

Bis(3-acetyl-6-methyl-2-oxo-2H-pyran-4-olato)bis(dimethyl sulfoxide)nickel(II)

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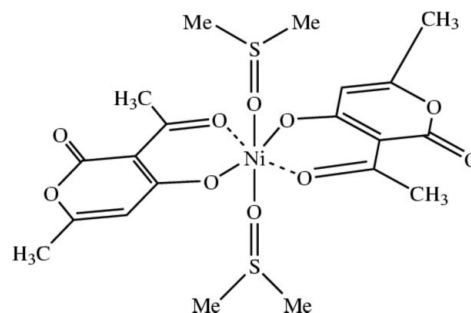
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.098; data-to-parameter ratio = 16.8.

In the title compound, $[\text{Ni}(\text{C}_8\text{H}_7\text{O}_4)_2(\text{CH}_3)_2\text{SO}]_2$, the Ni^{II} atom is located on a crystallographic centre of symmetry and has a distorted octahedral coordination geometry of type MO_6 . The bidentate dehydroacetic acid (DHA) ligands occupy the equatorial plane of the complex in a *trans* configuration, and the dimethyl sulfoxide (DMSO) ligands are weakly coordinated through their O atoms in the axial positions.

Related literature

3-Acetyl-4-hydroxy-6-methyl-2-oxo-2H-pyran (dehydroacetic acid) (Arndt *et al.*, 1936) is a versatile starting material for the synthesis of a wide variety of heterocyclic ring systems (Tan & Ang, 1988). It has been shown to possess modest antifungal properties, see: Rao *et al.* (1978). For natural fungicides possessing structures analogous to 5,6-dihydrodehydroacetic acid, see: Bartels-Keith (1960); Miyakado *et al.* (1982); Ayer *et al.* (1988). The complexes of DHA with copper and with several other transition metal cations are fungistatic, see: Rao *et al.* (1978). For the nickel-DHA complex, see: Casabò *et al.* (1987). The configuration of the complex molecule is similar to that found in $[\text{Zn}(\text{DHA})_2 \cdot 2(\text{DMSO})]$ and $[\text{Cd}(\text{DHA})_2 \cdot 2(\text{DMSO})]$ (Zucolotto Chalaça *et al.*, 2002), $[\text{Cu}(\text{DHA})_2 \cdot 2(\text{DMSO})]$ (Djedouani *et al.*, 2006) and bis(4,6-dibromo-2-formylphenolato- $\kappa^2\text{O},\text{O}'$)-bis(dimethyl sulfoxide)nickel(II) (Zhang *et al.*, 2007). For Ni—O_{DMSO} distances in similar structures, see: Ma *et al.* (2003); Tahir *et al.* (2007); Zhang *et al.* (2007).



Experimental

Crystal data

$[\text{Ni}(\text{C}_8\text{H}_7\text{O}_4)_2(\text{C}_2\text{H}_6\text{OS})_2]$
 $M_r = 549.24$
 Monoclinic, $P2_1/c$
 $a = 11.3850$ (10) Å
 $b = 6.2833$ (4) Å
 $c = 19.7434$ (15) Å
 $\beta = 123.525$ (6)°

$V = 1177.40$ (16) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.05$ mm⁻¹
 $T = 100$ K
 $0.25 \times 0.15 \times 0.10$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.902$, $T_{\max} = 0.902$

14084 measured reflections
 2628 independent reflections
 1962 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.071$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.098$
 $S = 1.11$
 2628 reflections

156 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.55$ e Å⁻³
 $\Delta\rho_{\min} = -0.97$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ni1—O2	1.9849 (16)	Ni1—O1	2.1255 (18)
Ni1—O3	2.0159 (15)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C1}-\text{H1B}\cdots\text{O4}^{\text{ii}}$	0.96	2.55	3.384 (4)	145
$\text{C2}-\text{H2B}\cdots\text{O4}^{\text{ii}}$	0.96	2.53	3.370 (4)	146
$\text{C2}-\text{H2C}\cdots\text{O2}^{\text{iii}}$	0.96	2.46	3.378 (4)	160

Symmetry codes: (ii) $x + 1, -y + \frac{3}{2}, z + \frac{1}{2}$; (iii) $x, y + 1, z$.

