Total synthesis of cortistatins A and J

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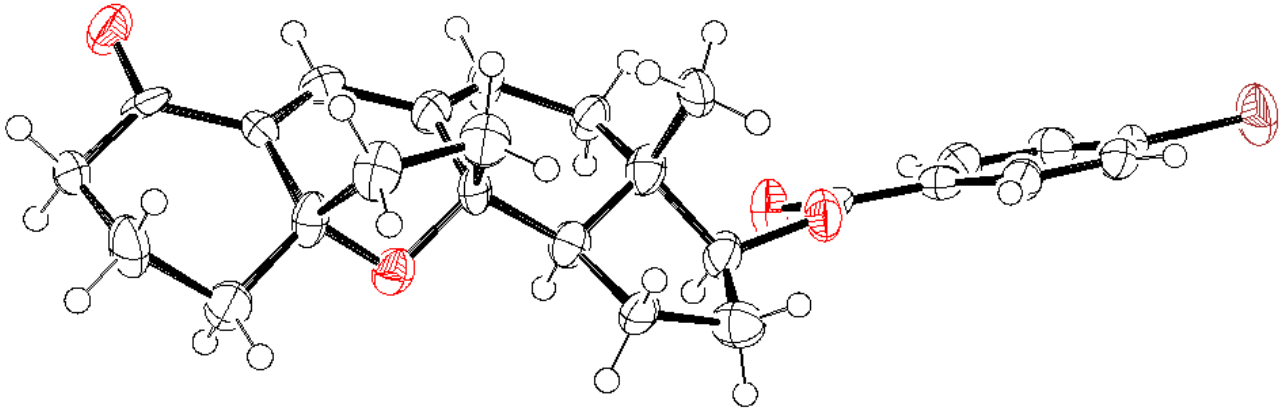

Data collection

A colorless prism crystal of $C_{26}H_{27}BrO_4$ having approximate dimensions of 0.28 × 0.14 × 0.05 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn CCD area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 0 images that were exposed for 0 seconds. The crystal-to-detector distance was 50.84 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions: a = 21.235(17) Å, b = 8.271(7) Å, c = 13.904(11) Å, β = 92.777(11)°, V = 2439(3) $Å^3$. For Z = 4 and F.W. = 483.40, the calculated density is 1.316 g/cm^3. Based on the systematic absences of: hkl: h+k ± 2n

Packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be C2 (#5).

The data were collected at a temperature of -100 ± 1 °C to a maximum 2θ value of 55.0°. A total of 2400 oscillation images were collected. A sweep of data was done using ω scans from -110.0° to 70.0° in 0.3° step, at χ = 45.0° and ϕ = 0.0°. The exposure rate was 53.3 [sec./°]. The detector swing

angle was -19.67°. A second sweep was performed using ω scans from -110.0° to 70.0° in 0.3° step, at χ = 45.0° and ϕ = 90.0°. The exposure rate was 53.3 [sec./°]. The detector swing angle was -19.67°. Another sweep was performed using ω scans from -110.0° to 70.0° in 0.3° step, at χ = 45.0° and ϕ = 180.0°. The exposure rate was 53.3 [sec./°]. The detector swing angle was -19.67°. Another sweep was performed using ω scans from -110.0° to 70.0° in 0.3° step, at χ = 45.0° and ϕ = 270.0°. The exposure rate was 53.3 [sec./°]. The detector swing angle was -19.67°. The crystal-to-detector distance was 50.84 mm. Readout was performed in the 0.547 mm pixel mode.

Data reduction

Of the 17216 reflections that were collected, 5457 were unique (R_{int} = 0.052); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku)[1]. The linear absorption coefficient, μ, for Mo-Kα radiation is 17.177 cm^{-1}. A numerical absorption correction was applied which resulted in transmission factors ranging from 0.751 to 0.918. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction[2] was applied (coefficient = 445.709992).

Structure Solution and Refinement

The structure was solved by direct methods[3] and expanded using Fourier techniques[4]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[5] on F^2 was based on 5457 observed reflections and 309 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of R1 = 0.0687, wR2 = 0.1967. The standard deviation of an observation of unit weight[6] was 1.01. A Sheldrick weighting scheme was used. Plots

of Σw ($|Fo| - |Fc|)^2$versus $|Fo|$, 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 3.03 and -0.94 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.006(19), refined using 2528 Friedel pairs.[7] Neutral atom scattering factors were taken from Cromer and Waber[8]. Anomalous dispersion effects were included in Fcalc[9]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[10]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[11]. All calculations were performed using the CrystalStructure[12,13] crystallographic software package.

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) Larson, A.C. (1970), Crystallographic Computing, 291-294. F.R. Ahmed, ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume).

(3) SHELX97: Sheldrick, G.M. (1997).

(4) DIRDIF99: Beurskens, P.T.; Admiraal, G., Beurskens, G., Bosman, W.P.; de Gelder, R.; Israel, R.; Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(5) Least Squares function minimized:

$$\Sigma w(Fo^2-Fc^2)^2 \qquad \text{where w = Least Squares weights.}$$

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

$$[\Sigma w(Fo^2-Fc^2)^2/(No-Nv)]^{1/2}$$

where: No = number of observations

Nv = number of variables

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{26}H_{27}BrO_4$
Formula Weight	483.40
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.28 X 0.14 X 0.05 mm
Crystal System	monoclinic
Lattice Type	C-centered
Detector Position	50.84 mm
Pixel Size	0.137 mm
Lattice Parameters	a = 21.235(17) Å b = 8.271(7) Å c = 13.904(11) Å β = 92.777(11) ^{o} V = 2439(3) Å^3
Space Group	C2 (#5)

Z value	4
D_{calc}	1.316 g/cm^3
F_{000}	1000.00
μ(MoKα)	17.177 cm^{-1}

B. Intensity Measurements

Detector	Rigaku Saturn
Goniometer	Rigaku AFC10
Radiation	MoKα (λ = 0.71070 Å) graphite monochromated
Detector Aperture	70 mm x 70 mm
Data Images	2400 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	-110.0 - 70.0^{o}
Exposure Rate	53.3 sec./^{o}
Detector Swing Angle	-19.67^{o}
ω oscillation Range (χ=45.0, ϕ=90.0)	-110.0 - 70.0^{o}
Exposure Rate	53.3 sec./^{o}
Detector Swing Angle	-19.67^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	-110.0 - 70.0^{o}
Exposure Rate	53.3 sec./^{o}
Detector Swing Angle	-19.67^{o}

ω oscillation Range (χ=45.0, ϕ=270.0)	-110.0 - 70.0^{o}
Exposure Rate	53.3 sec./^{o}
Detector Swing Angle	-19.67^{o}
Detector Position	50.84 mm
Pixel Size	0.137 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 17216 Unique: 5457 (R_{int} = 0.052) Friedel pairs: 2528
Corrections	Lorentz-polarization Absorption (trans. factors: 0.751 - 0.918) Secondary Extinction (coefficient: 4.45710e+002)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0012Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	5457
No. Variables	309
Reflection/Parameter Ratio	17.66
Residuals: R1 ($I>2.00\sigma(I)$)	0.0687
Residuals: R (All reflections)	0.0984
Residuals: wR2 (All reflections)	0.1967
Goodness of Fit Indicator	1.012
Flack Parameter (Friedel pairs = 2528)	0.006(19)
Max Shift/Error in Final Cycle	0.000

Maximum peak in Final Diff. Map	3.03 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.94 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Br1	0.98691(4)	-0.0326(2)	-0.37681(6)	4.33(2)
O1	0.7402(2)	-0.0644(7)	0.6918(3)	3.31(13)
O2	0.6637(2)	0.0893(6)	0.3780(3)	2.56(12)
O3	0.8073(2)	0.0535(6)	0.0126(3)	3.14(13)
O4	0.8866(2)	0.2300(7)	0.0578(4)	3.80(14)
C1	0.6963(3)	-0.0327(12)	0.6360(4)	2.51(14)
C2	0.6344(3)	0.0053(10)	0.6745(5)	2.95(19)
C3	0.5771(3)	-0.0104(14)	0.5988(4)	3.60(19)
C4	0.5914(3)	0.0821(11)	0.5050(5)	3.22(19)
C5	0.6456(3)	-0.0051(11)	0.4601(4)	2.60(17)
C6	0.6270(4)	-0.1683(10)	0.4106(6)	3.2(2)
C7	0.6741(4)	-0.1817(10)	0.3301(6)	3.3(2)
C8	0.7075(3)	-0.0151(11)	0.3293(4)	2.31(15)
C9	0.7698(3)	-0.0150(12)	0.3914(4)	2.60(16)
C10	0.7047(3)	-0.0258(12)	0.5315(4)	2.21(13)
C11	0.8258(3)	0.0103(11)	0.3548(5)	3.6(2)
C12	0.8364(3)	0.0451(10)	0.2504(4)	2.75(17)
C13	0.7780(3)	0.0007(9)	0.1857(5)	2.83(18)
C14	0.7182(3)	0.0614(10)	0.2326(5)	2.55(17)
C15	0.6680(3)	0.0504(9)	0.1534(4)	2.78(18)
C16	0.7019(3)	0.0929(10)	0.0630(6)	3.4(2)
C17	0.7683(4)	0.1041(9)	0.0932(5)	2.95(19)
C18	0.7766(4)	-0.1741(10)	0.1623(5)	3.6(2)
C19	0.7607(3)	-0.0280(13)	0.4945(4)	2.62(14)
C20	0.8618(3)	0.1332(9)	0.0014(5)	2.49(17)
C21	0.8894(3)	0.0882(8)	-0.0920(5)	2.43(17)
C22	0.9499(4)	0.1609(10)	-0.1113(6)	3.2(2)
C23	0.9766(4)	0.1259(10)	-0.1967(6)	3.4(2)
C24	0.9452(3)	0.0166(8)	-0.2634(5)	2.61(18)
C25	0.8887(3)	-0.0537(11)	-0.2458(4)	2.64(17)

C26	0.8600(3)	-0.0170(12)	-0.1609(4)	2.53(15)

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

Table 2. Atomic coordinates and B $_{iso}$ involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H1	0.6357	0.1132	0.6979	3.56
H2	0.6276	-0.0667	0.7262	3.57
H3	0.5405	0.0348	0.6251	4.36
H4	0.5698	-0.1212	0.5843	4.36
H5	0.9708	0.2308	-0.0658	3.85
H6	0.6030	0.1906	0.5198	3.88
H7	0.5554	0.0817	0.4617	3.87
H8	0.5848	-0.1650	0.3844	3.89
H9	0.8202	-0.0629	-0.1487	3.03
H10	0.6315	-0.2557	0.4547	3.90
H11	0.7969	-0.0399	0.5368	3.11
H12	0.7038	-0.2653	0.3444	3.99
H13	0.6528	-0.2029	0.2697	3.98
H14	0.8653	-0.0015	0.3973	4.30
H15	0.7246	0.1734	0.2446	3.05
H16	0.6351	0.1256	0.1636	3.30
H17	0.6509	-0.0557	0.1492	3.30
H18	0.6870	0.1930	0.0371	4.14
H20	0.6956	0.0103	0.0160	4.14
H21	0.8451	0.1570	0.2429	3.28
H22	0.8713	-0.0166	0.2312	3.29
H23	0.8185	-0.2144	0.1620	4.34
H24	0.7541	-0.2306	0.2093	4.35
H25	0.7562	-0.1895	0.1007	4.34
H26	0.8692	-0.1262	-0.2912	3.16
H27	1.0156	0.1738	-0.2119	4.14
H19	0.7779	0.2135	0.1087	3.55

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

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br1	0.0441(5)	0.0808(7)	0.0407(4)	-0.0006(6)	0.0138(3)	-0.0151(6)
O1	0.041(3)	0.057(4)	0.027(2)	0.003(3)	-0.004(2)	0.006(2)
O2	0.029(3)	0.040(3)	0.028(3)	-0.003(2)	-0.004(2)	-0.001(2)
O3	0.058(4)	0.035(3)	0.027(3)	-0.002(2)	0.012(2)	-0.003(2)
O4	0.052(3)	0.061(4)	0.032(3)	-0.012(3)	0.007(2)	-0.014(3)
C1	0.027(3)	0.036(4)	0.032(3)	0.006(4)	-0.008(2)	0.018(4)
C2	0.036(4)	0.049(6)	0.027(3)	0.014(4)	0.012(3)	0.005(3)
C3	0.036(4)	0.067(6)	0.035(3)	-0.001(5)	0.015(3)	-0.008(5)
C4	0.026(4)	0.061(6)	0.035(4)	0.006(4)	0.003(3)	0.005(4)
C5	0.027(3)	0.051(6)	0.021(3)	-0.011(4)	-0.001(2)	-0.005(4)
C6	0.049(5)	0.038(5)	0.036(4)	-0.016(4)	0.004(4)	0.001(4)
C7	0.047(5)	0.038(5)	0.041(5)	-0.005(4)	0.003(4)	0.000(4)
C8	0.034(3)	0.034(4)	0.020(3)	-0.004(4)	-0.001(2)	-0.010(4)
C9	0.033(3)	0.040(4)	0.026(3)	0.003(4)	-0.003(2)	0.000(4)
C10	0.030(3)	0.029(3)	0.026(3)	0.009(4)	0.002(2)	-0.004(4)
C11	0.023(3)	0.090(8)	0.022(3)	0.006(4)	-0.003(2)	-0.006(4)
C12	0.026(4)	0.053(5)	0.025(4)	0.002(3)	-0.000(3)	0.004(3)
C13	0.047(4)	0.033(5)	0.028(3)	-0.009(4)	0.006(3)	0.003(3)
C14	0.033(4)	0.034(4)	0.029(4)	-0.005(3)	-0.000(3)	0.000(3)
C15	0.045(5)	0.035(4)	0.024(3)	0.005(3)	-0.010(3)	-0.003(3)
C16	0.036(5)	0.044(5)	0.051(5)	0.003(4)	0.004(4)	0.004(4)
C17	0.056(5)	0.031(4)	0.025(4)	-0.000(4)	0.004(3)	0.003(3)
C18	0.068(6)	0.041(5)	0.028(4)	0.015(4)	0.019(4)	0.003(4)
C19	0.029(3)	0.038(4)	0.031(3)	0.006(5)	-0.007(2)	0.004(4)
C20	0.038(4)	0.034(4)	0.023(4)	0.011(3)	0.002(3)	0.010(3)
C21	0.028(4)	0.026(4)	0.037(4)	0.003(3)	0.002(3)	0.007(3)
C22	0.037(5)	0.045(5)	0.039(4)	0.000(4)	-0.004(3)	-0.002(4)
C23	0.039(5)	0.046(5)	0.046(5)	0.009(4)	0.017(4)	0.013(4)
C24	0.039(4)	0.038(5)	0.022(3)	0.009(3)	0.002(3)	-0.004(3)
C25	0.034(4)	0.039(5)	0.028(3)	0.006(4)	0.003(2)	0.002(4)

