

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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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(C₁₀H₆NO₂)₂(C₂H₆O)₂]

Organic formula index

All residues containing organic carbon are included in this index.

C₂₄H₂₄FeN₂O₆

Author index

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trans,trans,trans-Diethanoldiquinaldinatoiron(II)

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Key indicators

Single-crystal X-ray study
 $T = 123$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.004$ 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>.

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.

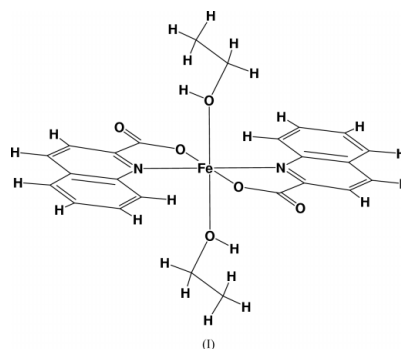
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Comment

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 picolinato 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 quinaldinate 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 ν(CO₂) band at 1628 cm⁻¹. The electronic spectrum in DMF exhibits an absorption maximum at 527 nm (ε = 795).

Crystal data

[Fe(C₁₀H₆NO₂)₂(C₂H₆O)₂]
M_r = 492.30
 Monoclinic, *P*₂₁/*n*
a = 5.816 (2) Å
b = 9.557 (3) Å
c = 19.948 (5) Å
 β = 91.461 (7)°
V = 1108.4 (6) Å³
Z = 2

D_x = 1.475 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 4502 reflections
 θ = 3.1–27.5°
 μ = 0.72 mm⁻¹
T = 123 K
 Prism, red-violet
 0.20 × 0.05 × 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 > 2σ(*F*²)
R_{int} = 0.039
 θ_{max} = 27.5°
h = -7 → 7
k = -12 → 12
l = -25 → 25

Refinement

Refinement on *F*
R = 0.039
wR = 0.069
S = 1.07
 2506 reflections
 151 parameters

H-atom parameters constrained
w = 1/[σ²(*F_o*) + 0.00168|*F_o*|²]
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.33 e Å⁻³
 Δρ_{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, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
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) ½ - *x*, *y* - ½, ½ - *z*.

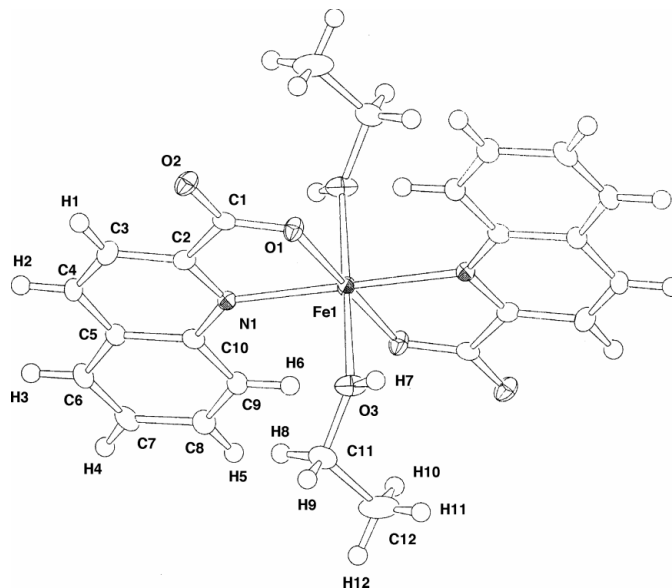


Figure 1

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.

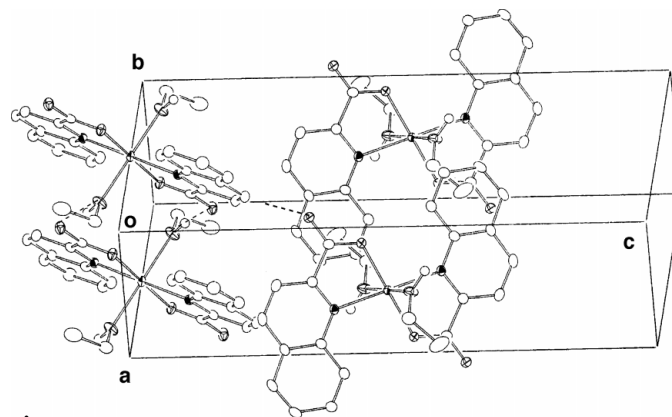


Figure 2

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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 Molecular Structure Corporation/Rigaku (2001). *CrystalClear*. Version 1.3. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA, and Rigaku Corporation, 3-9-12 Akishima, Tokyo, Japan.

C-H=0.96, O-H=0.96

H atoms were included at calculated positions (C—H = 0.96 Å, with isotropic displacement parameters of 1.2*U*_{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: *SHELXS86* (Sheldrick, 1985);

Sheldrick, G. M. (1985). *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger and R. Goddard, pp. 175–189. Oxford University Press.

Zhou, P., O'Hagan, D., Mocek, U., Zeng, Z., Yuen, L., Frenzel, T., Unkefer, C. J., Beale, J. M. & Floss, H. G. (1989). *J. Am. Chem. Soc.* **111**, 7274–7276.

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Rule	⊗/	
Chemical bond	— /	
Superior (e.g. superscript 2 or apostrophe)	$\vee/$ or $\vee/$	
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Capitals	<u>Cap</u>	} under characters
Small capitals	<u>Sc</u>	
Italic type	<u>Ital</u>	
Bold type	<u>Bold</u>	
Lower case letters	<u>Lc</u>	} Circle characters
Roman type	<u>Rom</u>	
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Delete and close up	$\overline{\text{?}}$	Cross out unwanted material and surround with $\overline{\quad}$
Close up	⌒	⌒ around space to be closed up
Invert type	⊖	Circle inverted characters
Transpose	⊗	⌞ between letters or words
Faulty setting (e.g. broken type)	X	Circle defective characters
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