Crystallographic Data 1

Ref. No.: JACS-ent-14

$C_{20}H_{32}O_4SSi$

STRUCTURAL REPORT

21-JUN-99

EXPERIMENTAL

DATA COLLECTION

A colorless plate of $C_{20}H_{32}O_4SSi$ having approximate dimensions of 0.38 x 0.30 x 0.25 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with Mo K_α radiation (λ = 0.71073 Å) on a Nonius KappaCCD.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 6700 reflections in the range $4 < \theta < 24°$. The orthorhombic cell parameters and calculated volume are: a = 10.072(1), b = 11.191(1), c = 19.830(2)Å, V = 2235.2Å^3. For Z = 4 and F.W. = 396.63 the calculated density is 1.18 g/cm^3. The space group was determined by the program ABSEN(ref 1). From the systematic presences of:

h00 h=2n
0k0 k=2n
00l l=2n

and from subsequent least-squares refinement, the space group was determined to be $P2_12_12_1$(# 19).

The data were collected at a temperature of 295 ± 1. Data were collected to a maximum 2θ of 48.3°.

DATA REDUCTION

A total of 6700 reflections were collected, of which 3473 were unique.

Lorentz and polarization corrections were applied to the data. The linear absorption coefficient is 2.1 /cm for Mo K radiation. An empirical absorption correction using SCALEPACK (ref 2) was applied. Transmission coefficients ranged from 0.755 to 0.949 with an average value of 0.890. Intensities of equivalent reflections were averaged. The agreement factor for the averaging was 5.1% based on intensity.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved by direct methods using SIR97 (ref 3). The remaining atoms were located in succeeding difference Fourier syntheses. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|^2-|Fc|^2)^2$ and the weight w is defined as $w=1/[\sigma^2(Fo^2)+(0.0360P)^2+0.0575P]$ where $P=(Fo^2+2Fc^2)/3$

Scattering factors were taken from the "International Tables for Crystallography" (ref 4). 3473 reflections were used in the refinements. However, only reflections with $F_o^2 > 2\sigma(F_o^2)$ were used in calculating R. The final cycle of refinement included 242 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, |Fo - Fc| \, / \, \Sigma \, Fo = 0.047$$

$$R2 = SQRT \left(\Sigma \, w \, (\, Fo^2 - Fc^2 \,)^2 \, / \, \Sigma \, w \, (Fo^2)^2 \, \right) = 0.099$$

The standard deviation of an observation of unit weight was 1.02. The highest peak in the final difference Fourier had a height of 0.19 e/A^3. The minimum negative peak had a height of -0.20 e/A^3. The factor for the determination of the absolute structure (ref 5) refined to 0.05.

Refinement was performed on a AlphaServer 2100 using SHELX-97 (ref 6). Crystallographic drawings were done using programs ORTEP (ref 7) ,and PLUTON (ref 8).

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(1) P. C. McArdle, J. Appl. Cryst., 239, 306 (1996).
(2) Z. Otwinowski and W. Minor, Methods Enzymol., 276, 307 (1996).
(3) A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, , A. G. G. Moliterni, M. C. Burla, G. Polidori, M. Camalli, and R. Spagna. , J. Appl. Cryst., 32, 115 (1999)
(4) "International Tables for Crystallography", Vol. C, Kluwer Academic Publishers, Dordrecht, The Netherlands, (199)2, Tables 4.2.6.8 and 6.1.1.4
(5) H. D. Flack, Acta Cryst., A39, 876 (1983).
(6) G. M. Sheldrick, SHELXS97. A Program for Crystal Structure Refinement. Univ. of Gottingen, Germany, (1997)
(7) C. K. Johnson, ORTEPII, Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA (1976)
(8) A. L. Spek, PLUTON. Molecular Graphics Program. Univ. of Ultrecht, The Netherlands (1991)

CRYSTALLOGRAPHIC DATA FOR

$C_{20}H_{32}O_4SSi$

$C_{20}H_{32}O_4SSi$

a = 10.0720(6)Å

b = 11.1909(11)Å

c = 19.8303(19)Å

V = 2235.2(6)Å^3

Z = 4

formula weight 396.63

space group $P2_12_12_1$ (No. 19)

T = 295. K

λ = 0.71073Å

ρ_{calc} = 1.179g cm^{-3}

μ = 0.210mm^{-1}

transmission coeff = 0.755-0.949

$R(F_o)^a$ = 0.047

$R_w(F_o^2)^b$ = 0.099

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[a] $R = \Sigma \, ||F_o| - |F_c|| / \Sigma \, |F_o|$ for $F_o^2 > 2\sigma(F_o^2)$

[b] $R_w = [\Sigma \, w \, (|F_o^2| - |F_c^2|)^2 / \Sigma \, w \, |F_o^2|^2]^{1/2}$

CRYSTAL DATA AND DATA COLLECTION PARAMETERS for

$C_{20}H_{32}O_4SSi$

formula	$C_{20}H_{32}O_4SSi$
formula weight	396.63
space group	$P2_12_12_1$ (No. 19)
a, Å	10.0720(6)
b, Å	11.1909(11)
c, Å	19.8303(19)
V, Å^3	2235.2(6)
Z	4
d_{calc}, g cm^{-3}	1.179
crystal dimensions, mm	0.38x0.30x0.25
temperature, K	295.
radiation (wavelength)	MO K_α(0.71073Å)
monochromator	graphite
linear abs coef, mm^{-1}	0.210
absorption correction applied	empirical[a]
transmission factors: min, max	0.76, 0.95
diffractometer	Nonius KappaCCD
h, k, l range	-11 to 11 -12 to 12 -22 to 22
2θ range, deg	8.00-48.25
programs used	SHELXL-97
F_{000}	856.0
weighting	
$w=1/[\sigma^2(Fo^2)+(0.0360P)^2+0.0575P]$ where $P=(Fo^2+2Fc^2)/3$	
data collected	6700
unique data	3473
R_{int}	0.051
data used in refinement	3473
cutoff used in R-factor calculations	$F_o^2>2.0\sigma(F_o^2)$
data with $I>2.0\sigma(I)$	2712
number of variables	242
largest shift/esd in final cycle	0.00
$R(F_o)$	0.047
$R_w(F_o^2)$	0.099
goodness of fit	1.024
absolute structure determination	Flack parameter[b] (0.1(1))

[a] Otwinowski Z. & Minor, W. Methods Enzymol., **1996**, 276, 307.

Positional Parameters and Their Estimated Standard Deviations

for $C_{20}H_{32}O_4SSi$

Atom	x	y	z	U(Å²)
S(31)	0.32131(8)	-0.03556(7)	0.07844(4)	0.0561(2)
Si(11)	0.80971(10)	0.16044(8)	0.13036(5)	0.0613(3)
O(11)	0.7209(2)	0.04518(19)	0.10360(10)	0.0595(7)
O(31)	0.2492(2)	-0.1327(2)	0.10890(12)	0.0749(8)
O(32)	0.2659(3)	0.0208(2)	0.01902(11)	0.0683(8)
O(51)	0.6157(3)	-0.38328(19)	0.07889(13)	0.0833(9)
C(1)	0.7104(4)	0.0100(3)	0.03420(15)	0.0613(12)
C(2)	0.5637(4)	0.0024(3)	0.01444(15)	0.0545(10)
C(3)	0.4832(3)	-0.0850(3)	0.05564(14)	0.0494(9)
C(4)	0.5088(3)	-0.1988(3)	0.07030(15)	0.0549(9)
C(5)	0.6273(4)	-0.2684(3)	0.04863(17)	0.0637(12)
C(6)	0.7586(4)	-0.2104(3)	0.0652(2)	0.0743(14)
C(7)	0.7942(4)	-0.1022(3)	0.0229(2)	0.0763(14)
C(11)	0.8497(3)	0.1288(3)	0.22094(19)	0.0663(12)
C(12)	0.7204(4)	0.1181(4)	0.2615(2)	0.0907(15)
C(13)	0.9330(5)	0.2309(4)	0.2510(3)	0.110(2)
C(14)	0.9275(6)	0.0116(4)	0.2258(2)	0.1030(17)
C(15)	0.9639(4)	0.1761(4)	0.0792(3)	0.0973(15)
C(16)	0.7107(5)	0.2993(3)	0.1216(2)	0.0920(16)
C(21)	0.5454(4)	-0.0210(3)	-0.06141(15)	0.0755(13)
C(31)	0.3403(3)	0.0776(3)	0.13940(16)	0.0570(10)
C(32)	0.3354(4)	0.1964(3)	0.1205(2)	0.0723(12)
C(33)	0.3444(4)	0.2837(4)	0.1696(3)	0.0920(17)
C(34)	0.3589(5)	0.2511(6)	0.2366(3)	0.102(2)
C(35)	0.3634(5)	0.1355(6)	0.2546(2)	0.105(2)
C(36)	0.3537(4)	0.0457(4)	0.20608(18)	0.0783(14)

$U_{eq} = (1/3)\Sigma_i\Sigma_j\ U_{ij}a^*_i a^*_j \mathbf{a}_i \cdot \mathbf{a}_j$

Positional Parameters and Their Estimated Standard Deviations

for $C_{20}H_{32}O_4SSi$

Atom	x	y	z	U(Å²)
H(1)	0.750	0.074	0.007	0.080
H(2)	0.526	0.082	0.023	0.071
H(4)	0.447	-0.239	0.097	0.071
H(5)	0.623	-0.279	0.000	0.083
H(32)	0.326	0.217	0.075	0.094
H(33)	0.341	0.364	0.158	0.119
H(34)	0.366	0.310	0.269	0.133
H(35)	0.373	0.115	0.300	0.136
H(36)	0.356	-0.034	0.218	0.102
H(51)	0.650	-0.433	0.054	0.108
H(6A)	0.828	-0.270	0.060	0.097
H(6B)	0.757	-0.187	0.112	0.097
H(7A)	0.787	-0.124	-0.024	0.099
H(7B)	0.886	-0.082	0.032	0.099
H(12A)	0.664	0.059	0.241	0.117
H(12B)	0.675	0.194	0.262	0.117
H(12C)	0.741	0.095	0.307	0.117
H(13A)	0.955	0.212	0.297	0.142
H(13B)	0.883	0.304	0.249	0.142
H(13C)	1.013	0.240	0.225	0.142
H(14A)	0.950	-0.003	0.272	0.134
H(14B)	1.007	0.018	0.199	0.134
H(14C)	0.874	-0.053	0.209	0.134
H(15A)	1.015	0.104	0.083	0.126
H(15B)	1.015	0.242	0.096	0.126
H(15C)	0.941	0.190	0.033	0.126
H(16A)	0.686	0.310	0.075	0.119
H(16B)	0.763	0.366	0.137	0.119
H(16C)	0.632	0.293	0.149	0.119
H(21A)	0.571	-0.102	-0.071	0.098
H(21B)	0.600	0.033	-0.087	0.098
H(21C)	0.454	-0.009	-0.073	0.098

Hydrogens included in calculation of structure factors but not refined
$B_{iso}(H) = 1.3 * B_{iso}(C)$

Anisotropic Temperature Factor Coefficients - U's

for $C_{20}H_{32}O_4SSi$

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
S(31)	0.0589(5)	0.0591(5)	0.0503(4)	0.0023(4)	-0.0020(4)	-0.0080(4)
Si(11)	0.0586(5)	0.0501(5)	0.0753(6)	-0.0033(5)	0.0052(5)	0.0063(5)
O(11)	0.0645(15)	0.0563(13)	0.0576(13)	-0.0074(11)	0.0061(10)	-0.0034(10)
O(31)	0.0683(15)	0.0735(16)	0.0830(17)	-0.0152(13)	0.0128(13)	-0.0009(13)
O(32)	0.0771(17)	0.0749(15)	0.0528(13)	0.0142(13)	-0.0187(11)	-0.0067(12)
O(51)	0.137(2)	0.0467(15)	0.0663(16)	0.0230(14)	0.0135(16)	0.0021(12)
C(1)	0.072(3)	0.060(2)	0.0518(19)	-0.0022(17)	0.0155(17)	-0.0018(16)
C(2)	0.068(2)	0.048(2)	0.0475(17)	0.0066(15)	0.0082(16)	-0.0009(14)
C(3)	0.063(2)	0.0501(19)	0.0351(16)	0.0006(15)	-0.0003(14)	-0.0063(13)
C(4)	0.075(2)	0.053(2)	0.0367(16)	0.0020(16)	-0.0016(16)	-0.0059(15)
C(5)	0.094(3)	0.048(2)	0.0491(18)	0.0152(19)	0.0045(18)	-0.0060(16)
C(6)	0.080(3)	0.066(2)	0.077(3)	0.029(2)	0.002(2)	-0.009(2)
C(7)	0.071(3)	0.075(2)	0.083(3)	0.014(2)	0.016(2)	-0.009(2)
C(11)	0.063(2)	0.051(2)	0.085(3)	-0.0048(17)	-0.0100(18)	-0.0060(18)
C(12)	0.096(3)	0.099(3)	0.077(3)	-0.011(2)	0.006(2)	0.008(2)
C(13)	0.111(4)	0.099(4)	0.119(4)	-0.040(3)	-0.012(3)	-0.020(3)
C(14)	0.122(4)	0.084(3)	0.103(3)	0.024(3)	-0.029(3)	0.009(3)
C(15)	0.075(3)	0.106(3)	0.111(3)	-0.023(2)	0.022(3)	0.009(3)
C(16)	0.094(3)	0.061(2)	0.121(4)	0.006(2)	0.003(3)	0.021(2)
C(21)	0.104(3)	0.077(3)	0.0454(19)	-0.001(2)	0.0104(18)	0.0016(18)
C(31)	0.053(2)	0.066(2)	0.052(2)	0.0083(16)	0.0016(16)	-0.0181(16)
C(32)	0.075(3)	0.071(2)	0.071(2)	0.011(2)	0.0045(19)	-0.019(2)
C(33)	0.089(3)	0.075(3)	0.112(4)	0.016(2)	-0.003(3)	-0.037(3)
C(34)	0.081(3)	0.131(5)	0.094(4)	0.017(3)	-0.005(3)	-0.061(3)
C(35)	0.109(4)	0.150(5)	0.055(3)	0.020(4)	-0.006(2)	-0.034(3)
C(36)	0.084(3)	0.099(3)	0.052(2)	0.009(2)	0.0004(18)	-0.012(2)

The form of the anisotropic temperature factor is:

$\exp[-2\pi \{h^2a^{*2}U(1,1) + k^2b^{*2}U(2,2) + l^2c^{*2}U(3,3) + 2hka^*b^*U(1,2) + 2hla^*c^*U(1,3) + 2klb^*c^*U(2,3)\}]$ where a^*, b^*, and c^* are reciprocal lattice constants.

Crystallographic Data 2

Ref. No.: JACS-ent-19

$C_{14}H_{16}O_6S_2$

STRUCTURAL REPORT

23-JUN-99

EXPERIMENTAL

DATA COLLECTION

A colorless chunk of $C_{14}H_{16}O_5S_2$ having approximate dimensions of 0.38 x 0.35 x 0.25 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with Mo K_α radiation (λ = 0.71073 Å) on a Nonius KappaCCD.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 6029 reflections in the range $4 < \theta < 27°$. The monoclinic cell parameters and calculated volume are: a = 9.697(1), b = 8.837(1), c = 10.196(1) Å, β = 116.92(0)°, V = 779.2Å^3. For Z = 2 and F.W. = 344.41 the calculated density is 1.47 g/cm^3. The space group was determined by the program ABSEN(ref 1). From the systematic presences of:

0k0 k=2n

and from subsequent least-squares refinement, the space group was determined to be $P2_1$(# 4).

The data were collected at a temperature of 193 ± 1. Data were collected to a maximum 2θ of 55.0°.

DATA REDUCTION

A total of 6029 reflections were collected, of which 3402 were unique.

Lorentz and polarization corrections were applied to the data. The linear absorption coefficient is 3.5 /cm for Mo K radiation. An empirical absorption correction using SCALEPACK (ref 2) was applied. Transmission coefficients ranged from 0.703 to 0.916 with an average value of 0.846. Intensities of equivalent reflections were averaged. The agreement factor for the averaging was 4.4% based on intensity.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved by direct methods using SIR97 (ref 3). The remaining atoms were located in succeeding difference Fourier syntheses. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|^2-|Fc|^2)^2$ and the weight w is defined as $w=1/[\sigma^2(Fo^2)+(0.0449P)^2+0.5109P]$ where $P=(Fo^2+2Fc^2)/3$

Scattering factors were taken from the "International Tables for Crystallography" (ref 4). 3402 reflections were used in the

refinements. However, only reflections with $F_o^2 > 2\sigma(F_o^2)$ were used in calculating R. The final cycle of refinement included 200 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, |Fo - Fc| \, / \, \Sigma \, Fo = 0.048$$

$$R2 = SQRT \left(\Sigma \, w \, (Fo^2 - Fc^2)^2 \, / \, \Sigma \, w \, (Fo^2)^2 \right) = 0.125$$

The standard deviation of an observation of unit weight was 1.14. The highest peak in the final difference Fourier had a height of 0.33 e/A^3. The minimum negative peak had a height of -0.52 e/A^3. The factor for the determination of the absolute structure (ref 5) refined to -0.11.

Refinement was performed on a AlphaServer 2100 using SHELX-97 (ref 6). Crystallographic drawings were done using programs ORTEP (ref 7) ,and PLUTON (ref 8).

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(1) P. C. McArdle, J. Appl. Cryst., 239, 306 (1996).
(2) Z. Otwinowski and W. Minor, Methods Enzymol., 276, 307 (1996).
(3) A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, , A. G. G. Moliterni, M. C. Burla, G. Polidori, M. Camalli, and R. Spagna. , J. Appl. Cryst., 32, 115 (1999)
(4) "International Tables for Crystallography", Vol. C, Kluwer Academic Publishers, Dordrecht, The Netherlands, (1992), Tables 4.2.6.8 and 6.1.1.4
(5) H. D. Flack, Acta Cryst., A39, 876 (1983).
(6) G. M. Sheldrick, SHELXS97. A Program for Crystal Structure Refinement. Univ. of Gottingen, Germany, (1997)
(7) C. K. Johnson, ORTEPII, Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA (1976
(8) A. L. Spek, PLUTON. Molecular Graphics Program. Univ. of Ultrecht, The Netherlands (1991)

CRYSTALLOGRAPHIC DATA FOR

$C_{14}H_{16}O_6S_2$

$C_{14}H_{16}O_6S_2$	formula weight 344.41
a = 9.6974(8)Å	space group $P2_1$ (No. 4)
b = 8.8375(5)Å	T = 193. K
c = 10.1963(9)Å	λ = 0.71073Å
β = 116.916(3)°	ρ_{calc} = 1.468g cm^{-3}
V = 779.2(2)$Å^3$	μ = 0.351mm^{-1}
Z = 2	transmission coeff = 0.703-0.916
	$R(F_o)^a$ = 0.048
	$R_w(F_o^2)^b$ = 0.125

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[a] $R = \Sigma \; ||F_o| - |F_c|| / \Sigma \; |F_o|$ for $F_o^2 > 2\sigma(F_o^2)$

[b] $R_w = [\Sigma \; w \; (|F_o^2| - |F_c^2|)^2 / \Sigma \; w \; |F_o^2|^2]^{1/2}$

CRYSTAL DATA AND DATA COLLECTION PARAMETERS for

$C_{14}H_{6}O_{6}S_{2}$

formula	$C_{14}H_{16}O_{6}S_{2}$
formula weight	344.41
space group	$P2_1$ (No. 4)
a, Å	9.6974(8)
b, Å	8.8375(5)
c, Å	10.1963(9)
β, deg	116.916(3)
V, Å^3	779.2(2)
Z	2
d_{calc}, g cm^{-3}	1.468
crystal dimensions, mm	0.38x0.35x0.25
temperature, K	193.
radiation (wavelength)	MO K_α(0.71073Å)
monochromator	graphite
linear abs coef, mm^{-1}	0.351
absorption correction applied	empirical[a]
transmission factors: min, max	0.70, 0.92
diffractometer	Nonius KappaCCD
h, k, l range	-12 to 12 -11 to 10 -13 to 13
2θ range, deg	8.00-55.00
programs used	SHELXL-97
F_{000}	360.0
weighting	
$w=1/[\sigma^2(Fo^2)+(0.0449P)^2+0.5109P]$ where $P=(Fo^2+2Fc^2)/3$	
data collected	6029
unique data	3402
R_{int}	0.044
data used in refinement	3402
cutoff used in R-factor calculations	$F_o^2>2.0\sigma(F_o^2)$
data with $I>2.0\sigma(I)$	3064
number of variables	200
largest shift/esd in final cycle	0.00
$R(F_o)$	0.048
$R_w(F_o^2)$	0.125
goodness of fit	1.143
absolute structure determination	Flack parameter[b] (-0.1(1))

[a] Otwinowski Z. & Minor, W. Methods Enzymol., **1996**, 276, 307.

Positional Parameters and Thei: Estimated Standard Deviations

for $C_{14}H_{16}C_5S_2$

Atom	x	y	z	U(Å²)
S(11)	0.38994(12)	1.24176(1))	0.34245(9)	0.0406(3)
S(22)	0.30330(9)	1.20410(9	-0.11408(8)	0.0289(3)
O(11)	0.3998(4)	1.2293(4)	0.4869(3)	0.0554(11)
O(12)	0.3253(4)	1.3766(3)	0.2586(3)	0.0531(11)
O(21)	0.3358(2)	1.1215(3)	0.0347(2)	0.0285(8)
O(23)	0.4313(3)	1.1625(3)	-0.1387(3)	0.0404(10)
O(24)	0.1497(3)	1.1676(3)	-0.2221(3)	0.0362(8)
O(36)	0.0315(3)	0.9122(3)	0.0784(3)	0.0380(9)
C(1)	0.2769(4)	1.0880(4)	0.2371(4)	0.0313(12)
C(2)	0.2071(4)	1.0968(4)	0.0715(3)	0.0274(10)
C(3)	0.1242(4)	0.9472(4)	0.0051(4)	0.0329(12)
C(4)	0.2319(5)	0.8125(5)	0.0351(5)	0.0465(16)
C(5)	0.2305(4)	0.7358(5)	0.1719(5)	0.0475(13)
C(6)	0.1435(4)	0.8497(5)	0.2171(4)	0.0408(13)
C(7)	0.2469(4)	0.9747(5)	0.3051(4)	0.0372(12)
C(11)	0.5797(4)	1.2159(4)	0.3619(3)	0.0397(12)
C(12)	0.6585(5)	1.0844(5)	0.4290(5)	0.0522(19)
C(13)	0.8095(6)	1.0704(7)	0.4544(6)	0.0646(19)
C(14)	0.8828(6)	1.1843(7)	0.4159(5)	0.0657(19)
C(15)	0.8018(6)	1.3141(7)	0.3498(5)	0.0643(19)
C(16)	0.6500(6)	1.3310(5)	0.3227(4)	0.0542(17)
C(25)	0.3132(5)	1.3965(4)	-0.0711(4)	0.0383(13)

$U_{eq} = (1/3)\Sigma_i\Sigma_j\ U_{ij}a^*_i a^*_j \mathbf{a}_i \cdot \mathbf{a}_j$

Positional Parameters and Their Estimated Standard Deviations

for $C_{14}H_{16}O_6S_2$

Atom	x	y	z	U(Å²)
H(2)	0.134	1.181	0.035	0.036
H(3)	0.058	0.959	-0.101	0.043
H(6)	0.093	0.801	0.270	0.053
H(7)	0.290	0.973	0.407	0.048
H(12)	0.610	1.008	0.456	0.068
H(13)	0.864	0.983	0.498	0.084
H(14)	0.985	1.173	0.434	0.086
H(15)	0.850	1.391	0.323	0.084
H(16)	0.596	1.419	0.279	0.070
H(4A)	0.335	0.845	0.056	0.060
H(4B)	0.194	0.744	-0.048	0.060
H(5A)	0.177	0.639	0.146	0.062
H(5B)	0.335	0.721	0.249	0.062
H(25A)	0.414	1.420	0.006	0.050
H(25B)	0.237	1.420	-0.039	0.050
H(25C)	0.294	1.455	-0.157	0.050

Hydrogens included in calculation of structure factors but not refined
$B_{iso}(H)=1.3*B_{iso}(C)$

Anisotropic Temperature Factor Coefficients - U's

for $C_{14}H_{16}O_6S_2$

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
S(11)	0.0607(6)	0.0256(5)	0.0281(4)	-0.0011(4)	0.0136(4)	-0.0029(3)
S(22)	0.0274(4)	0.0319(4)	0.0277(4)	0.0000(3)	0.0127(3)	0.0000(3)
O(11)	0.084(2)	0.0479(19)	0.0327(13)	-0.0025(16)	0.0250(14)	-0.0082(14)
O(12)	0.078(2)	0.0228(14)	0.0429(17)	0.0063(13)	0.0137(15)	0.0003(12)
O(21)	0.0240(11)	0.0303(13)	0.0284(12)	-0.0001(9)	0.0094(9)	0.0041(10)
O(23)	0.0381(13)	0.0463(17)	0.0450(15)	-0.0017(11)	0.0259(12)	-0.0034(12)
O(24)	0.0300(12)	0.0440(16)	0.0290(12)	-0.0024(10)	0.0085(10)	-0.0031(10)
O(36)	0.0235(12)	0.0409(15)	0.0452(15)	-0.0008(10)	0.0116(11)	0.0105(11)
C(1)	0.0370(19)	0.0274(18)	0.0298(17)	0.0062(13)	0.0153(15)	0.0045(14)
C(2)	0.0241(15)	0.0270(17)	0.0284(16)	0.0007(12)	0.0095(13)	0.0012(13)
C(3)	0.0289(17)	0.0309(19)	0.0379(19)	-0.0011(13)	0.0143(15)	0.0013(14)
C(4)	0.049(2)	0.0259(19)	0.063(3)	0.0026(17)	0.024(2)	-0.0041(18)
C(5)	0.0371(18)	0.027(2)	0.061(2)	-0.0017(15)	0.0070(17)	0.0094(19)
C(6)	0.0328(19)	0.040(2)	0.044(2)	-0.0034(15)	0.0125(16)	0.0156(18)
C(7)	0.0379(19)	0.039(2)	0.0344(19)	0.0035(15)	0.0162(16)	0.0087(15)
C(11)	0.053(2)	0.0264(19)	0.0254(15)	-0.0139(16)	0.0053(15)	0.0007(15)
C(12)	0.050(3)	0.043(3)	0.052(3)	-0.0048(19)	0.013(2)	0.010(2)
C(13)	0.050(3)	0.061(3)	0.061(3)	-0.004(2)	0.006(2)	0.012(3)
C(14)	0.050(3)	0.088(4)	0.045(2)	-0.020(3)	0.009(2)	0.004(3)
C(15)	0.056(3)	0.084(4)	0.037(2)	-0.034(3)	0.007(2)	0.006(2)
C(16)	0.068(3)	0.047(3)	0.030(2)	-0.020(2)	0.0067(19)	0.0048(18)
C(25)	0.044(2)	0.032(2)	0.041(2)	0.0018(16)	0.0211(17)	0.0061(16)

The form of the anisotropic temperature factor is:

$\exp[-2\pi \{h^2a^{*2}U(1,1) + k^2b^{*2}U(2,2) + l^2c^{*2}U(3,3) + 2hka^*b^*U(1,2) + 2hla^*c^*U(1,3) + 2klb^*c^*U(2,3)\}]$ where a^*, b^*, and c^* are reciprocal lattice constants.

