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$[Fe(thf)_6][Cl_3Fe–O–FeCl_3]$

Michael Bolte,[a]* Hans-Wolfram Lerner[b] and Stefan Scholz[b]

[a]Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and [b]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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The title compound, hexakis(tetrahydrofuran)iron(II) μ-oxo-bis[trichloroferrate(III)], $[Fe(C_4H_8O)_6][Fe_2Cl_6O]$, was obtained by oxidation of $FeCl_2$ in tetrahydrofuran. The O atom of the anion and the Fe atom of the cation are located on special positions of site symmetry $\bar{3}$; the Fe atoms of the anion are located on a threefold rotation axis and, as a result, there is just $\frac{1}{6}$ of both ions in the asymmetric unit.

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.009 Å
R factor = 0.056
wR factor = 0.133
Data-to-parameter ratio = 22.7

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

Comment

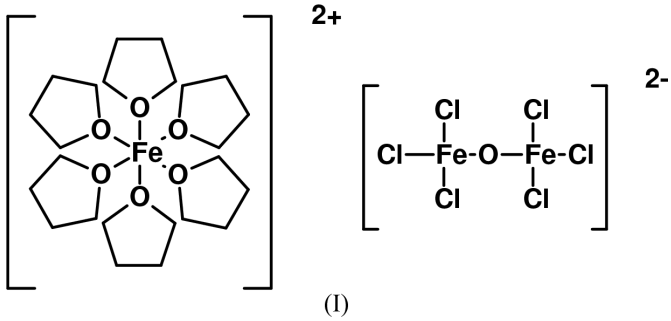
In contrast to established ferrocene, only a few compounds with two σ bonds, R—Fe—R, are known. Therefore, we became interested in the synthesis of the supersilylated compound ^{t}Bu$_3$Si—Fe—Si^{t}Bu$_3$, which we obtained from sodium supersilanide, ^{t}Bu$_3$SiNa, and iron(II) chloride, $FeCl_2$, in tetrahydrofuran at 195 K.

$$FeCl_2 + 2\,^t\mathrm{Bu_3SiNa} \rightarrow 2\,^t\mathrm{Bu_3Si{-}Fe{-}Si}\,^t\mathrm{Bu_3} + 2\mathrm{NaCl}$$

In this context, we have prepared a calibrated solution (0.5 M) of $FeCl_2$ in tetrahydrofuran. After several investigations, the Fe^{2+} in this calibrated solution was partially oxidized.

$$3FeCl_2 + 0.5O_2 \rightarrow [Fe(thf)_6]^{2+}[Cl_3Fe{-}O{-}FeCl_3]^{2-}$$

While in biological systems oxidation with oxygen from Fe^{2+} to Fe^{3+} is important, the molecular structure of the title compound, (I), is of general interest.



(I)

The O atom of the anion and the Fe atom of the cation are located on special positions of site symmetry $\bar{3}$; the Fe atoms of the anion are located on a threefold rotation axis and, as a result, there is just $\frac{1}{6}$ of both ions in the asymmetric unit. The Fe—O—Fe system in the anion is linear for symmetry reasons.

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Experimental

A solution of 1.072 g (8.51 mmol) $FeCl_2$ in 17 ml tetrahydrofuran was oxidized with dry air. Red cubic crystals of the title compound were obtained from this solution at 248 K.

Crystal data

$[Fe(C_4H_8O)_6][Fe_2Cl_6O]$
M_r = 828.87
Cubic, $Pa\bar{3}$
a = 15.244 (2) Å
V = 3542.4 (8) Å^3
Z = 4
D_x = 1.554 Mg m^{-3}
Mo $K\alpha$ radiation

Cell parameters from 502 reflections
θ = 2.7–20.1°
μ = 1.70 mm^{-1}
T = 173 (2) K
Plate, red
0.28 × 0.28 × 0.12 mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
T_{min} = 0.647, T_{max} = 0.822
36 645 measured reflections
1405 independent reflections
792 reflections with $I > 2\sigma(I)$

R_{int} = 0.068
θ_{max} = 28.2°
$h = -19 \rightarrow 19$
$k = -20 \rightarrow 17$
$l = -19 \rightarrow 20$
123 standard reflections
frequency: 1200 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.133
S = 1.03
1405 reflections
62 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 11.6567P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	1.7510 (12)	Fe2—O2	2.171 (3)
Fe1—Cl1	2.2273 (14)		
O1—Fe1—Cl1	110.54 (5)	O2—Fe2—O2^{i}	90.44 (11)

Symmetry code: (i) $\frac{3}{2} - z, 1 - x, \frac{1}{2} + y$.

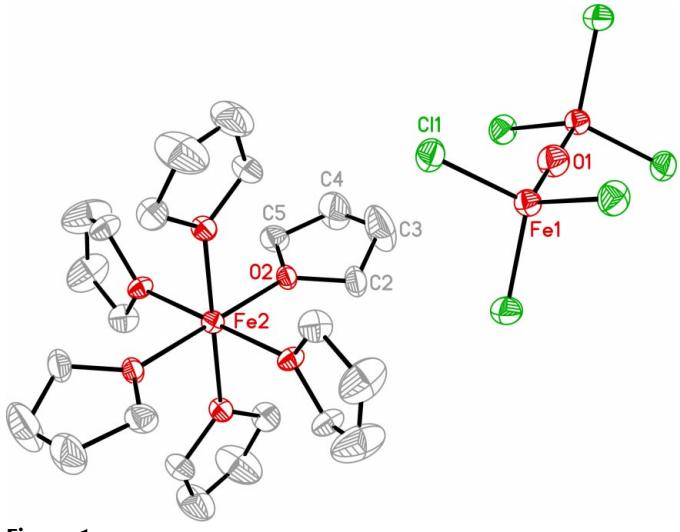

Figure 1
A perspective view of the title compound with the atom-numbering scheme and ellipsoids at the 50% probability level.

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [U(H) = 1.2U_{eq}(C)] using a riding model with C—H = 0.99 Å.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

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