

Poly[bis(μ_3 -naphthalene-1,4-dicarboxylato)(1,10-phenanthroline)dilead(II)]

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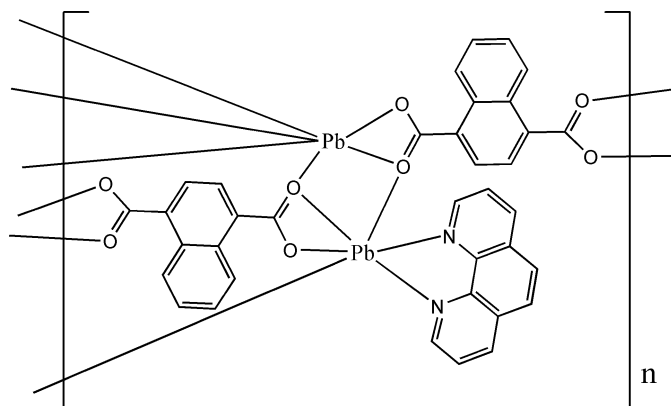
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.024; wR factor = 0.046; data-to-parameter ratio = 15.7.

In the title compound, $[\text{Pb}_2(\text{C}_{12}\text{H}_6\text{O}_4)_2(\text{C}_{12}\text{H}_8\text{N}_2)]_n$ or $[\text{Pb}_2(1,4\text{-NDC})_2(L)]_n$, where 1,4-NDC is naphthalene-1,4-dicarboxylate and L is 1,10-phenanthroline, the 1,4-NDC ligands link the Pb^{II} atoms into a layer structure. One Pb atom is ligated by L and also bonded to four 1,4-NDC O atoms and the other Pb atom bonds only to six 1,4-NDC O atoms. The lone pairs of the Pb atoms appear to be stereochemically active. Aromatic π - π stacking between L and 1,4-NDC ligands in adjacent layers helps to establish the packing [minimum centroid-centroid separation = 3.505 (3) Å].

Related literature

For studies on related lead(II) carboxylates, see Fan & Zhu (2006) and (2007).



Experimental

Crystal data

$[\text{Pb}_2(\text{C}_{12}\text{H}_6\text{O}_4)_2(\text{C}_{12}\text{H}_8\text{N}_2)]$
 $M_r = 1022.92$
Monoclinic, $P2_1$
 $a = 7.063$ (3) Å
 $b = 14.602$ (5) Å
 $c = 14.526$ (5) Å
 $\beta = 94.922$ (5)°

$V = 1492.6$ (10) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 11.33$ mm⁻¹
 $T = 293$ (2) K
 $0.29 \times 0.15 \times 0.09$ mm

Data collection

Rigaku R-Axis RAPID diffractometer
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\text{min}} = 0.141$, $T_{\text{max}} = 0.368$

14649 measured reflections
6786 independent reflections
6429 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.046$
 $S = 0.91$
6786 reflections
433 parameters
1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.60$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.06$ e Å⁻³
Absolute structure: Flack (1983), 3232 Friedel pairs
Flack parameter: -0.011 (5)

Table 1

Selected bond lengths (Å).

Pb2—N1	2.622 (4)	Pb1—O2	2.501 (4)
Pb2—N2	2.701 (4)	Pb1—O3	2.405 (3)
Pb2—O1	2.756 (4)	Pb1—O4	2.633 (3)
Pb2—O2	2.661 (4)	Pb1—O5 ⁱ	2.488 (4)
Pb2—O3	2.600 (4)	Pb1—O7 ⁱⁱ	2.588 (4)
Pb2—O6 ⁱ	2.464 (4)	Pb1—O8 ⁱⁱ	2.577 (5)

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2579).

References

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supplementary materials

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Poly[bis(μ_3 -naphthalene-1,4-dicarboxylato)(1,10-phenanthroline)dilead(II)]

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Comment

Lead(II), with its large radius, flexible coordination environment, and variable stereochemical activity, provides unique opportunities for formation of unusual coordination polymer network topologies with interesting properties (Fan & Zhu, 2006; 2007). Herein, we present a new Pb(II) coordination polymer, namely the title compound, (I), $[\text{Pb}_2(1,4\text{-NDC})_2(L)]$, where $L = 1,10\text{-phenanthroline}$ and $1,4\text{-NDC} = \text{naphthalene-1,4-dicarboxylate}$.

