (19)	C16—C17	1.349 (6)
O1—C1	1.254 (4)	C16—H16	0.9300
O2—C1	1.253 (4)	C17—C18	1.406 (6)
O3—C6	1.359 (4)	C17—H17	0.9300
O3—H3	0.8200	C18—C19	1.500 (5)
O4—C36	1.265 (3)	C19—H19A	0.9600
O5—C36	1.254 (3)	C19—H19B	0.9600
O6—C41	1.371 (4)	C19—H19C	0.9600
O6—H6	0.8200	C20—C21	1.437 (4)
O7—H1W	0.8331	C22—C23	1.501 (6)
O7—H2W	0.8357	C22—H22A	0.9600
O8—H3W	0.8493	C22—H22B	0.9600
O8—H4W	0.8274	C22—H22C	0.9600
O9—H5W	0.8310	C23—C24	1.409 (6)
O9—H6W	0.8336	C24—C25	1.351 (7)
N1—C18	1.334 (4)	C24—H24	0.9300
N1—C20	1.365 (4)	C25—C26	1.385 (7)
N2—C9	1.333 (3)	C25—H25	0.9300
N2—C21	1.375 (3)	C26—C35	1.416 (5)
N3—C23	1.331 (4)	C26—C27	1.430 (6)
N3—C35	1.357 (4)	C27—C28	1.329 (6)
N4—C32	1.325 (4)	C27—H27	0.9300
N4—C34	1.359 (4)	C28—C29	1.414 (6)
C1—C2	1.508 (4)	C28—H28	0.9300
C2—C7	1.386 (4)	C29—C30	1.401 (6)
C2—C3	1.393 (4)	C29—C34	1.411 (4)
C3—C4	1.396 (4)	C30—C31	1.344 (6)

C3—H3A	0.9300	C30—H30	0.9300
C4—C5	1.375 (5)	C31—C32	1.400 (6)
C4—H4	0.9300	C31—H31	0.9300
C5—C6	1.383 (5)	C32—C33	1.505 (5)
C5—H5	0.9300	C33—H33A	0.9600
C6—C7	1.396 (4)	C33—H33B	0.9600
C7—H7	0.9300	C33—H33C	0.9600
C8—C9	1.499 (4)	C34—C35	1.438 (4)
C8—H8A	0.9600	C36—C37	1.496 (3)
C8—H8B	0.9600	C37—C42	1.387 (4)
C8—H8C	0.9600	C37—C38	1.390 (4)
C9—C10	1.406 (4)	C38—C39	1.384 (4)
C10—C11	1.342 (5)	C38—H38	0.9300
C10—H10	0.9300	C39—C40	1.369 (4)
C11—C12	1.410 (5)	C39—H39	0.9300
