Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.00007$
$R = 0.049$	$\Delta\rho_{max} = 0.70$ e Å^{-3}
$wR = 0.040$	$\Delta\rho_{min} = -0.67$ e Å^{-3}
$S = 1.0$	Extinction correction: none
888 reflections	Atomic scattering factors
145 parameters	from *International Tables*
H atoms: see below	*for X-ray Crystallography*
$w = [1/\sigma^2(F)]$	(1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ag1	1.1920 (1)	−0.0784 (1)	0.46845 (4)	3.29 (1)
O1	0.9120 (9)	−0.0906 (8)	0.3648 (3)	2.8 (1)
O2	0.599 (1)	0.0267 (7)	0.4121 (3)	2.8 (1)
O3	−0.082 (1)	0.1523 (7)	0.2097 (4)	3.1 (1)
O4	−0.066 (1)	0.0990 (9)	0.0788 (4)	4.7 (2)
O5	0.557 (1)	−0.2139 (7)	−0.0021 (4)	3.3 (2)
O6	0.864 (1)	−0.2670 (7)	0.0866 (4)	3.6 (2)
N1	0.014 (1)	0.0975 (9)	0.1526 (4)	2.6 (2)
N2	0.665 (1)	−0.2095 (9)	0.0680 (4)	2.8 (2)
C1	0.562 (1)	−0.0565 (9)	0.2728 (5)	2.1 (2)
C2	0.345 (2)	0.0171 (9)	0.2548 (5)	1.9 (2)
C3	0.235 (2)	0.014 (1)	0.1730 (5)	2.1 (2)
C4	0.338 (2)	−0.0597 (9)	0.1111 (5)	2.3 (2)
C5	0.549 (2)	−0.134 (1)	0.1330 (5)	2.1 (2)
C6	0.663 (2)	−0.1302 (9)	0.2118 (5)	2.0 (2)
C7	0.703 (1)	−0.0395 (9)	0.3582 (5)	1.8 (2)

Table 2. *Selected geometric parameters (Å, °)*

Ag1···Ag1^i	2.835 (2)	Ag1—O2^i	2.218 (6)
Ag1···Ag1^{ii}	3.746 (2)	Ag1—O2^{iii}	2.655 (7)
Ag1—O1	2.176 (5)		
Ag1—O1—C7	128.9 (5)	O1—Ag1—O2^{iii}	108.0 (2)
Ag1^i—O2—C7	119.4 (5)	O2^i—Ag1—O2^{iii}	79.9 (2)
Ag1—O2^{iii}—C7^{iii}	118.07 (5)	Ag1—O2^i—Ag1^{ii}	100.1 (3)
O1—Ag1—O2^i	164.0 (2)		

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

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their corresponding C atoms. A riding model was used for all H atoms, with $U_{iso}(H) = 1.3U_{eq}(C)$.

Data collection: *CAD-4 Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Bis[N-(2,6-dimethylphenyl)amino-glyoximato-N,N']nickel(II)

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Abstract

The title compound consists of discrete centrosymmetric [Ni(C$_{10}$H$_{12}$N$_3$O$_2$)$_2$] molecules. The coordination around the Ni atom is square planar, involving four oxime N atoms of two chelating C$_2$H$_2$N$_2$O$_2$ groups. The oxime O atoms are linked by an intramolecular hydrogen bond [O···O 2.546 (2) Å]. The Ni—N distances are 1.891 (2) and 1.846 (2) Å, and the N—Ni—N angle within the NiC$_2$N$_2$ chelate ring is 82.75 (8)°. In the central portion of the molecule, the C=N distances are equal [1.297(3) and 1.298 (3) Å], while the two N—O distances are different [1.303 (3) and 1.386 (3) Å].

Comment

Aminoglyoximes and related ligands are of interest because of their free amino substituents, which gives them the ability to form new ligand systems or metal complexes. The structures of bis(oxamideoximato)-cobalt(II) oxamide oxime (Bekaroğlu, Sarısaban, Koray

& Ziegler, 1977), bis(oxamideoximato)nickel(II)–water–dimethylformamide (Enders, 1978) and tris(oxamide-oxime)cobalt(III) trichloride (Bekaroğlu et al., 1978) have been reported previously. The cobalt complexes have either tetragonal-bipyramidal or octahedral coordination, while the nickel complex exhibits a square-planar metal environment. The structure determination of the title complex, [Ni(C$_{10}$H$_{12}$N$_3$O$_2$)$_2$], (I), has been undertaken in order to compare its structure with those of related compounds.

