Travníček, Z., Pastorek, R., Šindelář, Z., Kameníček, J. & Marek, J. (1996). *Polyhedron*, **15**, 2975–2981.
Valigura, D., Ondrejovič, G. & Anderson, O. P. (1998). In preparation.

trans-Diaquabis(2-quinolinecarboxylato-N,O)iron(II)–Ethanol–Water (1/2/2)

NOBUO OKABE AND TOMOMI MAKINO

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan. E-mail: okabe@phar.kindai.ac.jp

Abstract

The title compound, $[Fe(C_{10}H_6NO_2)_2(H_2O)_2].2C_2H_6O.-2H_2O$, contains an Fe^{II} ion in a six-coordinate environment. The Fe^{II} ion lies on an inversion centre and displays distorted octahedral geometry with two 2-quinolinecarboxylate and two water ligands in a necessarily *trans* configuration. Complex molecules and uncoordinated water and ethanol molecules are linked together by a hydrogen-bonding network.

Comment

2-Quinolinecarboxylic acid (quinaldinic acid), (I), is one of the final products of tryptophan metabolism (Martin *et al.*, 1983). It is known to be a chelator of transition metal ions (Martell & Smith, 1974). It is converted to kynurenic acid (4-hydroxyquinoline-2-carboxylic acid) by quinaldinic acid 4-monooxygenase, the activity of which is enhanced by Fe^{II} ions (Bubeck *et al.*, 1996). Accordingly, we have undertaken the structure analysis of the chelate compounds of 2-quinolinecarboxylic acid and biologically important Fe^{II} ions in order to clarify the binding scheme of this metabolite to the metal ions. We report herein the structure of the title compound, *trans*-[diaquabis(2-quinolinecarboxylato)iron(II)] diethanol solvate dihydrate, (II). In previous papers, the structures of the Cu^{II} (Haendler, 1986) and Mn^{II} complexes (Haendler, 1996; Okabe & Koizumi, 1997) have been reported.
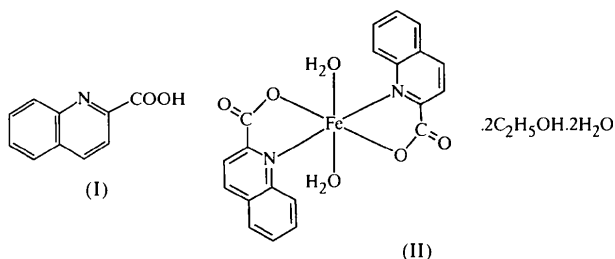
The molecular structure of the title compound with the atomic labelling is shown in Fig. 1. The title compound contains a six-coordinate Fe^{II} ion with distorted octahedral geometry formed by two 2-quinolinecarboxylate and two water ligands. The six-coordinate geometry found in this study is similar to that found for the Mn^{II} complex of (I) (Haendler, 1996; Okabe & Koizumi, 1997), but different from the pentacoordinate geometry of the Cu^{II} complex (Haendler, 1986). The Fe^{II} ion lies on an inversion centre and is coordinated by two *trans* quinoline N atoms [Fe(1)—N(1) 2.270(1) Å] and by two *trans* carboxylate O atoms [Fe(1)—O(2) 2.087(1) Å] in the equatorial plane, and by two *trans* O atoms of the water molecules in the axial positions [Fe(1)—O(3) 2.131(1) Å]. The carboxy group of the quinoline moiety is ionized and almost coplanar with the quinoline ring plane [O(2)—C(1)—C(2)—N(1) 3.5(2)°]. Molecules are connected by hydrogen bonds between the chelated molecules and the solvated water and ethanol molecules (Table 2).
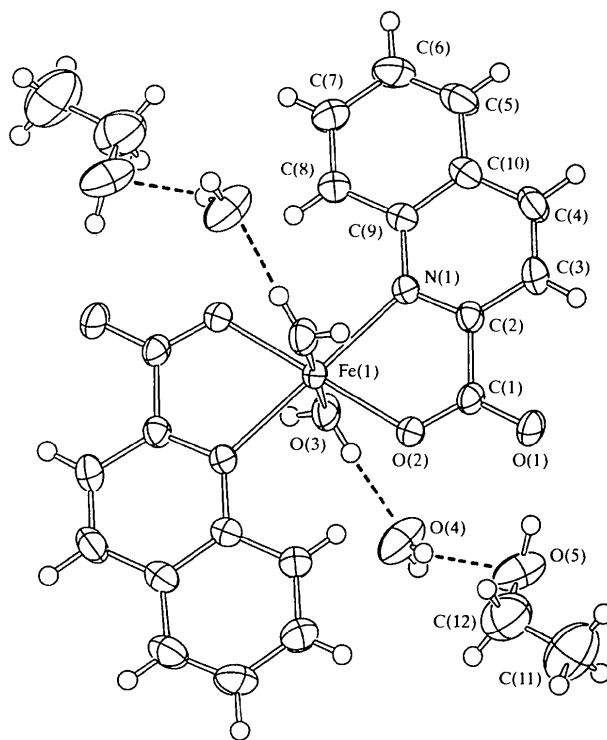

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Ellipsoids for non-H atoms correspond to 50% probability. Dashed lines indicate hydrogen bonds.

