

Bis(imidazo[4,5-*f*][1,10]phenanthroline)-dinitratolead(II)

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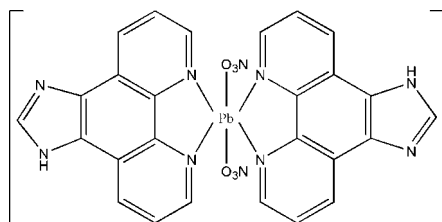
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Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.026; wR factor = 0.064; data-to-parameter ratio = 12.3.

In the title compound, $[\text{Pb}(\text{NO}_3)_2(\text{C}_{13}\text{H}_8\text{N}_4)_2]$, the Pb^{II} atom (site symmetry 2) is hexacoordinated by four N atoms from two N,N' -bidentate imidazo[4,5-*f*][1,10]phenanthroline (*L*) ligands and two O atoms from two weakly coordinated nitrate ions [$\text{Pb}-\text{O} = 2.872$ (5) Å] in an irregular arrangement, which may be ascribed to the stereochemically active lone pair of electrons on the metal ion. In the crystal, intermolecular bifurcated $\text{N}-\text{H}\cdots(\text{O},\text{O})$ hydrogen bonds connect the molecules into chains propagating along [100]. Adjacent chains interact by strong aromatic $\pi-\pi$ stacking interactions, with a centroid-centroid distance of 3.483 (2) Å.

Related literature

For the ligand synthesis, see: Steck & Day (1943). For background, see: Che *et al.* (2006, 2008); Thomas *et al.* (2008); Xu *et al.* (2008).



Experimental

Crystal data

$[\text{Pb}(\text{NO}_3)_2(\text{C}_{13}\text{H}_8\text{N}_4)_2]$
 $M_r = 771.68$
 Monoclinic, $C2/c$
 $a = 19.203$ (4) Å

$b = 7.3948$ (15) Å
 $c = 17.392$ (4) Å
 $\beta = 100.48$ (3)°
 $V = 2428.5$ (8) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 7.02$ mm⁻¹

$T = 292$ (2) K
 $0.56 \times 0.22 \times 0.11$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\text{min}} = 0.165$, $T_{\text{max}} = 0.453$

10060 measured reflections
 2402 independent reflections
 2170 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.064$
 $S = 1.05$
 2402 reflections

195 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.89$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.63$ e Å⁻³

Table 1

Selected bond lengths (Å).

Pb—N1	2.540 (3)	Pb—O1	2.872 (5)
Pb—N2	2.606 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N3}-\text{H3A}\cdots\text{O3}^{\text{i}}$	0.86	2.19	3.030 (6)	165
$\text{N3}-\text{H3A}\cdots\text{O2}^{\text{i}}$	0.86	2.35	3.059 (6)	140

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: HB2871).

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supplementary materials

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Bis(imidazo[4,5-*f*][1,10]phenanthroline)dinitratolead(II)

C.-X. Li, X.-L. Zha, C.-B. Liu, X.-Y. Li and G.-B. Che

Comment

1,10-Phenanthroline and its derivatives are very important heteroaromatic N-donor chelating ligands for the construction of coordination compounds. (Che *et al.*, 2008; Xu *et al.*, 2008; Che *et al.*, 2006). Whereas, only a handful of supramolecular architectures based on imidazo[4,5-*f*][1,10]phenanthroline ligand (*L*) have been described (Thomas *et al.*, 2008). The present attempt at synthesizing a new lead complex with *L* ligand gave the title complex, [Pb(C₁₃H₈N₄)₂(NO₃)₂] (I), whose structure is reported here.

In the title compound I, each Pb atom is hexacoordinated by four N atoms (N1, N1ⁱ, N2, N2ⁱ) from two *L* ligand and two O (O1, O1ⁱ) atoms from two NO₃⁻ ions (Fig. 1). The Pb—N bond lengths are normal (Table 1), however, O1 and O1ⁱ could be described as being semicoordinated with long Pb—O lengths of 2.872 (5) Å. The PbN₄O₂ coordination polyhedron approximates a distorted octahedral configuration.

In the crystal structure, the mononuclear complex molecules are linked *via* intermolecular N—H⋯O and N—H⋯N hydrogen bonds (Table 2) forming one-dimensional chains along [100] (Fig. 2). Withal, one-dimensional chains are stabilized by π-π interactions between the *L* rings planes of neighboring molecules with distances of 3.442 Å (Fig. 3), leading to a three-dimensional network.

