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Bis(μ-3-hydroxybenzoato-$\kappa^3O,O':O$)bis[aqua(3-hydroxybenzoato-κ^2O,O')(1,10-phenanthroline-κ^2N,N')lead(II)] monohydrate

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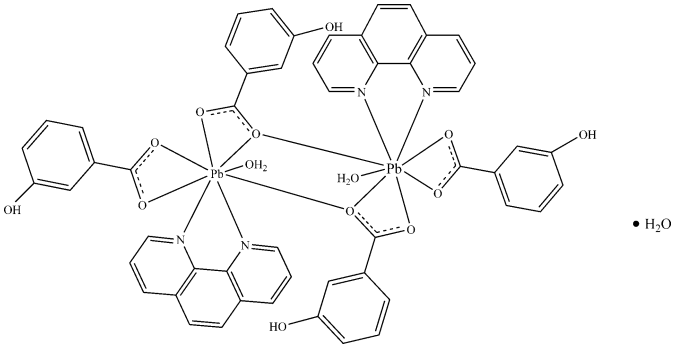
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.022; wR factor = 0.050; data-to-parameter ratio = 13.1.

In the centrosymmetric binuclear title complex, $[Pb_2(C_7H_5O_3)_4(C_{12}H_8N_2)_2(H_2O)_2]\cdot H_2O$, each Pb atom is eight-coordinated in a PbO_6N_2 environment by two N atoms from the 1,10-phenanthroline (phen) ligand, five carboxylate O atoms from four 3-hydroxybenzoate anions and one O atom from the coordinated water molecule in a distorted bicapped trigonal-prismatic geometry. The benzoate groups coordinate each Pb^{II} atom in two different ways. Two benzoate ions behave as bidentate ligands to the Pb atom, and another benzoate ion bridges the Pb atoms, forming a binuclear structure. The dimeric units are packed *via* O—H···O hydrogen bonds and π–π interactions between the aromatic rings of neighboring molecules, with centroid–centroid distances of 3.552 (2) and 3.641 (2) Å.

Related literature

For related structures, see: Li & Yang (2004); Mahjoub & Morsali (2002); Xuan *et al.* (2007); Zhu *et al.* (2004); For information on the coordination chemistry of lead, see: Shimoni-Livny *et al.* (1998).



Experimental

Crystal data

$[Pb_2(C_7H_5O_3)_4(C_{12}H_8N_2)_2(H_2O)_2]\cdot H_2O$
M_r = 1377.30
Triclinic, $P\overline{1}$
a = 8.5639 (16) Å
b = 12.152 (2) Å
c = 12.979 (3) Å
α = 62.652 (1)°
β = 82.762 (2)°
γ = 84.701 (2)°
V = 1189.2 (4) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 7.15 mm^{-1}
T = 293 (2) K
0.17 × 0.15 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.369, T_{max} = 0.512
(expected range = 0.328–0.456)
8820 measured reflections
4352 independent reflections
4009 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.050
S = 1.04
4352 reflections
333 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

Table 1
Selected bond lengths (Å).

Pb1—O1	2.472 (2)	Pb1—O5	2.720 (2)
Pb1—N1	2.551 (2)	Pb1—O6	2.815 (2)
Pb1—N2	2.585 (3)	Pb1—O6^{i}	2.880 (2)
Pb1—O2	2.630 (2)	Pb1—O7	2.914 (4)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O5^{ii}	0.82	1.84	2.658 (3)	176
O4—H4···O1^{iii}	0.82	1.93	2.738 (3)	167
O7—H2*W*···O8^{iv}	0.83	2.24	2.932 (6)	141
O7—H1*W*···O3^{v}	0.83	2.18	2.911 (5)	146
O8—H3*W*···O2^{vi}	0.83	2.06	2.840 (4)	157
O8—H4*W*···O2^{vii}	0.83	2.04	2.788 (4)	150
C15—H15···O8^{vi}	0.93	2.42	3.339 (5)	169

Symmetry codes: (ii) $-x+1, -y, -z$; (iii) $-x+1, -y, -z+1$; (iv) $x-1, y-1, z$; (v) $-x, -y, -z$; (vi) $-x+1, -y+1, -z$; (vii) $x+1, y+1, z$.

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: SI2047).

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supplementary materials

Acta Cryst. (2008). E**64**, m152-m153 [**doi:10.1107/S1600536807065075**]

Bis(μ-3-hydroxybenzoato-$\kappa^3O,O':O$)bis[aqua(3-hydroxybenzoato-κ^2O,O')(1,10-phenanthroline-κ^2N,N')lead(II)] monohydrate

X.-P. Xuan, P.-Z. Zhao and S.-X. Zhang

Comment

Lead(II) is capable of exhibiting a variable coordination number and geometry with or without a stereochemically active lone pair of electrons (Shimoni-Livny *et al.*1998). Among such compounds, a number of centrosymmetric dinuclear lead(II) compounds with 1,10-phenanthroline (phen) and oxygen donor ligands have been structurally characterized (Li & Yang, 2004, Mahjoub & Morsali 2002, Zhu *et al.* 2004). Recently, we obtained the title lead(II) complex, by reaction of lead acetate, 3-hydroxybenzoate acid, sodium hydroxide and in ethanol/water mixtures. The resulting complex is different to the polymeric chain-structure we previously reported using 2-hydroxybenzoate acid instead of 3-hydroxybenzoate acid (Xuan *et al.* 2007).

