$Cd(ReO_4)_2.4CH_4N_2S$

O3	−0.2540 (7)	0.0275 (3)	−0.3780 (4)	0.056 (1)
O4	−0.4875 (7)	−0.1361 (3)	−0.3711 (3)	0.060 (1)
S1	0.0038 (2)	−0.16998 (8)	0.12148 (9)	0.0326 (3)
S2	0.2084 (2)	−0.06799 (8)	−0.13997 (9)	0.0309 (3)
C1	−0.0845 (6)	−0.2643 (3)	0.0222 (4)	0.030 (1)
C2	0.3323 (6)	−0.1656 (3)	−0.0550 (4)	0.0266 (9)
N11	−0.2022 (7)	−0.2481 (3)	−0.0897 (3)	0.041 (1)
N12	−0.0394 (7)	−0.3563 (3)	0.0578 (4)	0.045 (1)
N21	0.4742 (6)	−0.1495 (3)	0.0481 (3)	0.037 (1)
N22	0.2900 (7)	−0.2565 (3)	−0.0927 (4)	0.040 (1)

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

Cd—O1	2.407 (3)	S1—C1	1.711 (4)
Cd—S1	2.701 (1)	C1—N11	1.330 (5)
Cd—S2	2.646 (1)	C1—N12	1.326 (6)
Re—O1	1.736 (3)	S2—C2	1.726 (4)
Re—O2	1.722 (4)	C2—N21	1.326 (5)
Re—O3	1.705 (5)	C2—N22	1.315 (6)
Re—O4	1.706 (4)		
O1—Cd—S1	95.20 (8)	O2—Re—O3	107.9 (2)
O1—Cd—S2	89.90 (9)	O2—Re—O4	111.5 (2)
S1—Cd—S2	95.49 (4)	O3—Re—O4	109.1 (2)
O1—Re—O2	110.3 (2)	N11—C1—N12	118.7 (4)
O1—Re—O3	109.7 (2)	N21—C2—N22	119.3 (4)
O1—Re—O4	108.3 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	H···A	D···A	D—H···A	A···H···A
N11—H111···O1	2.08 (1)	3.028 (5)	174 (1)	
N11—H112···O2[i]	2.13 (1)	2.962 (6)	146 (1)	
N12—H121···O2[i]	2.41 (1)	3.168 (5)	137 (1)	84 (1)
N12—H121···O3[ii]	2.60 (1)	2.983 (6)	105 (1)	
N12—H122···S2[ii]	2.62 (1)	3.562 (4)	168 (1)	
N21—H211···O1[iii]	2.52 (1)	3.194 (5)	128 (1)	
N21—H211···S2[iv]	2.81 (1)	3.667 (4)	150 (1)	82 (1)
N21—H212···O4[v]	2.24 (1)	3.049 (6)	143 (1)	
N22—H221···O3[vi]	2.41 (1)	2.961 (5)	117 (1)	93 (1)
N22—H221···O4[v]	2.12 (1)	2.961 (5)	147 (1)	
N22—H222···S1[vii]	2.54 (1)	3.467 (4)	164 (1)	

Symmetry codes: (i) $-1 - x, -\frac{1}{2} + y, -\frac{1}{2} - z$; (ii) $x, -\frac{1}{2} - y, \frac{1}{2} + z$; (iii) $-x, -y, -z$; (iv) $1 - x, -y, -z$; (v) $1 + x, -\frac{1}{2} - y, \frac{1}{2} + z$; (vi) $-x, -\frac{1}{2} + y, -\frac{1}{2} - z$; (vii) $x, -\frac{1}{2} - y, -\frac{1}{2} + z$.

The H atoms were situated at calculated positions and refined as riding with $U_{iso} = 0.0506$ Å^2.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Data reduction: *SDP/PDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SDP/PDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Macíček, unpublished).

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Bis(N-2-propylsalicylideneamino-N,O)-cobalt(II)

YALÇIN ELERMAN,[a] MEHMET KABAK[a] AND M. NAWAZ TAHIR[b]

[a]*Department of Engineering Physics, Faculty of Sciences, University of Ankara, 06100 Tandogan, Ankara, Turkey, and* [b]*Department of Engineering Physics, Faculty of Engineering, Hacettepe University, 06532 Beytepe, Ankara, Turkey. E-mail: elerman@eros.science.ankara.edu.tr*

Abstract

The title complex, bis[2-(2-propyliminomethyl)phenolato-N,O]cobalt(II), [Co(C$_{10}$H$_{12}$NO)$_2$], has two chelating Schiff base ligands coordinating through their N and O atoms to give a tetrahedral geometry distorted mainly by reduction of the chelate O—Co—N angles and by expansion of the N—Co—N angle because of steric hindrance of the isopropyl substituents. The Co—O bonds [mean 1.906 (5) Å] are shorter than the Co—N bonds [mean 1.992 (8) Å].