Data collection: COLLECT (Nonius, 2002); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg, 1998) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: WinGX (Farrugia, 1999) and PARST (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2559).

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supplementary materials

Acta Cryst. (2009). E65, m1205-m1206 [doi:10.1107/S1600536809034655]

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Comment

3-Acetyl-4-hydroxy-6-methyl-2-oxo-2H-pyran (dehydroacetic acid) (Arndt *et al.*, 1936) is a versatile starting material for the synthesis of a wide variety of heterocyclic ring systems (Tan & Ang, 1988). It has been shown to possess modest antifungal properties (Rao *et al.*, 1978). The importance of similar pyrones as potential fungicides is reinforced by the existence of several natural fungicides possessing structures analogous to 5,6-dihydrodehydroacetic acid, such as alternaric acid (Bartels-Keith, 1960), the podoblastins (Miyakado *et al.*, 1982) and lachnelluloic acid (Ayer *et al.*, 1988). Also, it has been shown that the complexes of DHA with copper and with several other transition metal cations are fungistatic (Rao *et al.*, 1978). This has motivated our study of the structural characterization of complexes of dehydroacetic acid. The complex of DHA with nickel was previously reported by Casabò *et al.* (1987), but their characterization of the compound was based only on thermal and elemental analysis, and on IR and NMR spectroscopy.

We present here the crystal structure determination of the title complex, [Ni(DHA)₂.2(DMSO)], (I) (DMSO = dimethylsulfoxide). The nature of the title compound, (I), was established by an X-ray structure determination and is shown in Fig. 1

The Ni atom lies on a crystallographic centre of symmetry with the ligands bonded to nickel in an all-*trans* fashion. The configuration of the complex molecule is similar to that found in [Zn(DHA)₂. 2(DMSO); Cd(DHA)₂.2(DMSO)] (Zucolotto Chalaça *et al.*, 2002), [Cu(DHA)₂. 2(DMSO)] (Djedouani *et al.*, 2006), with (DHA: dehydroacetic acid) and Bis(4,6-dibromo-2-formylphenolato-κ² O,O')-bis(dimethyl sulfoxide)nickel(II), [Ni(C₇H₃Br₂O₂)₂(C₂H₆OS)₂] (Zhang *et al.*, 2007).

The coordination polyhedron around the Ni atom is a slightly distorted octahedron (Table 1), with the O atoms of the DMSO groups in axial positions; and the Ni—O_{DMSO} distance is in agreement with literature values: [2.1139 (12) Å - 1.9897 (13) Å (Tahir *et al.*, 2007), 1.998 (3) Å - 2.105 (3) Å (Zhang *et al.* 2007), 2.030 (2) Å - 2.057 (2) Å (Ma *et al.*, 2003)].

The orientation of the DMSO molecule can be described by the torsion angles O3—Ni—O1—S [43.32 (4) °] and O2—Ni—O1—S [-137.70 (4) °]. The packing of (I) is stabilized by weak intermolecular C—H...O hydrogen bonds (Table 2) which form a three-dimensional network (Fig. 2).

Experimental

Compound (I) was prepared by the reaction of dehydroacetic acid with nickel (II) chloride hexahydrate in the presence of sodium acetate (Casabò *et al.* 1987). Crystals of (I) were grown by slow evaporation of a dimethylsulfoxide solution..

Refinement

H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, but were allowed to rotate freely about the C—C bonds.

Figures

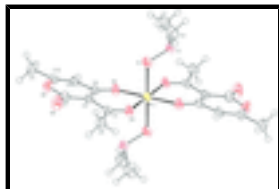


Fig. 1. The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

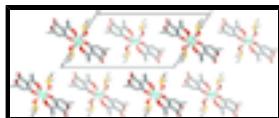


Fig. 2. The crystal packing of (I); Hydrogen atoms have been omitted for clarity.