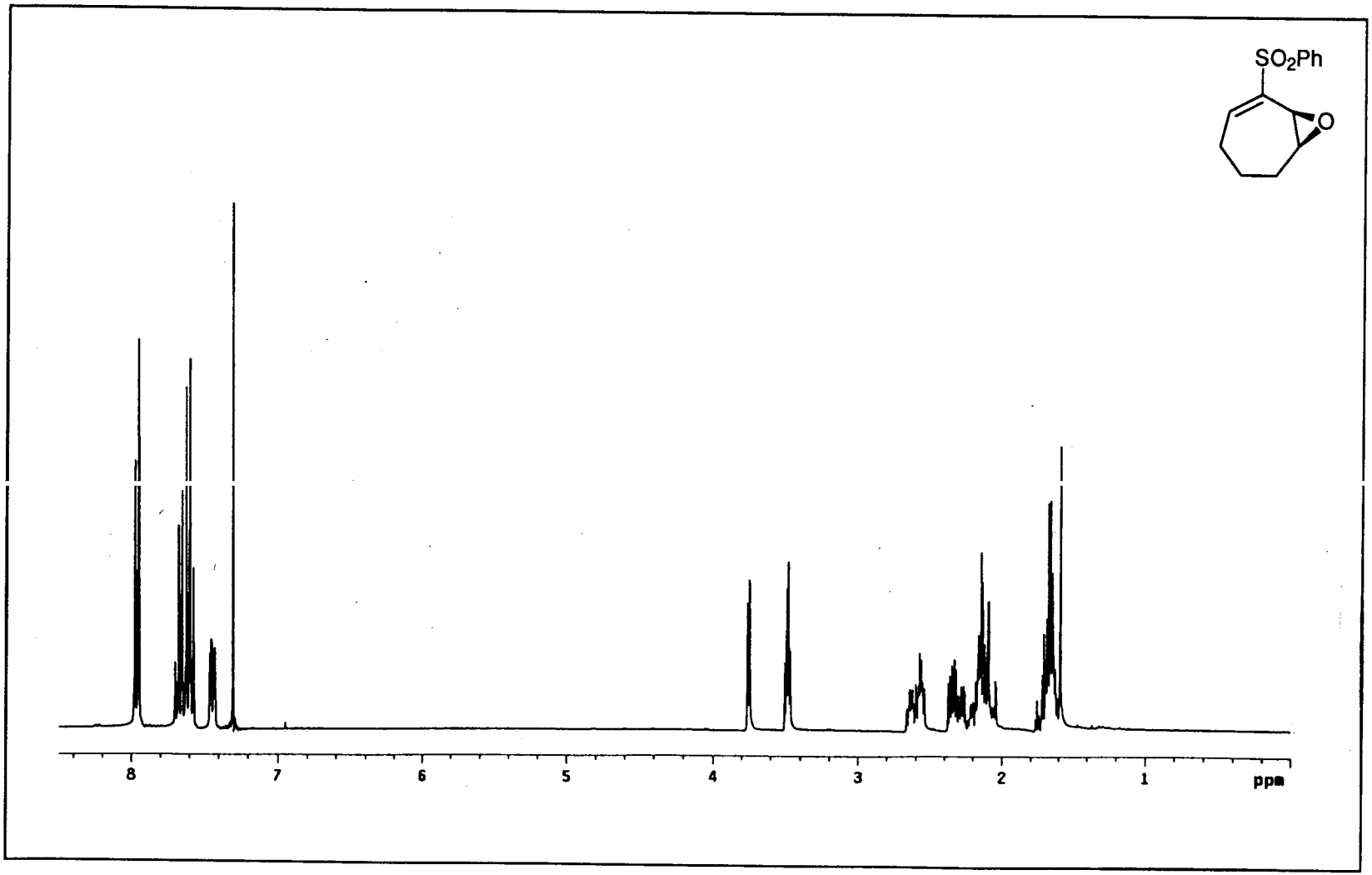

Figure 1 300MHz ^{1}H NMR of compound **5b** in $CDCl_3$

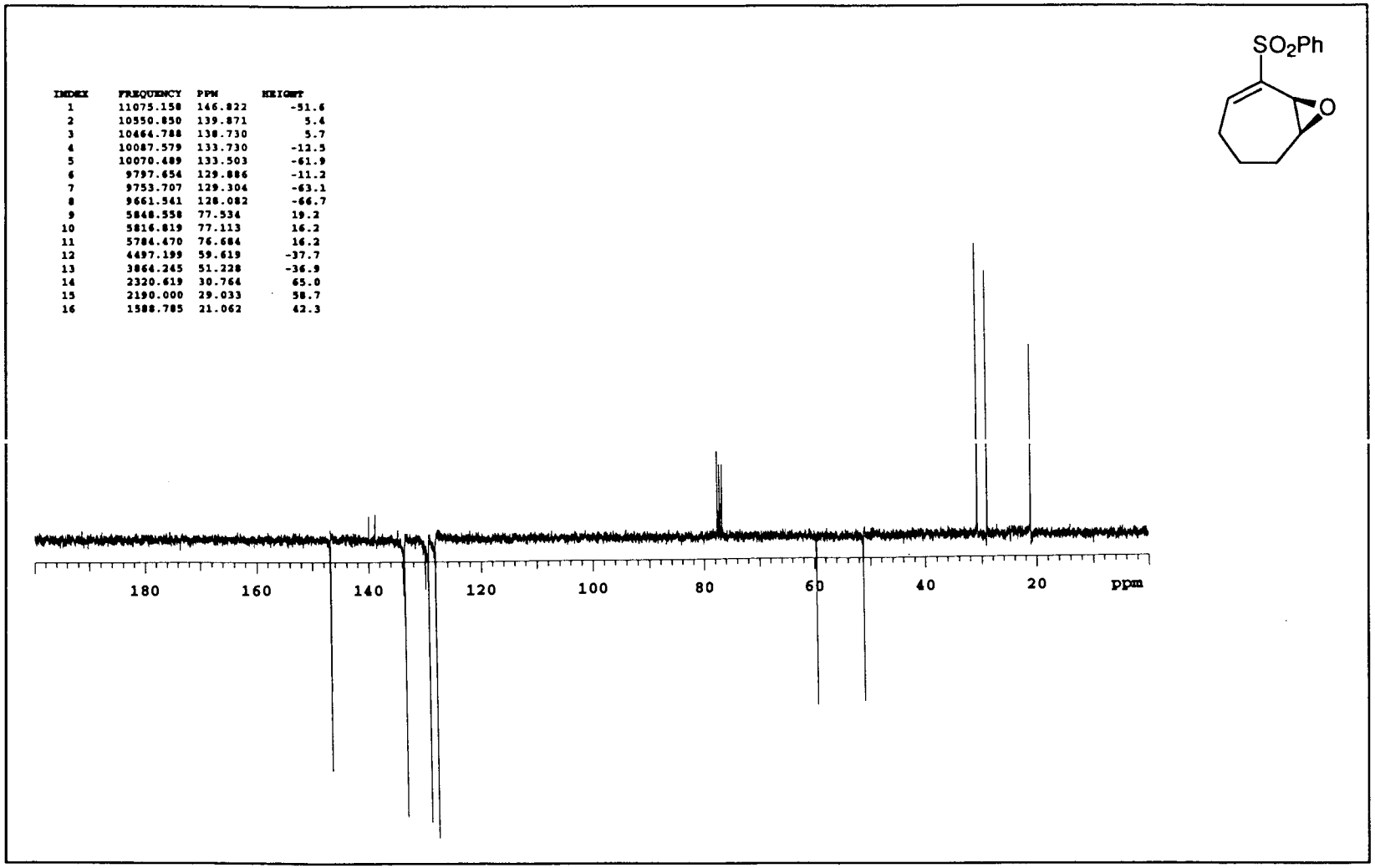

Figure 2 75MHz ^{13}C NMR of compound **5b** in $CDCl_3$

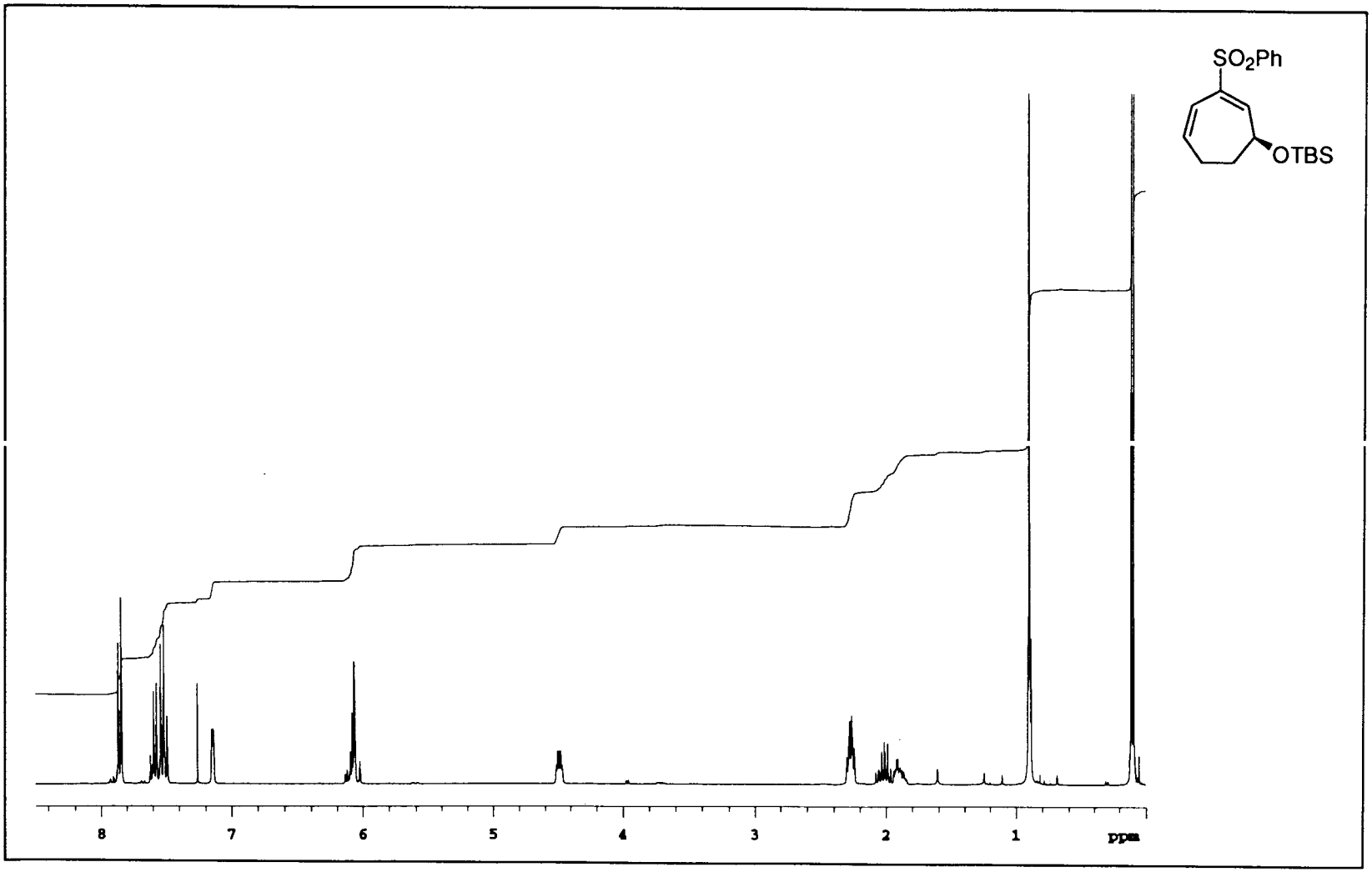

Figure 3 300MHz ^{1}H NMR of compound **6b** in $CDCl_3$

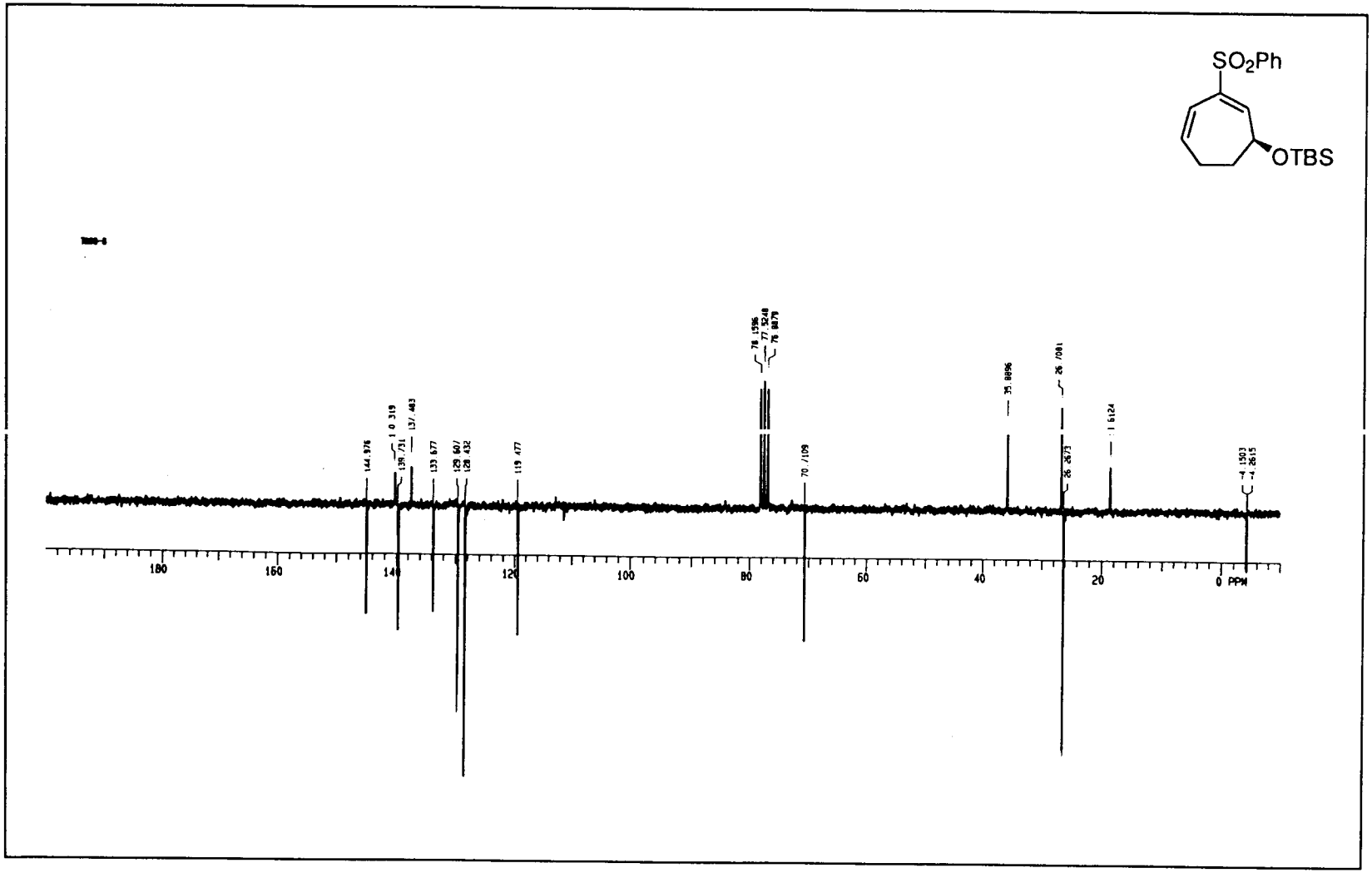

Figure 4 75MHz ^{13}C NMR of compound **6b** in $CDCl_3$

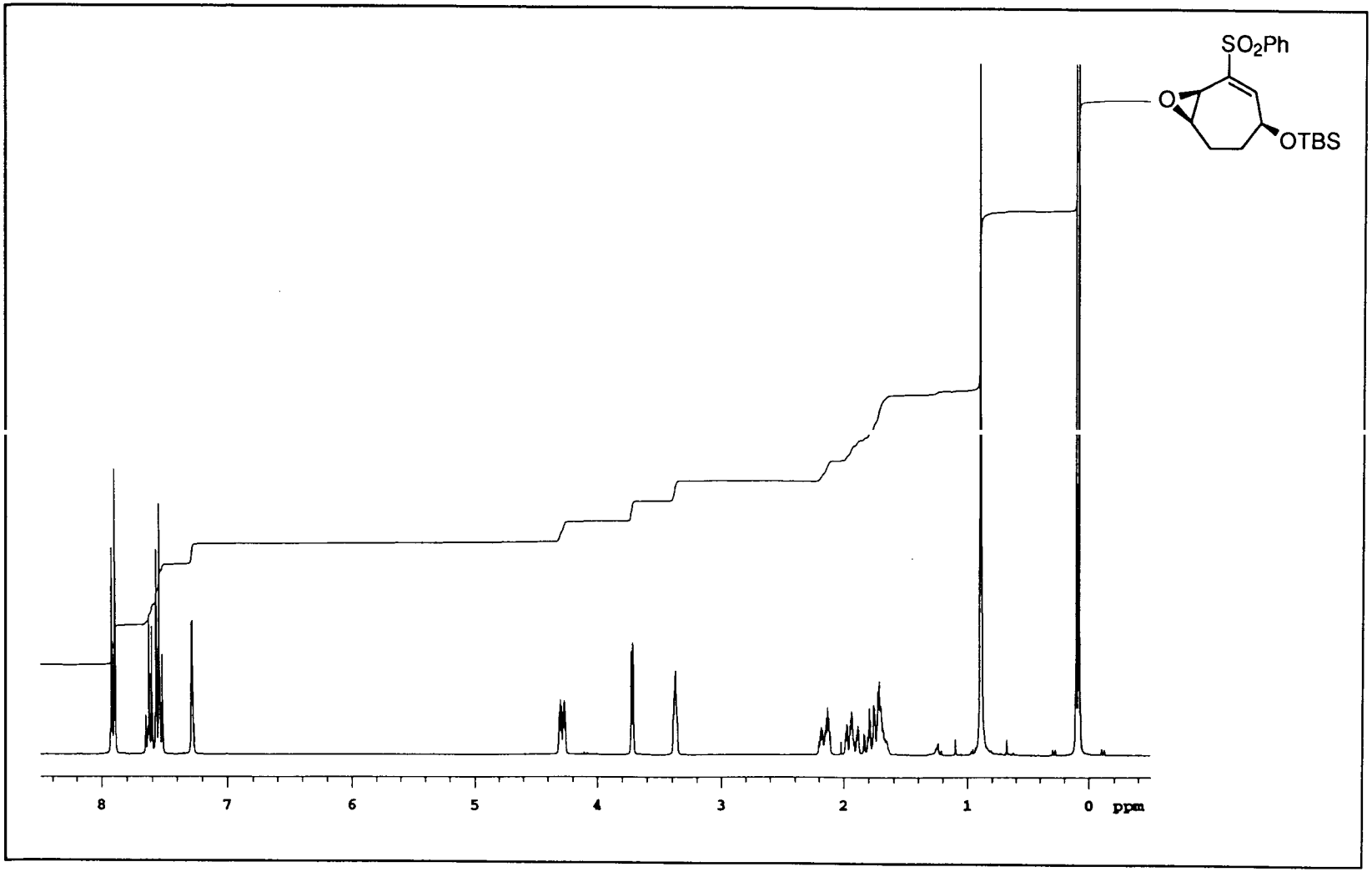

Figure 5 300MHz ^{1}H NMR of compound **syn-7b** in $CDCl_3$

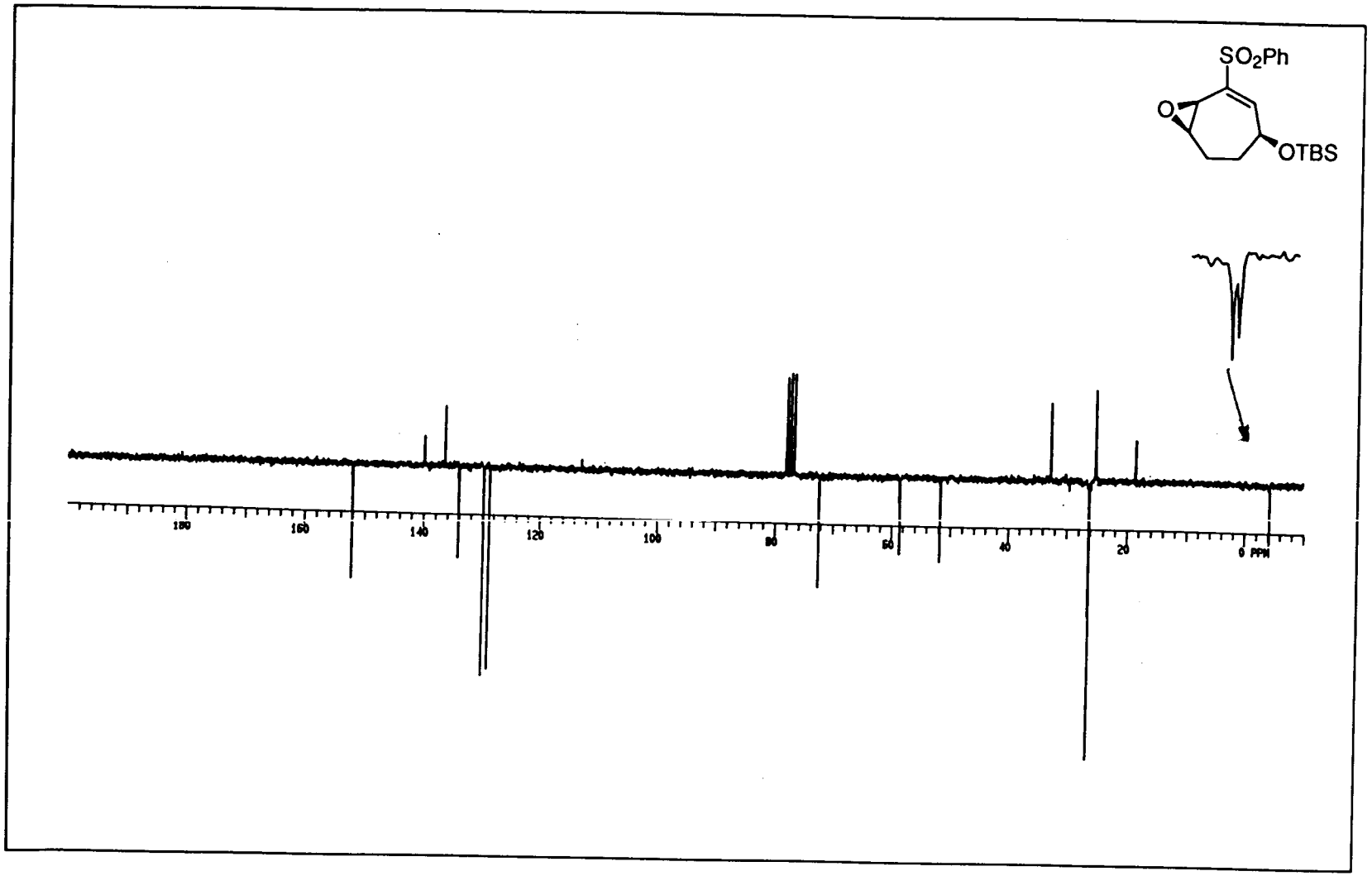

Figure 6 75MHz ^{13}C NMR of compound **syn-7b** in $CDCl_3$

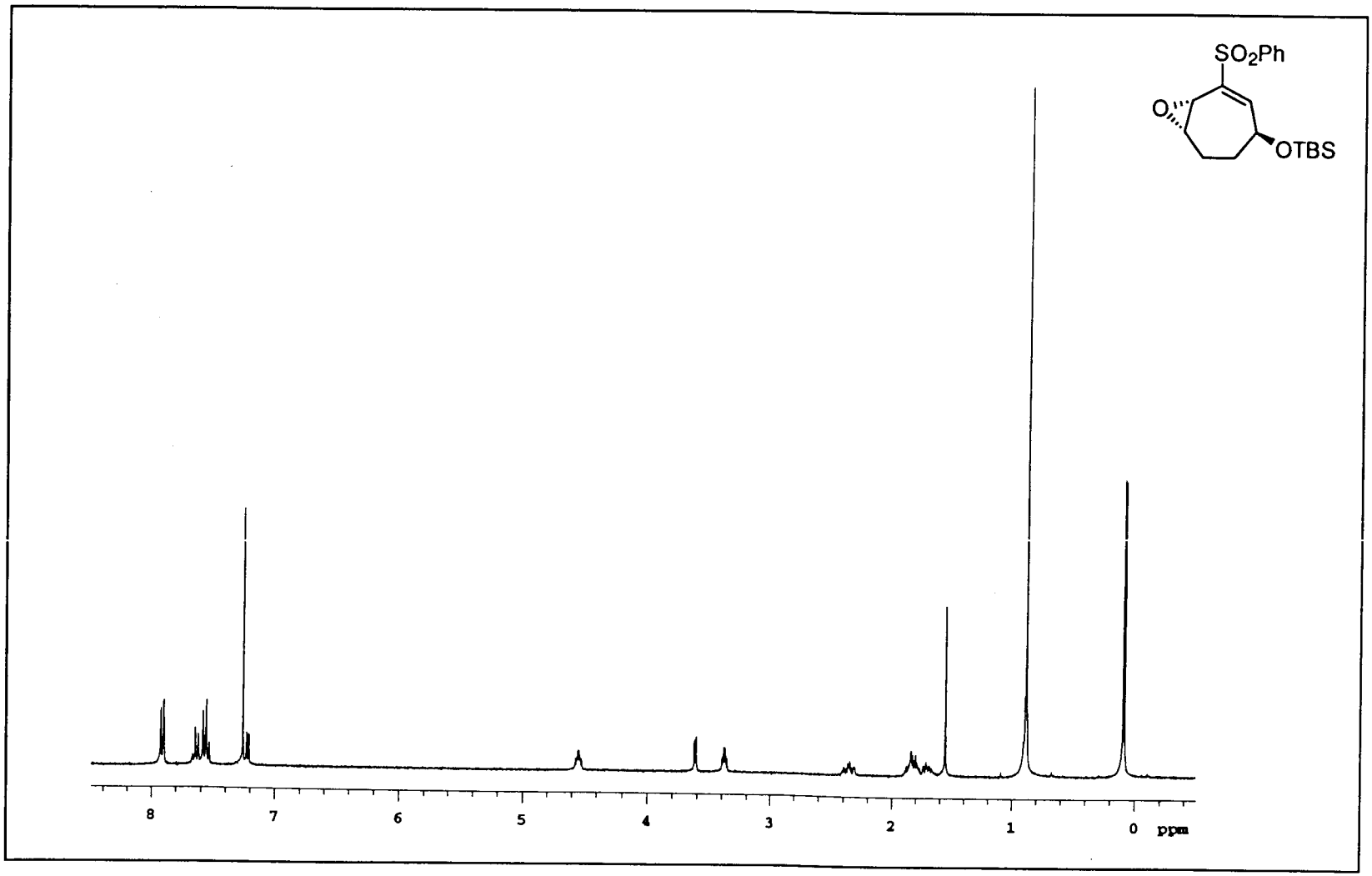

Figure 7 300MHz ^{1}H NMR of compound **anti-7b** in $CDCl_3$

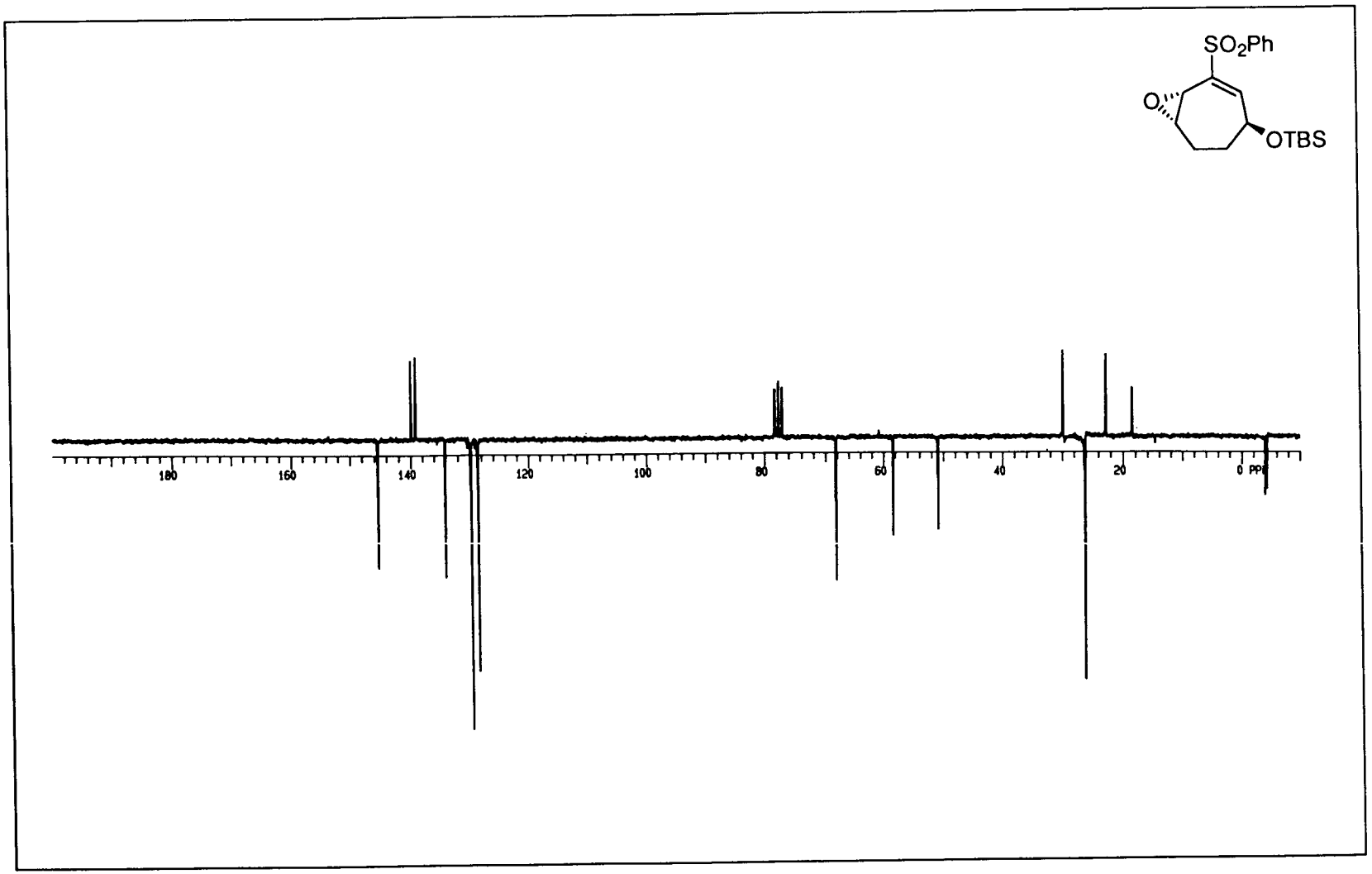