Selected bond lengths and angles are listed in Table 1. In compound (I) there exist two 1,4-NDC dianions, one L ligand and two crystallographically independent Pb(II) atoms (Fig. 1). Pb1 is six-coordinated by six carboxylate O atoms from four 1,4-NDC ligands, and its lone pair of electrons appears to be stereochemically active. Atom Pb2 is also six-coordinated, by two N atoms from one chelating L ligand, and four carboxylate O atoms from three 1,4-NDC ligands, while the lone pair of electrons completes its environment. The 1,4-NDC dianions link neighboring Pb(II) atoms to generate a layer structure and the L ligands are located on both sides of the layers (Fig. 2). Moreover, the neighboring layers interact through π - π forces between L and 1,4-NDC ligands, forming a three-dimensional supramolecular structure.

Experimental

A mixture of $\text{Pb}(\text{NO}_3)_2$ (1 mmol), 1,4- H_2NDC (1 mmol) and L (1 mmol) were dissolved in 15 ml distilled water, followed by addition of triethylamine until the pH value of the system was adjusted to about 6. The resulting solution was sealed in a 23-ml Teflon-lined stainless steel autoclave and heated at 453 K for 7 days under autogenous pressure. Afterwards, the reaction system was slowly cooled to room temperature. Colourless slabs and plates of (I) were collected.

Refinement

All the H atoms were generated geometrically ($\text{C}-\text{H} = 0.93 \text{ \AA}$) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

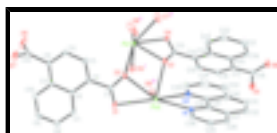


Fig. 1. The asymmetric unit of (I) expanded to show the lead coordination spheres, with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level. H atoms omitted for clarity. Symmetry codes: (i) $1 - x, y - 1/2, 1 - z$; (ii) $1 - x, y + 1/2, 2 - z$; (iii) $1 - x, y - 1/2, 1 - z$.

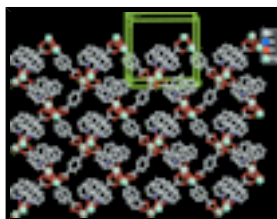


Fig. 2. : The extended layer structure of (I). The hydrogen atoms are omitted for clarity.

(1E,4E)-1,5-Bis(2-chlorophenyl)penta-1,4-dien-3-one

Crystal data

$[\text{Pb}_2(\text{C}_{12}\text{H}_6\text{O}_4)_2(\text{C}_{12}\text{H}_8\text{N}_2)]$	$F_{000} = 956$
$M_r = 1022.92$	$D_x = 2.276 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
Hall symbol: P 2yb	$\lambda = 0.71073 \text{ \AA}$
$a = 7.063 (3) \text{ \AA}$	Cell parameters from 14193 reflections
$b = 14.602 (5) \text{ \AA}$	$\theta = 3.0\text{--}27.5^\circ$
$c = 14.526 (5) \text{ \AA}$	$\mu = 11.33 \text{ mm}^{-1}$
$\beta = 94.922 (5)^\circ$	$T = 293 (2) \text{ K}$
$V = 1492.6 (10) \text{ \AA}^3$	Slab, colourless
$Z = 2$	$0.29 \times 0.15 \times 0.09 \text{ mm}$