C26	0.027(3)	0.039(4)	0.030(3)	0.001(4)	0.002(2)	-0.000(4)

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

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Br(1)	C(24)	1.890(7)	O(1)	C(1)	1.212(8)
O(2)	C(5)	1.450(9)	O(2)	C(8)	1.460(9)
O(3)	C(17)	1.485(9)	O(3)	C(20)	1.347(10)
O(4)	C(20)	1.222(9)	C(1)	C(2)	1.476(10)
C(1)	C(10)	1.473(8)	C(2)	C(3)	1.577(9)
C(3)	C(4)	1.555(11)	C(4)	C(5)	1.519(11)
C(5)	C(6)	1.558(12)	C(5)	C(10)	1.571(9)
C(6)	C(7)	1.541(12)	C(7)	C(8)	1.550(12)
C(8)	C(9)	1.543(9)	C(8)	C(14)	1.513(10)
C(9)	C(11)	1.332(10)	C(9)	C(19)	1.460(9)
C(10)	C(19)	1.319(9)	C(11)	C(12)	1.508(9)
C(12)	C(13)	1.539(10)	C(13)	C(14)	1.541(11)
C(13)	C(17)	1.550(10)	C(13)	C(18)	1.482(11)
C(14)	C(15)	1.498(10)	C(15)	C(16)	1.520(11)
C(16)	C(17)	1.455(11)	C(20)	C(21)	1.498(10)
C(21)	C(22)	1.456(11)	C(21)	C(26)	1.416(10)
C(22)	C(23)	1.371(12)	C(23)	C(24)	1.435(11)
C(24)	C(25)	1.367(10)	C(25)	C(26)	1.388(9)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(2)	H(1)	0.950	C(2)	H(2)	0.950
C(3)	H(3)	0.950	C(3)	H(4)	0.950
C(4)	H(6)	0.950	C(4)	H(7)	0.950
C(6)	H(8)	0.950	C(6)	H(10)	0.950
C(7)	H(12)	0.950	C(7)	H(13)	0.950
C(11)	H(14)	1.008	C(12)	H(21)	0.950
C(12)	H(22)	0.950	C(14)	H(15)	0.950
C(15)	H(16)	0.950	C(15)	H(17)	0.950
C(16)	H(18)	0.950	C(16)	H(20)	0.950
C(17)	H(19)	0.950	C(18)	H(23)	0.950
C(18)	H(24)	0.950	C(18)	H(25)	0.950
C(19)	H(11)	0.950	C(22)	H(5)	0.950
C(23)	H(27)	0.950	C(25)	H(26)	0.950
C(26)	H(9)	0.950			

Table 6. Bond angles (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
C(5)	O(2)	C(8)	104.2(5)	C(17)	O(3)	C(20)	117.5(5)
O(1)	C(1)	C(2)	118.9(5)	O(1)	C(1)	C(10)	120.9(6)
C(2)	C(1)	C(10)	120.1(5)	C(1)	C(2)	C(3)	114.4(5)
C(2)	C(3)	C(4)	110.1(6)	C(3)	C(4)	C(5)	107.1(7)
O(2)	C(5)	C(4)	107.6(6)	O(2)	C(5)	C(6)	101.0(5)
O(2)	C(5)	C(10)	108.8(5)	C(4)	C(5)	C(6)	114.3(6)
C(4)	C(5)	C(10)	113.0(5)	C(6)	C(5)	C(10)	111.3(7)
C(5)	C(6)	C(7)	102.9(6)	C(6)	C(7)	C(8)	104.7(6)
O(2)	C(8)	C(7)	102.8(5)	O(2)	C(8)	C(9)	106.7(5)
O(2)	C(8)	C(14)	106.9(6)	C(7)	C(8)	C(9)	112.1(7)
C(7)	C(8)	C(14)	117.8(6)	C(9)	C(8)	C(14)	109.6(5)
C(8)	C(9)	C(11)	122.9(5)	C(8)	C(9)	C(19)	113.5(5)
C(11)	C(9)	C(19)	123.3(6)	C(1)	C(10)	C(5)	119.8(5)
C(1)	C(10)	C(19)	122.6(5)	C(5)	C(10)	C(19)	117.5(5)
C(9)	C(11)	C(12)	125.1(6)	C(11)	C(12)	C(13)	111.3(6)
C(12)	C(13)	C(14)	109.4(5)	C(12)	C(13)	C(17)	115.1(6)
C(12)	C(13)	C(18)	111.6(6)	C(14)	C(13)	C(17)	95.3(6)
C(14)	C(13)	C(18)	113.7(7)	C(17)	C(13)	C(18)	110.8(6)
C(8)	C(14)	C(13)	113.7(6)	C(8)	C(14)	C(15)	119.7(6)
C(13)	C(14)	C(15)	104.0(5)	C(14)	C(15)	C(16)	104.2(6)
C(15)	C(16)	C(17)	105.5(6)	O(3)	C(17)	C(13)	114.4(6)
O(3)	C(17)	C(16)	109.4(6)	C(13)	C(17)	C(16)	107.0(6)
C(9)	C(19)	C(10)	123.2(5)	O(3)	C(20)	O(4)	126.3(7)
O(3)	C(20)	C(21)	110.7(6)	O(4)	C(20)	C(21)	123.0(7)
C(20)	C(21)	C(22)	116.3(6)	C(20)	C(21)	C(26)	124.1(6)
C(22)	C(21)	C(26)	119.6(6)	C(21)	C(22)	C(23)	118.5(7)
C(22)	C(23)	C(24)	119.6(7)	Br(1)	C(24)	C(23)	116.9(5)
Br(1)	C(24)	C(25)	120.6(5)	C(23)	C(24)	C(25)	122.5(7)
C(24)	C(25)	C(26)	119.0(7)	C(21)	C(26)	C(25)	120.8(6)

Table 7. Bond angles involving hydrogens (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	C(2)	H(1)	108.2	C(1)	C(2)	H(2)	108.2
C(3)	C(2)	H(1)	108.3	C(3)	C(2)	H(2)	108.2
H(1)	C(2)	H(2)	109.5	C(2)	C(3)	H(3)	109.3
C(2)	C(3)	H(4)	109.5	C(4)	C(3)	H(3)	109.0
C(4)	C(3)	H(4)	109.5	H(3)	C(3)	H(4)	109.5
C(3)	C(4)	H(6)	110.0	C(3)	C(4)	H(7)	110.2
C(5)	C(4)	H(6)	110.2	C(5)	C(4)	H(7)	109.9
H(6)	C(4)	H(7)	109.5	C(5)	C(6)	H(8)	111.1
C(5)	C(6)	H(10)	111.0	C(7)	C(6)	H(8)	110.8
C(7)	C(6)	H(10)	111.5	H(8)	C(6)	H(10)	109.5
C(6)	C(7)	H(12)	110.3	C(6)	C(7)	H(13)	110.9
C(8)	C(7)	H(12)	110.4	C(8)	C(7)	H(13)	110.9
H(12)	C(7)	H(13)	109.5	C(9)	C(11)	H(14)	119.7
C(12)	C(11)	H(14)	115.0	C(11)	C(12)	H(21)	109.3
C(11)	C(12)	H(22)	108.8	C(13)	C(12)	H(21)	108.9
C(13)	C(12)	H(22)	109.0	H(21)	C(12)	H(22)	109.5
C(8)	C(14)	H(15)	106.2	C(13)	C(14)	H(15)	106.1
C(15)	C(14)	H(15)	106.2	C(14)	C(15)	H(16)	110.6
C(14)	C(15)	H(17)	110.9	C(16)	C(15)	H(16)	110.6
C(16)	C(15)	H(17)	111.0	H(16)	C(15)	H(17)	109.5
C(15)	C(16)	H(18)	110.7	C(15)	C(16)	H(20)	110.3
C(17)	C(16)	H(18)	110.6	C(17)	C(16)	H(20)	110.2
H(18)	C(16)	H(20)	109.5	O(3)	C(17)	H(19)	108.6
C(13)	C(17)	H(19)	108.6	C(16)	C(17)	H(19)	108.7
C(13)	C(18)	H(23)	109.5	C(13)	C(18)	H(24)	109.5
C(13)	C(18)	H(25)	109.4	H(23)	C(18)	H(24)	109.5
H(23)	C(18)	H(25)	109.5	H(24)	C(18)	H(25)	109.5
C(9)	C(19)	H(11)	118.4	C(10)	C(19)	H(11)	118.4
C(21)	C(22)	H(5)	121.0	C(23)	C(22)	H(5)	120.5
C(22)	C(23)	H(27)	120.3	C(24)	C(23)	H(27)	120.1

C(24)	C(25)	H(26)	120.5	C(26)	C(25)	H(26)	120.5
C(21)	C(26)	H(9)	119.5	C(25)	C(26)	H(9)	119.7

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(5)	O(2)	C(8)	C(7)	44.3(6)	C(5)	O(2)	C(8)	C(9)	-73.9(7)
C(5)	O(2)	C(8)	C(14)	168.9(5)	C(8)	O(2)	C(5)	C(4)	-170.2(5)
C(8)	O(2)	C(5)	C(6)	-50.1(6)	C(8)	O(2)	C(5)	C(10)	67.1(7)
C(17)	O(3)	C(20)	O(4)	9.8(11)	C(17)	O(3)	C(20)	C(21)	-170.0(5)
C(20)	O(3)	C(17)	C(13)	-96.7(7)	C(20)	O(3)	C(17)	C(16)	143.2(6)
O(1)	C(1)	C(2)	C(3)	-161.4(8)	O(1)	C(1)	C(10)	C(5)	172.7(8)
O(1)	C(1)	C(10)	C(19)	-10.8(16)	C(2)	C(1)	C(10)	C(5)	-9.7(13)
C(2)	C(1)	C(10)	C(19)	166.8(9)	C(10)	C(1)	C(2)	C(3)	21.0(12)
C(1)	C(2)	C(3)	C(4)	-49.6(10)	C(2)	C(3)	C(4)	C(5)	66.0(9)
C(3)	C(4)	C(5)	O(2)	-174.3(6)	C(3)	C(4)	C(5)	C(6)	74.5(7)
C(3)	C(4)	C(5)	C(10)	-54.1(9)	O(2)	C(5)	C(6)	C(7)	35.3(7)
O(2)	C(5)	C(10)	C(1)	146.6(8)	O(2)	C(5)	C(10)	C(19)	-30.0(11)
C(4)	C(5)	C(6)	C(7)	150.4(6)	C(4)	C(5)	C(10)	C(1)	27.2(12)
C(4)	C(5)	C(10)	C(19)	-149.4(9)	C(6)	C(5)	C(10)	C(1)	-102.9(9)
C(6)	C(5)	C(10)	C(19)	80.4(10)	C(10)	C(5)	C(6)	C(7)	-80.1(7)
C(5)	C(6)	C(7)	C(8)	-9.1(7)	C(6)	C(7)	C(8)	O(2)	-20.0(7)
C(6)	C(7)	C(8)	C(9)	94.3(7)	C(6)	C(7)	C(8)	C(14)	-137.2(7)
O(2)	C(8)	C(9)	C(11)	-131.1(9)	O(2)	C(8)	C(9)	C(19)	42.8(10)
O(2)	C(8)	C(14)	C(13)	161.2(5)	O(2)	C(8)	C(14)	C(15)	-75.1(8)
C(7)	C(8)	C(9)	C(11)	117.1(9)	C(7)	C(8)	C(9)	C(19)	-69.0(9)
C(7)	C(8)	C(14)	C(13)	-83.8(8)	C(7)	C(8)	C(14)	C(15)	39.8(10)
C(9)	C(8)	C(14)	C(13)	45.9(9)	C(9)	C(8)	C(14)	C(15)	169.6(7)
C(14)	C(8)	C(9)	C(11)	-15.6(12)	C(14)	C(8)	C(9)	C(19)	158.3(8)
C(8)	C(9)	C(11)	C(12)	1.3(15)	C(8)	C(9)	C(19)	C(10)	-5.6(14)
C(11)	C(9)	C(19)	C(10)	168.3(10)	C(19)	C(9)	C(11)	C(12)	-172.0(9)
C(1)	C(10)	C(19)	C(9)	-177.6(9)	C(5)	C(10)	C(19)	C(9)	-1.1(15)
C(9)	C(11)	C(12)	C(13)	-16.3(12)	C(11)	C(12)	C(13)	C(14)	44.1(8)
C(11)	C(12)	C(13)	C(17)	149.9(7)	C(11)	C(12)	C(13)	C(18)	-82.6(8)
C(12)	C(13)	C(14)	C(8)	-62.3(8)	C(12)	C(13)	C(14)	C(15)	165.9(6)
C(12)	C(13)	C(17)	O(3)	80.9(8)	C(12)	C(13)	C(17)	C(16)	-157.7(6)

C(14)	C(13)	C(17)	O(3)	-164.7(6)	C(14)	C(13)	C(17)	C(16)	-43.4(7)
C(17)	C(13)	C(14)	C(8)	178.8(6)	C(17)	C(13)	C(14)	C(15)	46.9(7)
C(18)	C(13)	C(14)	C(8)	63.3(8)	C(18)	C(13)	C(14)	C(15)	-68.6(7)
C(18)	C(13)	C(17)	O(3)	-46.9(9)	C(18)	C(13)	C(17)	C(16)	74.5(8)
C(8)	C(14)	C(15)	C(16)	-163.6(7)	C(13)	C(14)	C(15)	C(16)	-35.3(7)
C(14)	C(15)	C(16)	C(17)	7.0(8)	C(15)	C(16)	C(17)	O(3)	148.3(6)
C(15)	C(16)	C(17)	C(13)	23.8(8)	O(3)	C(20)	C(21)	C(22)	-177.5(6)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(3)	C(20)	C(21)	C(26)	3.2(10)	O(4)	C(20)	C(21)	C(22)	2.7(11)
O(4)	C(20)	C(21)	C(26)	-176.6(7)	C(20)	C(21)	C(22)	C(23)	-178.8(7)
C(20)	C(21)	C(26)	C(25)	-179.7(6)	C(22)	C(21)	C(26)	C(25)	1.0(12)
C(26)	C(21)	C(22)	C(23)	0.5(11)	C(21)	C(22)	C(23)	C(24)	-1.4(11)
C(22)	C(23)	C(24)	Br(1)	-177.3(6)	C(22)	C(23)	C(24)	C(25)	0.9(12)
Br(1)	C(24)	C(25)	C(26)	178.8(6)	C(23)	C(24)	C(25)	C(26)	0.7(11)
C(24)	C(25)	C(26)	C(21)	-1.6(12)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Br(1)	Br(1)[1)]	3.4971(11)	O(1)	C(14)[2)]	3.373(10)
O(1)	C(25)[3)]	3.230(8)	O(1)	C(26)[3)]	3.211(8)
O(4)	C(22)[4)]	3.561(10)	O(4)	C(23)[4)]	3.517(10)
C(7)	C(25)[5)]	3.530(12)	C(14)	O(1)[6)]	3.373(10)
C(22)	O(4)[4)]	3.561(10)	C(23)	O(4)[4)]	3.517(10)
C(25)	O(1)[7)]	3.230(8)	C(25)	C(7)[8)]	3.530(12)
C(26)	O(1)[7)]	3.211(8)			