Experimental

The *L* ligand was synthesized according to the literature method of Steck & Day (1943). A mixture of Pb(NO₃)₂ (0.5 mmol) and the ligand (1 mmol) was dissolved in 15 ml distilled water, then followed by addition of NaOH until the pH value of the system approximately 6.0. Finally the resulting solution was sealed into a 23 ml Teflon-lined stainless steel autoclave and heated at 413 K for 3 d under autogenous pressure. The reaction autoclave slowly cooled to room temperature, yielding yellow block-like crystals of (I) in 62% yield based on Pb.

Refinement

All H atoms on C atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The hydrogen atoms of water molecules were located from difference Fourier maps and their positions and U_{iso} values were refined freely.

Figures

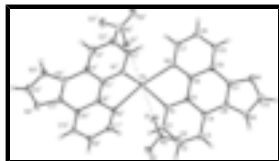


Fig. 1. A view of (I). Displacement ellipsoids are drawn at the 30% probability level (arbitrary spheres for the H atoms). Dashed lines indicate semicoordinated bonds [Symmetry code: (i) $-x + 1, y, -z + 1/2$].

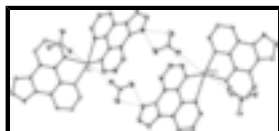


Fig. 2. A packing diagram of the title compound, showing a one-dimensional chain-like structure generated by the intermolecular hydrogen bonding. H atoms have been omitted [Symmetry code: (A) $-x + 1, -y - 1/2, z - 1/2$].

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Crystal data

$[\text{Pb}(\text{NO}_3)_2(\text{C}_{13}\text{H}_8\text{N}_4)_2]$

$M_r = 771.68$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 19.203\ (4)\ \text{\AA}$

$b = 7.3948\ (15)\ \text{\AA}$

$c = 17.392\ (4)\ \text{\AA}$

$\beta = 100.48\ (3)^\circ$

$V = 2428.5\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 1488$

$D_x = 2.111\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3904 reflections

$\theta = 2.2\text{--}26.1^\circ$

$\mu = 7.02\ \text{mm}^{-1}$

$T = 292\ \text{K}$

Block, yellow

$0.56 \times 0.22 \times 0.11\ \text{mm}$

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2002)

$T_{\min} = 0.165$, $T_{\max} = 0.453$

10060 measured reflections

2402 independent reflections

2170 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$

$\theta_{\max} = 26.1^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -23 \rightarrow 23$

$k = -9 \rightarrow 9$

$l = -20 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

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

$wR(F^2) = 0.064$

$S = 1.05$

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

$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2]$

2402 reflections
195 parameters
0 restraints

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.005$$

$$\Delta\rho_{\max} = 0.89 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.63 \text{ e } \text{\AA}^{-3}$$

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	0.3676 (2)	-0.2885 (6)	0.1841 (2)	0.0361 (10)
H1	0.3576	-0.2644	0.2334	0.043*
C2	0.3181 (3)	-0.3843 (7)	0.1316 (3)	0.0472 (12)
H2	0.2772	-0.4283	0.1466	0.057*
C3	0.3303 (2)	-0.4130 (6)	0.0577 (3)	0.0411 (11)
H3	0.2973	-0.4749	0.0214	0.049*
C4	0.3927 (2)	-0.3486 (5)	0.0372 (2)	0.0270 (9)
C5	0.4423 (2)	-0.2606 (5)	0.0951 (2)	0.0242 (8)
C6	0.5099 (2)	-0.1997 (5)	0.0783 (2)	0.0242 (8)
C7	0.6190 (2)	-0.0678 (6)	0.1228 (3)	0.0329 (9)
H7	0.6505	-0.0117	0.1627	0.040*
C8	0.6394 (2)	-0.0921 (6)	0.0507 (2)	0.0354 (10)
H8	0.6845	-0.0580	0.0439	0.043*
C9	0.4103 (2)	-0.3613 (5)	-0.0386 (2)	0.0284 (9)
C10	0.5268 (2)	-0.2217 (5)	0.0028 (2)	0.0251 (8)
C11	0.4729 (2)	-0.2999 (5)	-0.0565 (2)	0.0272 (9)
C12	0.4129 (3)	-0.3952 (6)	-0.1631 (2)	0.0386 (11)
H12	0.3985	-0.4253	-0.2155	0.046*
C13	0.5931 (2)	-0.1660 (5)	-0.0100 (2)	0.0296 (9)
H13	0.6055	-0.1790	-0.0590	0.035*
N5	0.2959 (2)	0.1066 (5)	0.1716 (2)	0.0387 (9)
O2	0.2564 (2)	0.0783 (8)	0.2184 (3)	0.0874 (14)
N1	0.42843 (17)	-0.2294 (4)	0.16763 (18)	0.0268 (7)
N2	0.55658 (17)	-0.1214 (4)	0.13726 (18)	0.0262 (7)
N3	0.3723 (2)	-0.4235 (5)	-0.1083 (2)	0.0359 (8)
H3A	0.3307	-0.4711	-0.1156	0.043*
N4	0.4751 (2)	-0.3210 (4)	-0.13538 (18)	0.0341 (8)
O1	0.35914 (17)	0.1449 (5)	0.19411 (19)	0.0508 (9)

