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(*N*,*N*,*N*',*N*'-Tetramethylethylenediamine-*N*,*N*')bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato-*O*,*O*')iron(II)

M. H. Dickman

Abstract

The title compound consists of discrete neutral complexes with crystallographic C_2 symmetry. Fe—O bond distances are 2.0556 (12) and 2.0937 (13) Å. The Fe—N distance is 2.2290 (16) Å.

Comment

The structure of the title compound confirms the presence of neutral monomers in the solid state as previously reported on the basis of mass spectrometry results (Bailey *et al.*, 1981). In contrast, addition of a bidentate nitrone to bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato-O,*O*')iron(II) resulted in ligand exchange to produce a complex salt (Villamena *et al.*, 1998).

Experimental

The synthesis has been reported previously (Bailey *et al.*, 1981). Crystals were grown by evaporation from a $CHCl_3$/n-heptane solution.

Computing details

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990b); software used to prepare material for publication: *SHELXTL*.

(*N*,*N*,*N*',*N*'-Tetramethylethylenediamine-*N*,*N*')bis(1,1,1,5,5,5- hexafluoropentane-2,4-dionato-O,*O*')iron(II)

Crystal data

$[Fe(C_5HF_6O_2)_2(C_6H_{16}N_2)]$	$V = 2334.9\ (2)$ Å^3
$M_r = 586.17$	$Z = 4$
Monoclinic, $C2/c$	Mo $K\alpha$
$a = 9.1202\ (5)$ Å	$\mu = 0.77$ mm^{-1}
$b = 14.7430\ (8)$ Å	$T = 173\ (2)$ K
$c = 17.7793\ (9)$ Å	0.50 × 0.38 × 0.30 mm
$\beta = 102.389\ (1)^o$	

Data collection

Siemens CCD	2774 independent reflections

CIF access

diffractometer

Absorption correction: multi-scan (Blessing, 1995)

T_{min} = 0.654, T_{max} = 0.795

7427 measured reflections

2439 reflections with $I > 2\sigma(I)$

R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037

$wR(F^2)$ = 0.098

S = 1.04

2774 reflections

195 parameters

All H-atom parameters refined

$\Delta\rho_{max}$ = 0.56 e Å^{-3}

$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1

Selected geometric parameters (Å, °)

Fe1—O1	2.0556 (12)	C3—C3^{i}	1.515 (5)
Fe1—O2	2.0937 (13)	C3—H3A	0.97 (3)
Fe1—N	2.2290 (16)	C3—H3B	1.01 (3)
N—C1	1.477 (3)	C4—F1	1.309 (2)
N—C3	1.480 (3)	C4—F2	1.324 (2)
N—C2	1.484 (3)	C4—F3	1.335 (2)
O1—C5	1.257 (2)	C4—C5	1.533 (2)
O2—C7	1.244 (2)	C5—C6	1.390 (2)
C1—H1A	1.00 (3)	C6—C7	1.399 (2)
C1—H1B	0.92 (3)	C6—H6	0.93 (2)
C1—H1C	0.96 (3)	C7—C8	1.537 (3)
C2—H2A	1.06 (3)	C8—F4	1.314 (3)
C2—H2B	0.97 (3)	C8—F5	1.325 (3)
C2—H2C	0.93 (3)	C8—F6	1.325 (3)
O1^{i}—Fe1—O1	175.01 (7)	H2B—C2—H2C	107 (2)
O1^{i}—Fe1—O2	91.02 (5)	N—C3—C3^{i}	111.08 (16)
O1—Fe1—O2	85.44 (5)	N—C3—H3A	107.7 (15)
O2—Fe1—O2^{i}	89.98 (8)	C3^{i}—C3—H3A	109.6 (16)
O1—Fe1—N^{i}	90.11 (5)	N—C3—H3B	107.9 (15)
O2—Fe1—N^{i}	173.97 (6)	C3^{i}—C3—H3B	109.5 (15)
O1—Fe1—N	93.66 (6)	H3A—C3—H3B	111 (2)
O2—Fe1—N	94.18 (6)	F1—C4—F2	108.83 (19)
N^{i}—Fe1—N	82.02 (8)	F1—C4—F3	106.73 (19)
C1—N—C3	110.76 (17)	F2—C4—F3	105.90 (15)
C1—N—C2	107.55 (17)	F1—C4—C5	111.17 (15)
C3—N—C2	109.98 (18)	F2—C4—C5	110.55 (17)
C1—N—Fe1	111.96 (13)	F3—C4—C5	113.41 (15)
C3—N—Fe1	104.90 (11)	O1—C5—C6	128.49 (16)
C2—N—Fe1	111.71 (13)	O1—C5—C4	113.27 (15)
C5—O1—Fe1	127.93 (11)	C6—C5—C4	118.22 (16)
C7—O2—Fe1	127.60 (11)	C5—C6—C7	121.22 (16)

