solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982) and refined by full-matrix least-squares calculations, initially with isotropic and finally with anisotropic displacement parameters for non H-atoms. All H atoms were found from a difference Fourier map and included in the structure-factor calculations. All calculations were performed on DEC MicroVAX II and IBM RS/6000 computers using the *Enraf–Nonius SDP-Plus* (Frenz, 1985) and *Xtal*3.2 (Hall, Flack & Stewart, 1992) systems. Molecular graphics were prepared using *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and root-mean-square amplitudes have been deposited with the IUCr (Reference: NA1171). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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fac-Tris(isothiocyanato)tris(triphenylphosphine oxide)iron(III) at 80 K

ZOFIA OLEJNIK AND TADEUSZ LIS

Institute of Chemistry, University of Wrocław, ul. Joliot-Curie 14, 50-383 Wrocław, Poland

IVETA ONDREJKOVIČOVÁ

Department of Inorganic Chemistry, Slovak Technical University, 9 Radlinského St., 812 37 Bratislava, Slovakia

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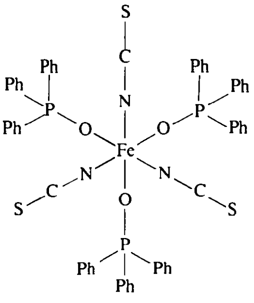
Abstract

The iron(III) complex $[Fe(NCS)_3(C_{18}H_{15}PO)_3]$ crystallizes in space group $R3$. One complex molecule with no crystallographically imposed symmetry and one-third of another molecule arranged about a threefold axis are contained in the asymmetric unit. Both molecules have almost the same geometry for the octahedral FeO_3N_3 chromophores, but opposite chirality of the arrangement of the ligands around the Fe atoms.

Comment

Iron(III) triphenylphosphine oxide complexes of composition $FeX_3(OPPh_3)_2$ (X = Cl^-, Br^- or SCN^-) act as oxidation catalysts of PPh_3 with O_2 in acetonitrile solutions (Ondrejkovičová, Vančová & Ondrejovič, 1983, 1991). These complexes show different catalytic activity, which depends not only on the redox potentials X_2/X^- but also on their crystal structures. Chloro and bromo complexes have the ionic structure $[FeX_2(OPPh_3)_4][FeX_4]$ (Durčanská, Głowiak, Kožišek, Ondrejkovičová & Ondrejovič, 1989; Durčanská, Głowiak, Gyepes, Ondrejkovičová & Ondrejovič, 1991) and $[Fe(NCS)_3(OPPh_3)_2]$ has a non-ionic structure (Cotton & Gibson, 1971).



(I)

The title compound, (I), the crystal structure of which we present here, exhibits catalytic properties similar to those of $[Fe(NCS)_3(OPPh_3)_2]$ (Vančová, Ondrejkovičová & Ondrejovič, 1984). The unit cell contains twelve molecules of $[Fe(NCS)_3(OPPh_3)_3]$, in each of which three NCS^- and three $OPPh_3$ ligands are coordinated at facial sites octahedrally around the Fe atom. Three of the complex molecules (type A) have crystallographic threefold symmetry and the remaining nine (type B), situated at general positions, have approximate threefold symmetry. In Fig. 1 an *ORTEP*II (Johnson, 1976) view of both molecules projected along the c axis is presented. As can be seen, molecules A and B have opposite chirality, probably imposed by the crystal packing and/or the arrangement of triphenylphosphine oxide ligands. More pronounced differences between the independent molecules A and B are observed in the Fe—N—C and Fe—O—P angles (see Table 2), which are more acute in A. Both molecules have very similar coordination octahedra, the weighted r.m.s. deviation for the FeO_3N_3 cores being 0.0129 Å. All Fe—N and Fe—O bond distances are almost equal and the N—Fe—N and O—Fe—O bond angles do not exhibit deviations from

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90° larger than 3°. Slightly greater deviations from octahedral symmetry involve some of the O—Fe—N angles, indicating a twisting of the coordination octahedra about their threefold (for *A*) and pseudo threefold (for *B*) axes. The triangular O_3 face is rotated by *ca* 55° with respect to the N_3 face in the same direction (which is surprising) for both molecules.

