References

Albada, G. A. van, de Graaff, R. A. G., Haasnoot, J. G., Schild, J. & Reedijk, J. (1991). *Acta Cryst.* C**47**, 946–949.

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package.* College Station, Texas, USA.

Bamidele Sanni, S., Smits, J. M. M., Beurskens, P. T., Haasnoot, J. G., Schild, J. & Lenstra, A. T. H. (1986). *J. Crystallogr. Spectrosc. Res.* **16**, 823–832.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Cornelissen, J. P., Haasnoot, J. G. & Reedijk, J. (1986). *Acta Cryst.* C**42**, 1296–1298.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Cornelissen, J. P., Haasnoot, J. G. & Reedijk, J. (1987). *Inorg. Chim. Acta*, **129**, 217–222.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Haasnoot, J. G. & Reedijk, J. (1983). *Inorg. Chim. Acta*, **72**, 81–88.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Haasnoot, J. G. & Reedijk, J. (1984). *Inorg. Chim. Acta*, **86**, 137–143.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Reedijk, J. & Haasnoot, J. (1986). *Acta Cryst.* C**42**, 427–430.

Cornelissen, J. P., de Graaff, R. A. G., Haasnoot, J. G., Prins, R., Reedijk, J., Biagini Cingi, M., Manotti Lanfredi, A. M. & Tiripicchio, A. (1989). *Polyhedron*, **8**, 2313–2320.

Dillen, J., Lenstra, A. T. H., Haasnoot, J. G. & Reedijk, J. (1983). *Polyhedron*, **2**, 195–201.

Favre, T. L. F., Haasnoot, J. G. & Reedijk, J. (1986). *Polyhedron*, **5**, 1405–1411.

Haasnoot, J. G., Driessen, W. L. & Reedijk, J. (1984). *Inorg. Chem.* **23**, 2803–2807.

Haasnoot, J. G., Favre, T. L. F., Hinrichs, W. & Reedijk, J. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 856–858.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* Univ. of Cambridge, England.

Sierra, R., Romero, M. A., Salas, J. M., Sanchez, M. P. & Moreno, M. N. (1991). *J. Inorg. Biochem.* **43**, 229.

Acta Cryst. (1993). C**49**, 1905–1906

An *N*,*N*′-Bis(salicylidene)-1,3-propanediamine–Nickel Complex

YALCIN ELERMAN* AND MEHMET KABAK

Department of Engineering Physics, Faculty of Sciences, University of Ankara, Besevler, Ankara, Turkey

ORHAN ATAKOL

Department of Chemistry, Faculty of Sciences, University of Ankara, Besevler, Ankara, Turkey

(*Received* 21 *August* 1992; *accepted* 6 *May* 1993)

Abstract

In the crystal structure of this Ni^{II} complex with bis(salicylidene)-1,3-diaminopropane, aqua{2,2′-[1,3-propanediylbis(nitrilomethylidyne)]diphenolato}-nickel(II), nickel is five-coordinate with a water molecule at the apex of a square pyramid. The complex has symmetry *m* and the two Schiff base moieties, which in themselves are planar, are inclined at 38.6 (4)° to each other, forming a shallow umbrella form. The configuration at the N atoms is planar, as would be expected of sp^2 hybridization.

Comment

Metal derivatives of Schiff bases have been extensively studied and copper(II) and nickel(II) complexes have played a major role in both synthesis and structural research. Copper(II) and nickel(II) react with tridentate anionic Schiff bases, giving dimerized complexes with square-planar configurations (Maggio, Pizzino & Romano, 1974). *N*-(2-Hydroxyphenyl)salicylideneamine reacts with the nickel(II) ion giving a pseudo-octahedral complex by polymerization. The present study concerns the complex formed when nickel(II) reacts with bis(salicylidene)-1,3-diaminopropane.

The two Schiff base moieties containing O1, N1, C1, C2, C3, C4, C5, C6, C7 and O1*A*, N1*A*, C1*A*, C2*A*, C3*A*, C4*A*, C5*A*, C6*A*, C7*A*, which in themselves are planar [maximum deviation from planes being 0.042 (6) Å], are inclined at 38.6 (4)° to each other, giving a shallow umbrella form as reported in other similar structures. The angles at the C atoms of the propane bridge, C8 112.8 (5) and C9 115.4 (7)°, indicate a small deviation from a regular tetrahedral configuration. The water molecule contributes to the molecular packing through hydrogen bridges.

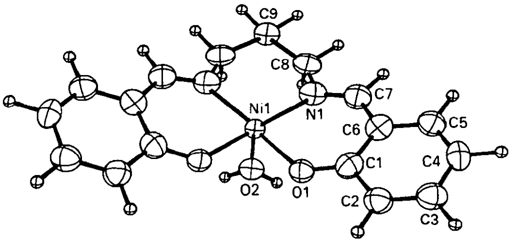

Fig. 1. Molecular structure of the title compound showing the numbering scheme and 50% probability thermal ellipsoids for the non-H atoms.