Figure 8 75MHz ^{13}C NMR of compound **anti-7b** in $CDCl_3$

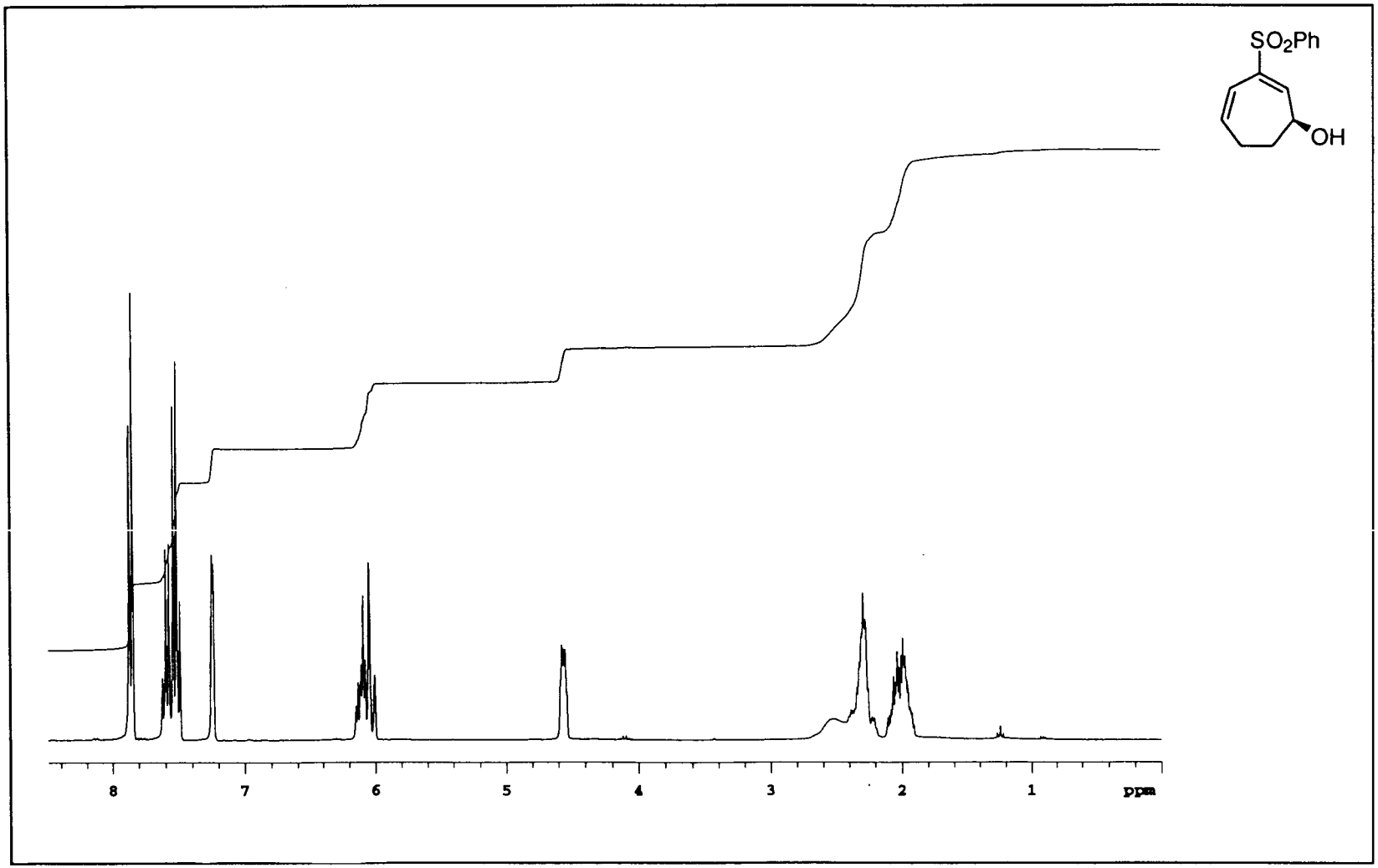

Figure 9 300MHz ^{1}H NMR of compound **8b** in $CDCl_3$

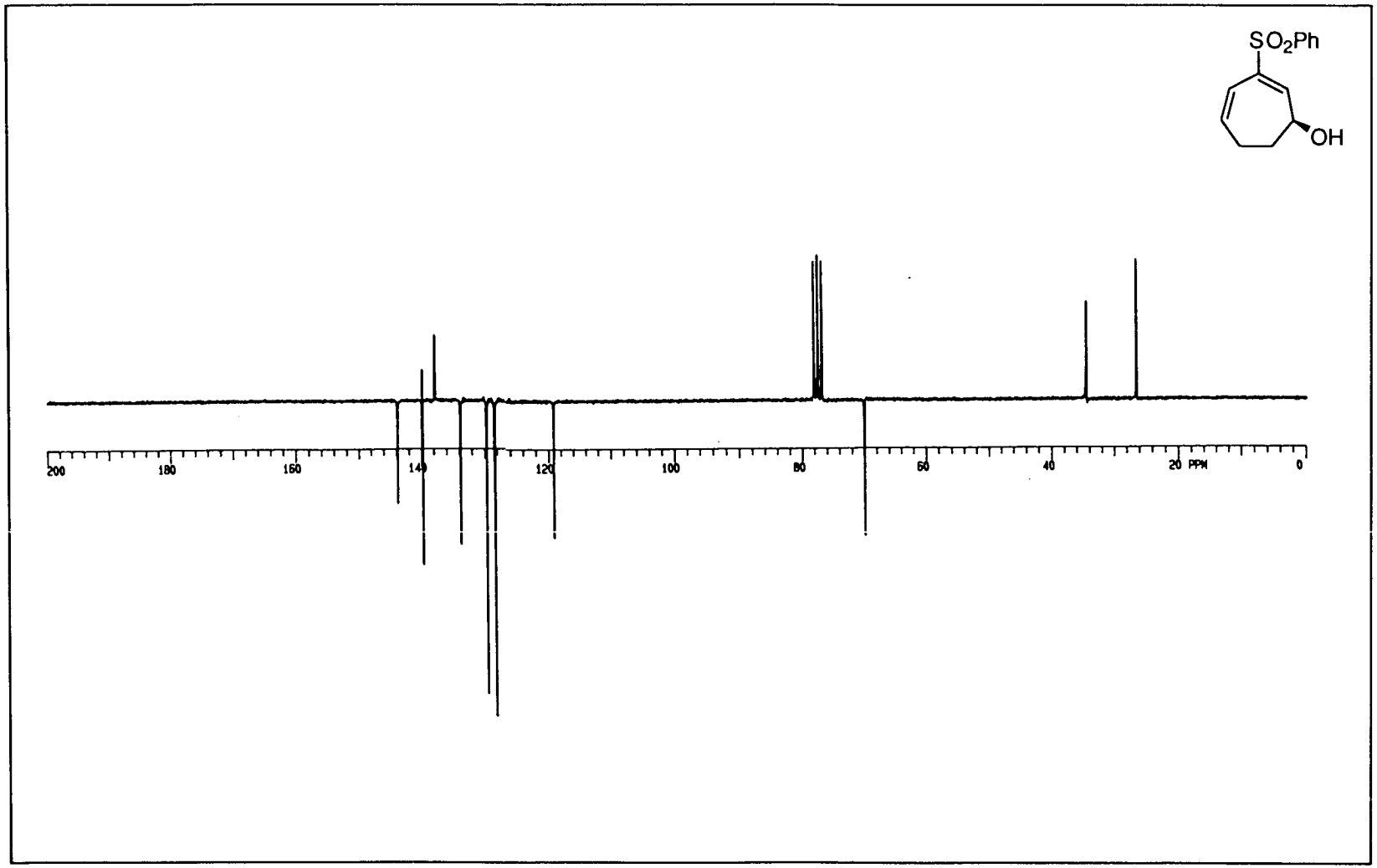

Figure 10 75MHz ^{13}C NMR of compound **8b** in $CDCl_3$

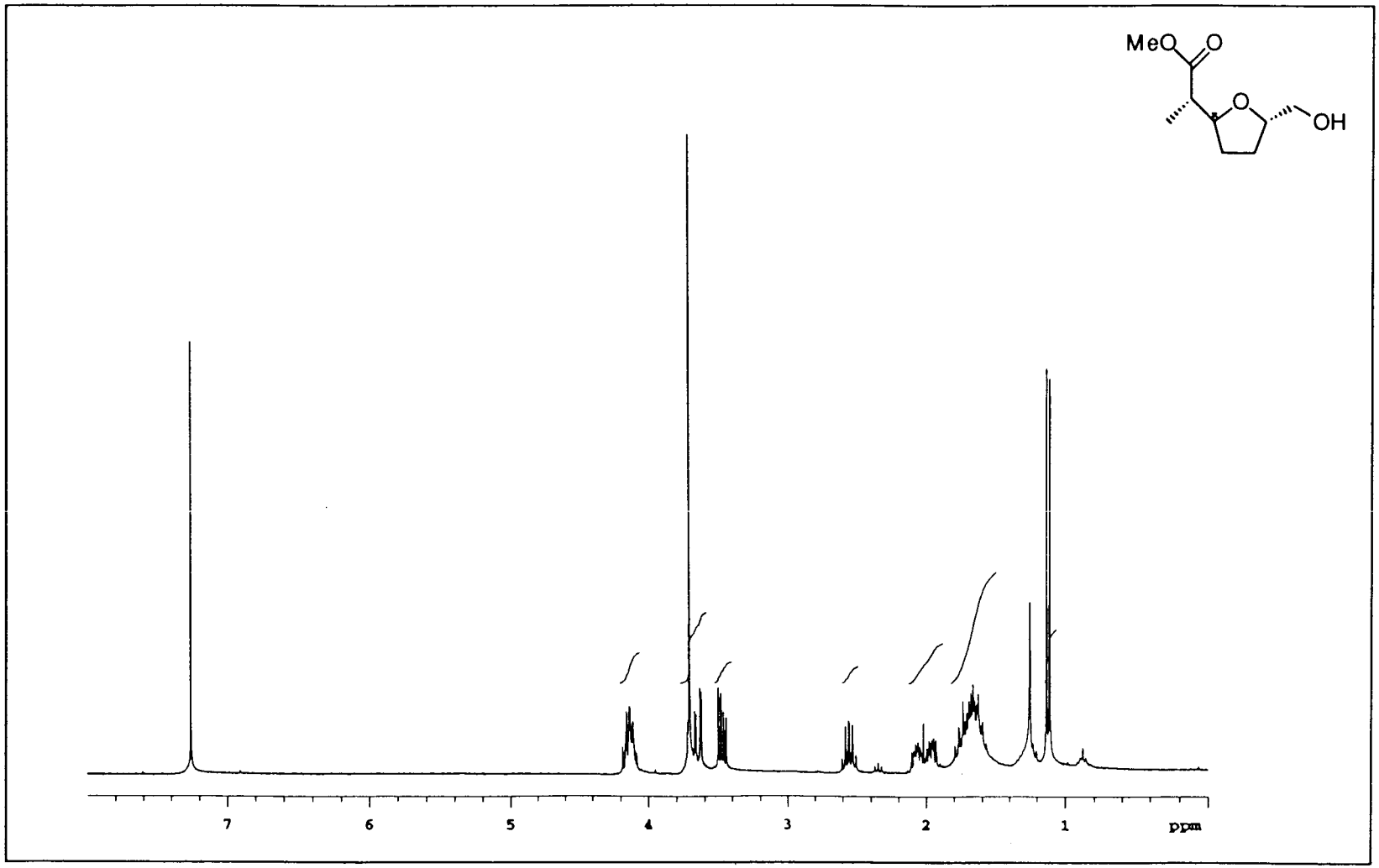

Figure 11 300MHz ^{1}H NMR of compound **10** in $CDCl_3$

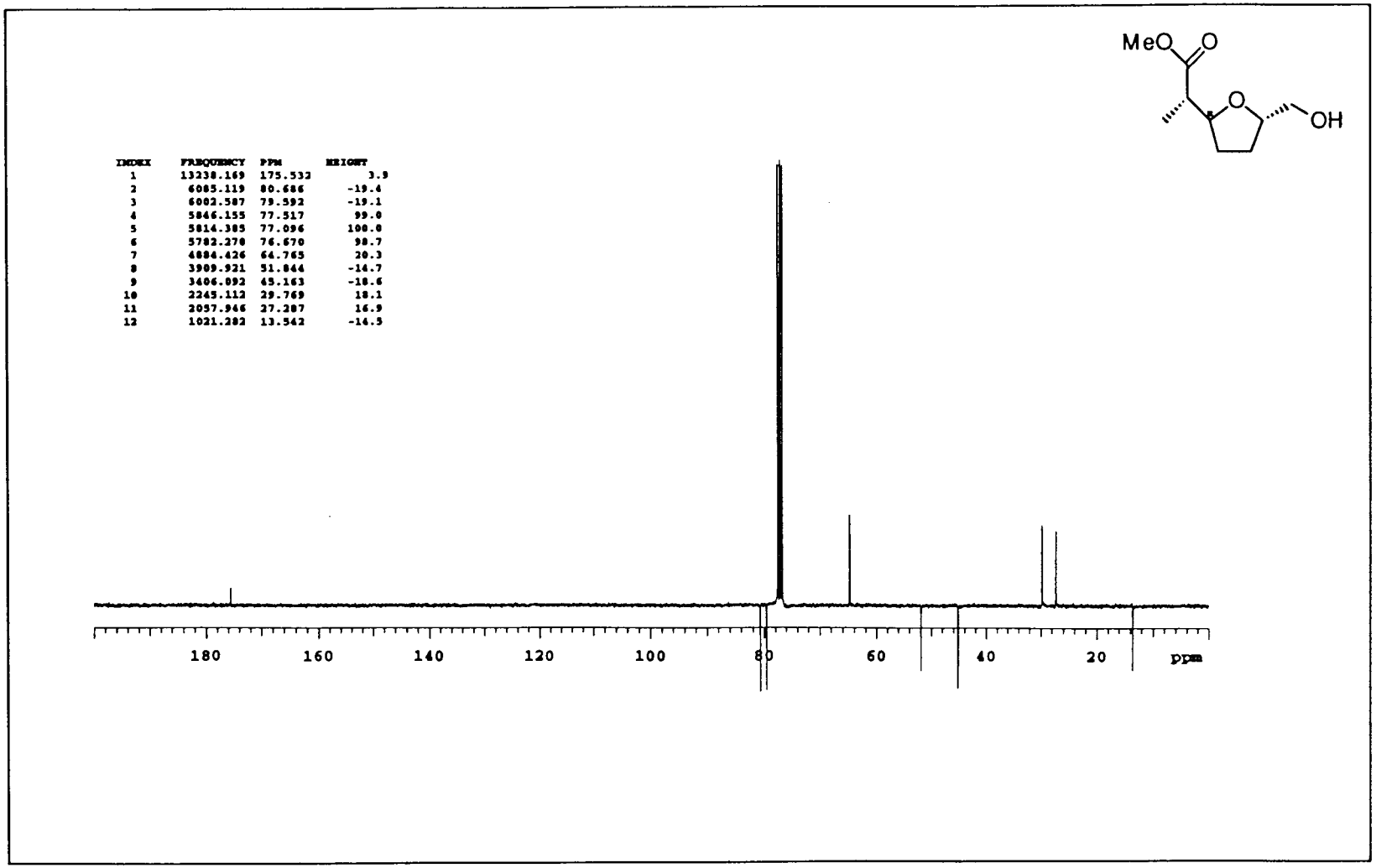

Figure 12 75MHz ^{13}C NMR of compound **10** in $CDCl_3$

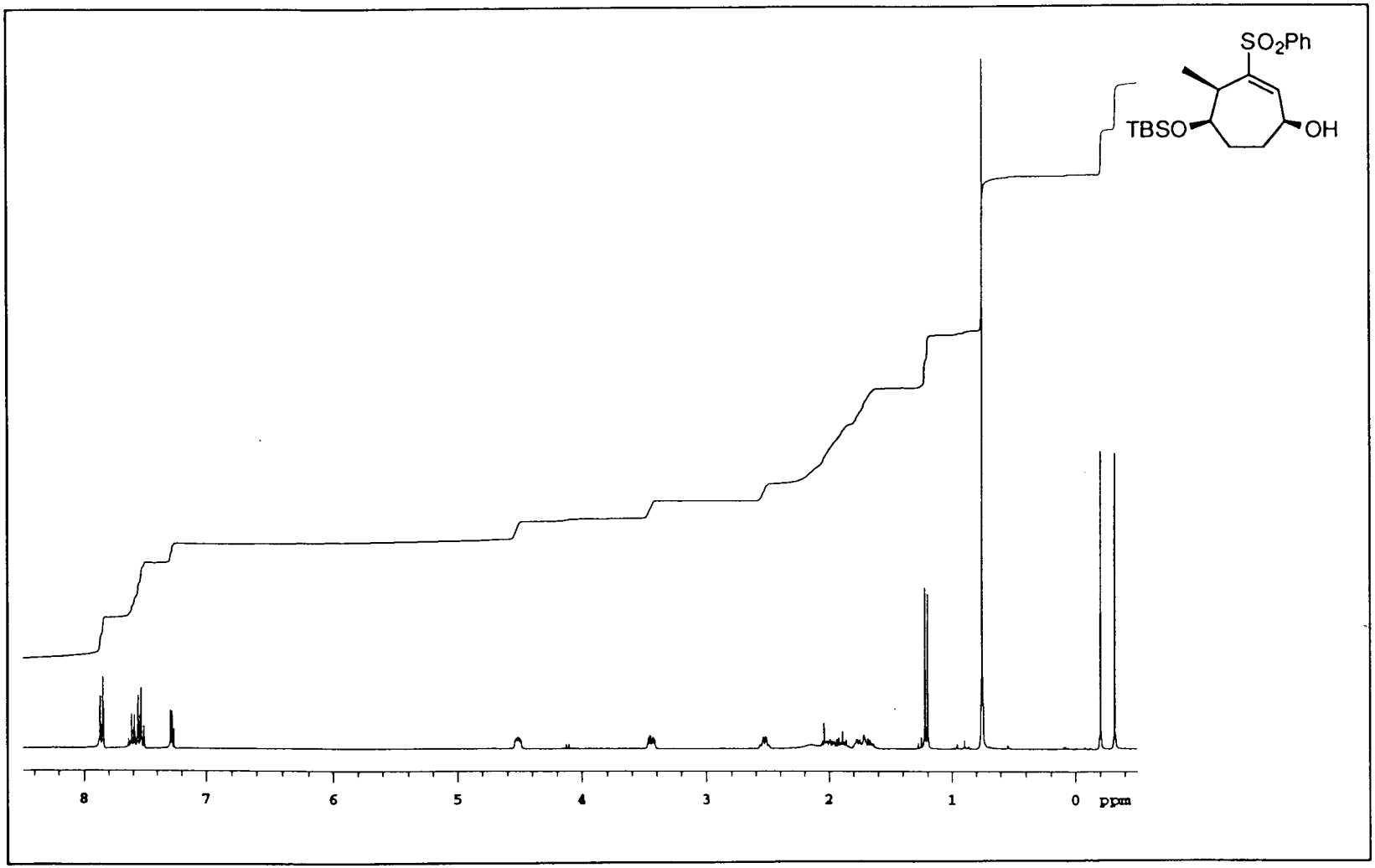

Figure 13 300MHz ^{1}H NMR of compound **11** in $CDCl_3$

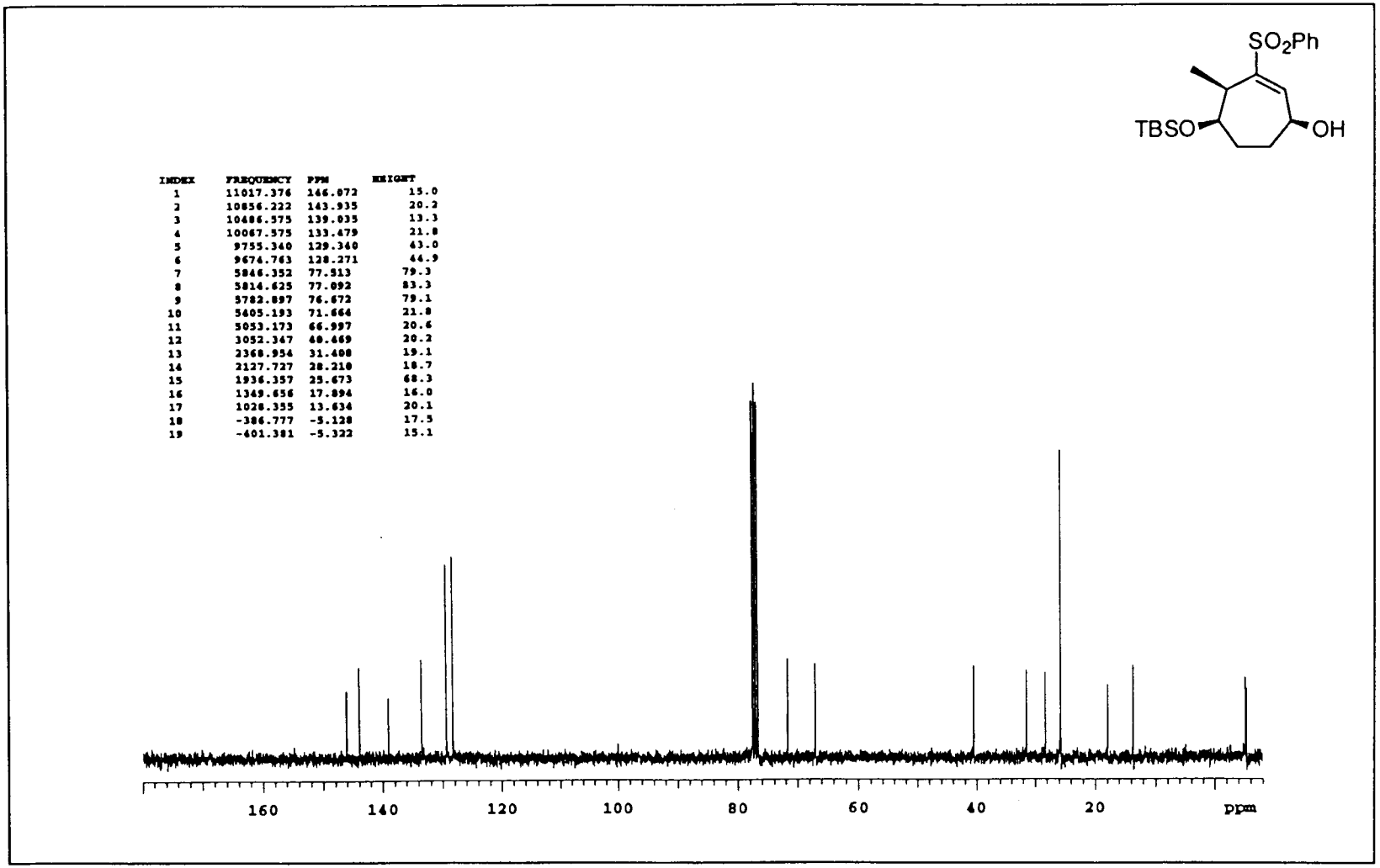

Figure 14 75MHz ^{13}C NMR of compound **11** in $CDCl_3$

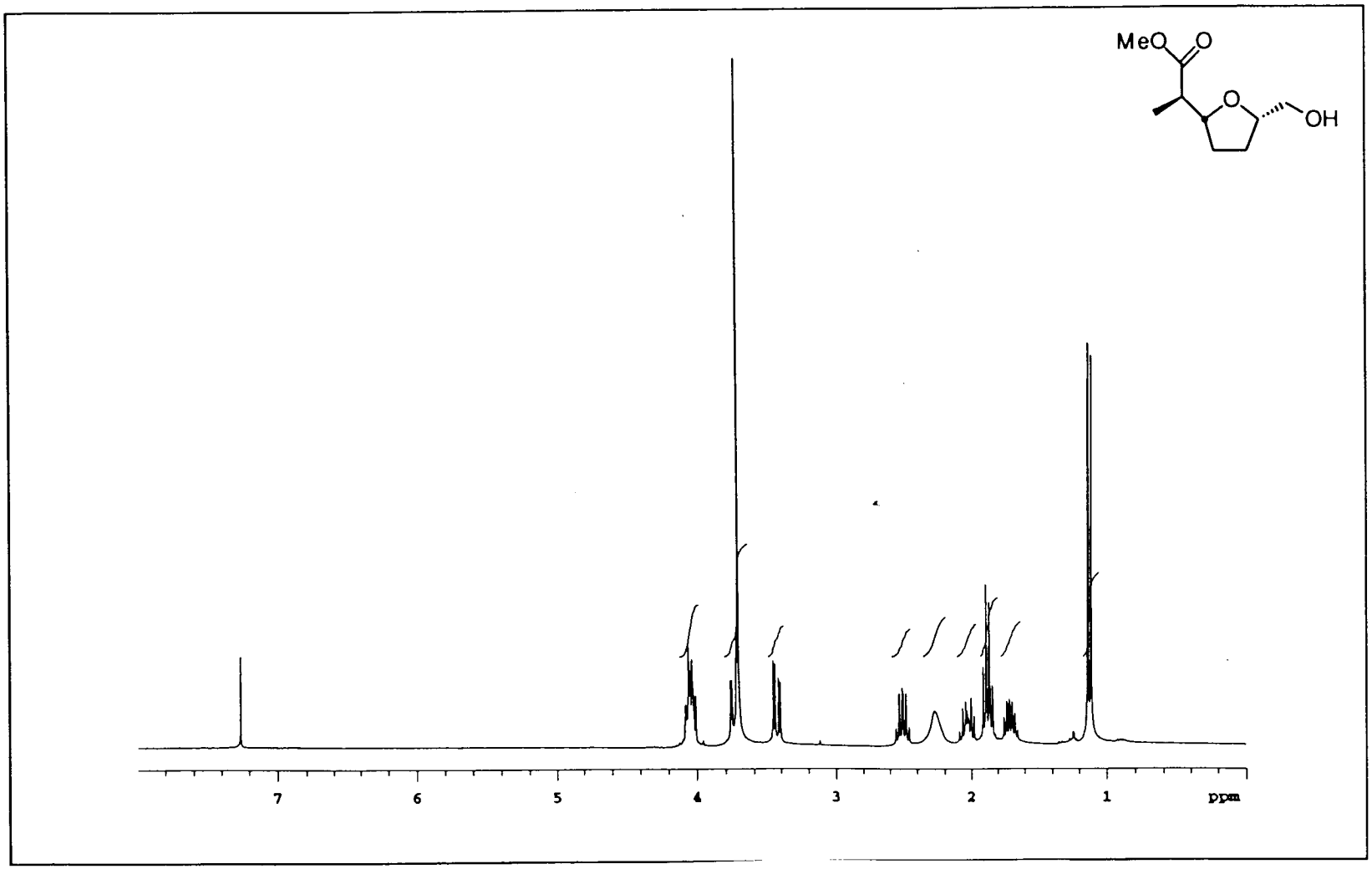

Figure 15 300MHz ^{1}H NMR of compound **12** in $CDCl_3$

