

catena-Poly[[*(4,7-diphenyl-1,10-phenanthroline)lead(II)*]- μ -benzene-1,4-dicarboxylato]

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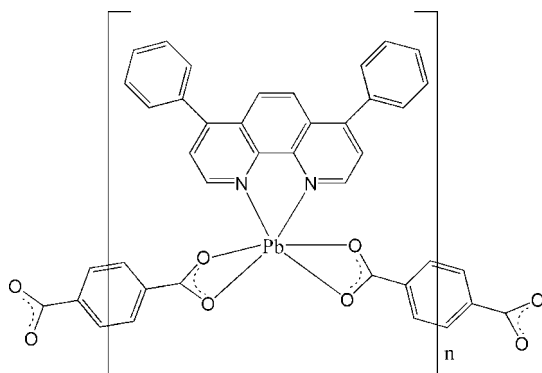
Received 19 October 2007; accepted 19 October 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.026; wR factor = 0.082; data-to-parameter ratio = 16.6.

In the title compound, $[\text{Pb}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{24}\text{H}_{16}\text{N}_2)]_n$, the benzene-1,4-dicarboxylate (1,4-BDC) dianions link the Pb^{II} atoms to form a chain structure. The Pb^{II} atom is coordinated by 4,7-diphenyl-1,10-phenanthroline (*L*) and is also bonded to four 1,4-BDC O atoms, resulting in a very distorted *cis*- PbN_2O_4 octahedron. There are two 1,4-BDC half-anions in the asymmetric unit, both completed by inversion. Aromatic π - π stacking between *L* ligands in adjacent chains results in a two-dimensional supramolecular layer structure [minimum centroid-centroid separation 3.464 (5) Å].

Related literature

For studies on related lead(II) carboxylates, see: Fan & Zhu (2007); Yang *et al.* (2007).



Experimental

Crystal data

$[\text{Pb}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{24}\text{H}_{16}\text{N}_2)]$
 $M_r = 703.69$
 Triclinic, $P\bar{1}$
 $a = 9.855$ (2) Å
 $b = 10.046$ (2) Å
 $c = 13.539$ (3) Å
 $\alpha = 100.36$ (3)°
 $\beta = 101.14$ (3)°

$\gamma = 92.04$ (3)°
 $V = 1290.2$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 6.58$ mm⁻¹
 $T = 293$ (2) K
 $0.27 \times 0.17 \times 0.08$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\text{min}} = 0.275$, $T_{\text{max}} = 0.592$

12741 measured reflections
 5855 independent reflections
 5326 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.082$
 $S = 1.12$
 5855 reflections

352 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.80$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.98$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Pb1—N1	2.686 (4)	Pb1—O2	2.418 (3)
Pb1—N2	2.677 (4)	Pb1—O3	2.506 (4)
Pb1—O1	2.503 (4)	Pb1—O4	2.663 (4)
O2—Pb1—O1	52.76 (11)	N2—Pb1—N1	60.74 (12)
O3—Pb1—O4	50.42 (12)		

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*.

The authors thank Qiqihar University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2600).

References

- Fan, S. R. & Zhu, L.-G. (2007). *Inorg. Chem.* **46**, 6785–6793.
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 Yang, J. Ma, J.-F., Liu, Y.-Y., Ma, J.-C. & Batten, S. R. (2007). *Inorg. Chem.* **46**, 6542–6555.

supplementary materials

Acta Cryst. (2007). E63, m2803 [doi:10.1107/S1600536807051859]

***catena*-Poly[[*(4,7-diphenyl-1,10-phenanthroline)lead(II)- μ -benzene-1,4-dicarboxylato*]**

Y.-B. Yin and H.-X. Yu

Comment

The design and synthesis of coordination polymers is one of the most active areas of materials research. In this regard, many of the efforts have so far been devoted to the study of transition-metal based coordination polymers. However, relatively little attention has been paid to the coordination polymers of main group metal ions despite their interesting network topologies and properties (Fan & Zhu, 2007; Yang *et al.*, 2007). Herein, we present a new Pb(II) coordination polymer, namely the title compound, (I), [Pb(1,4-BDC)(*L*)], where 1,4-BDC = the benzene-1,4-dicarboxylate dianion and *L* = 4,7-diphenyl-1,10-phenanthroline.

