Unexpected Formation of a Silicon Centered Spirocyclic Oligosiloxane Bearing Eight Pendant Ferrocene Units

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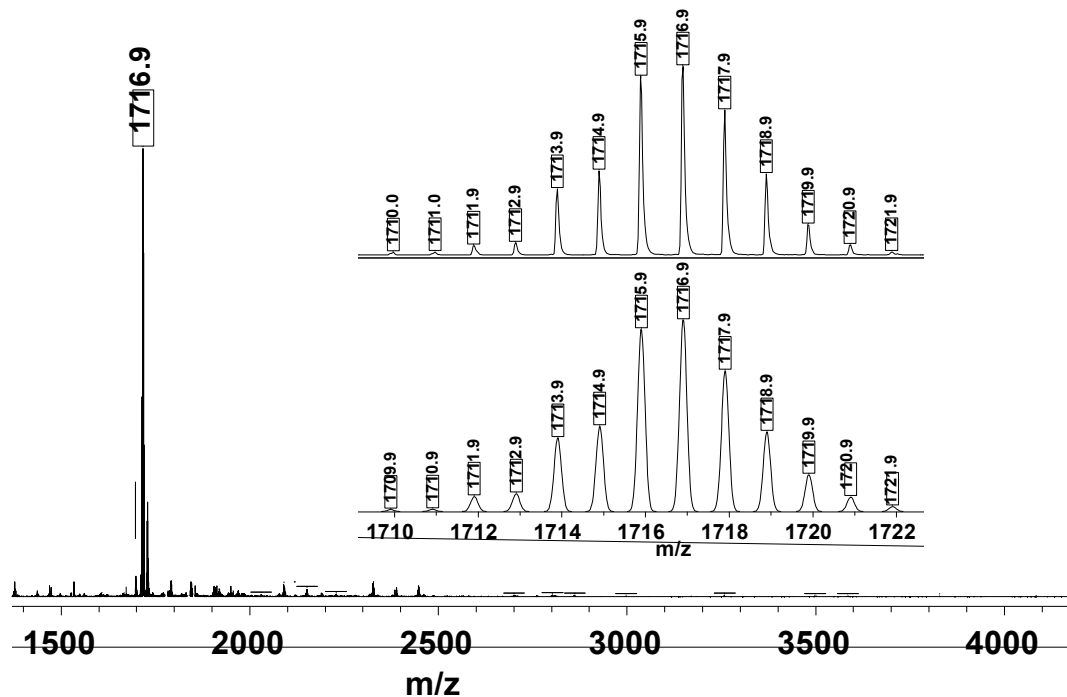
1. Characterization of 4

Figure S1. MALDI-TOF mass spectrometry of octaferrocenyl spirosilicate **4**. Inset: isotopic distribution of molecular ion peak (top: experimental; bottom: calculated).
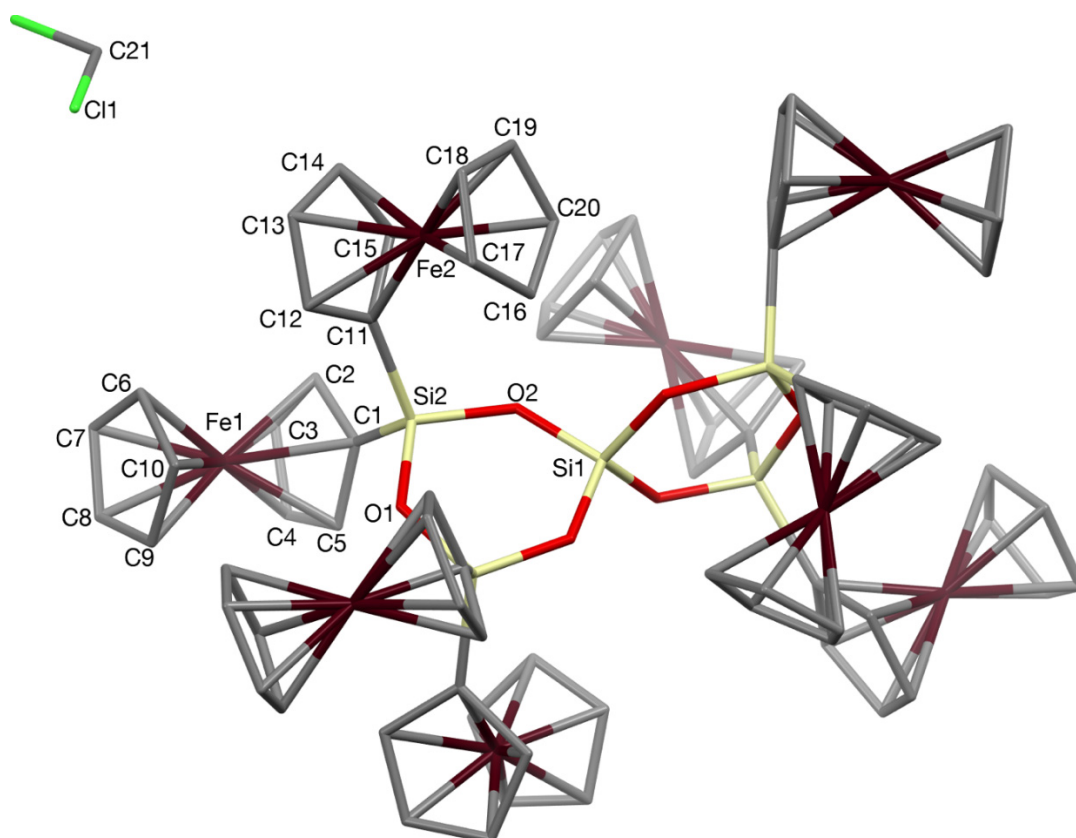
2. Single Crystal X-ray Diffraction data

