

X-ray Structural Data for 2c.

Data Collection

A colorless prismatic crystal of $\text{Ru}_3\text{SiC}_{27}\text{O}_6\text{H}_{22}$ having approximate dimensions of $0.15 \times 0.20 \times 0.30$ mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-K α radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $50.30 < 2\theta < 54.65^\circ$ corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned}a &= 10.865(2) \text{ \AA} \\b &= 17.050(4) \text{ \AA} & \beta &= 101.36(2)^\circ \\c &= 14.384(3) \text{ \AA} \\V &= 2612.4(9) \text{ \AA}^3\end{aligned}$$

For $Z = 4$ and F.W. = 773.76, the calculated density is 1.97 g/cm^3 . The systematic absences of:

$$\begin{aligned}h0l: \quad h+l &= 2n \\0k0: \quad k &= 2n\end{aligned}$$

uniquely determine the space group to be:

P2₁/n (#14)

The data were collected at a temperature of $20 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 55.1° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.28° with a take-off angle of 6.0° . Scans of $(1.26 + 0.30 \tan \theta)^\circ$ were made at a speed of $32.0^\circ/\text{min}$ (in ω). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 3 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm and the crystal to detector distance was 400 mm. The computer-controlled slits were set to 9.0 mm (horizontal) and 13.0 mm (vertical).

Data Reduction

Of the 6557 reflections which were collected, 6241 were unique ($R_{\text{int}} = 0.013$). The intensities of three representative reflections were measured after every 150 reflections. No decay

correction was applied.

The linear absorption coefficient, μ , for Mo-K α radiation is 18.0 cm^{-1} . The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ on F was based on 5146 observed reflections ($I > 3.00\sigma(I)$) and 422 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum | |F_O| - |F_C| | / \sum |F_O| = 0.029$$

$$R_w = [\sum w (|F_O| - |F_C|)^2 / \sum w F_O^2]^{1/2} = 0.028$$

The standard deviation of an observation of unit weight⁴ was 2.45. The weighting scheme was based on counting statistics and included a factor ($p = 0.005$) to downweight the intense reflections. Plots of $\sum w (|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.64 and $-1.14 \text{ e}^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for Δf and $\Delta f'$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the teXsan⁹ crystallographic software package of Molecular Structure Corporation.

References

- (1) PATTY: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., Garcia-Granda, S., Gould, R.O., Smits, J.M.M. and Smykalla, C. (1992). The DIRIDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (2) DIRIDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRIDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (3) Least Squares function minimized:

$$\Sigma w (|F_O| - |F_C|)^2 \text{ where}$$

$$w = 1/[\sigma^2(F_o)] = [\sigma_c^2(F_o) + p^2 F_o^2/4]^{-1}$$

$\sigma_c(F_o)$ = e.s.d. based on counting statistics
 p = p-factor

(4) Standard deviation of an observation of unit weight:

$$[\sum w(|F_o| \cdot |F_c|)^2 / (N_o \cdot N_v)]^{1/2}$$

where: N_o = number of observations
 N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan for Windows version 1.06: Crystal Structure Analysis Package, Molecular Structure Corporation (1997-9).

EXPERIMENTAL DETAILS**A. Crystal Data**

Empirical Formula	Ru ₃ SiC ₂₇ O ₆ H ₂₂
Formula Weight	773.76
Crystal Color, Habit	red, plate
Crystal Dimensions	0.15 X 0.20 X 0.30 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (50.3 - 54.7°)
Omega Scan Peak Width at Half-height	0.28°
Lattice Parameters	$a = 10.865(2) \text{ \AA}$ $b = 17.050(4) \text{ \AA}$ $c = 14.384(3) \text{ \AA}$ $\beta = 101.36(2)^\circ$ $V = 2612.4(9) \text{ \AA}^3$
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.967 g/cm ³
F ₀₀₀	1512.00
μ(MoKα)	18.00 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC7R
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated
Attenuator	Zr foil (factor = 8.40)
Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	400 mm
Voltage, Current	50kV, 40mA
Temperature	20.0°C
Scan Type	ω -2 θ
Scan Rate	32.0°/min (in ω) (up to 3 scans)
Scan Width	(1.26 + 0.30 tan θ)°
2 θ_{\max}	55.1°
No. of Reflections Measured	Total: 6557
Corrections	Unique: 6241 ($R_{\text{int}} = 0.013$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF92 PATTY)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (F_o - F_c)^2$
Least Squares Weights	$1/\sigma^2(F_o) = 4F_o^2/\sigma^2(F_o^2)$
p-factor	0.0054
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	5146
No. Variables	422
Reflection/Parameter Ratio	12.19
Residuals: R; R_w	0.029 ; 0.028
Goodness of Fit Indicator	2.45
Max Shift/Error in Final Cycle	0.01
Maximum peak in Final Diff. Map	$0.64 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-1.14 \text{ e}^-/\text{\AA}^3$

Table S1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B _{eq}
Ru(1)	0.15545(2)	0.72339(1)	0.36929(1)	2.704(5)
Ru(2)	0.34287(2)	0.66757(1)	0.25976(1)	2.325(4)
Ru(3)	0.23299(2)	0.56038(1)	0.37441(1)	2.333(4)
Si(1)	0.47023(6)	0.58835(4)	0.17475(5)	2.44(1)
O(1)	0.2028(2)	0.7140(2)	0.5827(2)	6.69(7)
O(2)	0.3543(2)	0.8477(1)	0.3921(2)	6.59(7)
O(3)	0.5692(2)	0.7049(1)	0.4100(2)	5.90(6)
O(4)	0.4254(2)	0.7975(1)	0.1463(2)	5.83(6)
O(5)	0.3770(2)	0.5744(2)	0.5751(1)	6.35(7)
O(6)	0.3152(2)	0.3927(1)	0.3843(2)	5.61(6)
C(1)	-0.0436(2)	0.7661(2)	0.3219(2)	3.69(7)
C(2)	0.0231(3)	0.7781(2)	0.2474(2)	3.47(7)
C(3)	0.1428(2)	0.6782(2)	0.1555(2)	2.85(6)
C(4)	0.1757(2)	0.5994(2)	0.1569(2)	2.66(5)
C(5)	0.1363(2)	0.5425(2)	0.2196(2)	2.53(5)
C(6)	0.0159(2)	0.5217(2)	0.3523(2)	3.10(6)
C(7)	-0.0649(2)	0.5583(2)	0.4090(2)	3.62(7)
C(8)	-0.0954(2)	0.6340(2)	0.4048(2)	3.48(7)
C(9)	-0.0479(2)	0.6836(2)	0.3399(2)	3.02(6)
C(10)	0.0185(2)	0.6466(1)	0.2755(2)	2.42(5)
C(11)	0.0643(2)	0.7035(2)	0.2188(2)	2.83(6)
C(12)	0.0482(2)	0.5655(1)	0.2768(2)	2.46(5)
C(13)	0.1884(3)	0.7147(2)	0.5017(2)	4.02(7)
C(14)	0.2846(3)	0.7965(2)	0.3823(2)	4.12(7)
C(15)	0.4837(3)	0.6896(2)	0.3547(2)	3.62(7)
C(16)	0.3896(2)	0.7481(2)	0.1871(2)	3.57(7)
C(17)	0.3176(3)	0.5726(2)	0.5010(2)	3.90(7)
C(18)	0.2805(2)	0.4558(2)	0.3793(2)	3.37(6)
C(19)	0.6442(3)	0.5956(2)	0.2284(2)	3.45(7)
C(20)	0.7240(2)	0.5409(2)	0.1840(2)	2.99(6)
C(21)	0.7772(3)	0.5636(2)	0.1091(2)	3.47(7)
C(22)	0.8494(2)	0.5136(2)	0.0673(2)	3.76(7)
C(23)	0.8703(3)	0.4383(2)	0.1005(2)	4.05(7)
C(24)	0.8187(3)	0.4142(2)	0.1751(2)	3.98(7)
C(25)	0.7461(2)	0.4651(2)	0.2165(2)	3.56(7)
C(26)	0.4303(3)	0.4824(2)	0.1797(3)	3.87(8)
C(27)	0.4526(3)	0.6158(2)	0.0472(2)	3.80(8)
H(1)	-0.075(2)	0.810(1)	0.356(2)	3.7(6)
H(2)	0.041(2)	0.825(1)	0.226(2)	2.9(6)
H(3)	0.161(2)	0.714(1)	0.109(2)	3.3(6)

