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Poly[(2,2-bipyridine)bis(μ_4-naphthalene-1,4-dicarboxylato)dilead(II)]

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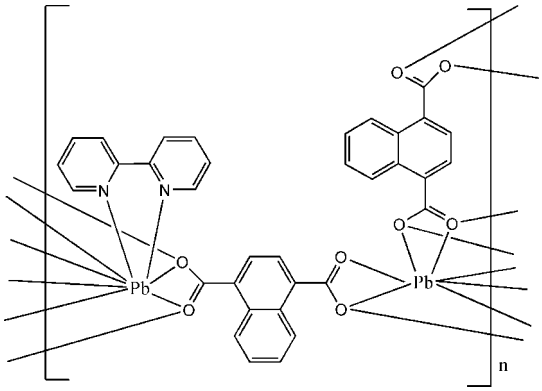
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.028; wR factor = 0.061; data-to-parameter ratio = 15.7.

In the title compound, $[Pb_2(C_{12}H_6O_4)_2(C_{10}H_8N_2)]_n$, neighboring Pb^{II} atoms are linked by naphthalene-1,4-dicarboxylate (1,4-NDC) ligands to form a three-dimensional polymeric structure. One Pb^{II} atom is seven-coordinated by seven carboxylate O atoms from five 1,4-NDC ligands, while the other is eight-coordinated by six carboxylate O atoms from five 1,4-NDC ligands and two N atoms from one 2,2-bipyridine ligand. Aromatic π–π stacking between 2,2-bipyridine and 1,4-NDC ligands helps to establish the packing [minimum centroid–centroid separation 3.475 (6) Å].

Related literature

For studies on related lead(II) carboxylates, see: Fan & Zhu (2007). For background, see: Pan *et al.* (2001); Liu *et al.* (2004).



Experimental

Crystal data

$[Pb_2(C_{12}H_6O_4)_2(C_{10}H_8N_2)]$
M_r = 998.90
Monoclinic, $P2_1$
a = 6.960 (5) Å
b = 14.094 (5) Å
c = 14.947 (5) Å
β = 95.099 (5)°
V = 1460.4 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 11.57 mm^{-1}
T = 293 (2) K
0.34 × 0.24 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.046, T_{max} = 0.124
14420 measured reflections
6518 independent reflections
6203 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.061
S = 1.02
6518 reflections
415 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.96 e Å^{-3}
$\Delta\rho_{min}$ = −1.66 e Å^{-3}
Absolute structure: Flack (1983), 3036 Friedel pairs
Flack parameter: −0.001 (7)

Table 1
Selected bond lengths (Å).

Pb1—O5	2.418 (4)	Pb2—N2	2.630 (6)
Pb1—O8^{i}	2.499 (4)	Pb2—O7^{iv}	2.434 (5)
Pb1—O2^{ii}	2.501 (5)	Pb2—O5^{v}	2.571 (4)
Pb1—O4	2.557 (6)	Pb2—O2	2.649 (4)
Pb1—O6	2.618 (4)	Pb2—O1	2.744 (5)
Pb1—O3	2.631 (5)	Pb2—O4^{vi}	2.856 (4)
Pb1—O1^{iii}	2.841 (4)	Pb2—O6^{vi}	2.785 (5)
Pb2—N1	2.678 (7)		

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

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: HB2578).

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supplementary materials

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Poly[(2,2-bipyridine)bis(μ_4-naphthalene-1,4-dicarboxylato)dilead(II)]

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Comment

There has been tremendous interest in the design and synthesis of coordination polymers due to their potential applications in catalysis, separations, and ion exchange processes (Pan *et al.*, 2001). In this regard, extensive work has been carried out using carboxylate-containing ligands (Liu *et al.*, 2004). naphthalene-1,4-dicarboxylic acid (1,4-H_2NDC), as a rigid linker, seems to be a promising organic ligand. So far, compared with transition metals, the analogous lead(II) complexes containing 1,4-NDC are still undeveloped (Fan & Zhu, 2007). Herein, we present a new Pb(II) coordination polymer, namely the title compound, [Pb_2(1,4-NDC)$_2$(*L*)], (I), where *L*= 2,2-bipyridine.

