

Synthesis and characterization of new oligosilane derivatives of iron and molybdenum

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Abstract

By use of salt elimination, the transition metal substituted oligosilanes $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **1**, $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Br}$ **2**, $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2(\text{SiMe}_2)_6(\text{CO})_2\text{Fe}(\eta^5\text{-C}_5\text{Me}_4\text{Et})$ **3** and $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2(\text{SiMe}_2)_6\text{Br}$ **4** were prepared and characterized. Compound **1** is well crystallized from pentane and its structure has been determined by X-ray diffraction analysis. © 1997 Elsevier Science S.A.

Keywords: Iron; Molybdenum; Oligosilanes; Transition metal silicon compounds

1. Introduction

Within the last few years transition metal silicon complexes with silane ligands have been the focus of much attention [1]. The chemistry of transition metal silylene complexes is researched very intensively [2]. Furthermore, triphenylsilyl and tris(trimethylsilyl)silyl ligands are applied to stabilize transition metal compounds due to their steric properties [3] (also, see for instance Ref. [4]).

Transition metal silicon compounds are useful as precursors in metal silicide CVD processes [5], they are involved in a number of syntheses, for instance in hydrosilylation reactions [6], and in σ -bond metathesis processes [7] like the catalytic dehydrocoupling of silanes [8]. Organosilicon polymers in which silicon atoms are joined with transition metals represent an area of considerable current interest, since unusual physical and chemical properties are to be expected of the resultant materials [9]. The chemistry of transition metal oligosilanes, i.e. one transition metal centre bonded to a chain with at least two silicon atoms, is not very well explored. There are a number of compounds of this type derived from $\text{CpFe}(\text{CO})_2^-$ (Fp) [10]. The Fp-fragment seems to be an ideal substrate to join with oligosilanes,

but there exist only a few examples of transition metal oligosilanes with transition metal centres other than iron [11,12]. In 1974, Malisch reported about the difficulties with the production of Group VIB-metal silicon compounds [12]. These transition metal substituted disilanes are in fact very sensitive to light, heat and air. We are presently re-investigating this area of chemistry and our aim was to prepare more-stable and well-crystallized compounds. For this reason we selected ligand systems other than cyclopentadiene, like the steric pretentious ethyltetramethylcyclopentadiene, which should enable us to synthesize transition metal substituted disilanes with improved stability. A conceptually simple way in which an M–Si bond may be formed is the reaction between a metallate complex and a silicon halide with elimination of one equivalent of a salt [13].

Halogeno-oligosilane complexes of the type $\text{L}_n\text{M}(\text{SiMe}_2)_m\text{X}$ (X = Cl, Br) are interesting as synthons for metal-containing polysilanes. For that purpose reactive halide groups are highly recommended.

2. Results and discussion

2.1. Synthesis of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **1**

The reaction of one equivalent of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ with $\text{ClMe}_2\text{SiSiMe}_2\text{Cl}$ in THF–

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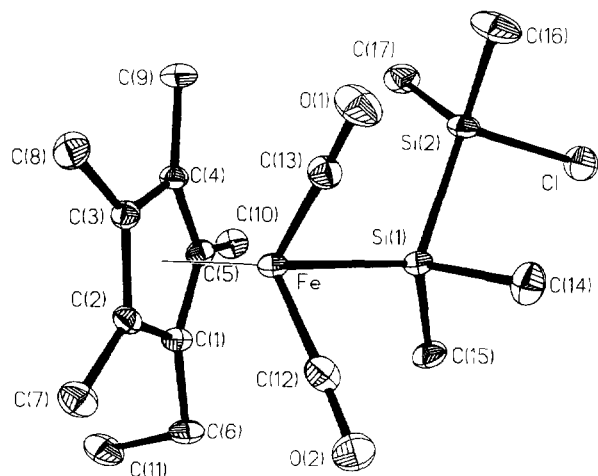
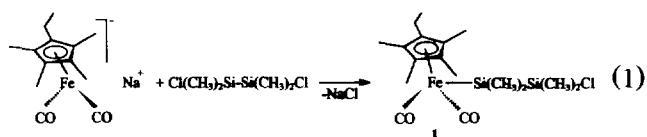


Fig. 1. Molecular structure of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **1**; hydrogen atoms omitted; measurement at -150°C .

pentane at ambient temperature results in $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ (**1**, Eq. (1)).



