

$\omega$ - $2\theta$  scans  
Absorption correction:  
empirical via  $\psi$  scans  
(*XPREP* in *SHELXTL-Plus*; Sheldrick, 1990)  
 $T_{\min} = 0.640$ ,  $T_{\max} = 0.710$   
6000 measured reflections  
4572 independent reflections

$R_{\text{int}} = 0.0298$   
 $\theta_{\text{max}} = 25.01^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -18 \rightarrow 0$   
 $l = -12 \rightarrow 3$   
3 standard reflections  
frequency: 60 min  
intensity decay: 12%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.111$   
 $S = 1.034$   
4571 reflections  
253 parameters  
H atoms riding with C—H =  
0.96 Å

$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 3.8139P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = -0.001$   
 $\Delta\rho_{\text{max}} = 0.440 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.664 \text{ e } \text{Å}^{-3}$   
Extinction correction: none  
Scattering factors from  
*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Nb1—O21	2.289 (4)	Nb1'—O21'	2.303 (4)
Nb1—S1'	2.324 (2)	Nb1'—S1'	2.330 (2)
Nb1—S1	2.336 (2)	Nb1'—S1''	2.331 (2)
Nb1—Cl1	2.370 (2)	Nb1'—O1'	2.335 (4)
Nb1—Cl2	2.3747 (15)	Nb1'—Cl2'	2.377 (2)
Nb1—O11	2.385 (4)	Nb1'—Cl1'	2.3879 (15)
Nb1—Nb1'	2.8685 (10)	Nb1'—Nb1''	2.8647 (10)
S1'—Nb1—S1	104.02 (5)	S1'—Nb1'—S1''	104.14 (5)
Cl1—Nb1—Cl2	158.20 (6)	O21'—Nb1'—O1'	78.88 (15)
O21—Nb1—O11	82.55 (14)	Cl2'—Nb1'—Cl1'	160.19 (6)
Nb1'—S1—Nb1	75.97 (5)		

Symmetry codes: (i)  $1-x, 1-y, -z$ ; (ii)  $2-x, 1-y, -z$ .

The structure was solved using direct methods with the program *SHELXTL-Plus* (Sheldrick, 1990). According to systematic absences, the space group  $P2_1/c$  was selected. All non-H atoms were located directly from the *E* map and refined with anisotropic displacement parameters. All H atoms were treated as riding atoms with C—H distances of 0.96 Å and with individual group isotropic displacement parameters.

Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1039). Services for accessing these data are described at the back of the journal.

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## Tris(8-hydroxyquinolinato)iron(III) Ethanol Solvate

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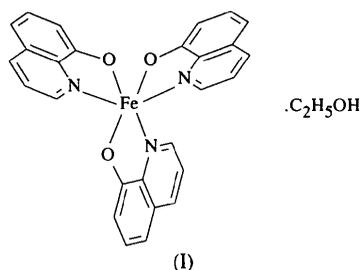
### Abstract

The title compound, tris(8-quinolinolato-*N,O*)iron(III) ethanol solvate,  $[\text{Fe}(\text{C}_9\text{H}_6\text{NO})_3] \cdot \text{C}_2\text{H}_5\text{OH}$ , has a slightly distorted octahedral coordination about the central Fe atom which bonds to the N and O atoms of each of the three 8-hydroxyquinoline ligands. The ethanol solvate molecule forms a hydrogen bond with the O atom which is least strongly bound to Fe. The Fe—O and Fe—N bond distances are in the ranges 1.936 (5)–1.996 (5) and 2.125 (6)–2.172 (5) Å, respectively.

### Comment

The present study is part of systematic crystallographic research of internal complexes of 8-hydroxyquinoline and its derivatives with transition metals (Bankovsky, Belsky, Pech & Ashaks, 1993; Kuzmina *et al.*, 1995).

The monomeric iron complex, (I), crystallizes together with an ethanol molecule which is not included in the coordination sphere of iron. The O and N atoms of the bidentate ligands form three five-membered chelate rings. The Fe atom thereby attains a distorted octahedral coordination. The intra-ligand bond angles at the Fe atom are in the range 78.9–79.5°, whereas all inter-ligand angles at Fe are greater than 87° (see Table 1). Similar results have been obtained in the case of tris(8-quinolinolato) complexes of chromium(III) (Foltling, Cox, Moore & Merritt, 1968) and manganese(III) (Hems & Mackay, 1975; Xiong, You, Wu & Huang, 1995).



The dihedral angles between the coordination planes O1—Fe—N1 and O2—Fe—N2, O1—Fe—N1 and O3—Fe—N3, and O2—Fe—N2 and O3—Fe—N3 are 81.1 (2), 81.0 (2) and 82.2 (2)°, respectively.