Selected bond lengths are listed in Table 1. In the asymmetric unit of compound (I) there are two 1,4-NDC dianions, one *L* linagd and two Pb(II) centers (Fig. 1). Pb1 is seven-coordinated by seven carboxylate O atoms from five 1,4-NDC ligands, while Pb2 is eight-coordinated by six carboxylate O atoms from five 1,4-NDC ligands and two N atoms from one *L* ligand.

The 1,4-NDC ligands bridge neighboring Pb(II) atoms to generate a three-dimensional structure (Fig. 2). In addition, π-π interactions between *L* and 1,4-NDC ligands, help to stabilize the structure of (I).

Experimental

A mixture of $Pb(NO_3)_2$ (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 458 K for 6 days under autogenous pressure. Afterward, the reaction system was slowly cooled to room temperature. Colourless blocks of (I) were collected.

Refinement

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

Figures

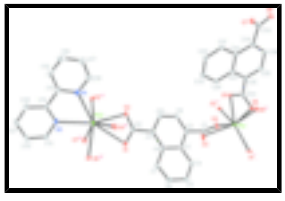

Fig. 1. The asymmetric unit of (I), expanded to show the lead coordination polyhedra. Displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity. Symmetry codes: (i) 1 − *x*, *y* + 1/2, −*z*; (ii) −*x*, *y* + 1/2, 1 − *z*; (iii) 1 − *x*, *y* − 1/2, −*z*; (iv) −*x*, *y* − 1/2, −*z*; (v) −*x*, *y* + 1/2, −*z*; (vi) *x*, *y*, *z* − 1]

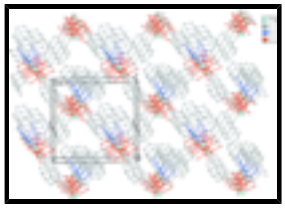

Fig. 2. : View of the packing in (I). The hydrogen atoms are omitted for clarity.

Poly[(2,2-bipyridine)bis(μ$_4$-naphthalene-1,4-dicarboxylato)dilead(II)]

Crystal data

$[Pb_2(C_{12}H_6O_4)_2(C_{10}H_8N_2)]$	F_{000} = 932
M_r = 998.90	D_x = 2.272 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 14029 reflections
a = 6.960 (5) Å	θ = 3.0–27.5º
b = 14.094 (5) Å	μ = 11.57 mm^{-1}
c = 14.947 (5) Å	T = 293 (2) K
β = 95.099 (5)º	Block, colourless
V = 1460.4 (13) Å^3	0.34 × 0.24 × 0.18 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID diffractometer	6518 independent reflections
Radiation source: rotating anode	6203 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.040
Detector resolution: 10.0 pixels mm^{-1}	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 3.1º
ω scans	h = −9→9
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	k = −18→18
T_{min} = 0.046, T_{max} = 0.124	l = −19→19
14420 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.028	$w = 1/[\sigma^2(F_o^2) + (0.0187P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.061	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.02	$\Delta\rho_{max}$ = 1.96 e Å^{-3}
6518 reflections	$\Delta\rho_{min}$ = −1.65 e Å^{-3}

415 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 3036 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.001 (7)
Secondary atom site location: difference Fourier map	