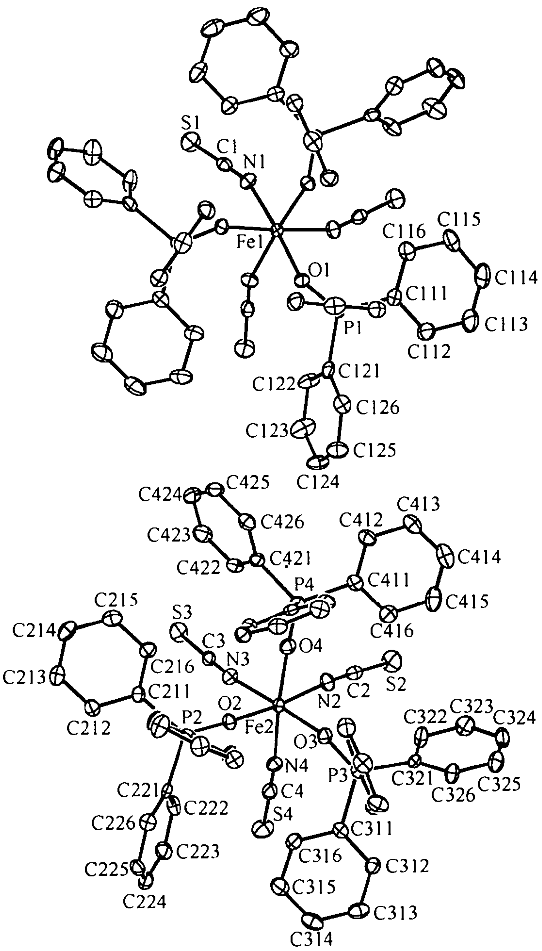

Fig. 1. View of the two crystallographically independent molecules of $[Fe(NCS)_3(OPPh_3)_3]$ projected along the *c* axis. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

The title compound was prepared by autocatalytic oxidation of PPh_3 (6 mmol) by dioxygen in the presence of $Fe_2(SO_4)_3.9H_2O$ (1 mmol) and KSCN (6 mmol) in acetonitrile (30 ml). The reaction mixture was stirred with heating at 353 K under a reflux condenser and O_2 was supplied until all the PPh_3 was oxidized (*ca* 2 d). After oxidation, K_2SO_4 was filtered off and well formed crystals were grown from the solution obtained. The crystals are not very stable at room temperature and therefore the X-ray data were collected at low temperature. [At 293 (1) K, a = 38.59 (1), c = 12.310 (9) Å, V = 15876 (13) Å^3, D_x = 1.337 (2), D_m = 1.33 Mg m^{-3} (flotation in C_6H_6/CH_2Cl_2).] A small specimen was cut from a large crystal for X-ray studies.

Crystal data

$[Fe(NCS)_3(C_{18}H_{15}PO)_3]$
M_r = 1064.9
Trigonal
$R3$
a = 38.40 (1) Å
c = 12.092 (9) Å
V = 15442 (13) Å^3
Z = 12
D_x = 1.374 (2) Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 9–11°
μ = 0.556 mm^{-1}
T = 80 (2) K
Rhombohedron
0.6 × 0.4 × 0.4 mm
Red–brown

Data collection

Kuma KM4 computer-controlled four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF
$T_{\min}$ = 0.75, $T_{\max}$ = 1.00
14523 measured reflections
12020 independent reflections (Friedel pairs not merged)
7641 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.0263
$\theta_{\max}$ = 25°
$h = -45 \rightarrow 29$
$k = 0 \rightarrow 45$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 100 reflections
intensity decay: 14%

Refinement

Refinement on F^2
$R(F)$ = 0.0289
$wR(F^2)$ = 0.0759
S = 1.030
7641 reflections
3478 Friedel opposites
835 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 19.13P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max}$ = −0.166
$\Delta\rho_{\max}$ = 0.308 e Å^{-3}
$\Delta\rho_{\min}$ = −0.405 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = 0.192 (13)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Fe1	0	0	0.17948 (9)	0.0134 (2)
Fe2	0.16769 (2)	0.33511 (2)	0.5	0.0140 (2)
N1	−0.00044 (10)	−0.04315 (10)	0.0819 (3)	0.0199 (7)
N2	0.12081 (9)	0.31616 (10)	0.3949 (3)	0.0213 (7)
N3	0.19114 (10)	0.30915 (10)	0.4013 (3)	0.0196 (7)
N4	0.19766 (10)	0.38639 (10)	0.4132 (3)	0.0210 (7)
O1	0.00421 (8)	0.04408 (7)	0.2809 (2)	0.0188 (6)
O2	0.21222 (7)	0.34849 (8)	0.6101 (2)	0.0182 (6)
O3	0.14708 (8)	0.36285 (8)	0.6018 (2)	0.0196 (6)
O4	0.13412 (8)	0.28408 (8)	0.5864 (2)	0.0206 (6)
C1	0.01001 (11)	−0.05771 (11)	0.0147 (3)	0.0195 (9)
C2	0.09649 (12)	0.30866 (11)	0.3250 (3)	0.0188 (8)
C3	0.19887 (11)	0.29294 (11)	0.3321 (3)	0.0183 (8)