Data collection

Rigaku R-Axis RAPID diffractometer	6786 independent reflections
Radiation source: rotating anode	6429 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.036$
Detector resolution: $10.0 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 293(2) \text{ K}$	$\theta_{\text{min}} = 3.1^\circ$
ω scans	$h = -9 \rightarrow 7$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -18 \rightarrow 18$
$T_{\text{min}} = 0.141, T_{\text{max}} = 0.368$	$l = -18 \rightarrow 18$
14649 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.024$	$w = 1/[\sigma^2(F_o^2)]$
$wR(F^2) = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.91$	$(\Delta/\sigma)_{\text{max}} = 0.001$
6786 reflections	$\Delta\rho_{\text{max}} = 0.60 \text{ e \AA}^{-3}$
433 parameters	$\Delta\rho_{\text{min}} = -1.06 \text{ e \AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 3232 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: $-0.011 (5)$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8600 (7)	-0.3121 (4)	0.8347 (4)	0.0310 (11)
C2	0.8383 (7)	-0.3959 (3)	0.8940 (4)	0.0262 (11)
C3	0.6962 (7)	-0.3962 (4)	0.9510 (4)	0.0375 (12)
H3	0.6189	-0.3448	0.9524	0.045*
C4	0.6608 (8)	-0.4704 (4)	1.0076 (4)	0.0396 (13)
H4	0.5589	-0.4686	1.0441	0.048*
C5	0.7767 (7)	-0.5462 (3)	1.0094 (4)	0.0339 (12)
C6	0.9682 (7)	-0.4729 (3)	0.8950 (3)	0.0254 (10)
C7	1.1281 (7)	-0.4789 (3)	0.8432 (4)	0.0349 (11)
H7	1.1488	-0.4335	0.8004	0.042*
C8	1.2530 (8)	-0.5502 (3)	0.8549 (5)	0.0453 (15)
H8	1.3593	-0.5514	0.8213	0.054*
C9	1.2242 (8)	-0.6206 (4)	0.9159 (4)	0.0447 (14)
H9	1.3119	-0.6681	0.9238	0.054*
C10	1.0669 (8)	-0.6206 (3)	0.9645 (4)	0.0435 (13)
H10	1.0460	-0.6695	1.0033	0.052*
C13	0.3703 (7)	-0.1061 (3)	0.6647 (4)	0.0242 (10)
C14	0.3748 (6)	-0.0248 (3)	0.6024 (3)	0.0212 (9)
C15	0.4044 (6)	-0.0375 (3)	0.5120 (4)	0.0289 (10)
H16	0.4243	-0.0962	0.4900	0.035*
C16	0.4051 (7)	0.0378 (3)	0.4518 (4)	0.0305 (11)
H17	0.4309	0.0285	0.3908	0.037*
C17	0.3689 (6)	0.1242 (3)	0.4810 (4)	0.0273 (10)
C20	0.3402 (6)	0.1398 (3)	0.5757 (3)	0.0242 (10)
C24	0.3427 (6)	0.0644 (3)	0.6368 (4)	0.0251 (10)
C23	0.3107 (7)	0.0790 (3)	0.7304 (4)	0.0320 (11)
H23	0.3109	0.0291	0.7702	0.038*
C19	0.3103 (7)	0.2291 (3)	0.6133 (4)	0.0351 (12)
H13	0.3100	0.2802	0.5751	0.042*
C21	0.2826 (8)	0.2399 (4)	0.7039 (4)	0.0438 (13)
H21	0.2652	0.2985	0.7268	0.053*
C31	0.7904 (8)	0.2644 (4)	0.6450 (6)	0.056 (2)
H31	0.7716	0.3260	0.6578	0.067*