Table S1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
H(4)	0.207(2)	0.578(1)	0.112(1)	1.4(4)
H(5)	0.146(2)	0.491(1)	0.208(1)	1.7(5)
H(6)	0.026(2)	0.467(1)	0.353(2)	3.1(6)
H(7)	-0.091(2)	0.525(2)	0.449(2)	4.1(7)
H(8)	-0.141(2)	0.652(1)	0.441(2)	3.1(6)
H(9)	0.656(2)	0.584(1)	0.293(2)	3.3(6)
H(10)	0.667(3)	0.644(2)	0.222(2)	4.3(7)
H(11)	0.764(2)	0.611(1)	0.089(2)	2.6(6)
H(12)	0.886(2)	0.527(1)	0.019(2)	2.3(5)
H(13)	0.918(2)	0.406(1)	0.071(2)	3.4(6)
H(14)	0.831(2)	0.367(1)	0.197(2)	2.0(5)
H(15)	0.720(2)	0.448(1)	0.272(2)	2.7(5)
H(16)	0.434(3)	0.466(2)	0.234(2)	5.9(9)
H(17)	0.355(3)	0.474(2)	0.149(2)	6.3(9)
H(18)	0.477(3)	0.455(2)	0.146(2)	4.1(7)
H(19)	0.387(3)	0.623(2)	0.023(2)	6(1)
H(20)	0.493(3)	0.587(2)	0.018(2)	6.1(9)
H(21)	0.478(3)	0.670(2)	0.042(2)	6.4(9)
H(22)	0.363(2)	0.588(1)	0.338(2)	3.3(5)

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table S2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ru(1)	0.0261(1)	0.0350(1)	0.0428(1)	-0.00218(9)	0.00945(9)	-0.0099(1)
Ru(2)	0.0250(1)	0.0306(1)	0.0342(1)	-0.00160(8)	0.00967(8)	-0.00143(9)
Ru(3)	0.0232(1)	0.0342(1)	0.0312(1)	-0.00229(8)	0.00522(8)	0.00178(9)
Si(1)	0.0258(3)	0.0366(4)	0.0315(4)	0.0010(3)	0.0082(3)	-0.0018(3)
O(1)	0.088(2)	0.120(2)	0.045(1)	0.007(2)	0.011(1)	-0.020(1)
O(2)	0.056(1)	0.057(2)	0.144(2)	-0.028(1)	0.035(2)	-0.040(2)
O(3)	0.045(1)	0.096(2)	0.074(2)	-0.014(1)	-0.011(1)	-0.033(1)
O(4)	0.071(2)	0.052(1)	0.107(2)	-0.001(1)	0.039(1)	0.032(1)
O(5)	0.070(2)	0.127(2)	0.035(1)	0.003(2)	-0.011(1)	-0.000(1)
O(6)	0.065(1)	0.041(1)	0.108(2)	0.007(1)	0.019(1)	0.014(1)
C(1)	0.033(1)	0.049(2)	0.058(2)	0.012(1)	0.008(1)	-0.010(2)
C(2)	0.041(2)	0.035(2)	0.056(2)	0.006(1)	0.009(1)	0.006(1)
C(3)	0.028(1)	0.046(2)	0.032(1)	-0.001(1)	0.003(1)	0.007(1)
C(4)	0.027(1)	0.045(2)	0.027(1)	-0.000(1)	0.001(1)	-0.007(1)
C(5)	0.026(1)	0.033(1)	0.034(1)	-0.002(1)	0.000(1)	-0.010(1)
C(6)	0.024(1)	0.048(2)	0.044(2)	-0.008(1)	0.002(1)	0.004(1)
C(7)	0.026(1)	0.072(2)	0.041(2)	-0.011(1)	0.008(1)	0.009(2)
C(8)	0.022(1)	0.069(2)	0.042(2)	-0.003(1)	0.009(1)	-0.006(2)
C(9)	0.019(1)	0.053(2)	0.042(2)	-0.000(1)	0.005(1)	-0.004(1)
C(10)	0.022(1)	0.037(1)	0.032(1)	-0.004(1)	0.003(1)	-0.008(1)
C(11)	0.028(1)	0.042(2)	0.036(1)	0.004(1)	0.004(1)	0.002(1)
C(12)	0.018(1)	0.040(1)	0.033(1)	-0.006(1)	-0.003(1)	-0.003(1)
C(13)	0.041(2)	0.060(2)	0.054(2)	-0.003(1)	0.014(1)	-0.016(2)
C(14)	0.041(2)	0.042(2)	0.075(2)	-0.004(1)	0.017(2)	-0.016(2)
C(15)	0.041(2)	0.049(2)	0.050(2)	-0.008(1)	0.014(1)	-0.010(1)
C(16)	0.037(2)	0.043(2)	0.059(2)	0.006(1)	0.017(1)	0.009(1)
C(17)	0.041(2)	0.070(2)	0.038(2)	-0.001(1)	0.011(1)	-0.000(2)
C(18)	0.033(1)	0.044(2)	0.050(2)	-0.005(1)	0.008(1)	0.008(1)
C(19)	0.032(1)	0.053(2)	0.045(2)	0.003(1)	0.005(1)	-0.007(2)
C(20)	0.022(1)	0.048(2)	0.041(1)	0.004(1)	0.000(1)	-0.006(1)
C(21)	0.031(1)	0.049(2)	0.052(2)	0.004(1)	0.007(1)	0.001(2)
C(22)	0.028(1)	0.073(2)	0.042(2)	-0.001(1)	0.008(1)	-0.007(2)
C(23)	0.027(1)	0.073(2)	0.051(2)	0.010(1)	-0.001(1)	-0.022(2)
C(24)	0.036(2)	0.045(2)	0.063(2)	0.008(1)	-0.009(1)	-0.002(2)
C(25)	0.030(1)	0.062(2)	0.042(2)	0.001(1)	0.003(1)	0.001(1)
C(26)	0.045(2)	0.043(2)	0.065(2)	0.002(1)	0.025(2)	-0.008(2)
C(27)	0.042(2)	0.071(3)	0.033(2)	0.005(2)	0.010(1)	0.004(2)

