Crystal structure of {*N*,*N*′-di-(2-hydroxy-1-naphthylidene)-1-methyl-1,2-diaminoethane-*N*,*N*′,O,O′}nickel(II), ($C_{25}H_{20}N_2O_2$)Ni(II)

F. Ercan

Hacettepe University, Department of Engineering Physics, 06532 Beytepe, Ankara, Turkey

M. Sari, R. Yağbasan

Gazi University, Gazi Education Faculty, Department of Physics Education, 06500 Besevler, Ankara, Turkey

O. Atakol and M. A. Akay

Ankara University, Department of Chemistry, 06100 Tandoğan, Ankara, Turkey

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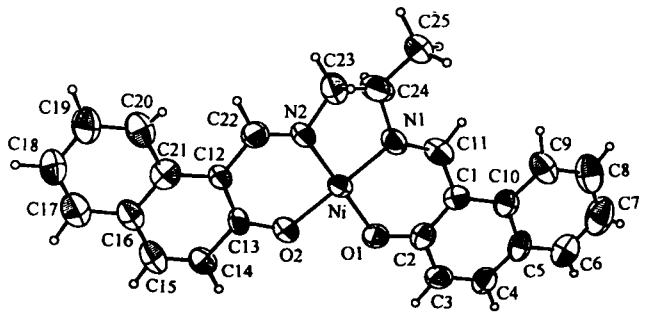

Source of material: 0.344 g 2-hydroxy-1-naphthaldehyde (2 mmol) was dissolved in 60 ml hot dioxane and a solution of 0.248 g $Ni(CH_3CO)_2{\cdot}4H_2O$ (1 mmol) and 0.075 g 1,2-diamino ethane (1 mmol) in 30 ml hot methanol was added to it (templat synthesis). The mixture was kept 48 h at room temperature. Dark red crystals which formed as a result of this process were filtered and dried in an oven at 353 K.

H atoms of the carbons were bonded geometrically 0.95 Å from the corresponding atoms. Thermal parameters of all H atoms were taken 1.5 times of the corresponding atoms and treated fixed. A riding model was used for all hydrogen atoms. All non-H atoms were refined anisotropically. There have been many crystal structure determinations of complexes between nickel(II) and tetradentate ligands with two O and two N donor atoms. The geometry of the inner coordination sphere is usually planar in the case of nickel(II). The aim of this study is to study the change on the coordination sphere when a symmetry broker group has been added to the ligand. In the literature, symmetric ligands have been studied more than asymmetric ligands (see refs. 1-2). The average Ni–N and Ni–O bond lengths are 1.812(11) Å and 1.837(9) Å, respectively, which are in agreement with those reported (see ref. 1) and of other similar structures (see refs. 1-2). The Ni atom has square-planar coordination with average angles of 94.3(4)° in the six-membered rings and 85.7(5)° in the five-membered rings. The average values of the other bond lengths in the six-membered rings are d(C–O) = 1.305(15) Å, d(C–N) = 1.285(2) Å and d(C–C) = 1.414(2) Å and 1.431(2) Å. The average interatomic distances in the two naphthyl rings are found to be the nearly same, 1.396(2) Å. The molecules have the *cis* form as imposed by the geometry of the ligand; the coordination of the metal atom is square-planar, the dihedral angle between the two Ni, N, O planes being 5.4(7)°. The two rings Ni-O1-C2-C1-C11-N1 and Ni-O2-C13-C12-C22-N2 are found nearly planar, the largest deviations of the atoms from the best plane passing through the molecule being C1 –0.002 Å and C13 –0.023 Å. The dihedral angle between the planes is 3.7°. The two six-membered rings are also found to have slightly different structures.

$C_{25}H_{20}N_2NiO_2$, monoclinic, $P12_1/n1$ (No. 14), a =14.384(2) Å, b =8.254(3) Å, c =17.626(2) Å, β =111.820(1)°, V =1942.7 Å^3, Z =4, $R(F)$ =0.053, $R_w(F^2)$ =0.126.

