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# *trans*-Bis(isoquinoline-3-carboxylato-$\kappa^2N,O$)bis(methanol-$\kappa O$)iron(II)

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**Key indicators**

Single-crystal X-ray study
$T$ = 296 K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.039
$wR$ factor = 0.141
Data-to-parameter ratio = 16.1

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

The title complex, $[Fe(C_{10}H_6NO_2)_2(CH_4O)_2]$, contains an $Fe^{II}$ ion at a center of inversion. The $Fe^{II}$ ion has a distorted octahedral coordination geometry, and is coordinated by two bidentate isoquinoline-3-carboxylate ligands through N and O atoms, and by two methanol O atoms. The two isoquinoline-3-carboxylate ligands lie in a *trans* position with respect to one another in the equatorial plane, and the two methanol ligands occupy the axial positions. The complex molecules are linked together by hydrogen bonds between the methanol ligands and the carboxylate groups.

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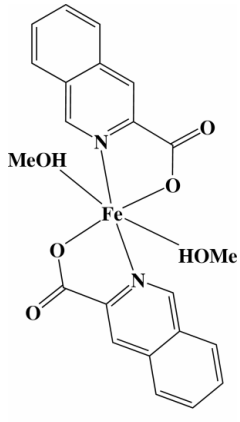
## Comment

Isoquinoline-3-carboxylic acid is known to be a potent non-peptidyl inhibitor of the insulin-like growth factor binding proteins (Zhu *et al.*, 2003). Its metal complexes act as catalysts in alkane oxidations, by so-called Gif-systems in solution (Shul'pin, 2002). These oxidation reactions occur in the presence of $H_2O_2$ and complexes of transition metals (mainly iron). In this study, the crystal structure of an iron(II) complex of isoquinoline-3-carboxylate, (I), has been determined in order to clarify the structural features of the complex.




(I)

The structure of (I) is shown in Fig. 1. The central $Fe^{II}$ ion has a distorted octahedral coordination geometry. The two bidentate ligands lie *trans* to one another and are coordinated to the central $Fe^{II}$ ion by N and O atoms, forming a five-membered ring in the equatorial plane. This type of coordination mode is commonly observed in analogous compounds that contain ring N and carboxylate O atoms, *e.g.* quinoline-2-carboxylic acid (Okabe & Muranishi, 2003*a*), pyridine-2-carboxylic acid (Okabe, Isomoto *et al.*, 2002) and pyridine-2,6-carboxylic acid (Okabe, Kyoyama *et al.*, 2002). In the centro-

symmetric complex (I), two O atoms of the methanol ligands complete the octahedron at the axial positions. The coordination Fe—N bond is about 0.08 Å shorter than the corresponding bond in the analogous iron(II) complex of quinoline-2-carboxylate (Okabe & Makino, 1998).

In (I), the N1—C1, N1—C9 and C1—C2 bond lengths are shorter than the others in the same ring (C2—C3, C3—C8 and C8—C9, see Table 1). Therefore, the $\pi$ electrons are delocalized over the first three bonds, which have double-bond character. In the quinoline-2-carboxylate complex, only bonds on either side of the N atom have double-bond character [N1—C1 = 1.321 (2) Å and N1—C9 = 1.375 (2) Å; Okabe & Makino, 1998]. A similar structural feature is observed in other metal complexes of isoquinoline-2-carboxylate, such as those of cobalt(II) [N1—C1 = 1.326 (3) Å and N1—C9 = 1.373 (3) Å; Okabe & Makino, 1999], nickel(II) [N1—C1 = 1.323 (3) Å and N1—C9 = 1.375 (2) Å; Odoko *et al.*, 2001] and zinc(II) [N1—C1 = 1.312 (7) Å and N1—C9 1.378 (6) Å; Okabe & Muranishi, 2003*b*]. The difference in the double-bond character around the N atom of the isoquinoline ring may be one of the reasons for the different Fe—N bond lengths in the Fe complex of isoquinoline-3-carboxylate and in that of quinoline-2-carboxylate.

The crystal structure of complex (I) is stabilized by an intermolecular hydrogen-bonding network between methanol ligands and the neighboring carboxylate groups, as listed in Table 2. A stacking interaction is also observed between the ligands, with a mean distance of 3.367 (4) Å.