N—C1—H1A	110.6 (15)	C5—C6—H6	120.7 (14)
N—C1—H1B	110.6 (15)	C7—C6—H6	118.1 (14)
H1A—C1—H1B	106 (2)	O2—C7—C6	128.02 (16)
N—C1—H1C	109.1 (14)	O2—C7—C8	113.81 (15)
H1A—C1—H1C	108.0 (19)	C6—C7—C8	118.15 (16)
H1B—C1—H1C	113 (2)	F4—C8—F5	107.08 (19)
N—C2—H2A	106.4 (16)	F4—C8—F6	108.1 (2)
N—C2—H2B	108.9 (17)	F5—C8—F6	105.8 (2)
H2A—C2—H2B	111 (2)	F4—C8—C7	111.72 (18)
N—C2—H2C	106.7 (16)	F5—C8—C7	110.06 (18)
H2A—C2—H2C	117 (2)	F6—C8—C7	113.72 (16)

Symmetry codes: (i) $-x$, y, $-z+1/2$.

Acknowledgements

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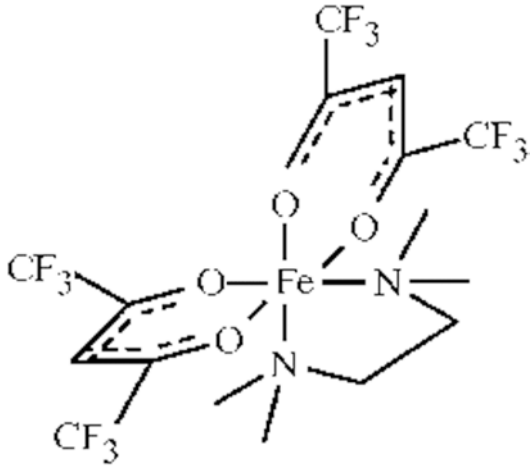
CIF access

Scheme 1



supplementary materials

(*N*,*N*,*N*',*N*'-Tetramethylethylenediamine-*N*,*N*')bis(1,1,1,5,5,5- hexafluoropentane-2,4-dionato-*O*,*O*')iron(II)

Crystal data

$[Fe(C_5HF_6O_2)_2(C_6H_{16}N_2)]$	F_{000} = 1176
M_r = 586.17	D_x = 1.667 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 9.1202 (5) Å	Cell parameters from 4164 reflections
b = 14.7430 (8) Å	θ = 2.4–28.3º
c = 17.7793 (9) Å	μ = 0.77 mm^{-1}
β = 102.389 (1)º	*T* = 173 (2) K
V = 2334.9 (2) Å^3	Block, red
Z = 4	0.50 × 0.38 × 0.30 mm