The general temperature factor expression: $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table S3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Ru(1)	Ru(2)	2.9662(6)	Ru(1)	Ru(3)	2.9009(8)
Ru(1)	C(1)	2.256(4)	Ru(1)	C(2)	2.241(4)
Ru(1)	C(9)	2.270(4)	Ru(1)	C(10)	2.226(4)
Ru(1)	C(11)	2.222(4)	Ru(1)	C(13)	1.874(5)
Ru(1)	C(14)	1.858(4)	Ru(2)	Ru(3)	2.8742(6)
Ru(2)	Si(1)	2.429(1)	Ru(2)	C(3)	2.392(4)
Ru(2)	C(4)	2.404(4)	Ru(2)	C(15)	1.877(4)
Ru(2)	C(16)	1.856(4)	Ru(2)	H(22)	1.75(3)
Ru(3)	C(5)	2.287(4)	Ru(3)	C(6)	2.409(4)
Ru(3)	C(12)	2.214(3)	Ru(3)	C(17)	1.883(4)
Ru(3)	C(18)	1.854(4)	Ru(3)	H(22)	1.67(3)
Si(1)	C(19)	1.900(4)	Si(1)	C(26)	1.862(5)
Si(1)	C(27)	1.866(5)	O(1)	C(13)	1.144(5)
O(2)	C(14)	1.146(5)	O(3)	C(15)	1.128(5)
O(4)	C(16)	1.137(5)	O(5)	C(17)	1.132(5)
O(6)	C(18)	1.138(5)	C(1)	C(2)	1.421(6)
C(1)	C(9)	1.433(6)	C(1)	H(1)	1.00(4)
C(2)	C(11)	1.436(5)	C(2)	H(2)	0.90(4)
C(3)	C(4)	1.388(5)	C(3)	C(11)	1.432(5)
C(3)	H(3)	0.96(4)	C(4)	C(5)	1.446(5)

Table S3. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(4)	H(4)	0.86(3)	C(5)	C(12)	1.434(5)
C(5)	H(5)	0.90(3)	C(6)	C(7)	1.452(6)
C(6)	C(12)	1.418(5)	C(6)	H(6)	0.93(4)
C(7)	C(8)	1.331(6)	C(7)	H(7)	0.89(4)
C(8)	C(9)	1.430(6)	C(8)	H(8)	0.85(4)
C(9)	C(10)	1.428(5)	C(10)	C(11)	1.419(5)
C(10)	C(12)	1.420(5)	C(19)	C(20)	1.499(6)
C(19)	H(9)	0.94(4)	C(19)	H(10)	0.87(4)
C(20)	C(21)	1.376(6)	C(20)	C(25)	1.379(6)
C(21)	C(22)	1.375(6)	C(21)	H(11)	0.86(4)
C(22)	C(23)	1.372(7)	C(22)	H(12)	0.90(3)
C(23)	C(24)	1.367(7)	C(23)	H(13)	0.92(4)
C(24)	C(25)	1.384(6)	C(24)	H(14)	0.87(3)
C(25)	H(15)	0.94(3)	C(26)	H(16)	0.83(5)
C(26)	H(17)	0.87(5)	C(26)	H(18)	0.90(4)
C(27)	H(19)	0.74(5)	C(27)	H(20)	0.82(5)
C(27)	H(21)	0.98(5)			

Table S4. Bond Angles($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
Ru(2)	Ru(1)	Ru(3)	58.65(1)	Ru(2)	Ru(1)	C(1)	131.3(1)
Ru(2)	Ru(1)	C(2)	96.9(1)	Ru(2)	Ru(1)	C(9)	123.4(1)
Ru(2)	Ru(1)	C(10)	86.36(9)	Ru(2)	Ru(1)	C(11)	69.8(1)
Ru(2)	Ru(1)	C(13)	120.1(1)	Ru(2)	Ru(1)	C(14)	71.6(1)
Ru(3)	Ru(1)	C(1)	125.0(1)	Ru(3)	Ru(1)	C(2)	123.9(1)
Ru(3)	Ru(1)	C(9)	89.2(1)	Ru(3)	Ru(1)	C(10)	66.9(1)
Ru(3)	Ru(1)	C(11)	87.1(1)	Ru(3)	Ru(1)	C(13)	84.4(1)
Ru(3)	Ru(1)	C(14)	115.5(1)	C(1)	Ru(1)	C(2)	36.8(2)
C(1)	Ru(1)	C(9)	36.9(1)	C(1)	Ru(1)	C(10)	61.3(1)
C(1)	Ru(1)	C(11)	62.5(2)	C(1)	Ru(1)	C(13)	108.2(2)
C(1)	Ru(1)	C(14)	118.3(2)	C(2)	Ru(1)	C(9)	61.8(2)
C(2)	Ru(1)	C(10)	61.4(2)	C(2)	Ru(1)	C(11)	37.5(1)
C(2)	Ru(1)	C(13)	142.5(2)	C(2)	Ru(1)	C(14)	98.9(2)
C(9)	Ru(1)	C(10)	37.0(1)	C(9)	Ru(1)	C(11)	62.8(1)
C(9)	Ru(1)	C(13)	98.6(2)	C(9)	Ru(1)	C(14)	155.1(2)
C(10)	Ru(1)	C(11)	37.2(1)	C(10)	Ru(1)	C(13)	122.7(2)
C(10)	Ru(1)	C(14)	148.9(2)	C(11)	Ru(1)	C(13)	159.7(2)
C(11)	Ru(1)	C(14)	112.5(2)	C(13)	Ru(1)	C(14)	87.8(2)
Ru(1)	Ru(2)	Ru(3)	59.54(2)	Ru(1)	Ru(2)	Si(1)	164.68(3)
Ru(1)	Ru(2)	C(3)	71.0(1)	Ru(1)	Ru(2)	C(4)	88.1(1)

Table S4. Bond Angles($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
Ru(1)	Ru(2)	C(15)	95.5(1)	Ru(1)	Ru(2)	C(16)	110.8(1)
Ru(1)	Ru(2)	H(22)	85(1)	Ru(3)	Ru(2)	Si(1)	106.11(3)
Ru(3)	Ru(2)	C(3)	88.8(1)	Ru(3)	Ru(2)	C(4)	72.3(1)
Ru(3)	Ru(2)	C(15)	94.7(1)	Ru(3)	Ru(2)	C(16)	169.8(1)
Ru(3)	Ru(2)	H(22)	32(1)	Si(1)	Ru(2)	C(3)	105.3(1)
Si(1)	Ru(2)	C(4)	81.8(1)	Si(1)	Ru(2)	C(15)	90.8(1)
Si(1)	Ru(2)	C(16)	83.2(1)	Si(1)	Ru(2)	H(22)	83(1)
C(3)	Ru(2)	C(4)	33.6(1)	C(3)	Ru(2)	C(15)	162.0(2)
C(3)	Ru(2)	C(16)	84.6(2)	C(3)	Ru(2)	H(22)	117(1)
C(4)	Ru(2)	C(15)	162.4(2)	C(4)	Ru(2)	C(16)	105.6(2)
C(4)	Ru(2)	H(22)	90(1)	C(15)	Ru(2)	C(16)	89.3(2)
C(15)	Ru(2)	H(22)	73(1)	C(16)	Ru(2)	H(22)	157(1)
Ru(1)	Ru(3)	Ru(2)	61.81(2)	Ru(1)	Ru(3)	C(5)	91.6(1)
Ru(1)	Ru(3)	C(6)	89.3(1)	Ru(1)	Ru(3)	C(12)	74.0(1)
Ru(1)	Ru(3)	C(17)	90.2(1)	Ru(1)	Ru(3)	C(18)	178.9(1)
Ru(1)	Ru(3)	H(22)	89(1)	Ru(2)	Ru(3)	C(5)	71.9(1)
Ru(2)	Ru(3)	C(6)	127.2(1)	Ru(2)	Ru(3)	C(12)	92.2(1)
Ru(2)	Ru(3)	C(17)	107.8(1)	Ru(2)	Ru(3)	C(18)	119.3(1)
Ru(2)	Ru(3)	H(22)	34(1)	C(5)	Ru(3)	C(6)	65.5(1)
C(5)	Ru(3)	C(12)	37.1(1)	C(5)	Ru(3)	C(17)	177.8(2)