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C32	0.8171 (8)	0.2400 (4)	0.5597 (5)	0.0478 (15)
H32	0.8174	0.2846	0.5140	0.057*
C27	0.8455 (7)	0.1461 (4)	0.5359 (5)	0.0411 (14)
C26	0.8780 (7)	0.1184 (4)	0.4484 (4)	0.0460 (14)
H26	0.8808	0.1618	0.4017	0.055*
C28	0.8420 (6)	0.0786 (3)	0.6056 (4)	0.0327 (12)
C29	0.8154 (7)	0.1041 (3)	0.6991 (4)	0.0313 (11)
C35	0.7991 (8)	0.0630 (4)	0.8511 (4)	0.0467 (14)
H35	0.8008	0.0178	0.8963	0.056*
C36	0.8966 (7)	-0.0346 (4)	0.4998 (4)	0.0423 (13)
H36	0.9139	-0.0962	0.4864	0.051*
C18	0.3599 (8)	0.1990 (3)	0.4078 (4)	0.0370 (12)
C30	0.7893 (8)	0.1983 (4)	0.7199 (5)	0.0459 (16)
C12	0.7355 (8)	-0.6235 (3)	1.0724 (4)	0.0381 (12)
C22	0.2796 (8)	0.1646 (4)	0.7639 (4)	0.0425 (13)
H22	0.2568	0.1729	0.8255	0.051*
C34	0.7771 (10)	0.1539 (5)	0.8771 (5)	0.0610 (19)
H34	0.7673	0.1689	0.9388	0.073*
C11	0.9371 (7)	-0.5482 (3)	0.9567 (4)	0.0280 (11)
C25	0.9060 (8)	0.0290 (5)	0.4290 (5)	0.0552 (17)
H25	0.9309	0.0105	0.3700	0.066*
C33	0.7700 (10)	0.2208 (4)	0.8114 (6)	0.0587 (19)
H33	0.7522	0.2815	0.8279	0.070*
N1	0.8641 (5)	-0.0117 (3)	0.5859 (3)	0.0305 (9)
N2	0.8177 (6)	0.0382 (3)	0.7636 (3)	0.0348 (10)
O1	0.9883 (6)	-0.3066 (3)	0.7806 (4)	0.0512 (12)
O2	0.7379 (5)	-0.2472 (2)	0.8418 (3)	0.0323 (8)
O3	0.5033 (4)	-0.1186 (2)	0.7271 (3)	0.0312 (8)
O4	0.2304 (4)	-0.1607 (2)	0.6549 (2)	0.0297 (8)
O5	0.4507 (6)	0.1824 (3)	0.3387 (3)	0.0577 (12)
O6	0.2628 (6)	0.2688 (2)	0.4181 (3)	0.0498 (11)
O7	0.8192 (7)	-0.6248 (3)	1.1507 (3)	0.0593 (12)
O8	0.6205 (9)	-0.6829 (4)	1.0446 (4)	0.102 (2)
Pb1	0.38951 (3)	-0.256319 (9)	0.794522 (12)	0.02560 (5)
Pb2	0.86603 (2)	-0.137997 (11)	0.714247 (12)	0.02589 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.028 (2)	0.036 (3)	0.028 (3)	-0.005 (2)	0.002 (2)	0.008 (2)
C2	0.028 (2)	0.026 (2)	0.025 (3)	-0.0022 (19)	0.002 (2)	0.008 (2)
C3	0.044 (3)	0.041 (3)	0.028 (3)	0.013 (2)	0.007 (2)	0.011 (2)
C4	0.040 (3)	0.051 (3)	0.030 (3)	0.004 (2)	0.014 (2)	0.011 (3)
C5	0.043 (3)	0.039 (3)	0.019 (3)	-0.003 (2)	-0.003 (2)	0.010 (2)
C6	0.030 (2)	0.024 (2)	0.022 (3)	0.0012 (18)	-0.001 (2)	0.0037 (19)
C7	0.044 (3)	0.028 (2)	0.035 (3)	-0.002 (2)	0.014 (2)	0.003 (2)
C8	0.046 (3)	0.035 (3)	0.057 (4)	0.003 (2)	0.013 (3)	-0.005 (3)
C9	0.050 (3)	0.038 (3)	0.045 (4)	0.017 (2)	0.000 (3)	0.000 (3)