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}
Pb1	0.11553 (3)	0.852058 (17)	0.206648 (12)	0.01977 (6)
Pb2	0.36408 (3)	0.224708 (16)	−0.285163 (12)	0.02040 (6)
C1	0.2458 (9)	0.7163 (6)	0.0764 (4)	0.0285 (14)
C2	0.2849 (9)	0.6357 (5)	0.0145 (4)	0.0260 (14)
C3	0.4448 (9)	0.6417 (5)	−0.0397 (4)	0.0237 (13)
C4	0.5760 (10)	0.7174 (6)	−0.0304 (4)	0.0339 (15)
H4	0.5547	0.7664	0.0092	0.041*
C5	0.7351 (10)	0.7205 (6)	−0.0783 (4)	0.0358 (15)
H5	0.8259	0.7687	−0.0688	0.043*
C6	0.7575 (10)	0.6494 (6)	−0.1422 (4)	0.0351 (16)
H6	0.8629	0.6522	−0.1762	0.042*
C7	0.6301 (10)	0.5763 (5)	−0.1562 (4)	0.0297 (14)
H7	0.6460	0.5319	−0.2010	0.036*
C8	0.4714 (9)	0.5677 (5)	−0.1017 (4)	0.0232 (13)
C9	0.1705 (10)	0.5580 (6)	0.0108 (4)	0.0329 (16)
H9	0.0700	0.5543	0.0477	0.039*
C10	0.2011 (9)	0.4835 (5)	−0.0474 (3)	0.0287 (14)
H10	0.1228	0.4300	−0.0474	0.034*
C11	0.3449 (9)	0.4871 (6)	−0.1050 (3)	0.0252 (14)
C12	0.3596 (9)	0.4025 (5)	−0.1662 (4)	0.0204 (12)
C13	0.3939 (9)	0.1196 (6)	−0.4927 (4)	0.0331 (16)
H13	0.4139	0.1838	−0.5029	0.040*
C14	0.3332 (9)	0.0000 (5)	−0.3950 (4)	0.0306 (15)
C15	0.3043 (10)	−0.0279 (6)	−0.2996 (5)	0.0349 (15)
C16	0.2939 (12)	0.0217 (7)	−0.1540 (5)	0.048 (2)
H16	0.2961	0.0710	−0.1126	0.058*
C17	0.2709 (13)	−0.0689 (9)	−0.1243 (6)	0.064 (3)
H17	0.2610	−0.0814	−0.0638	0.076*

C18	0.2629 (13)	−0.1406 (9)	−0.1861 (7)	0.063 (3)
H18	0.2470	−0.2028	−0.1676	0.076*
C19	0.2776 (12)	−0.1221 (6)	−0.2725 (7)	0.050 (2)
H19	0.2702	−0.1709	−0.3145	0.060*
C20	0.3406 (11)	−0.0661 (7)	−0.4635 (6)	0.048 (2)
H20	0.3231	−0.1301	−0.4517	0.057*
C21	0.3732 (11)	−0.0377 (8)	−0.5474 (5)	0.051 (2)
H21	0.3767	−0.0818	−0.5935	0.061*
C22	0.4017 (10)	0.0589 (8)	−0.5636 (5)	0.048 (2)
H22	0.4249	0.0809	−0.6203	0.057*