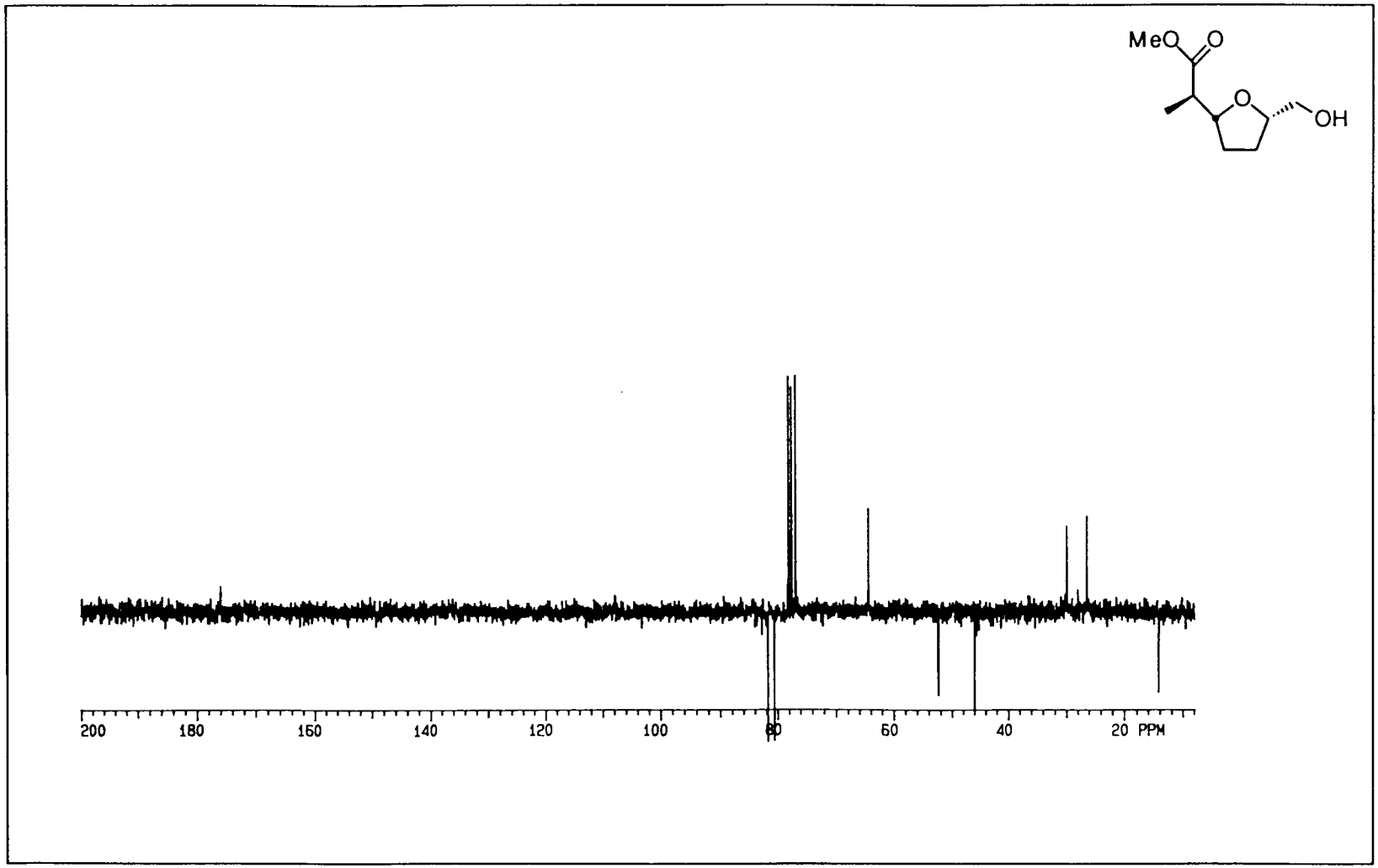

Figure 16 75MHz ^{13}C NMR of compound **12** in $CDCl_3$

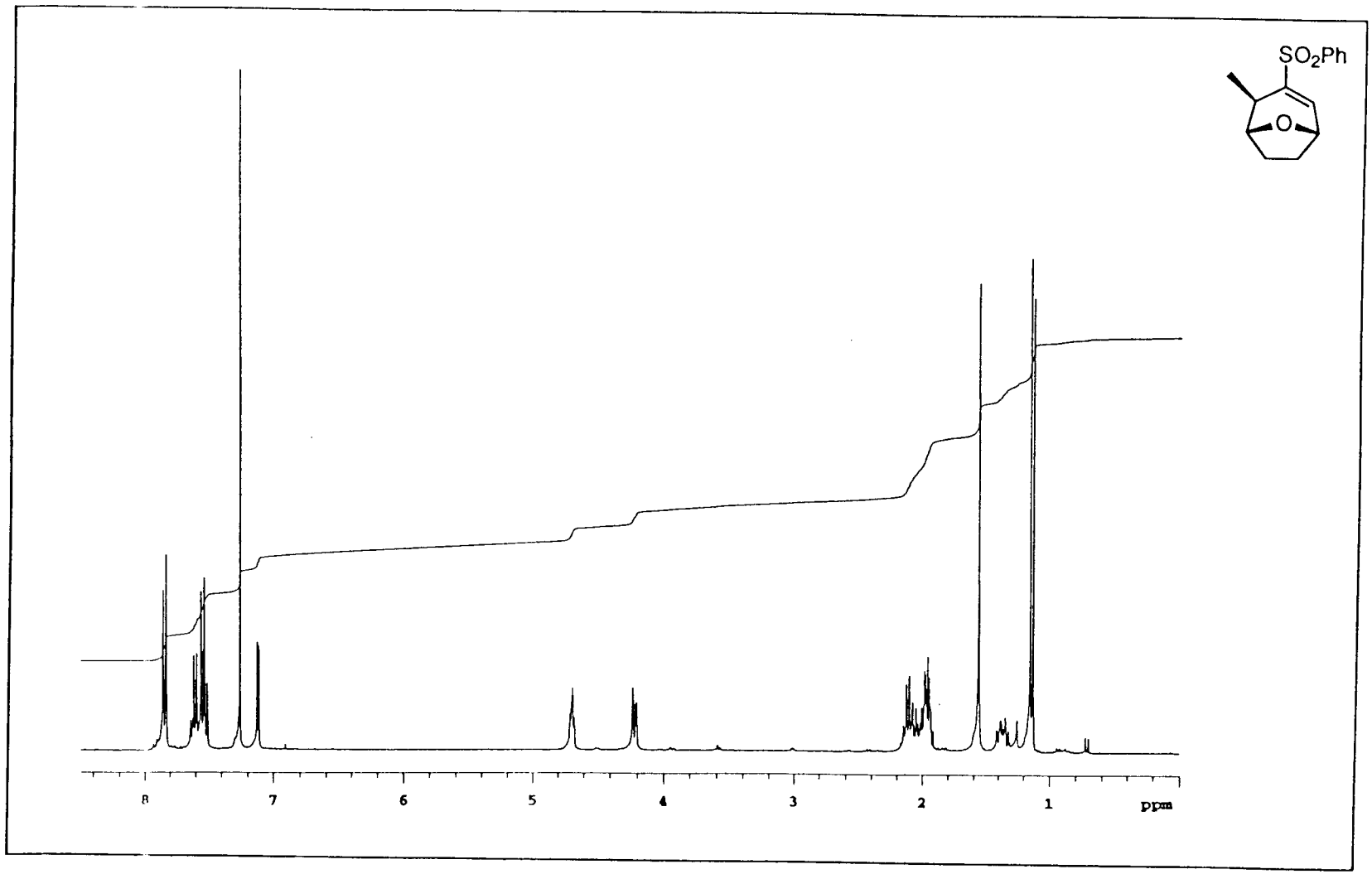

Figure 17 300MHz ^{1}H NMR of compound **13** in $CDCl_3$

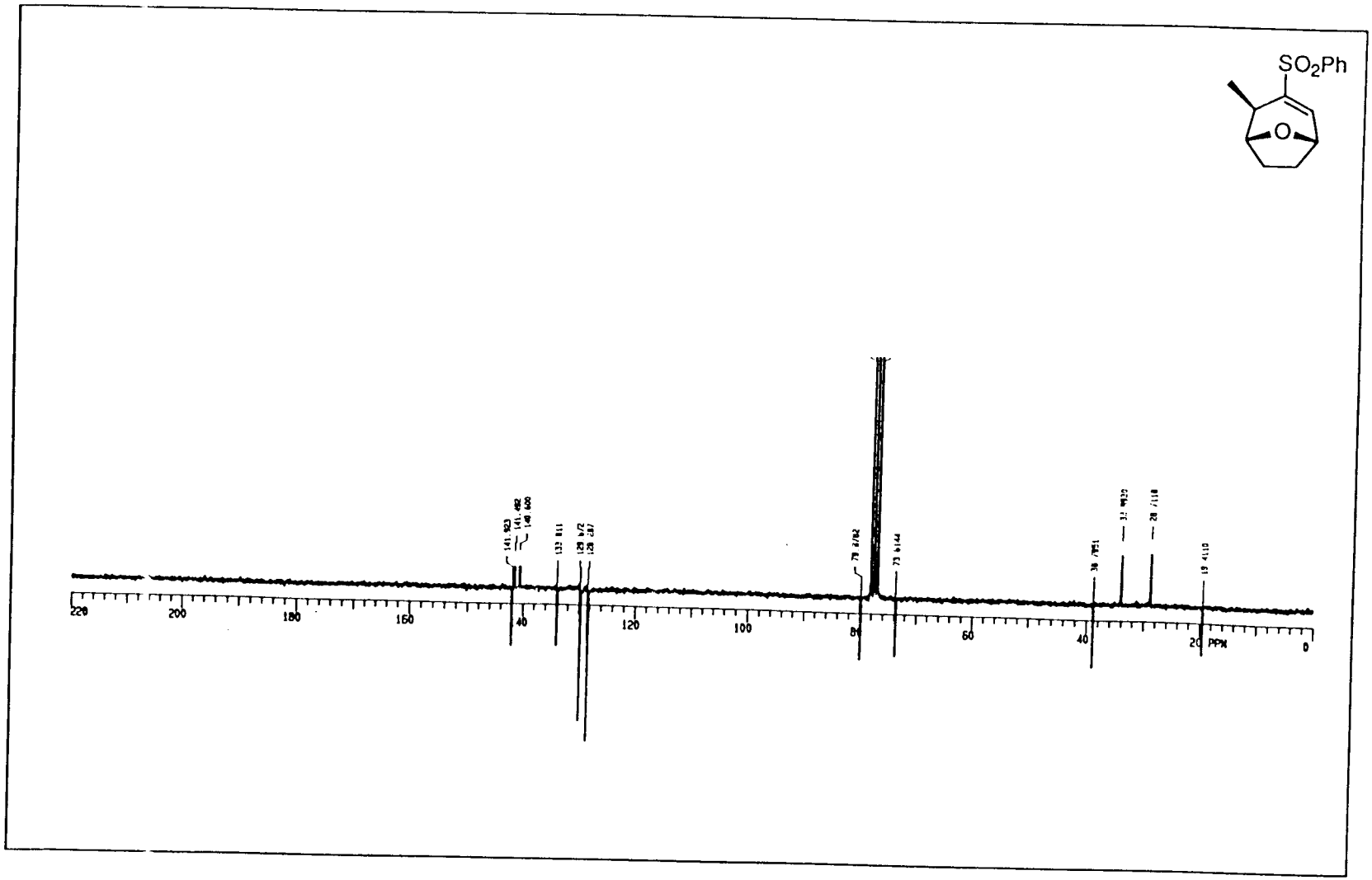

Figure 18 75MHz ^{13}C NMR of compound **13** in $CDCl_3$

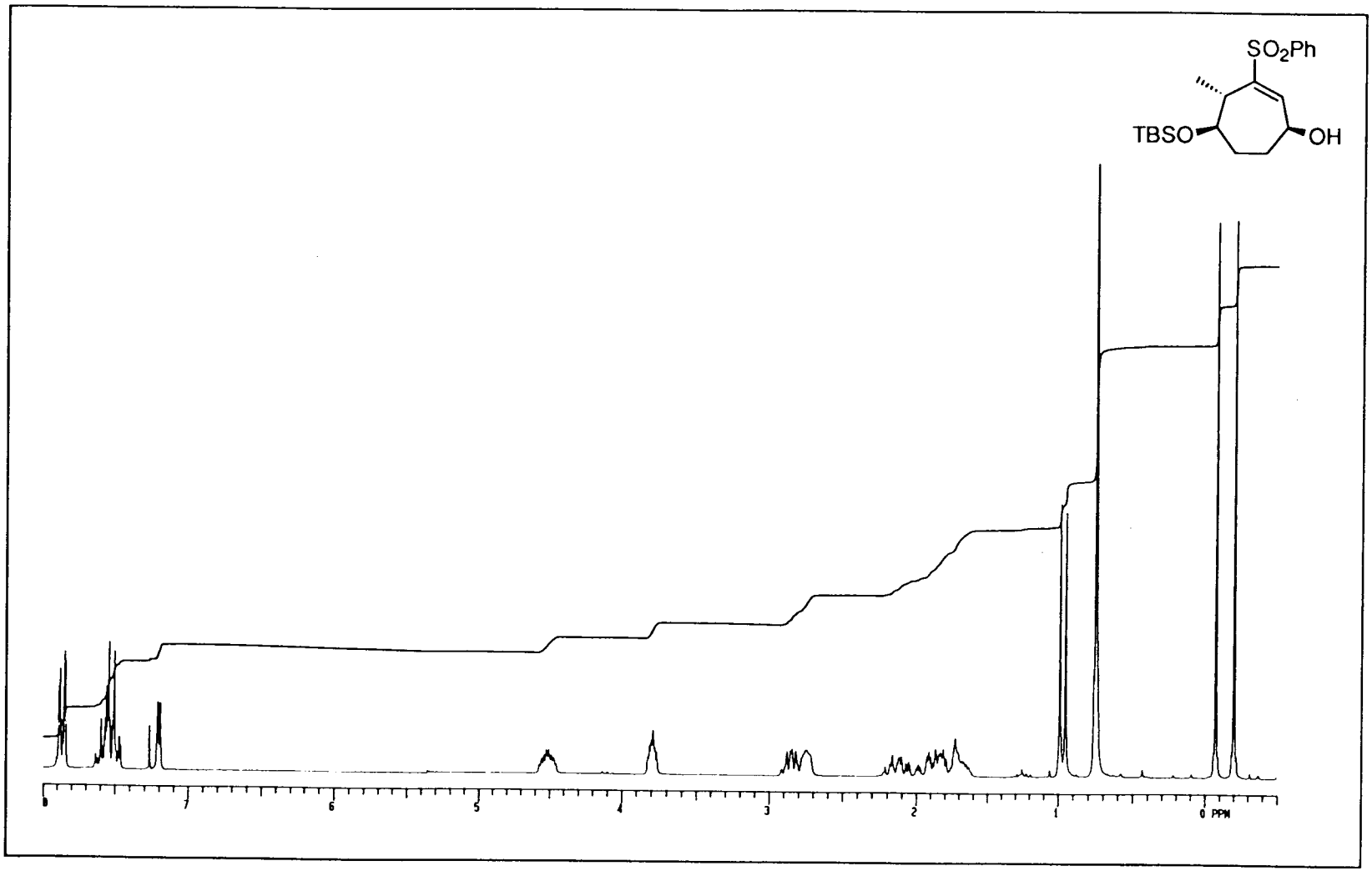

Figure 19 300MHz ^{1}H NMR of compound **14** in $CDCl_3$

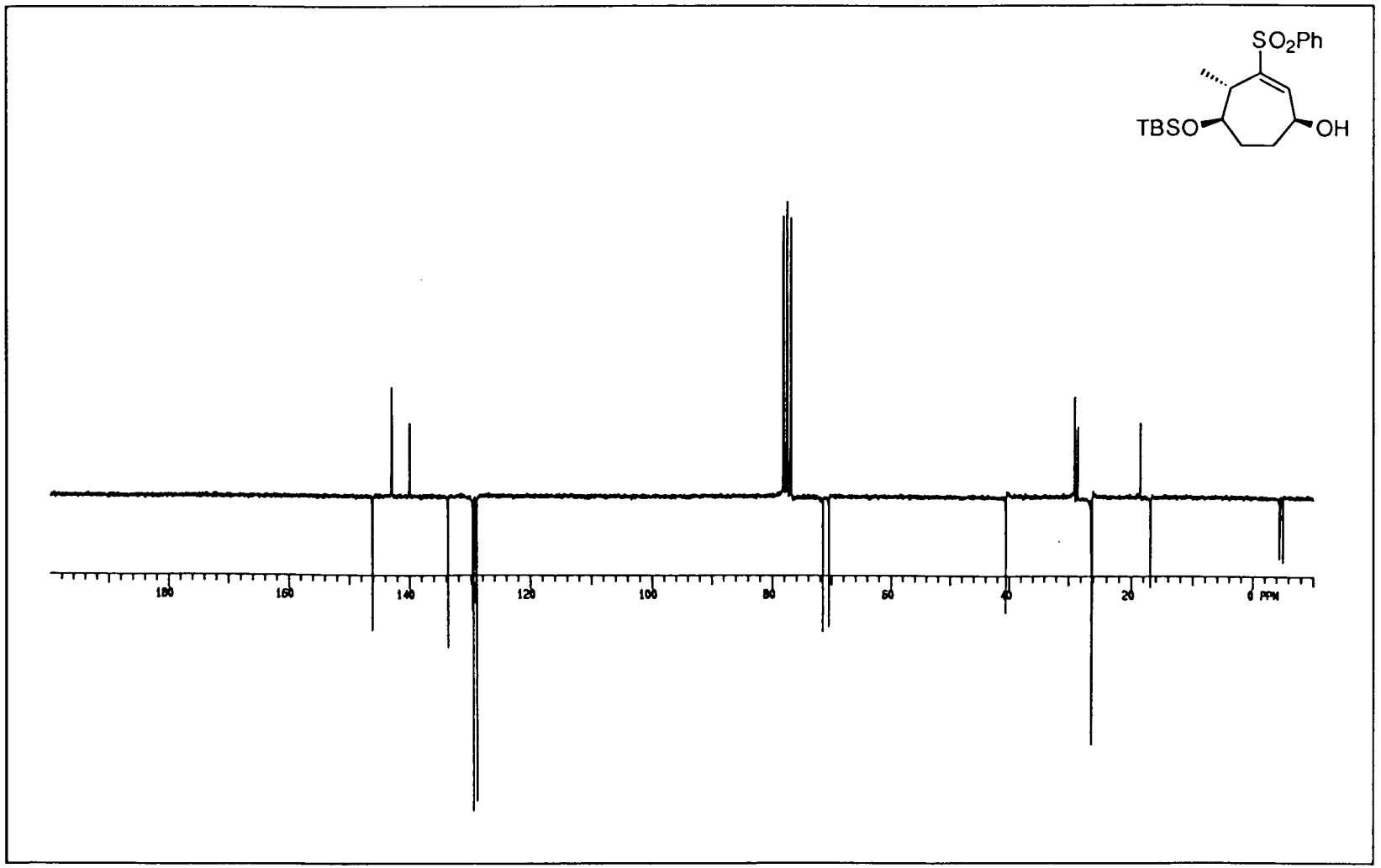

Figure 20 75MHz ^{13}C NMR of compound **14** in $CDCl_3$

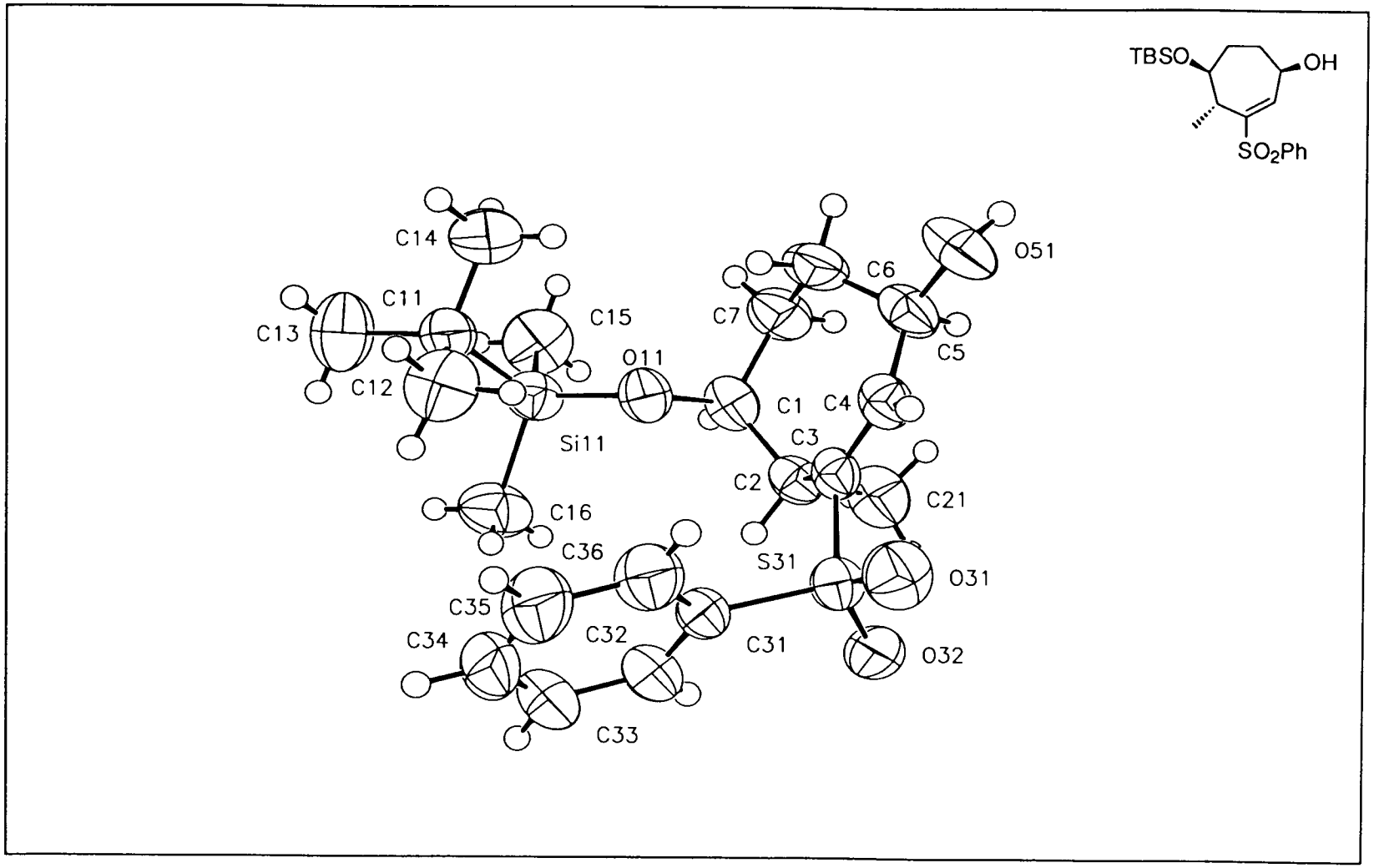

Figure 21 An ORTEP representation of compound **ent-14**

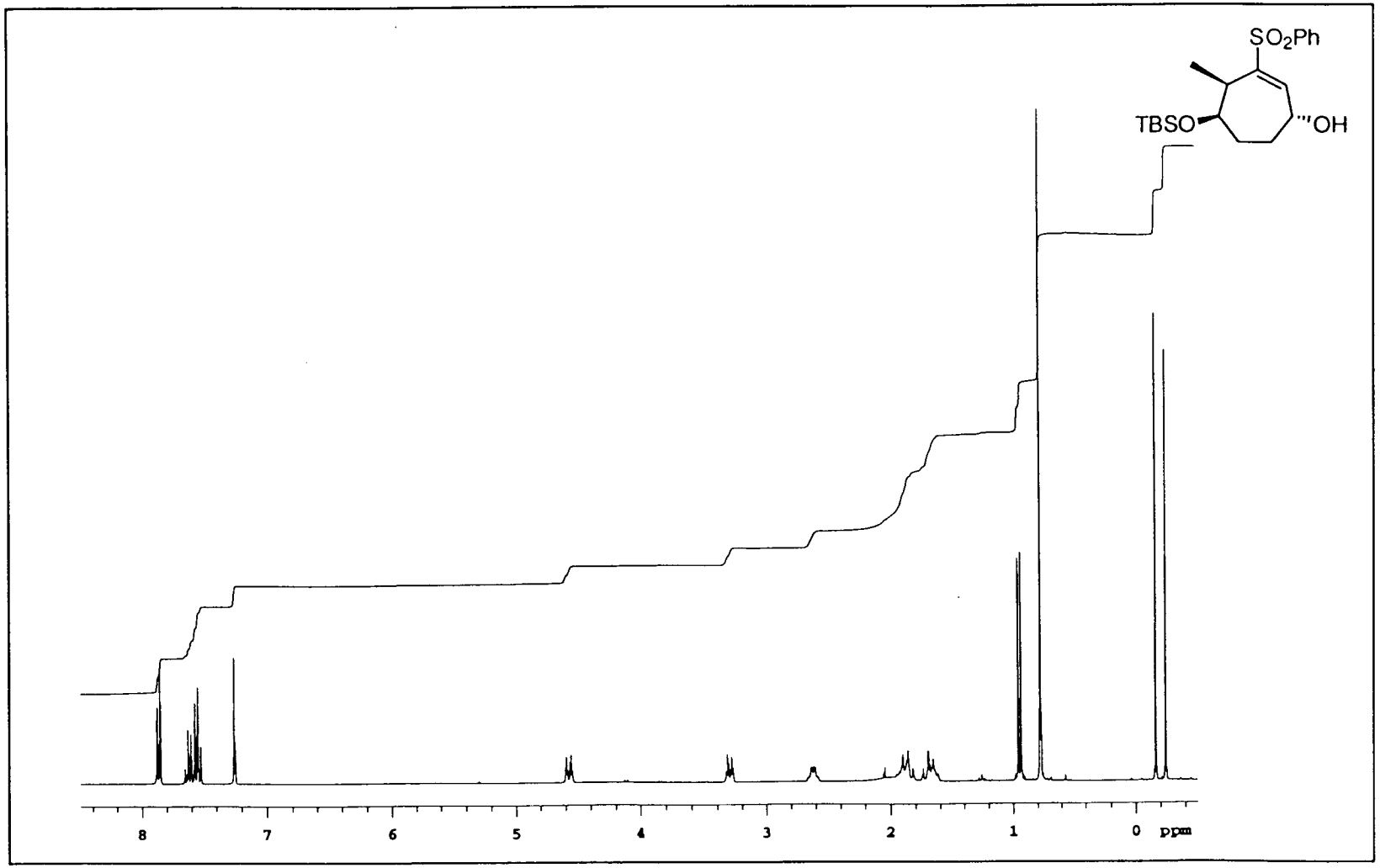

Figure 22 300MHz ^{1}H NMR of compound **15** in $CDCl_3$

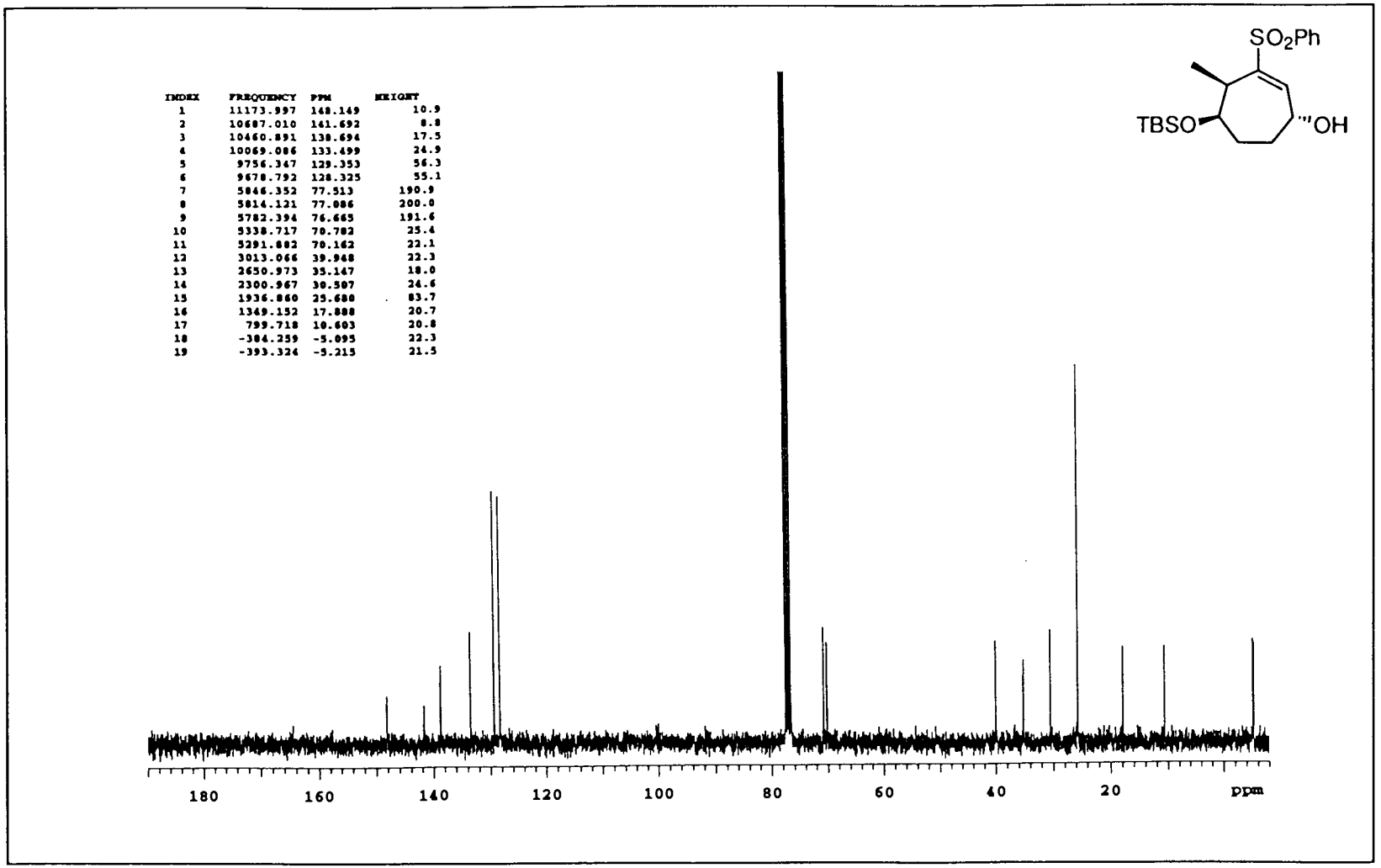

