Experimental

The synthesis of (I) was carried out by the reaction of (NEt_4)-$[Ni^{II}(SPh)_4]$ with $[S\text{-}o\text{-}(CF_3CONH)C_6H_4]_2$ in the presence of trimethylamine *N*-oxide in acetonitrile at room temperature (molar ratio 1:2:2). Dark-brown crystals were obtained from the solution.

Crystal data

$(C_8H_{20}N)_2[Ni(C_8H_4F_3NOS)_2]$
$M_r = 757.57$
Monoclinic
$P2_1/c$
$a = 11.951\ (4)$ Å
$b = 10.590\ (4)$ Å
$c = 28.852\ (2)$ Å
$\beta = 91.57\ (1)°$
$V = 3650\ (1)$ Å^3
$Z = 4$
$D_x = 1.378$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 14.4$–$14.9°$
$\mu = 0.711$ mm^{-1}
$T = 296.2$ K
Prismatic
$0.6 \times 0.5 \times 0.5$ mm
Dark brown

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
9268 measured reflections
8854 independent reflections
4485 reflections with $I > 3\sigma(I)$

$R_{int} = 0.036$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 13$
$l = -37 \rightarrow 37$
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.045$
$wR = 0.047$
$S = 1.828$
4485 reflections
425 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.00010|F_o|^2]$

$(\Delta/\sigma)_{max} = 0.078$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ni1—S1	2.177 (1)	S1—C11	1.762 (4)
Ni1—S2	2.171 (1)	S2—C21	1.749 (4)
Ni1—N1	1.954 (3)	N1—C12	1.434 (4)
Ni1—N2	1.959 (3)	N2—C22	1.428 (5)
S1—Ni1—S2	87.84 (5)	Ni1—S1—C11	95.6 (1)
S1—Ni1—N1	86.25 (9)	Ni1—S2—C21	96.0 (1)
S1—Ni1—N2	169.04 (10)	Ni1—N1—C12	111.7 (2)
S2—Ni1—N1	169.09 (9)	Ni1—N1—C17	131.2 (3)
S2—Ni1—N2	87.13 (9)	Ni1—N2—C22	112.3 (2)
N1—Ni1—N2	100.2 (1)	Ni1—N2—C27	130.7 (3)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1048). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1425–1427

1,1,3,3,14,14,16,16,18,18,29,29-Dodecamethyl-3,14,18,29-tetrasila-1,16-distanna-[5.5]ferrocenophane

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(Received 16 December 1997; accepted 27 April 1998)

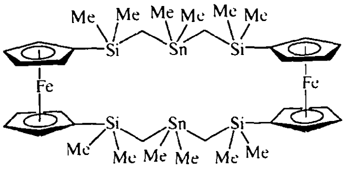
Abstract

The title compound, $[Fe_2\{Sn(C_9H_{15}Si)_2\}_2]$, is a new ferrocenophane containing two $(Me_2SiCH_2)_2SnMe_2$ bridges. The molecule lies about an inversion center. The intramolecular Si···Si distances are 7.827 (2) and 8.403 (2) Å, and the Sn···Sn intramolecular separation is 9.102 (1) Å.

Comment

In recent years, the host–guest chemistry of anion recognition has become a topic of increasing interest (Pierre & Baret, 1983; Dietrich, 1993; Schmidtchen & Berger, 1997). For some time we have been interested in the synthesis of tin- and silicon-containing multidentate Lewis acids for anion complexation (Altmann *et al.*, 1997). Some of our compounds were shown to function as carriers in phosphate-sensitive electrodes (Chaniotakis *et al.*, 1993; Tsagatakis *et al.*, 1994). With respect to their potential in such electrochemical applications, it was of interest to synthesize redox-active ferrocene-containing organotin and/or organosilicon derivatives. Although there are reports on organosilicon- as well as organotin-containing ferrocene derivatives (Herberhold *et al.*, 1996; Rulkens *et al.*, 1996; Zechel *et al.*, 1995; Park *et al.*, 1995; Foucher *et al.*, 1995; Finckh *et al.*, 1992; Clearfield *et al.*, 1983; Dong *et al.*, 1990; Yamin *et al.*, 1994), to the best of our knowledge, ferrocenophanes containing both Si and Sn as bridging atoms have not been reported so far.