C4	0.21355 (12)	0.40994 (11)	0.3421 (3)	0.0216 (9)
S1	0.02482 (3)	−0.07812 (3)	−0.07786 (11)	0.0303 (3)
S2	0.06300 (4)	0.29947 (4)	0.23182 (11)	0.0339 (3)
S3	0.20944 (3)	0.26971 (3)	0.23758 (10)	0.0288 (2)
S4	0.23578 (4)	0.44221 (3)	0.24412 (10)	0.0322 (3)
P1	−0.00953 (3)	0.07107 (3)	0.33058 (10)	0.0166 (2)
P2	0.25167 (3)	0.35858 (3)	0.66253 (9)	0.0151 (2)
P3	0.13943 (3)	0.39496 (3)	0.64585 (9)	0.0159 (2)
P4	0.10688 (3)	0.24598 (3)	0.64719 (9)	0.0160 (2)
C111	−0.05777 (12)	0.06045 (12)	0.2828 (3)	0.0203 (9)
C112	−0.06599 (13)	0.09140 (13)	0.2635 (4)	0.0288 (10)
C113	−0.10395 (14)	0.08254 (15)	0.2334 (4)	0.0390 (12)
C114	−0.13378 (14)	0.04332 (15)	0.2203 (4)	0.0370 (11)
C115	−0.12640 (12)	0.01219 (14)	0.2381 (4)	0.0318 (10)
C116	−0.08833 (12)	0.02053 (13)	0.2693 (3)	0.0246 (9)
C121	0.02539 (12)	0.12348 (12)	0.3005 (3)	0.0204 (8)
C122	0.04531 (13)	0.13291 (12)	0.1985 (4)	0.0267 (9)
C123	0.07081 (15)	0.17343 (14)	0.1717 (4)	0.0368 (11)
C124	0.07496 (13)	0.20318 (12)	0.2439 (4)	0.0299 (10)
C125	0.05453 (13)	0.19283 (12)	0.3442 (4)	0.0280 (10)
C126	0.03036 (12)	0.15357 (12)	0.3713 (3)	0.0237 (9)
C131	−0.01185 (11)	0.06718 (11)	0.4787 (3)	0.0177 (8)
C132	−0.04020 (11)	0.07129 (11)	0.5405 (3)	0.0196 (8)
C133	−0.03912 (12)	0.07066 (12)	0.6553 (3)	0.0229 (9)
C134	−0.00917 (13)	0.06685 (12)	0.7082 (3)	0.0265 (9)
C135	0.01856 (12)	0.06252 (12)	0.6479 (4)	0.0266 (9)
C136	0.01804 (12)	0.06262 (11)	0.5328 (3)	0.0212 (8)
C211	0.26751 (11)	0.32297 (11)	0.6300 (3)	0.0171 (8)
C212	0.30784 (11)	0.33375 (12)	0.6505 (3)	0.0218 (9)
C213	0.31927 (13)	0.30507 (13)	0.6366 (4)	0.0259 (9)
C214	0.29180 (13)	0.26647 (13)	0.6026 (3)	0.0269 (9)
C215	0.25264 (13)	0.25654 (13)	0.5786 (4)	0.0266 (10)
C216	0.24020 (12)	0.28454 (11)	0.5944 (3)	0.0210 (8)
C221	0.29041 (11)	0.40757 (11)	0.6221 (3)	0.0166 (8)
C222	0.29184 (12)	0.42031 (13)	0.5135 (3)	0.0227 (9)
C223	0.31955 (12)	0.45899 (12)	0.4821 (4)	0.0262 (9)
C224	0.34652 (12)	0.48511 (12)	0.5564 (4)	0.0260 (9)
C225	0.34667 (11)	0.47329 (12)	0.6653 (4)	0.0229 (9)
C226	0.31864 (11)	0.43449 (11)	0.6983 (3)	0.0211 (9)
C231	0.24837 (10)	0.35983 (11)	0.8111 (3)	0.0171 (8)
C232	0.26746 (13)	0.34673 (13)	0.8812 (4)	0.0256 (9)