Figure 23 75MHz ^{13}C NMR of compound **15** in $CDCl_3$

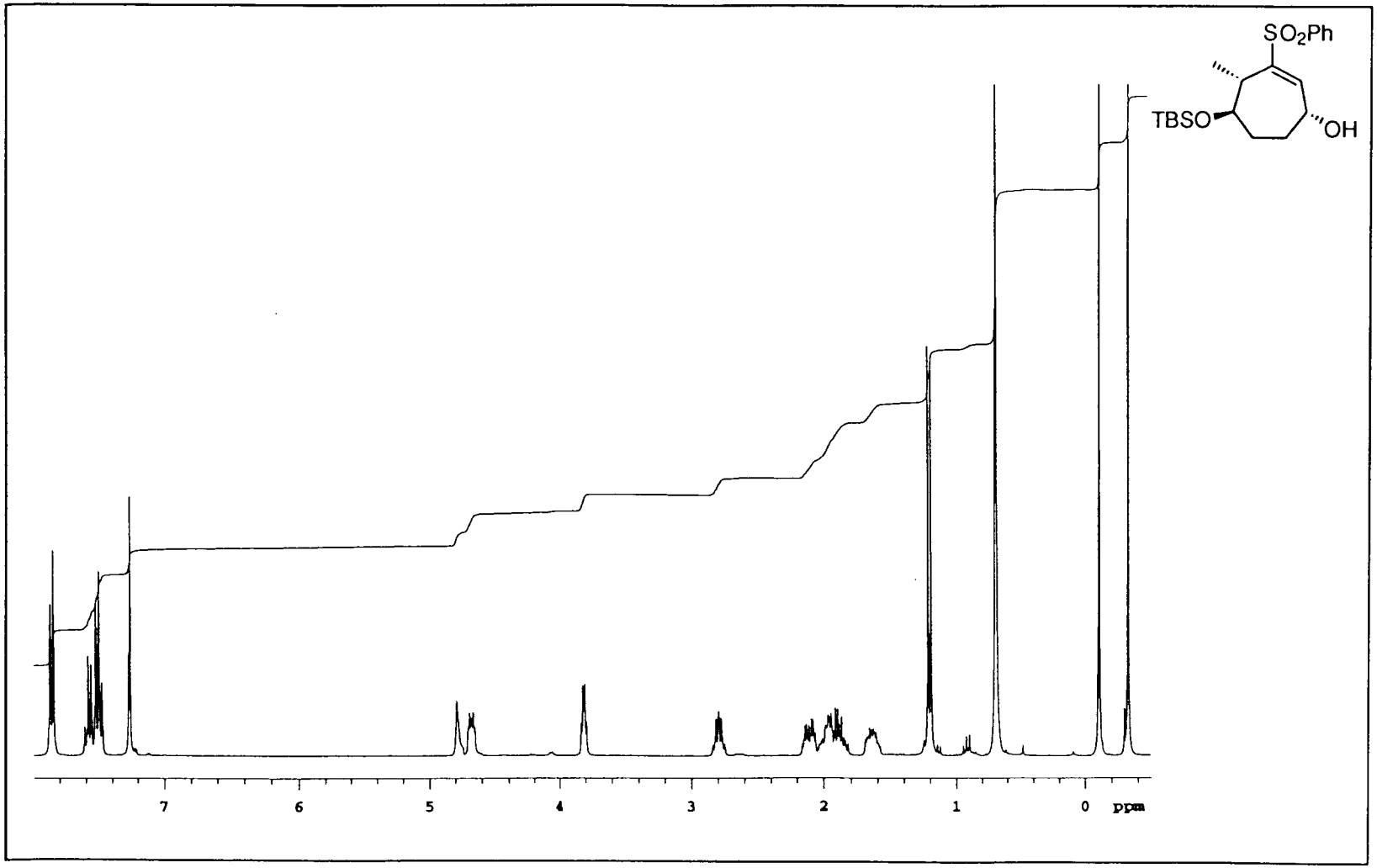

Figure 24 300MHz ^{1}H NMR of compound **16** in $CDCl_3$

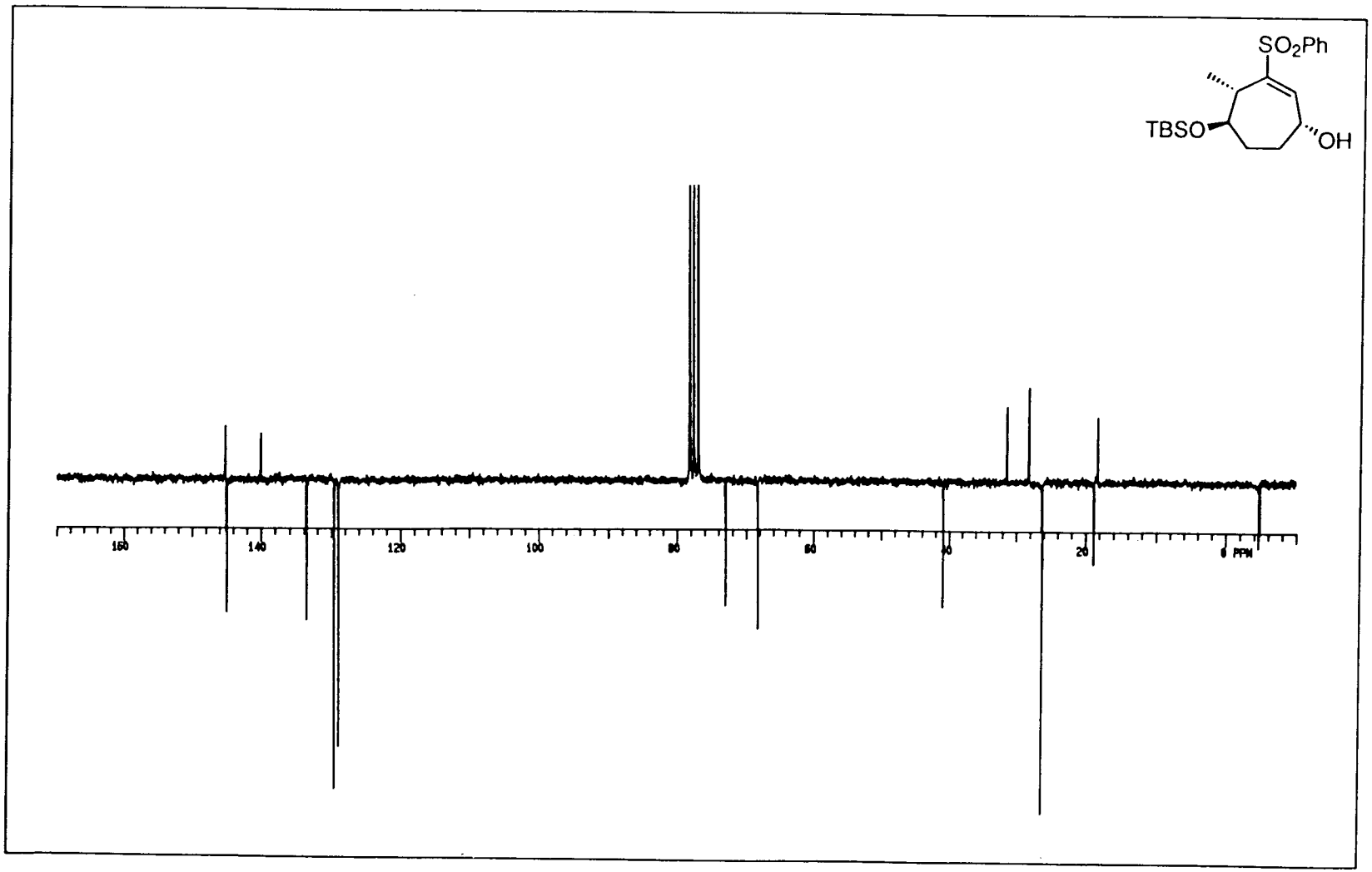

Figure 25 75MHz ^{13}C NMR of compound **16** in $CDCl_3$

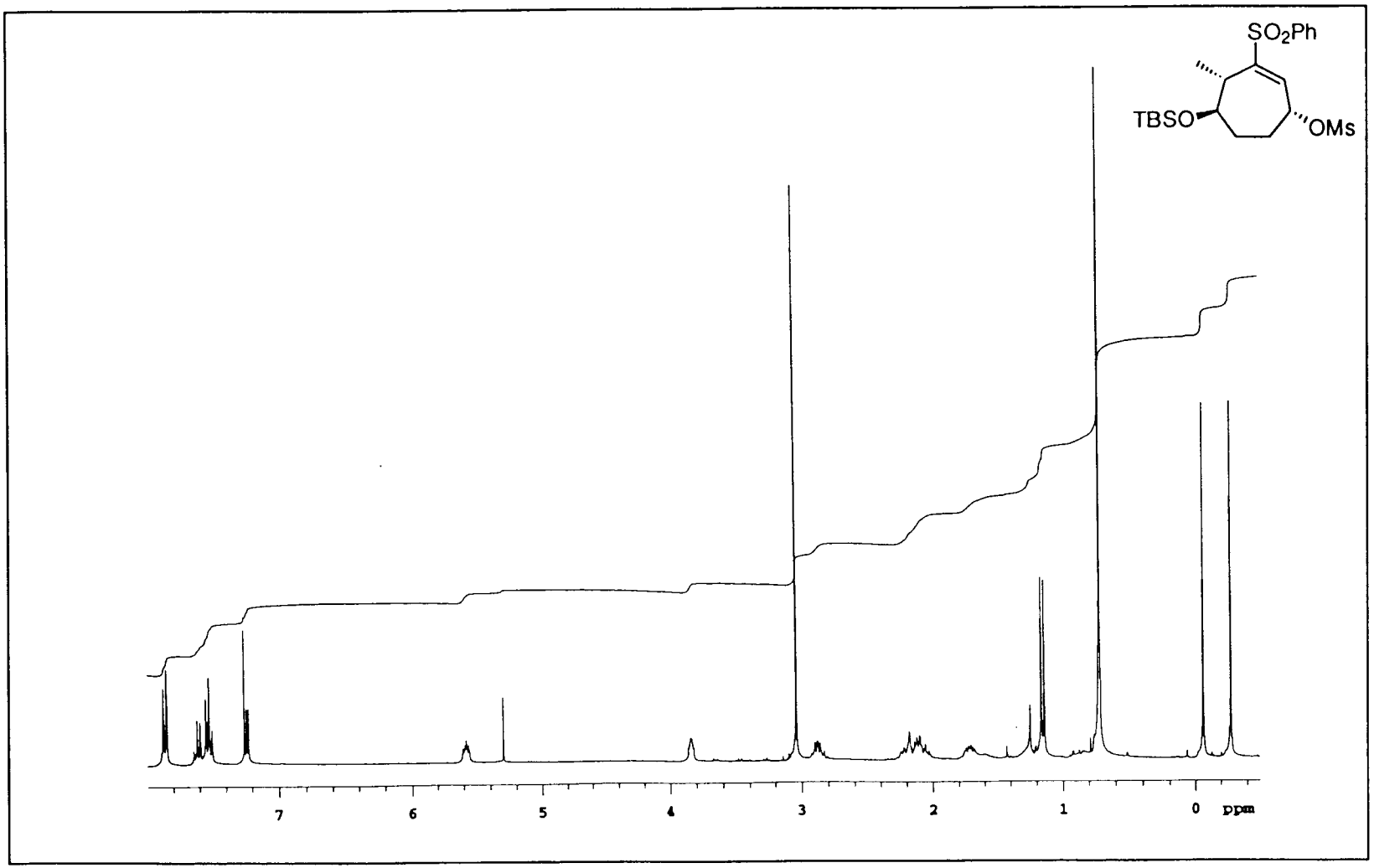

Figure 26 300MHz ^{1}H NMR of compound **17** in $CDCl_3$

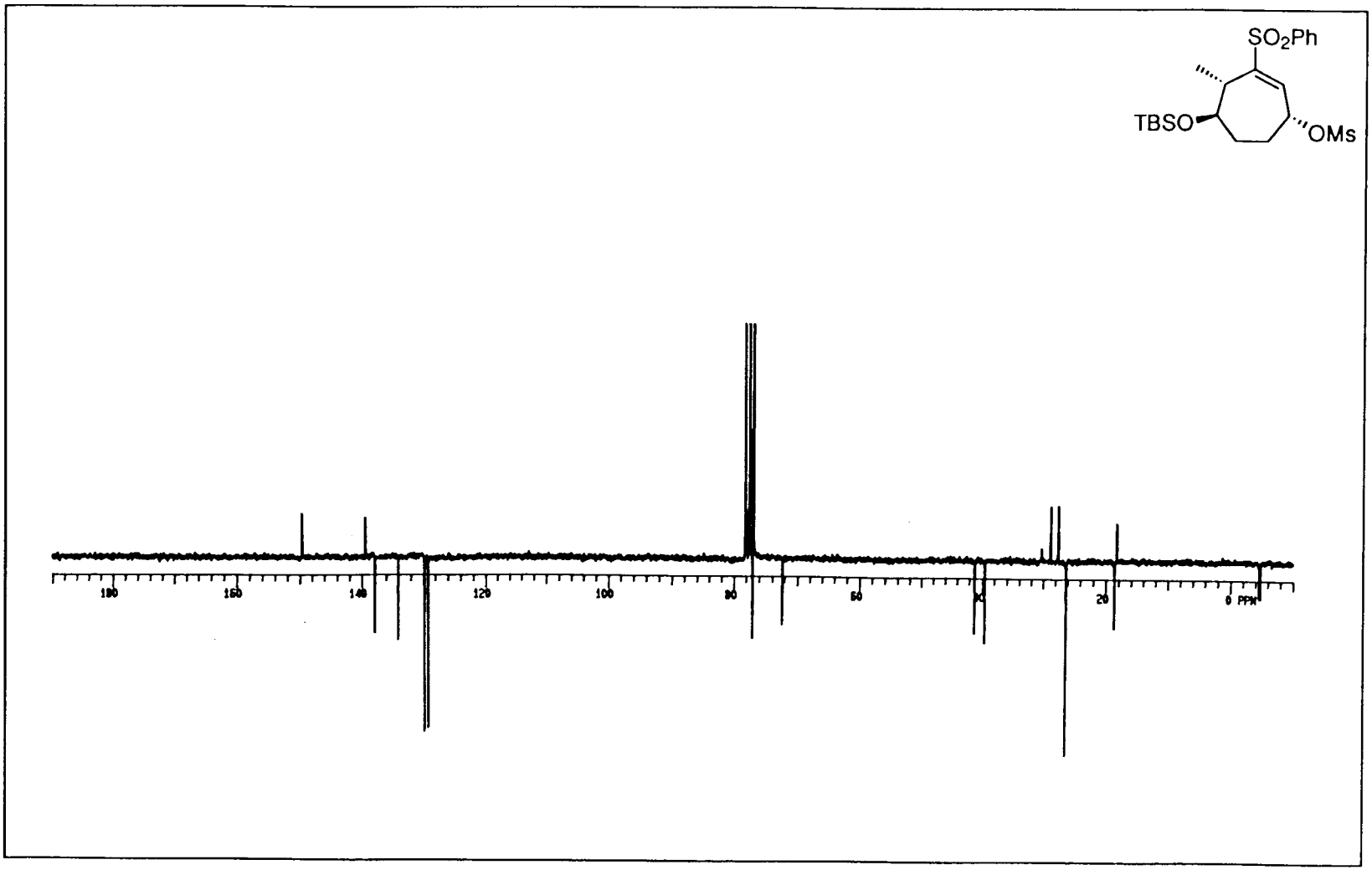

Figure 27 75MHz ^{13}C NMR of compound **17** in $CDCl_3$

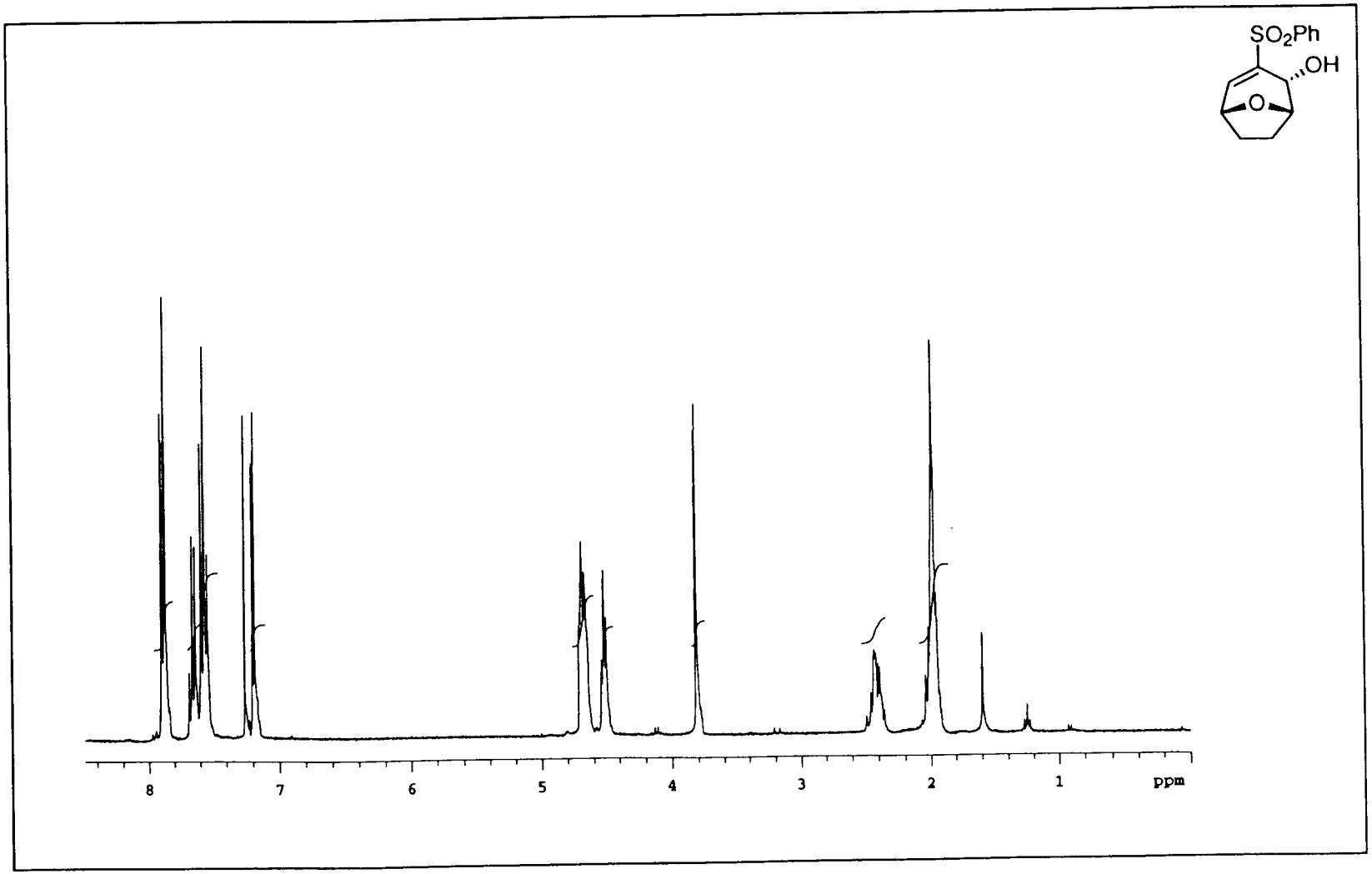

Figure 28 300MHz ^{1}H NMR of compound **18** in $CDCl_3$

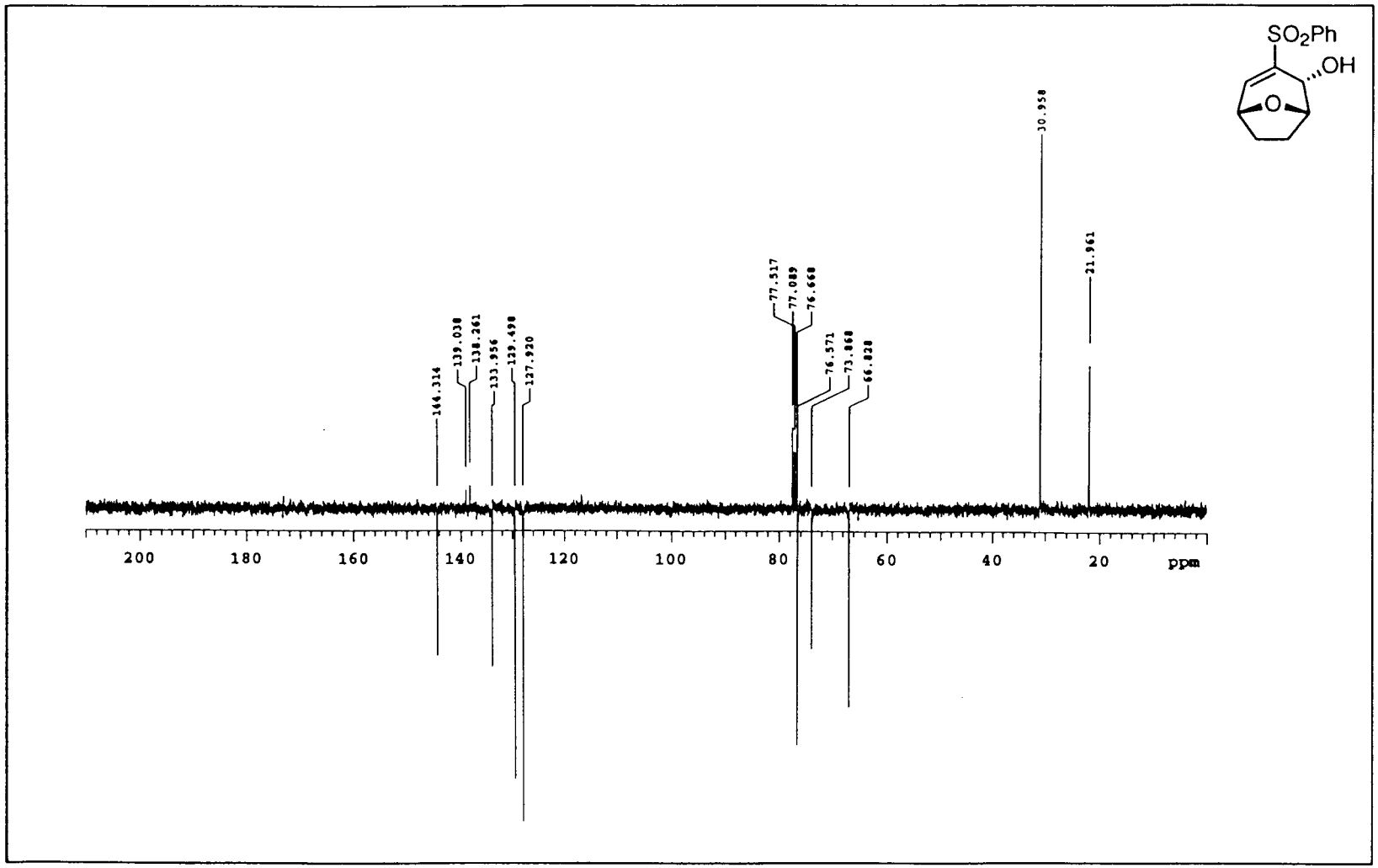

Figure 29 75MHz ^{13}C NMR of compound **18** in $CDCl_3$

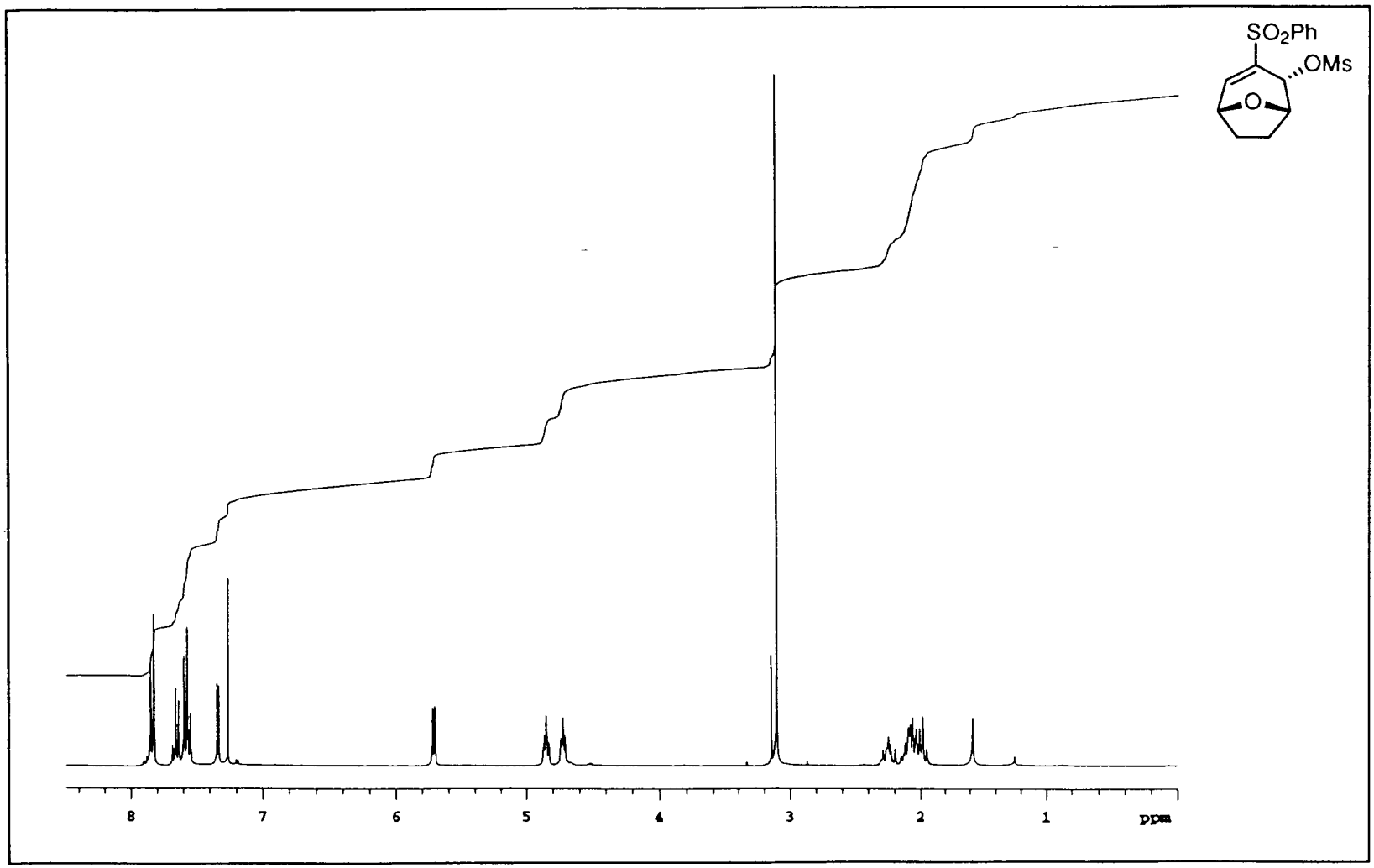