Table S4. Bond Angles($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(5)	Ru(3)	C(18)	88.8(2)	C(5)	Ru(3)	H(22)	89(1)
C(6)	Ru(3)	C(12)	35.4(1)	C(6)	Ru(3)	C(17)	115.9(2)
C(6)	Ru(3)	C(18)	90.0(2)	C(6)	Ru(3)	H(22)	155(1)
C(12)	Ru(3)	C(17)	144.9(2)	C(12)	Ru(3)	C(18)	105.8(2)
C(12)	Ru(3)	H(22)	121(1)	C(17)	Ru(3)	C(18)	89.4(2)
C(17)	Ru(3)	H(22)	89(1)	C(18)	Ru(3)	H(22)	92(1)
Ru(2)	Si(1)	C(19)	112.3(1)	Ru(2)	Si(1)	C(26)	111.1(2)
Ru(2)	Si(1)	C(27)	113.4(2)	C(19)	Si(1)	C(26)	105.6(2)
C(19)	Si(1)	C(27)	106.5(2)	C(26)	Si(1)	C(27)	107.5(3)
Ru(1)	C(1)	C(2)	71.0(2)	Ru(1)	C(1)	C(9)	72.1(2)
Ru(1)	C(1)	H(1)	120(2)	C(2)	C(1)	C(9)	108.5(4)
C(2)	C(1)	H(1)	122(2)	C(9)	C(1)	H(1)	129(2)
Ru(1)	C(2)	C(1)	72.2(3)	Ru(1)	C(2)	C(11)	70.5(2)
Ru(1)	C(2)	H(2)	119(3)	C(1)	C(2)	C(11)	108.8(4)
C(1)	C(2)	H(2)	125(3)	C(11)	C(2)	H(2)	126(3)
Ru(2)	C(3)	C(4)	73.6(2)	Ru(2)	C(3)	C(11)	101.9(3)
Ru(2)	C(3)	H(3)	102(2)	C(4)	C(3)	C(11)	117.8(4)
C(4)	C(3)	H(3)	123(2)	C(11)	C(3)	H(3)	118(2)
Ru(2)	C(4)	C(3)	72.7(2)	Ru(2)	C(4)	C(5)	102.8(2)
Ru(2)	C(4)	H(4)	108(2)	C(3)	C(4)	C(5)	123.5(4)

Table S4. Bond Angles^(o) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	C(4)	H(4)	122(2)	C(5)	C(4)	H(4)	113(2)
Ru(3)	C(5)	C(4)	112.4(3)	Ru(3)	C(5)	C(12)	68.7(2)
Ru(3)	C(5)	H(5)	105(2)	C(4)	C(5)	C(12)	119.2(4)
C(4)	C(5)	H(5)	118(2)	C(12)	C(5)	H(5)	119(2)
Ru(3)	C(6)	C(7)	119.6(3)	Ru(3)	C(6)	C(12)	64.7(2)
Ru(3)	C(6)	H(6)	100(2)	C(7)	C(6)	C(12)	118.2(4)
C(7)	C(6)	H(6)	120(2)	C(12)	C(6)	H(6)	119(2)
C(6)	C(7)	C(8)	124.4(4)	C(6)	C(7)	H(7)	114(3)
C(8)	C(7)	H(7)	122(3)	C(7)	C(8)	C(9)	119.0(4)
C(7)	C(8)	H(8)	120(3)	C(9)	C(8)	H(8)	121(3)
Ru(1)	C(9)	C(1)	71.0(2)	Ru(1)	C(9)	C(8)	121.2(3)
Ru(1)	C(9)	C(10)	69.8(2)	C(1)	C(9)	C(8)	136.7(4)
C(1)	C(9)	C(10)	106.1(4)	C(8)	C(9)	C(10)	117.1(4)
Ru(1)	C(10)	C(9)	73.1(2)	Ru(1)	C(10)	C(11)	71.2(2)
Ru(1)	C(10)	C(12)	116.2(2)	C(9)	C(10)	C(11)	110.5(3)
C(9)	C(10)	C(12)	124.4(4)	C(11)	C(10)	C(12)	124.7(3)
Ru(1)	C(11)	C(2)	71.9(2)	Ru(1)	C(11)	C(3)	117.3(3)
Ru(1)	C(11)	C(10)	71.6(2)	C(2)	C(11)	C(3)	135.1(4)
C(2)	C(11)	C(10)	106.0(3)	C(3)	C(11)	C(10)	118.7(4)
Ru(3)	C(12)	C(5)	74.2(2)	Ru(3)	C(12)	C(6)	79.8(2)

Table S4. Bond Angles($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
Ru(3)	C(12)	C(10)	102.8(2)	C(5)	C(12)	C(6)	126.1(4)
C(5)	C(12)	C(10)	115.8(3)	C(6)	C(12)	C(10)	115.7(4)
Ru(1)	C(13)	O(1)	174.9(4)	Ru(1)	C(14)	O(2)	172.2(4)
Ru(2)	C(15)	O(3)	177.7(4)	Ru(2)	C(16)	O(4)	175.7(4)
Ru(3)	C(17)	O(5)	172.9(4)	Ru(3)	C(18)	O(6)	176.7(4)
Si(1)	C(19)	C(20)	113.5(3)	Si(1)	C(19)	H(9)	108(2)
Si(1)	C(19)	H(10)	108(3)	C(20)	C(19)	H(9)	108(2)
C(20)	C(19)	H(10)	110(3)	H(9)	C(19)	H(10)	109(4)
C(19)	C(20)	C(21)	121.7(4)	C(19)	C(20)	C(25)	121.1(4)
C(21)	C(20)	C(25)	117.2(4)	C(20)	C(21)	C(22)	122.2(5)
C(20)	C(21)	H(11)	118(3)	C(22)	C(21)	H(11)	120(3)
C(21)	C(22)	C(23)	119.8(5)	C(21)	C(22)	H(12)	124(2)
C(23)	C(22)	H(12)	116(2)	C(22)	C(23)	C(24)	119.3(4)
C(22)	C(23)	H(13)	118(3)	C(24)	C(23)	H(13)	123(3)
C(23)	C(24)	C(25)	120.4(5)	C(23)	C(24)	H(14)	120(3)
C(25)	C(24)	H(14)	119(3)	C(20)	C(25)	C(24)	121.1(5)
C(20)	C(25)	H(15)	121(2)	C(24)	C(25)	H(15)	118(2)
Si(1)	C(26)	H(16)	114(4)	Si(1)	C(26)	H(17)	111(4)
Si(1)	C(26)	H(18)	109(3)	H(16)	C(26)	H(17)	107(5)
H(16)	C(26)	H(18)	114(4)	H(17)	C(26)	H(18)	102(4)