Experimental

The dark-red plate crystal used for analysis was obtained by the slow evaporation of a mixture of 2-quinolinecarboxylic acid and iron(II) sulfate in the molar ratio 4:1 in ethanol solution (*ca* 90%) at room temperature.

(I) (II) .2C₂H₅OH.2H₂O

Crystal data

[Fe(C$_{10}$H$_6$NO$_2$)$_2$(H$_2$O)$_2$].-
2C$_2$H$_6$O.2H$_2$O
M_r = 564.371
Triclinic
$P\bar{1}$
a = 9.057 (2) Å
b = 11.092 (3) Å
c = 7.299 (2) Å
α = 106.31 (2)°
β = 108.15 (2)°
γ = 77.88 (2)°
V = 662.7 (3) Å^3
Z = 1
D_x = 1.414 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25
reflections
θ = 21.65–23.85°
μ = 0.626 mm^{-1}
T = 296 K
Prism
0.5 × 0.5 × 0.1 mm
Dark red

Data collection

Rigaku AFC-5R diffractom-
eter
ω–2θ scans
Absorption correction:
ψ scan (North et al.,
1968)
T_{min} = 0.80, T_{max} = 0.94
3241 measured reflections
3049 independent reflections

2850 reflections with
$I > \sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°
$h = 0 \to 11$
$k = -13 \to 14$
$l = -9 \to 8$
3 standard reflections
every 150 reflections
intensity decay: −0.30%

Refinement

Refinement on F^2
$R(F)$ = 0.068
$wR(F^2)$ = 0.089
S = 1.68
2850 reflections
169 parameters
H atoms not refined
$w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: none
Scattering factors from Inter-
national Tables for X-ray
Crystallography (Vol. IV)

Table 1. Selected geometric parameters (Å, °)

Fe(1)—O(2)	2.087 (1)	N(1)—C(2)	1.321 (2)
Fe(1)—O(3)	2.131 (1)	N(1)—C(9)	1.375 (2)
Fe(1)—N(1)	2.270 (1)	C(1)—C(2)	1.517 (2)
O(1)—C(1)	1.245 (1)	C(2)—C(3)	1.407 (2)
O(2)—C(1)	1.256 (2)	C(11)—C(12)	1.437 (5)
O(5)—C(12)	1.412 (4)		
O(2)—Fe(1)—O(3)	88.79 (5)	O(1)—C(1)—O(2)	124.5 (2)
O(2)—Fe(1)—N(1)	75.55 (5)	O(1)—C(1)—C(2)	118.2 (2)
O(3)—Fe(1)—N(1)	90.57 (5)	O(2)—C(1)—C(2)	117.3 (1)
Fe(1)—O(2)—C(1)	119.5 (1)	N(1)—C(2)—C(1)	115.9 (1)

Fe(1)—N(1)—C(2)	111.5 (1)	N(1)—C(2)—C(3)	123.7 (2)
Fe(1)—N(1)—C(9)	130.2 (1)	C(1)—C(2)—C(3)	120.3 (2)
C(2)—N(1)—C(9)	118.2 (1)	O(5)—C(12)—C(11)	113.4 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O(5)—H(18)···O(1)	0.89	1.83	2.705 (2)	166
O(3)—H(9)···O(1^i)	0.75	1.98	2.727 (2)	178
O(3)—H(10)···O(4)	0.87	1.83	2.692 (2)	174
O(4)—H(11)···O(5^{ii})	0.86	1.91	2.766 (2)	175
O(4)—H(12)···O(5)	0.74	2.21	2.915 (2)	160

Symmetry codes: (i) $x, y, z - 1$; (ii) $1 - x, -y, 1 - z$.

H atoms were located from difference Fourier maps and included in the refinement calculations at fixed positions.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976).

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors*. Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Bubeck, B., Tshisuaka, B., Fetzner, S. & Lingens, F. (1996). *Biochim. Biophys. Acta*, **1293**, 39–44.

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Haendler, H. M. (1986). *Acta Cryst.* C42, 147–149.

Haendler, H. M. (1996). *Acta Cryst.* C52, 801–803.

Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Martell, A. E. & Smith, R. M. (1974). *Critical Stability Constants*, Vol. 1, pp. 78, 372; Vol. 2, p. 219. New York: Plenum Press.

Martin, D. W., Mayes, P. A. & Rodwell, V. W. (1983). *Harper's Review of Biochemistry*, pp. 464–479. Tokyo: Maruzen Asia.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A24, 351–359.

Okabe, N. & Koizumi, M. (1997). *Acta Cryst.* C53, 852–854.