Selected bond lengths and angles are listed in Table 1. In compound (I) the Pb^{II} atom is six-coordinated by four carboxylate O atoms from two different 1,4-BDC ligands, and two N atoms from one *L* ligand (Fig. 1). This results in a very distorted *cis*-PbN₂O₄ octahedron (Table 1). The centrosymmetric 1,4-BDC dianions bridge neighboring Pb(II) atoms to form a chain structure and the *L* ligands are attached on both sides of the chains (Fig. 2). In addition, the neighboring chains interact through π - π forces between *L* ligands, leading to a two-dimensional supramolecular layer structure [minimum centroid-centroid separation = 3.464 (5) Å].

Experimental

A mixture of Pb(NO₃)₂ (2 mmol), 1,4-H₂BDC (2 mmol) and *L* (2 mmol) were dissolved in 14 ml distilled water, followed by addition of triethylamine until the pH of the system was adjusted to about 5.8. The resulting solution was sealed in a 23-ml Teflon-lined stainless steel autoclave and heated at 455 K for 6 days under autogenous pressure. Afterwards, the reaction system was slowly cooled to room temperature. Colourless blocks of (I) were collected.

Refinement

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

Figures

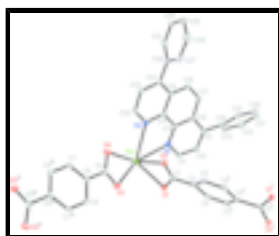


Fig. 1. The asymmetric unit of (I) expanded to show the complete 1,4-BDC molecules, with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level. H atoms omitted for clarity. Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $1 - x, 1 - y, 2 - z$.

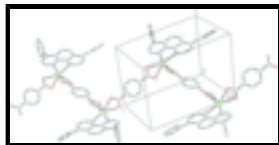


Fig. 2. View of the chain structure of (I). The hydrogen atoms are omitted for clarity.

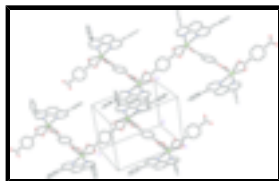


Fig. 3. View of the supramolecular layer structure of (I). The hydrogen atoms are omitted for clarity.

catena-Poly[[4,7-diphenyl-1,10-phenanthroline]lead(II)]- μ -benzene-1,4-dicarboxylato]

Crystal data

[Pb(C₈H₄O₄)(C₂₄H₁₆N₂)]

$M_r = 703.69$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.855$ (2) Å

$b = 10.046$ (2) Å

$c = 13.539$ (3) Å

$\alpha = 100.36$ (3)°

$\beta = 101.14$ (3)°

$\gamma = 92.04$ (3)°

$V = 1290.2$ (4) Å³

$Z = 2$

$F_{000} = 680$

$D_x = 1.811$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 11906 reflections

$\theta = 3.0$ – 27.5 °

$\mu = 6.58$ mm⁻¹

$T = 293$ (2) K

Block, colorless

$0.27 \times 0.17 \times 0.08$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: rotating anode

Monochromator: graphite

Detector resolution: 10.0 pixels mm⁻¹

$T = 293$ (2) K

ω scans

Absorption correction: multi-scan
(ABSCOR; Higashi, 1995)