Table S4. Bond Angles($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
Si(1)	C(27)	H(19)	114(4)	Si(1)	C(27)	H(20)	113(4)
Si(1)	C(27)	H(21)	110(3)	H(19)	C(27)	H(20)	114(5)
H(19)	C(27)	H(21)	95(5)	H(20)	C(27)	H(21)	109(4)

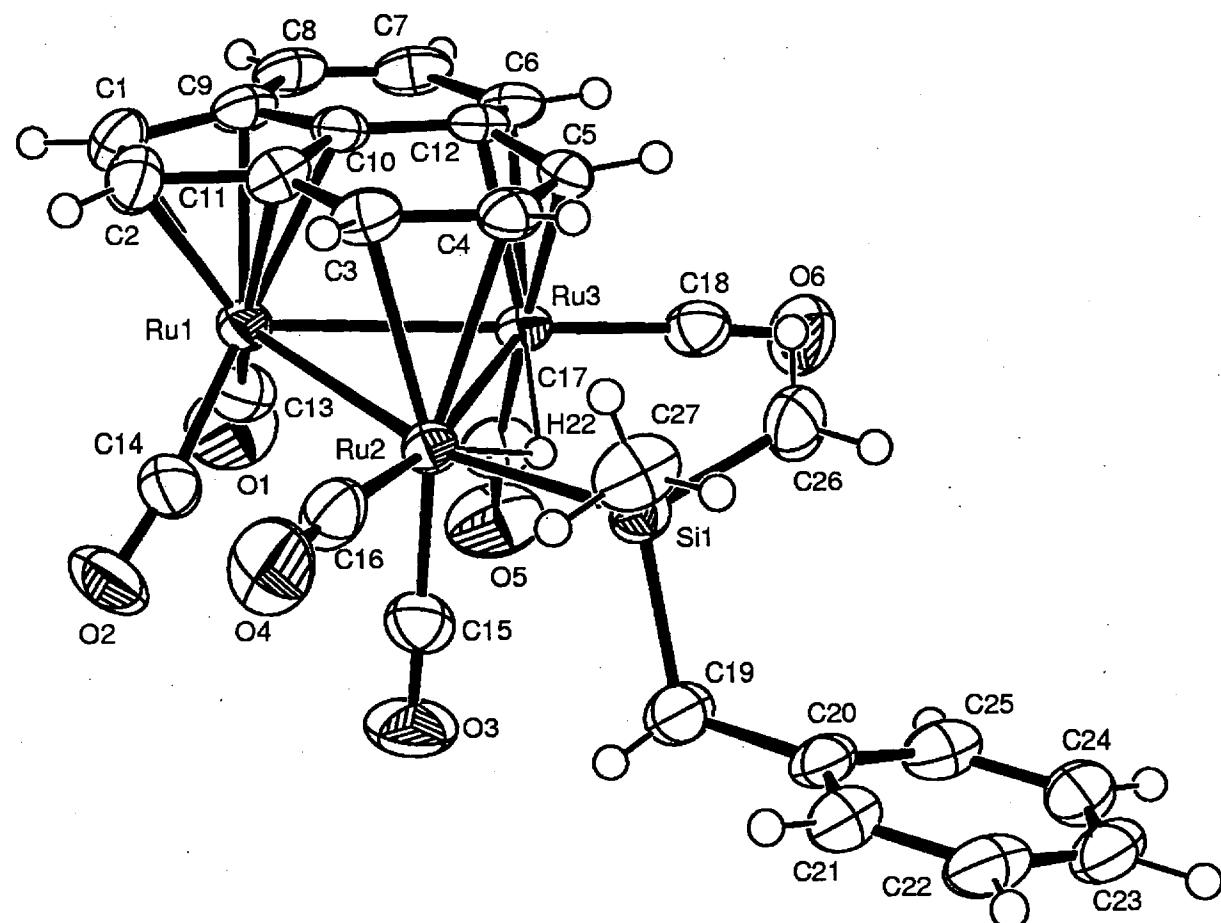


Figure S-1. ORTEP drawing of 2c. 50% probability of the thermal ellipsoids.