Recrystallization from pentane at -20°C gave analytically pure orange-yellow crystals of **1** suitable for an X-ray crystallographic study (Fig. 1). The infrared spectrum of **1** shows two carbonyl stretching modes at 1908 and 1963 cm^{-1} as expected for compounds of the type $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{L}$.

The ^1H NMR signals of the ethyltetramethylcyclopentadienyl ligand are observed at $\delta = 1.87$ ppm (s, methyl) and at $\delta = 1.02$ ppm (t) and 2.30 ppm (q) with coupling of the signals in the ethyl group. The ^1H NMR resonances for the SiMe_2 groups appear in a typical range at δ values near zero ($\delta = 0.43$ and 0.49 ppm). The ^{29}Si NMR spectrum of compound **1** exhibits two resonances for the disilyl ligand bonded to the metal atom (see Table 1). The ^{13}C NMR data of **1** are in agreement with the proposed structure (Table 1) [13].

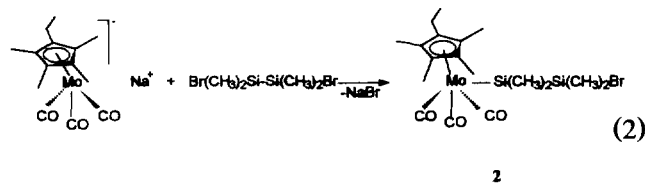
2.1.1. Molecular structure of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ **1**

The X-ray structure analysis of **1** reveals a distorted tetrahedral geometry around the iron atom with smaller angles between the silyl residue and the CO groups (82.7° and 85.4°) and rather large angles between the bulky $\text{C}_5\text{Me}_4\text{Et}$ group and the other substituents (see Fig. 1 and Table 2). The $\text{Fe-SiMe}_2\text{-SiMe}_2\text{-Cl}$ unit has a staggered conformation with the iron and the chlorine atoms at trans position to each other (torsion angle 179°). The Fe-Si bond length of 2.329 \AA is in good accordance with published data for $(\eta^5\text{-$

$\text{C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{-silyl}$ compounds (bond length between 2.348 and 2.365 \AA) [14]. The $\text{C}_5\text{Me}_4\text{Et}$ ligand occupies a definite conformation; no disorder was observed. Obviously the substituents (Me, Et) give rise to better complex crystallization in comparison to cyclopentadiene.

2.2. Synthesis of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Br}$ **2**

The treatment of a solution of $\text{BrMe}_2\text{SiSiMe}_2\text{Br}$ in pentane with a solution of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3]$ in THF yields $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Br}$ (**2**, Eq. (2)).



The recrystallization from pentane gives nice yellow crystals. Structure of **2** is clearly established by spectroscopic data (see Table 1). The substituted cyclopentadienyl ring is indicated by the ^1H NMR spectrum which exhibits a set of two singlets at 2.0 ppm (methyl groups), one triplet at 1.02 ppm and one quartet at 2.41 ppm for the ethyl group. The resonances of the silicon-bonded methyl groups are found at $\delta = 0.621$ and 0.622 ppm. The assigned structure was proved by the ^{13}C NMR spectrum (Table 1). The ^{29}Si NMR spectrum exhibits two resonances for the disilyl group with the metal-bonded silicon atom resonance at low field (28.6 ppm) due to the influence of the transition metal. The IR spectrum shows three absorptions of $\nu(\text{CO})$. This behaviour is typical for metal carbonylates of the type $\text{cis-L}_2\text{M}(\text{CO})_3$. The ^{29}Si NMR data show that the silicon-attached halogen effects a downfield shift of the

Table 1
NMR data of the new disilane derivatives of iron and molybdenum (in ppm relative to TMS at 0.0 ppm, recorded in CDCl_3)

