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High-spin [Fe(TTP)(THF)$_2$]

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The title compound, bis(tetrahydrofuran)(5,10,15,20-tetra-*p*-tolylporphyrinato)iron(II), [Fe($C_{48}H_{36}N_4$)(C_4H_8O)$_2$] or [Fe(TTP)(THF)$_2$] (TTP = dianion of *meso*-tetratolylporphyrin), is a six-coordinate high-spin iron(II) tetratolylporphyrin with two tetrahydrofuran molecules as axial ligands. The Fe^{II} atom is at an inversion center. The average Fe—N distance is 2.067 (7) Å and the Fe—O distance is 2.3208 (8) Å.

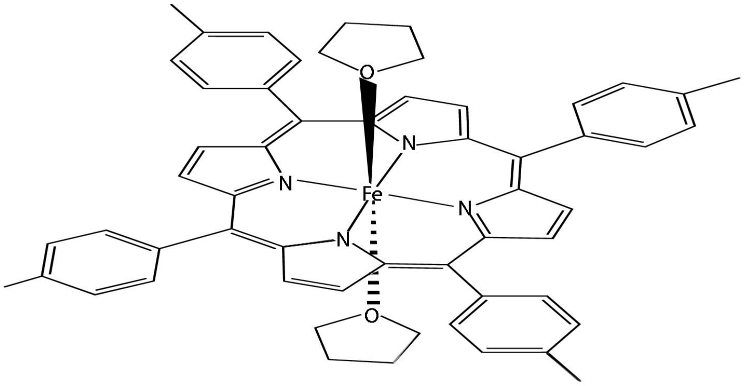
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 25.4

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

Comment

Reed *et al.* (1980) reported the structure of the first six-coordinate high-spin iron(II) porphyrinate, [Fe(TPP)(THF)$_2$]. Subsequently, Lecomte *et al.* (1986) performed an electron-density study of the compound. In this paper, we report the molecular structure of another six-coordinate high-spin iron(II) porphyrinate, the title compound, (I), [Fe(TTP)(THF)$_2$].



(I)

The molecular structure of (I), with the atom-numbering scheme, is shown in Fig. 1. The Fe^{II} atom is located at a crystallographic inversion center. As a result, the Fe^{II} atom is centered in the porphyrin plane. The independent Fe—N bond distances are 2.0619 (9) and 2.0713 (9) Å, which are similar to those in [Fe(TPP)(THF)$_2$] (2.054 and 2.060 Å; Reed *et al.*, 1980). These values are substantially longer than the value of 1.996 (6) Å in the low-spin six-coordinate iron(II) porphyrinate [Fe(TPP)(THT)$_2$] (THT is tetrahydrothiophene; Mashiko *et al.*, 1979).

The axial Fe—O bond distance [2.3208 (8) Å] is similar to the value of 2.351 (3) Å in [Fe(TPP)(THF)$_2$] (Reed *et al.*, 1980). This bond distance indicates that the THF ligand is a weak-field ligand. The Fe—O vector is tipped by 1.3 (1)° from the normal to the mean plane through the plane of the four N atoms. This is smaller than the 4.3° tip seen in

[Fe(TPP)(THF)$_2$] (Reed *et al.*, 1980). This deviation also breaks the D_{4h} symmetry of the FeN$_4$O$_2$ coordination. The THF ligand has an envelope configuration with approximate C_s symmetry. The O atom is displaced from the mean plane of the four C atoms by 0.56 (3) Å.

Experimental

All experimental manipulations were performed under a purified nitrogen atmosphere using standard Schlenk techniques. [Fe(TTP)]$_2$O (35 mg) was reduced in tetrahydrofuran (10 ml) in the presence of excess EtSH overnight. The solution was transfered into glass tubes and layered with hexane. A week later, high-quality red crystals of (I) formed.

Crystal data

[Fe(C$_{48}$H$_{36}$N$_4$)(C$_4$H$_8$O)$_2$]
M_r = 868.87
Monoclinic, $P2_1/n$
a = 13.6820 (2) Å
b = 9.7320 (1) Å
c = 17.1014 (2) Å
β = 104.964 (1)°
V = 2199.89 (5) Å^3
Z = 2
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7064 reflections
θ = 2.5–31.5°
μ = 0.39 mm^{-1}
T = 100 (2) K
Needle, red
0.49 × 0.15 × 0.09 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{\min}$ = 0.83, $T_{\max}$ = 0.97
31 446 measured reflections
7311 independent reflections
6283 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.028
$\theta_{\max}$ = 31.5°
$h = -20 \rightarrow 17$
$k = -11 \rightarrow 14$
$l = -22 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.04
7311 reflections
288 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 1.0408P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.53 e Å^{-3}
$\Delta\rho_{\min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N1	2.0619 (9)	Fe1—O1	2.3208 (8)
Fe1—N2	2.0713 (9)		
N1—Fe1—N2	89.57 (4)	N2—Fe1—O1	88.96 (3)
N1—Fe1—O1	89.34 (3)		

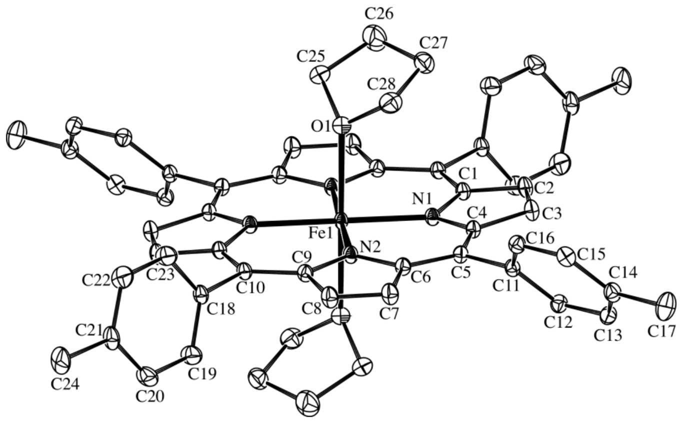


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted. Unlabeled atoms are related by the symmetry operation $-x, 1-y, -z$.

H atoms were positioned geometrically and treated as riding, with C—H distances in the range 0.95–0.99 Å and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *APEX2* (Bruker Nonius, 2004); cell refinement: *APEX2* and *SAINT* (Bruker Nonius, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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