

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

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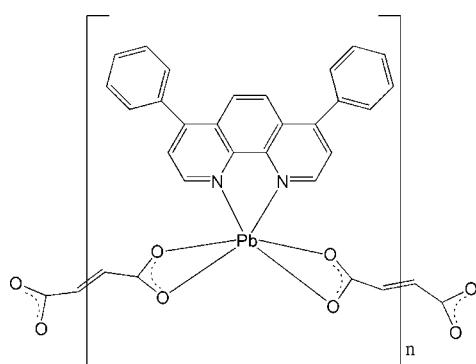
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Key indicators: single-crystal X-ray study; $T = 293\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$; R factor = 0.036; wR factor = 0.061; data-to-parameter ratio = 16.6.

In the title compound, $[\text{Pb}(\text{C}_4\text{H}_2\text{O}_4)(\text{C}_{24}\text{H}_{16}\text{N}_2)]_n$, the Pb^{II} atom is chelated by 4,7-diphenyl-1,10-phenanthroline (L) and also bonded to four O atoms from two fumarate dianions to yield an irregular PbN_2O_4 coordination polyhedron. The centrosymmetric fumarate ligands bridge neighboring Pb^{II} atoms to generate a chain structure. Aromatic $\pi-\pi$ stacking between L ligands in adjacent chains [minimum centroid–centroid separation = 3.462 (3) \AA] leads to a two-dimensional supramolecular array.

Related literature

For studies on related lead(II) carboxylates, see: Fan & Zhu (2006); Yang *et al.* (2007).



Experimental

Crystal data

| | |
|---|--|
| $[\text{Pb}(\text{C}_4\text{H}_2\text{O}_4)(\text{C}_{24}\text{H}_{16}\text{N}_2)]$ | $\gamma = 82.49 (3)^\circ$ |
| $M_r = 653.63$ | $V = 1154.7 (4)\text{ \AA}^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 8.3044 (17)\text{ \AA}$ | Mo $K\alpha$ radiation |
| $b = 11.021 (2)\text{ \AA}$ | $\mu = 7.35\text{ mm}^{-1}$ |
| $c = 12.746 (3)\text{ \AA}$ | $T = 293 (2)\text{ K}$ |
| $\alpha = 89.35 (3)^\circ$ | $0.18 \times 0.16 \times 0.15\text{ mm}$ |
| $\beta = 86.73 (3)^\circ$ | |

Data collection

| | |
|--|--|
| Rigaku R-AXIS RAPID diffractometer | 11422 measured reflections |
| Absorption correction: multi-scan (<i>ABSCOR</i> ; Higashi, 1995) | 5231 independent reflections |
| $T_{\min} = 0.254$, $T_{\max} = 0.333$ | 4411 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.041$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.037$ | 316 parameters |
| $wR(F^2) = 0.061$ | H-atom parameters constrained |
| $S = 1.06$ | $\Delta\rho_{\max} = 0.77\text{ e \AA}^{-3}$ |
| 5231 reflections | $\Delta\rho_{\min} = -0.70\text{ e \AA}^{-3}$ |

Table 1
Selected bond lengths (\AA).

| | | | |
|--------|-----------|--------|-----------|
| Pb1–N1 | 2.590 (4) | Pb1–O2 | 2.740 (4) |
| Pb1–N2 | 2.645 (4) | Pb1–O3 | 2.313 (3) |
| Pb1–O1 | 2.449 (4) | Pb1–O4 | 2.705 (4) |

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: HB2602).

References

- Fan, S. R. & Zhu, L.-G. (2006). *Inorg. Chem.* **45**, 7935–7942.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
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supplementary materials

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

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Comment

Metal cations with an s^2 electron configuration have important applications in electroluminescent devices or organic light-emitting diode technology (Fan & Zhu, 2006). Among them, the Pb^{II} coordination polymers have received much attention due to their interesting network topologies and properties (Yang *et al.*, 2007). Herein, we present a new $Pb(II)$ coordination polymer, namely the title compound, (I), $[Pb(fum)(L)]$, where fum = the fumarate dianion and L = 4,7-diphenyl-1,10-phenanthroline.