Structure unit	δ (ppm)	
	1	2
$^{13}\text{C}=\text{O}$	216.8	213.8, 216.8
$^{13}\text{CH}_3\text{-Si}$	2.0, 3.9	1.3, 3.9
$^{13}\text{CH}_3\text{-CH}_2$	18.4	18.5
$^{13}\text{CH}_2\text{-CH}_3$	14.5	14.5
$^{13}\text{C-CH}_3$	94.6, 95.5	94.7, 95.5
$^{13}\text{C-CH}_2\text{-}$	100.5	100.5
$^{13}\text{CH}_3\text{-C}$	9.8, 10.1	9.9, 10.1
$^{29}\text{Si-M}$	30.8	28.6
$^{29}\text{Si-Si-M}$	15.1	4.1
$^1\text{H}(\text{Si-Me})$	0.43 (s), 0.49 (s)	0.621 (s), 0.622 (s)
$^1\text{H}(\text{C-Me})$	1.86 (s,s)	2.0 (s,s)
$^1\text{H}(\text{C-Et})$	1.02 (t), 2.30 (q)	1.02 (t), 2.41 (q)

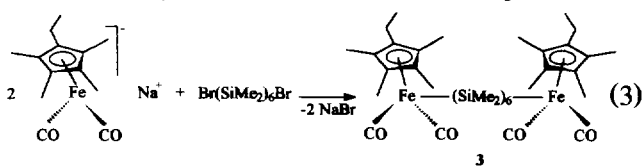
Table 2
Selected bond lengths (Å) and bond angles (deg) for $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$

Fe–MP(C ₅ Me ₄ Et)	1.726	Si(1)–Si(2)	2.363
Fe–C(12)	1.739	Fe–C(13)	1.755
Fe–Si(1)	2.329	Si(2)–Cl	2.112
Si(1)–C(14)	1.902	Si(2)–C(16)	1.860
Si(1)–C(15)	1.891	Si(2)–C(17)	1.866
Si(1)–Fe–MP(C ₅ Me ₄ Et)	128.5	Fe–Si(1)–Si(2)	118.78
Cl–Si(2)–Si(1)	103.5	C(12)–Fe–Si(1)	82.70
C(12)–Fe–C(13)	98.30	C(13)–Fe–Si(1)	85.37
Fe–Si(1)–Si(2)–Cl	179.03	MP(C ₅ Me ₄ Et)–Fe–Si(1)–Si(2)	72.9

silicon NMR signal. Results agree with values given in previous papers [15]. Compound 2 decomposes thermally above 150 °C and is stable against light. For this reason 2 is more stable than the analogous compound with the unsubstituted cyclopentadienyl ligand [12].

2.3. Reaction of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ with $\text{Br}(\text{SiMe}_2)_6\text{Br}$

Stimulated by the successful X-ray structure analysis of 1 the same route was applied on the synthesis of a compound with a longer silicon chain. A solution of $\text{Br}(\text{SiMe}_2)_6\text{Br}$ in pentane was added dropwise to a THF solution of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ at –78 °C. The solvent was removed, the orange residue was extracted with pentane, and the mixture was filtered. Recrystallization from pentane gave compound 3 (Eq. (3)).



The compound 3 was identified by ¹H-, ¹³C-, ²⁹Si-NMR and IR spectra (Table 3). The IR spectrum of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2(\text{SiMe}_2)_6(\text{CO})_2\text{Fe}(\eta^5\text{-C}_5\text{Me}_4\text{Et})$ exhibits two bands of $\nu(\text{CO})$ at 1924 and 1979 cm^{-1} according to A' and A'' modes for local C_s symmetry at iron. The formation of the diiron complex 3 is highly favoured in relation to that of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2(\text{SiMe}_2)_6\text{Br}$ 4. We were not successful in synthesizing $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2(\text{SiMe}_2)_6\text{Br}$ 4 in the same way as 1 and 2 by salt elimination. We tried to gain 4 in different solvents and time intervals but, unfortunately, the diiron complex 3 and $\text{Br}(\text{SiMe}_2)_6\text{Br}$ were recovered.

Using an excess of $\text{Br}(\text{SiMe}_2)_6\text{Br}$ in comparison to $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ we were able to obtain a mixture of 3 and 4. Table 3 shows the ¹³C- and ²⁹Si-NMR signals of 4. We were not able to separate 4 from 3 by recrystallization or column chromatography.

