Trans,trans,trans-Diethanoldiquinaldinatoiron(II)

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trans,trans,trans*-Diethanoldiquinaldinatoiron(II)*Kunitoyo Osawa *et al.*****Synopsis****Queries and comments**

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trans,trans,trans-Diethanoldiquinaldinatoiron(II)

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trans,trans,trans-Diethanoldiquinaldinatoiron(II)

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Key indicators

Single-crystal X-ray study
 $T = 123\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.039
 wR factor = 0.069
Data-to-parameter ratio = 16.6

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

The title complex, *trans,trans,trans*-[Fe^{II}(C₁₀H₆NO₂)₂-(C₂H₆O)₂], is centrosymmetric and the quinaldinate ligands form five-membered chelate rings. The geometry of the complex is distorted octahedral, with a *trans*-FeN₂O₄ chromophore. The hydroxy H atom forms an intermolecular hydrogen bond with the carbonyl O atom of the quinaldinate ligand.

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Comment

Quinaldic acid is associated with tryptophan metabolism (Zhou *et al.*, 1989) and is used as a reagent for solvent extraction of divalent transition metal ions (Högberg *et al.*, 1985). There are few structural studies of quinaldinate complexes in spite of numerous studies of related picolinate complexes. Only the Cu²⁺ (Haendler, 1986), Rh⁺ (Lamprecht *et al.*, 1986) and Ga³⁺ (Li *et al.*, 1996) complexes have been structurally characterized. Therefore, structural information of another transition metal complex is desired.

The title complex, (I), is monomeric and has a distorted octahedral structure, with the central atom lying on an inversion center (Fig. 1 and Table 1). The complex has a *trans,trans,trans*-geometry with respect to three kinds of donors. The quinaldinate acts as a planar *N,O*-bidentate ligand and forms a five-membered chelate ring upon coordination. Two quinaldinato ligands are connected by weak intramolecular hydrogen bonds; the distance between atoms C9 and O1ⁱ is 3.152 (3) Å [symmetry code: (i) $-x, 1-y, -z$].

There exists a strong hydrogen bond between an ethanol molecule and the uncoordinated O atom of a neighboring quinaldinate ligand. The distance between atoms O3 and O2ⁱⁱ is 2.694 (3) Å [symmetry code: (ii) $1-x, 1-y, -z$]. The hydrogen bonds form one-dimensional molecular chains parallel to the *a* axis. The chains are connected by weak hydrogen bonds (Table 2).

Experimental

The title complex was prepared under an N₂ atmosphere using Schlenk techniques. To a solution of Fe(BF₄)₂·6H₂O (0.134 g, 0.397 mmol) in 1.6 ml ethanol was added a solution containing quinaldic acid (0.173 g, 0.999 mmol) in ethanol (6 ml) and triethylamine (140 ml, 0.100 mmol). After vigorous stirring, the solution was allowed to stand for 2 d to afford red-violet crystals suitable for X-ray analysis. The IR spectrum shows a $\nu(\text{CO}_2)$ band at 1628 cm⁻¹. The electronic spectrum in DMF exhibits an absorption maximum at 527 nm ($\epsilon = 795$).

Crystal data

[Fe(C₁₀H₆NO₂)₂(C₂H₆O)₂]

$M_r = 492.30$

Monoclinic, $P2_1/n$

$a = 5.816$ (2) Å

$b = 9.557$ (3) Å

$c = 19.948$ (5) Å

$\beta = 91.461$ (7)°

$V = 1108.4$ (6) Å³

$Z = 2$

$D_x = 1.475$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 4502

reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.72$ mm⁻¹

$T = 123$ K

Prism, red-violet

$0.20 \times 0.05 \times 0.05$ mm

Data collection

Rigaku/MSM Mercury CCD
diffractometer

ω scans

Absorption correction: multi-scan
(Jacobson, 1998)

$T_{\min} = 0.783$, $T_{\max} = 0.964$

8886 measured reflections

2511 independent reflections

2006 reflections with $F^2 > 2\sigma(F^2)$

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

$\theta_{\max} = 27.5^\circ$

$h = -7 \rightarrow 7$

$k = -12 \rightarrow 12$

$l = -25 \rightarrow 25$

Refinement

Refinement on F

$R = 0.039$

$wR = 0.069$

$S = 1.07$

2506 reflections

151 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o) + 0.00168|F_o|^2]$

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

$\Delta\rho_{\max} = 0.33$ e Å⁻³

$\Delta\rho_{\min} = -0.27$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Fe—O1	2.032 (2)	Fe—N1	2.240 (2)
Fe—O3	2.154 (2)		
O1—Fe—O3	92.01 (8)	O3—Fe—N1	93.82 (8)
O1—Fe—N1	77.30 (8)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H6 $\cdots$ O1 ⁱ	0.96	2.27	3.152 (3)	153
O3—H7 $\cdots$ O1 ⁱⁱ	0.96	2.51	3.192 (3)	128
O3—H7 $\cdots$ O2 ⁱⁱ	0.96	1.74	2.694 (3)	172
C4—H2 $\cdots$ O2 ⁱⁱⁱ	0.96	2.50	3.359 (3)	149
C6—H3 $\cdots$ O2 ⁱⁱⁱ	0.96	2.57	3.410 (3)	146

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

Figure 1

ORTEP-3 drawing (Farrugia, 1997) of (I), half of which defines the asymmetric unit, showing the atomic numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 50% probability level.

Figure 2

Packing diagram of the title complex. Dotted lines show hydrogen bonding, which forms molecular chains parallel to the a axis.

program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN*.

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Chemical bond	⊕/	
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Small capitals	Sc	
Italic type	Ital	
Bold type	Bold	
Lower case letters	Lc	} Circle characters
Roman type	Rom	
Delete	?	Cross out unwanted material
Delete and close up	$\overline{?}$	Cross out unwanted material and surround with $\overline{\quad}$
Close up	$\cup$	$\cup$ around space to be closed up
Invert type	⊖	Circle inverted characters
Transpose	⊗	$\sqcap$ between letters or words
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