The crystal structure of the title compound consists of dimeric units of $[Pb(C_7H_5O_3)_2(C_{12}H_8N_2)(H_2O)]$, related by a crystallographic inversion centre (Fig. 1), and an uncoordinated (disordered) water molecule. Each lead atom is chelated by the two N atoms of 1,10-phenanthroline (phen) with Pb—N distances of 2.551 (2), and 2.583 (3) Å, and five carbonyl O atoms of 3-hydroxyl-benzoate anions, and is coordinated by the O atom of one water molecule. The shortest Pb—O distance is 2.472 (2) Å, and the longest is 2.914 (4) Å. The weak Pb—O bridging interactions form a four-membered Pb_2O_2 quadrilateral with a Pb—Pb separation of 4.1712 (8) Å. The uncoordinated water molecule is disordered over two sites close to a crystallographic inversion centre.

In the presented structure, the crystal is stabilized by intermolecular O—H···O and C—H···O hydrogen bonding contacts (Table 1 and Figure 2) and by two kinds of π-π stacking interactions in the sequence of benzoate - phen - phen - benzoate. The centroid-centroid distances between *Cg*1(N1/C1—C4/C12) and *Cg*2 (C21—C26)[symmetry code: -*x*, -*y*, 1 - *z*] and between *Cg*1 and *Cg*3 (N1/C1—C4/C12) [symmetry code: -*x*, 1 - *y*, -*z*] are 3.552 (2) and 3.641 (2) Å, respectively.

Experimental

To a solution of 1,10-phenanthroline (0.0906 g, 0.5 mmol), 3-hydroxybenzoate acid(0.1394 g, 1 mmol) and sodium hydroxide (0.0185 g,0.5 mmol) in ethanol/water (*v*:*v*=1:1,20 ml) was added a solution of $Pb(CH_3COO)_2.3H_2O$ (0.1903 g, 0.5 mmol) in distilled water (5 ml). The resulting solution was stirred for 5 h at 323 K and then a white precipitate was filtered. Block single crystals were obtained by slow evaporation of the filtrate after 2 d, one of which was selected for the X-ray experiment.

Refinement

The carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C aromatic). The hydroxyl H atoms were placed in calculated positions (O—H = 0.82 Å) and refined with free torsion angles to fit the electron density, with U_{iso}(H) = 1.5 U_{eq}(O). The

solvent water is disordered over two sites close to a crystallographic inversion centre, thus the site occupation factors for O8 and H3W, H4W were set 1/2. The water H atoms were restraint at distances O—H = 0.83 Å with U_{iso}(H) = 1.5U_{eq}(O), for both, the coordinated and uncoordinated water molecules.

Figures

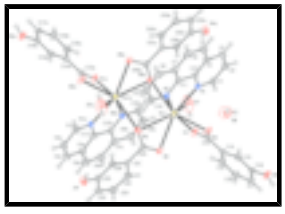


Fig. 1. The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for non-H atoms. [Symmetry codes for atoms labelled a: -*x*, -*y*, 1 - *z*].

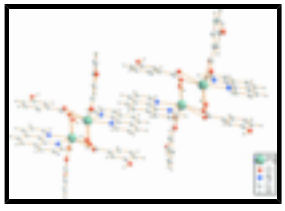


Fig. 2. The π-π interactions between the aromatic rings of the title compound.

Bis(μ-3-hydroxybenzoato-κ^3*O*,*O*':*O*)bis[aqua(3-hydroxybenzoato- κ^2*O*,*O*')(1,10-phenanthroline-κ^2*N*,*N*')lead(II)] monohydrate

Crystal data

$[Pb_2(C_7H_5O_3)_4(C_{12}H_8N_2)_2(H_2O)_2]\cdot H_2O$	Z = 1
M_r = 1377.30	F_{000} = 666
Triclinic, $P\overline{1}$	D_x = 1.923 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.5639 (16) Å	Cell parameters from 5362 reflections
b = 12.152 (2) Å	θ = 2.4–27.5º
c = 12.979 (3) Å	μ = 7.15 mm^{-1}
α = 62.652 (1)º	T = 293 (2) K
β = 82.762 (2)º	Block, yellow
γ = 84.701 (2)º	0.17 × 0.15 × 0.11 mm
V = 1189.2 (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	4352 independent reflections
Radiation source: fine-focus sealed tube	4009 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 293(2) K	θ_{max} = 25.5º
phi and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −10→10

T_{min} = 0.369, T_{max} = 0.512
8820 measured reflections
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.050
S = 1.04
4352 reflections
333 parameters
12 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
$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Pb1	0.062168 (13)	0.070072 (10)	0.321211 (9)	0.02816 (4)	
O1	0.3051 (3)	0.1587 (2)	0.19470 (18)	0.0387 (6)	
O2	0.1526 (3)	0.0939 (2)	0.1109 (2)	0.0450 (6)	
O3	0.4628 (3)	0.1416 (3)	−0.2612 (2)	0.0520 (7)	
H3	0.5442	0.1402	−0.3015	0.078*	
O4	0.5876 (3)	−0.3747 (2)	0.8277 (2)	0.0480 (7)	
H4	0.6085	−0.3047	0.8146	0.072*	
O5	0.2736 (3)	−0.1258 (2)	0.38683 (19)	0.0522 (7)	
O6	0.2135 (3)	−0.0679 (2)	0.5260 (2)	0.0434 (6)	
O7	−0.1913 (5)	−0.0008 (4)	0.2362 (4)	0.1267 (16)	
H2W	−0.1381	−0.0498	0.2158	0.190*	
H1W	−0.2871	−0.0133	0.2430	0.190*	
N1	−0.0483 (3)	0.2875 (2)	0.1953 (2)	0.0306 (6)	
N2	0.1144 (3)	0.2374 (2)	0.3819 (2)	0.0307 (6)	
C1	−0.1321 (4)	0.3108 (3)	0.1076 (3)	0.0361 (8)	

