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Bis{(*E*)-6-methoxy-2-[(4-methylphenyl)-iminomethyl]phenolato}cobalt(II)

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.076
Data-to-parameter ratio = 17.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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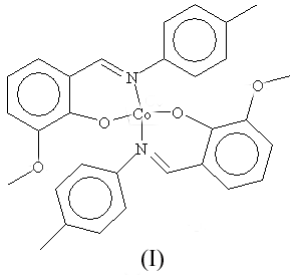
The title compound, $[Co(C_{14}H_{14}NO_2)_2]$ (CoSPT), crystallizes with one half molecule per asymmetric unit. The Co atom sits on a twofold axis. The tridentate ligands coordinate to the cobalt ion through the hydroxy O atom and the azomethine N atom, forming a distorted tetrahedral geometry around the metal ion.

Comment

Schiff base ligands derived from substituted salicylaldehyde and aniline and their metal complexes have been widely investigated because of their novel structural features (Burrows & Bailar, 1966; Nakao *et al.*, 1967; Theriot *et al.*, 1969, 1973; Moustakali-Mavridis *et al.*, 1980; Wu *et al.*, 1990; Wang & Chang, 1994). In a previous publication, Maurya *et al.* (1994) proposed an octahedral geometry for 2-hydroxybenzylideneanthranilic acid and 2-hydroxybenzylidene-2-aminothiophenol, where the ligands coordinated to the metal ions through all three donor atoms. This geometry was based on analytical data, conductance measurement, magnetic measurement and IR spectral studies. However, no X-ray crystallographic confirmations of these structures have been reported. We describe here the synthesis and crystal structure of a cobalt complex, (I), of a new analogous Schiff base derived from *o*-vanillin and *p*-toluidine. This work forms part of our studies of the crystal structures of new Schiff bases and their metal complexes, aiming to determine the true mode of coordination of these tridentate ligands.



(I)

The molecular structure of CoSPT is shown in Fig. 1. The molecule sits on a twofold axis. The ligands form a distorted tetrahedral geometry around the cobalt ion through the hydroxy O atom and the azomethine N atom (Fig. 2). There is no coordination of the cobalt ion by the methoxy O atom.

Experimental

All reagents were used as received without further purification. *o*-Vanillin and *p*-toluidine were purchased from Aldrich Chemical Company, while diethyl ether and cobalt(II) acetate were obtained from Fisher Scientific Company. *p*-Toluidine (4.28 g, 40 mmol) was placed in a beaker and heated gently on a hot-plate. *o*-Vanillin (3.34 g, 21 mmol) was added slowly until the mixture melted. The

mixture was then cooled to room temperature before diethyl ether (80 ml) was added. The resulting solution was filtered to remove unreacted materials. Crystals of the ligand were obtained by slow evaporation of the solution (yield 64.6%, m.p. 372–374 K). The ligand was characterized by IR, UV–vis, elemental and X-ray analyses. The cobalt(II) complex was prepared by mixing a methanol solution (20 ml) of cobalt acetate (0.1245 g) with a methanol solution (50 ml) of the ligand in a round-bottomed flask. The mixture was refluxed for 1 h and filtered. Slow evaporation of the filtrate yielded good quality red parallelepiped crystals (yield 60.8%, m.p. 503–506 K).