C11—H11	0.9300	C40—C41	1.379 (4)
C12—C21	1.410 (4)	C40—H40	0.9300
C12—C13	1.431 (5)	C41—C42	1.393 (4)
C13—C14	1.346 (5)	C42—H42	0.9300
N2—Pb1—N1	67.50 (7)	C18—C17—H17	119.9
N2—Pb1—O4	83.47 (7)	N1—C18—C17	120.2 (4)
N1—Pb1—O4	123.42 (7)	N1—C18—C19	117.6 (3)
N2—Pb1—O5	88.14 (7)	C17—C18—C19	122.2 (3)
N1—Pb1—O5	80.54 (6)	C18—C19—H19A	109.5
O4—Pb1—O5	49.89 (6)	C18—C19—H19B	109.5
N2—Pb1—O2	87.06 (7)	H19A—C19—H19B	109.5
N1—Pb1—O2	120.61 (7)	C18—C19—H19C	109.5
O4—Pb1—O2	104.22 (7)	H19A—C19—H19C	109.5
O5—Pb1—O2	154.07 (7)	H19B—C19—H19C	109.5
N2—Pb1—O1	98.49 (7)	N1—C20—C15	121.8 (3)
N1—Pb1—O1	81.39 (7)	N1—C20—C21	118.9 (2)
O4—Pb1—O1	152.86 (7)	C15—C20—C21	119.3 (3)
O5—Pb1—O1	156.62 (7)	N2—C21—C12	121.7 (3)
O2—Pb1—O1	49.22 (7)	N2—C21—C20	118.9 (2)
C1—O1—Pb1	94.05 (18)	C12—C21—C20	119.5 (3)
C1—O2—Pb1	94.19 (19)	C23—C22—H22A	109.5
C6—O3—H3	109.5	C23—C22—H22B	109.5
C36—O4—Pb1	93.58 (16)	H22A—C22—H22B	109.5
C36—O5—Pb1	91.03 (15)	C23—C22—H22C	109.5
C41—O6—H6	109.5	H22A—C22—H22C	109.5
H1W—O7—H2W	110.4	H22B—C22—H22C	109.5
H3W—O8—H4W	103.4	N3—C23—C24	121.2 (4)
H5W—O9—H6W	110.8	N3—C23—C22	117.0 (4)
C18—N1—C20	120.3 (3)	C24—C23—C22	121.7 (4)
C18—N1—Pb1	122.8 (2)	C25—C24—C23	120.3 (4)
C20—N1—Pb1	116.50 (17)	C25—C24—H24	119.8
C9—N2—C21	119.1 (2)	C23—C24—H24	119.8
C9—N2—Pb1	123.75 (18)	C24—C25—C26	120.2 (4)
C21—N2—Pb1	116.78 (17)	C24—C25—H25	119.9