$T_{\min} = 0.275$, $T_{\max} = 0.592$

12741 measured reflections

5855 independent reflections

5326 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\text{max}} = 27.5$ °

$\theta_{\text{min}} = 3.0$ °

$h = -12 \rightarrow 12$

$k = -13 \rightarrow 11$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.082$	$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.7324P]$
$S = 1.12$	where $P = (F_o^2 + 2F_c^2)/3$
5855 reflections	$(\Delta/\sigma)_{\max} = 0.001$
352 parameters	$\Delta\rho_{\max} = 0.80 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.98 \text{ e } \text{\AA}^{-3}$
	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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7524 (5)	0.5869 (5)	0.5700 (4)	0.0294 (9)
C2	0.8815 (4)	0.5438 (4)	0.5338 (3)	0.0277 (9)
C3	1.0097 (5)	0.5650 (5)	0.6012 (4)	0.0338 (10)
H3	1.0164	0.6088	0.6688	0.041*
C4	0.8726 (5)	0.4795 (5)	0.4329 (4)	0.0342 (10)
H4	0.7872	0.4660	0.3877	0.041*
C5	0.5244 (5)	0.6112 (5)	0.8194 (4)	0.0354 (10)
C6	0.5120 (5)	0.5544 (5)	0.9137 (3)	0.0306 (9)
C7	0.4760 (5)	0.6338 (5)	0.9975 (4)	0.0329 (10)
H7	0.4604	0.7243	0.9963	0.039*
C8	0.5374 (5)	0.4184 (5)	0.9160 (4)	0.0339 (10)
H8	0.5630	0.3638	0.8605	0.041*
C9	0.5850 (6)	0.7681 (5)	0.3916 (4)	0.0356 (11)
H9	0.5302	0.6869	0.3697	0.043*
C10	0.6482 (6)	0.8187 (5)	0.3207 (4)	0.0380 (11)
H10	0.6388	0.7685	0.2546	0.046*
C11	0.7240 (5)	0.9417 (5)	0.3473 (4)	0.0354 (10)
C12	0.7367 (5)	1.0139 (5)	0.4512 (3)	0.0323 (10)
C13	0.6757 (5)	0.9515 (4)	0.5188 (3)	0.0272 (9)
C14	0.6947 (5)	1.0147 (4)	0.6262 (3)	0.0278 (9)
C15	0.7765 (5)	1.1398 (4)	0.6614 (3)	0.0267 (9)
C16	0.8285 (5)	1.2039 (5)	0.5886 (4)	0.0348 (10)
H16	0.8764	1.2888	0.6105	0.042*
C17	0.8095 (5)	1.1436 (5)	0.4884 (3)	0.0331 (10)
H17	0.8448	1.1879	0.4431	0.040*

supplementary materials

C18	0.7880 (5)	0.9937 (5)	0.2696 (4)	0.0344 (10)
C19	0.9241 (6)	1.0477 (5)	0.2893 (4)	0.0373 (11)
H19	0.9778	1.0560	0.3549	0.045*
C20	0.9818 (6)	1.0897 (5)	0.2135 (4)	0.0419 (12)
H20	1.0733	1.1255	0.2280	0.050*
C21	0.9020 (7)	1.0776 (6)	0.1162 (5)	0.0539 (15)
H21	0.9395	1.1064	0.0649	0.065*
C22	0.7686 (7)	1.0238 (7)	0.0951 (4)	0.0540 (15)
H22	0.7161	1.0150	0.0290	0.065*
C23	0.7091 (6)	0.9817 (6)	0.1707 (4)	0.0445 (13)
H23	0.6175	0.9459	0.1553	0.053*
C24	0.8039 (5)	1.1944 (4)	0.7674 (3)	0.0289 (9)
C25	0.6614 (6)	1.0051 (5)	0.7875 (4)	0.0369 (11)
H25	0.6223	0.9610	0.8312	0.044*
C26	0.8990 (5)	1.3178 (5)	0.8113 (3)	0.0330 (10)
C27	1.0344 (7)	1.3022 (6)	0.8569 (5)	0.0517 (14)
H27	1.0640	1.2161	0.8609	0.062*
C28	1.1262 (7)	1.4166 (8)	0.8970 (6)	0.068 (2)
H28	1.2180	1.4064	0.9255	0.082*
C29	1.0815 (7)	1.5449 (6)	0.8945 (4)	0.0484 (14)
H29	1.1429	1.6208	0.9222	0.058*
C30	0.9472 (7)	1.5603 (6)	0.8513 (5)	0.0516 (15)
H30	0.9167	1.6468	0.8504	0.062*
C31	0.8564 (6)	1.4469 (5)	0.8087 (4)	0.0409 (11)
H31	0.7656	1.4579	0.7780	0.049*
C32	0.7425 (6)	1.1269 (5)	0.8302 (3)	0.0346 (10)
H32	0.7550	1.1622	0.8999	0.042*
N1	0.5995 (4)	0.8299 (4)	0.4880 (3)	0.0324 (8)
N2	0.6370 (4)	0.9488 (4)	0.6886 (3)	0.0303 (8)
O1	0.6379 (3)	0.5309 (4)	0.5201 (3)	0.0397 (8)
O2	0.7607 (3)	0.6793 (3)	0.6469 (2)	0.0337 (7)
O3	0.5268 (4)	0.5278 (4)	0.7384 (3)	0.0436 (9)
O4	0.5308 (5)	0.7365 (4)	0.8253 (3)	0.0573 (12)
Pb1	0.513080 (16)	0.698420 (16)	0.623266 (12)	0.02661 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.022 (2)	0.030 (2)	0.037 (2)	-0.0013 (17)	0.0068 (17)	0.007 (2)
C2	0.021 (2)	0.026 (2)	0.037 (2)	-0.0003 (17)	0.0054 (17)	0.0096 (19)
C3	0.026 (2)	0.039 (3)	0.034 (2)	-0.0047 (19)	0.0044 (18)	0.004 (2)
C4	0.022 (2)	0.041 (3)	0.038 (2)	-0.0032 (19)	0.0017 (18)	0.009 (2)
C5	0.035 (3)	0.040 (3)	0.033 (2)	-0.006 (2)	0.0092 (19)	0.010 (2)
C6	0.034 (2)	0.031 (2)	0.026 (2)	-0.0036 (19)	0.0058 (18)	0.0033 (19)
C7	0.041 (3)	0.024 (2)	0.033 (2)	0.0002 (19)	0.009 (2)	0.0032 (19)
C8	0.040 (3)	0.027 (2)	0.034 (2)	0.001 (2)	0.009 (2)	-0.0004 (19)
C9	0.046 (3)	0.025 (2)	0.030 (2)	-0.005 (2)	0.004 (2)	-0.0043 (19)
C10	0.052 (3)	0.034 (3)	0.025 (2)	0.000 (2)	0.008 (2)	-0.002 (2)