H1	−0.1418	0.2470	0.0884	0.043*	
C2	−0.2063 (4)	0.4251 (3)	0.0427 (3)	0.0441 (9)	
H2	−0.2649	0.4371	−0.0175	0.053*	
C3	−0.1904 (4)	0.5195 (3)	0.0703 (3)	0.0459 (10)	
H3A	−0.2386	0.5968	0.0283	0.055*	
C4	−0.1018 (4)	0.5004 (3)	0.1615 (3)	0.0374 (8)	
C5	−0.0782 (5)	0.5954 (3)	0.1930 (3)	0.0483 (10)	
H5	−0.1212	0.6747	0.1508	0.058*	
C6	0.0051 (5)	0.5725 (3)	0.2824 (3)	0.0478 (9)	
H6	0.0203	0.6366	0.2999	0.057*	
C7	0.0717 (4)	0.4497 (3)	0.3521 (3)	0.0378 (8)	
C8	0.1538 (4)	0.4193 (3)	0.4492 (3)	0.0441 (9)	
H8A	0.1677	0.4799	0.4718	0.053*	
C9	0.2131 (4)	0.3020 (3)	0.5105 (3)	0.0448 (9)	
H9	0.2658	0.2813	0.5758	0.054*	
C10	0.1931 (4)	0.2129 (3)	0.4735 (3)	0.0380 (8)	
H10	0.2364	0.1334	0.5143	0.046*	
C11	0.0534 (4)	0.3546 (3)	0.3214 (3)	0.0289 (7)	
C12	−0.0336 (4)	0.3809 (3)	0.2236 (3)	0.0295 (7)	
C13	0.2813 (4)	0.1357 (3)	0.1119 (3)	0.0308 (7)	
C14	0.4093 (4)	0.1573 (3)	0.0144 (2)	0.0280 (7)	
C15	0.3806 (4)	0.1410 (3)	−0.0805 (3)	0.0334 (7)	
H15	0.2814	0.1187	−0.0845	0.040*	
C16	0.4984 (4)	0.1575 (3)	−0.1691 (3)	0.0327 (8)	
C17	0.6472 (4)	0.1886 (3)	−0.1626 (3)	0.0358 (8)	
H17	0.7273	0.1990	−0.2215	0.043*	
C18	0.6748 (4)	0.2039 (3)	−0.0677 (3)	0.0418 (9)	
H18	0.7747	0.2245	−0.0630	0.050*	
C19	0.5578 (4)	0.1895 (3)	0.0204 (3)	0.0377 (8)	
H19	0.5784	0.2013	0.0833	0.045*	
C20	0.2779 (4)	−0.1430 (3)	0.4891 (3)	0.0322 (7)	
C21	0.3627 (4)	−0.2582 (3)	0.5717 (3)	0.0287 (7)	
C22	0.3647 (4)	−0.3663 (3)	0.5590 (3)	0.0362 (8)	
H22	0.3189	−0.3653	0.4972	0.043*	
C23	0.4342 (4)	−0.4746 (3)	0.6377 (3)	0.0407 (9)	
H23	0.4334	−0.5467	0.6297	0.049*	
C24	0.5050 (4)	−0.4762 (3)	0.7280 (3)	0.0380 (8)	
H24	0.5493	−0.5500	0.7819	0.046*	
C25	0.5104 (4)	−0.3681 (3)	0.7388 (3)	0.0328 (7)	
C26	0.4352 (3)	−0.2600 (3)	0.6630 (2)	0.0314 (7)	
H26	0.4332	−0.1888	0.6729	0.038*	
O8	0.9880 (3)	0.9421 (3)	0.0575 (2)	0.103 (2)	0.50
H4W	1.0384	1.0024	0.0467	0.154*	0.50
H3W	0.9574	0.9510	−0.0041	0.154*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

