C25	0.5112 (3)	0.8416 (3)	0.5733 (3)	0.0388 (7)
C26	0.4149 (4)	0.8250 (3)	0.4856 (4)	0.0502 (9)
C27	0.4692 (4)	0.7885 (3)	0.3782 (4)	0.0556 (10)
C28	0.6182 (4)	0.7616 (3)	0.3539 (3)	0.0509 (9)
C29	0.7139 (4)	0.7764 (3)	0.4354 (3)	0.0416 (7)
H1	0.826 (3)	0.653 (3)	0.798 (3)	0.052 (9)

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

Mo1—Mo2	3.2024 (7)	C4—C5	1.405 (5)
Mo1—C1	1.921 (4)	C5—C6	1.403 (5)
Mo1—C2	1.948 (4)	C6—C7	1.387 (5)
Mo1—C11	2.481 (3)	C11—C19	1.408 (5)
Mo1—C12	2.315 (4)	C11—C12	1.414 (5)
Mo1—C13	2.306 (3)	C11—C15	1.427 (5)
Mo1—C14	2.316 (3)	C12—C13	1.396 (6)
Mo1—C15	2.475 (3)	C13—C14	1.391 (6)
Mo1—C22	2.146 (3)	C14—C15	1.441 (5)
Mo1—H1	1.87 (3)	C15—C16	1.404 (5)
Mo2—C3	1.954 (4)	C16—C17	1.363 (6)
Mo2—C4	2.345 (3)	C17—C18	1.406 (6)
Mo2—C5	2.243 (3)	C18—C19	1.350 (6)
Mo2—C6	2.233 (3)	C21—C22	1.449 (4)
Mo2—C7	2.351 (3)	C21—C25	1.437 (4)
Mo2—C21	2.380 (3)	C21—C29	1.420 (4)
Mo2—C22	2.229 (3)	C22—C23	1.443 (4)
Mo2—C23	2.264 (3)	C23—C24	1.409 (4)
Mo2—C24	2.344 (3)	C24—C25	1.428 (5)
Mo2—C25	2.460 (3)	C25—C26	1.418 (4)
Mo2—H1	1.80 (3)	C26—C27	1.349 (5)
O1—C1	1.159 (4)	C27—C28	1.405 (5)
O2—C2	1.158 (4)	C28—C29	1.358 (5)
O3—C3	1.149 (4)		
C1—Mo1—C2	77.0 (2)	O1—C1—Mo1	178.2 (3)
C1—Mo1—C22	112.84 (13)	O2—C2—Mo1	176.9 (3)
C1—Mo1—*IN*1†	119.4	O3—C3—Mo2	175.2 (3)
C2—Mo1—C22	82.11 (13)	C23—C22—C21	104.9 (2)
C2—Mo1—*IN*1	118.3	C23—C22—Mo1	116.5 (2)
C22—Mo1—*IN*1	126.7	C21—C22—Mo1	133.0 (2)
C3—Mo2—C22	120.09 (12)	C23—C22—Mo2	72.6 (2)
C3—Mo2—*IN*2†	115.4	C21—C22—Mo2	77.5 (2)
C3—Mo2—*BU*†	98.9	Mo1—C22—Mo2	94.09 (11)
C22—Mo2—*BU*	140.0	Mo1—H1—Mo2	122 (3)
*IN*2—Mo2—*BU*	135.6		

† *IN*1, *IN*2 and *BU* are the unweighted centroids of the C11–C15 ring, the C21–C25 ring and the C4–C7 butadiene chain, respectively.

Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 4.2.4.2).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *XCAD*4 (Harms & Wocadlo, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1134). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis[(1,2,5,6-η)-1,5-cyclooctadiene]-di-μ-ethoxo-dirhodium(I)

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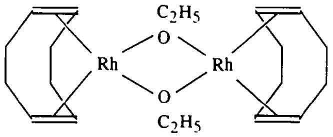
Abstract

The solvent-free title compound, $[Rh_2(C_2H_5O)_2(C_8H_{12})_2]$, was obtained by reaction of $[(cod)Rh(OH)]_2$ (cod is cyclooctadiene) with $Ti(OC_2H_5)_4$. The coordination of the Rh atoms is essentially square planar. The Rh and O atoms form a folded four-membered ring.