C10	0.059 (3)	0.034 (3)	0.036 (3)	0.007 (2)	-0.003 (3)	0.008 (2)
C13	0.030 (2)	0.020 (2)	0.024 (3)	0.0000 (18)	0.011 (2)	0.0026 (19)
C14	0.0170 (19)	0.022 (2)	0.025 (2)	-0.0028 (16)	0.0033 (18)	-0.0021 (19)
C15	0.029 (2)	0.028 (2)	0.029 (3)	0.0007 (19)	0.002 (2)	-0.002 (2)
C16	0.032 (2)	0.035 (2)	0.025 (3)	0.002 (2)	0.005 (2)	0.005 (2)
C17	0.023 (2)	0.026 (2)	0.032 (3)	-0.0041 (18)	0.004 (2)	0.009 (2)
C20	0.023 (2)	0.026 (2)	0.023 (2)	-0.0007 (17)	0.0015 (19)	0.0041 (19)
C24	0.022 (2)	0.026 (2)	0.028 (3)	-0.0006 (18)	0.002 (2)	0.001 (2)
C23	0.037 (3)	0.032 (2)	0.028 (3)	0.003 (2)	0.011 (2)	0.008 (2)
C19	0.040 (3)	0.021 (2)	0.044 (3)	0.0028 (19)	0.002 (2)	0.008 (2)
C21	0.055 (3)	0.029 (2)	0.047 (3)	0.008 (3)	0.004 (3)	-0.008 (3)
C31	0.046 (3)	0.026 (3)	0.093 (6)	0.000 (2)	-0.012 (4)	0.015 (3)
C32	0.040 (3)	0.033 (3)	0.069 (5)	-0.001 (2)	-0.006 (3)	0.023 (3)
C27	0.019 (2)	0.041 (3)	0.063 (4)	0.001 (2)	-0.001 (3)	0.020 (3)
C26	0.033 (3)	0.063 (4)	0.042 (4)	0.004 (3)	0.007 (3)	0.029 (3)
C28	0.022 (2)	0.033 (2)	0.043 (3)	0.0004 (19)	0.000 (2)	0.008 (2)
C29	0.027 (2)	0.022 (2)	0.044 (3)	-0.0011 (18)	-0.004 (2)	0.004 (2)
C35	0.051 (3)	0.055 (3)	0.033 (3)	0.001 (3)	0.001 (3)	-0.001 (3)
C36	0.035 (3)	0.054 (3)	0.038 (3)	0.005 (2)	0.008 (2)	0.006 (3)
C18	0.047 (3)	0.034 (3)	0.030 (3)	-0.012 (2)	0.001 (3)	0.008 (2)
C30	0.036 (3)	0.031 (3)	0.068 (5)	0.003 (2)	-0.010 (3)	-0.001 (3)
C12	0.045 (3)	0.033 (3)	0.037 (3)	-0.001 (2)	0.006 (2)	0.012 (2)
C22	0.057 (3)	0.041 (3)	0.030 (3)	0.003 (3)	0.008 (3)	-0.005 (3)
C34	0.062 (4)	0.069 (4)	0.049 (4)	0.010 (3)	-0.011 (3)	-0.023 (4)
C11	0.036 (3)	0.024 (2)	0.023 (3)	-0.003 (2)	-0.002 (2)	0.008 (2)
C25	0.039 (3)	0.090 (5)	0.038 (4)	0.011 (3)	0.013 (3)	0.015 (4)
C33	0.065 (4)	0.039 (3)	0.069 (5)	0.005 (3)	-0.012 (4)	-0.017 (3)
N1	0.0249 (19)	0.028 (2)	0.038 (3)	0.0016 (16)	-0.0030 (19)	0.0051 (19)
N2	0.036 (2)	0.033 (2)	0.035 (3)	0.0005 (17)	-0.003 (2)	0.002 (2)
O1	0.052 (2)	0.039 (2)	0.066 (3)	0.0082 (19)	0.027 (2)	0.028 (2)
O2	0.0352 (17)	0.0233 (16)	0.038 (2)	0.0018 (14)	-0.0003 (15)	0.0073 (16)
O3	0.0340 (17)	0.0242 (16)	0.034 (2)	-0.0008 (13)	-0.0019 (15)	0.0047 (15)
O4	0.0290 (17)	0.0328 (18)	0.0279 (19)	-0.0069 (13)	0.0045 (15)	0.0033 (15)
O5	0.081 (3)	0.050 (2)	0.046 (3)	0.002 (2)	0.027 (3)	0.022 (2)
O6	0.069 (3)	0.034 (2)	0.046 (3)	0.0107 (19)	0.000 (2)	0.0143 (18)
O7	0.090 (3)	0.052 (3)	0.034 (2)	-0.015 (2)	-0.007 (2)	0.014 (2)
O8	0.121 (5)	0.100 (4)	0.076 (4)	-0.071 (4)	-0.050 (4)	0.056 (3)
Pb1	0.03473 (10)	0.02015 (8)	0.02271 (9)	0.00014 (7)	0.00705 (7)	0.00040 (8)
Pb2	0.02927 (9)	0.02153 (8)	0.02683 (10)	-0.00054 (7)	0.00221 (7)	0.00271 (8)