In compound (I) the Pb^{II} atom is six-coordinated by four carboxylate O atoms from two different fum ligands and two N atoms from one L ligand (Table 1, Fig. 1). The resulting PbN_2O_6 polyhedron is irregular, perhaps caused, in part, by the stereochemical activity of the Pb lone pair of electrons. There are two fum half-molecules in the asymmetric unit, both being completed by inversion symmetry. The neighboring Pb^{II} atoms are linked by the fum dianions to form a chain structure and the L ligands are decoded on both sides of the chains (Fig. 2). Furthermore, π - π interactions [minimum centroid-centroid separation = 3.462 (3) Å] between L ligands in neighboring chains yields a two-dimensional supramolecular layer structure (Fig. 3).

Experimental

A mixture of $Pb(NO_3)_2$ (1 mmol), H_2fum (1 mmol) and L (1 mmol) were dissolved in 12 ml distilled water, followed by addition of triethylamine until the pH value 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 450 K for 10 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 $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

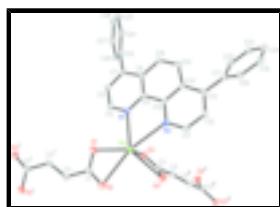


Fig. 1. The asymmetric unit of (I) expanded to show the complete fum dianions, with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level. H atoms omitted for clarity. Symmetry codes: (i) $1-x, -y, 1-z$; (ii) $-x, -y, -z$.

supplementary materials

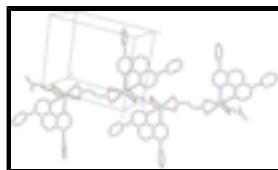


Fig. 2. View of the chain structure of (I). The hydrogen atoms are omitted for clarity.

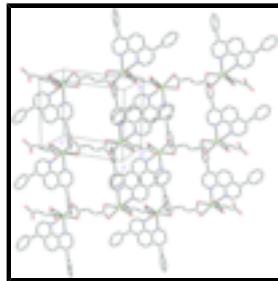


Fig. 3. The supramolecular layer structure of (I). The hydrogen atoms are omitted for clarity.

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

Crystal data

| | |
|--|---|
| [Pb(C ₄ H ₂ O ₄)(C ₂₄ H ₁₆ N ₂)] | $Z = 2$ |
| $M_r = 653.63$ | $F_{000} = 628$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.880 \text{ Mg m}^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation |
| $a = 8.3044 (17) \text{ \AA}$ | $\lambda = 0.71073 \text{ \AA}$ |
| $b = 11.021 (2) \text{ \AA}$ | Cell parameters from 10181 reflections |
| $c = 12.746 (3) \text{ \AA}$ | $\theta = 3.0\text{--}27.5^\circ$ |
| $\alpha = 89.35 (3)^\circ$ | $\mu = 7.35 \text{ mm}^{-1}$ |
| $\beta = 86.73 (3)^\circ$ | $T = 293 (2) \text{ K}$ |
| $\gamma = 82.49 (3)^\circ$ | Block, colorless |
| $V = 1154.7 (4) \text{ \AA}^3$ | $0.18 \times 0.16 \times 0.15 \text{ mm}$ |

Data collection

| | |
|---|--|
| Rigaku R-AXIS RAPID diffractometer | 5231 independent reflections |
| Radiation source: rotating anode | 4411 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.041$ |
| Detector resolution: 10.0 pixels mm ⁻¹ | $\theta_{\text{max}} = 27.5^\circ$ |
| $T = 293(2) \text{ K}$ | $\theta_{\text{min}} = 3.0^\circ$ |
| ω scan | $h = -10 \rightarrow 10$ |
| Absorption correction: multi-scan (ABSCOR; Higashi, 1995) | $k = -14 \rightarrow 14$ |
| $T_{\text{min}} = 0.254$, $T_{\text{max}} = 0.333$ | $l = -16 \rightarrow 14$ |
| 11422 measured reflections | |

