# Trans,trans,trans-Diethanoldiquinaldinatoiron(II)

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## Kunitoyo Osawa *et al.*

**Synopsis**

**Queries and comments**
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**Subject index**
The following terms will be used to index your paper. Authors wishing to recommend additional index entries should give these below.

*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**
Authors' names will normally be arranged alphabetically under their family name and this is commonly their last name. Prefixes (van, de *etc.*) will only be taken into account in the alphabetization if they begin with a capital letter. Authors wishing their names to be alphabetized differently should indicate this below.

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# *trans,trans,trans*-Diethanoldiquinaldinatoiron(II)

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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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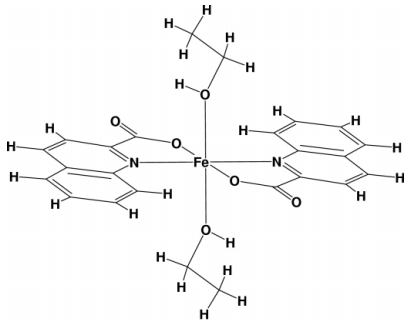
The title complex, *trans,trans,trans*-$[Fe^{II}(C_{10}H_6NO_2)_2(C_2H_6O)_2]$, is centrosymmetric and the quinaldinate ligands form five-membered chelate rings. The geometry of the complex is distorted octahedral, with a *trans*-$FeN_2O_4$ chromophore. The hydroxy H atom forms an intermolecular hydrogen 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 ($\varepsilon$ = 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 | $\theta$ = 3.1–27.5° |
| $b$ = 9.557 (3) Å | $\mu$ = 0.72 $mm^{-1}$ |
| $c$ = 19.948 (5) Å | $T$ = 123 K |
| $\beta$ = 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 $F^2 > 2\sigma(F^2)$ |
| $\omega$ scans | $R_{int}$ = 0.039 |
| Absorption correction: multi-scan (Jacobson, 1998) | $\theta_{max}$ = 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*.

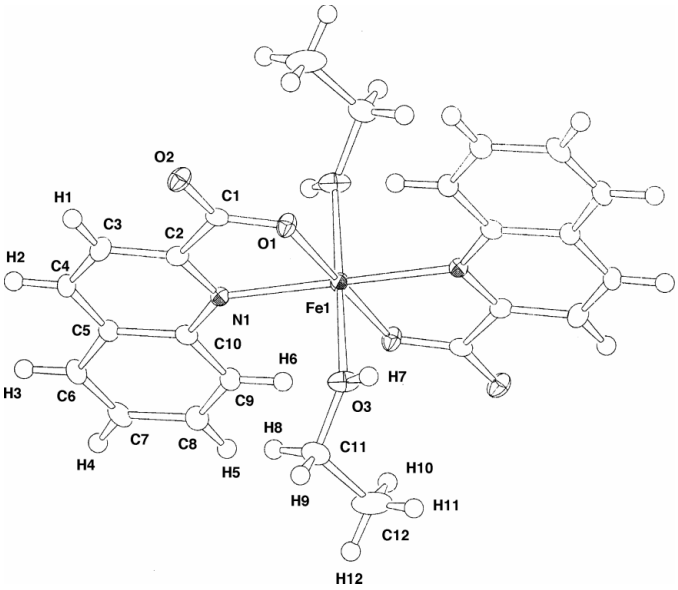



**Figure 1**
*ORTEP*-3 drawing (Farrugia, 1997) of (I), half of which defines the asymmetric unit, showing the atomic numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 50% probability level.

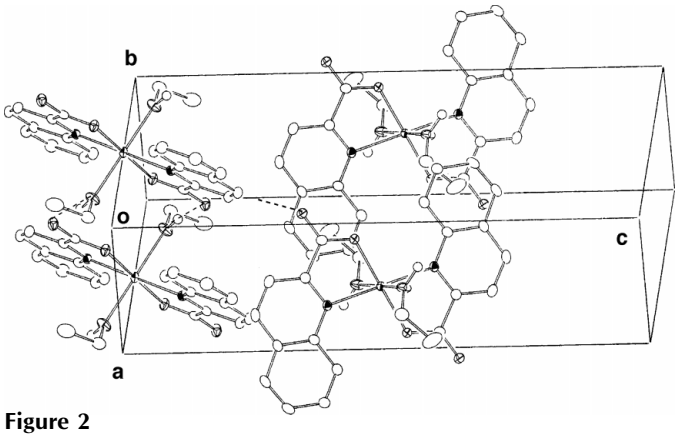



**Figure 2**
Packing diagram of the title complex. Dotted lines show hydrogen bonding, which forms molecular chains parallel to the *a* axis.

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| Italic type | ital | — |
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| Lower case letters | lc | Circle characters |
| Roman type | rom | |
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