Comment

The reaction between Co^{II} and bidentate anionic Schiff bases such as salicylidene results in stable organometal-

lic derivatives (Pahor, Calligaris, Delise, Dodic, Nardin & Randaccio, 1976); metal complexes of Schiff bases in general have been extensively studied (Calligaris, Nardin & Randaccio, 1972). We describe here the structure of the bis(*N*-isopropylsalicylideneamino)cobalt(II), (I), complex and compare it with related compounds having different substituents.

(I)
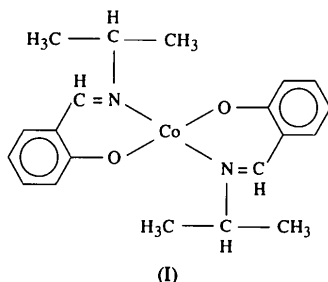
The two bidentate ligands are coordinated to the Co^{II} ion through their N and O atoms. The geometry around cobalt is somewhat distorted from ideal tetrahedal, mainly as a consequence of the restricted bite of the chelate ligands (reducing intra-ligand O—Co—N angles) and steric repulsion of the isopropyl groups (increasing the N—Co—N angle). The dihedral angle between the two Schiff base ligands, excluding the isopropyl groups, is 82.88 (5)°, so that the two ligands are almost perpendicular, the deviation from an ideal value

of 90° also being due to the isopropyl groups. Geometric differences between the two ligands are minor. The sum of the bond angles at the two N atoms is 360.0 (2)° in each case, which is appropriate for sp^2 hybridization.

The coordination geometry is very similar to that found for corresponding complexes in which the isopropyl groups are replaced by either *n*-butyl or *tert*-butyl groups, these complexes retaining the distorted tetrahedral coordination (Bahron, Larkworthy, Marecaux, Povey & Smith, 1994); the mean Co—O and Co—N distances in the present structure are 1.906 (5) and 1.992 (8) Å, respectively, with corresponding values of 1.894 (4) and 1.968 (4) Å for the averages of the *n*-butyl and *tert*-butyl structures. In contrast, linking the two ligands by a two-carbon bridge between the N atoms forces an approximate square-planar coordination geometry in which the Co—O bonds are shorter by about 0.06 Å and the Co—N bonds are drastically shortened by about 0.13 Å (Pahor *et al.*, 1976; Hiller, Nishinaga, Tsutsui & Rieker, 1993). Octahedral coordination of Co^{III} by three similar salicylideneamino ligands gives mean Co—O and Co—N bond lengths of 1.893 (3) and 1.948 (4) Å, respectively, the smaller intrinsic size of the Co^{III} ion being largely compensated for, as is generally observed, by an increase in the effective size with a larger coordination number (Elerman, Kabak, Svoboda & Geselle, 1994).

Experimental

Salicylaldehyde (0.2 mmol) and isopropylamine (1 ml) were dissolved in acetonitrile (50 ml). $CoCl_2.6H_2O$ (0.1 mmol) in boiling methanol (30 ml) was added and the solution refluxed for 48 h. Suitable crystals were obtained on cooling.

Crystal data

$[Co(C_{10}H_{12}NO)_2]$	Mo $K\alpha$ radiation
$M_r = 383.36$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25
Pbca	reflections
$a = 13.2001 (12)$ Å	$\theta = 10.1–18.1°$
$b = 19.4239 (15)$ Å	$\mu = 0.89$ mm^{-1}
$c = 15.1868 (13)$ Å	$T = 297$ K
$V = 3893.9 (4)$ Å^3	Prism
$Z = 8$	$0.35 \times 0.30 \times 0.30$ mm
$D_x = 1.308$ Mg m^{-3}	Red
D_m not measured	

Data collection

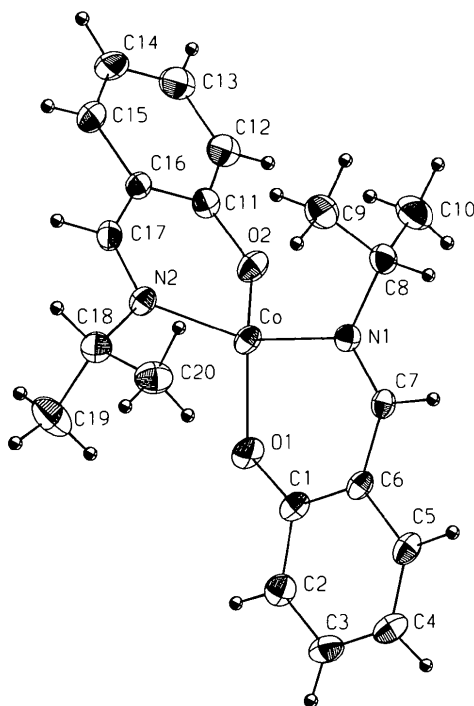|	
Enraf–Nonius CAD-4 diffractometer	3234 independent reflections
$\omega/2\theta$ scans	2156 observed reflections
Absorption correction:	$[I > 3\sigma(I)]$
empirical *via* ψ scan	$\theta_{max} = 26.3°$
(Kopfmann & Huber,	$h = 0 \rightarrow 16$
1968; North, Phillips &	$k = 0 \rightarrow 24$
Mathews, 1968)	$l = 0 \rightarrow 18$
$T_{min} = 0.72$, $T_{max} = 0.79$	3 standard reflections
3234 measured reflections	frequency: 120 min
	intensity decay: 1%

Fig. 1. The molecular structure of (I) showing the atom-labelling scheme and 50% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976).