C23—N3—C35	118.5 (3)	C26—C25—H25	119.9
C32—N4—C34	118.6 (3)	C25—C26—C35	117.0 (4)
O2—C1—O1	122.5 (3)	C25—C26—C27	123.9 (4)
O2—C1—C2	118.8 (3)	C35—C26—C27	119.1 (4)
O1—C1—C2	118.8 (3)	C28—C27—C26	121.4 (4)
C7—C2—C3	120.7 (3)	C28—C27—H27	119.3
C7—C2—C1	118.9 (3)	C26—C27—H27	119.3
C3—C2—C1	120.4 (3)	C27—C28—C29	121.6 (4)
C2—C3—C4	118.8 (3)	C27—C28—H28	119.2
C2—C3—H3A	120.6	C29—C28—H28	119.2
C4—C3—H3A	120.6	C30—C29—C34	116.9 (4)
C5—C4—C3	120.5 (4)	C30—C29—C28	123.6 (4)
C5—C4—H4	119.8	C34—C29—C28	119.5 (4)
C3—C4—H4	119.8	C31—C30—C29	120.4 (4)
C4—C5—C6	120.7 (3)	C31—C30—H30	119.8
C4—C5—H5	119.6	C29—C30—H30	119.8
C6—C5—H5	119.6	C30—C31—C32	119.6 (4)
O3—C6—C5	118.5 (3)	C30—C31—H31	120.2
O3—C6—C7	122.1 (4)	C32—C31—H31	120.2
C5—C6—C7	119.5 (4)	N4—C32—C31	122.3 (4)
C2—C7—C6	119.8 (3)	N4—C32—C33	117.3 (4)
C2—C7—H7	120.1	C31—C32—C33	120.4 (4)
C6—C7—H7	120.1	C32—C33—H33A	109.5
C9—C8—H8A	109.5	C32—C33—H33B	109.5
C9—C8—H8B	109.5	H33A—C33—H33B	109.5
H8A—C8—H8B	109.5	C32—C33—H33C	109.5
C9—C8—H8C	109.5	H33A—C33—H33C	109.5
H8A—C8—H8C	109.5	H33B—C33—H33C	109.5
H8B—C8—H8C	109.5	N4—C34—C29	122.2 (3)
N2—C9—C10	121.0 (3)	N4—C34—C35	118.4 (3)
N2—C9—C8	119.8 (3)	C29—C34—C35	119.4 (3)
C10—C9—C8	119.2 (3)	N3—C35—C26	122.8 (3)
C11—C10—C9	121.1 (3)	N3—C35—C34	118.3 (3)
C11—C10—H10	119.5	C26—C35—C34	119.0 (3)
C9—C10—H10	119.5	O5—C36—O4	121.1 (2)