Refinement

| | |
|--|---|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.037$ | H-atom parameters constrained |
| $wR(F^2) = 0.061$ | $w = 1/[\sigma^2(F_o^2) + (0.0096P)^2 + 1.8148P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{\max} = 0.002$ |
| 5231 reflections | $\Delta\rho_{\max} = 0.77 \text{ e \AA}^{-3}$ |
| 316 parameters | $\Delta\rho_{\min} = -0.70 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | 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 | 0.4593 (6) | 0.0099 (5) | 0.3502 (4) | 0.0427 (12) |
| C2 | 0.4835 (6) | -0.0340 (4) | 0.4607 (4) | 0.0432 (12) |
| H2 | 0.4744 | -0.1158 | 0.4750 | 0.052* |
| C3 | 0.1800 (6) | 0.0473 (4) | 0.0722 (4) | 0.0401 (12) |
| C4 | 0.0161 (6) | 0.0255 (4) | 0.0441 (4) | 0.0419 (12) |
| H4 | -0.0706 | 0.0494 | 0.0920 | 0.050* |
| C5 | 0.2750 (6) | 0.3741 (4) | 0.3041 (4) | 0.0400 (12) |
| H5 | 0.2981 | 0.3115 | 0.3526 | 0.048* |
| C6 | 0.1994 (7) | 0.4868 (4) | 0.3407 (4) | 0.0429 (12) |
| H6 | 0.1737 | 0.4979 | 0.4122 | 0.051* |
| C7 | 0.1623 (6) | 0.5819 (4) | 0.2720 (4) | 0.0384 (11) |
| C8 | 0.2041 (6) | 0.5598 (4) | 0.1626 (4) | 0.0377 (11) |
| C9 | 0.2769 (6) | 0.4420 (4) | 0.1324 (4) | 0.0342 (11) |
| C10 | 0.3062 (6) | 0.4128 (4) | 0.0226 (4) | 0.0356 (11) |
| C11 | 0.3919 (7) | 0.2663 (4) | -0.1023 (4) | 0.0492 (14) |
| H11 | 0.4357 | 0.1865 | -0.1197 | 0.059* |
| C12 | 0.3552 (7) | 0.3488 (4) | -0.1829 (4) | 0.0470 (13) |
| H12 | 0.3729 | 0.3224 | -0.2522 | 0.056* |