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Crystal data

[Ni(C₈H₇O₄)₂(C₂H₆OS)₂]

$M_r = 549.24$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.3850$ (10) Å

$b = 6.2833$ (4) Å

$c = 19.7434$ (15) Å

$\beta = 123.525$ (6)°

$V = 1177.40$ (16) Å³

$Z = 2$

$F_{000} = 572$

$D_x = 1.549$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2190 reflections

$\theta = 2.8$ – 27.3 °

$\mu = 1.05$ mm⁻¹

$T = 100$ K

Plates, colourless

$0.25 \times 0.15 \times 0.1$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed X-ray tube

Monochromator: graphite

$T = 100$ K

φ scans, and ω scans with κ offsets

Absorption correction: multi-scan
(SADABS; Bruker, 1998)

$T_{\min} = 0.902$, $T_{\max} = 0.902$

14084 measured reflections

2628 independent reflections

1962 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.071$

$\theta_{\max} = 27.5$ °

$\theta_{\min} = 3.5$ °

$h = -14 \rightarrow 14$

$k = -8 \rightarrow 7$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2 + 0.3428P)]$

where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.098$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.11$	$\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$
2628 reflections	$\Delta\rho_{\min} = -0.97 \text{ e } \text{\AA}^{-3}$
156 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0084 (19)
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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.5	0.5	0.5	0.02521 (16)
S1	0.75777 (6)	0.76611 (10)	0.53190 (4)	0.03283 (19)
O5	0.22643 (18)	0.1935 (3)	0.18093 (9)	0.0384 (4)
O1	0.62169 (17)	0.6626 (3)	0.46467 (10)	0.0387 (4)
O2	0.47029 (16)	0.2626 (3)	0.42577 (9)	0.0307 (4)
O3	0.32165 (16)	0.6292 (3)	0.40600 (9)	0.0316 (4)
O4	0.1020 (2)	0.4798 (3)	0.15900 (11)	0.0478 (5)
C6	0.2779 (2)	0.4022 (4)	0.29848 (13)	0.0252 (5)
C3	0.1192 (3)	0.7190 (4)	0.28219 (15)	0.0378 (6)
H3A	0.1101	0.8133	0.3173	0.057*
H3B	0.0368	0.631	0.2527	0.057*
H3C	0.1291	0.801	0.2446	0.057*
C10	0.3256 (3)	0.0457 (4)	0.23083 (15)	0.0317 (6)
C8	0.3859 (2)	0.2500 (4)	0.34964 (14)	0.0253 (5)
C9	0.3994 (2)	0.0651 (4)	0.31075 (14)	0.0296 (5)
H9	0.4616	-0.0426	0.3428	0.036*
C5	0.2474 (2)	0.5807 (4)	0.33237 (13)	0.0265 (5)
C2	0.7743 (3)	1.0036 (4)	0.48915 (18)	0.0469 (7)
H2A	0.7626	0.9718	0.4382	0.07*
H2B	0.8659	1.0642	0.5256	0.07*
H2C	0.7034	1.1033	0.4805	0.07*
C7	0.1958 (2)	0.3697 (4)	0.21184 (14)	0.0312 (6)
C4	0.3357 (3)	-0.1273 (5)	0.18287 (17)	0.0470 (7)
H4A	0.4053	-0.2289	0.2191	0.071*
H4B	0.3623	-0.0679	0.1484	0.071*
H4C	0.246	-0.1966	0.1501	0.071*
C1	0.8954 (3)	0.6199 (5)	0.53594 (19)	0.0526 (8)
H1A	0.8972	0.4776	0.5542	0.079*
H1B	0.9841	0.6879	0.573	0.079*