The title compound, (1), was obtained in good yield by reaction of equimolar quantities of [$Fe(C_5H_4SiMe_2CH_2MgCl)_2$] and [$Fe(C_5H_4SiMe_2CH_2SnMe_2Cl)_2$] in tetrahydrofuran (THF). Compound (1) consists of two ferrocene units which are linked by two $Me_2SiCH_2SnMe_2CH_2SiMe_2$ bridges resulting in a centrosymmetric ferrocenophane-type molecule. The cyclopentadiene rings are staggered as shown by the torsion angle C12—*Cnt*1—*Cnt*2—C15 of 15.8 (1)° (*Cnt*1 is the centroid of the C10–C14 ring and *Cnt*2 is the centroid of the C15–C19 ring). The torsion angle Si2—*Cnt*1—*Cnt*2—Si1^i amounts to 88.3 (1)° [symmetry code: (i) $1-x$, $-y$, $-z$]. The cyclopentadiene rings within one ferrocene unit are slightly tilted by 1.9 (1)°. The Si1 and Si1^i atoms are mutually *trans*. The Si—C, Sn—C and Fe—C bond lengths, as well as the bond angles at silicon and tin, are as expected and do not show any particularities. The intramolecular Si1—Si1^i, Si2—Si2^i and Sn1—Sn1^i distances amount to 8.403 (2), 7.827 (2) and 9.102 (1) Å, respectively.



(1)

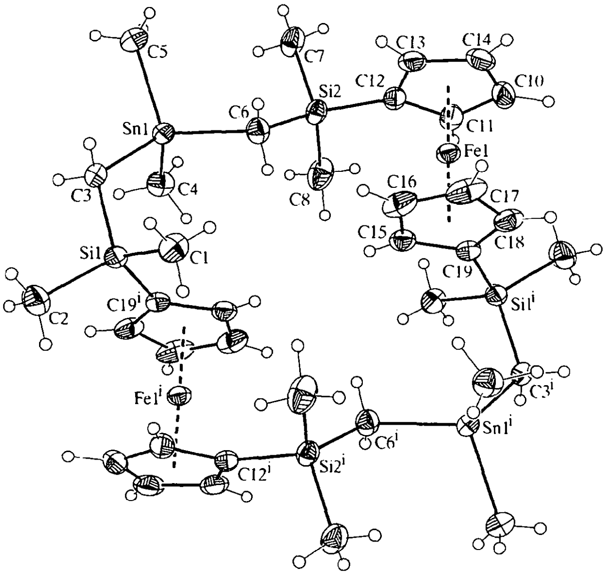


Fig. 1. View of the title compound showing the labeling of all non-H atoms. Displacement ellipsoids are shown at 50% probability levels. [Symmetry code: (i) $1-x$, $-y$, $-z$.]

Experimental

The Grignard reagent [$Fe(C_5H_4SiMe_2CH_2MgCl)_2$] was prepared from [$Fe(C_5H_4SiMe_2CH_2Cl)_2$] (1.99 g, 5 mmol) and excess magnesium (0.5 g, 20 mmol) in THF (10 ml). This reagent and a solution of [$Fe(C_5H_4SiMe_2CH_2SnMe_2Cl)_2$] (3.48 g, 5 mmol) in THF (20 ml) were added dropwise and synchronously to magnetically stirred THF (150 ml). The mixture was refluxed for 12 h and the solvent removed *in vacuo*. Dichloromethane (50 ml) was added and the mixture stirred for 5 min followed by filtration from $MgCl_2$. After size-exclusion chromatography of the filtrate using Sephadex, the title compound was obtained in 60% yield (2.86 g) as an orange oil. Single crystals of m.p. 406–409 K were obtained by slow evaporation from hexane. Spectroscopic details and elemental analyses will be provided in a forthcoming paper.