C23	0.1397 (9)	0.7015 (5)	0.3389 (4)	0.0227 (14)
C24	0.1379 (10)	0.3971 (5)	0.5987 (4)	0.0289 (15)
C25	0.1344 (8)	0.6205 (5)	0.4014 (3)	0.0194 (12)
C26	0.1083 (9)	0.6386 (5)	0.4902 (4)	0.0253 (13)
H26	0.0925	0.7007	0.5095	0.030*
C27	0.1627 (8)	0.5280 (5)	0.3715 (4)	0.0210 (13)
C28	0.1967 (9)	0.5088 (5)	0.2806 (4)	0.0233 (13)
H28	0.1987	0.5585	0.2397	0.028*
C29	0.1055 (9)	0.5641 (5)	0.5508 (4)	0.0279 (15)
H29	0.0836	0.5771	0.6100	0.033*
C30	0.1647 (8)	0.4506 (5)	0.4340 (3)	0.0199 (12)
C31	0.1917 (9)	0.3573 (6)	0.4014 (4)	0.0279 (13)
H31	0.1874	0.3060	0.4403	0.033*
C32	0.2239 (10)	0.3418 (6)	0.3137 (4)	0.0339 (15)
H32	0.2443	0.2804	0.2939	0.041*
C33	0.2265 (10)	0.4179 (6)	0.2532 (4)	0.0335 (16)
H33	0.2487	0.4063	0.1938	0.040*
C34	0.1345 (8)	0.4712 (5)	0.5257 (3)	0.0223 (12)
N1	0.3134 (9)	0.0430 (5)	−0.2403 (4)	0.0350 (14)
N2	0.3599 (8)	0.0932 (5)	−0.4102 (3)	0.0293 (12)
O1	0.4860 (7)	0.3992 (4)	−0.2213 (3)	0.0317 (11)
O2	0.2371 (6)	0.3358 (3)	−0.1602 (2)	0.0252 (10)
O3	0.1433 (11)	0.7825 (5)	0.0449 (4)	0.070 (2)
O4	0.3090 (8)	0.7106 (4)	0.1561 (3)	0.0426 (14)
O5	0.0016 (6)	0.7099 (3)	0.2755 (2)	0.0235 (9)
O6	0.2754 (6)	0.7594 (4)	0.3457 (3)	0.0267 (10)
O7	0.2291 (8)	0.3220 (4)	0.5901 (3)	0.0361 (12)
O8	0.0526 (7)	0.4206 (4)	0.6673 (3)	0.0365 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.02363 (12)	0.01570 (12)	0.02061 (9)	−0.00022 (10)	0.00555 (7)	0.00041 (9)
Pb2	0.02071 (11)	0.01725 (12)	0.02339 (10)	0.00086 (9)	0.00270 (7)	−0.00212 (9)
C1	0.031 (3)	0.028 (4)	0.026 (3)	−0.002 (3)	0.002 (2)	−0.009 (3)
C2	0.032 (4)	0.024 (4)	0.021 (3)	0.000 (3)	−0.002 (2)	−0.006 (2)
C3	0.022 (3)	0.020 (4)	0.028 (3)	−0.001 (3)	−0.004 (2)	−0.004 (2)
C4	0.040 (4)	0.031 (4)	0.030 (3)	0.004 (3)	−0.001 (2)	−0.010 (3)