Figure 30 300MHz ^{1}H NMR of compound **19** in $CDCl_3$

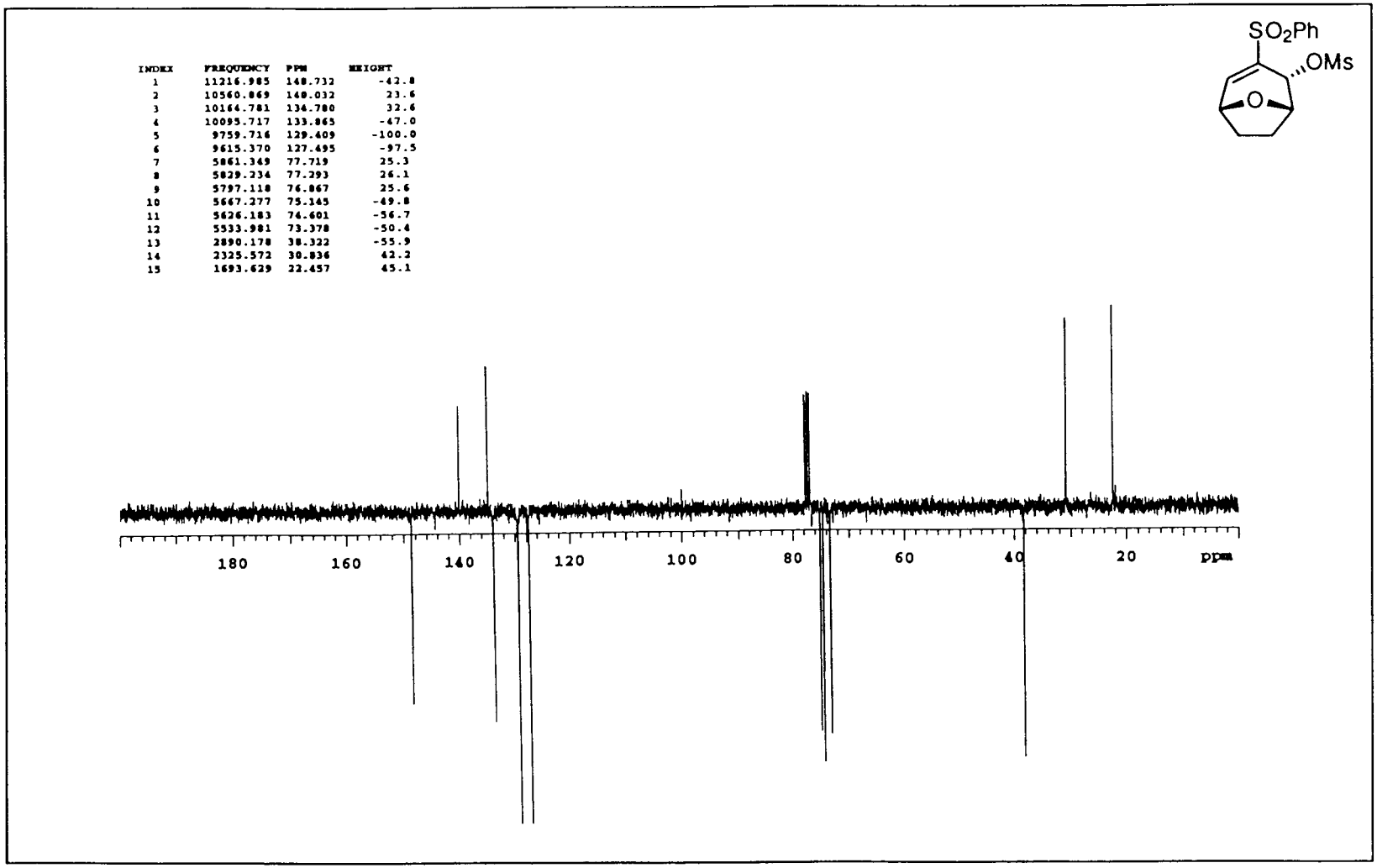

Figure 31 75MHz ^{13}C NMR of compound **19** in $CDCl_3$

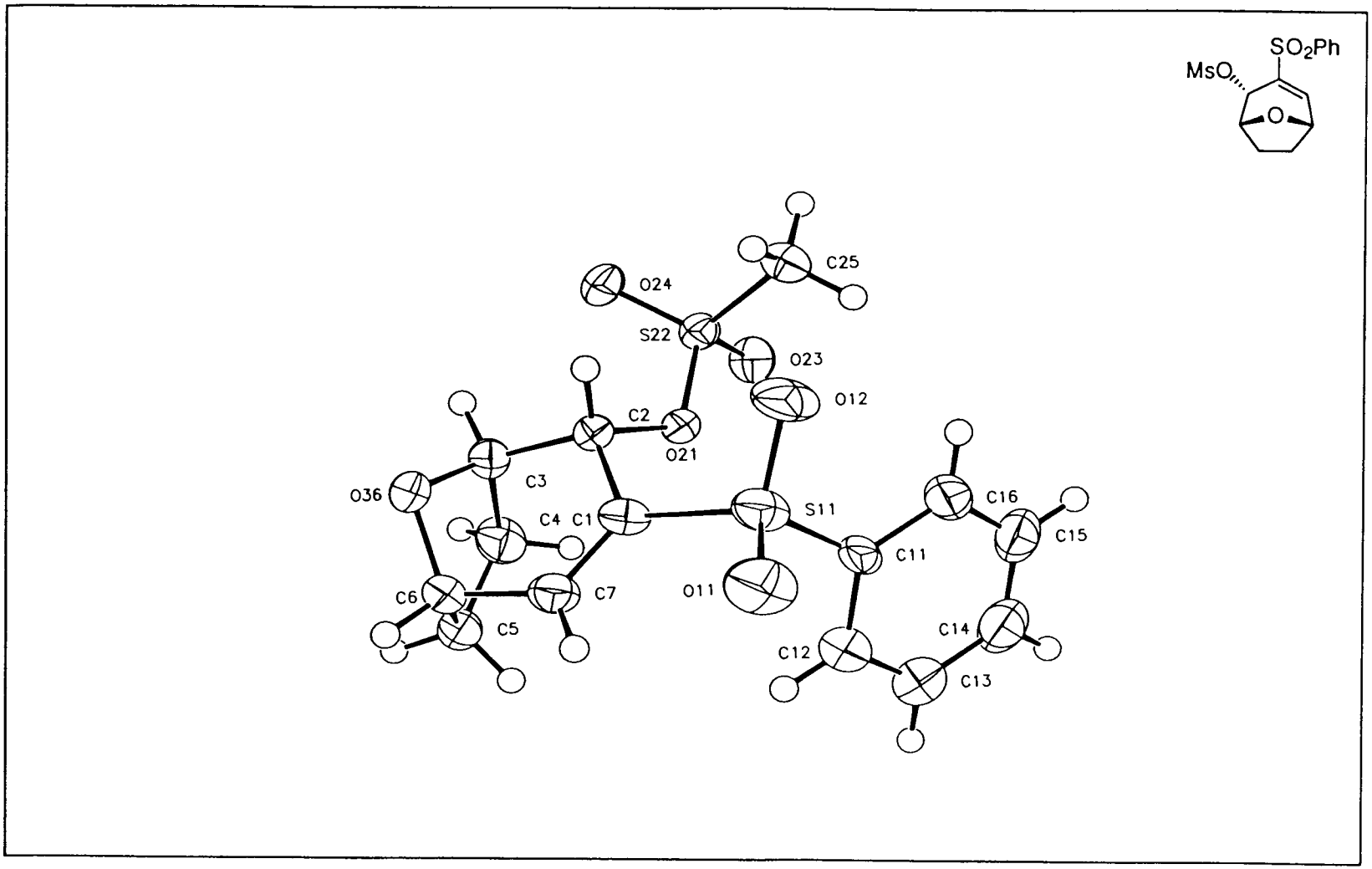

Figure 32 An ORTEP representation of compound **ent-19**

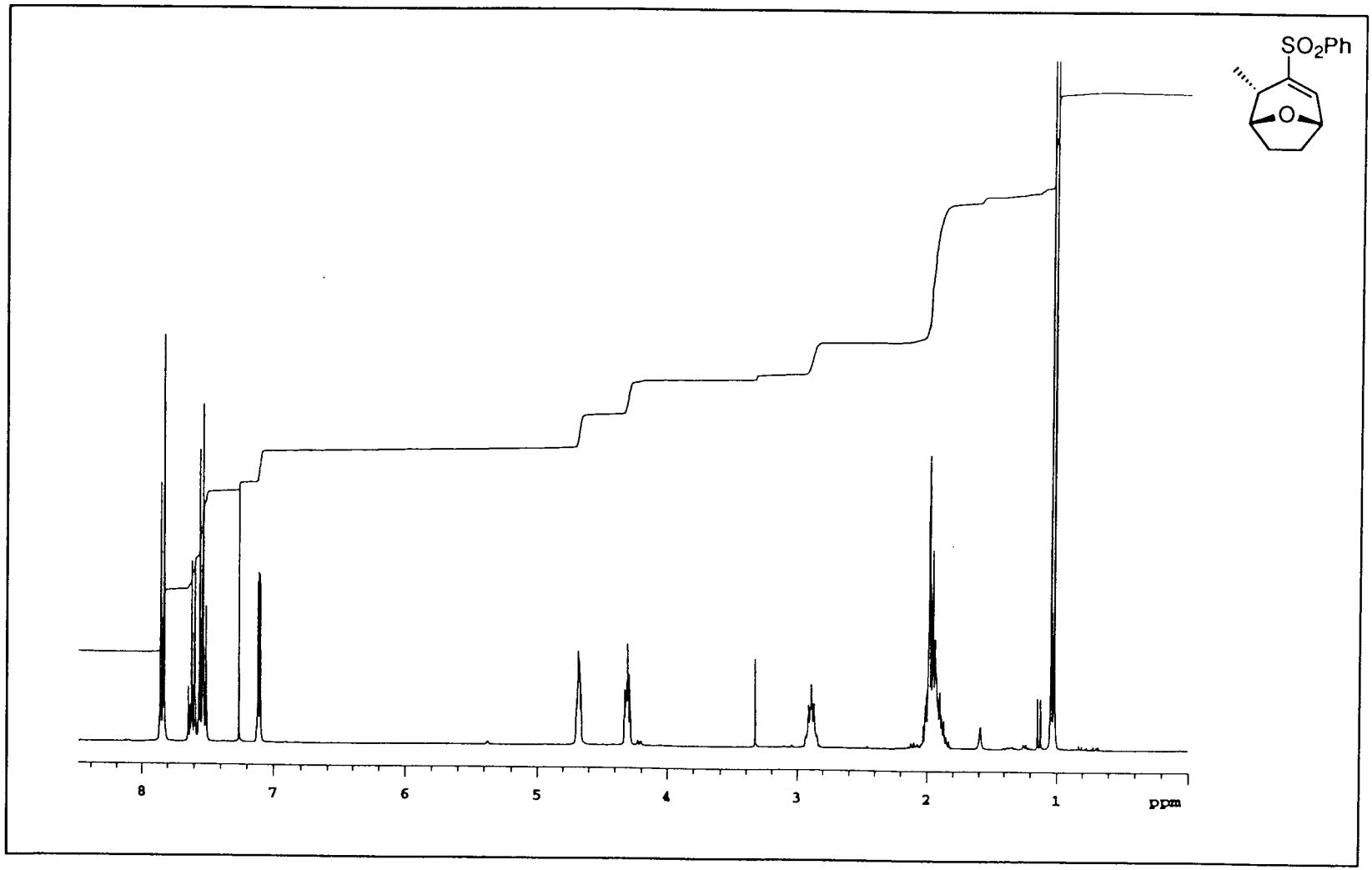

Figure 33 300MHz ^{1}H NMR of compound **20** in $CDCl_3$

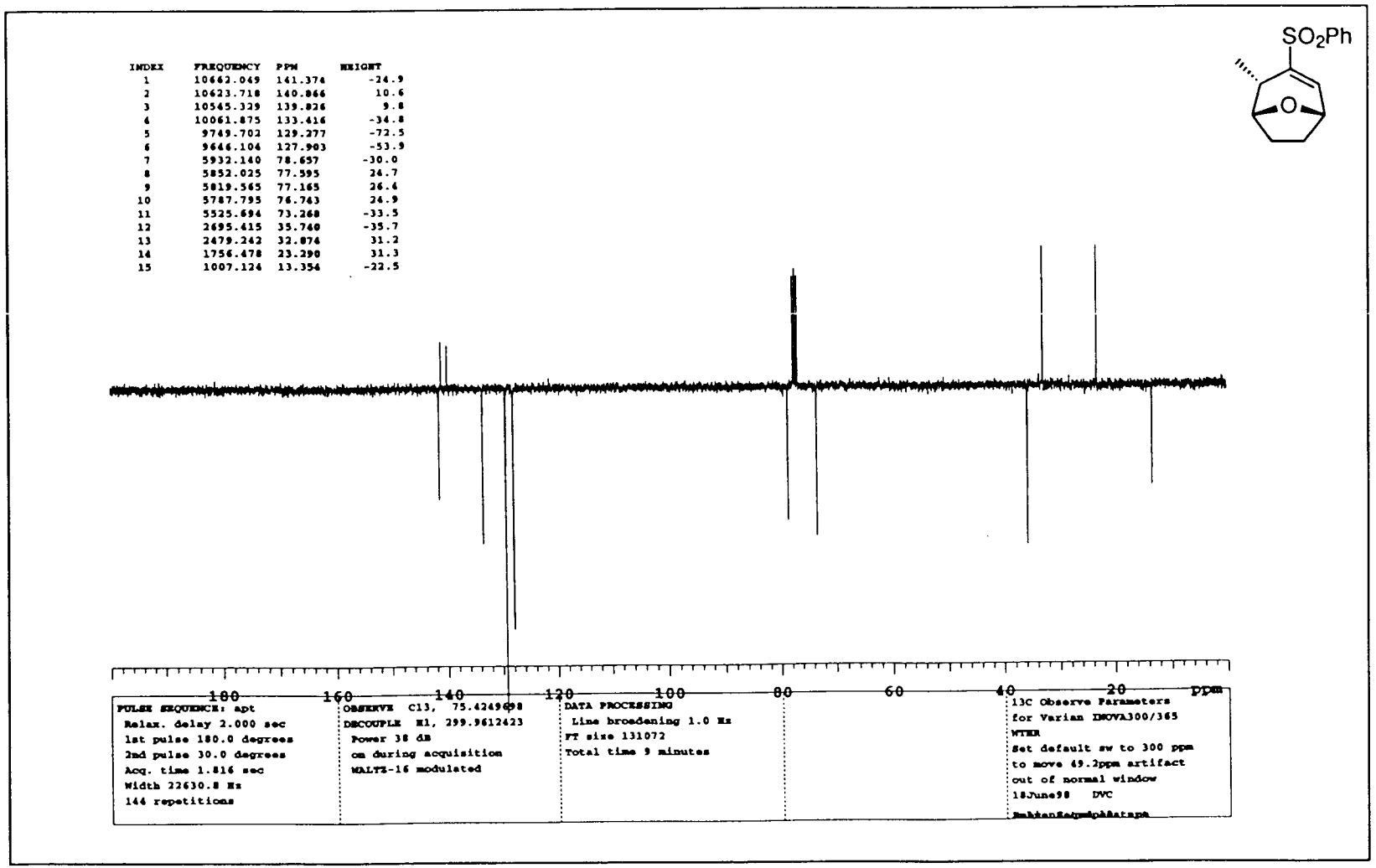

Figure 34 75MHz ^{13}C NMR of compound **20** in $CDCl_3$

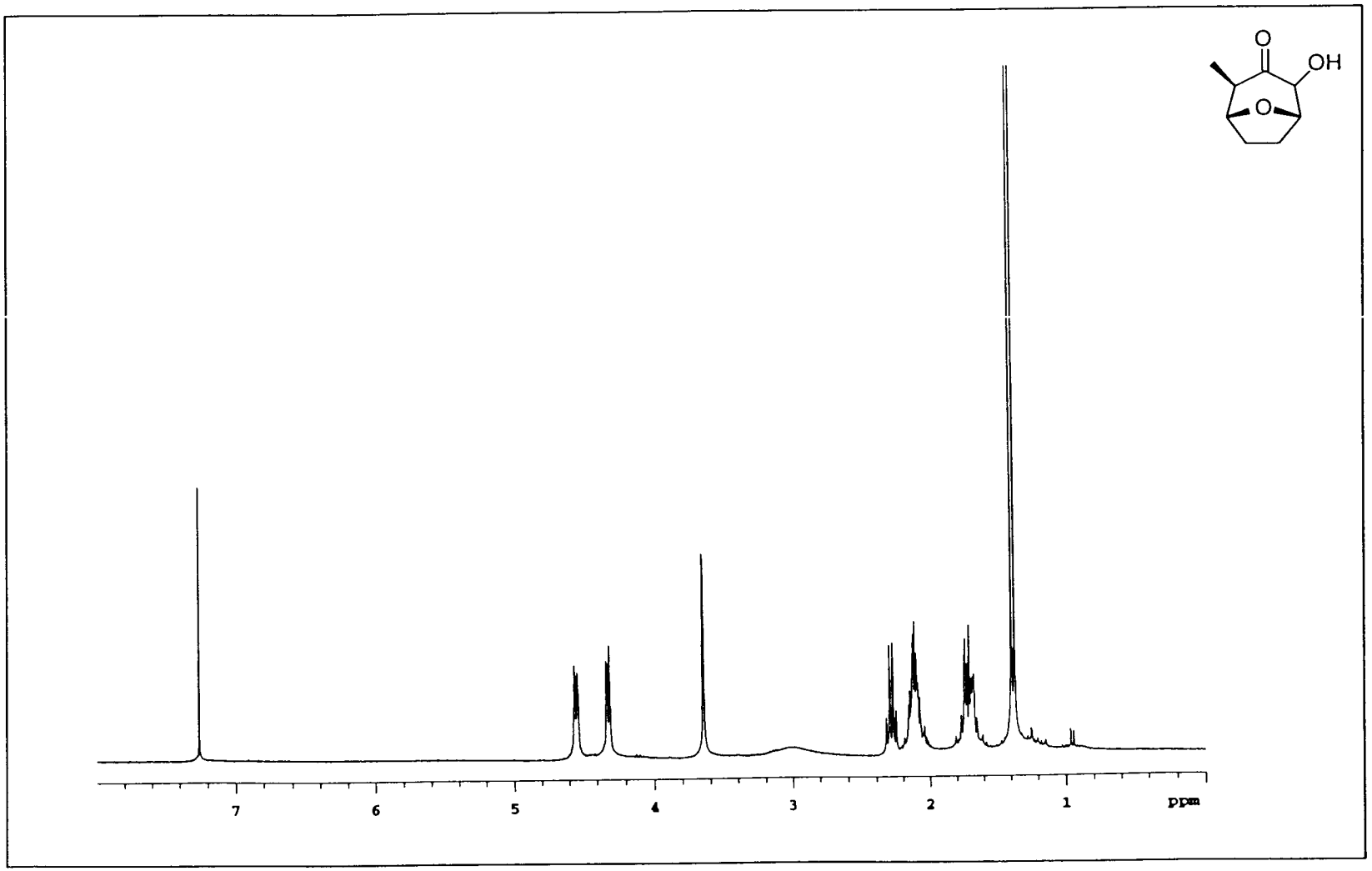

Figure 35 300MHz ^{1}H NMR of compound **21** in $CDCl_3$

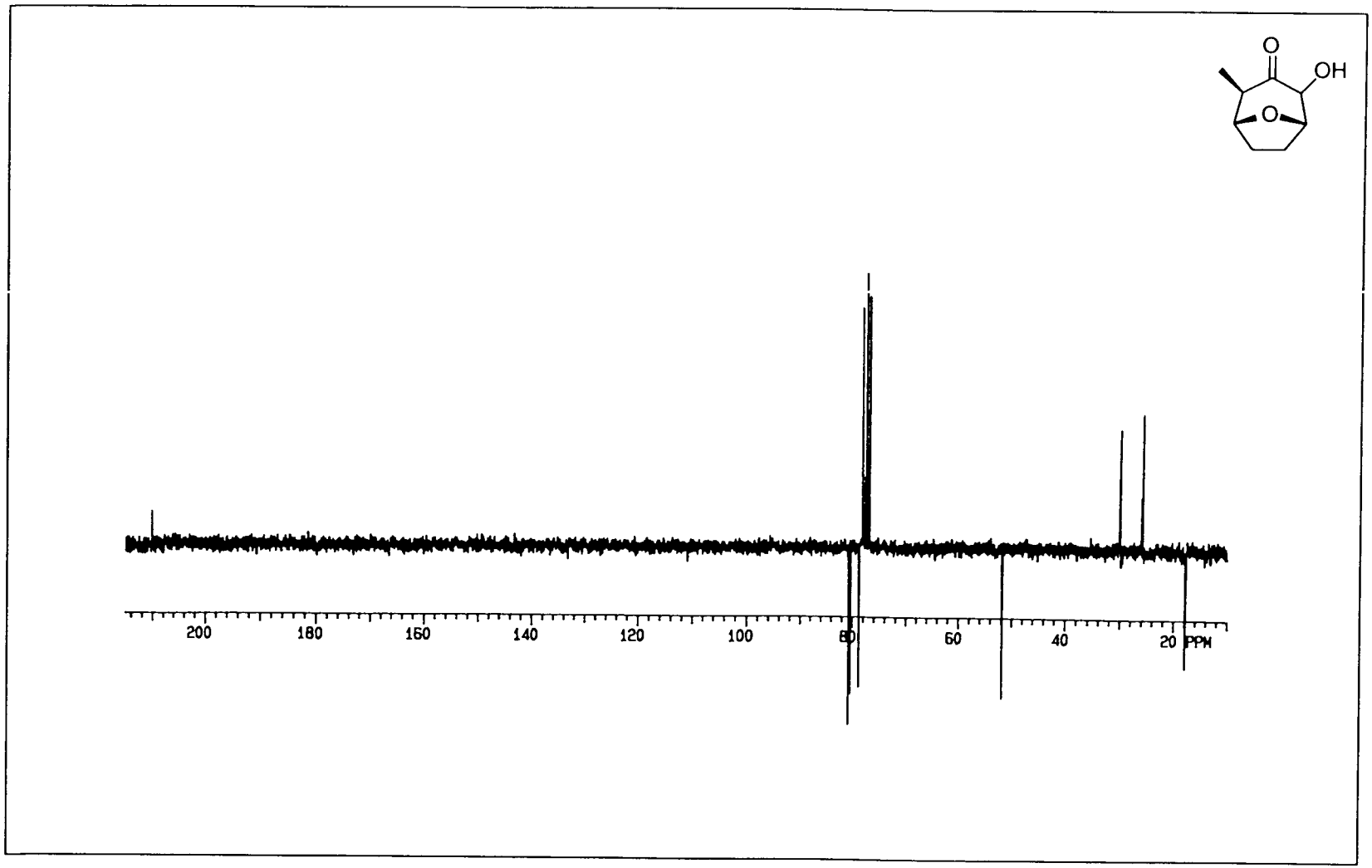

Figure 36 75MHz ^{13}C NMR of compound **21** in $CDCl_3$

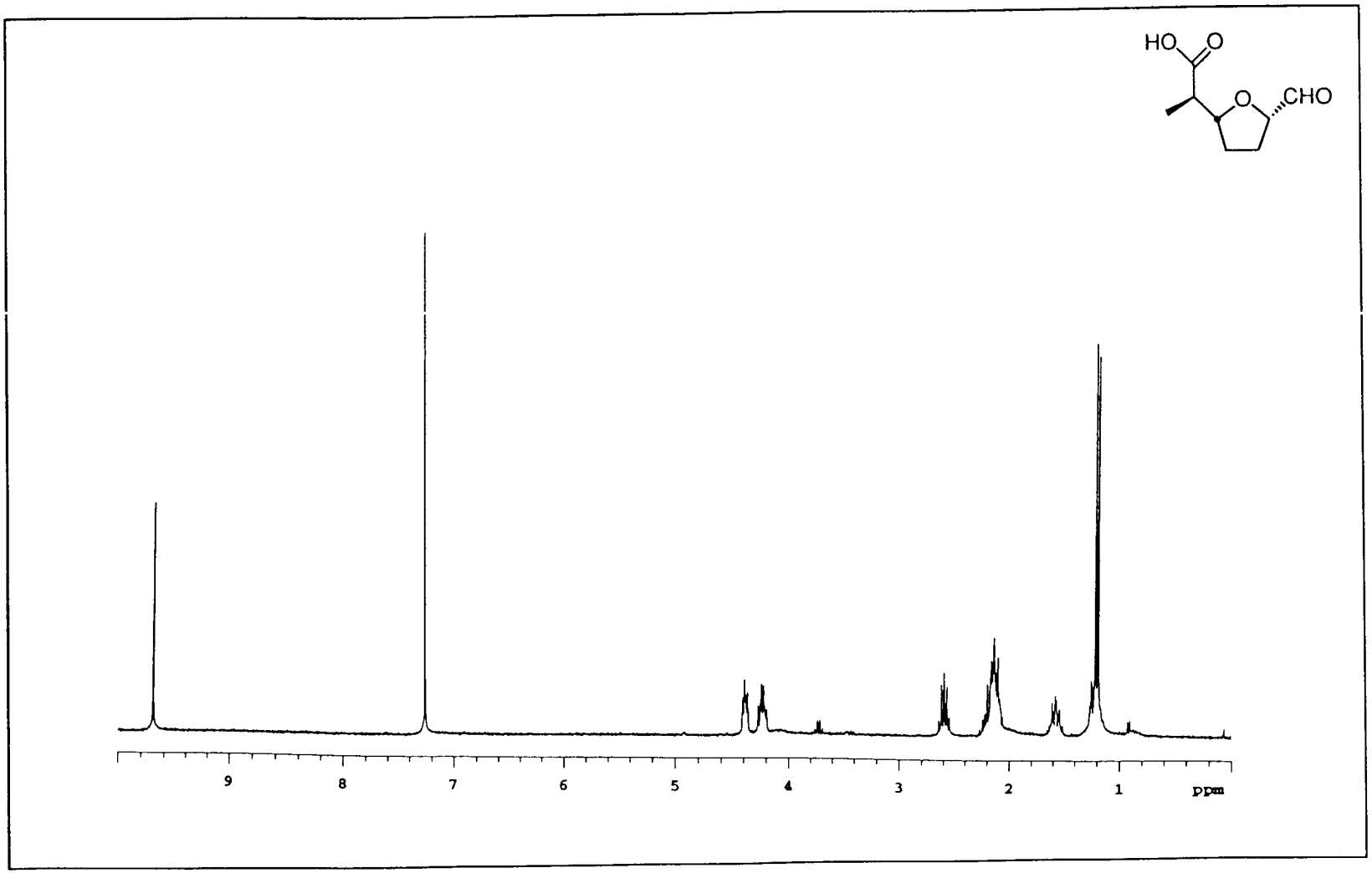

Figure 37 300MHz ^{1}H NMR of compound **22** in $CDCl_3$