Crystal data

$[Co(C_{14}H_{14}NO_2)_2]$
M_r = 539.47
Monoclinic, $C2/c$
a = 14.0496 (6) Å
b = 16.2459 (6) Å
c = 11.8597 (5) Å
β = 106.976 (2)°
V = 2589.01 (18) Å^3
Z = 4
D_x = 1.384 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9120 reflections
θ = 1.0–27.5°
μ = 0.70 mm^{-1}
T = 90.0 (2) K
Parallelpiped, red
0.25 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
ω scans at fixed χ = 55°
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.844, T_{max} = 0.872
9599 measured reflections
2955 independent reflections
2407 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.5°
$h = -18 \rightarrow 18$
$k = -21 \rightarrow 19$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.076
S = 1.11
2955 reflections
171 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0151P)^2 + 2.0584P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.014
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.9149 (12)	C4—C5	1.364 (3)
Co1—N1	2.0025 (14)	C5—C6	1.419 (2)
N1—C7	1.306 (2)	C6—C7	1.435 (2)
N1—C8	1.436 (2)	C8—C13	1.391 (2)
O1—C1	1.311 (2)	C8—C9	1.393 (2)
O2—C2	1.378 (2)	C9—C10	1.381 (2)
O2—C15	1.433 (2)	C10—C11	1.394 (3)
C1—C6	1.413 (2)	C11—C12	1.386 (3)
C1—C2	1.440 (2)	C11—C14	1.509 (3)
C2—C3	1.371 (2)	C12—C13	1.384 (3)
C3—C4	1.401 (3)		
O1—Co1—O1^{i}	117.54 (7)	C5—C4—C3	119.99 (17)
O1—Co1—N1	95.92 (5)	C4—C5—C6	121.08 (17)
O1^{i}—Co1—N1	111.59 (5)	C1—C6—C5	119.99 (16)
N1—Co1—N1^{i}	125.94 (8)	C1—C6—C7	124.40 (16)
C7—N1—C8	117.86 (15)	C5—C6—C7	115.56 (16)
C7—N1—Co1	120.14 (12)	N1—C7—C6	128.25 (17)
C8—N1—Co1	121.85 (11)	C13—C8—C9	118.42 (17)
C1—O1—Co1	125.80 (11)	C13—C8—N1	118.32 (16)
C2—O2—C15	116.03 (14)	C9—C8—N1	123.26 (16)
O1—C1—C6	124.55 (16)	C10—C9—C8	120.21 (17)
O1—C1—C2	118.29 (16)	C9—C10—C11	121.77 (18)
C6—C1—C2	117.15 (15)	C12—C11—C10	117.48 (17)
C3—C2—O2	124.82 (16)	C12—C11—C14	121.69 (17)
C3—C2—C1	121.20 (17)	C10—C11—C14	120.82 (18)
O2—C2—C1	113.97 (15)	C13—C12—C11	121.31 (17)
C2—C3—C4	120.55 (17)	C12—C13—C8	120.75 (18)

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

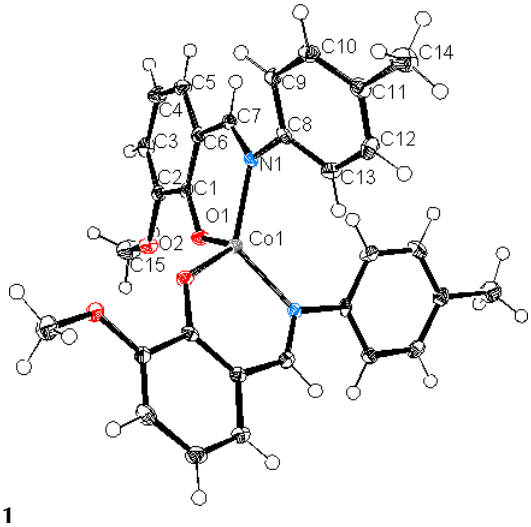


Figure 1
The molecular structure of (I), showing the atom numbering scheme for the asymmetric unit. Displacement ellipsoids are drawn at the 50% level.

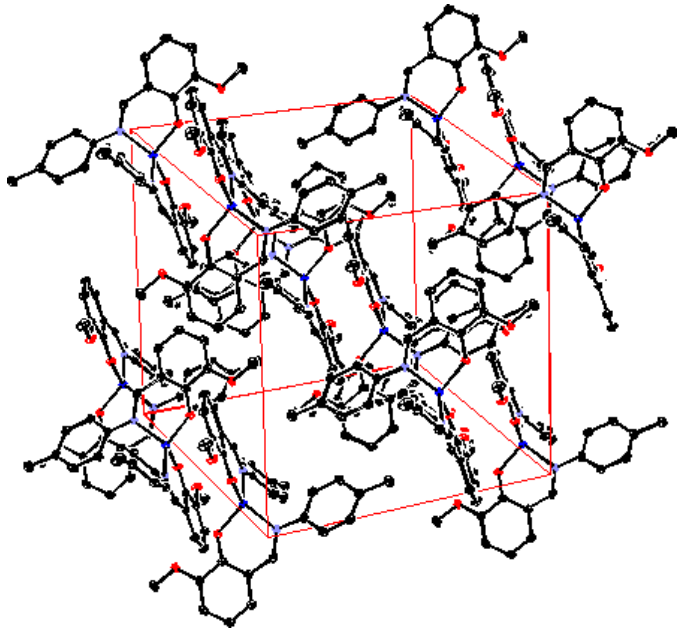


Figure 2
A view of the unit cell of (I).

All H atoms were found in difference Fourier maps and treated as riding [C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl atoms, and C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms].

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL*97 and local procedures.

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