Data collection

Siemens CCD diffractometer	2774 independent reflections
Radiation source: normal-focus sealed tube	2439 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 173(2) K	θ_{max} = 28.3º
ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (Blessing, 1995)	*h* = −11→11
T_{min} = 0.654, T_{max} = 0.795	*k* = −19→16
7427 measured reflections	*l* = −23→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.037	All H-atom parameters refined
$wR(F^2)$ = 0.098	$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 1.870P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
2774 reflections	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
195 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Crystal decay was monitored by re-collection of the first fifty frames after data collection was finished.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0	0.37441 (2)	0.25	0.02914 (12)
N	−0.11933 (17)	0.48850 (11)	0.18036 (9)	0.0365 (3)
O1	0.16610 (14)	0.36834 (9)	0.18817 (8)	0.0348 (3)
O2	−0.10672 (14)	0.27398 (9)	0.17444 (8)	0.0391 (3)
C1	−0.0701 (3)	0.50025 (17)	0.10709 (12)	0.0463 (5)
H1A	−0.126 (3)	0.5508 (19)	0.0765 (15)	0.059 (7)*
H1B	0.030 (3)	0.5165 (17)	0.1162 (13)	0.048 (6)*
H1C	−0.091 (3)	0.4459 (18)	0.0774 (13)	0.045 (6)*
C2	−0.2843 (2)	0.47429 (19)	0.16139 (16)	0.0530 (6)
H2A	−0.304 (3)	0.416 (2)	0.1258 (16)	0.068 (8)*
H2B	−0.332 (3)	0.527 (2)	0.1341 (15)	0.064 (8)*
H2C	−0.315 (3)	0.4714 (18)	0.2079 (16)	0.056 (7)*
C3	−0.0820 (3)	0.56959 (15)	0.22979 (13)	0.0472 (5)
H3A	−0.103 (3)	0.6227 (18)	0.1970 (15)	0.052 (7)*
H3B	−0.146 (3)	0.5685 (18)	0.2692 (15)	0.055 (7)*
C4	0.3306 (2)	0.32080 (14)	0.11194 (11)	0.0385 (4)
C5	0.18310 (18)	0.30999 (12)	0.13924 (9)	0.0296 (3)
C6	0.0855 (2)	0.24118 (13)	0.10697 (10)	0.0326 (4)
H6	0.110 (2)	0.2024 (16)	0.0703 (13)	0.041 (6)*
C7	−0.05323 (19)	0.22946 (12)	0.12727 (10)	0.0321 (4)
C8	−0.1544 (2)	0.15130 (15)	0.08958 (13)	0.0458 (5)
F1	0.44293 (15)	0.33654 (17)	0.16988 (9)	0.0883 (7)
F2	0.32056 (19)	0.38848 (10)	0.06215 (10)	0.0685 (5)
F3	0.36733 (13)	0.24772 (9)	0.07569 (8)	0.0506 (3)
F4	−0.29354 (16)	0.17820 (13)	0.06380 (12)	0.0886 (6)
F5	−0.1579 (2)	0.08638 (10)	0.14070 (10)	0.0745 (5)
F6	−0.10749 (18)	0.11187 (11)	0.03201 (9)	0.0713 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02652 (19)	0.02759 (19)	0.0347 (2)	0.000	0.00968 (13)	0.000
N	0.0307 (8)	0.0358 (8)	0.0408 (8)	−0.0001 (6)	0.0029 (6)	0.0052 (7)
O1	0.0307 (6)	0.0354 (7)	0.0405 (7)	−0.0094 (5)	0.0124 (5)	−0.0062 (5)
O2	0.0304 (6)	0.0382 (7)	0.0523 (8)	−0.0089 (5)	0.0170 (6)	−0.0075 (6)
C1	0.0475 (12)	0.0481 (12)	0.0416 (10)	−0.0041 (10)	0.0056 (9)	0.0098 (10)
C2	0.0306 (10)	0.0643 (16)	0.0596 (14)	0.0039 (10)	−0.0005 (9)	0.0184 (12)

C3	0.0543 (13)	0.0330 (10)	0.0506 (11)	0.0110 (9)	0.0033 (10)	0.0031 (9)
C4	0.0324 (9)	0.0480 (11)	0.0376 (9)	−0.0103 (8)	0.0131 (7)	−0.0027 (8)
C5	0.0257 (8)	0.0340 (9)	0.0296 (7)	−0.0023 (6)	0.0069 (6)	0.0040 (6)
C6	0.0319 (9)	0.0330 (9)	0.0342 (8)	−0.0052 (7)	0.0103 (7)	−0.0029 (7)
C7	0.0301 (8)	0.0299 (9)	0.0361 (8)	−0.0058 (7)	0.0062 (7)	−0.0010 (7)
C8	0.0359 (10)	0.0423 (11)	0.0608 (13)	−0.0131 (8)	0.0141 (9)	−0.0128 (9)
F1	0.0308 (7)	0.177 (2)	0.0591 (8)	−0.0329 (9)	0.0135 (6)	−0.0386 (11)
F2	0.0807 (11)	0.0491 (8)	0.0935 (11)	−0.0033 (7)	0.0578 (9)	0.0185 (7)
F3	0.0410 (6)	0.0527 (7)	0.0656 (8)	0.0026 (5)	0.0281 (6)	−0.0020 (6)
F4	0.0349 (7)	0.0790 (12)	0.1387 (16)	−0.0105 (7)	−0.0106 (8)	−0.0363 (11)
F5	0.0950 (12)	0.0456 (8)	0.0904 (11)	−0.0338 (8)	0.0365 (9)	−0.0057 (8)
F6	0.0719 (10)	0.0759 (11)	0.0713 (9)	−0.0348 (8)	0.0269 (8)	−0.0407 (8)