Figure S2. Structure of **4** with atoms labelled in the asymmetric unit.

Table S1. Sample and crystal data for 4.

Identification code	CCDC 2182627	
Chemical formula	$(C_{80}H_{72}Fe_8O_6Si_5)\cdot2(CH_2Cl_2)$	
Formula weight	1886.47.08 g/mol	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal size	0.040 x 0.040 x 0.190 mm	
Crystal habit	clear intense orange needle	
Crystal system	tetragonal	
Space group	$P4_2/n$	
Unit cell dimensions	$a = 15.650(1)$ Å	$\alpha = 90°$
	$b = 15.650(1)$ Å	$\beta = 90°$
	$c = 15.714(1)$ Å	$\gamma = 90°$
Volume	3848.6(6) Å^3	
Z	2	
Density (calculated)	1.628 g/cm^3	
Absorption coefficient	1.734 mm^{-1}	
F(000)	1924	

Table S2. Data collection and structure refinement for 4.

Theta range for data collection	2.25 to 24.75°
Index ranges	-18<=h<=14, -18<=k<=8, -12<=l<=17
Reflections collected	6429
Independent reflections	3175 [R(int) = 0.0601]
Max. and min. transmission	0.9380 and 0.7470
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2019/1 (Sheldrick, 2019)
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	3175 / 9 / 238
Goodness-of-fit on F^2	1.001
Final R indices	1755 data; I>2σ(I) $R_1 = 0.0528$, $wR_2 = 0.1234$
	all data $R_1 = 0.1290$, $wR_2 = 0.1527$
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0640P)^2]$ where $P=(F_o^2+2F_c^2)/3$
Largest diff. peak and hole	0.575 and -0.561 eÅ^{-3}
R.M.S. deviation from mean	0.119 eÅ^{-3}

Table S3. Si-O bond distances (Å).

O1-Si2	1.640(3)
O1-Si2^i	1.640(3)
O2-Si1	1.617(4)
O2-Si2	1.644(4)

(i)-x+1/2, -y+1/2, z

Table S4. Selected bond angles (°).

Si1-O2-Si2	132.7(3)
Si2-O1-Si2 ^{i}	132.0(4)
O2-Si1-O2 ^{i}	107.8(3)
O2-Si1-O2^{ii}	110.3(1)
O2-Si1-O2^{iii}	110.3(1)
O2 ^{i} -Si1-O2^{ii}	110.3(1)
O2^{iii}-Si1-O2 ^{i}	110.3(1)
O2^{iii}-Si1-O2^{ii}	107.8(3)
O1-Si2-O2	107.3(3)
O1-Si2-C1	108.3(2)

O2-Si2-C1	108.7(3)
O1-Si2-C11	113.4(2)
O2-Si2-C11	107.9(3)
C1-Si2-C11	111.2(3)

(i) -x+1/2, -y+1/2, z; (ii) y, -x+1/2, -z+3/2; (iii) -y+1/2, x, -z+3/2