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.003$
$R = 0.035$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$wR = 0.040$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
$S = 1.27$	Extinction correction: none
2156 reflections	Atomic scattering factors
226 parameters	from *International Tables*
H atoms riding with fixed	*for Crystallography* (1992,
$U_{iso} = 0.1$ Å^2	Vol. C, Tables 4.2.6.8 and
$w = 1/[\sigma^2(F)]$	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}
Co	0.45644 (3)	0.19228 (2)	0.61781 (3)	0.042 (1)
O1	0.4687 (1)	0.2759 (1)	0.6820 (1)	0.053 (1)
O2	0.3488 (1)	0.1307 (1)	0.6473 (1)	0.051 (1)
N1	0.5928 (2)	0.1549 (1)	0.6493 (2)	0.039 (1)
N2	0.4141 (2)	0.2085 (1)	0.4942 (2)	0.042 (1)
C1	0.5523 (2)	0.2993 (2)	0.7187 (2)	0.043 (1)
C2	0.5520 (2)	0.3662 (2)	0.7544 (2)	0.052 (2)
C3	0.6356 (3)	0.3941 (2)	0.7936 (2)	0.057 (2)
C4	0.7246 (2)	0.3564 (2)	0.8001 (2)	0.058 (2)
C5	0.7279 (2)	0.2911 (2)	0.7675 (2)	0.048 (2)
C6	0.6432 (2)	0.2606 (2)	0.7256 (2)	0.038 (1)
C7	0.6557 (2)	0.1915 (2)	0.6941 (2)	0.039 (2)
C8	0.6235 (2)	0.0840 (2)	0.6235 (2)	0.049 (2)
C9	0.6150 (3)	0.0763 (2)	0.5252 (2)	0.072 (2)
C10	0.5597 (3)	0.0327 (2)	0.6720 (3)	0.076 (2)
C11	0.2874 (2)	0.1018 (2)	0.5898 (2)	0.045 (2)
C12	0.2203 (2)	0.0504 (2)	0.6200 (2)	0.057 (2)
C13	0.1538 (2)	0.0184 (2)	0.5646 (3)	0.069 (2)
C14	0.1501 (3)	0.0348 (2)	0.4770 (3)	0.071 (2)
C15	0.2140 (2)	0.0846 (2)	0.4445 (2)	0.060 (2)
C16	0.2843 (2)	0.1183 (1)	0.4993 (2)	0.044 (2)
C17	0.3438 (2)	0.1719 (1)	0.4583 (2)	0.048 (2)
C18	0.4577 (3)	0.2651 (2)	0.4396 (2)	0.056 (2)
C19	0.4054 (3)	0.3322 (2)	0.4643 (3)	0.092 (3)
C20	0.5707 (3)	0.2688 (2)	0.4548 (2)	0.068 (2)

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

Co—O1	1.901 (2)	N1—C7	1.288 (4)
Co—O2	1.910 (2)	N1—C8	1.489 (4)
Co—N1	1.999 (2)	N2—C17	1.290 (4)
Co—N2	1.984 (2)	N2—C18	1.492 (4)
O1—C1	1.317 (3)	C1—C2	1.408 (4)
O2—C11	1.318 (4)		
O1—Co—O2	118.53 (9)	Co—O2—C11	124.7 (2)
O1—Co—N1	96.35 (9)	Co—N1—C7	120.5 (2)
O1—Co—N2	111.93 (9)	Co—N1—C8	121.2 (2)
O2—Co—N1	112.72 (9)	C7—N1—C8	118.3 (2)
O2—Co—N2	96.43 (9)	Co—N2—C17	121.0 (2)
N1—Co—N2	122.6 (1)	Co—N2—C18	122.3 (2)
Co—O1—C1	125.7 (2)	C17—N2—C18	116.7 (2)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1993). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SIMPEL* (Perchar & Schenk, 1987). Program(s) used to refine structure: *MolEN* (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1056). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Aquabis(p-nitrosalicylato-O,O')lead(II)

M. Nawaz Tahir,[a] Dinçer Ülkü[a] and Elman M. Mövsümov[b]†

[a]*Department of Physics Engineering, Hacettepe University, Beytepe 06532, Ankara, Turkey, and* [b]*Department of Chemistry, Hacettepe University, Beytepe 06532, Ankara, Turkey. E-mail: dulku@eti.cc.hun.edu.tr*

Abstract

In the crystal structure of the title compound, [Pb-(C₇H₄NO₅)₂(H₂O)], the sevenfold coordination around the Pb atom consists of four O atoms from two chelating carboxylate groups, one water O atom and two additional O atoms belonging to a carboxyl and a hydroxy group of neighbouring molecules. The Pb—O distances are in the range 2.419 (4)–3.084 (5) Å, the shortest distance being to a water O atom. Two intramolecular and two intermolecular hydrogen bonds are also observed in the polymeric structure.