C10—C11—C12	119.6 (3)	O5—C36—C37	120.3 (2)
C10—C11—H11	120.2	O4—C36—C37	118.6 (2)
C12—C11—H11	120.2	C42—C37—C38	119.4 (2)
C11—C12—C21	117.5 (3)	C42—C37—C36	120.3 (2)
C11—C12—C13	123.1 (3)	C38—C37—C36	120.2 (2)
C21—C12—C13	119.3 (3)	C39—C38—C37	120.0 (3)
C14—C13—C12	121.2 (4)	C39—C38—H38	120.0
C14—C13—H13	119.4	C37—C38—H38	120.0
C12—C13—H13	119.4	C40—C39—C38	120.4 (3)
C13—C14—C15	120.8 (3)	C40—C39—H39	119.8
C13—C14—H14	119.6	C38—C39—H39	119.8
C15—C14—H14	119.6	C39—C40—C41	120.4 (3)
C20—C15—C16	116.7 (4)	C39—C40—H40	119.8
C20—C15—C14	119.9 (3)	C41—C40—H40	119.8

C16—C15—C14	123.4 (4)	O6—C41—C40	117.7 (3)
C17—C16—C15	120.8 (4)	O6—C41—C42	122.5 (3)
C17—C16—H16	119.6	C40—C41—C42	119.8 (3)
C15—C16—H16	119.6	C37—C42—C41	120.0 (3)
C16—C17—C18	120.3 (4)	C37—C42—H42	120.0
C16—C17—H17	119.9	C41—C42—H42	120.0
N2—Pb1—O1—C1	80.06 (19)	C20—N1—C18—C19	178.1 (3)
N1—Pb1—O1—C1	145.6 (2)	Pb1—N1—C18—C19	−9.5 (4)
O4—Pb1—O1—C1	−12.2 (3)	C16—C17—C18—N1	1.1 (5)
O5—Pb1—O1—C1	−174.70 (17)	C16—C17—C18—C19	−179.2 (4)
O2—Pb1—O1—C1	1.44 (17)	C18—N1—C20—C15	1.9 (4)
N2—Pb1—O2—C1	−105.30 (19)	Pb1—N1—C20—C15	−170.99 (19)
N1—Pb1—O2—C1	−43.7 (2)	C18—N1—C20—C21	−177.4 (2)
O4—Pb1—O2—C1	172.20 (18)	Pb1—N1—C20—C21	9.7 (3)
O5—Pb1—O2—C1	175.06 (17)	C16—C15—C20—N1	−0.4 (4)
O1—Pb1—O2—C1	−1.44 (17)	C14—C15—C20—N1	−179.8 (3)
N2—Pb1—O4—C36	104.53 (17)	C16—C15—C20—C21	178.9 (3)
