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Diaquabis{3-methyl-1-phenyl-4-[(*E*)-phenyl(phenyliminio)methyl]-1*H*-pyrazol-5-olato-κ^2O,N^4}nickel(II) dimethyl sulfoxide disolvate

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In the title compound, $[Ni(C_{23}H_{18}N_3O)_2(H_2O)_2]\cdot 2C_2H_6OS$, the Ni atom is octahedrally coordinated by two N atoms and two O atoms from two organic ligands and two water molecules which are *cis* to each other. The complex has crystallographic twofold rotation symmetry.

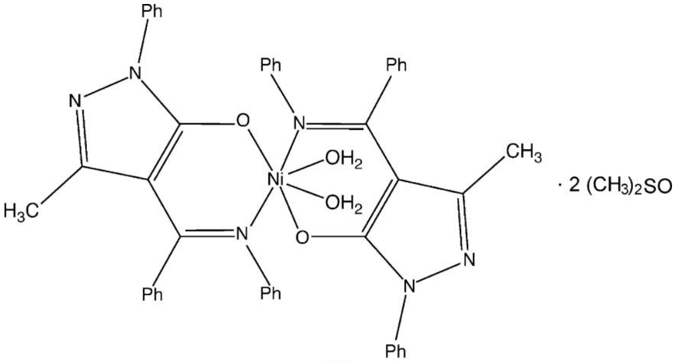
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.027
Data-to-parameter ratio = 16.9

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

Comment

3-Methyl-1-phenyl-4-[(*E*)-phenyl(phenyliminio)methyl]-1*H*-pyrazol-5-olate (*L*) reacts with nickel(II) acetate to give a complex $[Ni(L)_2(H_2O)_2]\cdot 2(CH_3)_2SO$, (I). The structure of complex (I) together with that of the free ligand was described briefly on a poster (Anderson *et al.*, 1999). The structures of metal complexes with ligands in which the phenyl group on the imino N of *L* is replaced by PhOCMe in $Cu(L^1)_2\cdot H_2O$ (Wang *et al.*, 2003), naphthyl in $Co(L^2)_2(DMF)_2$ (Zhang *et al.*, 2004) and PhCl in $Co(L^3)_2$ (Li & Yang, 2004) have been reported. The structure of the free ligand *L* has been described by Parsons *et al.* (2004) who found essentially the same structure as that found by us (la Cour & Hazell, 2006).



(I)

In complex (I), the Ni atom is octahedral, lying on a crystallographic twofold rotation axis, and is coordinated by two N atoms [Ni—N = 2.111 (1) Å] and two O atoms [Ni—O = 2.014 (1) Å] from two Schiff base ligands *L* and by two water molecules [Ni—O = 2.105 (1) Å] (Fig. 1). The water molecules are *cis* to each other, a configuration which occurs in complexes where the Schiff base ligands are too bulky to be *trans* to each other. As is in the free ligand (la Cour & Hazell, 2006), the pyrazole ring deviates slightly from planarity, with C7 0.051 (1) Å out of the plane defined by C3, C4, N5 and N6. The 1-phenyl group is approximately coplanar with the pyrazole group, whereas the other two phenyl groups are twisted out of the pyrazole plane. The torsion angles N5—N6—C20—C25, C2—N1—C30—C35 and C3—C2—C80—

C85 are −0.5 (3), 80.4 (2) and 71.6 (2)°, respectively. Complex (I) crystallizes with two DMSO molecules, which are hydrogen-bonded to water molecule O60 (Table 1). The biggest differences between the free ligand *L* and the complex (I) are in the chelating ring. The C2—C3 and C2—N1 bond lengths are 1.390 (2) and 1.344 (2) Å in *L* and 1.435 (2) and 1.306 (2) Å in (I), and the N1···O8 distance is 2.709 (1) Å in *L* and 2.944 (2) Å in (I).

Experimental

The ligand *L* was prepared as previously described (la Cour *et al.*, 1996). Addition of the ligand to a methanol solution of nickel(II) acetate tetrahydrate (in a molar ratio of 2:1) yielded a blue–green solid, which was recrystallized from DMSO to give crystals of (I).

Crystal data

$[Ni(C_{23}H_{18}N_3O)_2(H_2O)_2]\cdot 2C_2H_6OS$
$M_r = 955.86$
Monoclinic, $C2/c$
$a = 24.399\ (1)$ Å
$b = 11.9075\ (6)$ Å
$c = 16.8717\ (8)$ Å
$\beta = 107.421\ (1)^\circ$
$V = 4677.0\ (4)$ Å^3
$Z = 4$
$D_x = 1.357$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.56$ mm^{-1}
$T = 120$ K
Prism, blue–green
0.60 × 0.50 × 0.30 mm

Data collection

Siemens SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.634$, $T_{max} = 0.837$
20050 measured reflections
6483 independent reflections
5096 reflections with $I > 3\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 29.9^\circ$

Refinement

Refinement on F
$R[F^2 > 3\sigma(F^2)] = 0.030$
$wR(F^2) = 0.027$
$S = 1.01$
5096 reflections
302 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/\{[\sigma_{cs}(F^2) + 1 + 1.012F^2]^{1/2} - |F|\}^2$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20\ (3)$ e Å^{-3}
$\Delta\rho_{min} = -0.17\ (3)$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O60—H60*a*···N5^i	0.83 (2)	2.00 (2)	2.820 (2)	171 (2)
O60—H60*b*···O90	0.82 (2)	2.06 (2)	2.873 (2)	168 (2)

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

H atoms of the ligand were positioned geometrically and refined as riding atoms, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms of the water molecule were located in a difference map and refined isotropically.

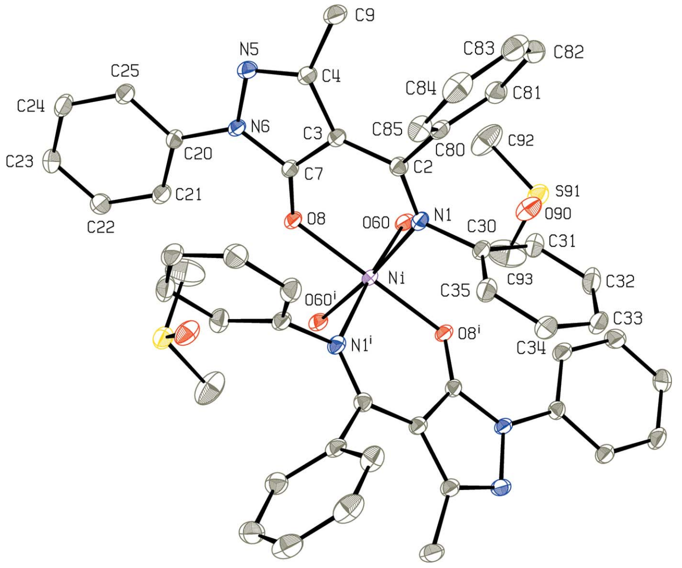


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x, y, \frac{1}{2} - z$.]

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *KRYSTAL* (Hazell, 1995); molecular graphics: *KRYSTAL*; software used to prepare material for publication: *KRYSTAL*.

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