Geometric parameters (Å, °)

C1—O1	1.252 (7)	C31—C32	1.319 (10)
C1—O2	1.291 (6)	C31—C30	1.454 (9)
C1—C2	1.512 (7)	C31—H31	0.9300
C2—C3	1.355 (7)	C32—C27	1.432 (8)
C2—C6	1.450 (7)	C32—H32	0.9300
C3—C4	1.396 (7)	C27—C26	1.372 (9)
C3—H3	0.9300	C27—C28	1.413 (7)

supplementary materials

C4—C5	1.375 (7)	C26—C25	1.354 (9)
C4—H4	0.9300	C26—H26	0.9300
C5—C11	1.420 (7)	C28—N1	1.362 (6)
C5—C12	1.498 (7)	C28—C29	1.436 (8)
C6—C7	1.412 (7)	C29—N2	1.341 (7)
C6—C11	1.447 (6)	C29—C30	1.425 (7)
C7—C8	1.365 (7)	C35—N2	1.339 (8)
C7—H7	0.9300	C35—C34	1.392 (8)
C8—C9	1.383 (8)	C35—H35	0.9300
C8—H8	0.9300	C36—N1	1.333 (7)
C9—C10	1.366 (9)	C36—C25	1.391 (9)
C9—H9	0.9300	C36—H36	0.9300
C10—C11	1.397 (7)	C18—O6	1.245 (6)
C10—H10	0.9300	C18—O5	1.261 (7)
C13—O3	1.261 (6)	C30—C33	1.386 (10)
C13—O4	1.268 (6)	C12—O8	1.232 (7)
C13—C14	1.495 (6)	C12—O7	1.236 (7)
C14—C15	1.359 (7)	C22—H22	0.9300
C14—C24	1.420 (6)	C34—C33	1.364 (10)
C15—C16	1.406 (7)	C34—H34	0.9300
C15—H16	0.9300	C25—H25	0.9300
C16—C17	1.362 (7)	C33—H33	0.9300
C16—H17	0.9300	Pb2—N1	2.622 (4)
C17—C20	1.426 (7)	Pb2—N2	2.701 (4)
C17—C18	1.522 (7)	Pb2—O1	2.756 (4)
C20—C24	1.413 (6)	Pb2—O2	2.661 (4)
C20—C19	1.436 (6)	Pb2—O3	2.600 (4)
C24—C23	1.415 (7)	Pb2—O6 ⁱ	2.464 (4)
C23—C22	1.366 (7)	Pb1—O2	2.501 (4)
C23—H23	0.9300	Pb1—O3	2.405 (3)
C19—C21	1.356 (8)	Pb1—O4	2.633 (3)
C19—H13	0.9300	Pb1—O5 ⁱ	2.488 (4)
C21—C22	1.404 (8)	Pb1—O7 ⁱⁱ	2.588 (4)
C21—H21	0.9300	Pb1—O8 ⁱⁱ	2.577 (5)
O1—C1—O2	121.8 (5)	N2—C35—C34	122.5 (6)
O1—C1—C2	121.6 (4)	N2—C35—H35	118.7
O2—C1—C2	116.6 (4)	C34—C35—H35	118.7
C3—C2—C6	119.5 (4)	N1—C36—C25	123.3 (6)
C3—C2—C1	117.9 (4)	N1—C36—H36	118.4
C6—C2—C1	122.6 (4)	C25—C36—H36	118.4
C2—C3—C4	122.8 (5)	O6—C18—O5	125.2 (5)
C2—C3—H3	118.6	O6—C18—C17	119.6 (5)
C4—C3—H3	118.6	O5—C18—C17	115.2 (5)
C5—C4—C3	120.0 (5)	C33—C30—C29	117.2 (6)
C5—C4—H4	120.0	C33—C30—C31	124.6 (6)
C3—C4—H4	120.0	C29—C30—C31	118.2 (6)
C4—C5—C11	120.5 (5)	O8—C12—O7	122.7 (5)
C4—C5—C12	118.1 (5)	O8—C12—C5	119.2 (5)