Geometric parameters (Å, °)

Fe1—O1^{i}	2.0556 (12)	C2—H2C	0.93 (3)
Fe1—O1	2.0556 (12)	C3—C3^{i}	1.515 (5)
Fe1—O2	2.0937 (13)	C3—H3A	0.97 (3)
Fe1—O2^{i}	2.0937 (13)	C3—H3B	1.01 (3)
Fe1—N^{i}	2.2290 (16)	C4—F1	1.309 (2)
Fe1—N	2.2290 (16)	C4—F2	1.324 (2)
N—C1	1.477 (3)	C4—F3	1.335 (2)
N—C3	1.480 (3)	C4—C5	1.533 (2)
N—C2	1.484 (3)	C5—C6	1.390 (2)
O1—C5	1.257 (2)	C6—C7	1.399 (2)
O2—C7	1.244 (2)	C6—H6	0.93 (2)
C1—H1A	1.00 (3)	C7—C8	1.537 (3)
C1—H1B	0.92 (3)	C8—F4	1.314 (3)
C1—H1C	0.96 (3)	C8—F5	1.325 (3)
C2—H2A	1.06 (3)	C8—F6	1.325 (3)
C2—H2B	0.97 (3)		
O1^{i}—Fe1—O1	175.01 (7)	H2A—C2—H2B	111 (2)
O1^{i}—Fe1—O2	91.02 (5)	N—C2—H2C	106.7 (16)
O1—Fe1—O2	85.44 (5)	H2A—C2—H2C	117 (2)
O1^{i}—Fe1—O2^{i}	85.44 (5)	H2B—C2—H2C	107 (2)
O1—Fe1—O2^{i}	91.02 (5)	N—C3—C3^{i}	111.08 (16)
O2—Fe1—O2^{i}	89.98 (8)	N—C3—H3A	107.7 (15)
O1^{i}—Fe1—N^{i}	93.66 (6)	C3^{i}—C3—H3A	109.6 (16)
O1—Fe1—N^{i}	90.11 (5)	N—C3—H3B	107.9 (15)
O2—Fe1—N^{i}	173.97 (6)	C3^{i}—C3—H3B	109.5 (15)
O2^{i}—Fe1—N^{i}	94.18 (6)	H3A—C3—H3B	111 (2)
O1^{i}—Fe1—N	90.11 (5)	F1—C4—F2	108.83 (19)
O1—Fe1—N	93.66 (6)	F1—C4—F3	106.73 (19)
O2—Fe1—N	94.18 (6)	F2—C4—F3	105.90 (15)
O2^{i}—Fe1—N	173.97 (5)	F1—C4—C5	111.17 (15)
N^{i}—Fe1—N	82.02 (8)	F2—C4—C5	110.55 (17)

C1—N—C3	110.76 (17)	F3—C4—C5	113.41 (15)
C1—N—C2	107.55 (17)	O1—C5—C6	128.49 (16)
C3—N—C2	109.98 (18)	O1—C5—C4	113.27 (15)
C1—N—Fe1	111.96 (13)	C6—C5—C4	118.22 (16)
C3—N—Fe1	104.90 (11)	C5—C6—C7	121.22 (16)
C2—N—Fe1	111.71 (13)	C5—C6—H6	120.7 (14)
C5—O1—Fe1	127.93 (11)	C7—C6—H6	118.1 (14)
C7—O2—Fe1	127.60 (11)	O2—C7—C6	128.02 (16)
N—C1—H1A	110.6 (15)	O2—C7—C8	113.81 (15)
N—C1—H1B	110.6 (15)	C6—C7—C8	118.15 (16)
H1A—C1—H1B	106 (2)	F4—C8—F5	107.08 (19)
N—C1—H1C	109.1 (14)	F4—C8—F6	108.1 (2)
H1A—C1—H1C	108.0 (19)	F5—C8—F6	105.8 (2)
H1B—C1—H1C	113 (2)	F4—C8—C7	111.72 (18)
N—C2—H2A	106.4 (16)	F5—C8—C7	110.06 (18)
N—C2—H2B	108.9 (17)	F6—C8—C7	113.72 (16)

Symmetry codes: (i) $-x, y, -z+1/2$.