Figure S2 SEC chart of silylated PolyTHF

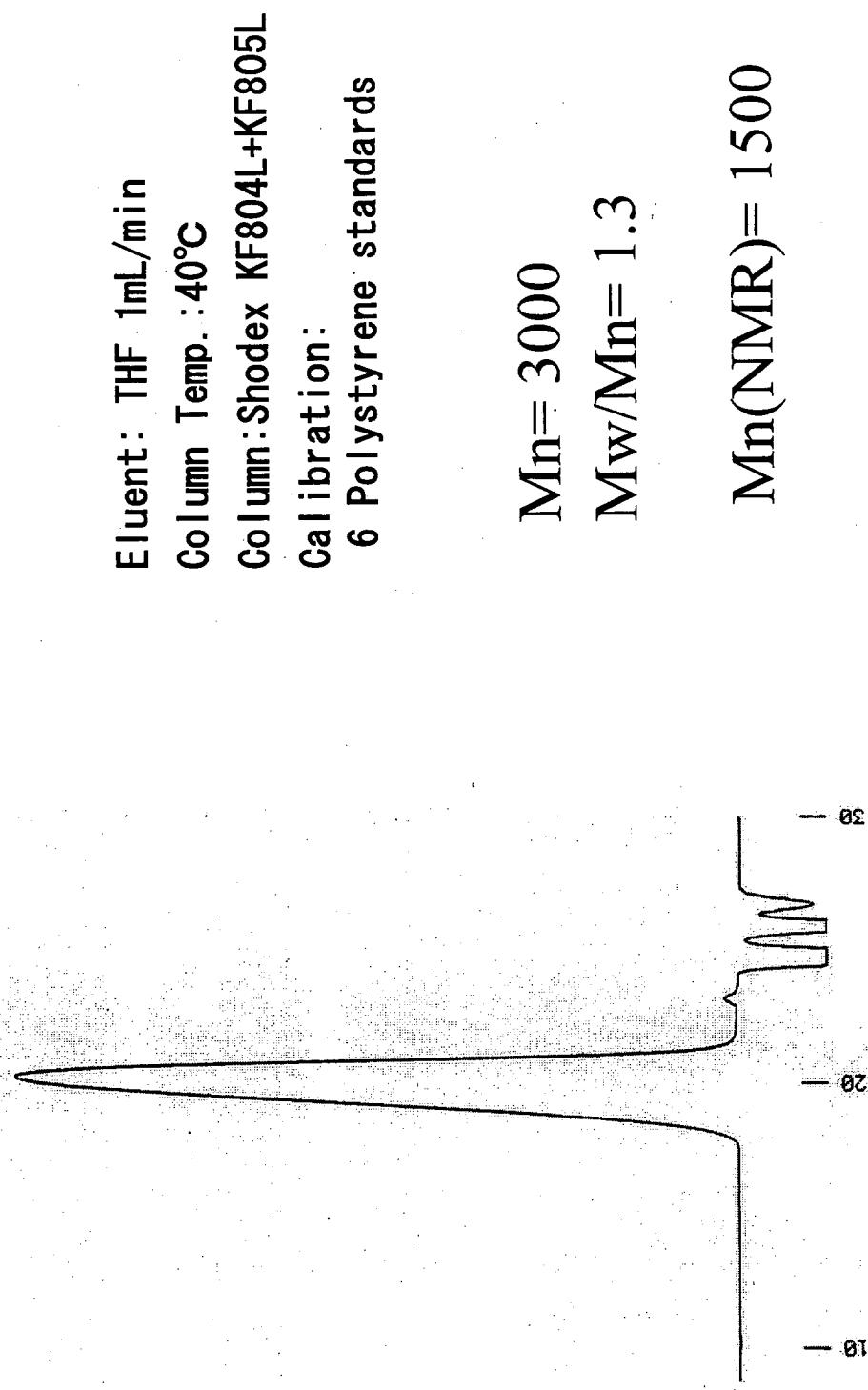


Figure S3 $^1\text{H-NMR}$ spectra of $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2[\text{O}(\text{CH}_2)_4]_n\text{-OSiPhMe}_2$

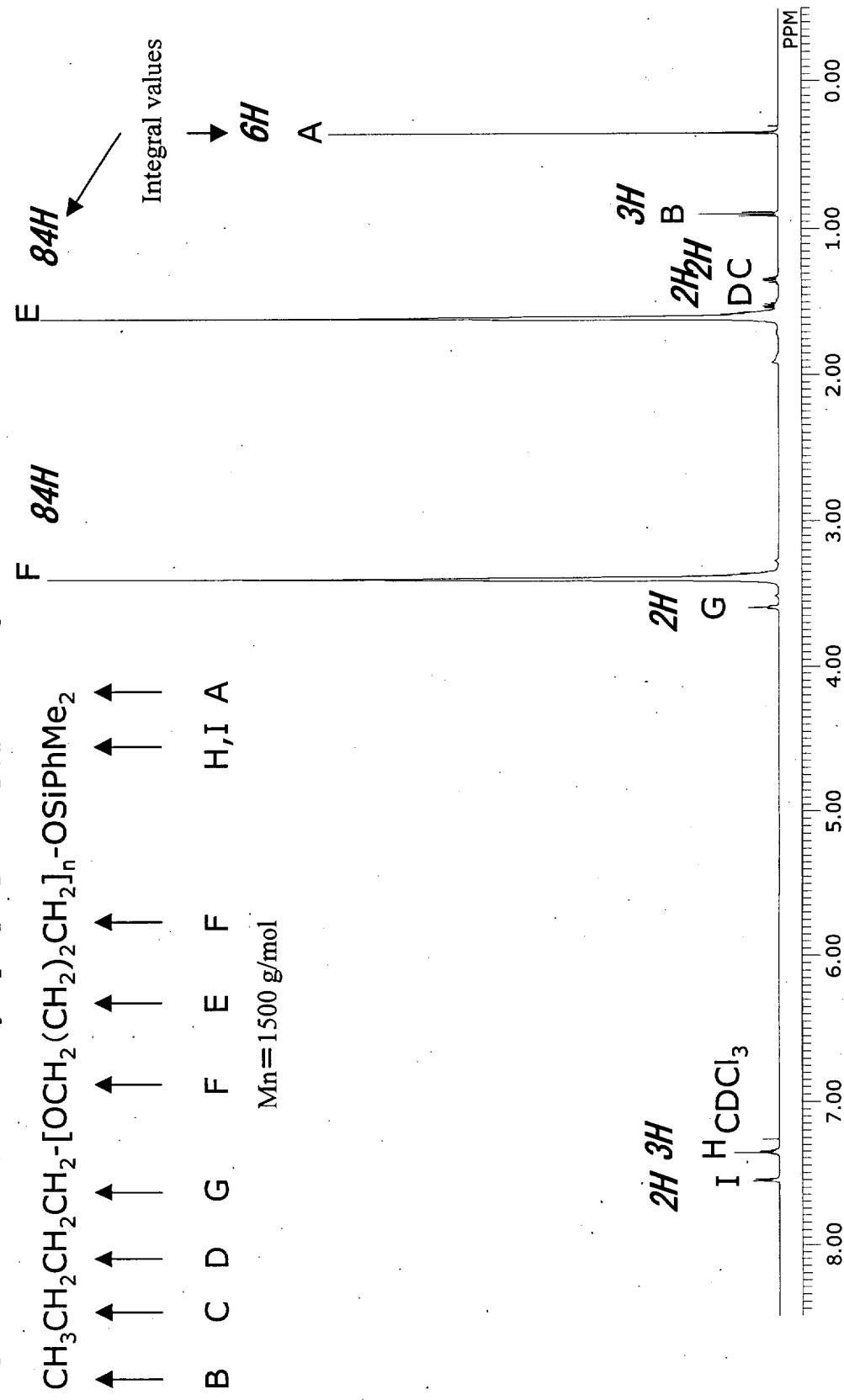


Figure S4 ^{13}C and ^{29}Si NMR spectra of $\text{CH}_3\text{CH}_2\text{CH}_2-\text{[O(CH}_2)_4\text{]}_{n-1}-\text{OSiPhMe}_2$