Symmetry Operators:

(1) -X+2,Y,-Z-1
(2) -X+1/2+1,Y+1/2-1,-Z+1
(3) X,Y,Z+1
(4) -X+2,Y,-Z
(5) -X+1/2+1,Y+1/2-1,-Z
(6) -X+1/2+1,Y+1/2,-Z+1
(7) X,Y,Z-1
(8) -X+1/2+1,Y+1/2,-Z

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Br(1)	H(6)[1)]	3.532	Br(1)	H(7)[1)]	3.504
Br(1)	H(8)[2)]	3.400	Br(1)	H(10)[2)]	3.532
Br(1)	H(14)[3)]	3.174	Br(1)	H(22)[3)]	3.548
O(1)	H(9)[4)]	2.726	O(1)	H(12)[5)]	2.801
O(1)	H(15)[6)]	2.446	O(1)	H(21)[6)]	3.095
O(1)	H(24)[5)]	3.084	O(1)	H(26)[4)]	2.785
O(1)	H(19)[6)]	3.365	O(2)	H(11)[5)]	3.378
O(2)	H(26)[2)]	2.721	O(3)	H(18)[1)]	3.065
O(3)	H(25)[2)]	2.935	O(4)	H(2)[5)]	3.468
O(4)	H(5)[3)]	3.026	O(4)	H(17)[2)]	3.441
O(4)	H(20)[2)]	3.050	O(4)	H(27)[3)]	2.949
C(1)	H(12)[5)]	3.068	C(1)	H(15)[6)]	3.349
C(1)	H(21)[6)]	3.215	C(1)	H(24)[5)]	3.430
C(2)	H(21)[6)]	3.124	C(2)	H(23)[5)]	3.366
C(2)	H(24)[5)]	3.552	C(3)	H(7)[7)]	2.995
C(4)	H(3)[7)]	3.283	C(4)	H(7)[7)]	3.173
C(6)	H(11)[6)]	3.533	C(7)	H(11)[6)]	3.532
C(10)	H(12)[5)]	3.328	C(11)	H(1)[6)]	3.471
C(11)	H(6)[6)]	3.474	C(11)	H(10)[5)]	3.370
C(12)	H(2)[5)]	3.313	C(12)	H(27)[3)]	3.387
C(14)	H(9)[2)]	3.404	C(14)	H(26)[2)]	3.306
C(15)	H(9)[2)]	3.209	C(15)	H(26)[2)]	3.406
C(16)	H(9)[2)]	3.130	C(16)	H(23)[2)]	3.519
C(16)	H(25)[2)]	3.067	C(17)	H(9)[2)]	3.442
C(17)	H(25)[2)]	3.212	C(18)	H(1)[6)]	3.160
C(18)	H(18)[1)]	3.115	C(19)	H(10)[5)]	3.264
C(19)	H(12)[5)]	3.185	C(20)	H(17)[2)]	3.320
C(20)	H(20)[2)]	3.353	C(20)	H(25)[2)]	3.178
C(21)	H(13)[2)]	3.111	C(21)	H(17)[2)]	3.158
C(21)	H(25)[2)]	3.595	C(22)	H(5)[3)]	2.973

C(22)	H(13)[2)]	3.227	C(22)	H(17)[2)]	3.201
C(23)	H(8)[2)]	3.342	C(23)	H(13)[2)]	3.212
C(23)	H(22)[3)]	3.492	C(24)	H(8)[2)]	3.173
C(24)	H(10)[2)]	3.586	C(24)	H(13)[2)]	3.115
C(25)	H(11)[8)]	3.518	C(25)	H(12)[2)]	3.340
C(25)	H(13)[2)]	3.045	C(25)	H(15)[1)]	3.299
C(25)	H(16)[1)]	2.942	C(26)	H(12)[2)]	3.513

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(26)	H(13)[2)]	3.013	C(26)	H(15)[1)]	3.307
C(26)	H(16)[1)]	2.958	C(26)	H(18)[1)]	3.143
C(26)	H(24)[2)]	3.432	H(1)	C(11)[5)]	3.471
H(1)	C(18)[5)]	3.160	H(1)	H(14)[5)]	3.451
H(1)	H(22)[5)]	3.223	H(1)	H(23)[5)]	2.568
H(1)	H(24)[5)]	2.918	H(2)	O(4)[6)]	3.468
H(2)	C(12)[6)]	3.313	H(2)	H(21)[6)]	2.392
H(2)	H(23)[5)]	3.472	H(2)	H(27)[9)]	3.348
H(2)	H(19)[6)]	3.487	H(3)	C(4)[7)]	3.283
H(3)	H(7)[7)]	2.348	H(3)	H(8)[7)]	3.131
H(4)	H(7)[7)]	3.182	H(4)	H(8)[7)]	3.353
H(4)	H(14)[6)]	3.438	H(4)	H(21)[6)]	3.462
H(4)	H(27)[9)]	3.542	H(5)	O(4)[3)]	3.026
H(5)	C(22)[3)]	2.973	H(5)	H(5)[3)]	2.162
H(5)	H(17)[2)]	3.293	H(6)	Br(1)[2)]	3.532
H(6)	C(11)[5)]	3.474	H(6)	H(7)[7)]	3.503
H(6)	H(11)[5)]	3.206	H(6)	H(14)[5)]	2.863
H(6)	H(26)[2)]	3.597	H(7)	Br(1)[2)]	3.504
H(7)	C(3)[7)]	2.995	H(7)	C(4)[7)]	3.173
H(7)	H(3)[7)]	2.348	H(7)	H(4)[7)]	3.182
H(7)	H(6)[7)]	3.503	H(7)	H(7)[7)]	2.629
H(8)	Br(1)[1)]	3.400	H(8)	C(23)[1)]	3.342
H(8)	C(24)[1)]	3.173	H(8)	H(3)[7)]	3.131
H(8)	H(4)[7)]	3.353	H(8)	H(27)[1)]	3.402
H(9)	O(1)[8)]	2.726	H(9)	C(14)[1)]	3.404
H(9)	C(15)[1)]	3.209	H(9)	C(16)[1)]	3.130
H(9)	C(17)[1)]	3.442	H(9)	H(13)[2)]	3.482
H(9)	H(15)[1)]	2.704	H(9)	H(16)[1)]	2.757
H(9)	H(18)[1)]	2.556	H(9)	H(24)[2)]	3.260

H(9)	H(25)[2)]	3.567	H(9)	H(19)[1)]	2.859
H(10)	Br(1)[1)]	3.532	H(10)	C(11)[6)]	3.370
H(10)	C(19)[6)]	3.264	H(10)	C(24)[1)]	3.586
H(10)	H(11)[6)]	2.800	H(10)	H(14)[6)]	2.892
H(11)	O(2)[6)]	3.378	H(11)	C(6)[5)]	3.533
H(11)	C(7)[5)]	3.532	H(11)	C(25)[4)]	3.518
H(11)	H(6)[6)]	3.206	H(11)	H(10)[5)]	2.800
H(11)	H(12)[5)]	2.809	H(11)	H(26)[4)]	2.869

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(12)	O(1)[6)]	2.801	H(12)	C(1)[6)]	3.068
H(12)	C(10)[6)]	3.328	H(12)	C(19)[6)]	3.185
H(12)	C(25)[1)]	3.340	H(12)	C(26)[1)]	3.513
H(12)	H(11)[6)]	2.809	H(12)	H(26)[1)]	3.427
H(13)	C(21)[1)]	3.111	H(13)	C(22)[1)]	3.227
H(13)	C(23)[1)]	3.212	H(13)	C(24)[1)]	3.115
H(13)	C(25)[1)]	3.045	H(13)	C(26)[1)]	3.013
H(13)	H(9)[1)]	3.482	H(13)	H(26)[1)]	3.547
H(14)	Br(1)[3)]	3.174	H(14)	H(1)[6)]	3.451
H(14)	H(4)[5)]	3.438	H(14)	H(6)[6)]	2.863
H(14)	H(10)[5)]	2.892	H(15)	O(1)[5)]	2.446
H(15)	C(1)[5)]	3.349	H(15)	C(25)[2)]	3.299
H(15)	C(26)[2)]	3.307	H(15)	H(9)[2)]	2.704
H(15)	H(26)[2)]	2.694	H(16)	C(25)[2)]	2.942
H(16)	C(26)[2)]	2.958	H(16)	H(9)[2)]	2.757
H(16)	H(26)[2)]	2.718	H(17)	O(4)[1)]	3.441
H(17)	C(20)[1)]	3.320	H(17)	C(21)[1)]	3.158
H(17)	C(22)[1)]	3.201	H(17)	H(5)[1)]	3.293
H(18)	O(3)[2)]	3.065	H(18)	C(18)[2)]	3.115
H(18)	C(26)[2)]	3.143	H(18)	H(9)[2)]	2.556
H(18)	H(23)[2)]	2.869	H(18)	H(25)[2)]	2.508
H(20)	O(4)[1)]	3.050	H(20)	C(20)[1)]	3.353
H(20)	H(23)[2)]	3.365	H(20)	H(25)[2)]	3.163
H(20)	H(19)[1)]	3.073	H(21)	O(1)[5)]	3.095
H(21)	C(1)[5)]	3.215	H(21)	C(2)[5)]	3.124
H(21)	H(2)[5)]	2.392	H(21)	H(4)[5)]	3.462
H(21)	H(27)[3)]	3.014	H(22)	Br(1)[3)]	3.548
H(22)	C(23)[3)]	3.492	H(22)	H(1)[6)]	3.223
H(22)	H(27)[3)]	2.895	H(23)	C(2)[6)]	3.366

H(23)	C(16)[1)]	3.519	H(23)	H(1)[6)]	2.568
H(23)	H(2)[6)]	3.472	H(23)	H(18)[1)]	2.869
H(23)	H(20)[1)]	3.365	H(24)	O(1)[6)]	3.084
H(24)	C(1)[6)]	3.430	H(24)	C(2)[6)]	3.552
H(24)	C(26)[1)]	3.432	H(24)	H(1)[6)]	2.918
H(24)	H(9)[1)]	3.260	H(25)	O(3)[1)]	2.935
H(25)	C(16)[1)]	3.067	H(25)	C(17)[1)]	3.212
H(25)	C(20)[1)]	3.178	H(25)	C(21)[1)]	3.595

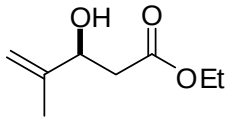
Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(25)	H(9)[1)]	3.567	H(25)	H(18)[1)]	2.508
H(25)	H(20)[1)]	3.163	H(25)	H(19)[1)]	3.072
H(26)	O(1)[8)]	2.785	H(26)	O(2)[1)]	2.721
H(26)	C(14)[1)]	3.306	H(26)	C(15)[1)]	3.406
H(26)	H(6)[1)]	3.597	H(26)	H(11)[8)]	2.869
H(26)	H(12)[2)]	3.427	H(26)	H(13)[2)]	3.547
H(26)	H(15)[1)]	2.694	H(26)	H(16)[1)]	2.718
H(27)	O(4)[3)]	2.949	H(27)	C(12)[3)]	3.387
H(27)	H(2)[10)]	3.348	H(27)	H(4)[10)]	3.542
H(27)	H(8)[2)]	3.402	H(27)	H(21)[3)]	3.014
H(27)	H(22)[3)]	2.895	H(19)	O(1)[5)]	3.365
H(19)	H(2)[5)]	3.487	H(19)	H(9)[2)]	2.859
H(19)	H(20)[2)]	3.073	H(19)	H(25)[2)]	3.072

Symmetry Operators:

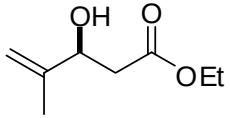
(1) -X+1/2+1,Y+1/2-1,-Z
(2) -X+1/2+1,Y+1/2,-Z
(3) -X+2,Y,-Z
(4) X,Y,Z+1
(5) -X+1/2+1,Y+1/2,-Z+1
(6) -X+1/2+1,Y+1/2-1,-Z+1
(7) -X+1,Y,-Z+1
(8) X,Y,Z-1
(9) X+1/2-1,Y+1/2-1,Z+1
(10) X+1/2,Y+1/2,Z-1