supplementary materials

O3	0.2706 (2)	0.0901 (7)	0.1014 (2)	0.0747 (12)
Pb	0.5000	0.01345 (3)	0.2500	0.02722 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.034 (3)	0.047 (2)	0.030 (2)	-0.005 (2)	0.013 (2)	0.0003 (19)
C2	0.032 (3)	0.068 (3)	0.044 (3)	-0.018 (2)	0.013 (2)	-0.006 (2)
C3	0.032 (3)	0.055 (3)	0.035 (3)	-0.017 (2)	0.003 (2)	-0.008 (2)
C4	0.024 (2)	0.0303 (19)	0.026 (2)	-0.0021 (16)	0.0025 (17)	0.0008 (16)
C5	0.022 (2)	0.0264 (18)	0.024 (2)	0.0013 (16)	0.0046 (17)	0.0018 (15)
C6	0.024 (2)	0.0247 (18)	0.023 (2)	0.0014 (15)	0.0024 (17)	0.0016 (15)
C7	0.025 (2)	0.041 (2)	0.031 (2)	-0.0063 (19)	0.0020 (19)	-0.0061 (18)
C8	0.027 (3)	0.046 (2)	0.035 (3)	-0.005 (2)	0.010 (2)	0.0001 (19)
C9	0.026 (2)	0.032 (2)	0.025 (2)	-0.0007 (17)	-0.0023 (18)	-0.0004 (16)
C10	0.023 (2)	0.0264 (19)	0.025 (2)	0.0021 (17)	0.0033 (16)	0.0028 (15)
C11	0.032 (2)	0.0283 (19)	0.021 (2)	0.0018 (17)	0.0034 (17)	0.0013 (15)
C12	0.047 (3)	0.043 (2)	0.022 (2)	0.000 (2)	-0.003 (2)	-0.0042 (18)
C13	0.030 (2)	0.041 (2)	0.020 (2)	0.0006 (18)	0.0111 (18)	-0.0019 (16)
N5	0.035 (2)	0.051 (2)	0.030 (2)	0.0030 (18)	0.0059 (18)	-0.0036 (16)
O2	0.048 (3)	0.171 (4)	0.049 (3)	-0.009 (3)	0.024 (2)	0.008 (3)
N1	0.0184 (18)	0.0355 (17)	0.0273 (18)	-0.0007 (14)	0.0065 (14)	0.0000 (13)
N2	0.0235 (19)	0.0331 (17)	0.0217 (17)	-0.0045 (14)	0.0031 (14)	-0.0022 (13)
N3	0.031 (2)	0.0431 (19)	0.032 (2)	-0.0073 (17)	0.0007 (17)	-0.0029 (16)
N4	0.041 (2)	0.0406 (19)	0.0202 (18)	-0.0029 (17)	0.0032 (16)	-0.0022 (14)
O1	0.0265 (19)	0.068 (2)	0.053 (2)	-0.0076 (16)	-0.0049 (16)	-0.0099 (17)
O3	0.054 (3)	0.133 (4)	0.034 (2)	-0.016 (3)	-0.0015 (19)	-0.009 (2)
Pb	0.02505 (15)	0.03545 (14)	0.02129 (13)	0.000	0.00458 (9)	0.000

Geometric parameters (\AA , $^\circ$)