Crystal data

[$Fe_2Sn_2(C_9H_{15}Si)_4$]
$M_r = 954.28$
Monoclinic
$P2_1/c$
$a = 13.180$ (2) Å
$b = 13.555$ (3) Å
$c = 13.267$ (2) Å
$\beta = 118.23$ (2)°
$V = 2088.3$ (6) Å^3
$Z = 2$
$D_x = 1.518$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12.28$–14.56°
$\mu = 2.001$ mm^{-1}
$T = 200$ (1) K
Rectangular block
0.20 × 0.15 × 0.13 mm
Orange

Data collection

Nonius MACH3 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*CORINC*; Dräger & Gattow, 1971)
$T_{min} = 0.659$, $T_{max} = 0.771$
5857 measured reflections
4786 independent reflections

3961 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 27.47$°
$h = -1 \rightarrow 17$
$k = -1 \rightarrow 17$
$l = -17 \rightarrow 15$
3 standard reflections frequency: 60 min intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.054$
$S = 1.054$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.384$ e Å^{-3}
$\Delta\rho_{min} = -0.357$ e Å^{-3}
Extinction correction: none

4786 reflections
207 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0195P)^2 + 0.8546P]$
where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Si1—C19^{i}	1.866 (2)	Sn1—C6	2.161 (2)
Si1—C3	1.869 (2)	Si2—C6	1.865 (2)
Sn1—C3	2.161 (2)	Si2—C12	1.865 (2)
C19^{i}—Si1—C3	108.59 (9)	Sn1—C6—Si2	115.6 (1)
Si1—C3—Sn1	118.0 (1)	C6 Si2—C12	111.5 (1)
C3—Sn1—C6	111.50 (8)		

Symmetry code: (i) $1 - x, -y, -z$.

All H atoms were located in the difference Fourier map, but were placed in calculated positions using a riding model and refined with common isotropic displacement parameters for different C—H types [methylene C—H 0.97 Å and U_{iso} 0.058 (2) Å^2; methyl C—H 0.96 Å and U_{iso} 0.058 (2) Å^2; aromatic C—H 0.93 Å and U_{iso} 0.043 (3) Å^2].

Data collection: Nonius MACH3 Argus software. Cell refinement: Nonius MACH3 Argus software. Data reduction: *CORINC* (Dräger & Gattow, 1971). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93.

We thank the Human Capital & Mobility (HCM) Programme (Contract ERBCHRX-CT94-0610) of the European Union, the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1430). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1427–1431

An Adduct Between Tetraammine(1,10-phenanthroline)ruthenium(II) and Dibenzo-42-crown-14

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(*Received 18 December 1997; accepted 15 April 1998*)

Abstract

The crystal structure of the adduct dibenzo-42-crown-14 (*OC*-6-22)-tetraammine(1,10-phenanthroline-*N*,*N*′)-ruthenium(II) bis(hexafluorophosphate) dichloromethane solvate, $[Ru(C_{12}H_8N_2)(NH_3)_4](PF_6)_2.C_{36}H_{56}O_{14}.CH_2Cl_2$, is reported. The guest Ru complex is located deep within the U-shaped cavity formed by folding of the flexible crown host. The ammine ligands of the Ru complex participate in an extensive network of hydrogen bonds with the ether O atoms of the crown. Weak π stacking appears to occur between the aromatic rings of the phenanthroline ligand and the benzene groups of the crown ether.

Comment

Hydrogen-bonding interactions between crown O atoms and ammine protons play an important role in the molecular recognition of ammine–metal complexes by crown ethers. For a recent list of references on this subject, see Zhang *et al.* (1994); for earlier references, see Colquhoun *et al.* (1986). We have observed that binding of ruthenium–ammine complexes by large cyclic crown ethers increases with the size

Acta Crystallographica Section C
ISSN 0108-2701 © 1998