^{1}H-NMR
400 MHz
$CDCl_3$

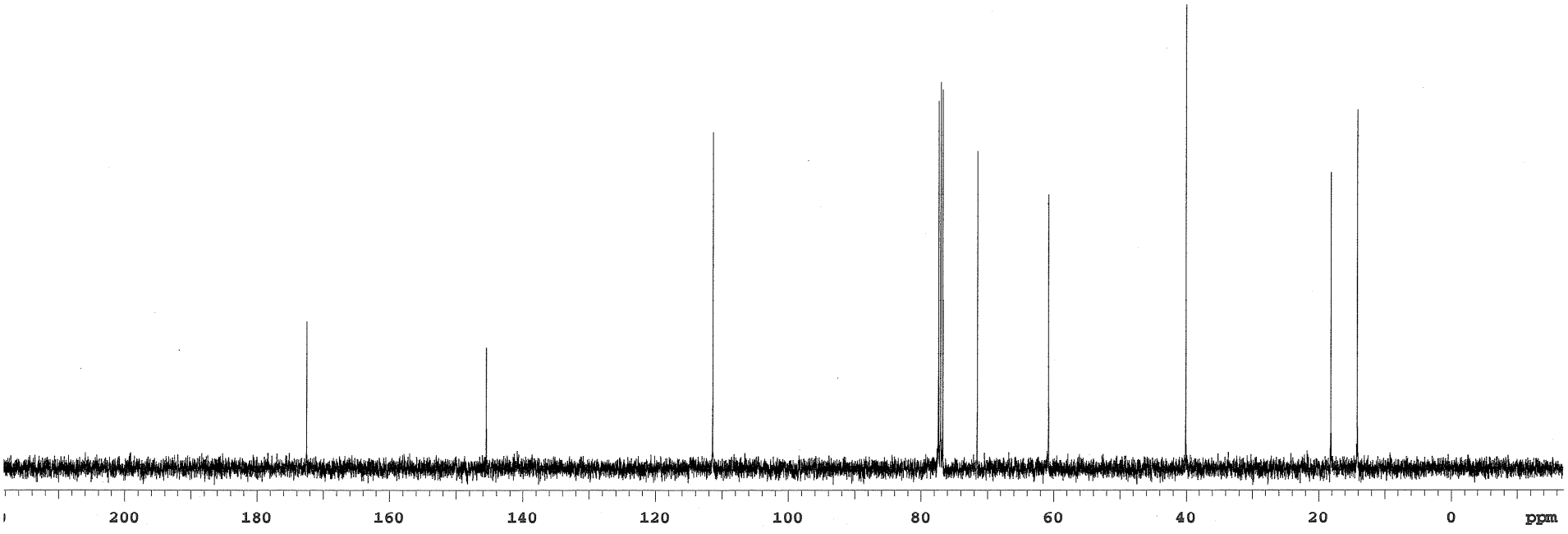


(-)-17

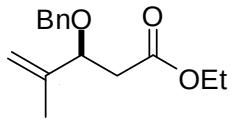
^{13}C-NMR
100 MHz
$CDCl_3$



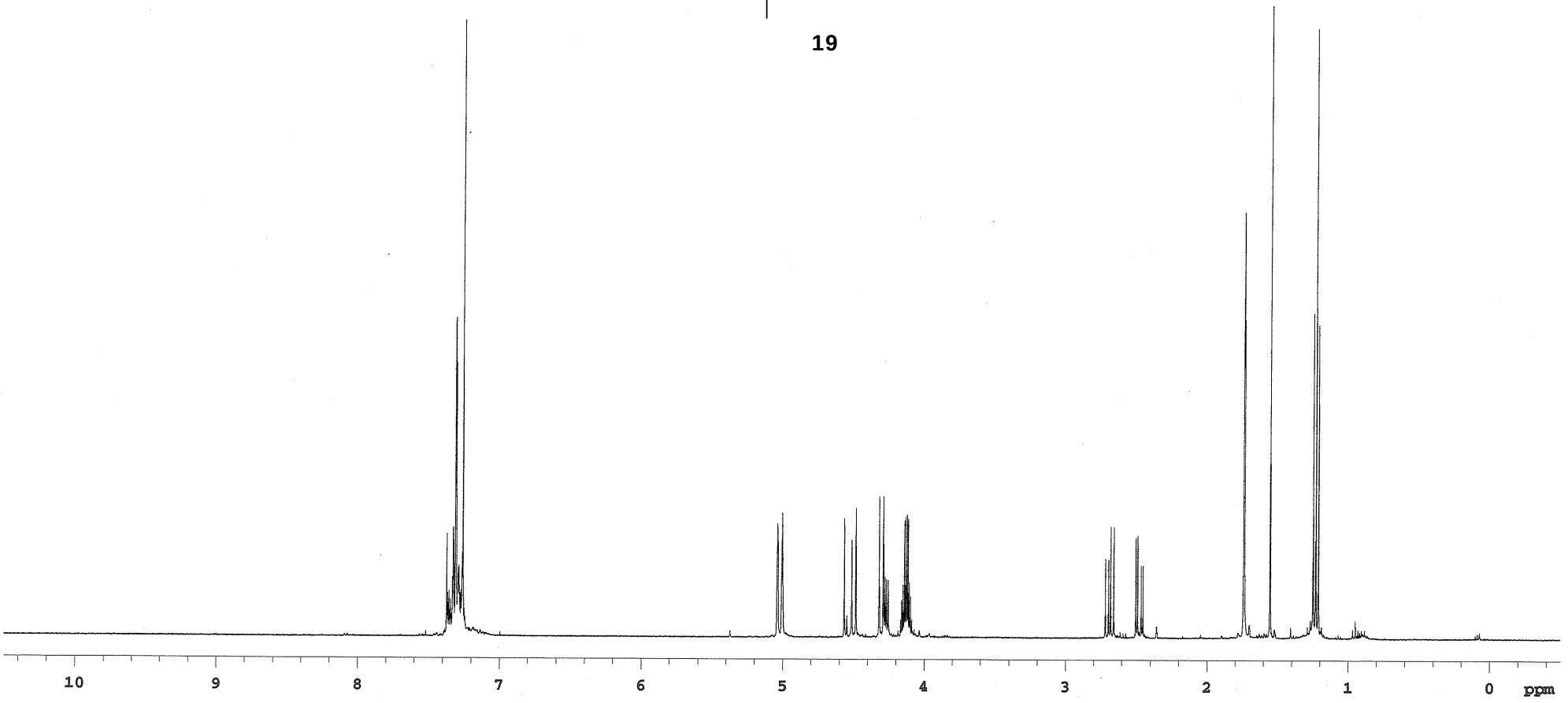
(-)-17



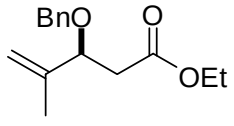
^{1}H-NMR
400 MHz
$CDCl_3$



19

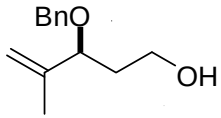


^{13}C-NMR
100 MHz
$CDCl_3$



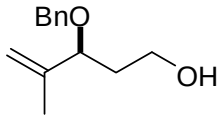
19

^{1}H-NMR
400 MHz
$CDCl_3$

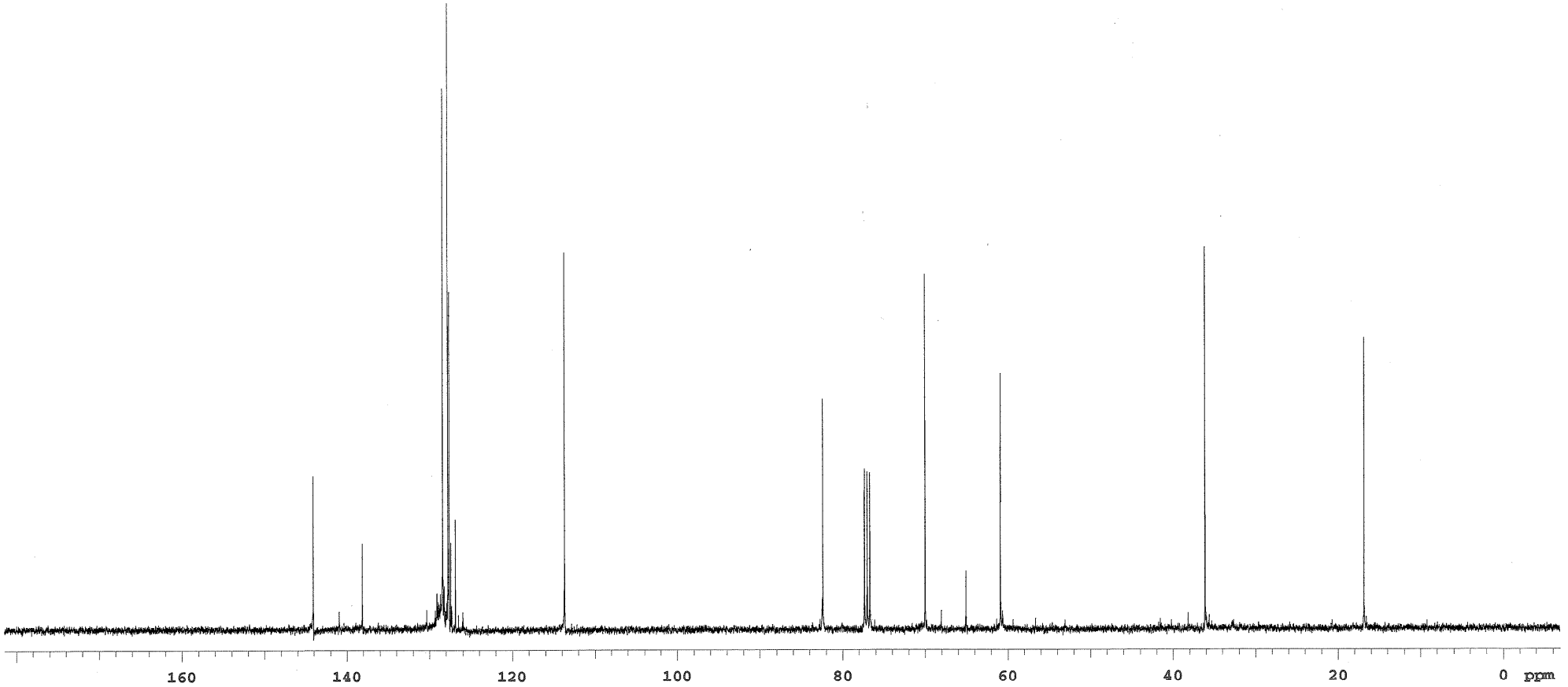


20

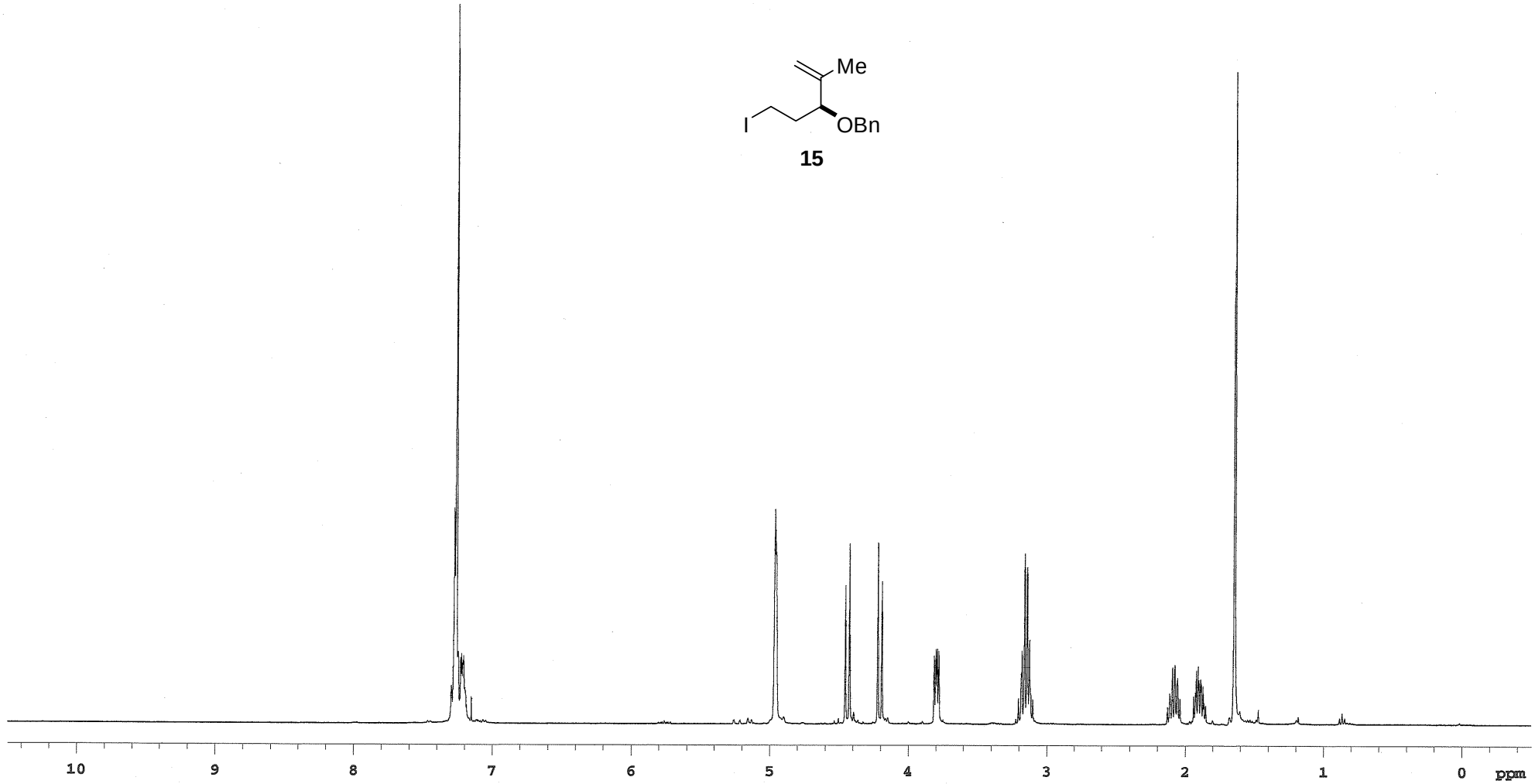
^{13}C-NMR
100 MHz
$CDCl_3$



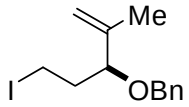
20



^{1}H-NMR
400 MHz
$CDCl_3$



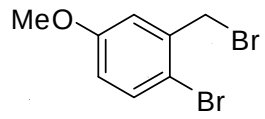
^{13}C-NMR
100 MHz
$CDCl_3$



15

40

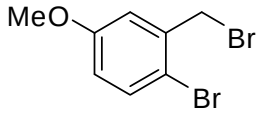
^{1}H-NMR
500 MHz
$CDCl_3$



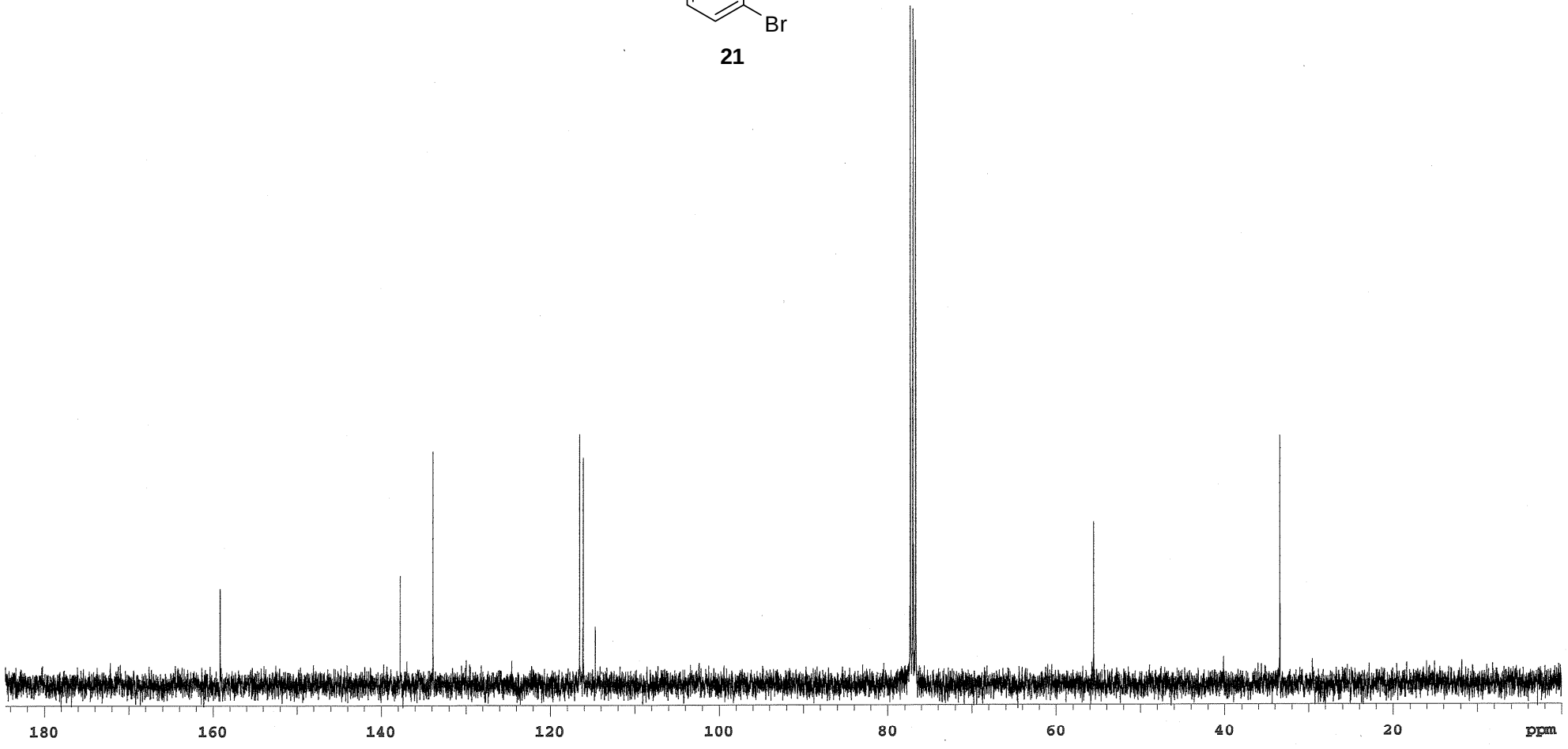
21

41

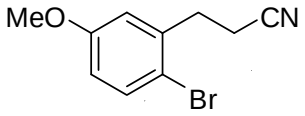
^{13}C-NMR
100 MHz
$CDCl_3$



21

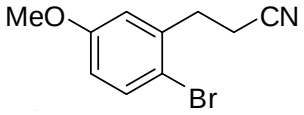


^{1}H-NMR
500 MHz
$CDCl_3$

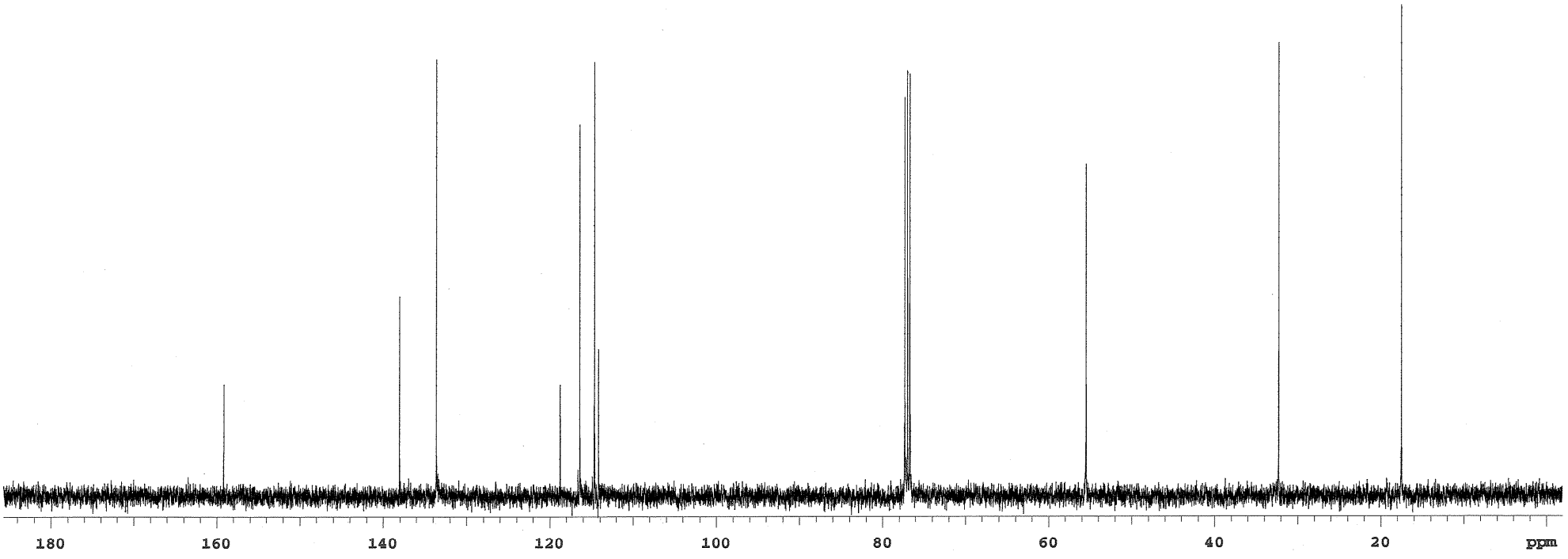


22

^{13}C-NMR
100 MHz
$CDCl_3$

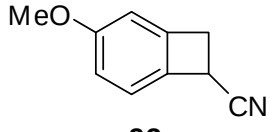


22



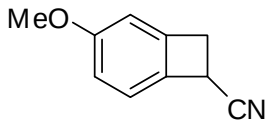
44

^{1}H-NMR
500 MHz
$CDCl_3$



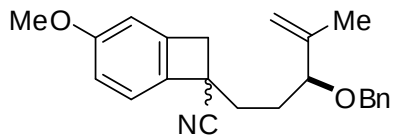
23

^{13}C-NMR
100 MHz
$CDCl_3$



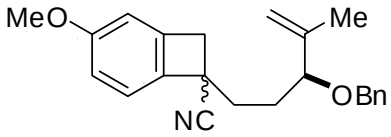