3. Experimental section

All reactions were performed in an atmosphere of Ar and in dry, oxygen-free solvents. Syntheses of

Table 3
NMR spectral properties of the new hexasilane derivatives of iron (data in ppm are relative to TMS at 0.0 ppm, recorded in CDCl₃)

Structure unit	δ (ppm)	
	3	4
¹³ C=O	217.7	214.0
¹³ CH ₃ –Si	–3.4, –2.7, 4.3	3.45, –4.08, –4.44, –5.5, –6.15, –6.29
¹³ CH ₃ –CH ₂	18.6	18.2
¹³ CH ₂ –CH ₃	14.6	14.05
¹³ C–CH ₃	94.2, 95.1	94.4, 95.4
¹³ C–CH ₂ –	100.1	101.0
¹³ CH ₃ –C	9.9, 10.1	9.1, 9.6
²⁹ Si–M	20.8	24.5
²⁹ Si–Si–M	–31.7	
²⁹ Si–Si–Si–M	–36.4	–39.17, –39.86, –47.11
²⁹ Si–Si–Si–Si–M		
²⁹ Si–Br		8.27
²⁹ Si–Si–Br		23.38
¹ H(Si–Me)	0.18(s), 0.20(s), 0.43(s)	0.13 (s), 0.22 (s), 0.23 (s), 0.24 (s), 0.65 (s), 0.66 (s)
¹ H(C–Me)	1.82 (s), 1.83 (s)	1.71 (s), 1.60 (s)
¹ H(C–Et)	1.00(t), 2.25 (q)	0.87 (t), 2.18 (q)

$\text{ClMe}_2\text{SiSiMe}_2\text{Cl}$ [16], $\text{BrMe}_2\text{SiSiMe}_2\text{Br}$ [17], $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ [18], $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3]$ [19] and $\text{Br}(\text{SiMe}_2)_6\text{Br}$ [13] were carried out according to known procedures.

NMR spectra were recorded on Bruker MSL 200. IR spectra were measured from 4000 to 400 cm^{-1} on a Specord 75 IR instrument.

3.1. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$ 1

A solution of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ was prepared from 50 ml THF, 3 mmol (1.56 g) of bis-(1-ethyl-2,3,4,5-tetramethylcyclopentadienyl-dicarbonyl-iron) and Na–Hg [19]. This solution was added to a mixture of 6 mmol (1.12 g) of $\text{ClMe}_2\text{SiSiMe}_2\text{Cl}$ and 50 ml pentane. After stirring for 4 h at room temperature all the solvents were immediately removed in vacuo and the residue was extracted five times with 10 ml portions of pentane. The combined extracts were immediately concentrated and after cooling to 4°C 1 crystallized as an orange-yellow solid. Yield: 1.8 g (74%), m.p.: 64°C ; anal. found: C, 48.07; H, 6.85. $\text{C}_{17}\text{H}_{29}\text{ClFeO}_2\text{Si}_2$ (412) calc.: C, 49.51; H, 7.03.

3.2. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3\text{SiMe}_2\text{SiMe}_2\text{Br}$ 2

The mixture of 50 ml pentane and 6 mmol of $\text{BrMe}_2\text{SiSiMe}_2\text{Br}$ (1.65 g) was added dropwise to a tetrahydrofuran solution containing 6 mmol of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Mo}(\text{CO})_3]$ (2.1 g). The solution was stirred at room temperature for 1 h. The solvent was removed under reduced pressure and the brown residue was extracted three times with 25 ml portions of pentane. The solvent was reduced to 20 ml and the dark orange-coloured solution was cooled to -15°C . The product 2 crystallized as a yellow solid. Yield: 1.9 g (63%), m.p.: 106°C ; anal. found: C, 39.86; H, 5.72. $\text{C}_{18}\text{H}_{29}\text{BrMoO}_3\text{Si}_2$ (525) calc.: C, 41.14; H, 5.52.