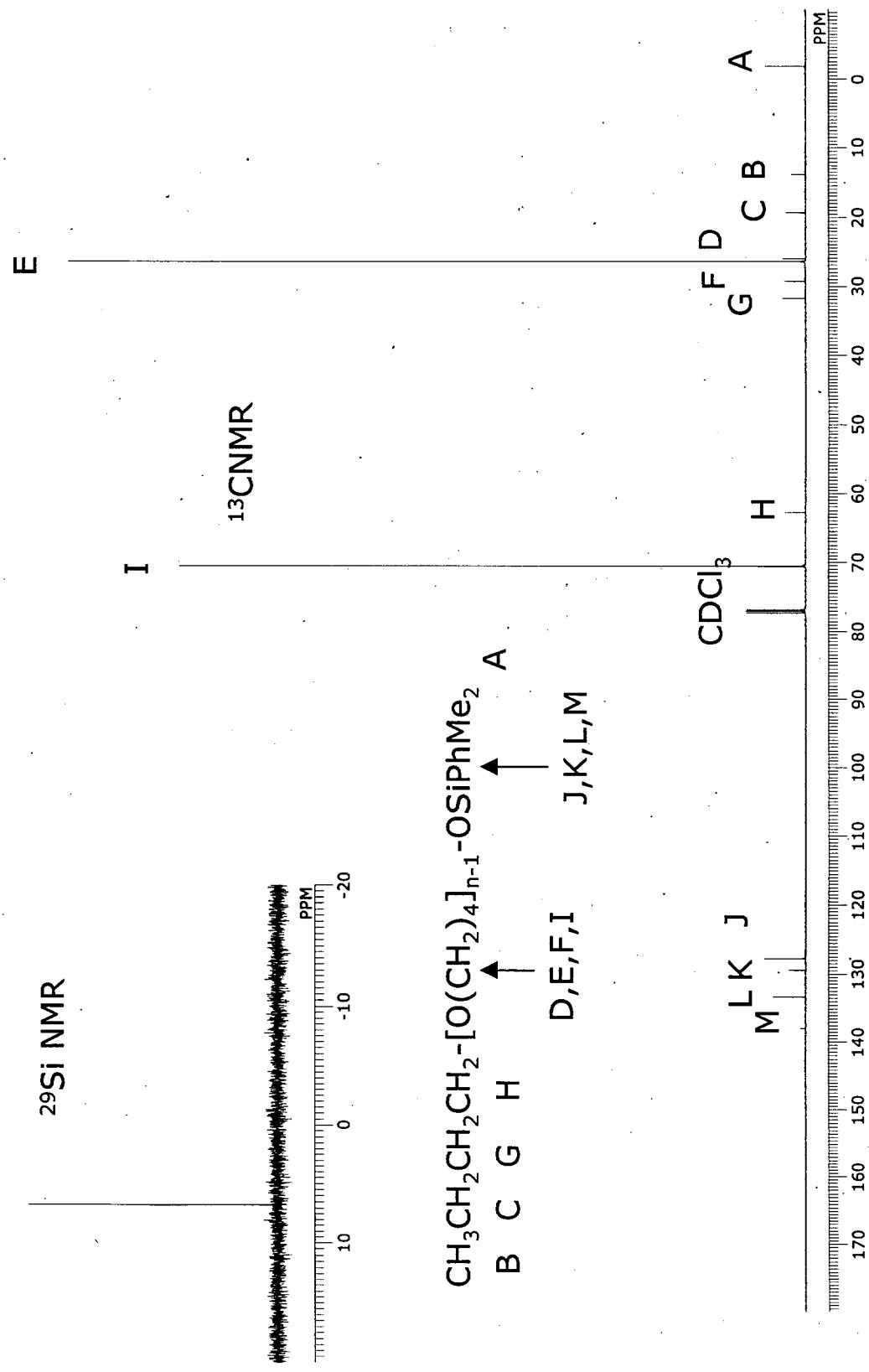


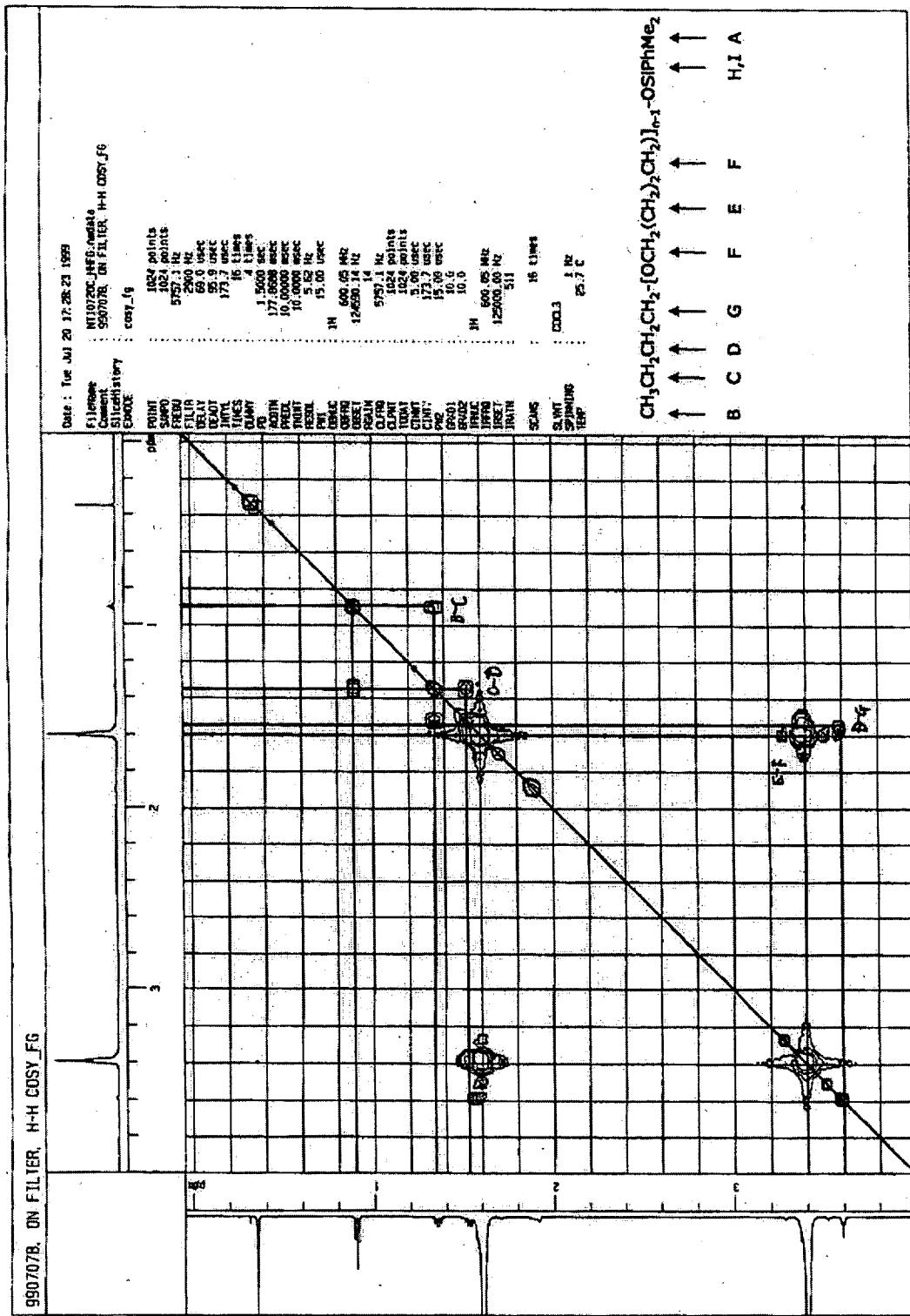
Figure S5 H-H COSY of $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-[O}(\text{CH}_2)_4\text{]}_n\text{-OSiPhMe}_2$ 