23

^{1}H-NMR
400 MHz
$CDCl_3$

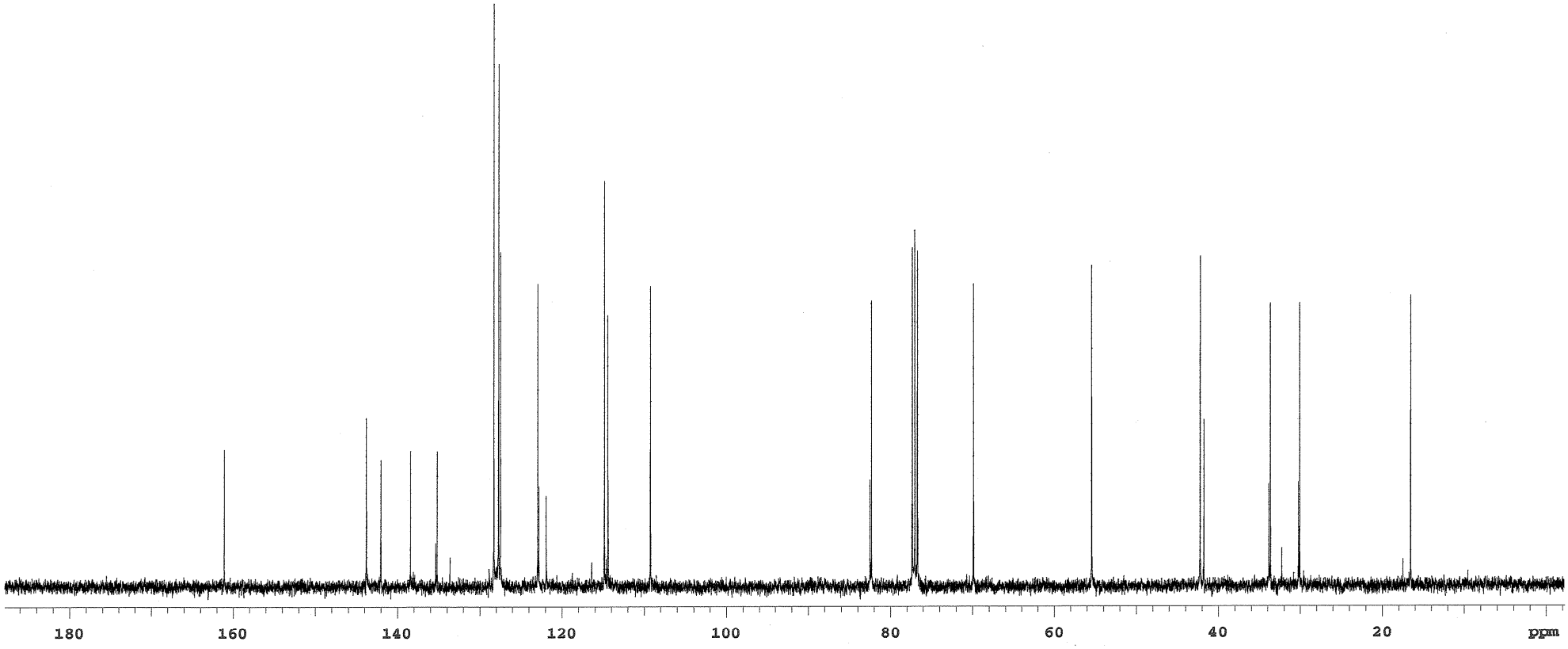


24 (dr = 2:1)

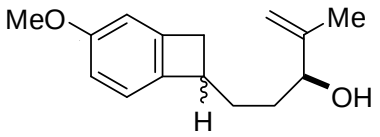
^{13}C-NMR
100 MHz
$CDCl_3$



24 (dr = 2:1)

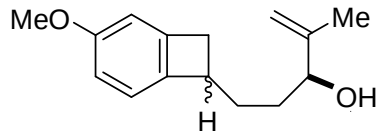


^{1}H-NMR
500 MHz
$CDCl_3$



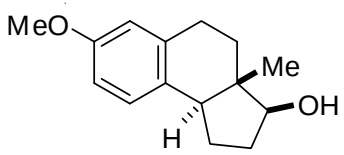
25 (dr = 1:1)

^{13}C-NMR
100 MHz
$CDCl_3$



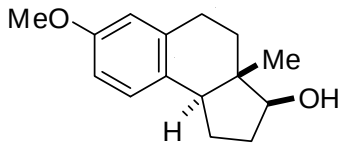
25 (dr = 1:1)

^{1}H-NMR
500 MHz
$CDCl_3$

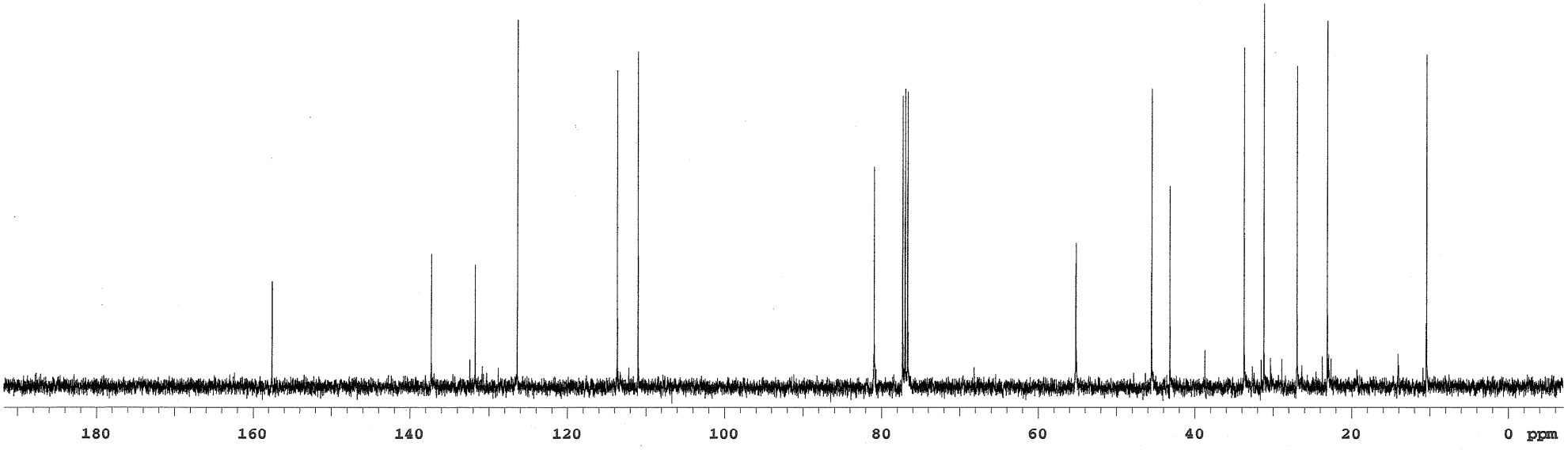


27

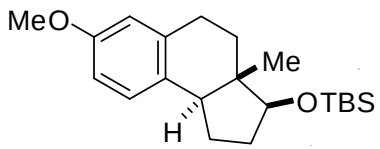
^{13}C-NMR
100 MHz
$CDCl_3$



27

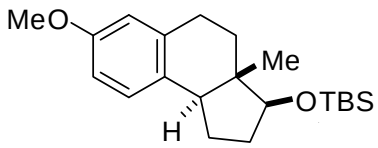


^{1}H-NMR
500 MHz
$CDCl_3$



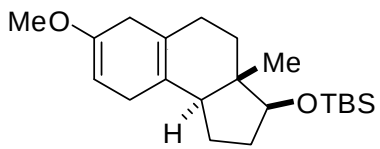
28

^{13}C-NMR
100 MHz
$CDCl_3$



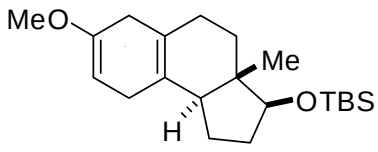
28

^{1}H-NMR
500 MHz
$CDCl_3$



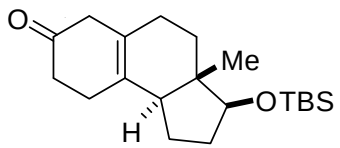
29

^{13}C-NMR
100 MHz
$CDCl_3$



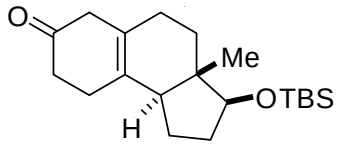
29

^{1}H-NMR
400 MHz
$CDCl_3$



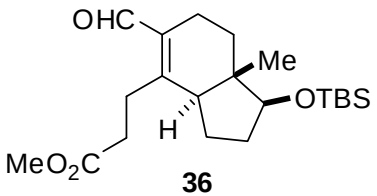
33

^{13}C-NMR
100 MHz
$CDCl_3$



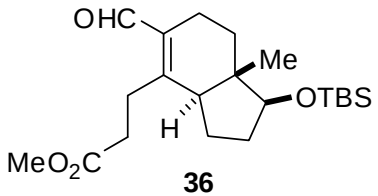
33

^{1}H-NMR
400 MHz
$CDCl_3$



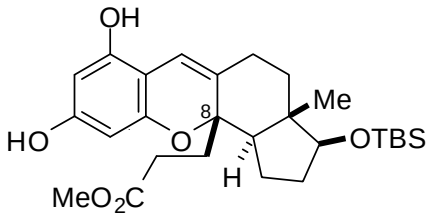
36

^{13}C-NMR
100 MHz
$CDCl_3$



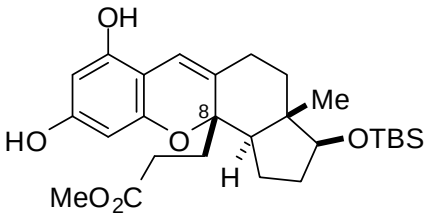
36

^{1}H-NMR
400 MHz
$CDCl_3$



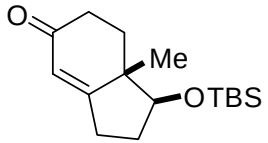
37 (dr = 3.3:1)