Table 1. Parameters used for the X-ray data collection

Crystal:	dark red, prism, size 0.06 x 0.16 x 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	103 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω/2θ
$T_{measurement}$:	296 K
$2\theta_{max}$:	49°
N(hkl)$_{unique}$:	2233
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
N($param$)$_{refined}$:	273
Program:	SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.251(1)	–0.106(2)	0.2098(9)	0.103
H(4)	4*e*	0.247(1)	–0.206(2)	0.3255(9)	0.103
H(6)	4*e*	0.171(1)	–0.229(2)	0.4310(9)	0.115
H(7)	4*e*	0.045(2)	–0.172(2)	0.475(1)	0.127
H(8)	4*e*	–0.089(2)	–0.020(2)	0.397(1)	0.129
H(9)	4*e*	–0.095(1)	0.080(2)	0.276(1)	0.109
H(11)	4*e*	–0.107(1)	0.143(2)	0.1532(9)	0.093
H(14)	4*e*	0.258(1)	0.170(2)	–0.0852(9)	0.094
H(15)	4*e*	0.262(1)	0.272(2)	–0.1999(9)	0.105
H(17)	4*e*	0.184(1)	0.430(2)	–0.327(1)	0.117

Table 2. (Continued)

Atom	Site	x	y	z	U_{iso}
H(18)	4e	0.058(1)	0.589(2)	−0.409(1)	0.119
H(19)	4e	−0.080(1)	0.645(2)	−0.374(1)	0.125
H(20)	4e	−0.083(1)	0.552(2)	−0.2553(9)	0.107
H(22)	4e	−0.0967(9)	0.439(2)	−0.1548(9)	0.091
H(23A)	4e	−0.138(1)	0.465(2)	−0.0171(9)	0.120
H(23B)	4e	−0.202(1)	0.362(2)	−0.0937(9)	0.120
H(24)	4e	−0.190(1)	0.148(2)	−0.025(1)	0.124
H(25A)	4e	−0.256(6)	0.188(4)	0.068(6)	0.144
H(25B)	4e	−0.299(3)	0.32(1)	0.001(2)	0.144
H(25C)	4e	−0.215(3)	0.37(1)	0.085(5)	0.144