supplementary materials

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|-----|-------------|---------------|---------------|-------------|
| C13 | 0.2933 (6) | 0.4686 (4) | -0.1631 (4) | 0.0382 (11) |
| C14 | 0.2685 (6) | 0.5034 (4) | -0.0553 (4) | 0.0362 (11) |
| C15 | 0.2079 (7) | 0.6242 (4) | -0.0196 (4) | 0.0513 (15) |
| H15 | 0.1918 | 0.6868 | -0.0690 | 0.062* |
| C16 | 0.1732 (7) | 0.6510 (4) | 0.0830 (4) | 0.0517 (15) |
| H16 | 0.1285 | 0.7300 | 0.1022 | 0.062* |
| C17 | 0.2590 (6) | 0.5552 (4) | -0.2513 (4) | 0.0372 (11) |
| C18 | 0.3689 (6) | 0.5503 (5) | -0.3385 (4) | 0.0447 (13) |
| H18 | 0.4610 | 0.4922 | -0.3403 | 0.054* |
| C19 | 0.3443 (8) | 0.6296 (5) | -0.4222 (4) | 0.0549 (15) |
| H19 | 0.4189 | 0.6241 | -0.4798 | 0.066* |
| C20 | 0.2103 (7) | 0.7165 (5) | -0.4208 (4) | 0.0511 (14) |
| H20 | 0.1956 | 0.7715 | -0.4763 | 0.061* |
| C21 | 0.0971 (7) | 0.7223 (5) | -0.3369 (4) | 0.0497 (14) |
| H21 | 0.0047 | 0.7800 | -0.3367 | 0.060* |
| C22 | 0.1207 (6) | 0.6426 (4) | -0.2533 (4) | 0.0441 (12) |
| H22 | 0.0435 | 0.6470 | -0.1972 | 0.053* |
| C23 | 0.0785 (6) | 0.7009 (4) | 0.3128 (4) | 0.0387 (11) |
| C24 | 0.1379 (7) | 0.8109 (4) | 0.2931 (4) | 0.0446 (13) |
| H24 | 0.2331 | 0.8126 | 0.2513 | 0.054* |
| C25 | 0.0570 (8) | 0.9177 (5) | 0.3352 (5) | 0.0553 (16) |
| H25 | 0.0978 | 0.9913 | 0.3217 | 0.066* |
| C26 | -0.0836 (8) | 0.9160 (5) | 0.3970 (5) | 0.0584 (16) |
| H26 | -0.1388 | 0.9888 | 0.4242 | 0.070* |
| C27 | -0.1433 (7) | 0.8071 (5) | 0.4188 (5) | 0.0572 (16) |
| H27 | -0.2372 | 0.8059 | 0.4621 | 0.069* |
| C28 | -0.0632 (6) | 0.6999 (4) | 0.3762 (4) | 0.0454 (13) |
| H28 | -0.1043 | 0.6264 | 0.3900 | 0.054* |
| N1 | 0.3158 (5) | 0.3512 (3) | 0.2036 (3) | 0.0363 (9) |
| N2 | 0.3676 (5) | 0.2954 (3) | -0.0021 (3) | 0.0416 (10) |
| O1 | 0.4253 (6) | 0.1240 (3) | 0.3391 (3) | 0.0647 (12) |
| O2 | 0.4774 (5) | -0.0635 (3) | 0.2768 (3) | 0.0553 (10) |
| O3 | 0.1859 (4) | 0.1193 (3) | 0.1493 (3) | 0.0448 (9) |
| O4 | 0.3050 (5) | -0.0021 (3) | 0.0238 (3) | 0.0571 (10) |
| Pb1 | 0.46035 (2) | 0.137456 (17) | 0.147460 (15) | 0.03580 (6) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-----------|-----------|-----------|------------|------------|--------------|
| C1 | 0.046 (3) | 0.047 (3) | 0.032 (3) | 0.007 (2) | -0.004 (2) | 0.000 (2) |
| C2 | 0.049 (3) | 0.034 (3) | 0.043 (3) | 0.006 (2) | 0.000 (3) | 0.002 (2) |
| C3 | 0.045 (3) | 0.037 (3) | 0.039 (3) | -0.007 (2) | -0.002 (2) | 0.001 (2) |
| C4 | 0.038 (3) | 0.046 (3) | 0.041 (3) | -0.006 (2) | -0.002 (2) | -0.009 (2) |
| C5 | 0.058 (3) | 0.033 (2) | 0.029 (3) | -0.005 (2) | -0.005 (2) | -0.001 (2) |
| C6 | 0.059 (3) | 0.039 (3) | 0.028 (3) | -0.001 (2) | -0.001 (2) | -0.008 (2) |
| C7 | 0.048 (3) | 0.031 (2) | 0.037 (3) | -0.007 (2) | -0.001 (2) | -0.006 (2) |
| C8 | 0.049 (3) | 0.033 (2) | 0.030 (3) | -0.004 (2) | -0.001 (2) | -0.004 (2) |
| C9 | 0.040 (3) | 0.032 (2) | 0.031 (3) | -0.004 (2) | -0.004 (2) | -0.0037 (19) |