^{13}C-NMR
100 MHz
$CDCl_3$



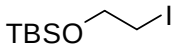
37 (dr = 3.3:1)

^{1}H-NMR
500 MHz
$CDCl_3$



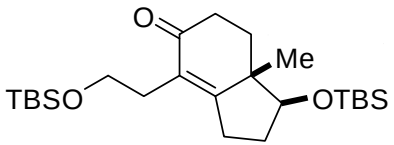
50

^{1}H-NMR
500 MHz
$CDCl_3$



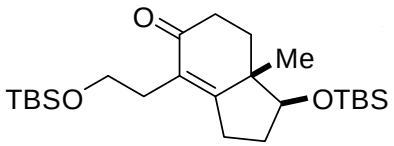
47

^{1}H-NMR
500 MHz
$CDCl_3$



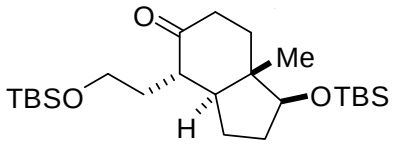
51

^{13}C-NMR
125 MHz
$CDCl_3$



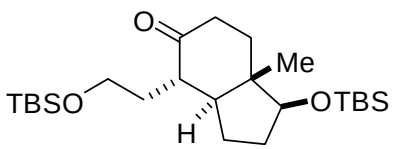
51

^{1}H-NMR
500 MHz
$CDCl_3$



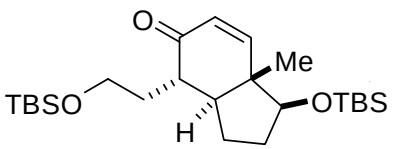
52

^{13}C-NMR
125 MHz
$CDCl_3$



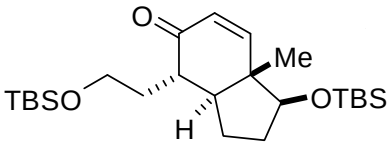
52

^{1}H-NMR
500 MHz
C_6D_6



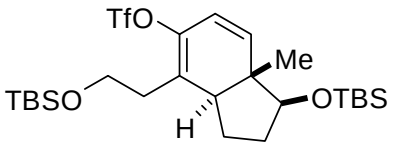
57

^{13}C-NMR
125 MHz
C_6D_6



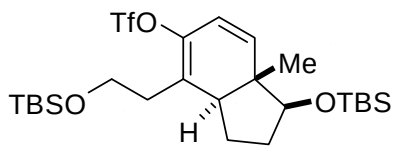
57

^{1}H-NMR
500 MHz
$CDCl_3$

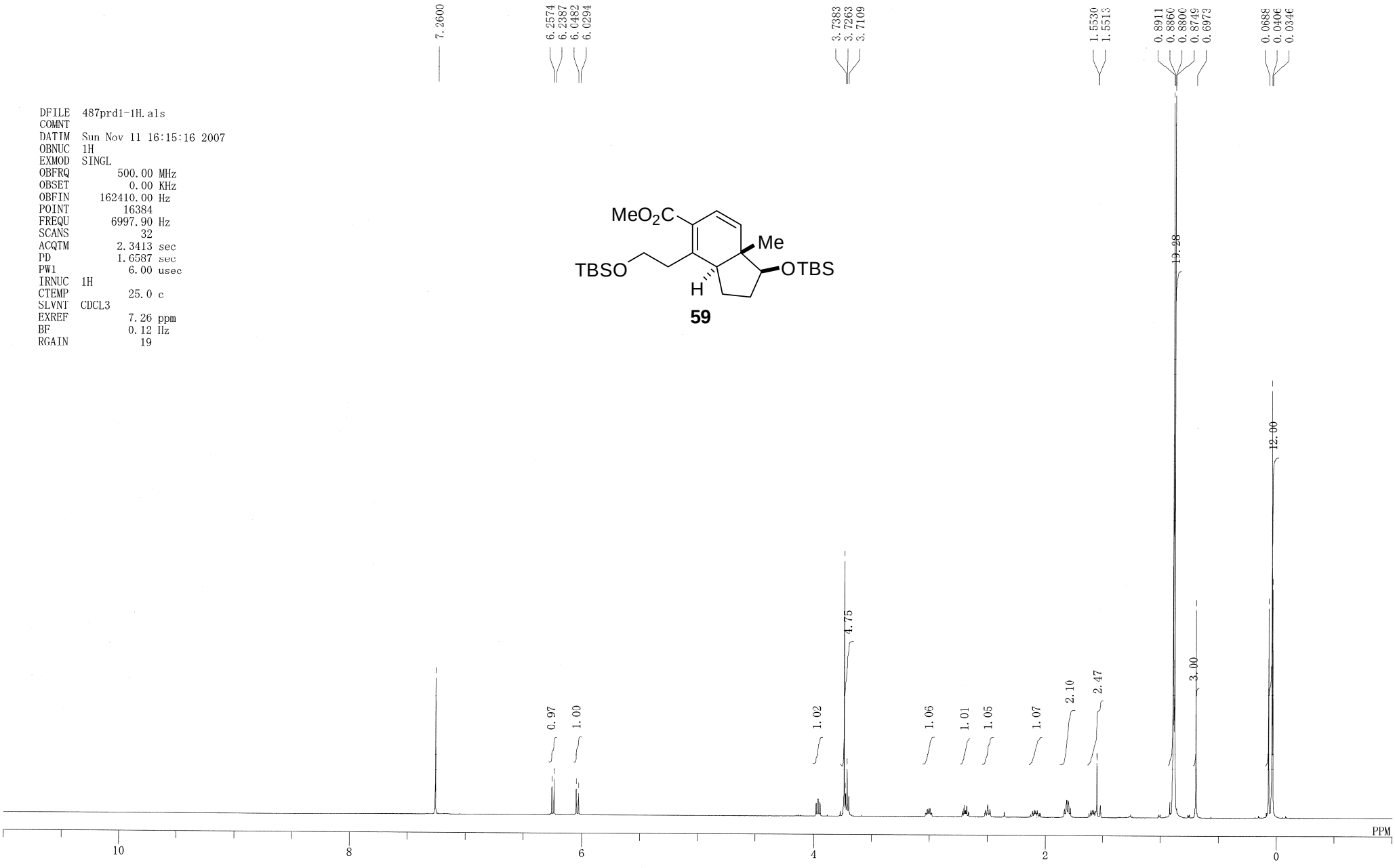


58

^{13}C-NMR
125 MHz
$CDCl_3$



58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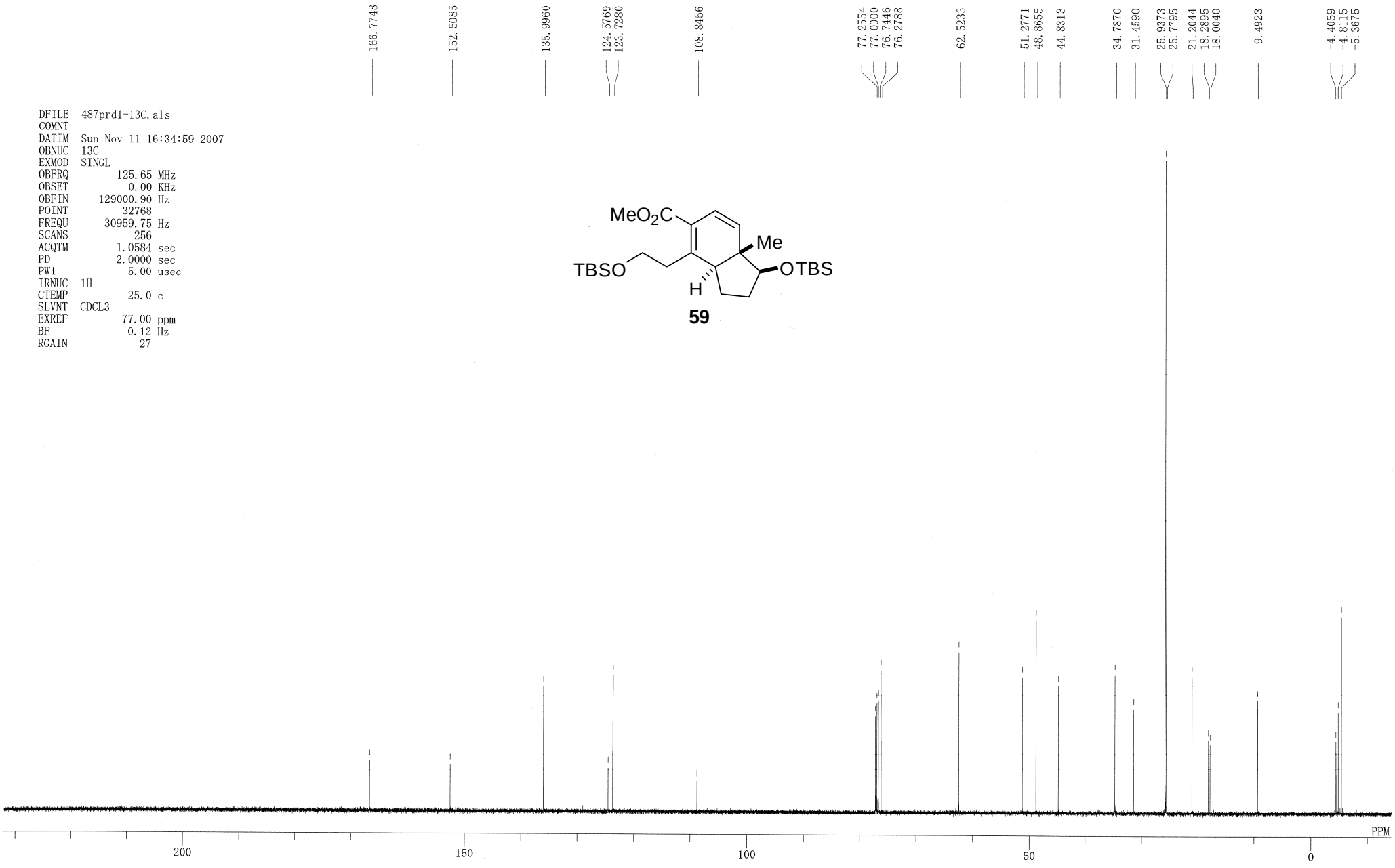
6.2574
6.2387
6.0482
6.0294
3.7383
3.7263
3.7109
1.5530
1.5513
0.8911
0.8860
0.8800
0.8749
0.6973
0.0688
0.0406
0.0346
DFILE 487prd1-1H.als
COMNT
DATIM Sun Nov 11 16:15:16 2007
OBNUC 1H
EXMOD SINGL
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162410.00 Hz
POINT 16384
FREQU 6997.90 Hz
SCANS 32
ACQTM 2.3413 sec
PD 1.6587 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 19
MeO2C
Me
TBSO
OTBS
H
59
19.28
12.00
4.75
3.00
2.47
2.10
1.07
1.06
1.02
1.01
1.05
1.00
0.97
PPM
10
8
6
4
2
0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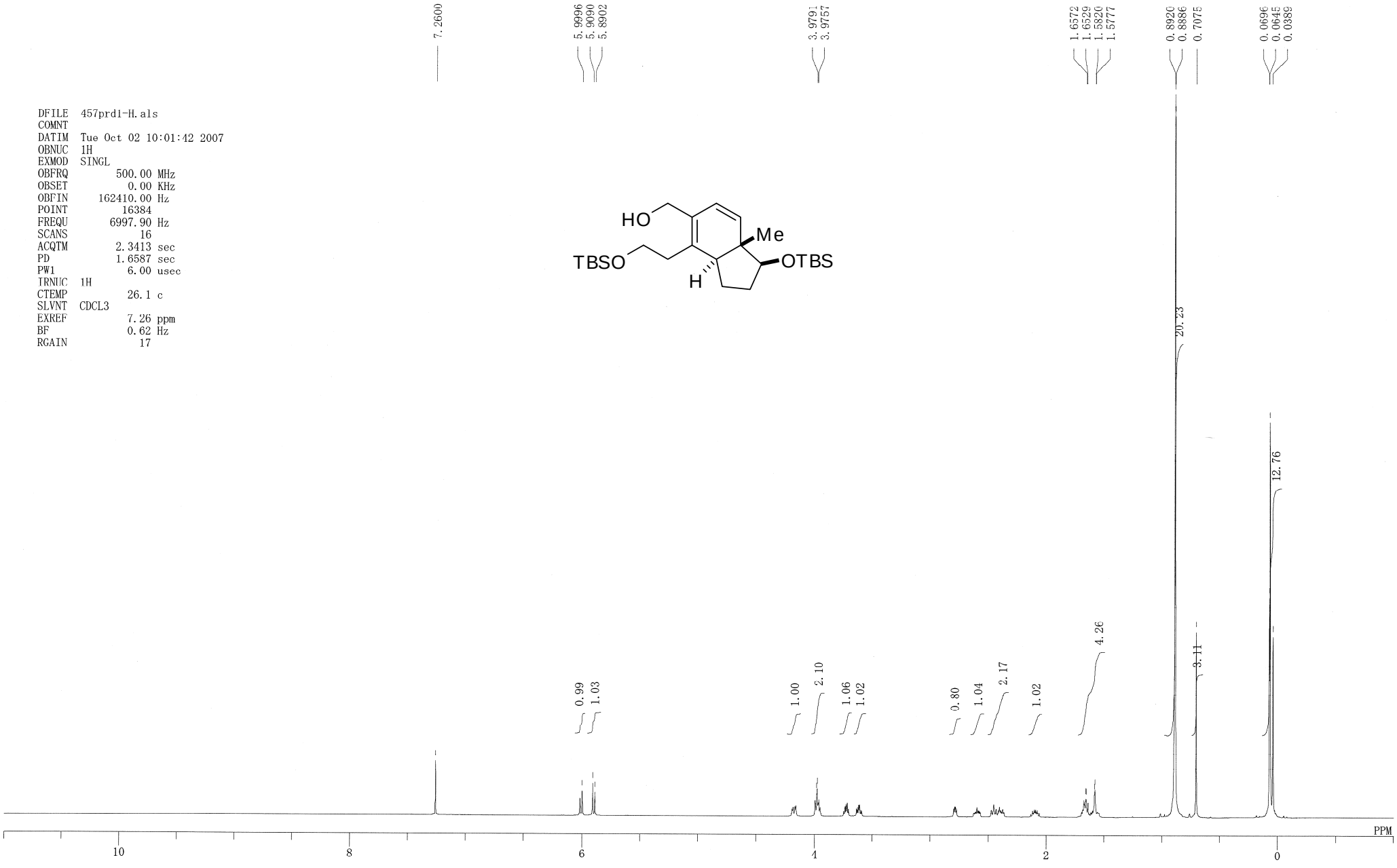
152.5085
135.9960
124.5769
123.7280
108.8456
77.2554
77.0000
76.7446
76.2788
62.5233
51.2771
48.8655
44.8313
34.7870
31.4590
25.9373
25.7795
21.2044
18.2895
18.0040
9.4923
-4.4059
-4.8115
-5.3675
DFILE 487prd1-13C.als
COMNT
DATIM Sun Nov 11 16:34:59 2007
OBNUC 13C
EXMOD SINGL
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 129000.90 Hz
POINT 32768
FREQU 30959.75 Hz
SCANS 256
ACQTM 1.0584 sec
PD 2.0000 sec
PW1 5.00 usec
TRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27
MeO2C
Me
TBSO
OTBS
H
59
PPM
200
150
100
50
0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COMNT
DATIM Tue Oct 02 10:01:42 2007
OBNUC 1H
EXMOD SINGL
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162410.00 Hz
POINT 16384
FREQU 6997.90 Hz
SCANS 16
ACQTM 2.3413 sec
PD 1.6587 sec
PW1 6.00 usec
TRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.62 Hz