Pb1	0.03033 (7)	0.03078 (6)	0.02384 (6)	0.00001 (5)	−0.00074 (4)	−0.01346 (4)
O1	0.0418 (13)	0.0503 (12)	0.0318 (11)	−0.0092 (11)	0.0047 (10)	−0.0258 (10)
O2	0.0358 (13)	0.0690 (15)	0.0427 (12)	−0.0154 (12)	0.0085 (10)	−0.0365 (11)
O3	0.0463 (14)	0.0861 (17)	0.0366 (12)	0.0002 (14)	−0.0001 (11)	−0.0404 (12)
O4	0.0576 (15)	0.0449 (13)	0.0401 (13)	−0.0025 (12)	−0.0190 (12)	−0.0144 (11)
O5	0.0574 (16)	0.0655 (16)	0.0298 (12)	0.0260 (13)	−0.0087 (11)	−0.0221 (11)
O6	0.0515 (14)	0.0408 (12)	0.0395 (12)	0.0126 (11)	−0.0056 (11)	−0.0218 (10)
O7	0.131 (3)	0.131 (3)	0.103 (3)	−0.056 (3)	−0.046 (2)	−0.022 (2)
N1	0.0349 (14)	0.0329 (13)	0.0244 (12)	−0.0006 (11)	−0.0032 (11)	−0.0134 (10)
N2	0.0330 (14)	0.0342 (13)	0.0273 (12)	−0.0009 (11)	−0.0040 (11)	−0.0159 (11)
C1	0.0363 (18)	0.0417 (17)	0.0321 (16)	−0.0016 (15)	−0.0064 (14)	−0.0174 (14)
C2	0.0355 (18)	0.056 (2)	0.0338 (17)	0.0024 (16)	−0.0093 (15)	−0.0136 (16)
C3	0.042 (2)	0.0385 (19)	0.0391 (19)	0.0104 (16)	−0.0059 (16)	−0.0042 (16)
C4	0.0339 (17)	0.0318 (16)	0.0377 (18)	0.0040 (14)	0.0045 (14)	−0.0110 (14)
C5	0.053 (2)	0.0311 (17)	0.056 (2)	0.0018 (16)	0.0011 (18)	−0.0178 (16)
C6	0.053 (2)	0.0379 (17)	0.062 (2)	−0.0040 (16)	0.0052 (19)	−0.0321 (16)
C7	0.0340 (18)	0.0421 (17)	0.0423 (18)	−0.0092 (14)	0.0076 (14)	−0.0248 (15)
C8	0.044 (2)	0.0540 (19)	0.0528 (19)	−0.0092 (16)	0.0033 (16)	−0.0403 (16)
C9	0.0399 (19)	0.066 (2)	0.0413 (18)	−0.0040 (17)	−0.0087 (15)	−0.0332 (17)
C10	0.0393 (18)	0.0449 (18)	0.0315 (16)	−0.0023 (15)	−0.0077 (14)	−0.0175 (14)
C11	0.0262 (15)	0.0340 (15)	0.0259 (14)	−0.0047 (13)	0.0056 (12)	−0.0143 (12)
C12	0.0252 (15)	0.0335 (15)	0.0272 (14)	−0.0019 (13)	0.0043 (12)	−0.0132 (13)
C13	0.0339 (17)	0.0332 (15)	0.0280 (15)	0.0006 (13)	−0.0014 (13)	−0.0169 (13)
C14	0.0308 (16)	0.0264 (14)	0.0255 (14)	−0.0002 (12)	0.0007 (12)	−0.0116 (12)
C15	0.0303 (16)	0.0347 (16)	0.0356 (16)	0.0043 (13)	−0.0066 (13)	−0.0164 (13)
C16	0.0411 (18)	0.0348 (16)	0.0232 (14)	0.0035 (14)	−0.0013 (13)	−0.0153 (13)
C17	0.0363 (18)	0.0410 (17)	0.0296 (15)	−0.0027 (14)	0.0079 (14)	−0.0180 (14)
C18	0.0264 (16)	0.058 (2)	0.0475 (19)	−0.0090 (15)	0.0015 (15)	−0.0297 (16)
C19	0.0385 (18)	0.0504 (18)	0.0347 (16)	−0.0089 (15)	0.0001 (14)	−0.0278 (14)
C20	0.0292 (16)	0.0397 (17)	0.0277 (15)	−0.0002 (14)	0.0016 (13)	−0.0166 (13)
C21	0.0268 (15)	0.0351 (15)	0.0258 (14)	−0.0024 (13)	0.0055 (12)	−0.0171 (12)
C22	0.0376 (18)	0.0427 (17)	0.0359 (16)	−0.0009 (15)	−0.0046 (14)	−0.0242 (14)
C23	0.049 (2)	0.0305 (16)	0.0469 (19)	−0.0009 (15)	−0.0002 (16)	−0.0221 (14)
C24	0.0401 (19)	0.0307 (16)	0.0395 (18)	0.0051 (14)	−0.0032 (15)	−0.0141 (14)
C25	0.0321 (16)	0.0363 (16)	0.0290 (15)	−0.0035 (14)	−0.0029 (13)	−0.0136 (13)
C26	0.0384 (18)	0.0300 (15)	0.0278 (15)	−0.0020 (13)	−0.0008 (13)	−0.0154 (12)
O8	0.086 (4)	0.127 (5)	0.137 (5)	−0.018 (4)	−0.024 (4)	−0.089 (4)

Geometric parameters (Å, °)

Pb1—O1	2.472 (2)	C5—H5	0.9300
Pb1—N1	2.551 (2)	C6—C7	1.450 (5)
Pb1—N2	2.585 (3)	C6—H6	0.9300
Pb1—O2	2.630 (2)	C7—C8	1.402 (5)
Pb1—O5	2.720 (2)	C7—C11	1.411 (5)
Pb1—O6	2.815 (2)	C8—C9	1.360 (5)
Pb1—O6^{i}	2.880 (2)	C8—H8A	0.9300
Pb1—O7	2.914 (4)	C9—C10	1.402 (5)
Pb1—C13	2.922 (3)	C9—H9	0.9300