Comment

The reaction of the halide derivative $[(cod)RhCl]_2$ (cod is cyclooctadiene) with Na_2CO_3/ethanol or KOH/ethanol led to $[(cod)Rh(OC_2H_5)]_2$, *i.e.* bis(1,5-cyclooctadiene)-di-μ-ethoxo-dirhodium(I), (1). Such reactions may result, however, in hydrated (1) (Green & Meek, 1989) and the solvate water is not removable in these cases. Pure (1) is accessible from $[(cod)_2Rh_2O_2]$ by reaction with ethanol (Sakurai, Suzuki, Moro-oka & Ikawa, 1980). We have found that solvent-free (1) can also be formed by subsequent ion metathesis reaction of $[(cod)Rh(OH)]_2$ with $Ti(OC_2H_5)_4$

and $Sn(OC_2H_5)_4$, respectively. The mixed hydroxo–ethoxo complex $[(cod)_2Rh_2(OH)(OC_2H_5)]$.0.5toluene, was obtained as an intermediate and characterized by spectroscopic and X-ray structure analyses (Selent, Zabel & Ramm, 1996). Furthermore, complex (1) reacts with additional alkoxide to form the new $[Rh(cod)]_2[Ti(OEt)_6]$ (Selent *et al.*, 1996) and the previously described $[Rh(cod)]_2[Sn(OEt)_6]$ (Wark, Gulliver, Hampden-Smith & Rheingold, 1990). Only a few alkoxo-bridged rhodium(I) complexes, including the methoxy homologue $[(cod)Rh(OCH_3)]_2$ (Tanaka, Jin-no, Kushida, Tsutsui, Ashida, Suzuki, Sakurai, Moro-oka & Ikawa, 1983) have been characterized by X-ray structure analysis.



(1)

The molecule of (1) displays crystallographic C_2 symmetry. The coordination of the Rh atom is essentially square planar; it lies 0.046 (6) Å from the least-squares plane formed by the two O atoms and the midpoints of the C3=C10 and C6=C7 double bonds. The Rh and O atoms form a folded four-membered ring; the planes defined by the atoms Rh1, O1, O1^i and Rh1^i, O1, O1^i subtend a dihedral angle of about 121.3 (4)° [symmetry code: (i) $\frac{3}{2}-x, y, \frac{3}{2}-z$]. This value corresponds to those found in other cyclooctadiene complexes of rhodium(I). The Rh—O bond lengths are 2.065 (2) and 2.074 (2) Å, and may be considered normal (see, for example, Selent & Ramm, 1995). Atoms O1, C1 and C2, and their symmetry equivalents do not occupy the same plane because of rotation of the ethyl group about the O1—C1 bond, resulting in different Rh—O1—C1—C2 torsion angles [−138.4 (3) and 101.0 (4)°]. The C4—C5 and C8—C9 single-bond lengths are relatively short, which may be a result of the larger thermal motion of these atoms.

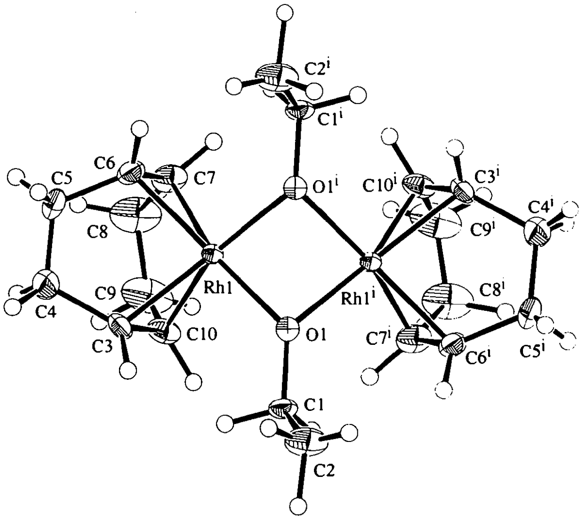


Fig. 1. An *ORTEP* (Johnson, 1971) drawing of the molecular structure of (1) showing the crystallographic numbering scheme (50% probability displacement ellipsoids).

Experimental

Crystals of (1) were obtained from toluene at 250 K.

Crystal data

$[Rh_2(C_2H_5O)_2(C_8H_{12})_2]$
$M_r = 512.3$
Monoclinic
$P2/n$
$a = 12.474$ (2) Å
$b = 6.3201$ (8) Å
$c = 12.418$ (3) Å
$\beta = 94.93$ (3)°
$V = 975.4$ (3) Å^3
$Z = 2$
$D_x = 1.744$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 42 reflections
$\theta = 8.1$–12.1°
$\mu = 1.701$ mm^{-1}
$T = 193$ (1) K
Prism
0.57 × 0.24 × 0.11 mm
Yellow

Data collection

Siemen AED diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration from crystal shape (*Xtal*3.2; Hall, Flack & Stewart, 1992)
$T_{min} = 0.469$, $T_{max} = 0.847$
2215 measured reflections
2120 independent reflections

