

Tris(8-quinolinolato- κ^2N,O)cobalt(III) methanol solvate

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Key indicators

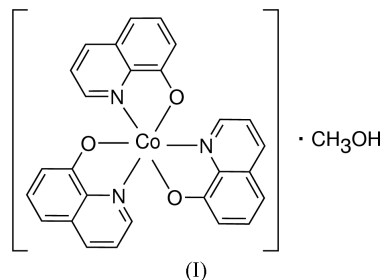
Single-crystal X-ray study
 $T = 298\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 H-atom completeness 82%
 Disorder in solvent or counterion
 R factor = 0.042
 wR factor = 0.130
 Data-to-parameter ratio = 16.8

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

The title cobalt(III) complex, $[\text{Co}(\text{C}_9\text{H}_6\text{NO})_3]\cdot\text{CH}_3\text{OH}$, features three 8-quinolinolate ligands that chelate the cobalt(III) atom with an octahedral coordination geometry. The methanol solvent molecule is disordered in the structure. The $\text{O}\cdots\text{O}$ distances 2.726 (7) and 2.871 (8) \AA between the quinolinol hydroxyl and disordered methanol suggest the existence of hydrogen bonding. The separation distances of 3.404 (15) and 3.386 (7) \AA between parallel quinoline rings indicate π - π stacking between neighboring complex molecules.

Comment

As part of an investigation into π - π stacking interactions in metal complexes, several 8-quinolinolate metal complexes have been prepared in our laboratory (Li *et al.*, 2003). Compound (I) is a representative of this group.



The structure of (I) is shown in Fig. 1. Three 8-quinolinolate monoanions chelate to the Co^{III} atom with an octahedral coordination geometry. The planar 8-quinolinolate monoanions are nearly perpendicular to each other, with dihedral angles of 93.80 (7), 95.34 (6) and 91.49 (7) $^\circ$. The overlapped arrangement of neighboring parallel quinoline rings is shown in Fig. 2. The quinoline plane containing atom N11 is separated from the quinoline plane containing N11(1 - x, 1 - y, 1 - z) by 3.404 (15) \AA . Likewise, the quinoline plane containing atom N31 is separated from the quinoline plane containing N31(1 - x, 1 - y, -z) by 3.386 (7) \AA . These findings indicate the existence of π - π stacking between the neighboring complex molecules.

The methanol solvent molecule is disordered, both C and O atoms being located in two sites with 0.5 occupancy factors. Although H atoms of the disordered methanol were not located, the $\text{O}1a\cdots\text{O}11$ distance of 2.726 (7) \AA and the $\text{O}1b\cdots\text{O}11$ distance of 2.871 (8) \AA suggest the existence of hydrogen bonding between the quinolinol hydroxyl group and the disordered methanol molecule. The $\text{C}24-\text{H}24\cdots\text{O}1b(\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z)$ angle of 133 $^\circ$ and the

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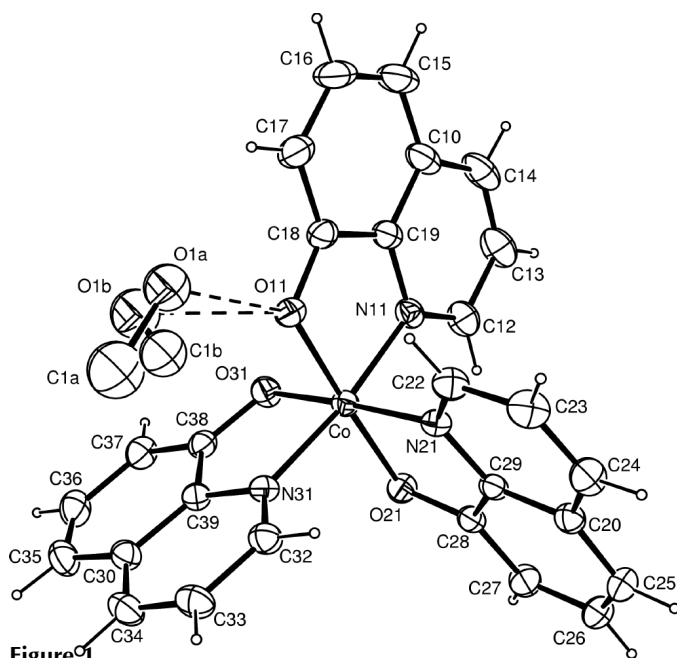


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. Dashed lines indicate hydrogen bonds. Both disorder components of methanol are shown.