N1—Pb1—O4—C36	47.02 (18)	C14—C15—C20—C21	−0.6 (4)
O5—Pb1—O4—C36	11.45 (15)	C9—N2—C21—C12	−1.2 (4)
O2—Pb1—O4—C36	−170.18 (16)	Pb1—N2—C21—C12	172.30 (19)
O1—Pb1—O4—C36	−159.58 (17)	C9—N2—C21—C20	177.7 (2)
N2—Pb1—O5—C36	−94.55 (17)	Pb1—N2—C21—C20	−8.8 (3)
N1—Pb1—O5—C36	−162.04 (17)	C11—C12—C21—N2	0.7 (4)
O4—Pb1—O5—C36	−11.52 (15)	C13—C12—C21—N2	−179.4 (3)
O2—Pb1—O5—C36	−15.1 (3)	C11—C12—C21—C20	−178.2 (2)
O1—Pb1—O5—C36	158.15 (18)	C13—C12—C21—C20	1.7 (4)
N2—Pb1—N1—C18	177.4 (2)	N1—C20—C21—N2	−0.7 (3)
O4—Pb1—N1—C18	−117.5 (2)	C15—C20—C21—N2	−180.0 (2)
O5—Pb1—N1—C18	−90.7 (2)	N1—C20—C21—C12	178.2 (2)
O2—Pb1—N1—C18	105.4 (2)	C15—C20—C21—C12	−1.1 (4)
O1—Pb1—N1—C18	74.4 (2)	C35—N3—C23—C24	0.6 (5)
N2—Pb1—N1—C20	−9.94 (16)	C35—N3—C23—C22	−177.3 (3)
O4—Pb1—N1—C20	55.17 (19)	N3—C23—C24—C25	−0.7 (6)
O5—Pb1—N1—C20	81.98 (17)	C22—C23—C24—C25	177.1 (4)
O2—Pb1—N1—C20	−81.91 (18)	C23—C24—C25—C26	−0.1 (7)
O1—Pb1—N1—C20	−112.91 (18)	C24—C25—C26—C35	1.0 (6)
N1—Pb1—N2—C9	−177.3 (2)	C24—C25—C26—C27	−178.7 (4)
O4—Pb1—N2—C9	52.4 (2)	C25—C26—C27—C28	179.1 (4)
O5—Pb1—N2—C9	102.2 (2)	C35—C26—C27—C28	−0.6 (6)
O2—Pb1—N2—C9	−52.3 (2)	C26—C27—C28—C29	1.1 (6)
O1—Pb1—N2—C9	−100.3 (2)	C27—C28—C29—C30	−179.9 (4)
N1—Pb1—N2—C21	9.58 (16)	C27—C28—C29—C34	−0.7 (5)
O4—Pb1—N2—C21	−120.78 (17)	C34—C29—C30—C31	−0.2 (5)
O5—Pb1—N2—C21	−70.95 (17)	C28—C29—C30—C31	179.0 (4)