C11	0.040 (3)	0.033 (2)	0.031 (2)	-0.004 (2)	-0.0015 (19)	0.012 (2)
C12	0.041 (3)	0.026 (2)	0.028 (2)	-0.0030 (19)	0.0024 (19)	0.0066 (19)
C13	0.031 (2)	0.023 (2)	0.025 (2)	-0.0009 (17)	0.0011 (17)	0.0046 (17)
C14	0.031 (2)	0.024 (2)	0.027 (2)	0.0005 (17)	0.0020 (17)	0.0042 (17)
C15	0.031 (2)	0.024 (2)	0.0244 (19)	0.0007 (17)	0.0064 (17)	0.0023 (17)
C16	0.045 (3)	0.025 (2)	0.035 (2)	-0.007 (2)	0.009 (2)	0.0064 (19)
C17	0.047 (3)	0.026 (2)	0.027 (2)	-0.002 (2)	0.0149 (19)	0.0019 (18)
C18	0.042 (3)	0.033 (2)	0.029 (2)	0.003 (2)	0.006 (2)	0.010 (2)
C19	0.043 (3)	0.037 (3)	0.030 (2)	0.005 (2)	0.009 (2)	0.001 (2)
C20	0.044 (3)	0.036 (3)	0.047 (3)	0.004 (2)	0.021 (2)	-0.001 (2)
C21	0.067 (4)	0.053 (4)	0.051 (3)	-0.001 (3)	0.023 (3)	0.025 (3)
C22	0.062 (4)	0.072 (4)	0.032 (3)	0.004 (3)	0.010 (3)	0.019 (3)
C23	0.042 (3)	0.057 (3)	0.034 (3)	-0.001 (3)	0.004 (2)	0.013 (3)
C24	0.035 (2)	0.022 (2)	0.029 (2)	0.0057 (18)	0.0069 (18)	-0.0001 (17)
C25	0.044 (3)	0.037 (3)	0.030 (2)	-0.005 (2)	0.012 (2)	0.004 (2)
C26	0.035 (3)	0.032 (2)	0.028 (2)	-0.0051 (19)	0.0024 (18)	0.0037 (19)
C27	0.045 (3)	0.037 (3)	0.063 (4)	0.008 (2)	0.001 (3)	-0.007 (3)
C28	0.044 (4)	0.069 (5)	0.076 (5)	-0.011 (3)	-0.017 (3)	0.008 (4)
C29	0.058 (4)	0.037 (3)	0.042 (3)	-0.013 (3)	0.007 (3)	-0.006 (2)
C30	0.066 (4)	0.030 (3)	0.056 (3)	-0.010 (3)	0.005 (3)	0.009 (3)
C31	0.040 (3)	0.034 (3)	0.046 (3)	-0.002 (2)	0.003 (2)	0.008 (2)
C32	0.048 (3)	0.032 (2)	0.023 (2)	-0.001 (2)	0.0112 (19)	-0.0010 (19)
N1	0.040 (2)	0.029 (2)	0.0270 (18)	-0.0053 (17)	0.0057 (16)	0.0033 (16)
N2	0.035 (2)	0.0245 (18)	0.0317 (19)	-0.0026 (16)	0.0066 (16)	0.0074 (16)
O1	0.0213 (16)	0.044 (2)	0.049 (2)	-0.0040 (14)	0.0113 (14)	-0.0056 (17)
O2	0.0278 (17)	0.0351 (18)	0.0357 (17)	0.0032 (14)	0.0082 (13)	-0.0018 (15)
O3	0.063 (3)	0.0363 (19)	0.0321 (17)	0.0070 (18)	0.0110 (17)	0.0062 (16)
O4	0.100 (4)	0.0288 (19)	0.045 (2)	-0.008 (2)	0.019 (2)	0.0076 (17)
Pb1	0.02255 (9)	0.02771 (10)	0.03195 (10)	0.00146 (6)	0.00919 (6)	0.00811 (7)