C11—C5—C12	121.2 (4)	O7—C12—C5	118.1 (5)
C7—C6—C11	117.0 (4)	C23—C22—C21	118.9 (5)
C7—C6—C2	125.2 (4)	C23—C22—H22	120.5
C11—C6—C2	117.8 (4)	C21—C22—H22	120.5
C8—C7—C6	121.2 (5)	C33—C34—C35	119.4 (7)
C8—C7—H7	119.4	C33—C34—H34	120.3
C6—C7—H7	119.4	C35—C34—H34	120.3
C7—C8—C9	121.2 (6)	C10—C11—C5	121.4 (5)
C7—C8—H8	119.4	C10—C11—C6	119.4 (5)
C9—C8—H8	119.4	C5—C11—C6	119.2 (4)
C10—C9—C8	120.0 (5)	C26—C25—C36	118.2 (6)
C10—C9—H9	120.0	C26—C25—H25	120.9
C8—C9—H9	120.0	C36—C25—H25	120.9
C9—C10—C11	121.1 (5)	C34—C33—C30	120.1 (6)
C9—C10—H10	119.5	C34—C33—H33	120.0
C11—C10—H10	119.5	C30—C33—H33	120.0
O3—C13—O4	121.0 (4)	C36—N1—C28	118.2 (5)
O3—C13—C14	119.8 (4)	C36—N1—Pb2	120.0 (3)
O4—C13—C14	119.2 (4)	C28—N1—Pb2	121.7 (4)
C15—C14—C24	120.5 (5)	C35—N2—C29	118.3 (5)
C15—C14—C13	119.3 (4)	C35—N2—Pb2	122.3 (4)
C24—C14—C13	120.2 (4)	C29—N2—Pb2	119.4 (4)
C14—C15—C16	120.2 (4)	C1—O1—Pb2	93.1 (3)
C14—C15—H16	119.9	C1—O2—Pb1	125.9 (3)
C16—C15—H16	119.9	C1—O2—Pb2	96.6 (3)
C17—C16—C15	121.3 (5)	Pb1—O2—Pb2	103.08 (12)
C17—C16—H17	119.3	C13—O3—Pb1	99.2 (3)
C15—C16—H17	119.3	C13—O3—Pb2	130.0 (3)
C16—C17—C20	119.6 (4)	Pb1—O3—Pb2	107.69 (11)
C16—C17—C18	116.4 (5)	C13—O4—Pb1	88.3 (3)
C20—C17—C18	124.0 (4)	C18—O5—Pb1 ⁱⁱⁱ	147.8 (4)
C24—C20—C17	119.1 (4)	C18—O6—Pb2 ⁱⁱⁱ	121.9 (4)
C24—C20—C19	117.6 (5)	C12—O7—Pb1 ^{iv}	93.1 (3)
C17—C20—C19	123.3 (4)	C12—O8—Pb1 ^{iv}	93.7 (4)
C20—C24—C23	119.4 (4)	O3—Pb1—O5 ⁱ	78.12 (14)
C20—C24—C14	119.2 (5)	O3—Pb1—O2	73.26 (11)
C23—C24—C14	121.4 (4)	O5 ⁱ —Pb1—O2	74.42 (14)
C22—C23—C24	121.6 (5)	O3—Pb1—O8 ⁱⁱ	93.29 (19)
C22—C23—H23	119.2	O5 ⁱ —Pb1—O8 ⁱⁱ	154.69 (18)
C24—C23—H23	119.2	O2—Pb1—O8 ⁱⁱ	80.32 (17)
C21—C19—C20	120.8 (5)	O3—Pb1—O7 ⁱⁱ	73.96 (14)
C21—C19—H13	119.6	O5 ⁱ —Pb1—O7 ⁱⁱ	144.51 (15)
C20—C19—H13	119.6	O2—Pb1—O7 ⁱⁱ	116.68 (13)
C19—C21—C22	121.5 (5)	O8 ⁱⁱ —Pb1—O7 ⁱⁱ	49.58 (14)
C19—C21—H21	119.2	O3—Pb1—O4	51.57 (10)
C22—C21—H21	119.2	O5 ⁱ —Pb1—O4	77.52 (14)