(I)
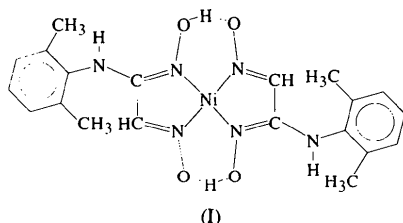
The molecule of (I) is a centrosymmetric monomer (Fig. 1) like the Ni complex mentioned above. The coordination around the Ni atom is square planar, involving four oxime N atoms of two chelating C$_2$H$_2$N$_2$O$_2$ moieties. The hydrogen bonds between the oxime O atoms are not symmetrical, with an O· · ·O distance of 2.546 (2) Å and an O—H—O angle of 167 (3)°. The N—Ni—N angle within the NiC$_2$N$_2$ chelate ring is 82.75 (8)°. Although the angles at the Ni atom and one Ni—N distance have practically the same values as in bis(oximideoximato)nickel(II)–water–dimethyl–formamide, the second Ni—N distance is slightly longer [Ni—N2 1.891 (2) versus 1.860 (4) Å]. The coordination plane around the Ni atom and the plane of the chelating moiety coincide within experimental error. The planes of the phenyl ring and the coordination plane are perpendicular to one another [dihedral angle 89.39 (6)°]. A
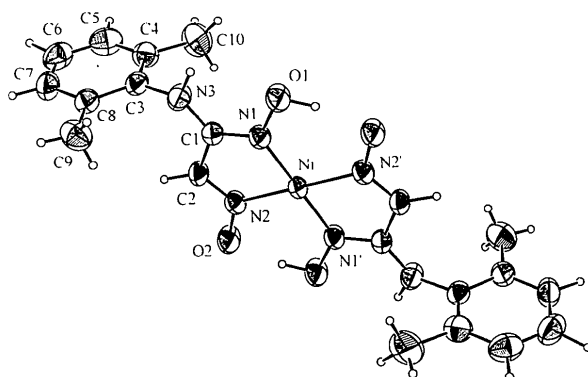
comparison of the C=N and N—O distances with those of free oximes shows that at least one of the N—O distances [N2—O2 1.303 (3) Å] is considerably shortened on complex formation, while the C=N distance stays practically the same. On complex formation, the C—N—O angle is also affected, opening by up to ca 10°. These observations are in agreement with those of Chakravorty (1974) for transition metal complexes of oximes and related ligands.

Experimental

A solution of NiCl$_2$.6H$_2$O (1.20 g, 5 mmol) in ethanol–water (1:1) was added dropwise to a solution of N-(2,6-dimethyl-phenyl)aminoglyoxime (2.07 g, 10 mmol) in ethanol (40 ml) (pH 2.5–3.0). Then a 1% solution of KOH in water was dripped slowly into the mixture until the pH reached 5.5. The resulting precipitate was removed by filtration, washed and dried in vacuo. Recrystallization from a chloroform–ethanol mixture (2:1) gave reddish prismatic crystals.

Crystal data

[Ni(C$_{10}$H$_{12}$N$_3$O$_2$)$_2$]	Mo Kα radiation
M_r = 471.15	λ = 0.71073 Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
a = 7.9572 (7) Å	θ = 11–18°
b = 8.2383 (4) Å	μ = 0.96 mm^{-1}
c = 8.5846 (6) Å	T = 296 K
α = 110.416 (5)°	Prism
β = 92.067 (6)°	0.48 × 0.33 × 0.13 mm
γ = 92.165 (6)°	Dark red
V = 526.32 (4) Å^3	
Z = 1	
D_x = 1.486 Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	2150 observed reflections [$I > 3\sigma(I)$]
ω/2θ scans	R_{int} = 0.016
Absorption correction:	θ_{max} = 26.3°
empirical via ψ scans	h = 0 → 9
(North, Phillips &	k = −10 → 10
Mathews, 1968)	l = −10 → 10
T_{min} = 0.78, T_{max} = 0.88	3 standard reflections
2283 measured reflections	frequency: 120 min
2196 independent reflections	intensity decay: 1%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.002
R = 0.029	$\Delta\rho_{max}$ = 0.61 e Å^{-3}
wR = 0.033	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
S = 1.25	Extinction correction: none
2150 reflections	Atomic scattering factors
145 parameters	from International Tables
If $F \geq$ 197.30 then	for X-ray Crystallography
w = $(197.30/F)^2$,	(1974, Vol. IV)
otherwise w = 1.0	

