Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Aqua(nitrilotriacetato)(1,10-phenanthroline)iron(III) monohydrate: a seven-coordinate iron(III) complex

Yaoyu Feng,* Say-Leong Ong, Jiangyong Hu and Wun-Jern Ng

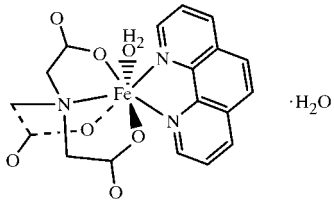
Department of Civil Engineering, National University of Singapore, Singapore 119260, Singapore
Correspondence e-mail: cvefyy@nus.edu.sg

Received 26 September 2001
Accepted 12 October 2001
Online 14 December 2001

Reaction of 1,10-phenanthroline (phen) with iron trichloride in the presence of sodium nitrilotriacetate (NTA) resulted in the formation of red crystals of the title complex, $[Fe(C_6H_6NO_6)(C_{12}H_8N_2)(H_2O)]\cdot H_2O$. The Fe atom has a distorted capped trigonal prismatic coordination comprised of one tetradentate NTA, one bidentate phen molecule and a water molecule. Intermolecular O—H···O hydrogen bonds link the molecules into infinite chains. The chains are crosslinked by hydrogen bonds involving the solvent water molecule, leading to an infinite ladder packing mode.

Comment

The structures of several iron(III) complexes containing coordinated tetradentate NTA (Clegg *et al.*, 1984; White *et al.*, 1984; Heath *et al.*, 1992; Fujita *et al.*, 1994; Powell *et al.*, 1995) have been reported due to the unique reactivity of Fe^{III}–NTA solutions (Awai *et al.*, 1979; Goddard *et al.*, 1986). With the exception of the seven-coordinate $[Fe(NTA)_2]^{3-}$ ion (Clegg *et al.*, 1984), the Fe atoms in all known Fe–NTA complexes are six-coordinate. To the best of our knowledge, seven-coordinate iron(III) compounds are also rare (Marlin *et al.*, 2000; Sanchiz *et al.*, 1997; Finnen *et al.*, 1991; Fleischer & Hawkinson, 1967). Herein we report the X-ray structure of a seven-coordinate iron-centred compound, $[Fe(NTA)(phen)(H_2O)]\cdot H_2O$, (I).



(I)

Unlike most seven-coordinate Fe^{III} complexes, which usually have pentagonal bipyramidal geometries, the Fe atom in (I) lies near the centre of a distorted capped trigonal prism: O1/O3/N2 and O5/O7/N1 make up the two triangular ends of the distorted trigonal prism, while atom N3 (of NTA) occupies the capped position (Fig. 1). The Fe—O7(water) bond length

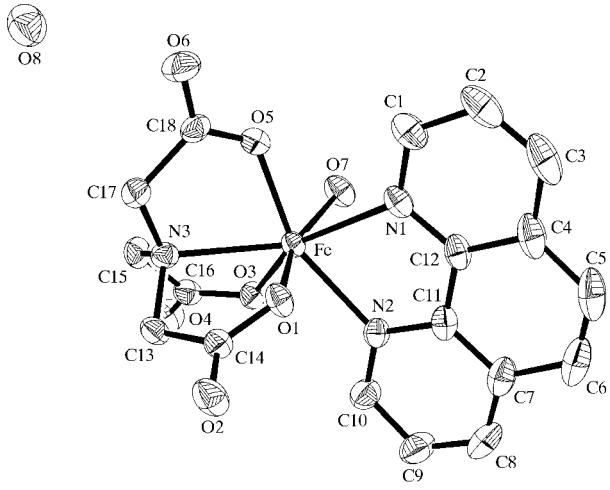


Figure 1
A view (*SHELXTL*; Sheldrick, 1998) of (I) showing the atom labelling and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

is significantly longer than the Fe—O bond lengths to NTA (Table 1), but the latter are similar to those previously observed in Fe(NTA) complexes (Clegg *et al.*, 1984; White *et al.*, 1984; Heath *et al.*, 1992; Fujita *et al.*, 1994; Powell *et al.*, 1995). The Fe—N3 bond length is significantly longer than the average Fe—N(phen) bond lengths [2.2340 (13) Å]. Steric effects due to the constraints on the NTA ligand, required by its tetradentate ligation, may be responsible for the difference in the Fe—N bond lengths. The molecules are packed in such a way that the intermolecular O—H···O hydrogen-bonding interconnections [2.696 (2) Å] between the coordinated water molecule and the carboxylate oxygen (O2) in the NTA ligand (Table 2) results in infinite chains of molecules. Each solvent water molecule bridges two iron complex molecules through long-range intermolecular O8—H···O hydrogen bonds and forms the rung of the infinite ladder packing mode.