C1—N1	1.327 (5)	C9—N3	1.376 (5)
C1—C2	1.387 (6)	C10—C13	1.393 (6)
C1—H1	0.9300	C10—C11	1.442 (5)
C2—C3	1.364 (6)	C11—N4	1.390 (5)
C2—H2	0.9300	C12—N4	1.323 (5)
C3—C4	1.396 (6)	C12—N3	1.353 (6)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.413 (5)	C13—H13	0.9300
C4—C9	1.423 (5)	N5—O2	1.227 (5)
C5—N1	1.355 (5)	N5—O3	1.235 (5)
C5—C6	1.453 (5)	N5—O1	1.239 (5)
C6—N2	1.362 (5)	Pb—N1	2.540 (3)
C6—C10	1.418 (5)	Pb—N2	2.606 (3)
C7—N2	1.329 (5)	Pb—O1	2.872 (5)
C7—C8	1.392 (6)	N3—H3A	0.8600
C7—H7	0.9300	Pb—N1 ⁱ	2.540 (3)
C8—C13	1.365 (6)	Pb—N2 ⁱ	2.606 (3)

C8—H8	0.9300	O1—Pb ⁱ	2.872 (5)
C9—C11	1.372 (6)		
N1—C1—C2	123.5 (4)	C9—C11—N4	111.8 (4)
N1—C1—H1	118.3	C9—C11—C10	121.0 (3)
C2—C1—H1	118.3	N4—C11—C10	127.1 (4)
C3—C2—C1	119.0 (4)	N4—C12—N3	113.9 (4)
C3—C2—H2	120.5	N4—C12—H12	123.0
C1—C2—H2	120.5	N3—C12—H12	123.0
C2—C3—C4	119.4 (4)	C8—C13—C10	118.7 (4)
C2—C3—H3	120.3	C8—C13—H13	120.6
C4—C3—H3	120.3	C10—C13—H13	120.6
C3—C4—C5	118.3 (4)	O2—N5—O3	117.3 (4)
C3—C4—C9	125.0 (4)	O2—N5—O1	121.2 (4)
C5—C4—C9	116.6 (4)	O3—N5—O1	121.4 (4)
N1—C5—C4	121.4 (3)	C1—N1—C5	118.4 (3)
N1—C5—C6	118.0 (3)	C1—N1—Pb	121.2 (3)
C4—C5—C6	120.7 (3)	C5—N1—Pb	117.9 (2)
N2—C6—C10	121.1 (4)	C7—N2—C6	118.5 (3)
N2—C6—C5	118.0 (3)	C7—N2—Pb	122.8 (3)
C10—C6—C5	120.9 (3)	C6—N2—Pb	115.0 (2)
N2—C7—C8	122.9 (4)	C12—N3—C9	106.7 (4)
N2—C7—H7	118.6	C12—N3—H3A	126.7
C8—C7—H7	118.6	C9—N3—H3A	126.7
C13—C8—C7	119.9 (4)	C12—N4—C11	102.6 (4)
C13—C8—H8	120.1	N1—Pb—N1 ⁱ	90.01 (14)
C7—C8—H8	120.1	N1—Pb—N2 ⁱ	84.09 (10)
C11—C9—N3	105.0 (3)	N1 ⁱ —Pb—N2 ⁱ	64.03 (10)
C11—C9—C4	123.6 (4)	N1—Pb—N2	64.03 (10)
N3—C9—C4	131.4 (4)	N1 ⁱ —Pb—N2	84.09 (10)
C13—C10—C6	118.9 (4)	N2 ⁱ —Pb—N2	135.02 (14)
C13—C10—C11	124.2 (3)	O1—Pb—N1	70.65 (10)
C6—C10—C11	116.9 (4)	O1—Pb—N2	111.75 (10)
N1—C1—C2—C3	-3.2 (7)	C11—C10—C13—C8	179.3 (4)
C1—C2—C3—C4	1.2 (7)	C2—C1—N1—C5	1.6 (6)
C2—C3—C4—C5	2.1 (7)	C2—C1—N1—Pb	163.1 (4)
C2—C3—C4—C9	-176.7 (4)	C4—C5—N1—C1	2.0 (5)
C3—C4—C5—N1	-3.8 (6)	C6—C5—N1—C1	-178.4 (3)
C9—C4—C5—N1	175.1 (3)	C4—C5—N1—Pb	-160.2 (3)
C3—C4—C5—C6	176.6 (4)	C6—C5—N1—Pb	19.5 (4)
C9—C4—C5—C6	-4.5 (5)	C8—C7—N2—C6	-1.5 (6)
N1—C5—C6—N2	2.6 (5)	C8—C7—N2—Pb	-158.6 (3)
C4—C5—C6—N2	-177.7 (3)	C10—C6—N2—C7	-0.9 (5)
N1—C5—C6—C10	-177.6 (3)	C5—C6—N2—C7	178.9 (3)
C4—C5—C6—C10	2.0 (5)	C10—C6—N2—Pb	157.9 (3)
N2—C7—C8—C13	3.2 (7)	C5—C6—N2—Pb	-22.3 (4)
C3—C4—C9—C11	-178.6 (4)	N4—C12—N3—C9	0.6 (5)
C5—C4—C9—C11	2.6 (6)	C11—C9—N3—C12	-0.5 (4)