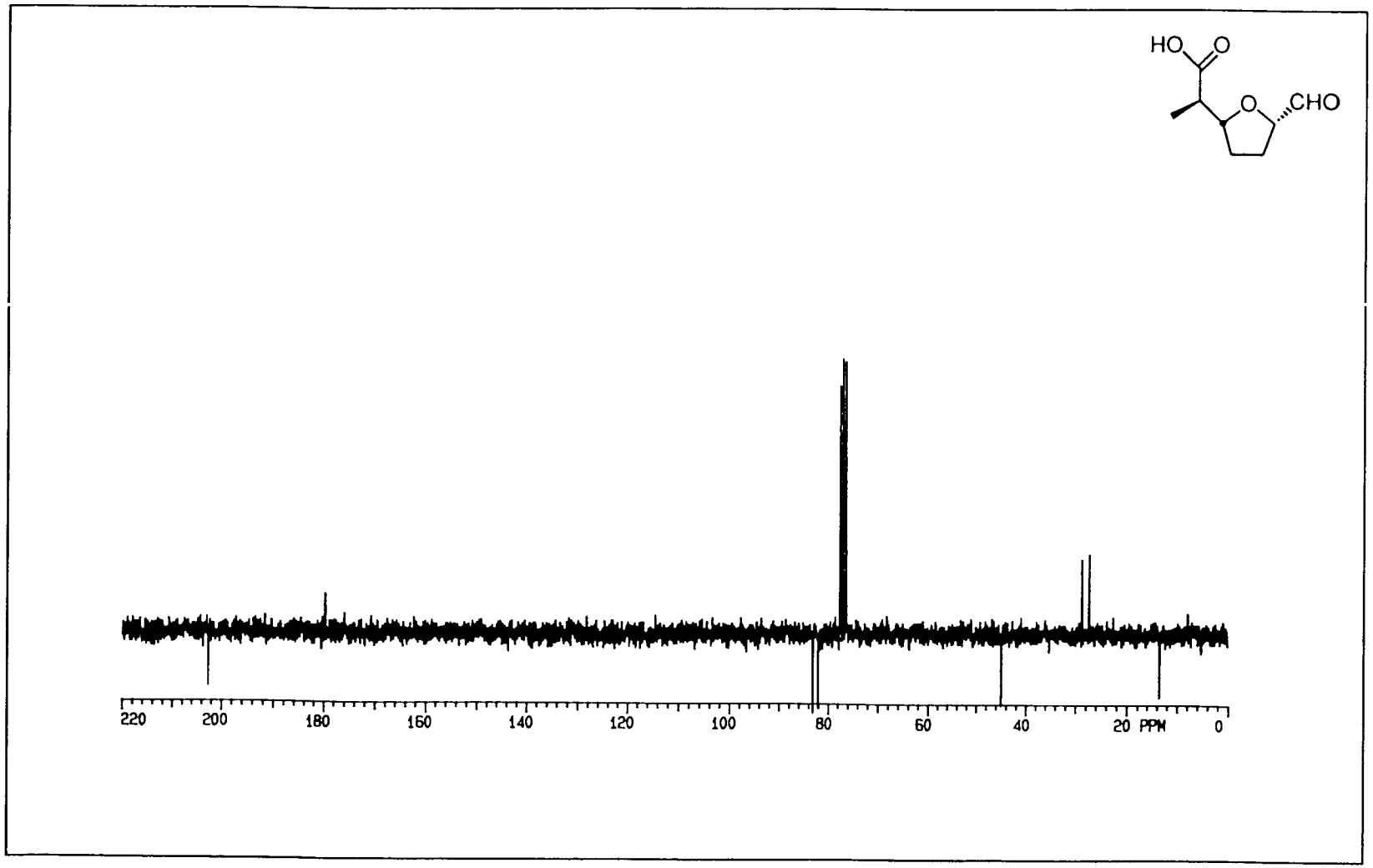

Figure 38 75MHz ^{13}C NMR of compound **22** in $CDCl_3$

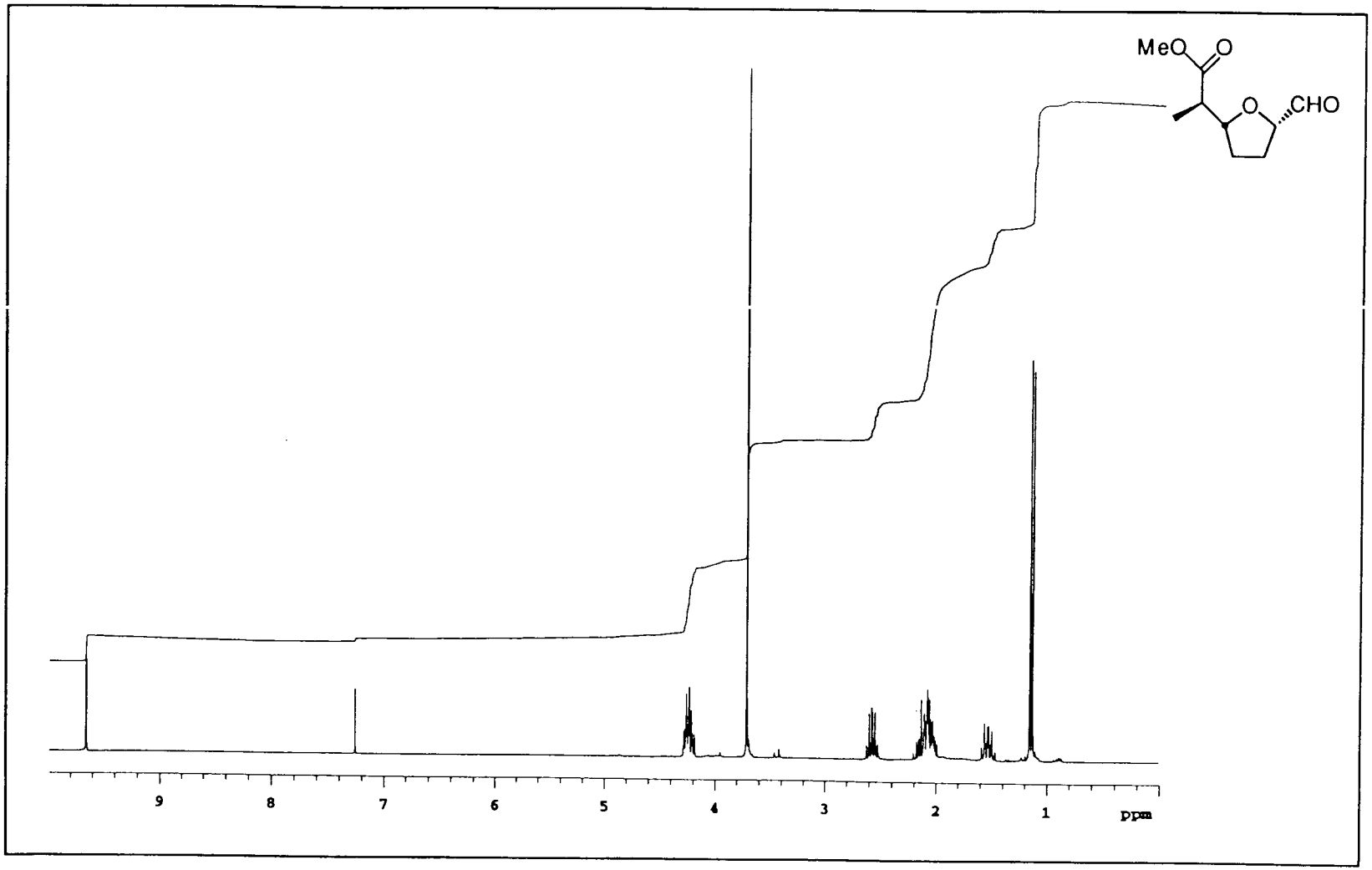

Figure 39 300MHz ^{1}H NMR of compound **23** in $CDCl_3$

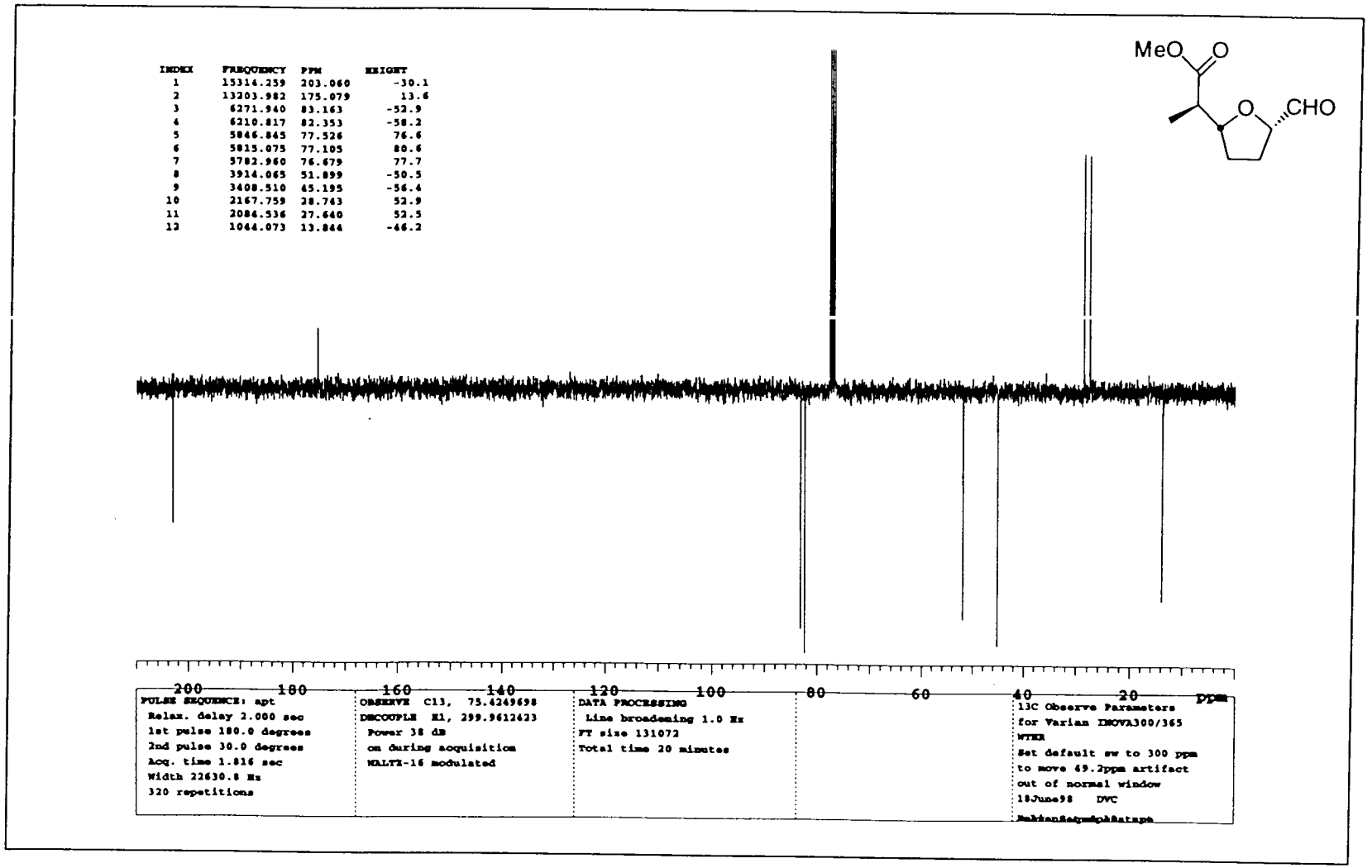

Figure 40 75MHz ^{13}C NMR of compound **23** in $CDCl_3$

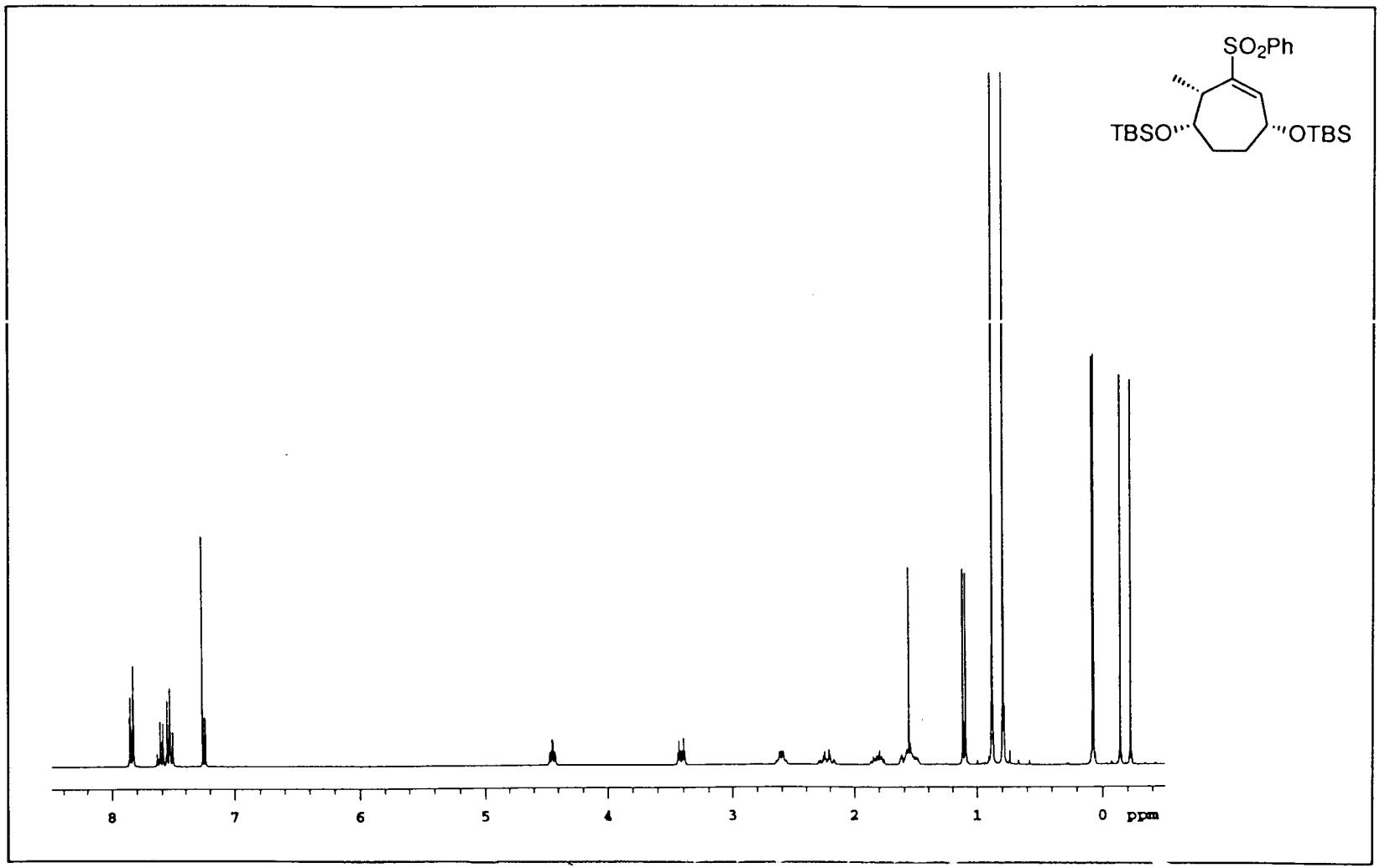

Figure 41 300MHz ^{1}H NMR of compound **24** in $CDCl_3$

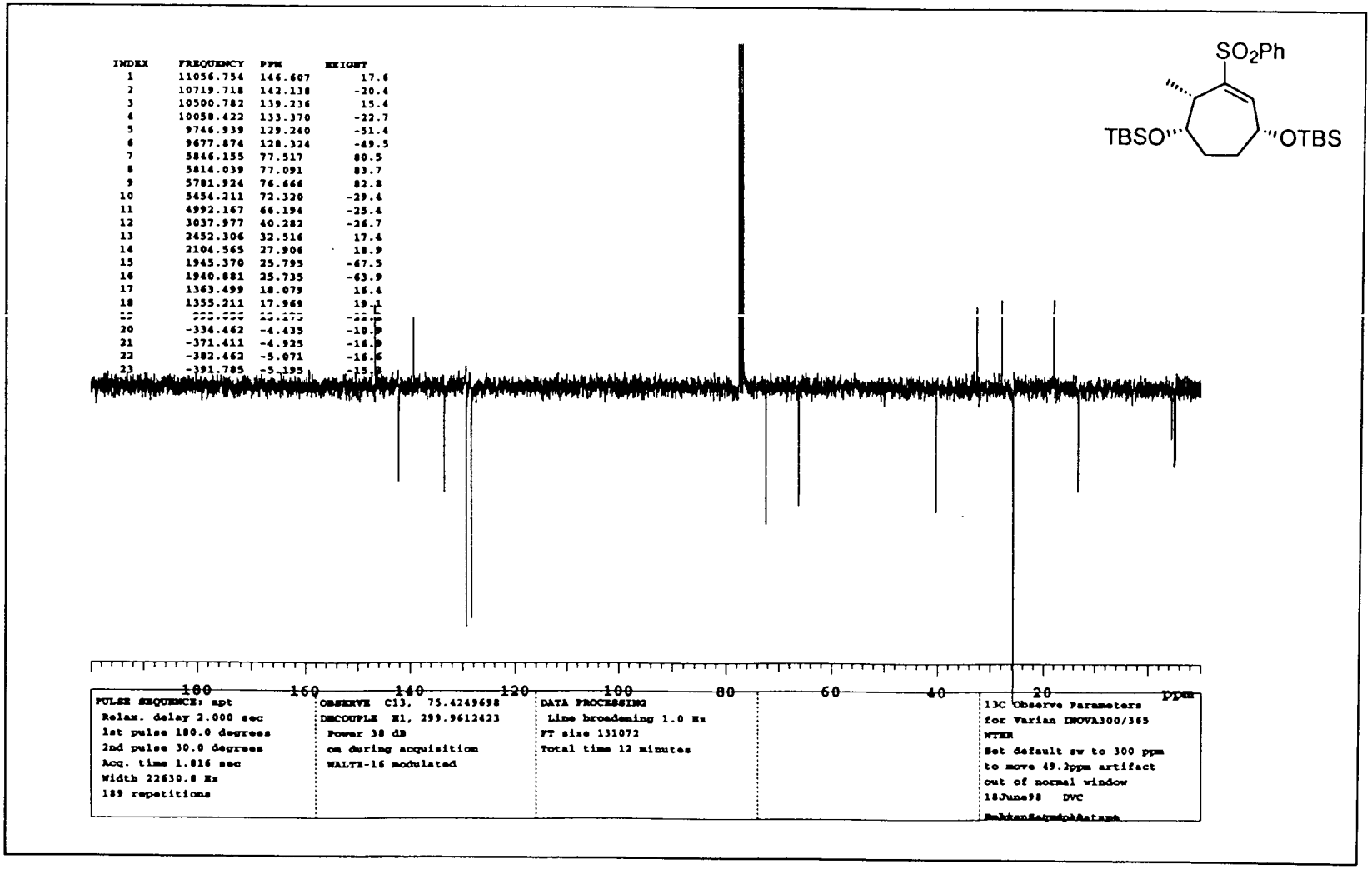

Figure 42 75MHz ^{13}C NMR of compound **24** in $CDCl_3$

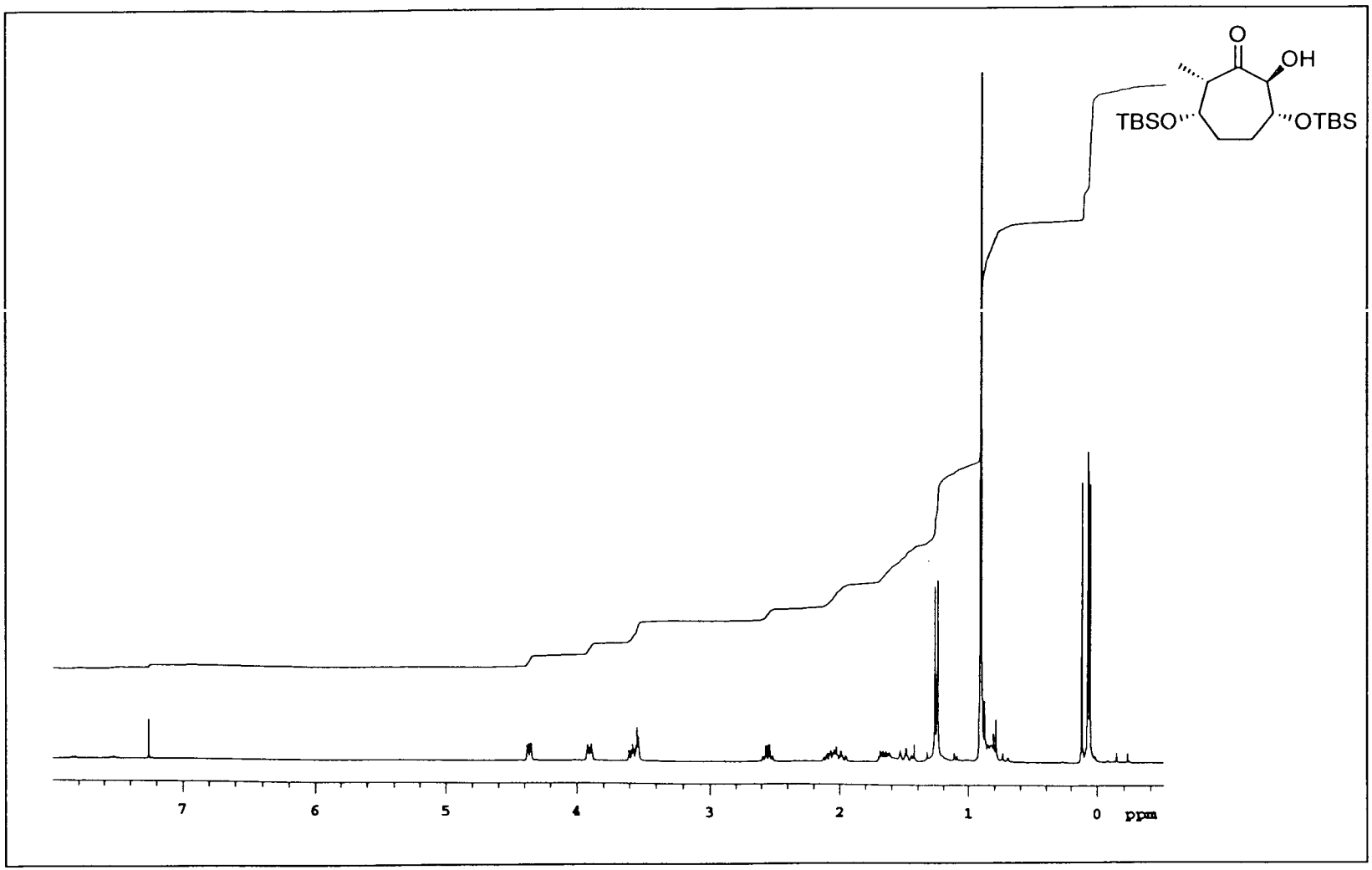

Figure 43 300MHz ^{1}H NMR of compound **25** in $CDCl_3$

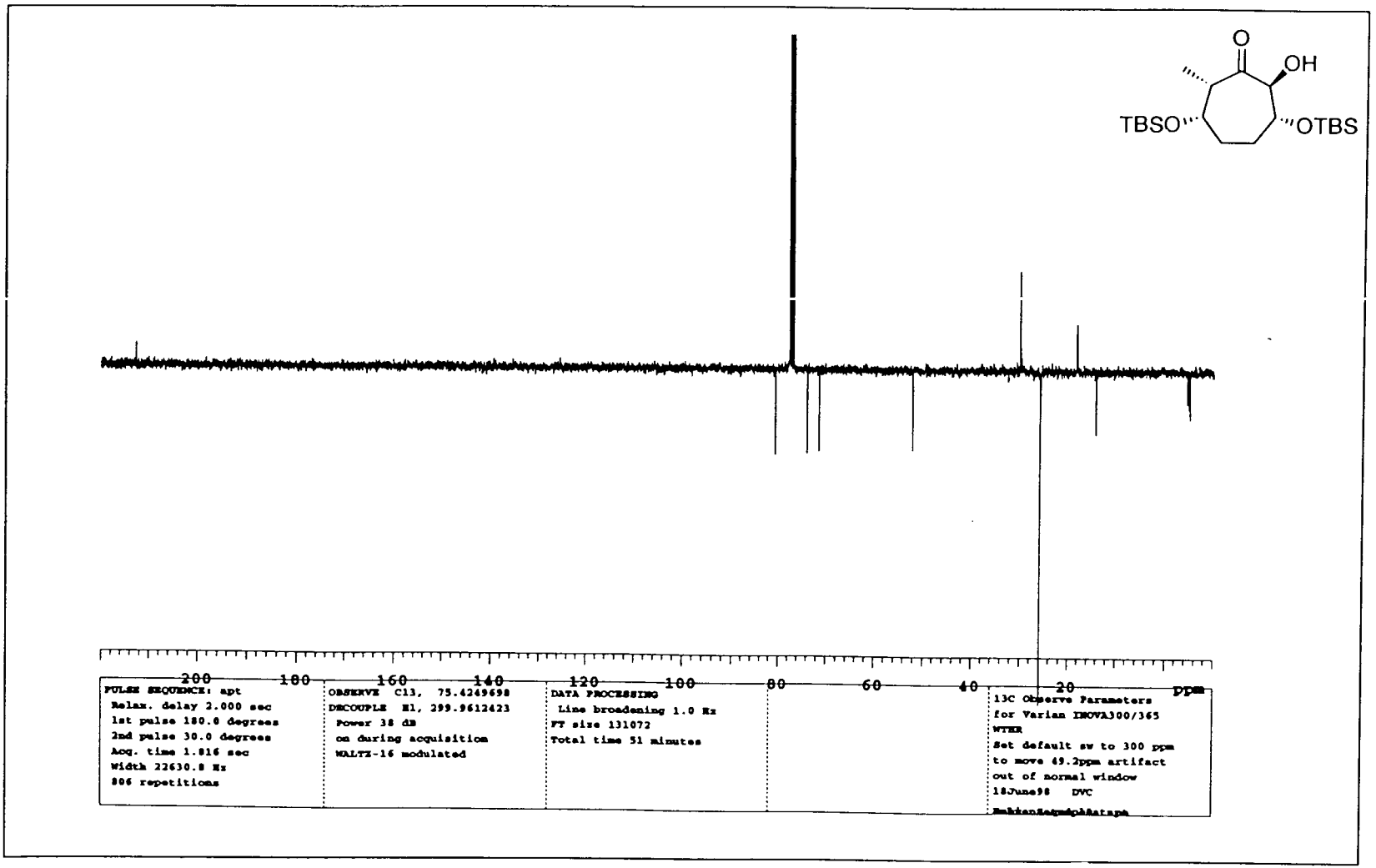

Figure 44 75MHz ^{13}C NMR of compound **25** in $CDCl_3$

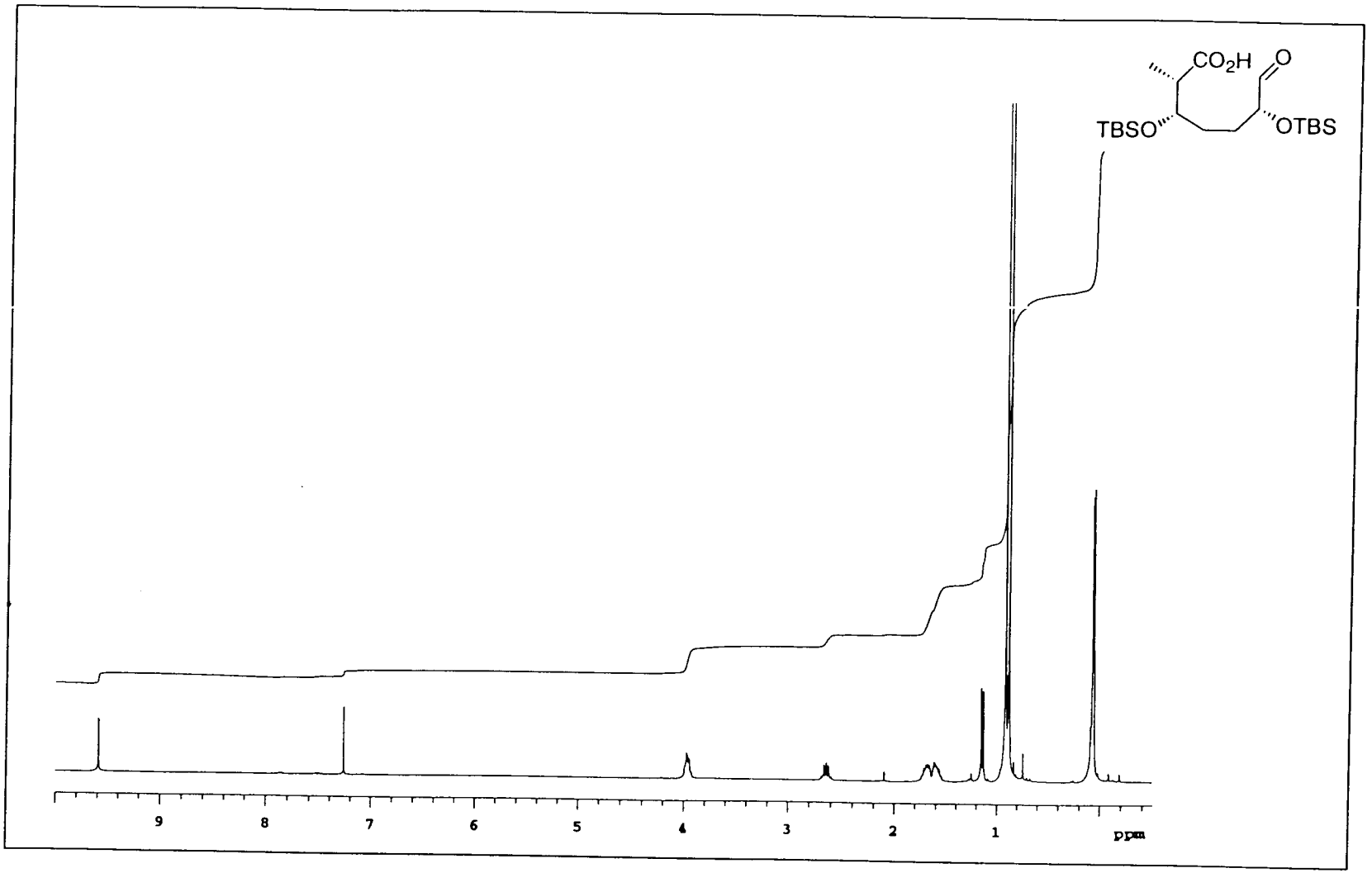