| | | | | | | |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| C10 | 0.043 (3) | 0.033 (2) | 0.031 (3) | -0.005 (2) | -0.003 (2) | -0.0052 (19) |
| C11 | 0.075 (4) | 0.034 (3) | 0.035 (3) | 0.005 (3) | 0.002 (3) | -0.006 (2) |
| C12 | 0.069 (4) | 0.043 (3) | 0.027 (3) | -0.002 (3) | 0.002 (3) | -0.008 (2) |
| C13 | 0.045 (3) | 0.036 (2) | 0.034 (3) | -0.006 (2) | -0.001 (2) | -0.001 (2) |
| C14 | 0.046 (3) | 0.033 (2) | 0.029 (3) | -0.003 (2) | 0.000 (2) | -0.0039 (19) |
| C15 | 0.084 (4) | 0.033 (3) | 0.034 (3) | 0.001 (3) | 0.003 (3) | 0.001 (2) |
| C16 | 0.082 (4) | 0.029 (2) | 0.041 (3) | 0.003 (3) | -0.007 (3) | -0.002 (2) |
| C17 | 0.045 (3) | 0.039 (3) | 0.030 (3) | -0.012 (2) | -0.003 (2) | -0.003 (2) |
| C18 | 0.046 (3) | 0.052 (3) | 0.034 (3) | -0.003 (2) | 0.004 (2) | -0.003 (2) |
| C19 | 0.069 (4) | 0.064 (4) | 0.033 (3) | -0.015 (3) | 0.005 (3) | -0.002 (3) |
| C20 | 0.073 (4) | 0.049 (3) | 0.034 (3) | -0.015 (3) | -0.009 (3) | 0.005 (2) |
| C21 | 0.054 (4) | 0.046 (3) | 0.048 (4) | -0.001 (3) | -0.010 (3) | 0.005 (2) |
| C22 | 0.046 (3) | 0.046 (3) | 0.039 (3) | 0.000 (2) | -0.002 (2) | -0.005 (2) |
| C23 | 0.050 (3) | 0.039 (3) | 0.026 (3) | -0.001 (2) | -0.004 (2) | -0.008 (2) |
| C24 | 0.064 (4) | 0.036 (3) | 0.034 (3) | -0.009 (3) | 0.006 (3) | -0.005 (2) |
| C25 | 0.080 (4) | 0.031 (3) | 0.053 (4) | -0.003 (3) | 0.004 (3) | -0.008 (2) |
| C26 | 0.071 (4) | 0.041 (3) | 0.059 (4) | 0.012 (3) | -0.001 (3) | -0.015 (3) |
| C27 | 0.054 (4) | 0.055 (3) | 0.058 (4) | 0.006 (3) | 0.011 (3) | -0.004 (3) |
| C28 | 0.052 (3) | 0.036 (3) | 0.049 (3) | -0.007 (2) | -0.002 (3) | -0.003 (2) |
| N1 | 0.048 (2) | 0.0306 (19) | 0.030 (2) | -0.0017 (18) | -0.0039 (18) | -0.0042 (16) |
| N2 | 0.058 (3) | 0.034 (2) | 0.031 (2) | 0.0030 (19) | -0.004 (2) | -0.0079 (17) |
| O1 | 0.104 (3) | 0.046 (2) | 0.036 (2) | 0.020 (2) | -0.007 (2) | -0.0002 (17) |
| O2 | 0.077 (3) | 0.045 (2) | 0.043 (2) | -0.0011 (19) | -0.007 (2) | -0.0109 (17) |
| O3 | 0.050 (2) | 0.048 (2) | 0.037 (2) | -0.0123 (17) | -0.0021 (17) | -0.0103 (16) |
| O4 | 0.043 (2) | 0.065 (2) | 0.063 (3) | -0.0025 (19) | -0.003 (2) | -0.022 (2) |
| Pb1 | 0.03826 (11) | 0.03501 (10) | 0.03301 (10) | 0.00104 (7) | -0.00548 (7) | -0.00209 (7) |