Experimental

A methanol solution of phen (36.02 mg, 0.2 mmol) was added slowly to a methanol solution of $FeCl_3\cdot 6H_2O$ (54.06 mg, 0.2 mmol), followed by a water solution of $Na[N(CH_2COO)_3]$ (51.42 mg, 0.2 mmol). Well-shaped red polyhedral crystals of (I) separated from the mother liquor by slow evaporation at room temperature after three weeks. They were filtered off, washed with a small amount of water, and dried in air; yield 25%. Analysis calculated for $C_{18}H_{18}FeN_3O_8$: C 46.77, H 4.36, N 9.09%; found C 46.93, H 4.62, N 9.35%.

 © 2002 International Union of Crystallography DOI: 10.1107/S0108270101017036

Crystal data

$[Fe(C_6H_6NO_6)(C_{12}H_8N_2)(H_2O)]\cdot H_2O$
$M_r = 460.20$
Monoclinic, $P2_1/c$
$a = 11.7051\ (5)$ Å
$b = 8.0381\ (3)$ Å
$c = 19.8739\ (7)$ Å
$\beta = 92.879\ (2)^\circ$
$V = 1867.51\ (12)$ Å^3
$Z = 4$
$D_x = 1.637$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 260 reflections
$\theta = 3.5$–27.9°
$\mu = 0.86$ mm^{-1}
$T = 293\ (2)$ K
Polyhedron, red
$0.27 \times 0.16 \times 0.11$ mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: empirical (Blessing, 1995, 1997)
$T_{min} = 0.789$, $T_{max} = 0.909$
24 260 measured reflections
4467 independent reflections
3086 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.9^\circ$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 10$
$l = -25 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.098$
$S = 1.02$
4467 reflections
343 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.1433P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

Fe—O3	2.0054 (15)	Fe—N2	2.2311 (19)
Fe—O5	2.0208 (15)	Fe—N1	2.2370 (19)
Fe—O1	2.0316 (15)	Fe—N3	2.3381 (19)
Fe—O7	2.0744 (16)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H20···O6	0.82 (3)	2.00 (3)	2.818 (3)	176 (4)
O8—H21···O3^{i}	0.83 (3)	2.16 (3)	2.971 (3)	164 (4)
O7—H22···O2^{ii}	0.92 (3)	1.79 (3)	2.696 (2)	168 (3)
O7—H23···O8^{iii}	0.78 (4)	1.95 (4)	2.734 (3)	176 (4)

Symmetry codes: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $x, 1+y, z$; (iii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$.

All H atoms were located in difference maps and refined isotropically [C—H = 0.81 (3)–1.02 (3) Å)].

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1352). Services for accessing these data are described at the back of the journal.

References

Awai, M., Narasaki, M., Yamanoi, Y. & Seno, S. (1979). *Am. J. Pathol.* **95**, 663–672.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Clegg, W., Powell, A. K. & Ware, M. J. (1984). *Acta Cryst.* C**40**, 1822–1823.
Finnen, D. C., Pinkerton, A. A., Dunham, W. R., Sands, R. H. & Funk, M. O. (1991). *Inorg. Chem.* **30**, 3960–3964.
Fleischer, E. B. & Hawkinson, S. W. (1967). *J. Am. Chem. Soc.* **89**, 720–721.
Fujita, T., Ohba, S., Nishida, Y., Goto, A. & Tokii, T. (1994). *Acta Cryst.* C**50**, 544–546.
Goddard, J. G., Basford, D. & Sweeney, G. D. (1986). *Biochem. Pharmacol.* **35**, 2381–2387.
Heath, S. L., Powell, A. K., Utting, H. L. & Helliwell, M. (1992). *J. Chem. Soc. Dalton Trans.* pp. 305–307.
Mackay, S., Gilmore, C. J., Edwards, C., Tremayne, M., Stuart, N. & Shankland, K. (1998). *maXus*. University of Glasgow, Scotland, Nonius BV, Delft, The Netherlands, and MacScience Co. Ltd, Yokohama, Japan.
Marlin, D. S., Olmstead, M. M. & Mascharak, P. K. (2000). *Inorg. Chim. Acta*, **297**, 106–114.
Nonius (1997–2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter & R. M. Sweet, pp. 307–326. London: Academic Press.
Powell, A. K., Heath, S. L., Gatteschi, D., Pardi, L., Sessoli, R., Spina, G., Giallo, F. D. & Pieralli, F. (1995). *J. Am. Chem. Soc.* **117**, 2491–2502.
Sanchiz, J., Dominguez, S., Mederos, A., Brito, F. & Arrieta, J. M. (1997). *Inorg. Chem.* **36**, 4108–4114.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. PC Versions. University of Göttingen, Germany.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
White, L. S., Nillson, P. V., Pignolet, L. H. & Que, L. (1984). *J. Am. Chem. Soc.* **106**, 8312–8313.