Figure 45 300MHz ^{1}H NMR of compound **26** in $CDCl_3$

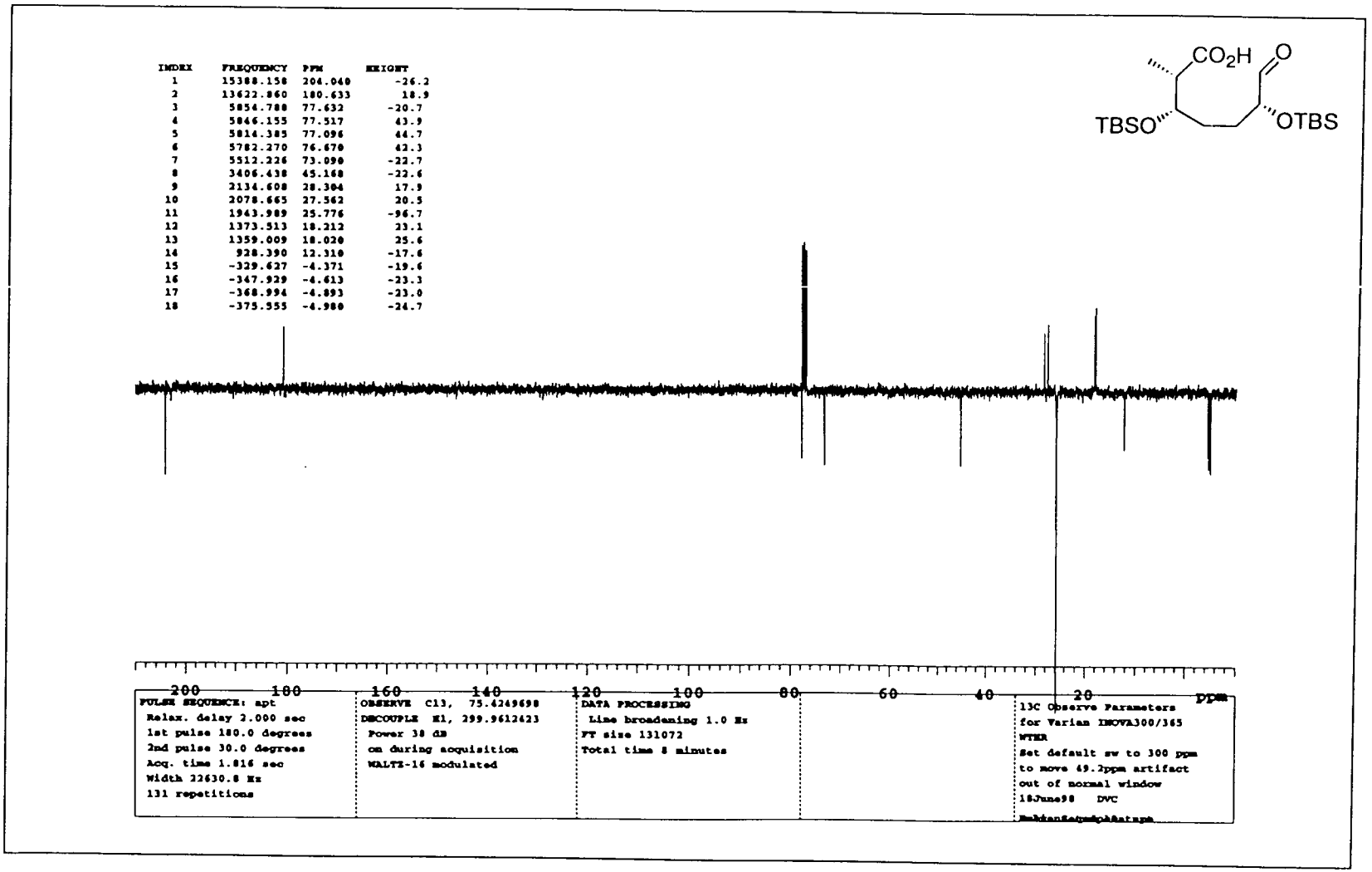

Figure 46 75MHz ^{13}C NMR of compound **26** in $CDCl_3$

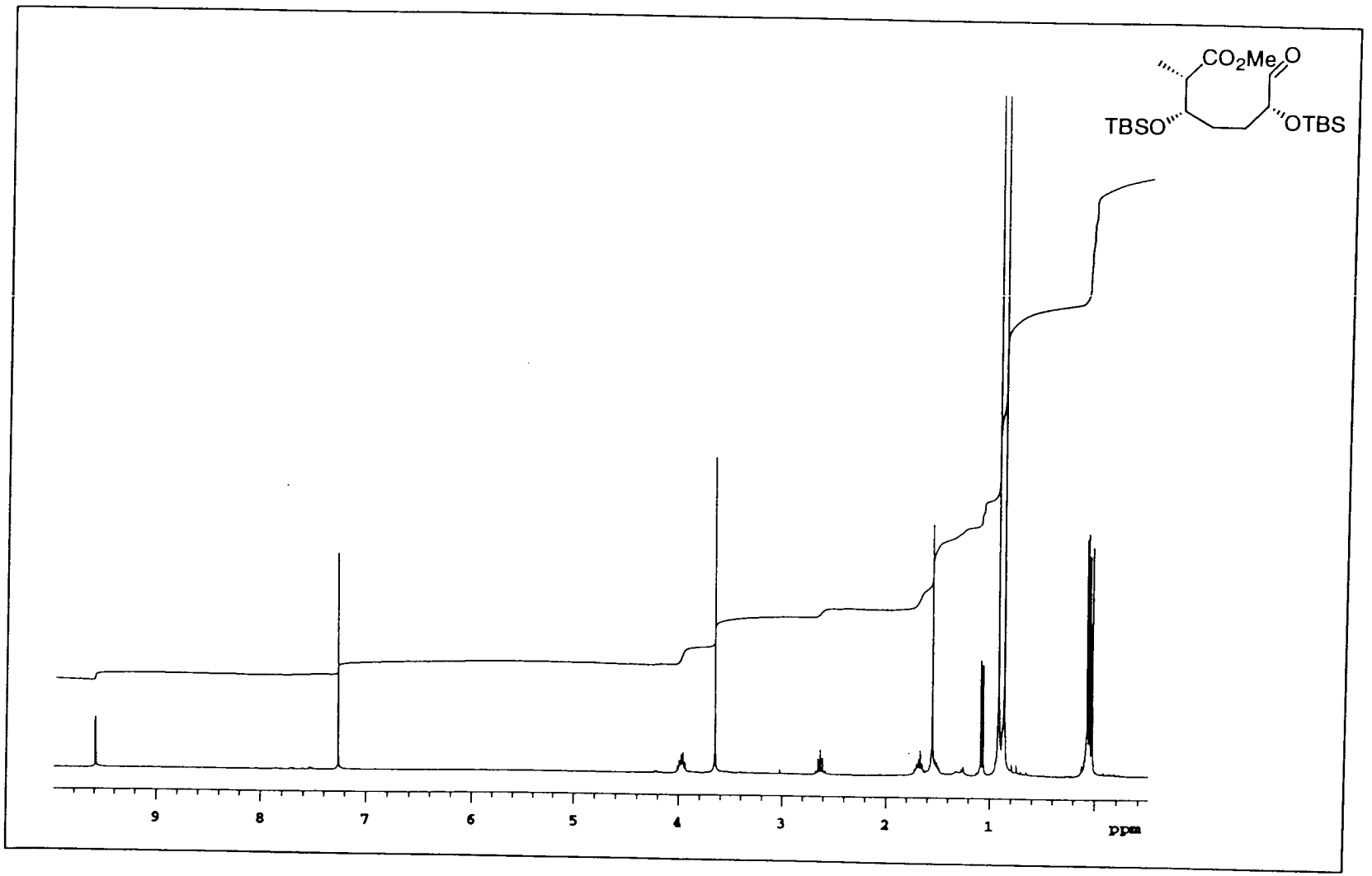

Figure 47 300MHz ^{1}H NMR of compound **27** in $CDCl_3$

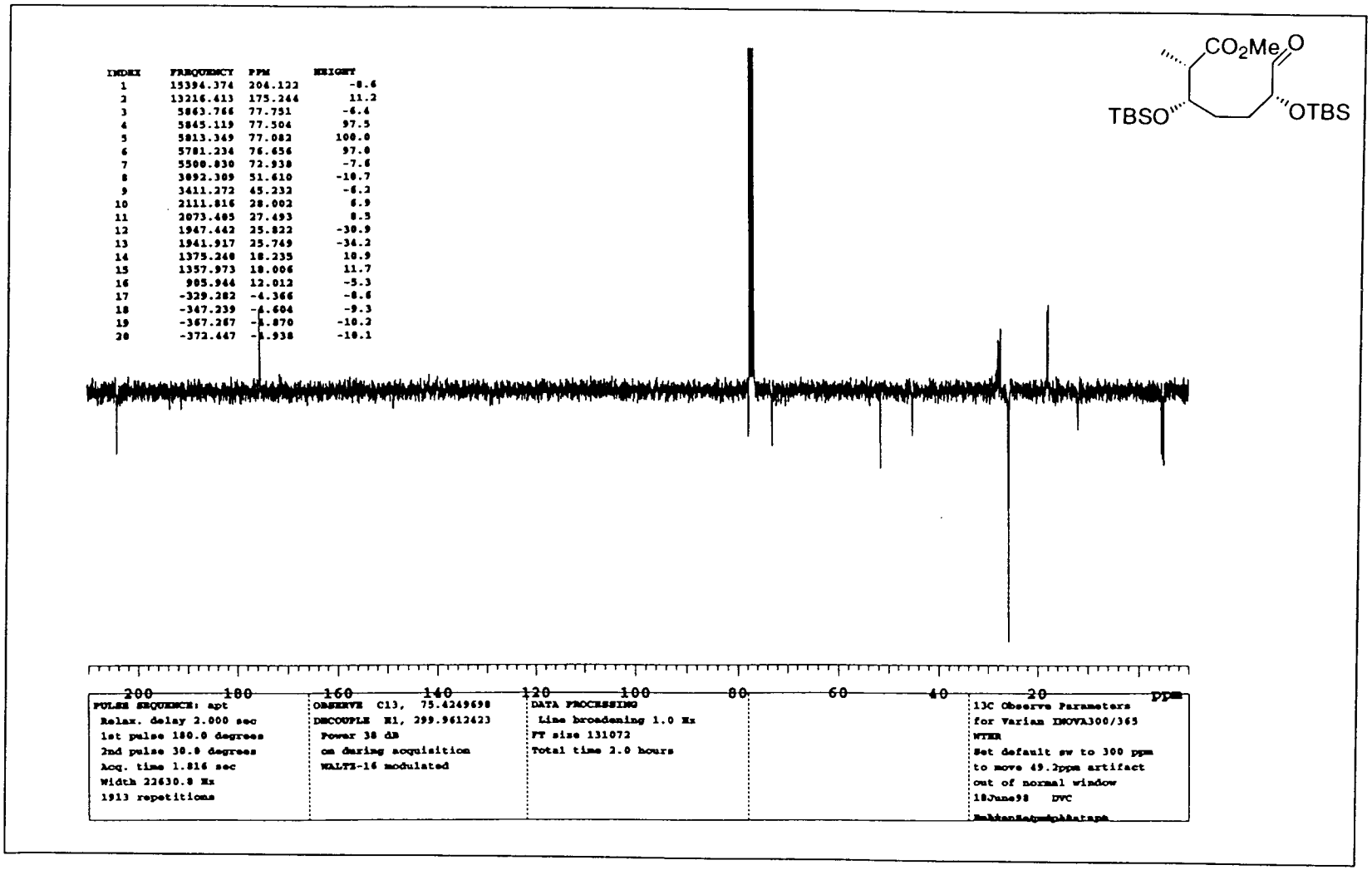

Figure 48 75MHz ^{13}C NMR of compound **27** in $CDCl_3$

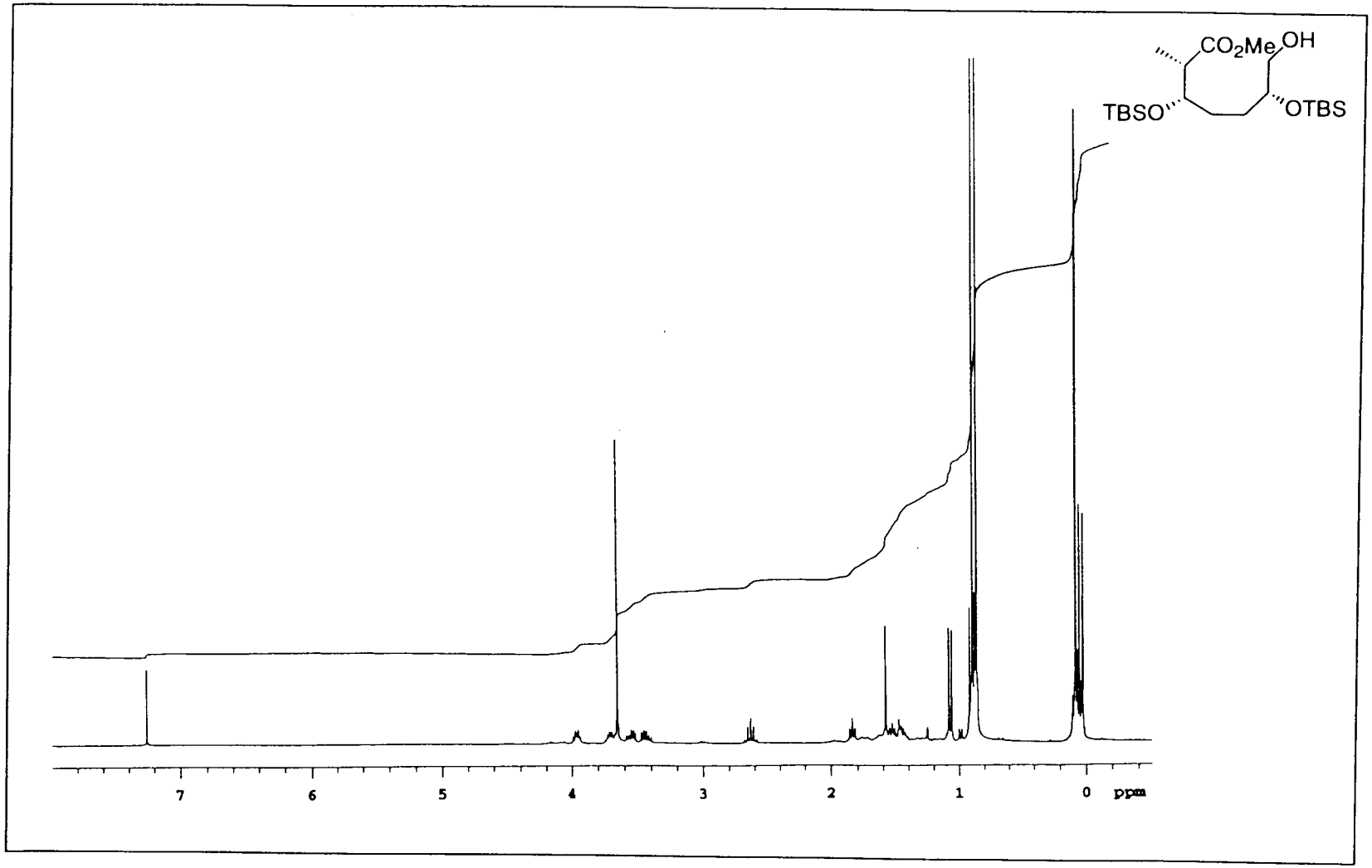

Figure 49 300MHz ^{1}H NMR of compound **28** in $CDCl_3$

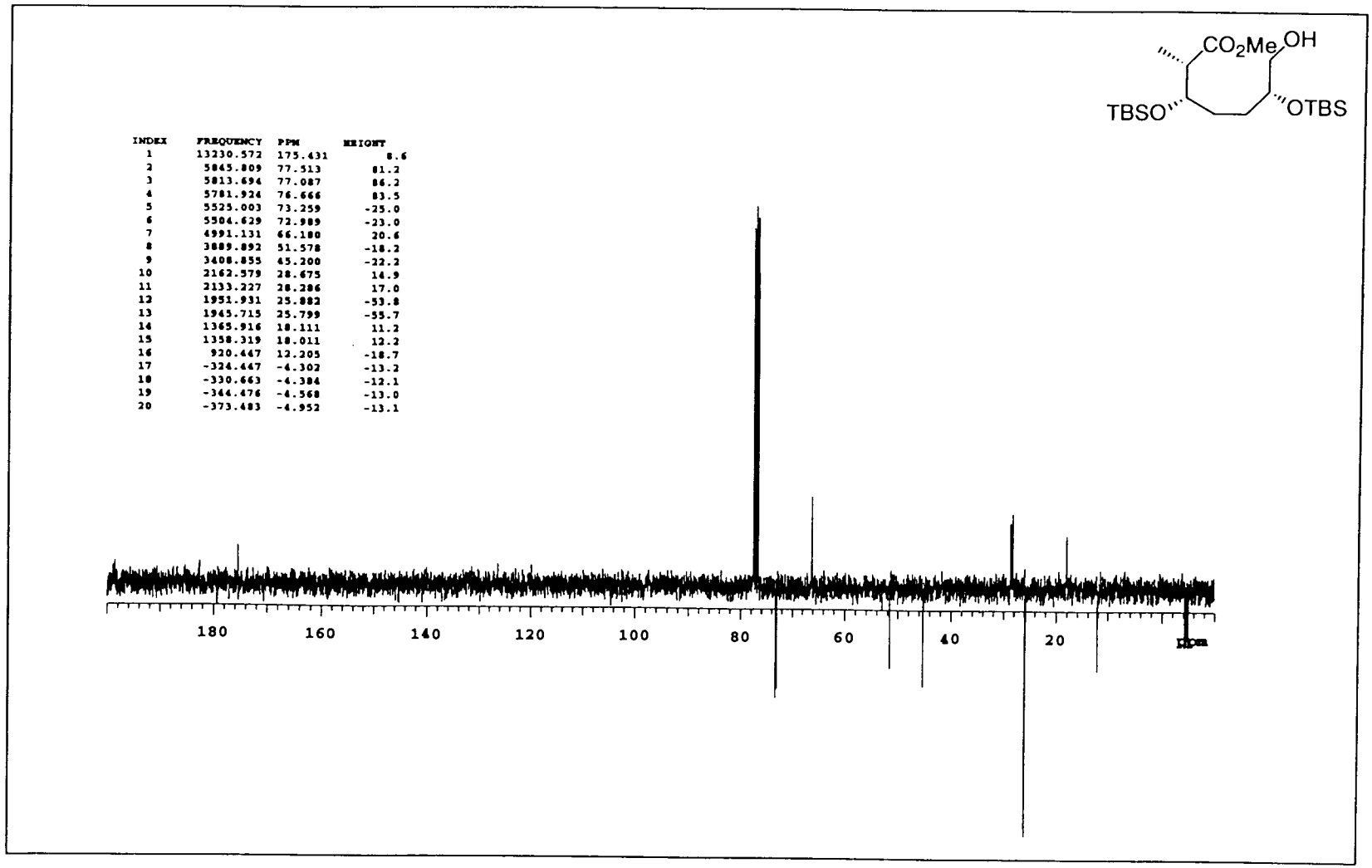

Figure 50 75MHz ^{13}C NMR of compound **28** in $CDCl_3$

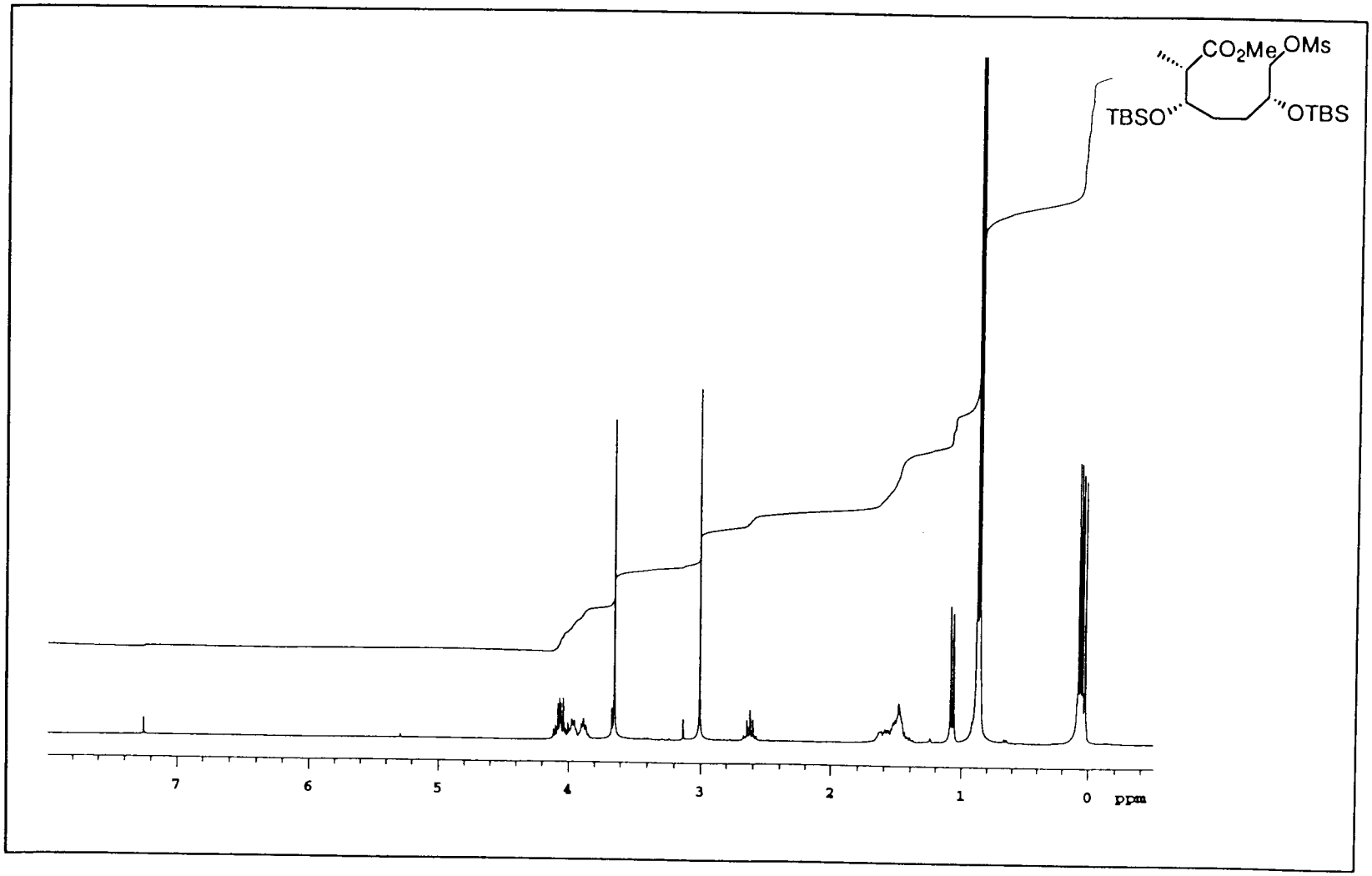

Figure 51 300MHz ^{1}H NMR of compound **29** in $CDCl_3$

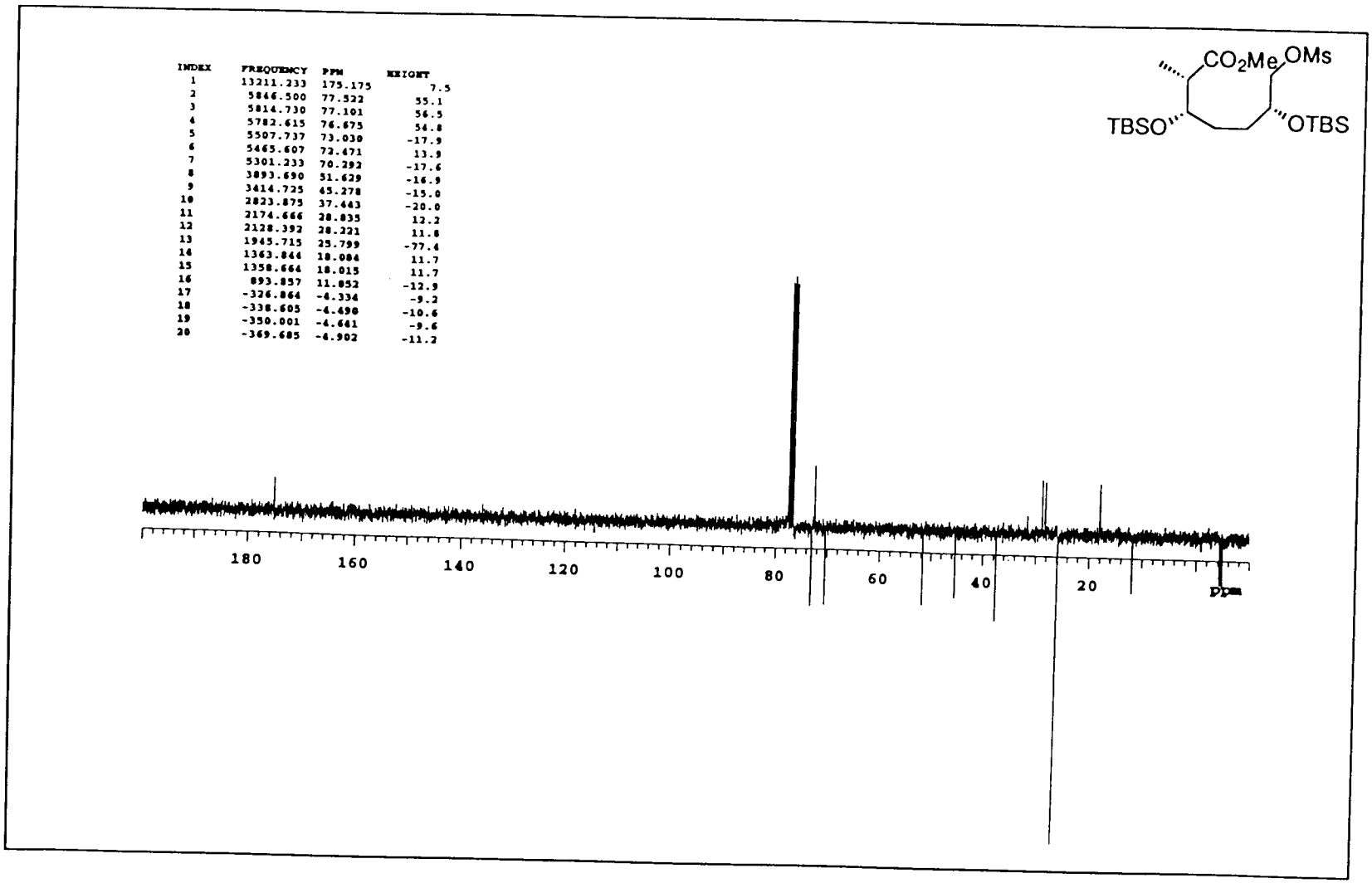

Figure 52 75MHz ^{13}C NMR of compound **29** in $CDCl_3$