Fig. 1. ORTEPII (Johnson, 1976) drawing showing the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ni	0	0	0	2.635 (7)
N1	0.1230 (3)	−0.0968 (2)	−0.1853 (2)	3.18 (4)
O1	0.1776 (2)	−0.0196 (2)	−0.2957 (2)	4.29 (4)
C1	0.1783 (3)	−0.2488 (2)	−0.2064 (2)	2.98 (4)
C2	0.1188 (3)	−0.3209 (2)	−0.0846 (3)	3.32 (4)
N2	0.0314 (3)	−0.2148 (2)	0.0277 (2)	3.22 (4)
O2	−0.0345 (3)	−0.2594 (2)	0.1447 (2)	4.57 (4)
N3	0.2754 (3)	−0.3352 (2)	−0.3301 (2)	3.64 (4)
C3	0.3832 (3)	−0.4663 (3)	−0.3165 (2)	3.13 (4)
C4	0.5340 (3)	−0.4165 (3)	−0.2233 (3)	3.96 (5)
C5	0.6395 (4)	−0.5440 (4)	−0.2187 (3)	5.00 (6)
C6	0.5949 (4)	−0.7160 (3)	−0.3014 (3)	5.02 (6)
C7	0.4427 (4)	−0.7626 (3)	−0.3893 (3)	4.51 (6)
C8	0.3323 (3)	−0.6407 (3)	−0.4010 (3)	3.47 (5)
C9	0.1677 (4)	−0.6917 (3)	−0.4951 (3)	5.11 (7)
C10	0.5811 (4)	−0.2276 (4)	−0.1289 (4)	6.28 (8)

Table 2. *Selected geometric parameters ($\mathring{A}$, °)*

Ni—N2	1.891 (2)	C1—N3	1.344 (3)
Ni—N1	1.846 (2)	N1—O1	1.386 (3)
N2—O2	1.303 (3)	N3—C3	1.438 (3)
N2—C2	1.298 (3)	C4—C10	1.508 (4)
C1—C2	1.455 (3)	C8—C9	1.479 (4)
C1—N1	1.297 (3)	O1—H1	0.95 (3)
N2—Ni—N1	82.75 (8)	Ni—N1—O1	127.6 (1)
Ni—N2—O2	123.9 (1)	C1—N1—O1	115.7 (2)
Ni—N2—C2	114.7 (2)	C1—N3—C3	121.9 (2)
O2—N2—C2	121.3 (2)	N3—C3—C4	119.2 (2)
C2—C1—N1	112.9 (2)	N3—C3—C8	118.4 (2)
C2—C1—N3	122.7 (2)	C3—C4—C10	120.7 (2)
N1—C1—N3	124.4 (2)	C5—C4—C10	121.0 (2)
N2—C2—C1	113.1 (2)	C3—C8—C9	121.6 (2)
Ni—N1—C1	116.3 (2)	C7—C8—C9	121.9 (2)
Ni—N2—C2—C1	1.6 (3)	C2—C1—N3—C3	−26.1 (3)
N3—C1—C2—N2	177.9 (2)	C1—N3—C3—C4	−77.5 (3)

H-atom positions were obtained from difference maps. For all H atoms, $U_{iso} = 1.3U_{eq}$ of the parent atom and a riding model was adopted except that the fractional coordinates of H1 (bonded to O1) were refined.

Data collection: *CAD-4 Express Software* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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(Diethylamine-N){4-methyl-2-[(2-oxido-benzylidene)amino]phenolato(2-)-O,N,O'}-nickel(II)

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Abstract

In the title compound, [Ni(C₁₄H₁₁NO₂)(C₄H₁₁N)], the coordination of the Ni atom is distorted square planar, with Ni—O distances of 1.839 (7) and 1.812 (7) Å, Ni—N distances of 1.852 (8) and 1.951 (9) Å, and O—Ni—N angles between 86.7 (4) and 96.2 (4)°.

Comment

Salicylaldimine and its biologically interesting complexes have been extensively studied over the years (Stewart & Lingafelter, 1959; Calligaris, Nardin & Randaccio, 1972; Bhatia, Bindlish, Saini & Jain, 1981; Kessissoglu, Raptopoulou, Bakalbassis, Terzis & Mrozinski, 1992). In biological systems such as jack-bean urease and in some hydrogenases, nickel is in a related complexed form (Walsh & Orme-Johnson, 1987). Schiff base complexes have also been used in electrochemical research and catalytic reactions (Hamilton, Drago & Zombeck, 1987; Costamagna, Vargas, Latorre, Alvarado & Mena, 1992). The title compound, (I), is one of the square-planar complexes formed by the reaction of Ni^{II} with both a ligand having an ONO donor set and an aliphatic amine. The structure of this Ni^{II} complex was determined in order to compare the geometrical features with those of related compounds.