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Bis[*N*-(2,6-dimethylphenyl)aminophenylglyoximato-κ^2N,N']nickel(II) dimethyl sulfoxide solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.037
wR factor = 0.084
Data-to-parameter ratio = 13.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[Ni(C_{16}H_{16}N_3O_2)_2]\cdot C_2H_6OS$, the Ni^{II} atom lies on a crystallographic centre of symmetry and its coordination geometry is distorted square-planar, involving four N atoms from two oxime ligands. The dimethyl sulfoxide solvent is disordered over a crystallographic twofold axis.

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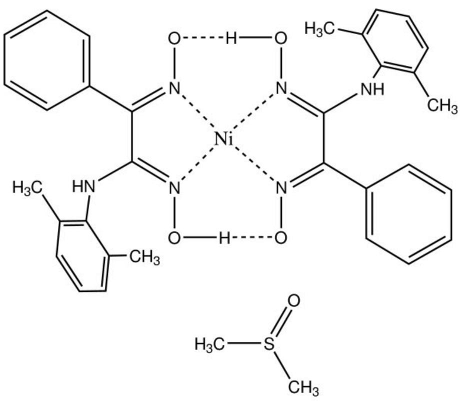
Comment

Transition metal complexes of vicinal dioximes are structurally well known compounds in which metal ions coordinate through N atoms (Chakravorty, 1974). Generally, coordination occurs through deprotonation of two ligands, giving neutral metal complexes which are soluble only in organic solvents. The most common example of a dioxime complex is dimethylglyoxime–nickel(II), which is also used for the analytical determination of Ni^{II} ions. Aminoglyoximes and related ligands are of interest because of their free amine substituents, which give them the ability to form new ligand systems or metal complexes. In this paper, we report the structure of the title compound, (I).



(I)

In (I), the Ni complex is centrosymmetric (Fig. 1). The coordination around the Ni atom is distorted square planar, involving four oxime N atoms of two chelating moieties, with Ni—N distances of 1.8370 (16) and 1.8827 (15) Å. These distances are typical of Ni–oxime complexes (Ülkü *et al.*, 1996; Işık *et al.*, 2000). The coordination plane around the Ni atom and the plane of the chelating ligand coincide within experimental error. The hydroxyl H atom is involved in an intramolecular hydrogen bond [O1···O2^{i} = 2.496 (2) Å; symmetry code: (i) $-x$, $1-y$, $-z$]. The hydrogen bonds between the oxime O atoms are not symmetric. The H atom in complexes with O···O distances of 2.42–2.43 Å is almost centred between the O atoms (Chakravorty, 1974).

A comparision of the C═N and N—O distances with those of free oximes [N—O = 1.396 (3) Å; Hökelek *et al.*, 2001]

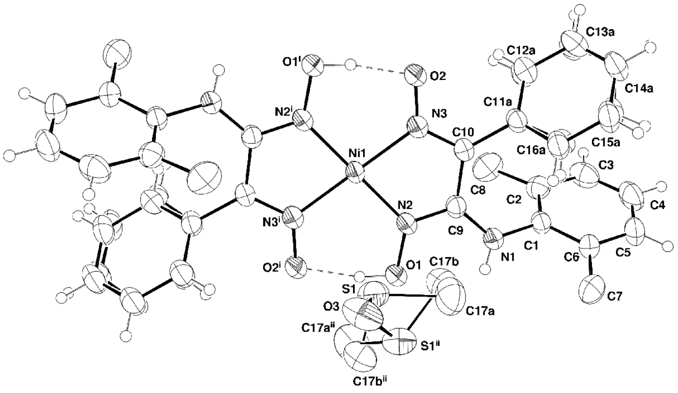


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 40% probability level. Dashed lines indicate hydrogen bonds. Only one of the disordered phenyl group is labelled and methyl H atoms have been omitted for clarity. [Symmetry codes: (i) $-x, 1-y, -z$; (ii) $-x, y, \frac{1}{2}-z$.]

shows that at least one of the N—O distances [N3—O2 = 1.309 (2) Å] is considerably shortened in (I), while the C═N distance is increased by about 0.01 Å.