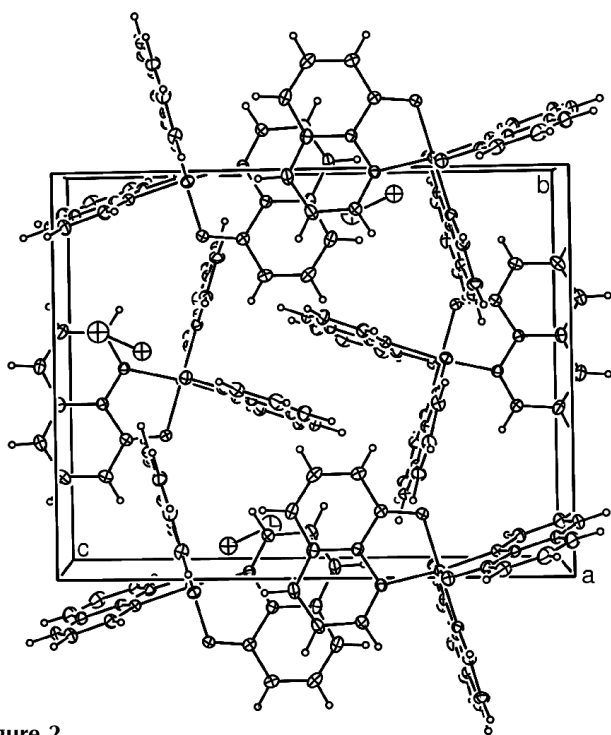


Figure 2
A packing diagram, showing the π - π interaction between neighboring quinoline rings.

$C24 \cdots O1b$ distance of 3.154 (8) Å suggest that the disordered methanol molecule is also involved in weak C—H \cdots O hydrogen bonding.

Compound (I) is isomorphous with the manganese(III) compound (Hems & Mackay, 1975). The major difference between the Co and Mn complexes is the length of the coordination bonds: Mn—O distances range from 1.905 to

1.924 Å and Co—O distances range from 1.8975 (19) to 1.9066 (19) Å; Mn—N distances range from 2.059 to 2.266 Å, while Co—N distances range from 1.921 (2) to 1.936 (2) Å. The other difference between the two structures is that the solvent methanol molecule in the Mn structure was not treated with a disorder model, even though the C—O distance of 1.352 Å is shorter than the expected value.

Experimental

The complex was prepared by refluxing a methanol solution (15 ml) containing $CoCl_2 \cdot 6H_2O$ (0.24 g, 1 mmol) and 8-quinolinol (0.15 g, 1 mmol) for 2 h. The resulting solution was filtered at room temperature. Brown single crystals were obtained from the filtrate after two weeks.

Crystal data

$[Co(C_9H_6NO)_3] \cdot CH_4O$
 $M_r = 523.42$
 Monoclinic, $P2_1/n$
 $a = 10.9235$ (12) Å
 $b = 13.1172$ (14) Å
 $c = 16.6861$ (14) Å
 $\beta = 97.325$ (8)°
 $V = 2371.4$ (4) Å³
 $Z = 4$

$D_x = 1.466$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 9762 reflections
 $\theta = 3.0$ – 27.0°
 $\mu = 0.77$ mm⁻¹
 $T = 298$ (2) K
 Prism, brown
 $0.36 \times 0.20 \times 0.10$ mm

Data collection

Rigaku R-AXIS-RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{min} = 0.750$, $T_{max} = 0.918$
 10460 measured reflections

5432 independent reflections
 4009 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.020$
 $\theta_{max} = 27.5^\circ$
 $h = -14 \rightarrow 14$
 $k = -17 \rightarrow 17$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.130$
 $S = 1.04$
 5432 reflections
 323 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.1393P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.003$
 $\Delta\rho_{max} = 0.59$ e Å⁻³
 $\Delta\rho_{min} = -0.42$ e Å⁻³

Table 1

Selected geometric parameters (Å).

Co—O31	1.8975 (19)	Co—N11	1.921 (2)
Co—O21	1.8994 (18)	Co—N21	1.924 (2)
Co—O11	1.9066 (19)	Co—N31	1.936 (2)

The disordered methanol molecule was refined isotropically and its H atoms were not located. Other H atoms were placed in calculated positions with C—H = 0.93 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atoms.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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