C5	0.037 (4)	0.024 (4)	0.046 (3)	−0.005 (4)	0.001 (2)	0.006 (3)
C6	0.034 (4)	0.024 (4)	0.048 (4)	−0.003 (3)	0.013 (3)	−0.002 (3)
C7	0.035 (4)	0.020 (4)	0.035 (3)	0.003 (3)	0.011 (2)	−0.002 (3)
C8	0.023 (3)	0.021 (4)	0.026 (3)	0.000 (3)	−0.001 (2)	0.002 (2)
C9	0.040 (4)	0.034 (4)	0.026 (3)	−0.001 (3)	0.012 (2)	−0.007 (3)
C10	0.034 (4)	0.026 (4)	0.026 (3)	−0.006 (3)	0.005 (2)	−0.004 (3)
C11	0.034 (4)	0.024 (4)	0.017 (3)	0.001 (3)	0.000 (2)	−0.002 (3)
C12	0.024 (3)	0.015 (3)	0.021 (3)	−0.004 (3)	−0.002 (2)	0.006 (2)
C13	0.022 (3)	0.035 (5)	0.043 (3)	0.004 (3)	0.008 (2)	−0.013 (3)
C14	0.016 (3)	0.022 (4)	0.053 (4)	0.001 (3)	−0.002 (2)	−0.011 (3)
C15	0.017 (3)	0.025 (4)	0.062 (4)	0.008 (3)	−0.001 (3)	−0.007 (3)
C16	0.038 (5)	0.058 (6)	0.047 (4)	−0.006 (4)	−0.001 (3)	0.010 (4)
C17	0.039 (5)	0.086 (9)	0.064 (5)	−0.008 (5)	−0.008 (4)	0.038 (6)
C18	0.040 (5)	0.046 (6)	0.101 (7)	−0.003 (5)	−0.007 (4)	0.035 (6)
C19	0.028 (4)	0.025 (5)	0.096 (6)	−0.002 (3)	0.003 (4)	−0.004 (4)
C20	0.019 (4)	0.042 (6)	0.081 (6)	0.003 (4)	−0.005 (3)	−0.031 (4)
C21	0.029 (4)	0.067 (7)	0.056 (4)	0.005 (4)	0.004 (3)	−0.042 (5)
C22	0.022 (4)	0.080 (8)	0.042 (4)	−0.001 (4)	0.003 (3)	−0.027 (4)
C23	0.023 (3)	0.016 (4)	0.030 (3)	0.006 (2)	0.006 (2)	−0.005 (2)
C24	0.029 (4)	0.030 (4)	0.028 (3)	−0.010 (3)	0.004 (2)	0.006 (3)
C25	0.015 (3)	0.023 (4)	0.021 (2)	−0.003 (2)	0.0039 (18)	0.006 (2)
C26	0.027 (3)	0.017 (3)	0.032 (3)	0.006 (3)	0.004 (2)	0.004 (2)
C27	0.009 (3)	0.027 (4)	0.027 (3)	0.000 (2)	0.0005 (19)	0.002 (2)
C28	0.024 (3)	0.025 (4)	0.021 (3)	−0.004 (3)	0.0037 (19)	0.005 (2)
C29	0.023 (3)	0.038 (4)	0.023 (3)	0.001 (3)	0.005 (2)	0.004 (3)
C30	0.013 (3)	0.020 (3)	0.028 (3)	−0.003 (2)	0.0030 (18)	−0.001 (2)
C31	0.027 (3)	0.023 (3)	0.034 (3)	−0.002 (3)	0.000 (2)	0.004 (3)
C32	0.045 (4)	0.021 (4)	0.035 (3)	0.005 (3)	0.001 (2)	−0.003 (3)
C33	0.031 (4)	0.039 (5)	0.030 (3)	0.000 (3)	0.000 (2)	0.001 (3)
C34	0.019 (3)	0.025 (4)	0.023 (3)	−0.002 (3)	0.0017 (18)	0.004 (2)
N1	0.036 (3)	0.030 (4)	0.039 (3)	0.000 (3)	−0.001 (2)	0.004 (3)
N2	0.026 (3)	0.029 (3)	0.032 (3)	0.004 (3)	−0.0004 (19)	−0.015 (2)
O1	0.030 (3)	0.030 (3)	0.038 (2)	0.005 (2)	0.0158 (19)	−0.016 (2)
O2	0.026 (2)	0.024 (3)	0.0258 (18)	0.0056 (19)	0.0036 (14)	−0.0058 (17)
O3	0.103 (6)	0.055 (5)	0.046 (3)	0.043 (4)	−0.027 (3)	−0.033 (3)
O4	0.071 (4)	0.040 (4)	0.0174 (19)	0.007 (3)	0.0022 (18)	−0.007 (2)
O5	0.018 (2)	0.019 (3)	0.033 (2)	0.0017 (18)	0.0018 (14)	0.0048 (17)
O6	0.022 (2)	0.028 (3)	0.031 (2)	−0.005 (2)	0.0033 (15)	0.0010 (18)
O7	0.046 (3)	0.028 (3)	0.034 (2)	0.006 (2)	0.0032 (19)	0.0059 (19)
O8	0.046 (3)	0.032 (3)	0.033 (2)	−0.002 (2)	0.011 (2)	0.013 (2)