supplementary materials

C32—C31—C30	122.3 (6)	O2—Pb1—O4	121.87 (10)
C32—C31—H31	118.8	O8 ⁱⁱ —Pb1—O4	115.74 (14)
C30—C31—H31	118.8	O7 ⁱⁱ —Pb1—O4	68.07 (13)
C31—C32—C27	121.3 (6)	O6 ⁱ —Pb2—O3	79.40 (14)
C31—C32—H32	119.4	O6 ⁱ —Pb2—N1	81.63 (14)
C27—C32—H32	119.4	O3—Pb2—N1	91.69 (11)
C26—C27—C28	118.2 (5)	O6 ⁱ —Pb2—O2	95.01 (13)
C26—C27—C32	122.9 (6)	O3—Pb2—O2	67.62 (10)
C28—C27—C32	118.9 (6)	N1—Pb2—O2	159.28 (11)
C25—C26—C27	121.1 (6)	O6 ⁱ —Pb2—N2	133.35 (14)
C25—C26—H26	119.5	O3—Pb2—N2	74.31 (11)
C27—C26—H26	119.5	N1—Pb2—N2	61.68 (14)
N1—C28—C27	121.0 (5)	O2—Pb2—N2	109.14 (13)
N1—C28—C29	118.4 (5)	O6 ⁱ —Pb2—O1	82.13 (15)
C27—C28—C29	120.6 (5)	O3—Pb2—O1	110.68 (10)
N2—C29—C30	122.5 (6)	N1—Pb2—O1	149.26 (13)
N2—C29—C28	118.8 (4)	O2—Pb2—O1	48.41 (11)
C30—C29—C28	118.7 (5)	N2—Pb2—O1	143.27 (15)

Symmetry codes: (i) $-x+1, y-1/2, -z+1$; (ii) $-x+1, y+1/2, -z+2$; (iii) $-x+1, y+1/2, -z+1$; (iv) $-x+1, y-1/2, -z+2$.

Fig. 1

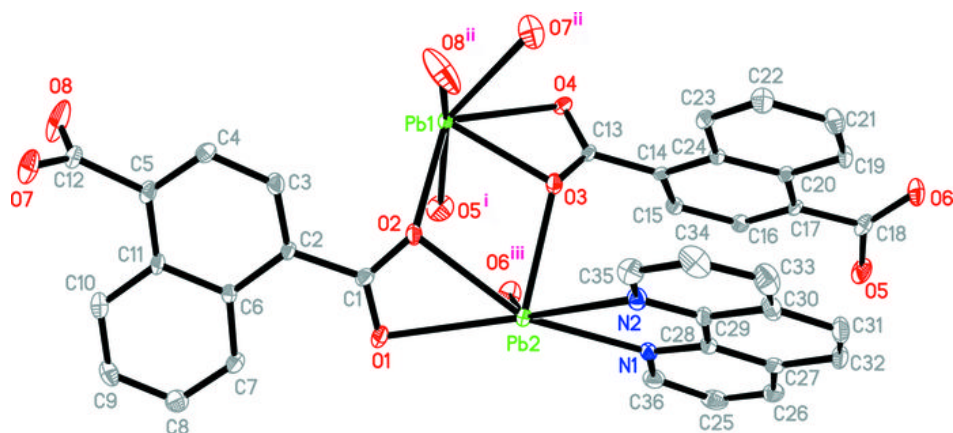


Fig. 2