Table 3. Final atomic coordinates and displacement parameters (in $Å^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	4e	0.0338(1)	0.1952(2)	0.0247(1)	0.0632(9)	0.070(1)	0.073(1)	0.001(1)	0.0269(7)	−0.007(1)
O(1)	4e	0.1237(6)	0.074(1)	0.1057(6)	0.076(6)	0.074(6)	0.092(6)	0.004(5)	0.042(5)	−0.005(5)
O(2)	4e	0.1289(6)	0.196(1)	−0.0225(5)	0.070(5)	0.085(6)	0.074(6)	−0.002(5)	0.024(4)	−0.003(5)
N(1)	4e	−0.0624(7)	0.192(1)	0.0688(6)	0.081(7)	0.072(7)	0.074(7)	−0.006(6)	0.032(6)	0.017(6)
N(2)	4e	−0.0537(7)	0.315(2)	−0.0555(7)	0.071(7)	0.092(8)	0.080(7)	−0.005(7)	0.039(6)	−0.017(7)
C(1)	4e	0.034(1)	0.055(2)	0.1950(8)	0.080(9)	0.064(8)	0.065(8)	−0.009(7)	0.025(7)	0.008(7)
C(2)	4e	0.117(1)	0.023(2)	0.1728(8)	0.079(9)	0.068(9)	0.071(9)	−0.005(7)	0.028(7)	−0.005(7)
C(3)	4e	0.197(1)	−0.080(2)	0.2252(9)	0.074(9)	0.10(1)	0.076(9)	0.010(8)	0.021(7)	−0.008(9)
C(4)	4e	0.195(1)	−0.139(2)	0.2945(9)	0.082(9)	0.09(1)	0.072(9)	−0.002(8)	0.017(7)	0.000(8)
C(5)	4e	0.118(1)	−0.106(2)	0.3240(9)	0.10(1)	0.064(8)	0.078(9)	−0.003(8)	0.026(8)	−0.014(8)
C(6)	4e	0.118(1)	−0.166(2)	0.3987(9)	0.11(1)	0.09(1)	0.072(9)	0.01(1)	0.020(9)	0.011(9)
C(7)	4e	0.042(2)	−0.132(2)	0.425(1)	0.14(2)	0.10(1)	0.08(1)	−0.01(1)	0.04(1)	0.02(1)
C(8)	4e	−0.037(2)	−0.041(2)	0.379(1)	0.15(2)	0.10(1)	0.09(1)	−0.00(1)	0.07(1)	0.02(1)
C(9)	4e	−0.040(1)	0.018(2)	0.306(1)	0.09(1)	0.09(1)	0.11(1)	−0.014(8)	0.050(9)	−0.016(9)
C(10)	4e	0.036(1)	−0.007(2)	0.273(1)	0.09(1)	0.064(8)	0.10(1)	−0.008(8)	0.028(8)	−0.014(8)
C(11)	4e	−0.052(1)	0.134(2)	0.1378(9)	0.074(9)	0.063(8)	0.10(1)	0.011(7)	0.037(8)	0.007(8)
C(12)	4e	0.0417(9)	0.358(2)	−0.1424(8)	0.063(7)	0.079(9)	0.070(8)	0.000(7)	0.026(6)	−0.012(7)
C(13)	4e	0.123(1)	0.262(2)	−0.0924(8)	0.076(8)	0.09(1)	0.060(7)	−0.001(7)	0.031(7)	−0.016(7)
C(14)	4e	0.205(1)	0.234(2)	−0.1179(9)	0.072(8)	0.08(1)	0.088(9)	−0.001(7)	0.036(7)	−0.014(8)
C(15)	4e	0.207(1)	0.294(2)	−0.1860(9)	0.10(1)	0.09(1)	0.10(1)	−0.01(1)	0.063(9)	−0.01(1)
C(16)	4e	0.130(1)	0.389(2)	−0.2380(9)	0.09(1)	0.076(9)	0.082(9)	−0.018(8)	0.050(8)	−0.021(8)
C(17)	4e	0.130(1)	0.453(2)	−0.312(1)	0.11(1)	0.10(1)	0.10(1)	0.01(1)	0.06(1)	−0.00(1)
C(18)	4e	0.055(1)	0.547(2)	−0.361(1)	0.12(1)	0.10(1)	0.10(1)	−0.01(1)	0.06(1)	0.02(1)
C(19)	4e	−0.028(1)	0.582(2)	−0.340(1)	0.10(1)	0.12(1)	0.11(1)	−0.01(1)	0.05(1)	0.03(1)
C(20)	4e	−0.029(1)	0.524(2)	−0.2699(9)	0.09(1)	0.10(1)	0.09(1)	−0.011(9)	0.045(8)	0.012(9)
C(21)	4e	0.047(1)	0.423(2)	−0.2160(9)	0.10(1)	0.062(8)	0.079(9)	−0.010(8)	0.029(8)	−0.004(7)
C(22)	4e	−0.0438(9)	0.377(2)	−0.1202(9)	0.059(7)	0.079(9)	0.083(9)	0.004(7)	0.019(7)	−0.011(8)
C(23)	4e	−0.145(1)	0.359(2)	−0.0419(9)	0.10(1)	0.13(2)	0.078(9)	0.00(1)	0.046(8)	0.01(1)
C(24)	4e	−0.163(1)	0.243(2)	0.010(1)	0.071(9)	0.15(2)	0.10(1)	0.03(1)	0.044(9)	0.04(1)
C(25)	4e	−0.240(1)	0.283(2)	0.0438(9)	0.085(9)	0.11(1)	0.10(1)	0.003(9)	0.039(8)	0.01(1)

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