O2—Pb1—N2—C21	134.55 (17)	C29—C30—C31—C32	0.8 (6)
O1—Pb1—N2—C21	86.53 (17)	C34—N4—C32—C31	−0.1 (5)
Pb1—O2—C1—O1	2.7 (3)	C34—N4—C32—C33	−178.2 (3)
Pb1—O2—C1—C2	−177.5 (2)	C30—C31—C32—N4	−0.6 (6)
Pb1—O1—C1—O2	−2.7 (3)	C30—C31—C32—C33	177.4 (4)

Pb1—O1—C1—C2	177.5 (2)	C32—N4—C34—C29	0.8 (4)
O2—C1—C2—C7	−19.0 (4)	C32—N4—C34—C35	−178.9 (3)
O1—C1—C2—C7	160.8 (3)	C30—C29—C34—N4	−0.6 (4)
O2—C1—C2—C3	163.1 (3)	C28—C29—C34—N4	−179.9 (3)
O1—C1—C2—C3	−17.1 (4)	C30—C29—C34—C35	179.0 (3)
C7—C2—C3—C4	1.8 (5)	C28—C29—C34—C35	−0.3 (4)
C1—C2—C3—C4	179.7 (3)	C23—N3—C35—C26	0.3 (4)
C2—C3—C4—C5	−1.5 (5)	C23—N3—C35—C34	179.2 (3)
C3—C4—C5—C6	−1.1 (5)	C25—C26—C35—N3	−1.2 (5)
C4—C5—C6—O3	−176.1 (3)	C27—C26—C35—N3	178.6 (3)
C4—C5—C6—C7	3.3 (5)	C25—C26—C35—C34	−180.0 (3)
C3—C2—C7—C6	0.4 (5)	C27—C26—C35—C34	−0.3 (4)
C1—C2—C7—C6	−177.6 (3)	N4—C34—C35—N3	1.4 (4)
O3—C6—C7—C2	176.4 (3)	C29—C34—C35—N3	−178.2 (3)
C5—C6—C7—C2	−2.9 (5)	N4—C34—C35—C26	−179.7 (3)
C21—N2—C9—C10	0.6 (4)	C29—C34—C35—C26	0.7 (4)
Pb1—N2—C9—C10	−172.4 (2)	Pb1—O5—C36—O4	21.3 (3)
C21—N2—C9—C8	−179.7 (3)	Pb1—O5—C36—C37	−157.8 (2)
Pb1—N2—C9—C8	7.3 (4)	Pb1—O4—C36—O5	−21.8 (3)
N2—C9—C10—C11	0.5 (5)	Pb1—O4—C36—C37	157.3 (2)
C8—C9—C10—C11	−179.2 (3)	O5—C36—C37—C42	5.5 (4)
C9—C10—C11—C12	−0.9 (5)	O4—C36—C37—C42	−173.6 (3)
C10—C11—C12—C21	0.3 (4)	O5—C36—C37—C38	−175.4 (3)