3.3. Preparation of $(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2(\text{SiMe}_2)_6(\text{CO})_2\text{Fe}(\eta^5\text{-C}_5\text{Me}_4\text{Et})$ 3

4 mmol (2.03 g) of $\text{Si}_6\text{Me}_{12}\text{Br}_2$ was dissolved in 100 ml pentane. A solution of $\text{Na}[(\eta^5\text{-C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2]$ was prepared from 50 ml THF; 2 mmol (1.04 g) of bis-(1-ethyl-2,3,4,5-tetramethylcyclopentadienyl-dicarbonyl-iron) [19] was added dropwise. After stirring for 1 day the solvents were evaporated to dryness under vacuum. The orange residue was extracted with 50 ml of pentane. The resulting orange solution was cooled to -20°C . After 1 week the compound 3 was crystallized. Yield: 2.8 g (80.5%); anal. found: C, 50.32; H, 7.91 $\text{C}_{38}\text{H}_{70}\text{Fe}_2\text{O}_4\text{Si}_6$ (870) calc.: C, 52.41; H, 8.04.

Table 4
Crystal data and structure refinement for $(\text{C}_5\text{Me}_4\text{Et})\text{Fe}(\text{CO})_2\text{SiMe}_2\text{SiMe}_2\text{Cl}$

Empirical formula	$\text{C}_{17}\text{H}_{29}\text{ClFeO}_2\text{Si}_2$
Formula weight	412.88
Temperature (K)	293(2)
Wavelength (\AA)	1.54180
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	
<i>a</i> (\AA)	8.4728(7)
<i>b</i> (\AA)	8.8670(14)
<i>c</i> (\AA)	14.640(3)
α (deg)	99.963(14)
β (deg)	102.578(10)
γ (deg)	96.288(11)
Volume (\AA^3)	1044.8(3)
<i>Z</i>	2
Density (calculated) (Mg m^{-3})	1.312
Absorption coefficient (mm^{-1})	8.104
<i>F</i> (000)	436
Crystal size (mm^3)	$0.15 \times 0.12 \times 0.1$
θ range for data collection (deg)	3.16 to 74.81
Index ranges	$-10 \leq h \leq 10$, $-10 \leq k \leq 11$, $-18 \leq l \leq 18$
Reflections collected	8301
Independent reflections	4295 [$R(\text{int}) = 0.0630$]
Refinement method	Full matrix least squares on F^2
Data/restraints/parameters	4295/0/217
Goodness-of-fit on F^2	1.056
Final <i>R</i> indices [$I > 2\sigma(I)$]	$R1 = 0.0454$, $wR2 = 0.1139$
<i>R</i> indices (all data)	$R1 = 0.0525$, $wR2 = 0.1241$
Largest diff. peak and hole ($\text{e}^- \text{\AA}^{-3}$)	0.876 and -1.009

3.4. Crystal structure analysis of 1

An orange-yellow crystal of approximate dimensions $0.15 \times 0.12 \times 0.1\text{ mm}^3$ was immersed in Nujol under argon, then mounted on a glass fibre in a random orientation. Preliminary examination and data collection were performed using a CAD 4 single crystal X-ray diffractometer with graphite monochromated $\text{Cu K}\alpha$ radiation ($\lambda = 1.5418\text{ \AA}$) at 123(5) K. Final cell constants and the orientation matrix for data collection were calculated by least squares refinement of 24 reflections with $6.8 < \theta < 54^\circ$. Intensity data were collected using ω - 2θ scans. Crystal data and intensity data collection parameters are listed in Table 4. Data reduction was carried out with MOLEN (Enraf-Nonius), structure solution with SHELXS-86 [20], refinement with SHELXL-93 [21]. Empirical absorption correction was applied to the data with min/max correction factors of 0.632/0.99. The structure was solved by direct methods and refined in the triclinic space group $P\bar{1}$. A check for higher symmetries by MISSYM [22] proved the absence of any additional symmetry elements. Full matrix least squares refinement was carried out. The non-hydrogen atoms