RGAIN 17
HO
Me
TBSO
OTBS
H
7.2600
5.9996
5.9090
5.8902
3.9791
3.9757
1.6572
1.6529
1.5820
1.5777
0.8920
0.8886
0.7075
0.0696
0.0645
0.0389
0.99
1.03
1.00
2.10
1.06
1.02
0.80
1.04
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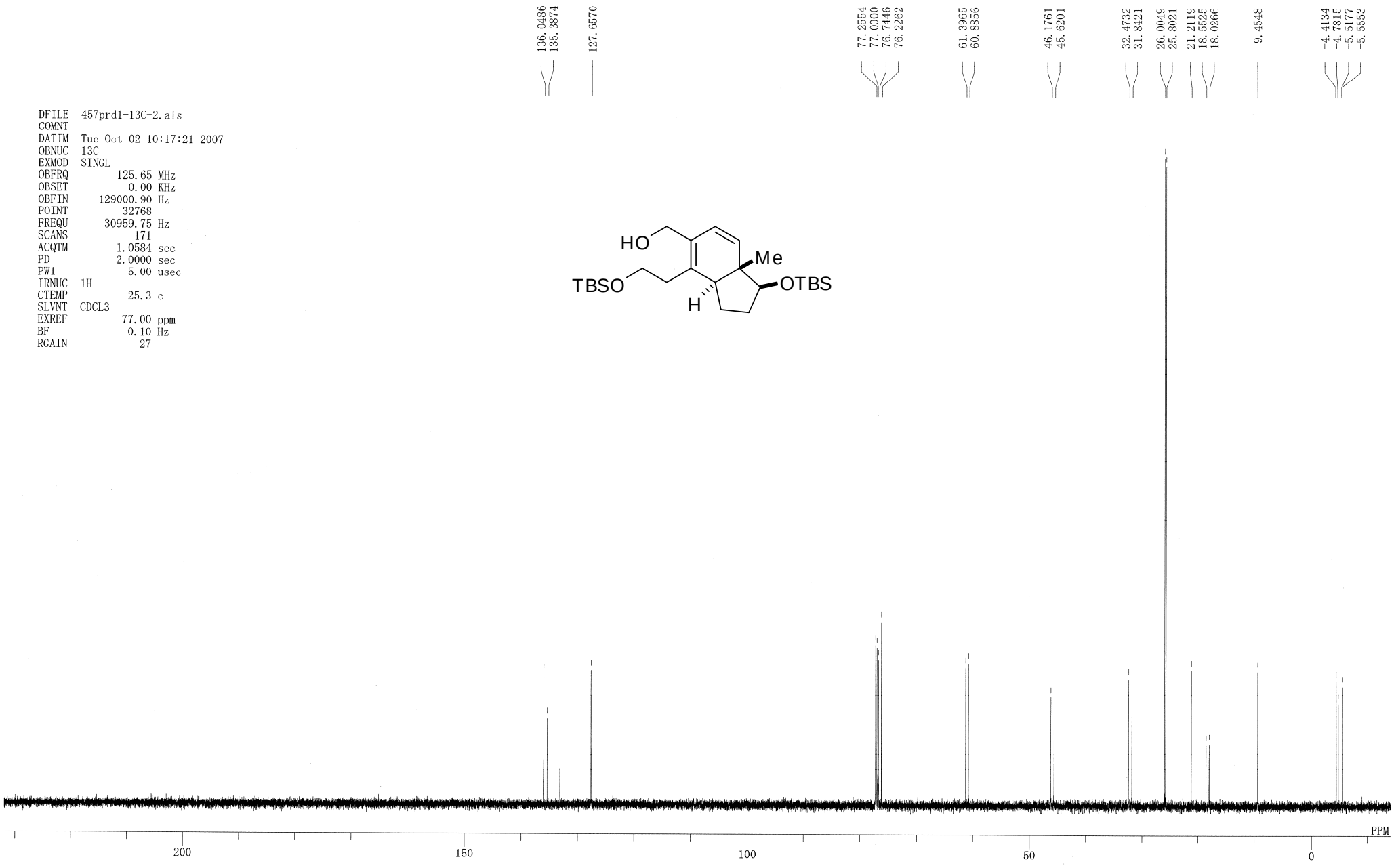
1.02
4.26
20.23
3.11
12.76
PPM
10
8
6
4
2
0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127.6570
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77.0000
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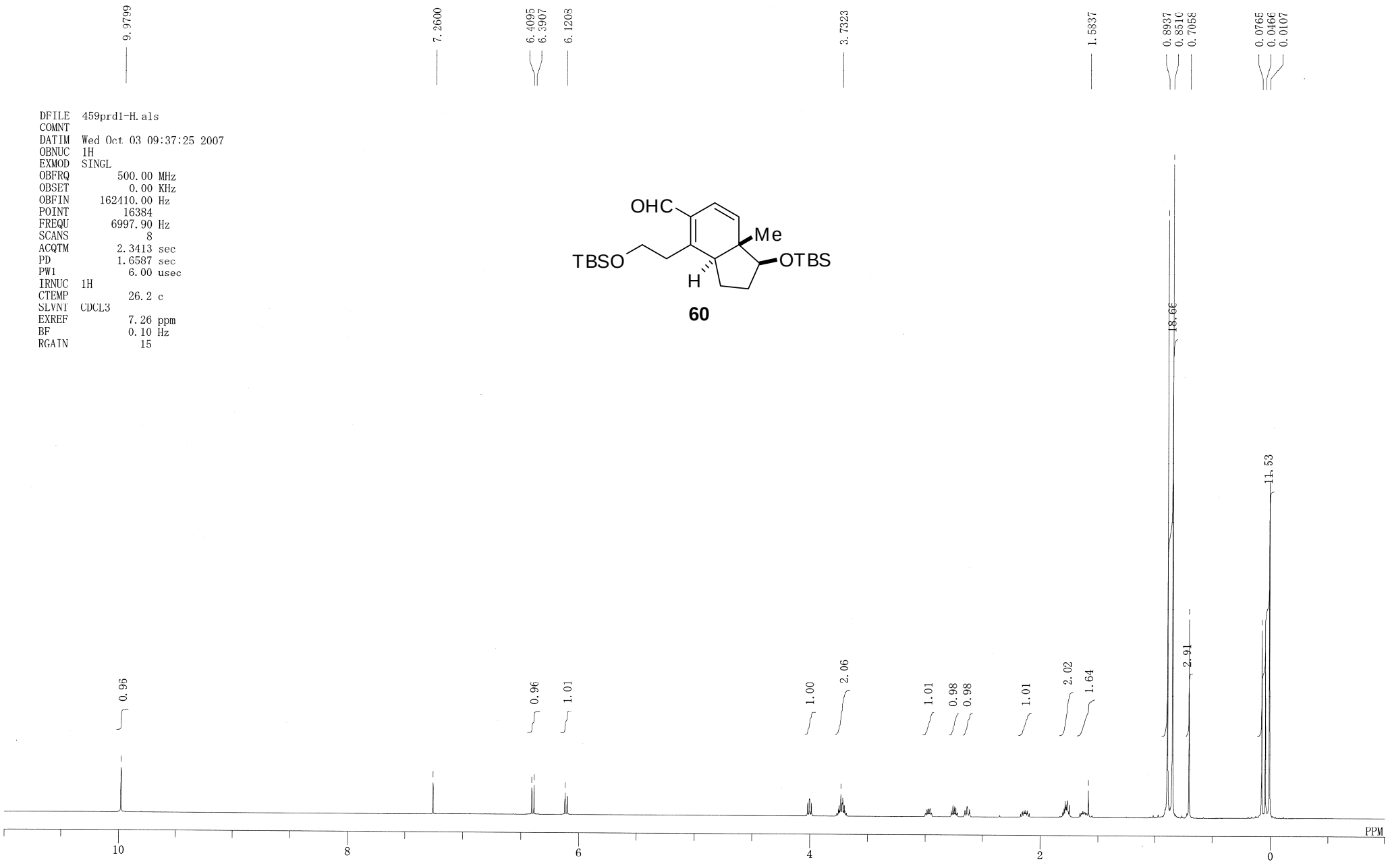
46.1761
45.6201
32.4732
31.8421
26.0049
25.8021
21.2119
18.5525
18.0266
9.4548
-4.4134
-4.7815
-5.5177
-5.5553
DFILE 457prd1-13C-2.als
COMNT
DATIM Tue Oct 02 10:17:21 2007
OBNUC 13C
EXMOD SINGL
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 129000.90 Hz
POINT 32768
FREQU 30959.75 Hz
SCANS 171
ACQTM 1.0584 sec
PD 2.0000 sec
PW1 5.00 usec
TRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 27
HO
Me
TBSO
OTBS
H
PPM
200
150
100
50
0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6.3907
6.1208
3.7323
1.5837
0.8937
0.8510
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0.0765
0.0466
0.0107
DFILE 459prd1-H.als
COMNT
DATIM Wed Oct 03 09:37:25 2007
OBNUC 1H
EXMOD SINGL
OBFRQ 500.00 MHz
OBSET 0.00 KHz
OBFIN 162410.00 Hz
POINT 16384
FREQU 6997.90 Hz
SCANS 8
ACQTM 2.3413 sec
PD 1.6587 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 26.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 15
OHC
Me
TBSO
OTBS
H
60
18.66
11.53
2.91
0.96
0.96
1.01
1.00
2.06
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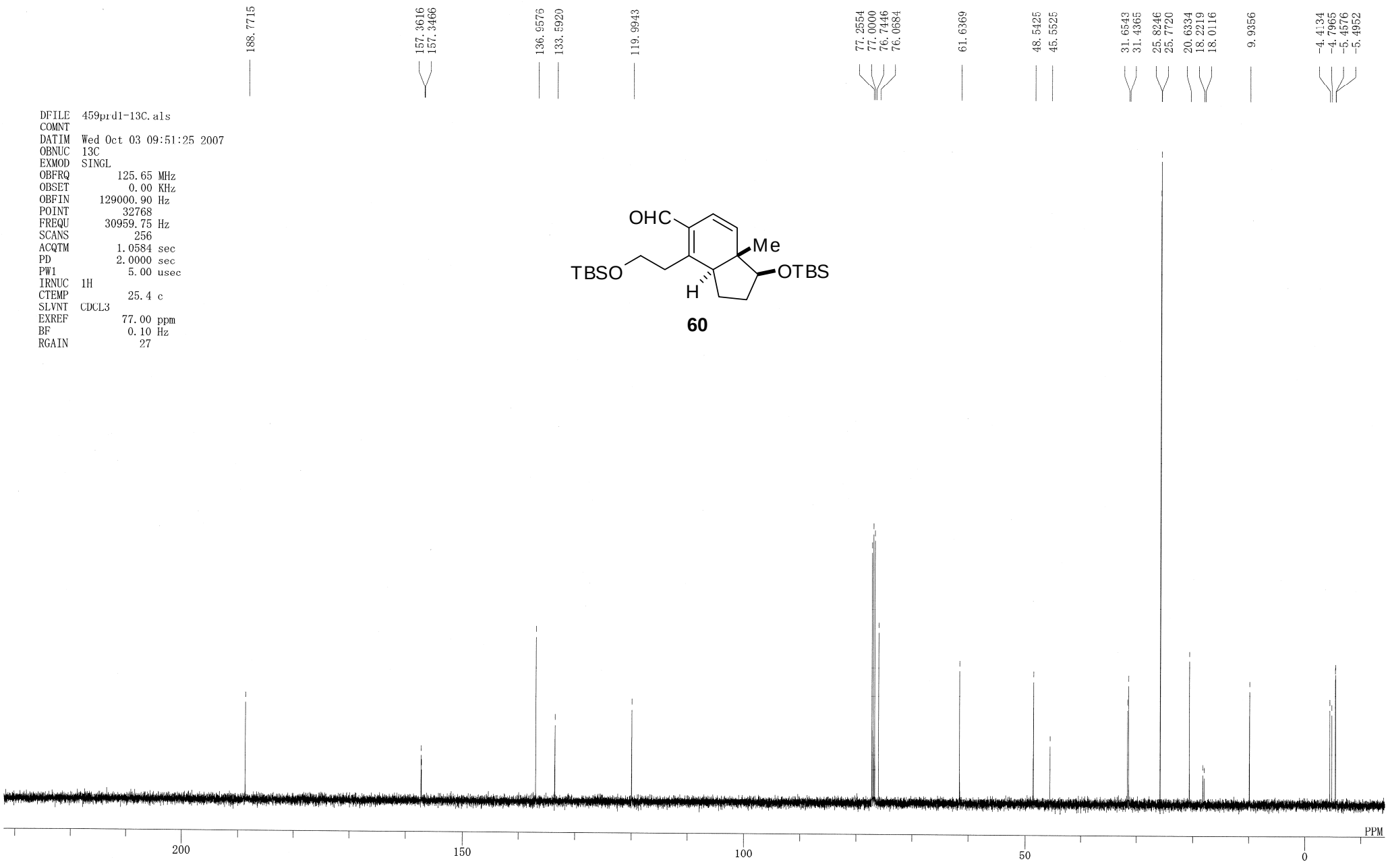
0.98
0.98
1.01
2.02
1.64
PPM
10
8
6
4
2
0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157.3616
157.3466
136.9576
133.5920
119.9943
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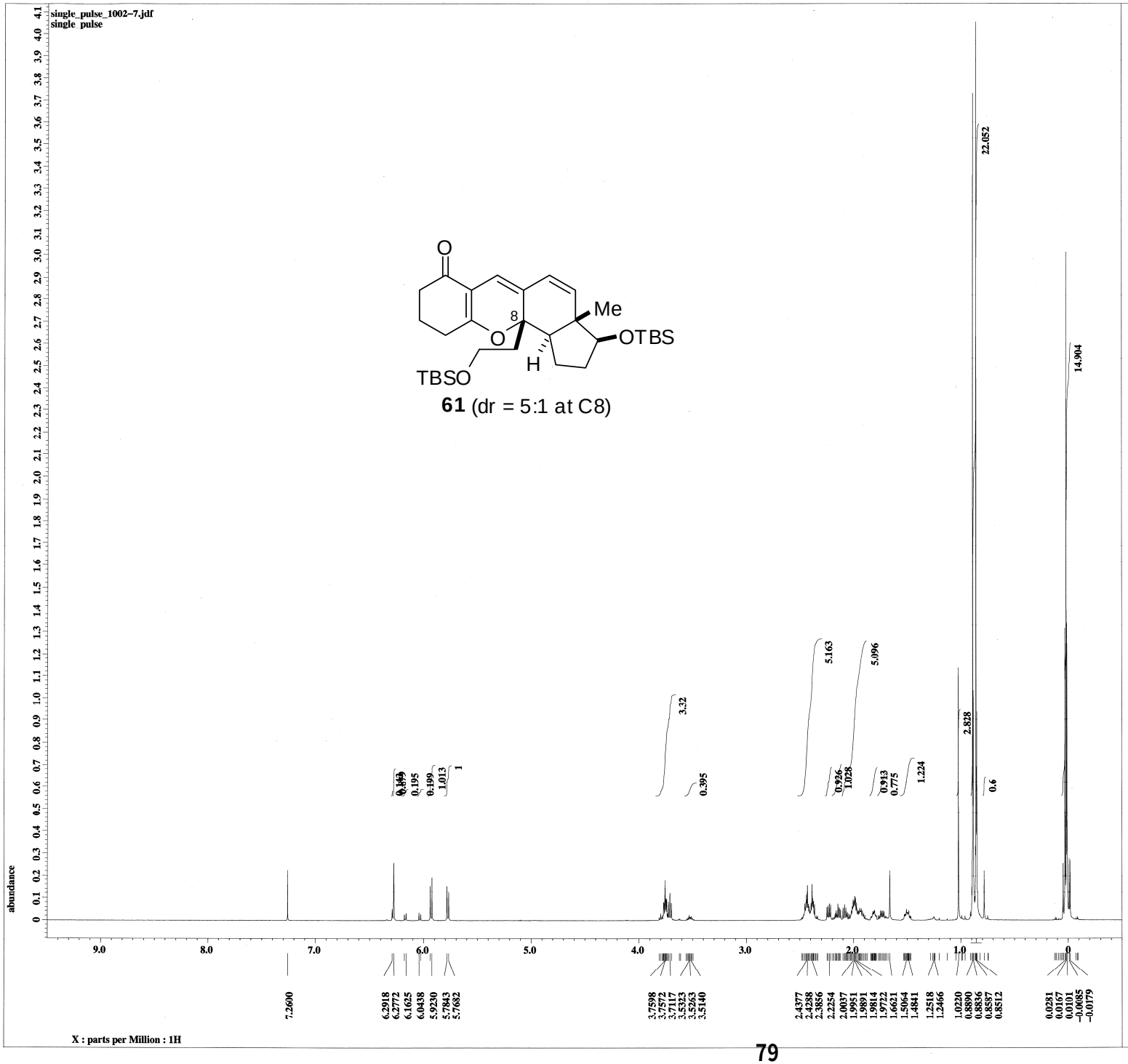
77.0000
76.7446
76.0684
61.6369
48.5425
45.5525
31.6543
31.4365
25.8246
25.7720
20.6334
18.2219
18.0116
9.9356
-4.4134
-4.7965
-5.4576
-5.4952
DFILE 459prd1-13C.als
COMNT
DATIM Wed Oct 03 09:51:25 2007
OBNUC 13C
EXMOD SINGL
OBFRQ 125.65 MHz
OBSET 0.00 KHz
OBFIN 129000.90 Hz
POINT 32768
FREQU 30959.75 Hz
SCANS 256
ACQTM 1.0584 sec
PD 2.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 25.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 27
OHC
Me
TBSO
OTBS
H
200
150
100
50
0
PPM