Geometric parameters (Å, °)

| | | | |
|---------------------|------------|---------|-----------|
| C1—O2 | 1.234 (6) | C15—C16 | 1.351 (7) |
| C1—O1 | 1.260 (6) | C15—H15 | 0.9300 |
| C1—C2 | 1.499 (7) | C16—H16 | 0.9300 |
| C2—C2 ⁱ | 1.321 (10) | C17—C18 | 1.394 (6) |
| C2—H2 | 0.9300 | C17—C22 | 1.400 (7) |
| C3—O4 | 1.242 (6) | C18—C19 | 1.379 (7) |
| C3—O3 | 1.277 (6) | C18—H18 | 0.9300 |
| C3—C4 | 1.477 (7) | C19—C20 | 1.370 (8) |
| C4—C4 ⁱⁱ | 1.319 (10) | C19—H19 | 0.9300 |
| C4—H4 | 0.9300 | C20—C21 | 1.378 (7) |
| C5—N1 | 1.324 (6) | C20—H20 | 0.9300 |
| C5—C6 | 1.390 (6) | C21—C22 | 1.380 (7) |
| C5—H5 | 0.9300 | C21—H21 | 0.9300 |
| C6—C7 | 1.373 (7) | C22—H22 | 0.9300 |
| C6—H6 | 0.9300 | C23—C24 | 1.382 (7) |
| C7—C8 | 1.433 (6) | C23—C28 | 1.391 (7) |
| C7—C23 | 1.487 (6) | C24—C25 | 1.375 (7) |
| C8—C9 | 1.407 (6) | C24—H24 | 0.9300 |
| C8—C16 | 1.430 (7) | C25—C26 | 1.373 (8) |
| C9—N1 | 1.363 (6) | C25—H25 | 0.9300 |

supplementary materials

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|-------------------------|-----------|-------------|-----------|
| C9—C10 | 1.439 (6) | C26—C27 | 1.377 (8) |
| C10—N2 | 1.361 (6) | C26—H26 | 0.9300 |
| C10—C14 | 1.419 (6) | C27—C28 | 1.379 (7) |
| C11—N2 | 1.318 (6) | C27—H27 | 0.9300 |
| C11—C12 | 1.385 (7) | C28—H28 | 0.9300 |
| C11—H11 | 0.9300 | Pb1—N1 | 2.590 (4) |
| C12—C13 | 1.374 (6) | Pb1—N2 | 2.645 (4) |
| C12—H12 | 0.9300 | Pb1—O1 | 2.449 (4) |
| C13—C14 | 1.426 (6) | Pb1—O2 | 2.740 (4) |
| C13—C17 | 1.483 (6) | Pb1—O3 | 2.313 (3) |
| C14—C15 | 1.431 (6) | Pb1—O4 | 2.705 (4) |
| O2—C1—O1 | 124.1 (5) | C20—C19—H19 | 119.9 |
| O2—C1—C2 | 120.2 (4) | C18—C19—H19 | 119.9 |
| O1—C1—C2 | 115.6 (4) | C19—C20—C21 | 120.0 (5) |
| C2 ⁱ —C2—C1 | 125.3 (6) | C19—C20—H20 | 120.0 |
| C2 ⁱ —C2—H2 | 117.3 | C21—C20—H20 | 120.0 |
| C1—C2—H2 | 117.3 | C20—C21—C22 | 120.1 (5) |
| O4—C3—O3 | 121.9 (5) | C20—C21—H21 | 120.0 |
| O4—C3—C4 | 121.8 (5) | C22—C21—H21 | 120.0 |
| O3—C3—C4 | 116.3 (4) | C21—C22—C17 | 121.1 (5) |
| C4 ⁱⁱ —C4—C3 | 124.6 (6) | C21—C22—H22 | 119.4 |
| C4 ⁱⁱ —C4—H4 | 117.7 | C17—C22—H22 | 119.4 |
| C3—C4—H4 | 117.7 | C24—C23—C28 | 119.1 (4) |
| N1—C5—C6 | 123.4 (4) | C24—C23—C7 | 123.0 (5) |
| N1—C5—H5 | 118.3 | C28—C23—C7 | 117.9 (4) |
| C6—C5—H5 | 118.3 | C25—C24—C23 | 120.3 (5) |
| C7—C6—C5 | 120.4 (5) | C25—C24—H24 | 119.8 |
| C7—C6—H6 | 119.8 | C23—C24—H24 | 119.8 |
| C5—C6—H6 | 119.8 | C26—C25—C24 | 120.3 (5) |
| C6—C7—C8 | 117.5 (4) | C26—C25—H25 | 119.9 |
| C6—C7—C23 | 119.5 (4) | C24—C25—H25 | 119.9 |
| C8—C7—C23 | 123.0 (4) | C25—C26—C27 | 120.2 (5) |
| C9—C8—C16 | 118.7 (4) | C25—C26—H26 | 119.9 |
| C9—C8—C7 | 118.1 (4) | C27—C26—H26 | 119.9 |
| C16—C8—C7 | 123.1 (4) | C26—C27—C28 | 119.7 (5) |
| N1—C9—C8 | 122.4 (4) | C26—C27—H27 | 120.2 |
| N1—C9—C10 | 117.8 (4) | C28—C27—H27 | 120.2 |
| C8—C9—C10 | 119.7 (4) | C27—C28—C23 | 120.4 (5) |
| N2—C10—C14 | 122.3 (4) | C27—C28—H28 | 119.8 |
| N2—C10—C9 | 117.2 (4) | C23—C28—H28 | 119.8 |
| C14—C10—C9 | 120.5 (4) | C5—N1—C9 | 118.1 (4) |
| N2—C11—C12 | 123.1 (5) | C5—N1—Pb1 | 119.9 (3) |
| N2—C11—H11 | 118.5 | C9—N1—Pb1 | 122.0 (3) |
| C12—C11—H11 | 118.5 | C11—N2—C10 | 118.1 (4) |
| C13—C12—C11 | 121.6 (5) | C11—N2—Pb1 | 121.4 (3) |
| C13—C12—H12 | 119.2 | C10—N2—Pb1 | 120.3 (3) |
| C11—C12—H12 | 119.2 | C1—O1—Pb1 | 99.2 (3) |
| C12—C13—C14 | 116.6 (4) | C1—O2—Pb1 | 86.1 (3) |