Geometric parameters (Å, °)

Pb1—O5	2.418 (4)	C14—C15	1.511 (10)
Pb1—O8^{i}	2.499 (4)	C15—N1	1.334 (10)
Pb1—O2^{ii}	2.501 (5)	C15—C19	1.404 (12)
Pb1—O4	2.557 (6)	C16—N1	1.342 (9)
Pb1—O6	2.618 (4)	C16—C17	1.367 (14)
Pb1—O3	2.631 (5)	C16—H16	0.9300

Pb1—O1^{iii}	2.841 (4)	C17—C18	1.366 (16)
Pb2—N1	2.678 (7)	C17—H17	0.9300
Pb2—N2	2.630 (6)	C18—C19	1.331 (13)
Pb2—O7^{iv}	2.434 (5)	C18—H18	0.9300
Pb2—O5^{v}	2.571 (4)	C19—H19	0.9300
Pb2—O2	2.649 (4)	C20—C21	1.354 (12)
Pb2—O1	2.744 (5)	C20—H20	0.9300
Pb2—O4^{vi}	2.856 (4)	C21—C22	1.401 (15)
Pb2—O6^{vi}	2.785 (5)	C21—H21	0.9300
C1—O4	1.236 (7)	C22—H22	0.9300
C1—O3	1.241 (9)	C23—O6	1.245 (8)
C1—C2	1.505 (9)	C23—O5	1.294 (7)
C2—C9	1.352 (10)	C23—C25	1.477 (8)
C2—C3	1.436 (9)	C24—O7	1.247 (9)
C3—C4	1.403 (10)	C24—O8	1.274 (8)
C3—C8	1.418 (9)	C24—C34	1.509 (9)
C4—C5	1.373 (9)	C25—C26	1.381 (8)
C4—H4	0.9300	C25—C27	1.398 (9)
C5—C6	1.402 (10)	C26—C29	1.388 (9)
C5—H5	0.9300	C26—H26	0.9300
C6—C7	1.363 (10)	C27—C28	1.425 (8)
C6—H6	0.9300	C27—C30	1.435 (9)
C7—C8	1.435 (8)	C28—C33	1.367 (10)
C7—H7	0.9300	C28—H28	0.9300
C8—C11	1.435 (10)	C29—C34	1.382 (10)
C9—C10	1.392 (9)	C29—H29	0.9300
C9—H9	0.9300	C30—C31	1.421 (10)
C10—C11	1.378 (8)	C30—C34	1.435 (7)
C10—H10	0.9300	C31—C32	1.367 (8)
C11—C12	1.512 (9)	C31—H31	0.9300
C12—O1	1.259 (7)	C32—C33	1.403 (10)
C12—O2	1.278 (8)	C32—H32	0.9300
C13—N2	1.330 (9)	C33—H33	0.9300
C13—C22	1.366 (10)	O2—Pb1^{v}	2.501 (5)
C13—H13	0.9300	O5—Pb2^{ii}	2.571 (4)
C14—N2	1.348 (10)	O7—Pb2^{vii}	2.434 (5)
C14—C20	1.388 (10)	O8—Pb1^{viii}	2.499 (4)
O5—Pb1—O8^{i}	78.82 (17)	N2—C13—H13	117.7
O5—Pb1—O2^{ii}	72.02 (14)	C22—C13—H13	117.7
O8^{i}—Pb1—O2^{ii}	74.40 (16)	N2—C14—C20	121.0 (7)
O5—Pb1—O4	71.40 (16)	N2—C14—C15	116.4 (6)
O8^{i}—Pb1—O4	144.92 (16)	C20—C14—C15	122.5 (8)
O2^{ii}—Pb1—O4	112.08 (17)	N1—C15—C19	121.1 (7)
O5—Pb1—O6	51.76 (14)	N1—C15—C14	115.4 (7)
O8^{i}—Pb1—O6	77.81 (16)	C19—C15—C14	123.4 (7)