Geometric parameters (Å, °)

C1—O2	1.251 (6)	C17—H17	0.9300
C1—O1	1.253 (6)	C18—C19	1.387 (7)
C1—C2	1.500 (6)	C18—C23	1.394 (7)
C2—C4	1.387 (7)	C19—C20	1.385 (7)
C2—C3	1.393 (6)	C19—H19	0.9300
C3—C4 ⁱ	1.388 (7)	C20—C21	1.380 (8)
C3—H3	0.9300	C20—H20	0.9300
C4—C3 ⁱ	1.388 (7)	C21—C22	1.361 (9)
C4—H4	0.9300	C21—H21	0.9300
C5—O4	1.245 (6)	C22—C23	1.393 (8)
C5—O3	1.259 (6)	C22—H22	0.9300
C5—C6	1.512 (6)	C23—H23	0.9300
C6—C7	1.379 (6)	C24—C32	1.387 (7)
C6—C8	1.402 (7)	C24—C26	1.486 (7)
C7—C8 ⁱⁱ	1.392 (7)	C25—N2	1.329 (6)
C7—H7	0.9300	C25—C32	1.403 (7)

supplementary materials

C8—C7 ⁱⁱ	1.392 (7)	C25—H25	0.9300
C8—H8	0.9300	C26—C31	1.382 (7)
C9—N1	1.320 (6)	C26—C27	1.385 (8)
C9—C10	1.397 (7)	C27—C28	1.394 (9)
C9—H9	0.9300	C27—H27	0.9300
C10—C11	1.372 (7)	C28—C29	1.381 (9)
C10—H10	0.9300	C28—H28	0.9300
C11—C12	1.442 (7)	C29—C30	1.366 (9)
C11—C18	1.486 (7)	C29—H29	0.9300
C12—C13	1.409 (6)	C30—C31	1.387 (8)
C12—C17	1.423 (7)	C30—H30	0.9300
C13—N1	1.364 (6)	C31—H31	0.9300
C13—C14	1.452 (6)	C32—H32	0.9300
C14—N2	1.353 (6)	Pb1—N1	2.686 (4)
C14—C15	1.422 (6)	Pb1—N2	2.677 (4)
C15—C24	1.410 (6)	Pb1—O1	2.503 (4)
C15—C16	1.431 (6)	Pb1—O2	2.418 (3)
C16—C17	1.357 (6)	Pb1—O3	2.506 (4)
C16—H16	0.9300	Pb1—O4	2.663 (4)
O2—C1—O1	121.7 (4)	C19—C20—H20	120.4
O2—C1—C2	119.9 (4)	C22—C21—C20	120.1 (5)
O1—C1—C2	118.4 (4)	C22—C21—H21	119.9
O2—C1—Pb1	59.1 (2)	C20—C21—H21	119.9
O1—C1—Pb1	63.0 (2)	C21—C22—C23	121.2 (5)
C2—C1—Pb1	171.9 (3)	C21—C22—H22	119.4
C4—C2—C3	119.7 (4)	C23—C22—H22	119.4
C4—C2—C1	119.7 (4)	C22—C23—C18	119.5 (5)
C3—C2—C1	120.6 (4)	C22—C23—H23	120.3
C4 ⁱ —C3—C2	119.8 (5)	C18—C23—H23	120.3
C4 ⁱ —C3—H3	120.1	C32—C24—C15	118.0 (4)
C2—C3—H3	120.1	C32—C24—C26	120.8 (4)
C2—C4—C3 ⁱ	120.5 (4)	C15—C24—C26	121.2 (4)
C2—C4—H4	119.7	N2—C25—C32	124.1 (4)
C3 ⁱ —C4—H4	119.7	N2—C25—H25	118.0
O4—C5—O3	123.5 (5)	C32—C25—H25	118.0
O4—C5—C6	119.0 (5)	C31—C26—C27	119.3 (5)
O3—C5—C6	117.5 (4)	C31—C26—C24	122.1 (5)
C7—C6—C8	119.3 (4)	C27—C26—C24	118.6 (5)
C7—C6—C5	121.2 (4)	C26—C27—C28	119.6 (6)
C8—C6—C5	119.5 (4)	C26—C27—H27	120.2
C6—C7—C8 ⁱⁱ	121.5 (4)	C28—C27—H27	120.2
C6—C7—H7	119.3	C29—C28—C27	120.4 (6)
C8 ⁱⁱ —C7—H7	119.3	C29—C28—H28	119.8
C7 ⁱⁱ —C8—C6	119.3 (4)	C27—C28—H28	119.8
C7 ⁱⁱ —C8—H8	120.4	C30—C29—C28	120.0 (5)
C6—C8—H8	120.4	C30—C29—H29	120.0
N1—C9—C10	123.1 (5)	C28—C29—H29	120.0

