

Tris(8-quinolinolato- κ^2N,O)chromium(III) ethanol solvate

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Key indicators

Single-crystal X-ray study

$T = 298$ K

Mean $\sigma(C-C) = 0.006$ Å

R factor = 0.058

wR factor = 0.162

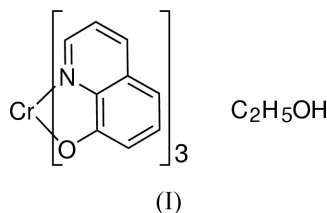
Data-to-parameter ratio = 13.1

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

The title chromium(III) complex, $[Cr(C_9H_6NO)_3] \cdot C_2H_5O$, is isomorphous with the manganese(III) compound [Xiong *et al.* (1995). *Acta Cryst.* **C51**, 1978–1980]. Three 8-quinolinolate ligands chelate the Cr^{III} atom to form an approximately octahedral coordination geometry. An ethanol solvent molecule hydrogen bonds to the complex with an $O \cdots O$ distance of 2.758 (5) Å and an $O-H \cdots O$ angle of 167°. The separation distances of 3.426 (19) and 3.368 (4) Å between parallel quinoline rings suggests the existence of π - π stacking between neighboring complex molecules.

Comment

As part of a series of investigations on π - π -stacking interactions in metal complexes, several 8-quinolinolate–metal complexes have been synthesized in the laboratory.



The structure of the title complex, (I), is shown in Fig. 1. Three 8-quinolinolate ligands chelate to the Cr^{III} atom in an octahedral coordination geometry. The planar 8-quinolinolate ligands are almost perpendicular to each other, with dihedral angles of 85.84 (10), 85.40 (11) and 81.91 (14)°. The overlapped disposition of neighboring parallel quinoline rings is shown in Fig. 2. The quinoline plane containing atom N21 is separated from the quinoline plane containing N21($-x, -y, 1 - z$) by 3.426 (19) Å. Likewise, the plane containing atom N31 and the plane containing N31($-x, -y, -z$) are separated by 3.368 (4) Å. These findings strongly suggest the existence of π - π stacking in the crystal structure (Fig. 2).

The ethanol solvent molecule is hydrogen bonded to the Cr^{III} complex, with an $O1 \cdots O31$ distance of 2.758 (5) Å and an $O1-H1 \cdots O31$ angle of 167° (Table 2). Weak $C-H \cdots O$ hydrogen bonding occurs between atom O11 and quinoline atoms C15 and C24.

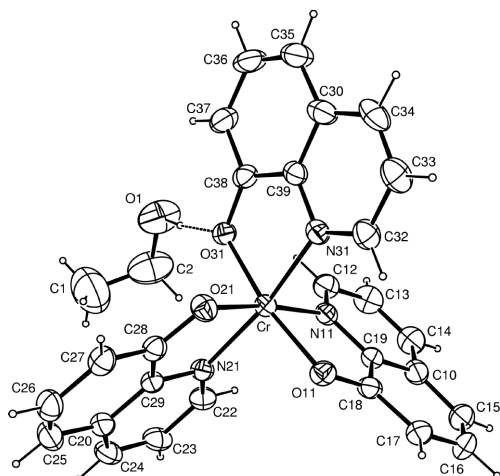
Experimental

The title complex was prepared by refluxing an ethanol solution (15 ml) containing $CrCl_3 \cdot 6H_2O$ (0.27 g, 1 mmol), 8-quinolinol (0.29 g, 2 mmol) and imidazole (0.14 g, 2 mmol) for 2 h. The resulting solution was filtered at room temperature. Green single crystals were obtained from the filtrate after 2 weeks.

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Figure 1

A view of the molecular structure of (I), with 30% probability displacement ellipsoids and the atom-numbering scheme. The dashed line indicates the hydrogen bond between the complex and the solvent molecule.

Crystal data

$[\text{Cr}(\text{C}_9\text{H}_6\text{NO})_3] \cdot \text{C}_2\text{H}_6\text{O}$

$M_r = 530.51$

Monoclinic, $P2_1/n$

$a = 11.2683$ (11) Å

$b = 13.2572$ (11) Å

$c = 16.8041$ (18) Å

$\beta = 94.783$ (6)°

$V = 2501.6$ (4) Å³

$Z = 4$

$D_x = 1.409$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 8818

reflections

$\theta = 2.5\text{--}24.5^\circ$

$\mu = 0.50$ mm⁻¹

$T = 298$ (2) K

Prism, green

$0.36 \times 0.32 \times 0.20$ mm

Data collection

Bruker SMART CCD

diffractometer

ω and φ scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 1999)

$T_{\min} = 0.84$, $T_{\max} = 0.91$

8913 measured reflections

4362 independent reflections

3346 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 25.0^\circ$

$h = -13 \rightarrow 8$

$k = -13 \rightarrow 15$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.162$

$S = 1.10$

4362 reflections

334 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2 + 2.5324P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

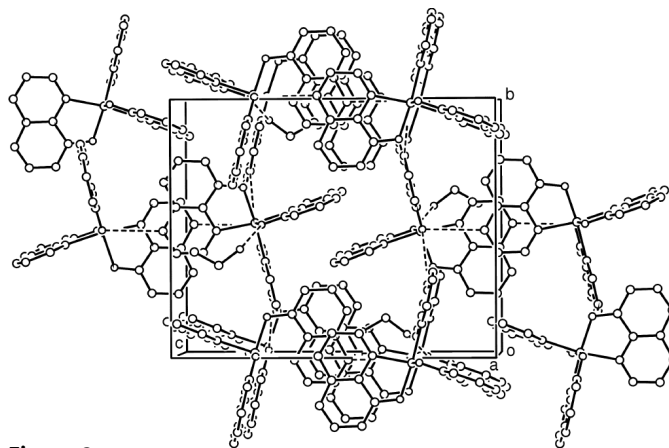
$\Delta\rho_{\max} = 0.47$ e Å⁻³

$\Delta\rho_{\min} = -0.44$ e Å⁻³

Table 1

Selected geometric parameters (Å).

Cr—O21	1.950 (3)	Cr—N31	2.053 (3)
Cr—O11	1.974 (3)	Cr—N11	2.068 (3)
Cr—O31	1.975 (2)	Cr—N21	2.072 (3)


Figure 2

A packing diagram, showing the π - π interactions between neighboring quinoline rings.

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 ⁱ ···O31	0.87	1.90	2.758 (5)	167
C15—H15 ⁱ ···O11 ⁱ	0.93	2.52	3.377 (5)	153
C24—H24 ⁱ ···O11 ⁱⁱ	0.93	2.59	3.401 (4)	146

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

The hydroxyl H atom was located in a difference Fourier map, and included in the final cycles of refinement with fixed positional parameters and displacement parameter $U_{\text{iso}} = 0.08$ Å². Other H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ or $1.5U_{\text{eq}}$ of the carrier atoms.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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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