O2^{ii}—Pb1—O6	120.81 (13)	N1—C16—C17	123.1 (9)
O4—Pb1—O6	69.37 (14)	N1—C16—H16	118.4
O5—Pb1—O3	97.8 (2)	C17—C16—H16	118.4
O8^{i}—Pb1—O3	156.09 (19)	C18—C17—C16	118.0 (9)
O2^{ii}—Pb1—O3	82.02 (19)	C18—C17—H17	121.0
O4—Pb1—O3	49.80 (17)	C16—C17—H17	121.0
O6—Pb1—O3	118.86 (17)	C19—C18—C17	120.6 (10)
O3^{iii}—Pb1—O1	88.38 (5)	C19—C18—H18	119.7
O4^{iii}—Pb1—O1	78.64 (5)	C17—C18—H18	119.7
O5^{iii}—Pb1—O1	70.77 (4)	C18—C19—C15	119.2 (9)
O6^{iii}—Pb1—O1	72.92 (4)	C18—C19—H19	120.4
O8^{i}—Pb1—O1^{iii}	111.8 (17)	C15—C19—H19	120.4
O7^{iv}—Pb2—O5^{v}	76.83 (16)	C21—C20—C14	120.3 (9)
O7^{iv}—Pb2—N2	82.76 (18)	C21—C20—H20	119.9
O5^{v}—Pb2—N2	91.97 (15)	C14—C20—H20	119.9
O7^{iv}—Pb2—O2	94.42 (15)	C20—C21—C22	119.0 (7)
O5^{v}—Pb2—O2	67.30 (13)	C20—C21—H21	120.5
N2—Pb2—O2	159.11 (15)	C22—C21—H21	120.5
O7^{iv}—Pb2—N1	132.84 (18)	C13—C22—C21	117.3 (8)
O5^{v}—Pb2—N1	75.84 (17)	C13—C22—H22	121.4
N2—Pb2—N1	60.71 (19)	C21—C22—H22	121.4
O2—Pb2—N1	109.19 (16)	O6—C23—O5	120.5 (6)
O7^{iv}—Pb2—O1	81.24 (17)	O6—C23—C25	121.1 (6)
O5^{v}—Pb2—O1	109.27 (13)	O5—C23—C25	118.4 (6)
N2—Pb2—O1	149.53 (16)	O7—C24—O8	125.5 (6)
O2—Pb2—O1	48.51 (13)	O7—C24—C34	119.3 (6)
N1—Pb2—O1	144.39 (16)	O8—C24—C34	115.1 (7)
N1^{vi}—Pb2—O4	97.67 (4)	C26—C25—C27	120.9 (5)
N2^{vi}—Pb2—O4	87.33 (4)	C26—C25—C23	118.6 (6)
O1^{vi}—Pb2—O4	77.84 (5)	C27—C25—C23	120.5 (5)
O2^{vi}—Pb2—O4	87.36 (4)	C25—C26—C29	119.9 (6)
O5^{v}—Pb2—O4^{vi}	133.61 (12)	C25—C26—H26	120.1
N1^{vi}—Pb2—O6	90.45 (4)	C29—C26—H26	120.1
N2^{vi}—Pb2—O6	80.04 (4)	C25—C27—C28	121.4 (6)
O1^{vi}—Pb2—O6	70.54 (4)	C25—C27—C30	119.7 (5)
O2^{vi}—Pb2—O6	80.49 (3)	C28—C27—C30	118.9 (6)
O5^{v}—Pb2—O6^{vi}	163.26 (12)	C33—C28—C27	120.4 (6)
O4^{vi}—Pb2—O6^{vi}	62.94 (14)	C33—C28—H28	119.8
O4—C1—O3	123.9 (7)	C27—C28—H28	119.8
O4—C1—C2	118.4 (7)	C34—C29—C26	122.0 (5)
O3—C1—C2	117.6 (5)	C34—C29—H29	119.0
C9—C2—C3	120.5 (6)	C26—C29—H29	119.0
C9—C2—C1	119.9 (6)	C31—C30—C27	118.3 (5)