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H1C 0.8792 0.6154 0.4828 0.079*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0190 (2)	0.0297 (3)	0.0197 (2)	0.00047 (17)	0.00610 (17)	-0.00322 (16)
S1	0.0280 (3)	0.0421 (4)	0.0247 (3)	-0.0061 (3)	0.0122 (3)	-0.0037 (3)
O5	0.0403 (10)	0.0450 (11)	0.0241 (9)	0.0070 (8)	0.0141 (8)	-0.0024 (7)
O1	0.0292 (9)	0.0534 (11)	0.0275 (9)	-0.0120 (8)	0.0119 (8)	-0.0051 (8)
O2	0.0245 (8)	0.0336 (9)	0.0212 (8)	0.0057 (7)	0.0046 (7)	-0.0021 (7)
O3	0.0257 (8)	0.0336 (10)	0.0260 (9)	0.0043 (7)	0.0083 (7)	-0.0040 (7)
O4	0.0461 (12)	0.0539 (12)	0.0237 (9)	0.0148 (10)	0.0068 (8)	0.0052 (8)
C6	0.0210 (11)	0.0284 (13)	0.0226 (11)	-0.0001 (10)	0.0099 (9)	0.0003 (9)
C3	0.0292 (13)	0.0375 (15)	0.0335 (13)	0.0092 (11)	0.0090 (11)	-0.0011 (11)
C10	0.0273 (13)	0.0352 (14)	0.0342 (13)	-0.0012 (11)	0.0179 (11)	-0.0038 (10)
C8	0.0209 (11)	0.0285 (12)	0.0255 (11)	-0.0034 (10)	0.0122 (9)	-0.0008 (9)
C9	0.0225 (12)	0.0309 (13)	0.0286 (13)	0.0003 (10)	0.0098 (10)	-0.0023 (10)
C5	0.0202 (11)	0.0286 (13)	0.0266 (12)	-0.0018 (10)	0.0104 (10)	0.0025 (9)
C2	0.0358 (15)	0.0372 (16)	0.0524 (17)	0.0002 (12)	0.0148 (13)	0.0047 (12)
C7	0.0292 (13)	0.0347 (14)	0.0264 (12)	0.0010 (11)	0.0132 (10)	0.0013 (10)
C4	0.0510 (17)	0.0524 (18)	0.0395 (15)	0.0046 (15)	0.0262 (14)	-0.0127 (13)
C1	0.0350 (15)	0.0460 (18)	0.0587 (19)	0.0043 (14)	0.0144 (14)	-0.0056 (14)

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

Ni1—O2 ⁱ	1.9849 (16)	C3—C5	1.507 (3)
Ni1—O2	1.9849 (16)	C3—H3A	0.96
Ni1—O3	2.0159 (15)	C3—H3B	0.96
Ni1—O3 ⁱ	2.0159 (15)	C3—H3C	0.96
Ni1—O1 ⁱ	2.1255 (18)	C10—C9	1.321 (3)
Ni1—O1	2.1255 (18)	C10—C4	1.487 (4)
S1—O1	1.5211 (17)	C8—C9	1.447 (3)
S1—C2	1.775 (3)	C9—H9	0.93
S1—C1	1.780 (3)	C2—H2A	0.96
O5—C10	1.371 (3)	C2—H2B	0.96
O5—C7	1.398 (3)	C2—H2C	0.96
O2—C8	1.262 (3)	C4—H4A	0.96
O3—C5	1.250 (3)	C4—H4B	0.96
O4—C7	1.215 (3)	C4—H4C	0.96
C6—C8	1.440 (3)	C1—H1A	0.96
C6—C7	1.441 (3)	C1—H1B	0.96
C6—C5	1.443 (3)	C1—H1C	0.96
O2 ⁱ —Ni1—O2	180	C9—C10—C4	127.3 (2)
O2 ⁱ —Ni1—O3	92.94 (6)	O5—C10—C4	111.1 (2)
O2—Ni1—O3	87.06 (6)	O2—C8—C6	125.9 (2)
O2 ⁱ —Ni1—O3 ⁱ	87.06 (6)	O2—C8—C9	116.6 (2)
O2—Ni1—O3 ⁱ	92.94 (6)	C6—C8—C9	117.4 (2)

