<table>
<thead>
<tr>
<th>著者</th>
<th>古謁 イヒト・藤波 修平・鈴木 正樹</th>
</tr>
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<tbody>
<tr>
<td>著者別表示</td>
<td>古舘 英樹 □ 藤波 修平 □ 鈴木 正樹</td>
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<tr>
<td>出版物名</td>
<td>水素化物を用いた金属錯体の合成と性質</td>
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doi: https://doi.org/10.1107/s1600536803009607
trans,trans,trans-Diethanoldiquinaldinatoiron(II)

Kunitoyo Osawa et al.

Synopsis

Queries and comments
Please supply or correct as appropriate all bold underlined text.

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_{14}NO_{2})_{2}(C_{2}H_{6}O)_{2}]

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

C_{24}H_{24}FeN_{2}O_{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.

Osawa, K.
Furutachi, H.
Fujinami, S.
Suzuki, M.
trans,trans,trans-Diethanoldiquinaldinatoiron(II)

Kunitoyo Osawa, Hideki Furutachi, Shuhei Fujinami* and Masastatu Suzuki

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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.

The title complex, trans,trans,trans-[FeII(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.

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

Crystal data

\[ [\text{Fe}(_{2}C_{10}H_{6}NO_{2})(C_{2}H_{6}O)_{2}] \]

\[ M_r = 492.30 \]

Monoclinic, \( P2_1/n \)

\( a = 5.816 \) Å

\( b = 9.557 \) Å

\( c = 19.948 \) Å

\( \beta = 91.461 \)°

\( V = 1108.4 \) Å³

\( Z = 2 \)

Data collection

Rigaku/MSC Mercury CCD

diffactometer

\( \omega \) scans

2511 measured reflections

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

\( \theta_{max} = 25.5^\circ \)

\( h = -7 \rightarrow 7 \)

\( k = -12 \rightarrow 12 \)

\( l = -25 \rightarrow 25 \)

Refinement

H-atom parameters constrained

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

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

\( D_{calc} = 1.475 \) Mg m⁻³

\( D_{x} = 0.72 \) mm

\( T = 293 \) K

\( \mu = 0.72 \) mm⁻¹

\( R_{int} = 0.039 \)

\( S = 1.07 \)

2506 reflections

151 parameters

Table 1

<table>
<thead>
<tr>
<th></th>
<th>Fe–O1</th>
<th>Fe–O3</th>
<th>O1–Fe–O3</th>
<th>O1–Fe–N1</th>
</tr>
</thead>
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<td>Distance</td>
<td>2.032 (2)</td>
<td>2.154 (2)</td>
<td>92.01 (8)</td>
<td>77.30 (8)</td>
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</table>

Table 2

<table>
<thead>
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<th>Hydrogen-bonding geometry (Å, °)</th>
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<tr>
<td>D–H···A</td>
</tr>
<tr>
<td>C9–H9b···O1i</td>
</tr>
<tr>
<td>O3–H7b···O1ii</td>
</tr>
<tr>
<td>O3–H7···O2ii</td>
</tr>
<tr>
<td>C4–H4a···O2iii</td>
</tr>
<tr>
<td>C6–H6···O2iii</td>
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</tbody>
</table>

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.

References


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metal-organic papers
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<tr>
<th>Alteration</th>
<th>Marginal mark</th>
<th>Mark in text</th>
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<td>Insert or substitute:</td>
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<td>Space</td>
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- Change to
- Capitals
- Small capitals
- Italic type
- Bold type
- Lower case letters
- Roman type

- Delete
- Delete and close up
- Close up
- Invert type
- Transpose
- Faulty setting
- (e.g. broken type)
- Leave as printed
- New paragraph
- No new paragraph or line

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