Bis{4-[(Z)-(2,4-dimethylphenylamino)phenylmethylene]-3-methyl-1-phenyl-1H-pyrazol-5(4H)-onato-κ²N,O}(ethanol-κO)nickel(II)

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The Ni^{II} atom in the title compound, $[Ni(C_{25}H_{22}N_3O)_2(C_2H_6O)]$, is chelated by two deprotonated 4-[(Z)-(2,4-dimethylphenylamino)phenylmethylene]-3-methyl-1-phenyl-1H-pyrazol-5(4H)-one ligands through their amide N and carbonyl O atoms and coordinated by an ethanol molecule in a five-coordinate trigonal–bipyramidal geometry.

Comment

The 4-[(Z)-(2,4-dimethylphenylamino)phenylmethylene]-3-methyl-1-phenyl-1H-pyrazol-5(4H)-one ligand (Ma *et al.*, 2006) is a sterically crowded ligand that chelates to nickel in its deprotonated form; the 2,4-dimethylphenyl units are rotated so as to avoid interacting with the metal center. The metal is additionally linked to an ethanol molecule, (I) (Fig. 1). The trigonal–bipyramidal geometry has the axial sites occupied by the O atoms involved in chelation, in the expected configuration with the most electronegative atoms axial.

(I)
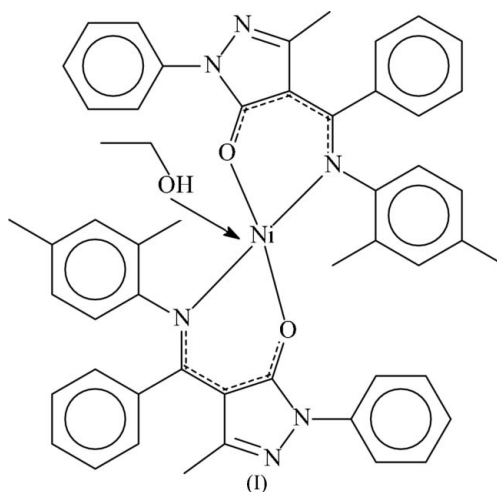
The ethanol serves as hydrogen-bond donor to the pyrazole N atom of an adjacent molecule, forming a zigzag chain running along the c axis.

Experimental

To a chloroform (5 ml) solution of 4-[(Z)-(2,4-dimethylphenylamino)phenylmethylene]-3-methyl-1-phenyl-1H-pyrazol-5(4H)-one (38 mg, 0.1 mmol) and triethylamine (14 ml, 0.1 mmol) was added nickel(II) chloride (6.5 mg, 0.05 mmol) dissolved in ethanol (5 ml). The mixture was filtered and the solution set aside for several days to give green crystals in about 70% yield. Elemental analysis calculated for $C_{52}H_{50}N_6NiO_3$: C 72.15, H 5.82, N 9.71%; found: C 72.20, H 5.79, N 9.82%.

Crystal data

$[Ni(C_{25}H_{22}N_3O)_2(C_2H_6O)]$

$M_r = 865.69$

Monoclinic, $P2_1/c$

$a = 24.478\ (1)$ Å

$b = 14.8835\ (6)$ Å

$c = 12.6757\ (5)$ Å

$\beta = 99.366\ (1)°$

$V = 4556.4\ (3)$ Å^3

$Z = 4$

$D_x = 1.262$ Mg m^{-3}

Mo $K\alpha$ radiation

$\mu = 0.48$ mm^{-1}

$T = 295\ (2)$ K

Block, green
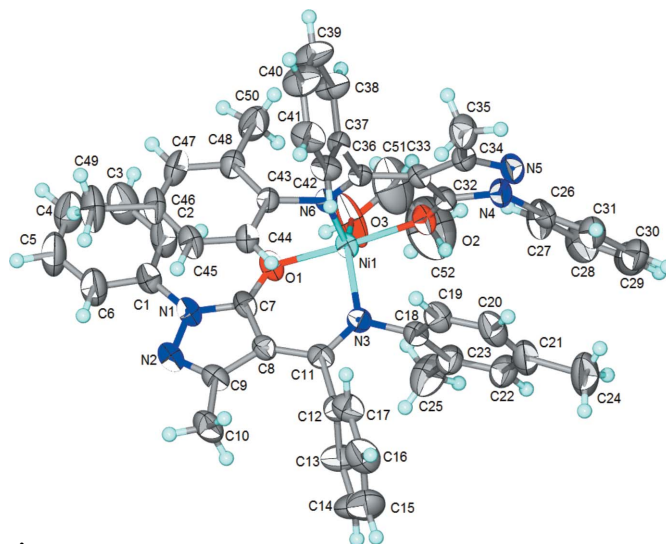
$0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEX area-detector diffractometer

ω and φ scans

Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
$T_{min} = 0.911$, $T_{max} = 0.954$

43522 measured reflections

8022 independent reflections

5347 reflections with $I > 2\sigma(I)$

$R_{int} = 0.097$

$\theta_{max} = 25.0°$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.151$

$S = 0.95$

8022 reflections

565 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.60$ e Å^{-3}

$\Delta\rho_{min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.982 (2)	Ni1—N3	2.020 (3)
Ni1—O2	1.967 (2)	Ni1—N6	2.025 (3)
Ni1—O3	2.094 (3)		
O1—Ni1—O2	163.8 (1)	O2—Ni1—N3	97.3 (1)
O1—Ni1—O3	80.1 (1)	O2—Ni1—N6	92.4 (1)
O1—Ni1—N3	92.4 (1)	O3—Ni1—N3	130.8 (1)
O1—Ni1—N6	98.8 (1)	O3—Ni1—N6	129.0 (1)
O2—Ni1—O3	83.7 (1)	N3—Ni1—N6	100.1 (1)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H3o$\cdots$N2^i	0.85	2.15	2.910 (4)	148

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

The ethanol molecule is somewhat disordered but a disorder model refinement did not significantly lower the *R* index. Distance restraints were applied to the O—C (1.400±0.005 Å) and C—C

Figure 1
The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

(1.500±0.005 Å) bonds to bring them to standard values of about 1.45 and 1.50 Å; the displacement parameters of the atoms were restrained to be nearly isotropic. H atoms were positioned geometrically and were included in the refinement in the riding-model approximation [phenyl C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C); methyl C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)]; the methyl groups were rotated to fit the electron density. The hydroxy H atom was similarly treated, with O—H = 0.85 Å.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.

Bruker (2003). *SAINT* (Version 6.45A) and *SMART* (Version 6.45A). Bruker AXS Inc., Madison, Wisconsin, USA.

Ma, R.-M., Sun, S.-F. & Ng, S. W. (2006). *Acta Cryst.* E**62**, o4679–o4680.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.