Each quinoline nucleus is approximately planar and the dihedral angles between their least-squares planes are 77.4 (2), 83.2 (2) and 83.4 (2)°. The Fe atom is close to one quinoline ring plane but displaced by 0.143 (2) and 0.061 (2) Å from the others. Each quinoline O—C bond lies close to the plane of its phenyl ring but the Fe—N bonds form angles with the pyridine ring planes of 3.7 (3), 0.8 (3) and 2.3 (2)° for Fe—N1, N2, N3, respectively. The Fe—O—C angles are 115.7 (4), 118.2 (4) and 117.8 (4)°.

The solvate ethanol molecule forms a hydrogen bond with O1 resulting in a lengthening of the Fe—O1 bond [O1E—H1E...O1 geometry: H1E...O1 1.89, O1E—H1E 0.82 Å and O1E—H1E...O1 166.1°].

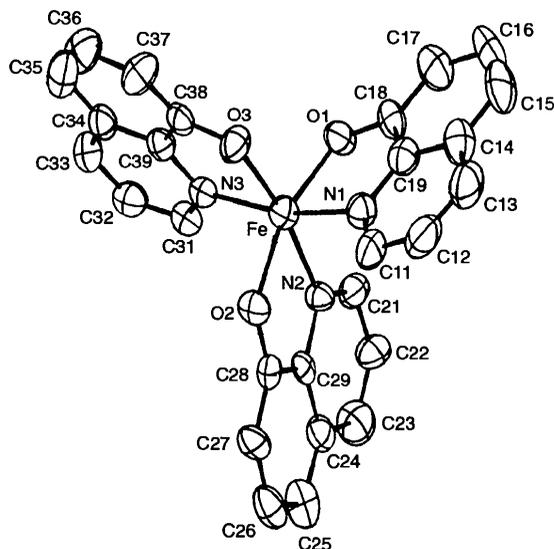


Fig. 1. View of the [Fe(C<sub>9</sub>H<sub>6</sub>NO)<sub>3</sub>] molecule showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level for the non-H atoms. H atoms have been omitted.

## Experimental

The title compound was prepared from Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and 8-hydroxyquinoline in ethanol solution. Crystals suitable for X-ray analysis were obtained by recrystallization from CHCl<sub>3</sub>/C<sub>2</sub>H<sub>5</sub>OH.

## Crystal data

[Fe(C<sub>9</sub>H<sub>6</sub>NO)<sub>3</sub>].C<sub>2</sub>H<sub>6</sub>O

*M<sub>r</sub>* = 534.3

Monoclinic

*P*2<sub>1</sub>/*c*

*a* = 11.189 (2) Å

*b* = 13.416 (3) Å

*c* = 19.409 (4) Å

β = 120.73 (2)°

*V* = 2504.4 (9) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.407 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

## Data collection

Syntex *P*2<sub>1</sub> diffractometer

θ/2θ scans

Absorption correction: none

2418 measured reflections

2418 independent reflections

1938 reflections with

*I* > 2σ(*I*)

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 20 reflections

θ = 14.0–17.5°

μ = 0.643 mm<sup>-1</sup>

*T* = 293 (2) K

Needle

0.55 × 0.15 × 0.10 mm

Dark green

## Refinement

Refinement on *F*<sup>2</sup>

*R* = 0.062

*wR*(*F*<sup>2</sup>) = 0.156

*S* = 0.944

2417 reflections

336 parameters

H atoms not refined

*w* = 1/[σ<sup>2</sup>(*F*<sub>o</sub><sup>2</sup>) + (0.1*P*)<sup>2</sup>]

where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.006

Δρ<sub>max</sub> = 0.28 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.30 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Fe—O3	1.936 (5)	O2—C28	1.315 (8)
Fe—O2	1.956 (5)	N2—C21	1.313 (9)
Fe—O1	1.996 (5)	N2—C29	1.376 (8)
Fe—N1	2.125 (6)	C28—C29	1.426 (9)
Fe—N2	2.166 (6)	O3—C38	1.328 (8)
Fe—N3	2.172 (5)	N3—C31	1.310 (9)
O1—C18	1.328 (8)	N3—C39	1.374 (9)
N1—C11	1.332 (9)	C38—C39	1.421 (10)
N1—C19	1.347 (9)	O1E—C1E	1.409 (11)
C18—C19	1.420 (10)	C1E—C2E	1.418 (14)
O3—Fe—O2	97.3 (2)	N1—C19—C14	121.9 (7)
O3—Fe—O1	99.0 (2)	C18—C19—C14	121.7 (7)
O2—Fe—O1	160.9 (2)	C28—O2—Fe	118.2 (4)
O3—Fe—N1	91.9 (2)	C21—N2—C29	117.5 (6)
O2—Fe—N1	90.4 (2)	C21—N2—Fe	132.4 (5)
O1—Fe—N1	79.2 (2)	C29—N2—Fe	110.0 (4)
O3—Fe—N2	168.4 (2)	O2—C28—C27	124.9 (7)
O2—Fe—N2	78.9 (2)	O2—C28—C29	117.6 (6)
O1—Fe—N2	86.9 (2)	C27—C28—C29	117.5 (7)
N1—Fe—N2	99.1 (2)	N2—C29—C24	122.9 (6)
O3—Fe—N3	79.5 (2)	N2—C29—C28	115.3 (6)
O2—Fe—N3	97.7 (2)	C24—C29—C28	121.8 (7)
O1—Fe—N3	95.0 (2)	C38—O3—Fe	117.8 (4)
N1—Fe—N3	168.8 (2)	C31—N3—C39	117.9 (6)
N2—Fe—N3	90.1 (2)	C31—N3—Fe	132.6 (5)
C18—O1—Fe	115.7 (4)	C39—N3—Fe	109.4 (4)
C11—N1—C19	119.1 (7)	O3—C38—C37	123.9 (7)
C11—N1—Fe	129.6 (6)	O3—C38—C39	117.9 (7)
C19—N1—Fe	111.0 (5)	C37—C38—C39	118.2 (7)
O1—C18—C17	124.5 (7)	N3—C39—C34	122.7 (7)
O1—C18—C19	117.3 (6)	N3—C39—C38	115.5 (6)
C17—C18—C19	118.2 (7)	C34—C39—C38	121.9 (7)
N1—C19—C18	116.3 (7)	O1E—C1E—C2E	113.6 (11)