N1—C9—H9	118.4	C29—C30—C31	119.9 (5)
C10—C9—H9	118.4	C29—C30—H30	120.1
C11—C10—C9	120.8 (4)	C31—C30—H30	120.1
C11—C10—H10	119.6	C26—C31—C30	120.8 (5)
C9—C10—H10	119.6	C26—C31—H31	119.6
C10—C11—C12	117.1 (4)	C30—C31—H31	119.6
C10—C11—C18	119.5 (4)	C24—C32—C25	119.4 (4)
C12—C11—C18	123.5 (4)	C24—C32—H32	120.3
C13—C12—C17	118.9 (4)	C25—C32—H32	120.3
C13—C12—C11	118.1 (4)	C9—N1—C13	118.3 (4)
C17—C12—C11	123.0 (4)	C9—N1—Pb1	119.5 (3)
N1—C13—C12	122.5 (4)	C13—N1—Pb1	121.6 (3)
N1—C13—C14	117.2 (4)	C25—N2—C14	117.1 (4)
C12—C13—C14	120.4 (4)	C25—N2—Pb1	120.4 (3)
N2—C14—C15	123.3 (4)	C14—N2—Pb1	121.8 (3)
N2—C14—C13	118.1 (4)	C1—O1—Pb1	90.5 (3)
C15—C14—C13	118.6 (4)	C1—O2—Pb1	94.5 (3)
C24—C15—C14	118.0 (4)	C5—O3—Pb1	96.3 (3)
C24—C15—C16	123.0 (4)	C5—O4—Pb1	89.3 (3)
C14—C15—C16	119.0 (4)	O2—Pb1—O1	52.76 (11)
C17—C16—C15	121.5 (4)	O2—Pb1—O3	83.20 (13)
C17—C16—H16	119.3	O1—Pb1—O3	83.93 (12)
C15—C16—H16	119.3	O2—Pb1—O4	90.28 (14)
C16—C17—C12	121.4 (4)	O1—Pb1—O4	126.20 (13)
C16—C17—H17	119.3	O3—Pb1—O4	50.42 (12)
C12—C17—H17	119.3	O2—Pb1—N2	72.33 (12)
C19—C18—C23	118.4 (5)	O1—Pb1—N2	116.45 (12)
C19—C18—C11	123.2 (4)	O3—Pb1—N2	121.73 (12)
C23—C18—C11	118.3 (5)	O4—Pb1—N2	77.13 (12)
C20—C19—C18	121.5 (5)	O2—Pb1—N1	73.90 (12)
C20—C19—H19	119.2	O1—Pb1—N1	74.30 (13)
C18—C19—H19	119.2	O3—Pb1—N1	154.98 (14)
C21—C20—C19	119.3 (5)	O4—Pb1—N1	137.65 (12)
C21—C20—H20	120.4	N2—Pb1—N1	60.74 (12)