Pb1—C20	3.133 (3)	C10—H10	0.9300
Pb1—Pb1^{i}	4.1712 (8)	C11—C12	1.445 (4)
O1—C13	1.272 (4)	C13—C14	1.507 (4)
O2—C13	1.258 (4)	C14—C19	1.387 (4)
O3—C16	1.369 (4)	C14—C15	1.388 (5)
O3—H3	0.82	C15—C16	1.383 (4)
O4—C25	1.368 (4)	C15—H15	0.9300
O4—H4	0.82	C16—C17	1.386 (5)
O5—C20	1.250 (4)	C17—C18	1.378 (5)
O6—C20	1.268 (4)	C17—H17	0.9300
O6—Pb1^{i}	2.880 (2)	C18—C19	1.379 (5)
O7—H2W	0.83	C18—H18	0.9300
O7—H1W	0.83	C19—H19	0.9300
N1—C1	1.327 (4)	C20—C21	1.505 (4)
N1—C12	1.365 (4)	C21—C22	1.396 (5)
N2—C10	1.341 (4)	C21—C26	1.396 (4)
N2—C11	1.362 (4)	C22—C23	1.380 (4)
C1—C2	1.393 (5)	C22—H22	0.9300
C1—H1	0.9300	C23—C24	1.377 (5)
C2—C3	1.372 (6)	C23—H23	0.9300
C2—H2	0.9300	C24—C25	1.390 (5)
C3—C4	1.405 (5)	C24—H24	0.9300
C3—H3A	0.9300	C25—C26	1.387 (4)
C4—C12	1.409 (4)	C26—H26	0.9300
C4—C5	1.427 (5)	O8—H4W	0.83
C5—C6	1.344 (6)	O8—H3W	0.83
O1—Pb1—N1	81.43 (8)	C2—C3—H3A	119.7
O1—Pb1—N2	78.87 (8)	C4—C3—H3A	119.7
N1—Pb1—N2	64.53 (8)	C3—C4—C12	117.3 (3)
O1—Pb1—O2	50.98 (7)	C3—C4—C5	123.2 (3)
N1—Pb1—O2	78.90 (8)	C12—C4—C5	119.4 (3)
N2—Pb1—O2	121.76 (8)	C6—C5—C4	121.3 (3)
O1—Pb1—O5	76.29 (8)	C6—C5—H5	119.4
N1—Pb1—O5	157.21 (8)	C4—C5—H5	119.4
N2—Pb1—O5	114.84 (8)	C5—C6—C7	121.3 (4)
O2—Pb1—O5	83.37 (7)	C5—C6—H6	119.3
O1—Pb1—O6	95.43 (7)	C7—C6—H6	119.3
N1—Pb1—O6	141.71 (8)	C8—C7—C11	117.6 (3)
N2—Pb1—O6	77.37 (7)	C8—C7—C6	123.7 (3)
O2—Pb1—O6	127.92 (7)	C11—C7—C6	118.7 (3)
O5—Pb1—O6	47.03 (7)	C9—C8—C7	120.5 (3)
O1—Pb1—O6^{i}	154.52 (8)	C9—C8—H8A	119.8
N1—Pb1—O6^{i}	81.97 (7)	C7—C8—H8A	119.8
N2—Pb1—O6^{i}	76.57 (8)	C8—C9—C10	118.7 (3)
O2—Pb1—O6^{i}	142.57 (7)	C8—C9—H9	120.6
O5—Pb1—O6^{i}	120.61 (7)	C10—C9—H9	120.6
O6—Pb1—O6^{i}	85.82 (7)	N2—C10—C9	122.8 (3)

O1—Pb1—O7	121.36 (10)	N2—C10—H10	118.6
N1—Pb1—O7	82.17 (10)	C9—C10—H10	118.6
N2—Pb1—O7	138.64 (12)	N2—C11—C7	122.0 (3)
O2—Pb1—O7	70.71 (11)	N2—C11—C12	118.5 (3)
O5—Pb1—O7	105.38 (11)	C7—C11—C12	119.5 (3)
O6—Pb1—O7	129.11 (9)	N1—C12—C4	122.0 (3)
O6^{i}—Pb1—O7	75.04 (11)	N1—C12—C11	118.3 (3)
O1—Pb1—C13	25.58 (8)	C4—C12—C11	119.7 (3)
N1—Pb1—C13	80.73 (8)	O2—C13—O1	120.8 (3)
N2—Pb1—C13	101.53 (9)	O2—C13—C14	119.6 (3)
O2—Pb1—C13	25.51 (8)	O1—C13—C14	119.6 (3)
O5—Pb1—C13	77.12 (8)	O2—C13—Pb1	64.14 (16)
O6—Pb1—C13	111.91 (8)	O1—C13—Pb1	57.02 (15)
O6^{i}—Pb1—C13	161.54 (8)	C14—C13—Pb1	172.4 (2)
O7—Pb1—C13	96.18 (11)	C19—C14—C15	119.5 (3)
O1—Pb1—C20	87.16 (8)	C19—C14—C13	120.5 (3)
N1—Pb1—C20	160.33 (9)	C15—C14—C13	120.0 (3)
N2—Pb1—C20	97.64 (8)	C16—C15—C14	120.6 (3)
O2—Pb1—C20	106.12 (8)	C16—C15—H15	119.7
O5—Pb1—C20	23.32 (8)	C14—C15—H15	119.7
O6—Pb1—C20	23.85 (8)	O3—C16—C15	118.0 (3)
O6^{i}—Pb1—C20	102.71 (7)	O3—C16—C17	122.0 (3)
O7—Pb1—C20	117.50 (11)	C15—C16—C17	120.0 (3)
C13—Pb1—C20	95.75 (8)	C18—C17—C16	119.0 (3)
O1—Pb1—Pb1^{i}	133.40 (5)	C18—C17—H17	120.5
N1—Pb1—Pb1^{i}	115.68 (6)	C16—C17—H17	120.5
N2—Pb1—Pb1^{i}	72.07 (5)	C17—C18—C19	121.6 (3)
O2—Pb1—Pb1^{i}	164.35 (5)	C17—C18—H18	119.2
O5—Pb1—Pb1^{i}	83.78 (5)	C19—C18—H18	119.2
O6—Pb1—Pb1^{i}	43.51 (5)	C18—C19—C14	119.3 (3)
O6^{i}—Pb1—Pb1^{i}	42.31 (4)	C18—C19—H19	120.3
O7—Pb1—Pb1^{i}	104.33 (9)	C14—C19—H19	120.3
C13—Pb1—Pb1^{i}	155.04 (6)	O5—C20—O6	122.8 (3)
C20—Pb1—Pb1^{i}	62.27 (6)	O5—C20—C21	118.3 (3)
C13—O1—Pb1	97.40 (19)	O6—C20—C21	118.9 (3)
C13—O2—Pb1	90.36 (19)	O5—C20—Pb1	59.49 (17)
C16—O3—H3	109.5	O6—C20—Pb1	63.89 (16)
C25—O4—H4	109.5	C21—C20—Pb1	171.4 (2)
C20—O5—Pb1	97.2 (2)	C22—C21—C26	119.4 (3)
C20—O6—Pb1	92.26 (18)	C22—C21—C20	119.4 (3)
C20—O6—Pb1^{i}	134.9 (2)	C26—C21—C20	121.2 (3)
Pb1—O6—Pb1^{i}	94.18 (7)	C23—C22—C21	120.2 (3)
Pb1—O7—H2W	97.5	C23—C22—H22	119.9
Pb1—O7—H1W	147.1	C21—C22—H22	119.9
H2W—O7—H1W	110.3	C24—C23—C22	120.3 (3)
C1—N1—C12	118.2 (3)	C24—C23—H23	119.9