C233	0.26572 (13)	0.35102 (14)	0.9966 (4)	0.0301 (10)
C234	0.24516 (12)	0.36911 (13)	1.0377 (3)	0.0263 (9)
C235	0.22559 (13)	0.38180 (12)	0.9687 (4)	0.0278 (9)
C236	0.22709 (12)	0.37727 (12)	0.8535 (3)	0.0213 (8)
C311	0.17814 (11)	0.44527 (11)	0.6139 (3)	0.0170 (8)
C312	0.17030 (12)	0.47705 (12)	0.6082 (3)	0.0234 (9)
C313	0.20142 (13)	0.51584 (13)	0.6053 (4)	0.0284 (10)
C314	0.24088 (13)	0.52439 (13)	0.6080 (3)	0.0292 (10)
C315	0.24938 (13)	0.49305 (13)	0.6095 (4)	0.0281 (9)
C316	0.21806 (12)	0.45372 (12)	0.6140 (3)	0.0240 (9)
C321	0.09140 (11)	0.38712 (11)	0.5998 (3)	0.0201 (9)
C322	0.07566 (12)	0.36633 (13)	0.5015 (4)	0.0251 (9)
C323	0.03741 (13)	0.35720 (14)	0.4697 (4)	0.0313 (10)
C324	0.01526 (12)	0.36848 (13)	0.5332 (4)	0.0285 (10)
C325	0.03080 (13)	0.38945 (14)	0.6295 (4)	0.0279 (10)
C326	0.06904 (12)	0.39869 (12)	0.6642 (3)	0.0229 (9)
C331	0.13783 (11)	0.39377 (12)	0.7945 (3)	0.0179 (8)
C332	0.14167 (12)	0.42594 (12)	0.8574 (4)	0.0257 (9)
C333	0.13806 (12)	0.42314 (13)	0.9706 (4)	0.0288 (10)
C334	0.13098 (12)	0.38764 (13)	1.0233 (3)	0.0275 (10)
C335	0.12727 (12)	0.35619 (13)	0.9624 (4)	0.0284 (10)
C336	0.12979 (11)	0.35818 (11)	0.8480 (3)	0.0215 (9)
C411	0.05465 (11)	0.22959 (12)	0.6260 (3)	0.0198 (8)
C412	0.02491 (12)	0.19037 (12)	0.6493 (3)	0.0242 (9)
C413	−0.01527 (12)	0.17943 (13)	0.6427 (4)	0.0284 (10)
C414	−0.02537 (13)	0.20779 (15)	0.6124 (4)	0.0334 (11)
C415	0.00335 (13)	0.24603 (15)	0.5853 (4)	0.0335 (11)
C416	0.04412 (13)	0.25819 (13)	0.5923 (3)	0.0252 (9)
C421	0.11747 (11)	0.20721 (11)	0.6077 (3)	0.0183 (8)
C422	0.13663 (12)	0.21161 (12)	0.5061 (3)	0.0221 (9)
C423	0.14619 (12)	0.18317 (13)	0.4730 (4)	0.0292 (10)
C424	0.13680 (12)	0.14998 (12)	0.5382 (4)	0.0284 (10)
C425	0.11738 (12)	0.14545 (11)	0.6391 (4)	0.0254 (9)
C426	0.10762 (11)	0.17372 (12)	0.6736 (3)	0.0223 (9)
C431	0.11510 (11)	0.25237 (10)	0.7944 (3)	0.0160 (8)
C432	0.15425 (11)	0.26626 (11)	0.8330 (3)	0.0194 (8)
C433	0.16237 (12)	0.27308 (11)	0.9439 (3)	0.0223 (9)
C434	0.13255 (12)	0.26653 (12)	1.0182 (4)	0.0245 (9)
C435	0.09341 (13)	0.25223 (13)	0.9803 (4)	0.0273 (10)
C436	0.08483 (12)	0.24594 (11)	0.8692 (3)	0.0226 (9)

