Trans,trans,trans-Diethanoldiquinaldinatoiron(II)

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Kunitoyo Osawa *et al.*

Synopsis

Queries and comments
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Subject index
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trans,trans,trans-Diethanoldiquinaldinatoiron(II)

Inorganic formula index
Note that, for coordination complexes, the ligands are listed in alphabetic order. This means that the indexing term may differ from the IUPAC formula used elsewhere in the paper.

$[Fe(C_{10}H_6NO_2)_2(C_2H_6O)_2]$

Organic formula index
All residues containing organic carbon are included in this index.

$C_{24}H_{24}FeN_2O_6$

Author index
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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.004 Å
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.

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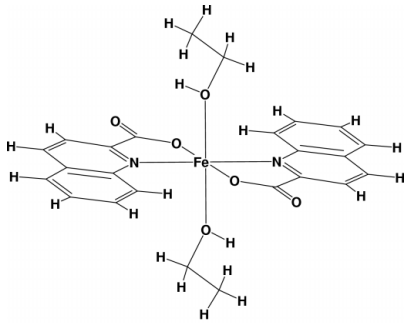
Online 9 May 2003

The title complex, *trans,trans,trans*-$[Fe^{II}(C_{10}H_6NO_2)_2(C_2H_6O)_2]$, is centrosymmetric and the quinaldinate ligands form five-membered chelate rings. The geometry of the complex is distorted octahedral, with a *trans*-FeN_2O_4 chromophore. The hydroxy H atom forms an intermolecular hydrogen bond with the carbonyl O atom of the quinaldinate ligand.

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 quialdinate complexes in spite of numerous studies of related picolinato complexes. Only the Cu^{2+} (Haendler, 1986), Rh^{+} (Lamprecht *et al.*, 1986) and Ga^{3+} (Li *et al.*, 1996) complexes have been structurally characterized. Therefore, structural information of another transition metal complex is desired.



(I)

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 $O1^{i}$ 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 $O2^{ii}$ 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 N_2 atmosphere using Schlenk techniques. To a solution of $Fe(BF_4)_2{\cdot}6H_2O$ (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 $\nu(CO_2)$ band at 1628 cm^{-1}. The electronic spectrum in DMF exhibits an absorption maximum at 527 nm (ε = 795).

Crystal data

$[Fe(C_{10}H_6NO_2)_2(C_2H_6O)_2]$	D_x = 1.475 Mg m^{-3}
M_r = 492.30	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4502 reflections
a = 5.816 (2) Å	θ = 3.1–27.5°
b = 9.557 (3) Å	μ = 0.72 mm^{-1}
c = 19.948 (5) Å	T = 123 K
β = 91.461 (7)°	Prism, red-violet
V = 1108.4 (6) $Å^3$	0.20 × 0.05 × 0.05 mm
Z = 2	

Data collection

Rigaku/MSC Mercury CCD diffractometer	2511 independent reflections
	2006 reflections with $F^2 > 2\sigma(F^2)$
ω scans	R_{int} = 0.039
Absorption correction: multi-scan (Jacobson, 1998)	θ_{max} = 27.5°
	$h = -7 \rightarrow 7$
T_{min} = 0.783, T_{max} = 0.964	$k = -12 \rightarrow 12$
8886 measured reflections	$l = -25 \rightarrow 25$

Refinement

Refinement on F	H-atom parameters constrained
R = 0.039	$w = 1/[\sigma^2(F_o) + 0.00168\|F_o\|^2]$
wR = 0.069	$(\Delta/\sigma)_{max} < 0.001$
S = 1.07	$\Delta\rho_{max}$ = 0.33 e $Å^{-3}$
2506 reflections	$\Delta\rho_{min}$ = −0.27 e $Å^{-3}$
151 parameters	

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

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

C-H=0.96, O-H=0.96

H atoms were included at calculated positions (~~C—H = 0.96 O— = ???~~ Å), with isotropic displacement parameters of $1.2U_{eq}$(parent atom).

Data collection: *CrystalClear* (Molecular Structure Corporation/Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation/Rigaku, 2000); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN*.

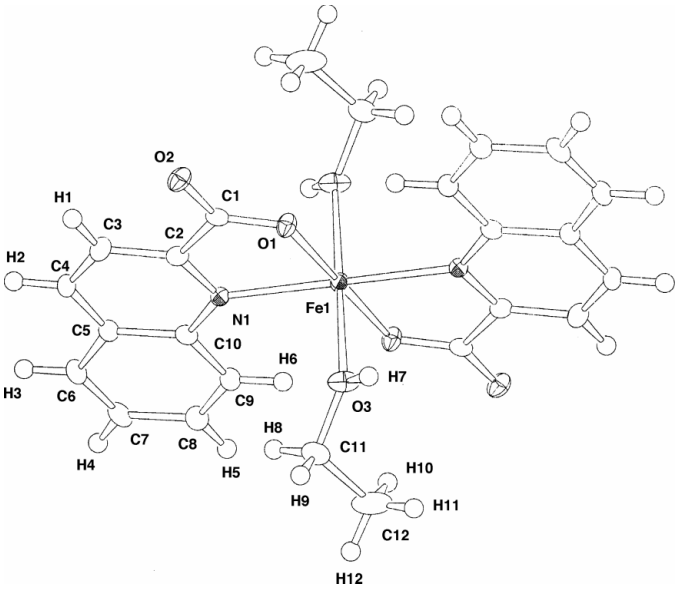


Figure 1
ORTEP-3 drawing (Farrugia, 1997) of (I), half of which defines the asymmetric unit, showing the atomic numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 50% probability level.

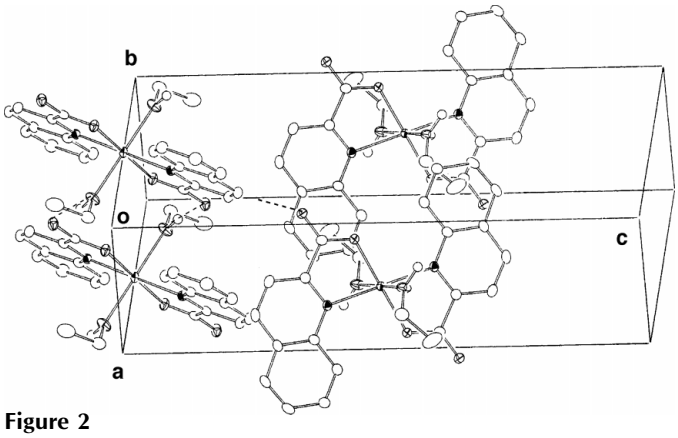


Figure 2
Packing diagram of the title complex. Dotted lines show hydrogen bonding, which forms molecular chains parallel to the *a* axis.

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Hyphen	⊢	
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Chemical bond	bond	
Superior (e.g. superscript 2 or apostrophe	x^2 or x'	
Inferior (e.g. subscript 2)	A_2	
Change to:		
Capitals	caps	≡ under characters
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	⌒	Cross out unwanted material and surround with ⌒
Close up	⌒	⌒ around space to be closed up
Invert type	◎	Circle inverted characters
Transpose	tr	⌐¬ between letters or words
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