supplementary materials

| | | | |
|-------------|-----------|-----------|-------------|
| C12—C13—C17 | 120.1 (4) | C3—O3—Pb1 | 102.0 (3) |
| C14—C13—C17 | 123.3 (4) | C3—O4—Pb1 | 84.5 (3) |
| C10—C14—C13 | 118.4 (4) | O3—Pb1—O1 | 85.17 (14) |
| C10—C14—C15 | 117.1 (4) | O3—Pb1—N1 | 75.35 (12) |
| C13—C14—C15 | 124.5 (4) | O1—Pb1—N1 | 75.70 (12) |
| C16—C15—C14 | 122.6 (5) | O3—Pb1—N2 | 79.49 (13) |
| C16—C15—H15 | 118.7 | O1—Pb1—N2 | 137.38 (12) |
| C14—C15—H15 | 118.7 | N1—Pb1—N2 | 62.03 (12) |
| C15—C16—C8 | 121.0 (5) | O3—Pb1—O4 | 51.31 (11) |
| C15—C16—H16 | 119.5 | O1—Pb1—O4 | 120.44 (14) |
| C8—C16—H16 | 119.5 | N1—Pb1—O4 | 118.64 (12) |
| C18—C17—C22 | 117.1 (5) | N2—Pb1—O4 | 78.77 (12) |
| C18—C17—C13 | 119.0 (5) | O3—Pb1—O2 | 84.60 (12) |
| C22—C17—C13 | 123.8 (4) | O1—Pb1—O2 | 49.85 (11) |
| C19—C18—C17 | 121.5 (5) | N1—Pb1—O2 | 123.32 (11) |
| C19—C18—H18 | 119.2 | N2—Pb1—O2 | 161.17 (12) |
| C17—C18—H18 | 119.2 | O4—Pb1—O2 | 83.44 (12) |
| C20—C19—C18 | 120.1 (5) | | |