Table 2. *Selected geometric parameters* (Å, °)

Fe1—N1	2.027 (3)	N3—C3	1.166 (5)
Fe1—O1	2.030 (3)	N4—C4	1.173 (5)
Fe2—N2	2.019 (3)	O1—P1	1.503 (3)
Fe2—N3	2.032 (3)	O2—P2	1.503 (3)
Fe2—N4	2.010 (3)	O3—P3	1.501 (3)
Fe2—O2	2.021 (3)	O4—P4	1.499 (3)
Fe2—O3	2.030 (3)	C1—S1	1.623 (4)
Fe2—O4	2.016 (3)	C2—S2	1.611 (4)
N1—C1	1.166 (5)	C3—S3	1.622 (4)
N2—C2	1.183 (5)	C4—S4	1.616 (4)
N1—Fe1—N1$^{\mathrm{i}}$	89.5 (2)	O4—Fe2—N3	93.0 (2)
N1—Fe1—O1	176.1 (2)	N2—Fe2—N3	89.3 (2)
N1$^{\mathrm{i}}$—Fe1—O1	89.1 (2)	O2—Fe2—N3	89.2 (2)
N1—Fe1—O1$^{\mathrm{i}}$	94.1 (2)	O3—Fe2—N3	177.1 (2)
O1—Fe1—O1$^{\mathrm{i}}$	87.3 (2)	C1—N1—Fe1	159.0 (3)
N4—Fe2—O4	175.9 (2)	C2—N2—Fe2	172.0 (3)
N4—Fe2—N2	89.7 (2)	C3—N3—Fe2	167.6 (3)
O4—Fe2—N2	87.4 (2)	C4—N4—Fe2	163.8 (3)
N4—Fe2—O2	95.6 (2)	P1—O1—Fe1	155.1 (2)
O4—Fe2—O2	87.4 (2)	P2—O2—Fe2	163.5 (2)
N2—Fe2—O2	174.5 (2)	P3—O3—Fe2	159.1 (2)
N4—Fe2—O3	89.2 (2)	P4—O4—Fe2	176.4 (2)
O4—Fe2—O3	88.2 (2)	N1—C1—S1	179.4 (3)
N2—Fe2—O3	93.4 (2)	N2—C2—S2	178.1 (3)
O2—Fe2—O3	88.2 (2)	N3—C3—S3	178.9 (4)
N4—Fe2—N3	89.8 (2)	N4—C4—S4	179.4 (3)

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

Three standard reflections decreased approximately 14% over the period of data collection and the data were rescaled using the intensities of the standards. The trigonal system and $\bar{3}$ Laue class determined from Weissenberg photographs suggested the space group to be either $R3$ or $R\bar{3}$. A reasonable solution could only be obtained in $R3$. Absorption corrections were applied by the method of Walker & Stuart (1983) as implemented by the locally modified program *ABSORB* (Ugozzoli, 1987) for the set of data $> 3\sigma(I)$. As the value of the Flack parameter (Flack, 1983) was more than three e.s.d.'s from the ideal value of 0.0, refinement of the inverted structure was carried out, giving $R(F) = 0.0354$ and $wR(F) = 0.0942$.

Data collection: Kuma (1991) KM4 diffractometer software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1168). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexakis(dimethyl sulfoxide-*O*)iron(III) Trinitrate

Ji-Ren Tzou, Matthew Mullaney and
Richard E. Norman

*Department of Chemistry and Biochemistry,
Duquesne University, Pittsburgh, PA 15282, USA*

Shih-Chi Chang

*Department of Physics, Duquesne University,
Pittsburgh, PA 15282, USA*

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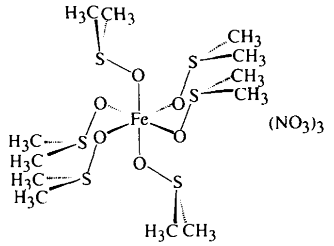
Abstract

The title compound, $[Fe(C_2H_6OS)_6](NO_3)_3$, was prepared by dissolving an iron(III) complex of *N*,*N*,*N'*,*N'*-tetrakis(2-benzimidazolylmethyl)-2-hydroxy-1,3-diaminopropane in a 20% *v*/*v* solution of dimethyl sulfoxide in acetonitrile for NMR studies. Crystals of hexakis(dimethyl sulfoxide-*O*)iron(III) trinitrate formed upon standing. The iron(III) atom sits on a site of $\bar{3}$ symmetry and has approximately octahedral coordination [the O—Fe—O angles are 87.40 (9) and 92.60 (9)°, and the Fe—O distance is 2.020 (2) Å]. One nitrate anion is centered on a site of 3 symmetry and the other is disordered about a site of $\bar{3}$ symmetry. The first nitrate anion appears twice per iron center, while the second appears only once.