## Experimental

Crystals of (I) were obtained by slow evaporation of a methanol solution of a mixture of isoquinoline-3-carboxylic acid and $FeCl_2{\cdot}4H_2O$ (molar ratio 4:1).

*Crystal data*

| | |
|---|---|
| $[Fe(C_{10}H_6NO_2)_2(CH_4O)_2]$ | $D_x$ = 1.538 Mg m$^{-3}$ |
| $M_r$ = 464.25 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/c$ | Cell parameters from 25 reflections |
| $a$ = 10.798 (2) Å | $\theta$ = 13.2–14.4° |
| $b$ = 6.281 (1) Å | $\mu$ = 0.80 mm$^{-1}$ |
| $c$ = 15.045 (2) Å | $T$ = 296.2 K |
| $\beta$ = 100.82 (1)° | Plate, dark orange |
| $V$ = 1002.2 (3) Å$^3$ | 0.40 × 0.10 × 0.10 mm |
| $Z$ = 2 | |

*Data collection*

| | |
|---|---|
| Rigaku AFC-5*R* diffractometer | $R_{int}$ = 0.024 |
| $\omega$–$2\theta$ scans | $\theta_{max}$ = 27.5° |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $h = 0 \rightarrow 14$ |
| $T_{min}$ = 0.909, $T_{max}$ = 0.923 | $k = 0 \rightarrow 8$ |
| 2641 measured reflections | $l = -19 \rightarrow 19$ |
| 2301 independent reflections | 3 standard reflections every 150 reflections |
| 1433 reflections with $I > 2\sigma(I)$ | intensity decay: 0.5% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters not refined |
| $R[F^2 > 2\sigma(F^2)]$ = 0.039 | $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.141 | $(\Delta/\sigma)_{max} < 0.001$ |
| $S$ = 0.86 | $\Delta\rho_{max}$ = 0.37 e Å$^{-3}$ |
| 2301 reflections | $\Delta\rho_{min}$ = −0.36 e Å$^{-3}$ |
| 143 parameters | |

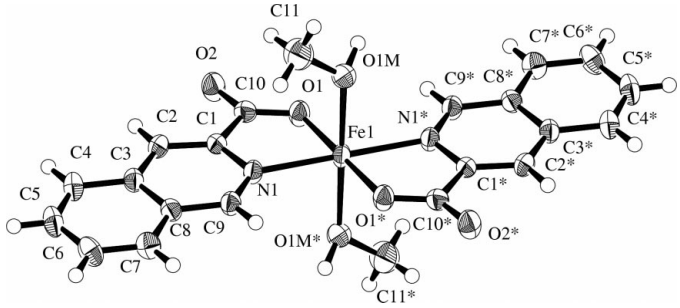



**Figure 1**
*ORTEP*II (Johnson, 1976) drawing of (I), showing the atomic numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Fe1—O1 | 2.050 (2) | C3—C4 | 1.420 (4) |
| Fe1—O1*M* | 2.196 (2) | C3—C8 | 1.415 (5) |
| Fe1—N1 | 2.167 (2) | C4—C5 | 1.356 (5) |
| N1—C1 | 1.372 (4) | C5—C6 | 1.415 (5) |
| N1—C9 | 1.315 (4) | C6—C7 | 1.362 (4) |
| C1—C2 | 1.366 (4) | C7—C8 | 1.405 (4) |
| C2—C3 | 1.414 (4) | C8—C9 | 1.416 (4) |
| | | | |
| O1—Fe1—O1*M* | 89.97 (9) | O1*M*—Fe1—N1 | 92.43 (9) |
| O1—Fe1—N1 | 78.86 (9) | | |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*M*—H1*M*···O2$^{i}$ | 0.97 | 1.66 | 2.617 (3) | 170 |

Symmetry code: (i) $-x, -1-y, -z$.

Initially, all H atoms were located in difference Fourier maps, and then all H atoms except that of the OH group of the methanol molecule were placed in idealized positions [C—H = 0.96 (methyl), 0.93 Å (other H atoms); $U_{iso}$(H) = 1.2 times $U_{eq}$ (methyl) and 1.5 times $U_{eq}$ (other H atoms)]. The hydroxy H atom was fixed at the position determined from the Fourier map and was not refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku, 2000); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999) and *DIRDIF*94 (Beurskens *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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