1815 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0177$
$\theta_{max} = 27°$
$h = 0 \rightarrow 15$
$k = -8 \rightarrow 0$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0287$
$wR(F^2) = 0.0780$
$S = 1.071$
2120 reflections
141 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 2.0877P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.887$ e Å^{-3}
$\Delta\rho_{min} = -0.816$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Rh1	0.63632 (2)	0.38112 (4)	0.75616 (2)	0.01485 (10)
O1	0.7353 (2)	0.2543 (4)	0.6478 (2)	0.0194 (5)
C1	0.7186 (3)	0.2565 (6)	0.5334 (3)	0.0238 (7)
C2	0.7475 (5)	0.0486 (8)	0.4854 (4)	0.0420 (11)
C3	0.4942 (3)	0.4100 (6)	0.6530 (3)	0.0265 (8)
C4	0.3928 (4)	0.4033 (10)	0.7138 (4)	0.0477 (13)
C5	0.4143 (3)	0.3694 (9)	0.8304 (4)	0.0446 (12)
C6	0.5290 (3)	0.4077 (6)	0.8759 (3)	0.0258 (8)

C7	0.5838 (4)	0.5983 (6)	0.8669 (3)	0.0305 (8)
C8	0.5304 (5)	0.7934 (7)	0.8135 (4)	0.0529 (14)
C9	0.5370 (5)	0.8028 (7)	0.6967 (4)	0.0530 (14)
C10	0.5564 (3)	0.5902 (6)	0.6458 (3)	0.0277 (8)

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

Rh1—O1	2.065 (2)	C3—C10	1.386 (5)
Rh1—O1^{i}	2.074 (2)	C3—C4	1.528 (6)
Rh1—C7	2.088 (4)	C4—C5	1.465 (6)
Rh1—C6	2.091 (4)	C5—C6	1.512 (6)
Rh1—C10	2.094 (4)	C6—C7	1.394 (6)
Rh1—C3	2.104 (4)	C7—C8	1.525 (6)
O1—C1	1.417 (4)	C8—C9	1.461 (7)
O1—Rh1^{i}	2.074 (2)	C9—C10	1.513 (5)
C1—C2	1.500 (6)		
O1—Rh1—O1^{i}	75.49 (11)	O1—C1—C2	111.7 (3)
C7—Rh1—C6	39.0 (2)	C10—C3—C4	123.1 (4)
C7—Rh1—C10	81.8 (2)	C5—C4—C3	113.8 (4)
C6—Rh1—C10	96.7 (2)	C4—C5—C6	115.6 (3)
C7—Rh1—C3	92.8 (2)	C7—C6—C5	124.2 (4)
C6—Rh1—C3	82.46 (15)	C6—C7—C8	122.4 (4)
C10—Rh1—C3	38.5 (2)	C9—C8—C7	113.8 (4)
C1—O1—Rh1	127.2 (2)	C8—C9—C10	113.8 (4)
C1—O1—Rh1^{i}	127.7 (2)	C3—C10—C9	126.3 (4)
Rh1—O1—Rh1^{i}	87.16 (9)		

Symmetry code: (i) $\frac{3}{2} - x, y, \frac{3}{2} - z$.

The C7—H7 bond length was restrained. All H-atom parameters were refined except for those of H41, H42, H51, H52, H81, H82, H91, H92 and H10, which were refined using a riding model.

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[*N*-(2-Mercaptophenyl)-2-hydroxynaphthaldiminato](pyridine)nickel(II)

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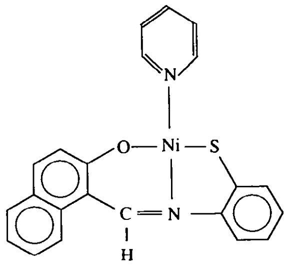
Abstract

The central Ni^{2+} ion in the title complex {systematic name: [1-(2-mercaptophenyliminomethyl)-2-naphtholato-*O*,*N*,*S*](pyridine-*N*)nickel(II); $[Ni(C_{17}H_{11}NOS)(C_5H_5N)]$} is in a slightly distorted square-planar environment of S, O and N atoms. The Ni^{2+} ion is 0.001 (1) Å out of the coordination plane.

Comment

Schiff base complexes have continued to be versatile stereochemical models in main group and transition metal coordination chemistry because of their facile preparation and structural variation (Garnovskii, Nivorozhkin & Minkin, 1993). The purpose of the present study is to elucidate what happens when the nickel(II) ion reacts with the monodentate pyridine ligand and the tridentate 2-mercaptophenyl-2-hydroxynaphthaldehyde ligand. The structure of the resulting complex, (I), has been determined.



(I)

The monodentate and tridentate ligands of (I) are coordinated to nickel(II) in a square-planar configuration (Fig. 1). The bond lengths of several complexes of *N*-substituted salicylaldimines are compared with the present results in Table 3.

The Ni—O and Ni—S distances are 1.843 (2) and 2.141 (2) Å, respectively. The Ni—N1 bond length of 1.862 (2) Å, however, is definitely shorter than Ni—N2 and other literature Ni—N distances. The reason for this

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