C1—N1—Pb1	121.6 (2)	C22—C23—H23	119.9
C12—N1—Pb1	119.89 (19)	C23—C24—C25	120.2 (3)
C10—N2—C11	118.3 (3)	C23—C24—H24	119.9
C10—N2—Pb1	122.9 (2)	C25—C24—H24	119.9
C11—N2—Pb1	118.7 (2)	O4—C25—C26	122.7 (3)
N1—C1—C2	123.9 (3)	O4—C25—C24	117.5 (3)
N1—C1—H1	118.1	C26—C25—C24	119.9 (3)
C2—C1—H1	118.1	C25—C26—C21	119.9 (3)
C3—C2—C1	118.0 (3)	C25—C26—H26	120.0
C3—C2—H2	121.0	C21—C26—H26	120.0
C1—C2—H2	121.0	H4W—O8—H3W	111.2
C2—C3—C4	120.6 (3)		
O2—Pb1—O1—C13	−3.8 (2)	O5—Pb1—O6^{i}—Pb1^{i}	33.16 (11)
O5—Pb1—O1—C13	88.8 (2)	O5—Pb1—O6^{i}—C20^{i}	130.5 (3)
O6—Pb1—O1—C13	132.1 (2)	O6—Pb1—O6^{i}—Pb1^{i}	0.00 (8)
O7—Pb1—O1—C13	−11.1 (3)	O6—Pb1—O6^{i}—C20^{i}	97.3 (3)
N1—Pb1—O1—C13	−86.4 (2)	O7—Pb1—O6^{i}—Pb1^{i}	132.41 (12)
N2—Pb1—O1—C13	−152.0 (2)	O7—Pb1—O6^{i}—C20^{i}	−130.3 (3)
O6^{i}—Pb1—O1—C13	−136.3 (2)	N1—Pb1—O6^{i}—Pb1^{i}	−143.59 (9)
O1—Pb1—O2—C13	3.8 (2)	N1—Pb1—O6^{i}—C20^{i}	−46.3 (3)
O5—Pb1—O2—C13	−73.9 (2)	N2—Pb1—O6^{i}—Pb1^{i}	−77.99 (8)
O6—Pb1—O2—C13	−57.7 (2)	N2—Pb1—O6^{i}—C20^{i}	19.3 (3)
O7—Pb1—O2—C13	177.2 (2)	Pb1—O1—C13—O2	7.2 (4)
N1—Pb1—O2—C13	91.7 (2)	Pb1—O1—C13—C14	−172.1 (3)
N2—Pb1—O2—C13	41.3 (2)	Pb1—O2—C13—O1	−6.7 (4)
O6^{i}—Pb1—O2—C13	152.4 (2)	Pb1—O2—C13—C14	172.6 (3)
O1—Pb1—O5—C20	115.9 (2)	Pb1—O5—C20—O6	−9.3 (4)
O2—Pb1—O5—C20	167.3 (2)	Pb1—O5—C20—C21	170.4 (3)
O6—Pb1—O5—C20	4.8 (2)	Pb1—O6—C20—O5	8.9 (4)
O7—Pb1—O5—C20	−124.9 (2)	Pb1—O6—C20—C21	−170.8 (3)
N1—Pb1—O5—C20	128.3 (2)	Pb1^{i}—O6—C20—O5	107.1 (4)
N2—Pb1—O5—C20	45.3 (2)	Pb1^{i}—O6—C20—C21	−72.7 (4)
C13—Pb1—O5—C20	142.2 (2)	Pb1—N1—C1—C2	174.3 (3)
O6^{i}—Pb1—O5—C20	−43.4 (2)	C12—N1—C1—C2	−0.1 (5)
O1—Pb1—O6—C20	−70.3 (2)	Pb1—N1—C12—C4	−175.6 (2)
O1—Pb1—O6—Pb1^{i}	154.47 (8)	Pb1—N1—C12—C11	3.6 (4)
O2—Pb1—O6—C20	−27.0 (2)	C1—N1—C12—C4	−1.2 (5)
O2—Pb1—O6—Pb1^{i}	−162.22 (8)	C1—N1—C12—C11	178.1 (3)
O5—Pb1—O6—C20	−4.69 (19)	Pb1—N2—C10—C9	−176.0 (2)
O5—Pb1—O6—Pb1^{i}	−139.97 (12)	C11—N2—C10—C9	1.0 (5)
O7—Pb1—O6—C20	68.4 (2)	Pb1—N2—C11—C7	177.5 (2)
O7—Pb1—O6—Pb1^{i}	−66.84 (15)	Pb1—N2—C11—C12	−1.9 (4)
N1—Pb1—O6—C20	−153.3 (2)	C10—N2—C11—C7	0.4 (5)
N1—Pb1—O6—Pb1^{i}	71.43 (13)	C10—N2—C11—C12	−179.1 (3)
N2—Pb1—O6—C20	−147.6 (2)	N1—C1—C2—C3	0.7 (5)