supplementary materials

C3—C4—C9—N3	4.8 (7)	C4—C9—N3—C12	176.5 (4)
C5—C4—C9—N3	-173.9 (4)	N3—C12—N4—C11	-0.4 (5)
N2—C6—C10—C13	1.6 (5)	C9—C11—N4—C12	0.1 (4)
C5—C6—C10—C13	-178.1 (3)	C10—C11—N4—C12	-178.9 (4)
N2—C6—C10—C11	-177.7 (3)	C1—N1—Pb—N1 ⁱ	93.6 (3)
C5—C6—C10—C11	2.5 (5)	C5—N1—Pb—N1 ⁱ	-104.8 (3)
N3—C9—C11—N4	0.2 (4)	C1—N1—Pb—N2 ⁱ	29.7 (3)
C4—C9—C11—N4	-177.1 (4)	C5—N1—Pb—N2 ⁱ	-168.7 (3)
N3—C9—C11—C10	179.3 (3)	C1—N1—Pb—N2	177.0 (3)
C4—C9—C11—C10	2.0 (6)	C5—N1—Pb—N2	-21.4 (2)
C13—C10—C11—C9	176.2 (4)	C7—N2—Pb—N1	179.9 (3)
C6—C10—C11—C9	-4.5 (5)	C6—N2—Pb—N1	22.2 (2)
C13—C10—C11—N4	-4.9 (6)	C7—N2—Pb—N1 ⁱ	-87.2 (3)
C6—C10—C11—N4	174.4 (4)	C6—N2—Pb—N1 ⁱ	115.1 (3)
C7—C8—C13—C10	-2.3 (6)	C7—N2—Pb—N2 ⁱ	-130.6 (3)
C6—C10—C13—C8	0.0 (6)	C6—N2—Pb—N2 ⁱ	71.7 (2)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3A \cdots O3 ⁱⁱ	0.86	2.19	3.030 (6)	165
N3—H3A \cdots O2 ⁱⁱ	0.86	2.35	3.059 (6)	140

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

Fig. 1

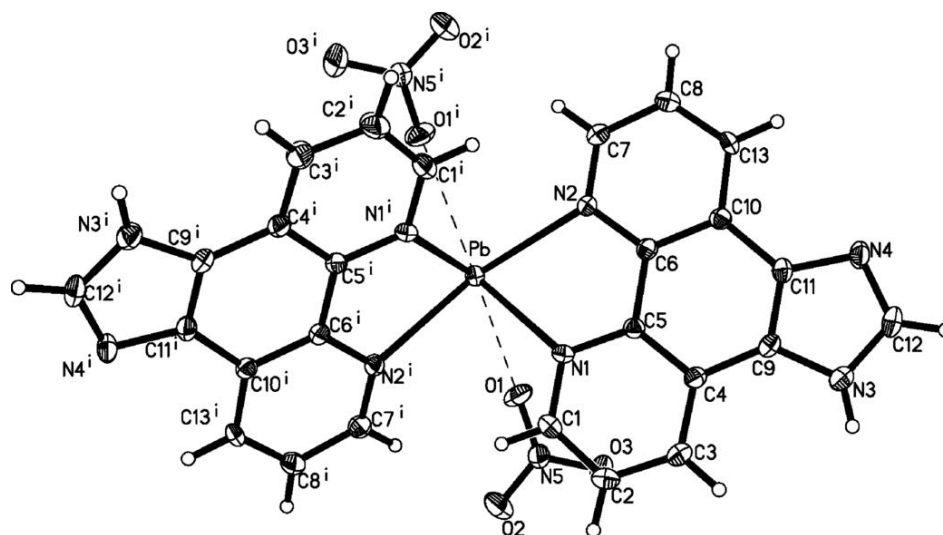


Fig. 2