Table 5

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1

	x	y	z	U_{eq}^a
Fe	4240(1)	6037(1)	1806(1)	14(1)
Cl	2315(1)	1531(1)	3864(1)	30(1)
Si(1)	4190(1)	4119(1)	2708(1)	16(1)
Si(2)	1709(1)	3258(1)	3077(1)	19(1)
O(1)	1777(3)	3880(3)	329(1)	32(1)
O(2)	7160(3)	4870(3)	1447(2)	36(1)
C(1)	5545(3)	8136(3)	2668(2)	17(1)
C(2)	5147(3)	8281(3)	1679(2)	16(1)
C(3)	3426(3)	8004(3)	1343(2)	16(1)
C(4)	2736(3)	7698(3)	2120(2)	16(1)
C(5)	4038(3)	7819(3)	2941(2)	16(1)
C(6)	7224(3)	8481(4)	3333(2)	23(1)
C(7)	6353(3)	8715(4)	1116(2)	25(1)
C(8)	2466(3)	8114(4)	374(2)	23(1)
C(9)	945(3)	7489(4)	2092(2)	23(1)
C(10)	3858(3)	7824(4)	3937(2)	21(1)
C(11)	7655(4)	10212(4)	3798(2)	30(1)
C(12)	5970(3)	5301(4)	1582(2)	23(1)
C(13)	2757(3)	4714(3)	919(2)	20(1)
C(14)	4517(4)	2199(4)	2021(2)	29(1)
C(15)	5870(3)	4489(4)	3843(2)	23(1)
C(16)	145(4)	2239(5)	1980(2)	35(1)
C(17)	669(3)	4504(4)	3847(2)	28(1)

^a U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

were refined anisotropically, all hydrogen atoms were geometrically positioned and refined isotropically. The final residual values and structure refinement parameters

Table 6

Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1^a

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Fe	11(1)	18(1)	12(1)	2(1)	2(1)	2(1)
Cl	29(1)	32(1)	30(1)	16(1)	4(1)	3(1)
Si(1)	12(1)	20(1)	16(1)	4(1)	2(1)	3(1)
Si(2)	13(1)	26(1)	17(1)	6(1)	0(1)	-1(1)
O(1)	30(1)	34(1)	23(1)	0(1)	-3(1)	-8(1)
O(2)	30(1)	34(1)	54(1)	10(1)	25(1)	12(1)
C(1)	12(1)	20(1)	19(1)	2(1)	3(1)	2(1)
C(2)	14(1)	14(1)	18(1)	1(1)	4(1)	-1(1)
C(3)	15(1)	15(1)	16(1)	2(1)	0(1)	2(1)
C(4)	11(1)	16(1)	18(1)	1(1)	1(1)	1(1)
C(5)	15(1)	18(1)	15(1)	1(1)	4(1)	4(1)
C(6)	12(1)	28(2)	23(1)	2(1)	-1(1)	0(1)
C(7)	22(1)	28(2)	26(1)	6(1)	12(1)	2(1)
C(8)	24(1)	26(2)	17(1)	6(1)	0(1)	3(1)
C(9)	12(1)	31(2)	25(1)	5(1)	3(1)	2(1)
C(10)	22(1)	27(2)	16(1)	4(1)	4(1)	5(1)
C(11)	23(1)	35(2)	26(1)	2(1)	-1(1)	-8(1)
C(12)	23(1)	21(2)	24(1)	2(1)	8(1)	-1(1)
C(13)	20(1)	20(1)	17(1)	2(1)	2(1)	1(1)
C(14)	36(2)	26(2)	30(2)	7(1)	12(1)	10(1)
C(15)	17(1)	32(2)	22(1)	11(1)	1(1)	7(1)
C(16)	23(1)	52(2)	21(1)	7(1)	-4(1)	-11(2)
C(17)	19(1)	36(2)	32(2)	10(1)	13(1)	5(1)

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^*U_{11} + \dots + 2hka^*b^*U_{12}]$.

ters are listed in Table 4. The selected bond distances and angles, the atomic coordinates for the non-hydrogen atoms, and the anisotropic displacement parameters are listed in Tables 2, 5 and 6 respectively. ¹ A view of the molecule with non-hydrogen atoms represented by 50% probability ellipsoids is presented in Fig. 1.

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¹ Further details of the X-ray determination may be obtained from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Germany. Requests should contain the deposition number CSD-405000, names of authors, and citation of this article.

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