N2—Pb1—O6—Pb1^{i}	77.17 (8)	C1—C2—C3—C4	−0.1 (5)
C13—Pb1—O6—C20	−50.1 (2)	C2—C3—C4—C5	178.7 (3)
C13—Pb1—O6—Pb1^{i}	174.66 (9)	C2—C3—C4—C12	−1.1 (5)
O6^{i}—Pb1—O6—C20	135.3 (2)	C3—C4—C5—C6	178.7 (4)
O6^{i}—Pb1—O6—Pb1^{i}	0.00 (8)	C12—C4—C5—C6	−1.4 (6)
O1—Pb1—N1—C1	100.8 (2)	C3—C4—C12—N1	1.8 (5)
O1—Pb1—N1—C12	−84.9 (2)	C3—C4—C12—C11	−177.5 (3)
O2—Pb1—N1—C1	49.1 (2)	C5—C4—C12—N1	−178.1 (3)
O2—Pb1—N1—C12	−136.6 (2)	C5—C4—C12—C11	2.7 (5)
O5—Pb1—N1—C1	88.7 (3)	C4—C5—C6—C7	−1.3 (6)
O5—Pb1—N1—C12	−97.1 (3)	C5—C6—C7—C8	−177.2 (4)
O6—Pb1—N1—C1	−171.3 (2)	C5—C6—C7—C11	2.7 (6)
O6—Pb1—N1—C12	3.0 (3)	C6—C7—C8—C9	−180.0 (3)
O7—Pb1—N1—C1	−22.7 (3)	C11—C7—C8—C9	0.1 (5)
O7—Pb1—N1—C12	151.6 (2)	C6—C7—C11—N2	179.2 (3)
N2—Pb1—N1—C1	−177.5 (3)	C6—C7—C11—C12	−1.4 (5)
N2—Pb1—N1—C12	−3.2 (2)	C8—C7—C11—N2	−0.9 (5)
C13—Pb1—N1—C1	75.0 (2)	C8—C7—C11—C12	178.6 (3)
C13—Pb1—N1—C12	−110.8 (2)	C7—C8—C9—C10	1.2 (5)
O6^{i}—Pb1—N1—C1	−98.6 (2)	C8—C9—C10—N2	−1.8 (5)
O6^{i}—Pb1—N1—C12	75.7 (2)	N2—C11—C12—N1	−1.1 (5)
O1—Pb1—N2—C10	−94.7 (2)	N2—C11—C12—C4	178.2 (3)
O1—Pb1—N2—C11	88.3 (2)	C7—C11—C12—N1	179.5 (3)
O2—Pb1—N2—C10	−123.5 (2)	C7—C11—C12—C4	−1.3 (5)
O2—Pb1—N2—C11	59.5 (2)	O1—C13—C14—C15	−175.4 (3)
O5—Pb1—N2—C10	−25.6 (3)	O1—C13—C14—C19	6.8 (5)
O5—Pb1—N2—C11	157.4 (2)	O2—C13—C14—C15	5.3 (5)
O6—Pb1—N2—C10	3.5 (2)	O2—C13—C14—C19	−172.5 (3)
O6—Pb1—N2—C11	−173.5 (2)	C13—C14—C15—C16	−178.4 (3)
O7—Pb1—N2—C10	139.9 (3)	C19—C14—C15—C16	−0.5 (6)
O7—Pb1—N2—C11	−37.1 (3)	C13—C14—C19—C18	177.4 (3)
N1—Pb1—N2—C10	179.6 (3)	C15—C14—C19—C18	−0.5 (6)
N1—Pb1—N2—C11	2.6 (2)	C14—C15—C16—O3	−179.2 (4)
C13—Pb1—N2—C10	−106.6 (2)	C14—C15—C16—C17	1.1 (6)
C13—Pb1—N2—C11	76.4 (2)	O3—C16—C17—C18	179.6 (4)
O6^{i}—Pb1—N2—C10	92.2 (2)	C15—C16—C17—C18	−0.7 (6)
O6^{i}—Pb1—N2—C11	−84.8 (2)	C16—C17—C18—C19	−0.3 (6)
O1—Pb1—C13—O2	−173.1 (4)	C17—C18—C19—C14	0.9 (6)
O2—Pb1—C13—O1	173.1 (4)	O5—C20—C21—C22	−31.7 (5)
O5—Pb1—C13—O1	−85.1 (2)	O5—C20—C21—C26	150.3 (3)
O5—Pb1—C13—O2	101.8 (2)	O6—C20—C21—C22	148.0 (3)
O6—Pb1—C13—O1	−52.8 (2)	O6—C20—C21—C26	−30.0 (5)
O6—Pb1—C13—O2	134.1 (2)	C20—C21—C22—C23	−176.4 (3)
O7—Pb1—C13—O1	170.5 (2)	C26—C21—C22—C23	1.6 (5)
O7—Pb1—C13—O2	−2.6 (2)	C20—C21—C26—C25	179.0 (3)
N1—Pb1—C13—O1	89.5 (2)	C22—C21—C26—C25	1.0 (5)
N1—Pb1—C13—O2	−83.6 (2)	C21—C22—C23—C24	−1.4 (5)

N2—Pb1—C13—O1	28.1 (2)	C22—C23—C24—C25	−1.6 (5)
N2—Pb1—C13—O2	−145.1 (2)	C23—C24—C25—O4	−177.6 (3)
O1—Pb1—O6^{i}—Pb1^{i}	−93.85 (17)	C23—C24—C25—C26	4.3 (5)
O1—Pb1—O6^{i}—C20^{i}	3.5 (4)	O4—C25—C26—C21	178.0 (3)
O2—Pb1—O6^{i}—Pb1^{i}	156.67 (10)	C24—C25—C26—C21	−4.0 (5)
O2—Pb1—O6^{i}—C20^{i}	−106.0 (3)		