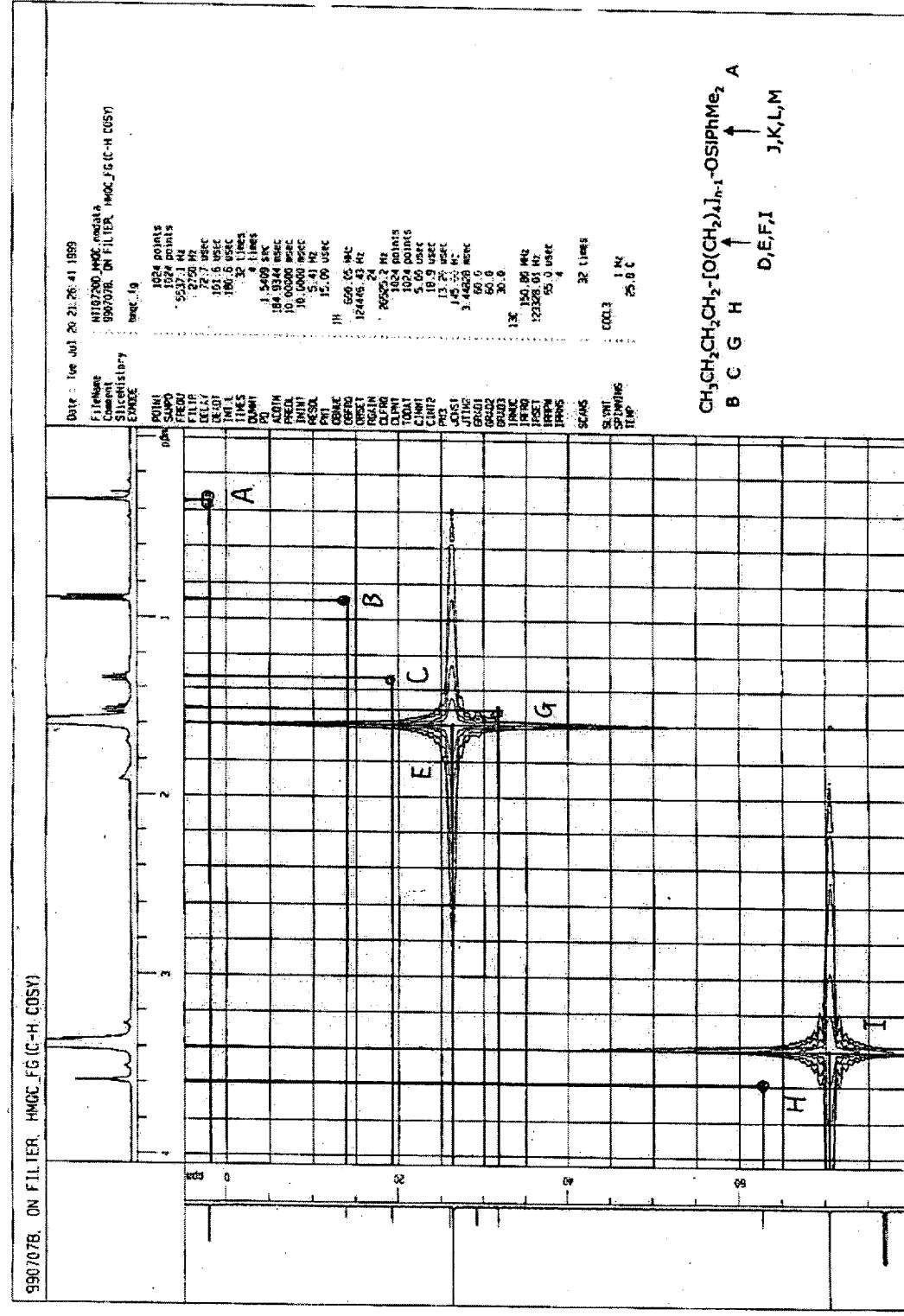
Figure S6 H-C COSY of $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-[O}(\text{CH}_2)_4\text{]-OSiPhMe}_2$ 

Figure S7 ^1H -NMR spectrum of D- $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{-}[\text{OCH}_2(\text{CH}_2)_2\text{CH}_2]\text{}_n\text{-OSiPhMe}_2$

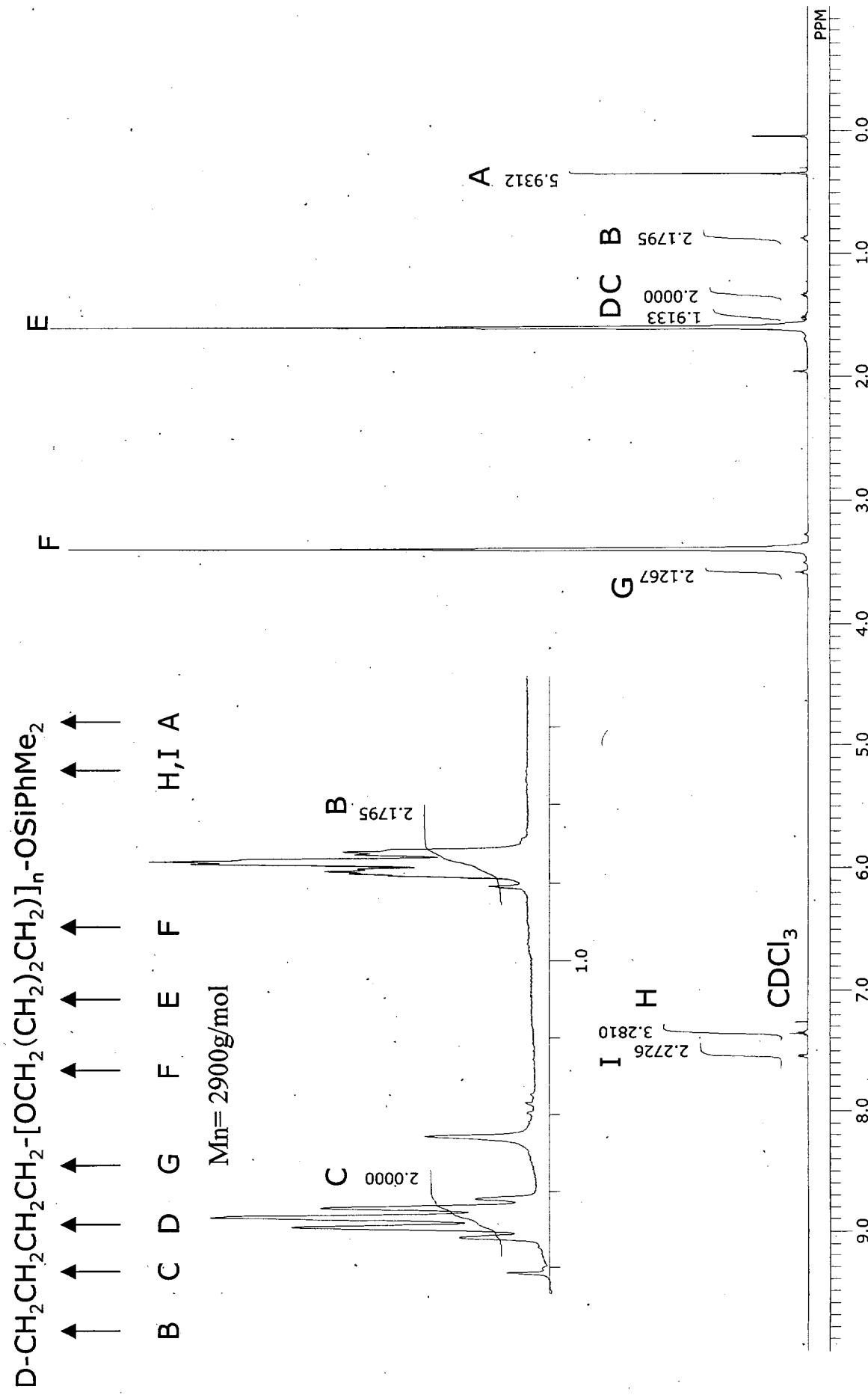


Figure S8 ^{13}C -NMR spectrum of D- $\text{CH}_3\text{CH}_2\text{CH}_2\text{-}[\text{O}(\text{CH}_2)_4]_n\text{-OSiPhMe}_2$