Experimental

Crystal data

[Ni($C_{17}H_{16}N_2O_2$)(H_2O)]
M_r = 357.04
Orthorhombic
Pbnm
a = 7.539 (1) Å
b = 8.871 (1) Å
c = 24.108 (10) Å

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 9–32°
μ = 1.22 mm^{-1}
T = 293 K

V = 1612.3 (2) Å^3
Z = 4
D_x = 1.471 Mg m^{-3}

Prism
0.20 × 0.15 × 0.10 mm
Green

Data collection

Nicolet $R3$ diffractometer
$\theta/2\theta$ scans
Absorption correction: Ψ method
$T_{\min}$ = 0.75, $T_{\max}$ = 0.87
1343 measured reflections
1343 observed reflections [$F > \sigma(F)$]

$\theta_{\max}$ = 33.18°
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 20$
1 standard reflection
frequency: 60 min
intensity variation: negligible

Refinement

Refinement on F
Final R = 0.048
wR = 0.045
S = 1.11
1106 reflections
109 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F)]$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.435 e Å^{-3}
$\Delta\rho_{\min}$ = −0.453 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ni1	0.0575 (1)	0.4838 (1)	0.2500	0.032 (1)
O1	0.1042 (4)	0.3182 (4)	0.3062 (2)	0.043 (2)
O2	0.3011 (6)	0.5773 (5)	0.2500	0.045 (3)
N1	−0.0382 (6)	0.6167 (5)	0.3119 (2)	0.053 (3)
C1	0.0514 (8)	0.3055 (6)	0.3567 (3)	0.054 (4)
C2	0.0850 (7)	0.1683 (6)	0.3877 (2)	0.053 (4)
C3	0.0305 (8)	0.1488 (6)	0.4406 (3)	0.056 (4)
C4	−0.0626 (8)	0.2636 (6)	0.4674 (2)	0.052 (4)
C5	−0.0959 (7)	0.3974 (6)	0.4411 (3)	0.051 (4)
C6	−0.0417 (7)	0.4226 (6)	0.3867 (3)	0.051 (4)
C7	−0.0717 (8)	0.5693 (6)	0.3625 (3)	0.055 (4)
C8	−0.0680 (7)	0.7824 (6)	0.3023 (3)	0.055 (3)
C9	−0.1671 (10)	0.8136 (9)	0.2500	0.055 (5)

Table 2. *Geometric parameters* (Å, °)

Ni1—O1	2.030 (4)	C4—C5	1.369 (8)
Ni1—O2	2.015 (5)	C5—C6	1.392 (9)
Ni1—N1	2.034 (5)	C6—C1	1.448 (8)
O1—C1	1.286 (7)	C6—C7	1.444 (8)
C1—C2	1.450 (7)	C7—N1	1.313 (8)
C2—C3	1.352 (9)	N1—C8	1.505 (7)
C3—C4	1.395 (8)	C8—C9	1.491 (7)
O1—Ni1—O2	98.0 (1)	C1—C6—C5	119.8 (5)
O1—Ni1—N1	89.5 (2)	C7—C6—C1	121.4 (5)
O1—Ni1—O1*A*	83.8 (2)	C7—C6—C5	118.7 (5)
O2—Ni1—N1	94.9 (2)	N1—C7—C6	129.3 (5)
N1—Ni1—N1*A*	94.4 (3)	C7—N1—Ni1	124.3 (4)
Ni1—O1—C1	130.0 (3)	C8—N1—Ni1	120.3 (4)
O1—C1—C6	124.0 (5)	C8—N1—C7	115.3 (5)
O1—C1—C2	120.5 (5)	C9—C8—N1	112.8 (5)
C6—C1—C2	115.5 (5)	C8—C9—C8*A*	115.4 (7)

2 g of the title complex was warmed in 80 ml 96.5% ethanol until completely dissolved. When this solution was left to cool, diamagnetic needle-shaped crystals began to precipitate, but as slow cooling continued, green paramagnetic prismatic crystals were obtained (48 h).

The phase problem was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). All calculations were performed on a NOVA 3/12 computer using *SHELXTL* (Sheldrick, 1983).

This work was supported by the Research Fund of University of Ankara under grant number 91-25-00-10.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71310 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1026]

References

Maggio, F., Pizzino, T. & Romano, V. (1974). *Inorg. Nucl. Chem. Lett.* **10**, 1005–1008.
Sheldrick, G. M. (1983). *SHELXTL Users Manual.* Revision 4. Nicolet XRD Corporation, Madison, Wisconsin, USA.
Sheldrick, G. M. (1986). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Acta Cryst. (1993). C49, 1906–1908

The First Dinuclear Cobalt–Rhenium Cluster Compound: Hexacarbonylbis(μ-dicyclohexylphosphanido)cobaltrhenium

ULRICH FLÖRKE AND HANS-JÜRGEN HAUPT

Anorganische und Analytische Chemie, Universität-GH Paderborn, Warburgerstrasse 100, D-4790 Paderborn, Germany

(*Received* 12 *March* 1993; *accepted* 18 *May* 1993)

Abstract

The title compound [hexacarbonyl-1κ^4C,2κ^2C-bis(μ-dicyclohexylphosphido-1:2κ^2P)cobaltrhenium(*Co*—*Re*) 0.5-methanol solvate] belongs to a type of edge-linked tetrahedron-octahedron coordination complex. The molecule has local C_{2v} symmetry with ecliptic conformation of the carbonyl ligands at both metal centres, and a Co—Re single-bond length of 2.786 (1) Å.

Comment

In the course of our work on phosphanido-bridged homo- and heteronuclear metal-atom cluster compounds (Haupt, Balsaa & Flörke, 1988; Haupt, Heinekamp & Flörke, 1989; Flörke & Haupt, 1993) we isolated the title compound with a Co—Re bond symmetrically bridged by two dicyclohexylphosphanido groups.