Data collection: Syntex  $P2_1$  software (Syntex, 1976). Cell refinement: Syntex  $P2_1$  software. Data reduction: Syntex  $P2_1$  software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976) implemented on an IBM-PC/AT.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1310). Services for accessing these data are described at the back of the journal.

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## (2,2'-Bipyridyl-*N,N'*)dibromopalladium(II)

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## Abstract

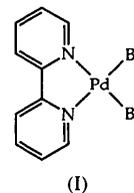
The title complex, [PdBr<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)], has square-planar geometry for palladium with Pd—N 2.047 (5) and Pd—Br 2.4102 (9) Å. The mean planes of the coordinated

pyridine groups form a dihedral angle of 1.7 (3)° with the coordination square plane; the parallel complexes stack with an interplanar spacing of 3.41 (1) Å and a Pd··Pd separation of 5.246 (1) Å to form a chain structure as reported for isomorphous PtI<sub>2</sub>(bipy) (where bipy is 2,2'-bipyridyl, C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>). The structure differs from those found for related chain structures in the 'red' form of PtCl<sub>2</sub>(bipy) and in PdCl<sub>2</sub>(bipy) which is isomorphous with the 'yellow' form of PtCl<sub>2</sub>(bipy).

## Comment

Molecules of stoichiometry  $MX_2(\text{bipy})$  ( $X = \text{halide}$  and  $\text{bipy} = 2,2'$ -bipyridyl) have been central to the development of the chemistry of Pd and Pt, *e.g.* PdCl<sub>2</sub>(bipy) was first reported in 1952 (Livingstone, 1952) and Pd(CH<sub>3</sub>)<sub>2</sub>(bipy) (Calvin & Coates, 1960) is the precursor to the first reported alkylpalladium(IV) complex (Byers, Canty, Skelton & White, 1986). In addition, the complexes exhibit interesting structural chemistry in which molecules are stacked in columnar arrays with different relationships between neighbouring molecules, *e.g.* PdCl<sub>2</sub>(bipy) is isomorphous with the 'yellow' form of PtCl<sub>2</sub>(bipy) and has a Pd··Pd distance of 4.587 (2) Å (Canty, Skelton, Traill & White, 1992), longer than observed in the closely related 'red' form of PtCl<sub>2</sub>(bipy), Pt··Pt 3.45 Å (Osborn & Rogers, 1974; Textor & Oswald, 1974), but shorter than a third structural type, PtI<sub>2</sub>(bipy) with Pt··Pt 5.291 (1) Å (Connick & Gray, 1994).

The title complex, (I), crystallized during the slow decomposition of an organopalladium(IV) complex; in view of current interest in the structural chemistry of  $MX_2(\text{bipy})$  complexes, it was examined by X-ray crystallography and found to be isomorphous with PtI<sub>2</sub>(bipy) (Connick & Gray, 1994), crystallizing in space group  $C2/c$  with similar cell dimensions. The complex PtI<sub>2</sub>(bipy) has  $a = 17.400$  (4),  $b = 9.809$  (2),  $c = 7.693$  (2) Å and  $\beta = 111.97$  (2)°.



The molecule lies on a twofold axis passing through the Pd atom and the centre of the bond between the two pyridyl moieties (Fig. 1), and Pd has regular square-planar geometry with Pd—N 2.047 (5) and Pd—Br 2.4102 (9) Å, and angles of 80.1 (2) (chelate) and 89.50 (3)° (PdBr<sub>2</sub>) at the Pd atom. The pyridine rings are planar [ $\chi^2$  9.7, maximum deviation from mean plane 0.014 (7) Å for C5]. The coordinated rings form a dihedral angle of 1.7 (3)° with the coordination mean