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

Fig. 1

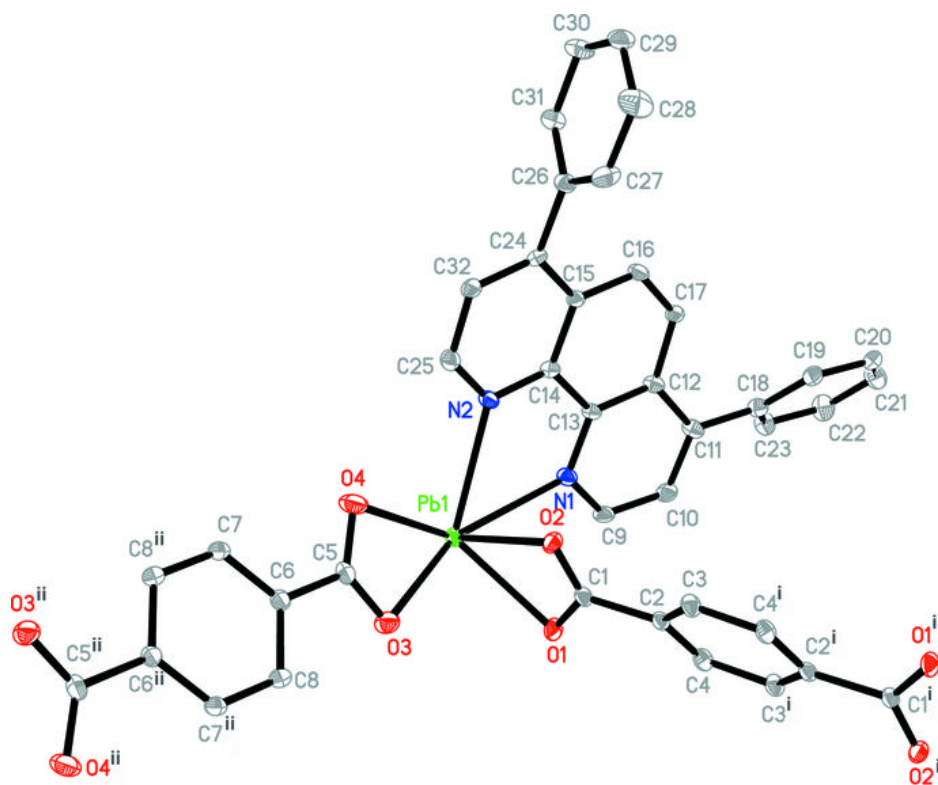


Fig. 2

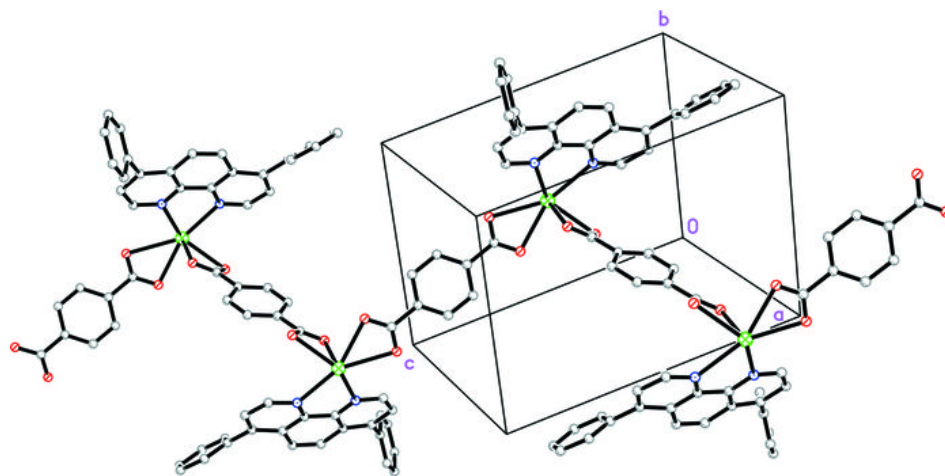


Fig. 3