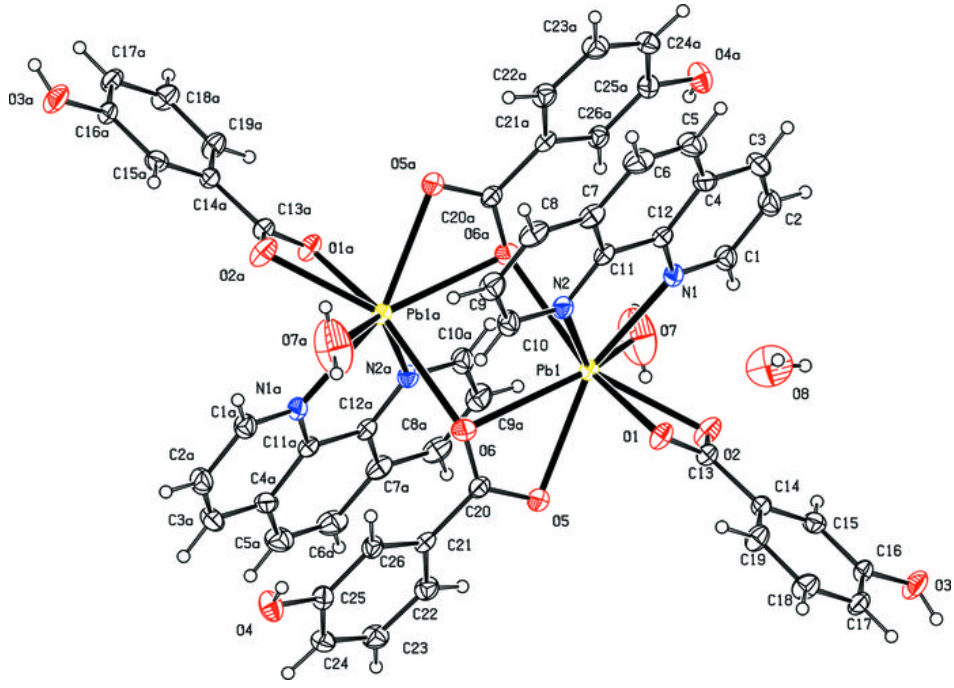
Symmetry codes: (i) −*x*, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O5^{ii}	0.82	1.84	2.658 (3)	176
O4—H4···O1^{iii}	0.82	1.93	2.738 (3)	167
O7—H2W···O8^{iv}	0.83	2.24	2.932 (6)	141
O7—H1W···O3^{v}	0.83	2.18	2.911 (5)	146
O8—H3W···O2^{vi}	0.83	2.06	2.840 (4)	157
O8—H4W···O2^{vii}	0.83	2.04	2.788 (4)	150
C15—H15···O8^{vi}	0.93	2.42	3.339 (5)	169

Symmetry codes: (ii) −*x*+1, −*y*, −*z*; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*−1, *y*−1, *z*; (v) −*x*, −*y*, −*z*; (vi) −*x*+1, −*y*+1, −*z*; (vii) *x*+1, *y*+1, *z*.

Fig. 1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O8
N2a
N1a
C12a
C8a
C9a
O1
C1a
C11a
O6
O2
C13
C2a
C4a
C7a
C14
C3a
C20
O5
C15
C5a
C6a
C26
C21
C19
C16
O3
C25
C18
C22
O4
C17
C24
C23
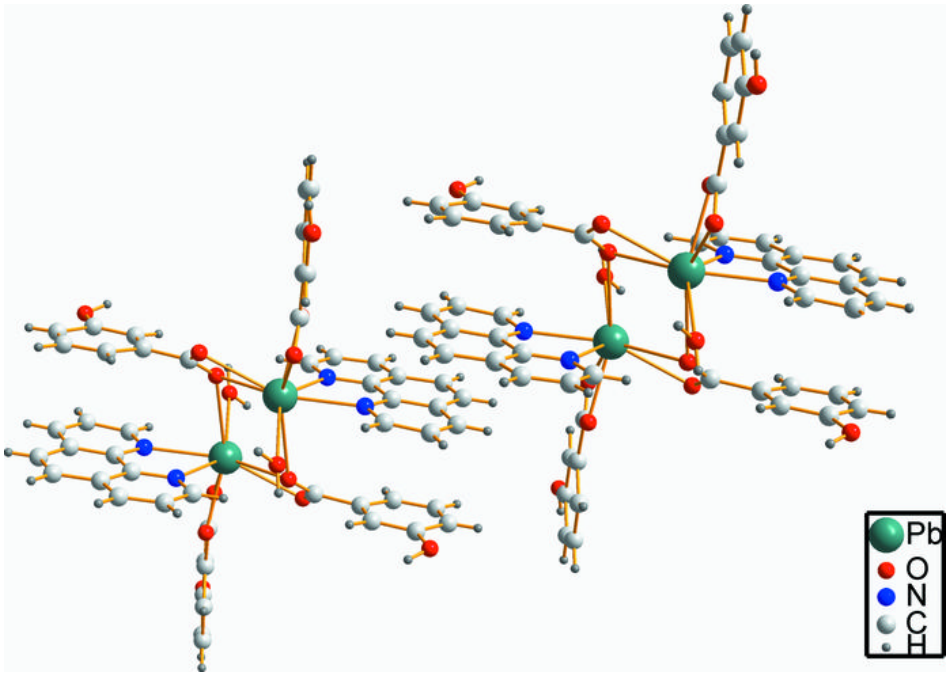
Fig. 2