C10—C11—C12—C13	−179.5 (3)	O4—C36—C37—C38	5.5 (4)
C11—C12—C13—C14	179.1 (3)	C42—C37—C38—C39	1.3 (5)
C21—C12—C13—C14	−0.7 (5)	C36—C37—C38—C39	−177.8 (3)
C12—C13—C14—C15	−0.9 (5)	C37—C38—C39—C40	−0.6 (5)
C13—C14—C15—C20	1.6 (5)	C38—C39—C40—C41	−0.2 (5)
C13—C14—C15—C16	−177.8 (3)	C39—C40—C41—O6	−178.4 (3)
C20—C15—C16—C17	−0.7 (5)	C39—C40—C41—C42	0.2 (5)
C14—C15—C16—C17	178.7 (3)	C38—C37—C42—C41	−1.2 (4)
C15—C16—C17—C18	0.4 (6)	C36—C37—C42—C41	177.9 (3)
C20—N1—C18—C17	−2.2 (4)	O6—C41—C42—C37	179.0 (3)
Pb1—N1—C18—C17	170.2 (2)	C40—C41—C42—C37	0.4 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O8^{i}	0.82	1.86	2.669 (4)	169
O6—H6···N4	0.82	2.15	2.916 (4)	155
O7—H1W···O1	0.83	1.88	2.702 (3)	172
O7—H2W···O6^{ii}	0.84	2.15	2.977 (3)	169
O8—H3W···O4^{iii}	0.85	1.94	2.784 (3)	180
O8—H4W···O9^{iv}	0.83	2.02	2.828 (4)	166
O9—H6W···O2^{v}	0.83	2.15	2.850 (4)	141
O9—H5W···O8^{iv}	0.83	2.19	2.828 (4)	133
O6—H6···N3	0.82	2.51	3.124 (4)	133

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

Fig. 1

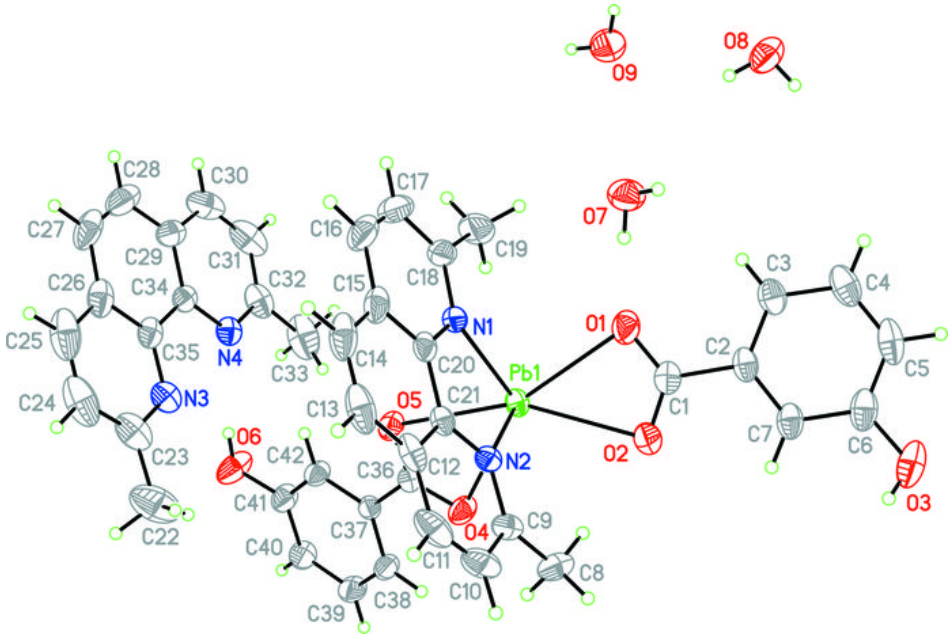
O9
O8
O7
C28
C30
C27
C29
C31
C26
C34
C32
C25
C35
N4
C33
C24
N3
C23
C22
C17
C16
C19
C15
C18
N1
C14
C20
C13
O5
C21
Pb1
O1
C2
C3
C4
C5
C1
O2
C7
C6
O3
O6
C42
C36
C12
N2
C41
C37
O4
C9
C40
C11
C8
C10
C38
C39

Fig. 2

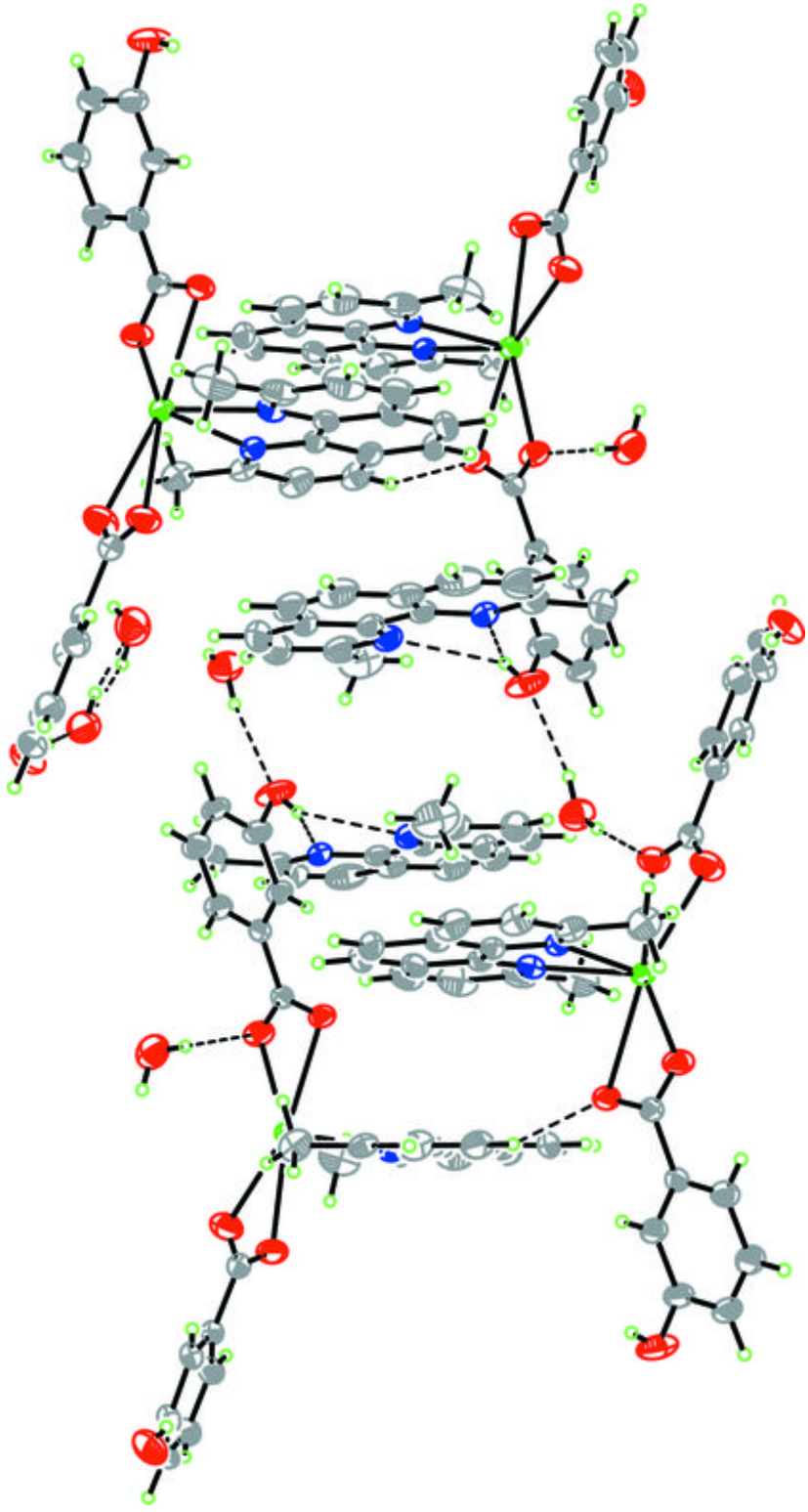

Fig. 3

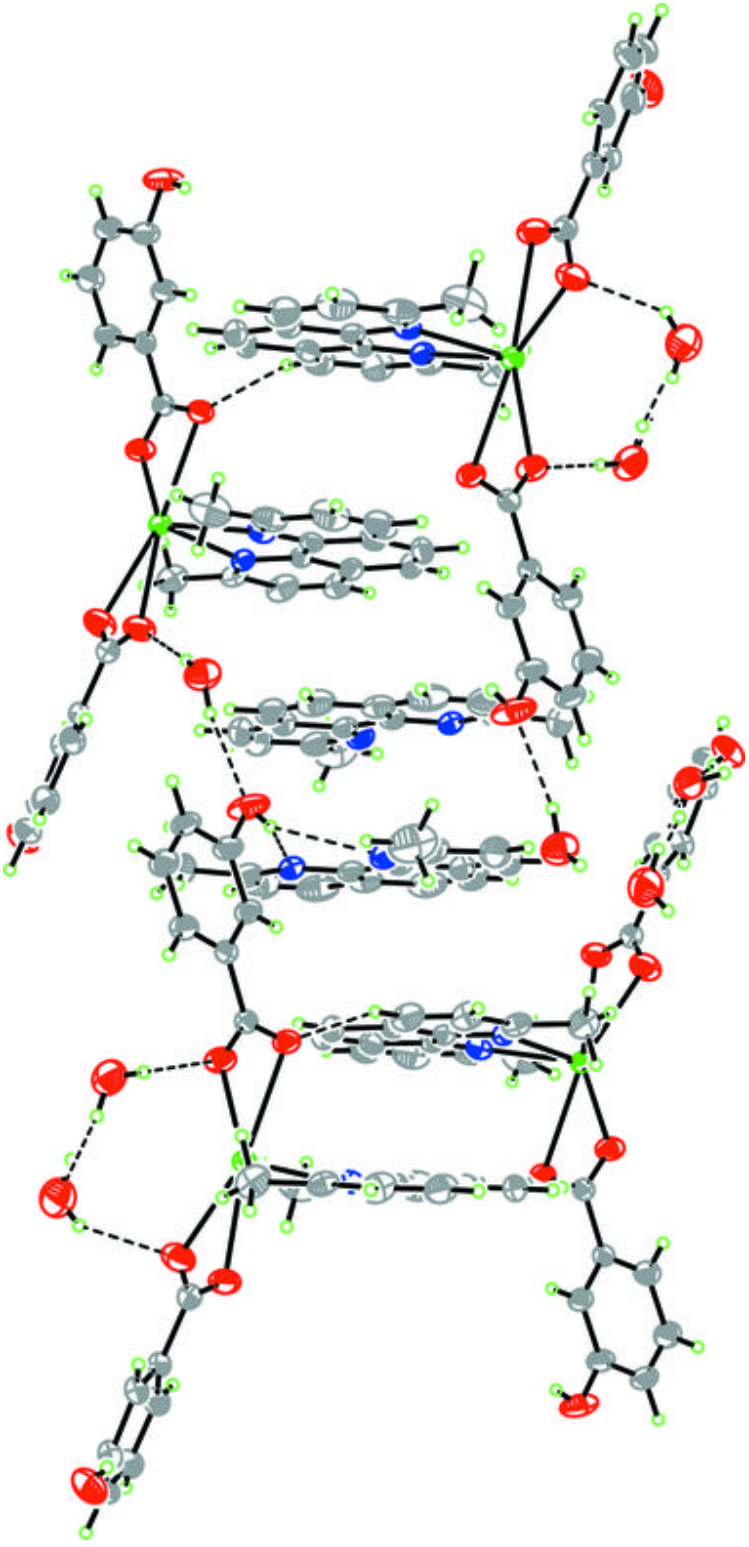

Fig. 4