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

supplementary materials

Fig. 1

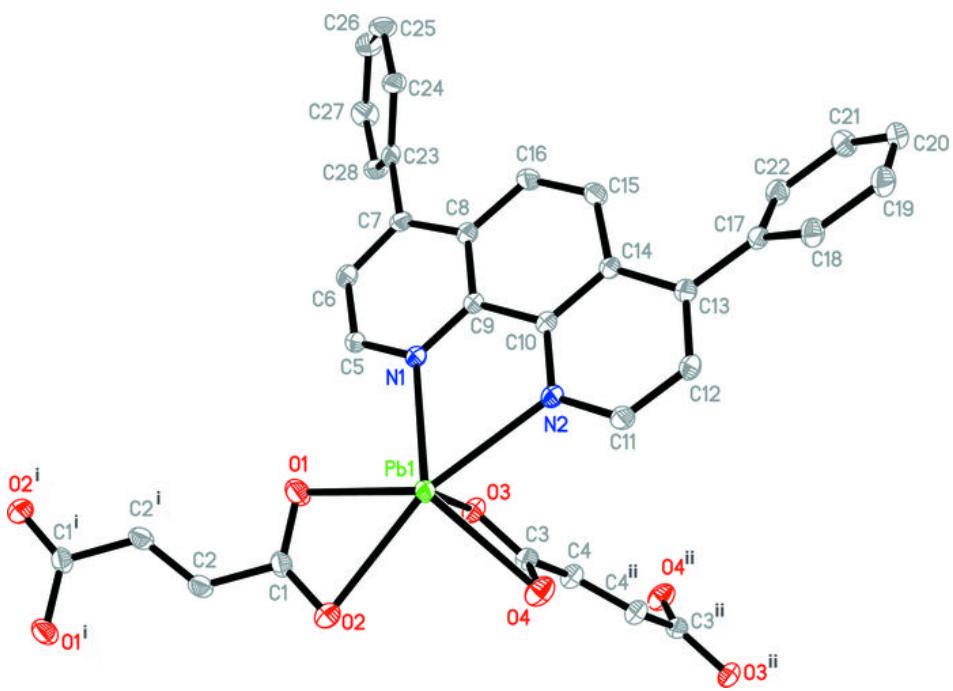
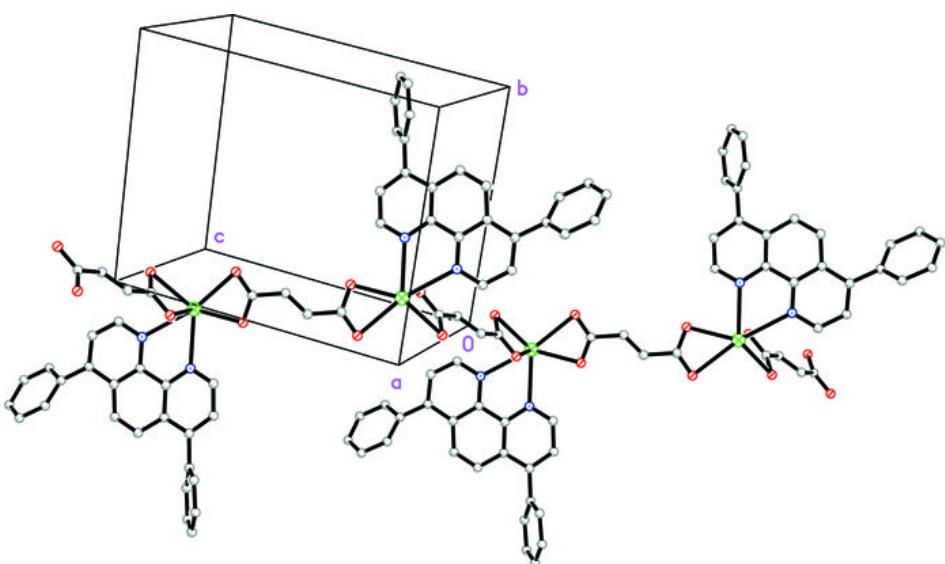


Fig. 2



supplementary materials

Fig. 3