Comment

Dimethyl sulfoxide (DMSO) is used widely as a solvent for metal complexes and is a known ligand for virtually all metal ions (Davies, 1981; Reynolds, 1970). During our studies of the reactions of iron(III) nitrate nonahydrate with *N*,*N*,*N'*,*N'*-tetrakis(2-benzimidazolylmethyl)-2-hydroxy-1,3-diaminopropane (HPTB) (Tzou & Norman, unpublished results; Tzou, Chang & Norman, 1993), we isolated four crystals of hexakis(dimethyl sulfoxide-*O*)iron(III) trinitrate, (I), the structure of which is reported herein. The preparation of this complex has been reported previously (Langford & Chung, 1968).



(I)

Only a few crystallographic determinations of the $[M(DMSO)_6]^{n+}$ unit have been carried out, namely, $[Hg(DMSO)_6](ClO_4)_2$ (Sandström & Persson, 1978), $[Cd(DMSO)_6](ClO_4)_2$ (Sandström, 1978; Lyubeznova & Ponomarev, 1989), $[Ru(DMSO)_6](BF_4)_2$ (Davies, Einstein, Farrell, James & McMillan, 1978), $[Zn(DMSO)_6](ClO_4)_2$ (Persson, 1982), $[Fe(DMSO)_6]^{2+}$ in a non-stoichiometric complex (Huang, Jian, Wang & Lu, 1985), $[Fe(DMSO)_6][Cl_2Fe(MoS_4)]$ and $[Fe(DMSO)_6][Cl_2Fe(MoOS_3)]$ (Müller *et al.*, 1989), $[In(DMSO)_6](ClO_4)_3$ (Harrowfield, Skelton & White, 1990), $[Mn(DMSO)_6][W_6O_{19}]$ and $[Cu(DMSO)_6]$-$[W_6O_{19}]$ (Chen, Zhu & Gu, 1990), and $[Cd(DMSO)_6]$-$[Cd(DMSO)I_3]_2.C_2H_5OH$ and $[Cd(DMSO)_6][CdI_4]$ (Nieuwenhuyzen, Wen & Wilkins, 1992). The title complex contains an approximately octahedral iron(III) center located at a site of $\bar{3}$ symmetry (Fig. 1) and two distinct nitrate ion environments, one ordered at a site of 3 symmetry and the other disordered at a site of $\bar{3}$ symmetry. Similarly, in $[In(DMSO)_6](ClO_4)_3$, the metal center is located at a site of $\bar{3}$ symmetry and the anions are disordered about sites of 3 and $\bar{3}$ symmetry (Harrowfield *et al.*, 1990). The Fe—O distance of 2.020 (2) Å compares well with the value of 2.006 (6) Å reported for *trans*-$[FeCl_2(DMSO)_4][FeCl_4]$ (Bennett, Cotton & Weaver, 1967) and the distances reported for Fe—O not *trans* to Cl [2.003 (4)–2.032 (4) Å] in $[FeCl(DMSO)_5][Fe_2Cl_6O]$ (Ponomarev, Arutyunyan & Atovmyan, 1984), and is, as expected, shorter than the distances of 2.132 (3), 2.102 (3) and 2.149 (3) Å found in $[Fe(DMSO)_6]^{2+}$ (Müller *et al.*, 1989). The metrical parameters of DMSO are typical for O-bonded metal complexes (Davies, 1981; Sandström & Persson, 1978; Sandström, 1978; Davies *et al.*, 1978; Persson, 1982; Lyubeznova & Ponomarev, 1989; Harrowfield *et al.*, 1990; Müller *et al.*, 1989; Ponomarev *et al.*, 1984; Bennett *et al.*, 1967; Chen *et al.*, 1990; Nieuwenhuyzen *et al.*, 1992).

The ordered nitrate (located at a site of 3 symmetry) is unremarkable. However, the nitrate disordered at the site of $\bar{3}$ symmetry deserves comment. One possible interpretation of this disordered anion would be as a

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