The oxime moieties in (I) have an *E* configuration, with an O1—N2—C9—C10 torsion angle of 178.21 (16)°. In this configuration, atom N1 participates in intermolecular hydrogen bonding (Table 2) with atom O3 atom of the dimethyl sulfoxide (DMSO) molecule. Atom O3 is located on a twofold axis, the molecule showing rotational disorder (Fig. 1). The C11–C16 phenyl group also shows orientational disorder. The three-dimensional framework is stabilized by a hydrogen-bonded network and other weak intermolecular interactions. A *PLATON* analysis (Spek, 2003) showed that unit cell contains some voids and that the total potential solvent volume is 35 Å^3, or 2.1%. The nearest atom to the void is O2, at a distance of 3.01 Å.

Experimental

N-(2,6-Dimethylphenyl)aminophenylglyoxime was prepared as reported in the literature (Hökelek *et al.*, 2001). A solution of $NiCl_2 \cdot 6H_2O$ (0.48 g, 2 mmol) in ethanol (10 ml) was added to a solution of *N*-(2,6-dimethylphenyl)aminophenylglyoxime (1.13 g, 4 mmol) in absolute ethanol (20 ml). The mixture was stirred for 3–4 h at ambient temperature. The original pale-yellow colour changed immediately to red and the pH value decreased from 6 to 2, which was then adjusted to about 5.0–5.5 with 1% KOH solution. The turbid mixture was stirred in a water bath at 313–323 K for 1 h in order to completely precipitate the complex. The precipitated complex was dissolved in DMSO and left at the ambient temperature for one year to obtain crystals of (I) suitable for X-ray study.

Crystal data

$[Ni(C_{16}H_{16}N_3O_2)_2] \cdot C_2H_6OS$
$M_r = 701.47$
Monoclinic, $P2/c$
$a = 13.2381$ (11) Å
$b = 7.8171$ (4) Å
$c = 16.5034$ (14) Å
$\beta = 94.654$ (7)°
$V = 1702.2$ (2) Å^3
$Z = 2$
$D_x = 1.369$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9778 reflections
$\theta = 2.5$–27.6°
$\mu = 0.68$ mm^{-1}
$T = 293$ (2) K
Plate, dark red
$0.70 \times 0.29 \times 0.05$ mm

Data collection

Stoe IPDS-2 diffractometer
φ scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\min} = 0.789$, $T_{\max} = 0.967$
18574 measured reflections
4027 independent reflections
2627 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.069$
$\theta_{\max} = 27.8°$
$h = -17 \rightarrow 17$
$k = -10 \rightarrow 9$
$l = -21 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.084$
$S = 0.91$
4027 reflections
291 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	1.8370 (16)	N1—C9	1.338 (3)
Ni1—N3	1.8827 (15)	N1—C1	1.427 (2)
O1—N2	1.384 (2)	N2—C9	1.302 (2)
O2—N3	1.309 (2)	N3—C10	1.305 (2)
N2—Ni1—N3^i	98.03 (7)	N2—Ni1—N3	81.97 (7)
O1—N2—C9—C10	178.21 (16)		

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···O2^i	0.92 (3)	1.59 (3)	2.496 (2)	169 (3)
N1—H21···O3^{ii}	0.81 (2)	2.21 (2)	2.933 (2)	149 (2)

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

The phenyl group of the oxime ligand shows orientational disorder and the refined site-occupancy factors of the disordered parts, C11*a*–C16*a* and C11*b*–C16*b*, are 46 (2) and 54 (2)%, respectively. The H atoms bonded to N and O atoms were located in difference-density maps and refined isotropically. Phenyl and methyl H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: X-RED32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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