C3—C2—C1	119.6 (6)	C31—C30—C34	123.3 (6)
C4—C3—C8	120.2 (6)	C27—C30—C34	118.3 (6)
C4—C3—C2	121.3 (6)	C32—C31—C30	121.0 (6)
C8—C3—C2	118.5 (6)	C32—C31—H31	119.5
C5—C4—C3	121.4 (7)	C30—C31—H31	119.5
C5—C4—H4	119.3	C31—C32—C33	120.5 (7)
C3—C4—H4	119.3	C31—C32—H32	119.7
C4—C5—C6	118.4 (7)	C33—C32—H32	119.7
C4—C5—H5	120.8	C28—C33—C32	120.8 (6)
C6—C5—H5	120.8	C28—C33—H33	119.6
C7—C6—C5	122.3 (6)	C32—C33—H33	119.6
C7—C6—H6	118.8	C29—C34—C30	119.2 (5)
C5—C6—H6	118.8	C29—C34—C24	116.9 (5)
C6—C7—C8	120.0 (6)	C30—C34—C24	123.9 (6)
C6—C7—H7	120.0	C15—N1—C16	117.9 (7)
C8—C7—H7	120.0	C15—N1—Pb2	123.4 (5)
C3—C8—C7	117.4 (6)	C16—N1—Pb2	118.8 (6)
C3—C8—C11	119.3 (5)	C13—N2—C14	117.8 (6)
C7—C8—C11	123.2 (6)	C13—N2—Pb2	118.1 (5)
C2—C9—C10	120.9 (6)	C14—N2—Pb2	124.1 (4)
C2—C9—H9	119.5	C12—O1—Pb2	92.8 (4)
C10—C9—H9	119.5	C12—O2—Pb1^{v}	123.9 (4)
C11—C10—C9	121.5 (7)	C12—O2—Pb2	96.8 (4)
C11—C10—H10	119.2	Pb1^{v}—O2—Pb2	103.83 (14)
C9—C10—H10	119.2	C1—O3—Pb1	91.1 (4)
C10—C11—C8	119.0 (6)	C1—O4—Pb1	94.7 (5)
C10—C11—C12	116.2 (6)	C23—O5—Pb1	97.9 (4)
C8—C11—C12	124.8 (5)	C23—O5—Pb2^{ii}	129.9 (4)
O1—C12—O2	121.9 (6)	Pb1—O5—Pb2^{ii}	108.70 (16)
O1—C12—C11	120.9 (6)	C23—O6—Pb1	89.8 (4)
O2—C12—C11	117.1 (5)	C24—O7—Pb2^{vii}	124.4 (4)
N2—C13—C22	124.6 (8)	C24—O8—Pb1^{viii}	142.2 (5)

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

Fig. 1

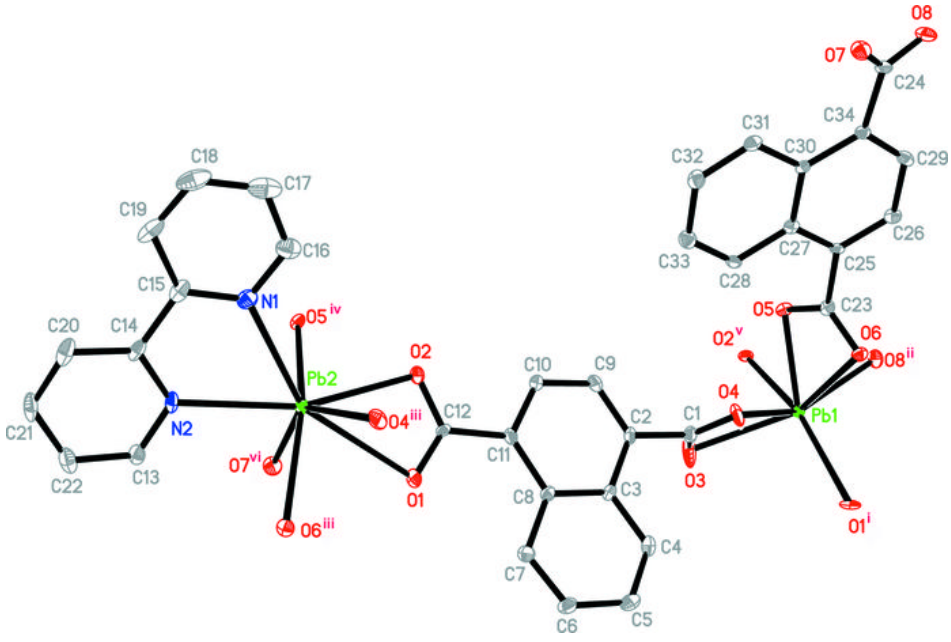
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Fig. 2