O3—Ni1—O3 ⁱ	180.0000 (10)	C10—C9—C8	121.6 (2)
O2 ⁱ —Ni1—O1 ⁱ	89.72 (7)	C10—C9—H9	119.2
O2—Ni1—O1 ⁱ	90.28 (7)	C8—C9—H9	119.2
O3—Ni1—O1 ⁱ	89.31 (7)	O3—C5—C6	123.2 (2)
O3 ⁱ —Ni1—O1 ⁱ	90.69 (7)	O3—C5—C3	114.3 (2)
O2 ⁱ —Ni1—O1	90.28 (7)	C6—C5—C3	122.51 (19)
O2—Ni1—O1	89.72 (7)	S1—C2—H2A	109.5
O3—Ni1—O1	90.69 (7)	S1—C2—H2B	109.5
O3 ⁱ —Ni1—O1	89.31 (7)	H2A—C2—H2B	109.5
O1 ⁱ —Ni1—O1	180	S1—C2—H2C	109.5
O1—S1—C2	105.60 (12)	H2A—C2—H2C	109.5
O1—S1—C1	105.41 (12)	H2B—C2—H2C	109.5
C2—S1—C1	97.65 (15)	O4—C7—O5	112.9 (2)
C10—O5—C7	121.85 (18)	O4—C7—C6	128.7 (2)
S1—O1—Ni1	116.97 (9)	O5—C7—C6	118.4 (2)
C8—O2—Ni1	129.32 (15)	C10—C4—H4A	109.5
C5—O3—Ni1	131.49 (15)	C10—C4—H4B	109.5
C8—C6—C7	118.8 (2)	H4A—C4—H4B	109.5
C8—C6—C5	121.45 (19)	C10—C4—H4C	109.5
C7—C6—C5	119.74 (19)	H4A—C4—H4C	109.5
C5—C3—H3A	109.5	H4B—C4—H4C	109.5
C5—C3—H3B	109.5	S1—C1—H1A	109.5
H3A—C3—H3B	109.5	S1—C1—H1B	109.5
C5—C3—H3C	109.5	H1A—C1—H1B	109.5
H3A—C3—H3C	109.5	S1—C1—H1C	109.5
H3B—C3—H3C	109.5	H1A—C1—H1C	109.5
C9—C10—O5	121.6 (2)	H1B—C1—H1C	109.5

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

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C1—H1B \cdots O4 ⁱⁱ	0.96	2.55	3.384 (4)	145
C2—H2B \cdots O4 ⁱⁱ	0.96	2.53	3.370 (4)	146
C2—H2C \cdots O2 ⁱⁱⁱ	0.96	2.46	3.378 (4)	160

Symmetry codes: (ii) $x+1, -y+3/2, z+1/2$; (iii) $x, y+1, z$.

Fig. 1

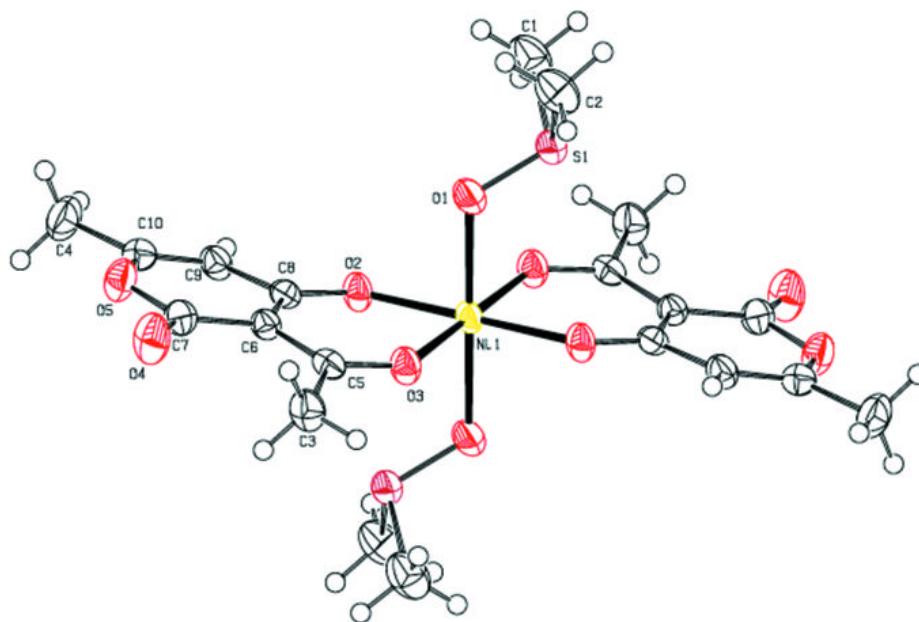


Fig. 2