60

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Author            = delta
Experiment        = single_pulse.ex2
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Solvent           = CHLOROFORM-D
Creation_time     = 27-SEP-2007 09:34:45
Revision_time     = 27-SEP-2007 09:45:56
Current_time      = 27-SEP-2007 09:46:25

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 52428
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA600SL
Spectrometer      = DELTA2_NMR

Field_strength    = 13.95540559[T] (590[M
X_acq_duration    = 5.88251136[s]
X_domain          = 1H
X_freq            = 594.17058168[MHz]
X_offset          = 5[ppm]
X_points          = 65536
X_prescans        = 1
X_resolution      = 0.16999542[Hz]
X_sweep           = 11.14081996[kHz]
Irr_domain        = 1H
Irr_freq          = 594.17058168[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 594.17058168[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 14[us]
X_acq_time        = 5.88251136[s]
X_angle           = 45[deg]
X_atn             = 8.5[dB]
X_pulse           = 7[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 2[s]
Repetition_time   = 7.88251136[s]
Temp_get          = 23.5[dC]
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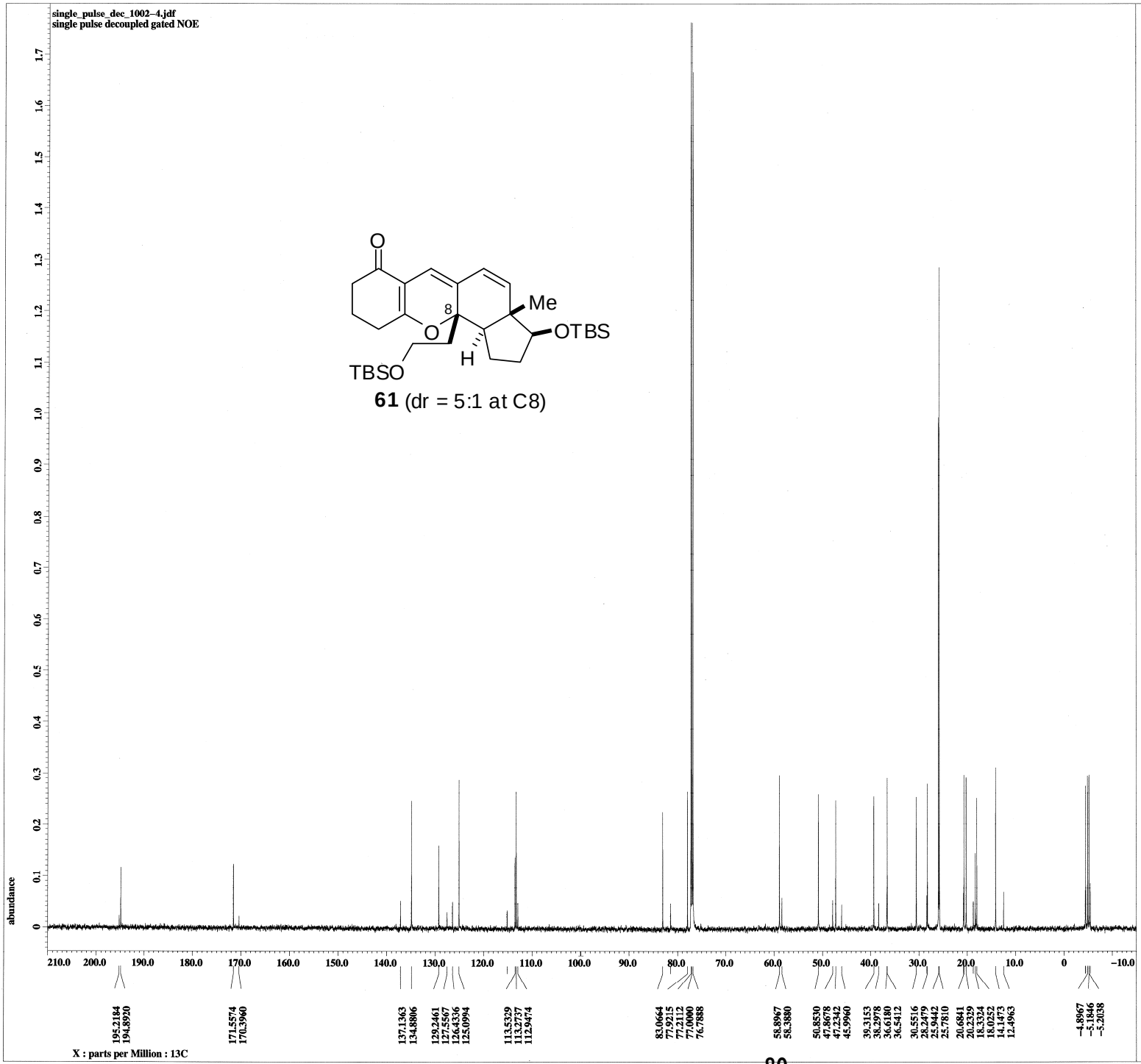


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Filename          = single_pulse_dec_1002
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = a
Solvent           = CHLOROFORM-D
Creation_time     = 27-SEP-2007 11:14:53
Revision_time     = 28-APR-2008 15:12:43
Current_time      = 28-APR-2008 15:13:46

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA600SL
Spectrometer      = DELTA2_NMR

Field_strength    = 13.95540559[T] (590[M
X_acq_duration    = 0.69730304[s]
X_domain          = 13C
X_freq            = 149.40429612[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.43409672[Hz]
X_sweep           = 46.9924812[kHz]
Irr_domain        = 1H
Irr_freq          = 594.17058168[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 11.2[us]
X_acq_time        = 0.69730304[s]
X_angle           = 40[deg]
X_atn             = 8[dB]
X_pulse           = 4.97777778[us]
Irr_atn_dec       = 20.5[dB]
Irr_atn_noe       = 20.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 58
Relaxation_delay  = 2[s]
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Temp_get          = 25.1[dC]
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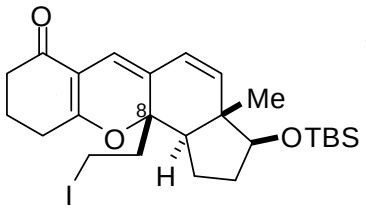
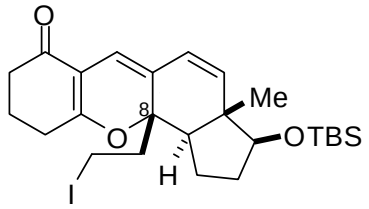
^{1}H-NMR
400 MHz
$CDCl_3$



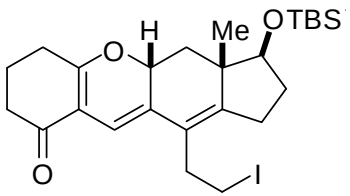
69 (dr=8:1 at C8)

^{13}C-NMR
100 MHz
$CDCl_3$

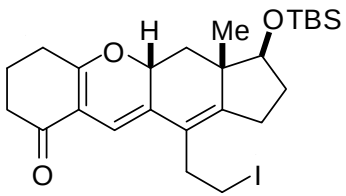


69 (dr=8:1 at C8)

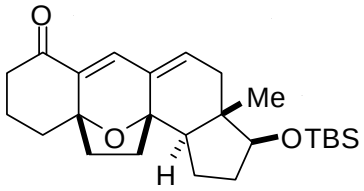
^{1}H-NMR
400 MHz
$CDCl_3$



^{13}C-NMR
100 MHz
$CDCl_3$

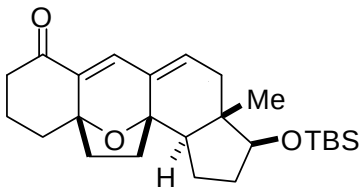


^{1}H-NMR
400 MHz
$CDCl_3$



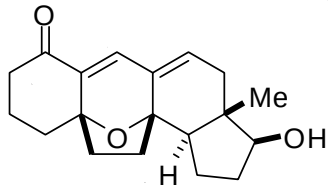
72

^{13}C-NMR
100 MHz
$CDCl_3$



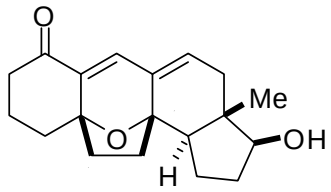
72

^{1}H-NMR
400 MHz
$CDCl_3$



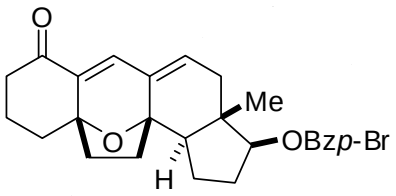
73

^{13}C-NMR
100 MHz
$CDCl_3$



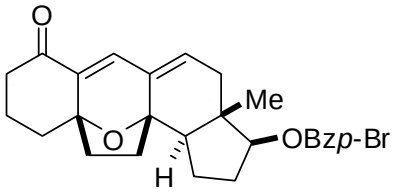
73

^{1}H-NMR
400 MHz
$CDCl_3$



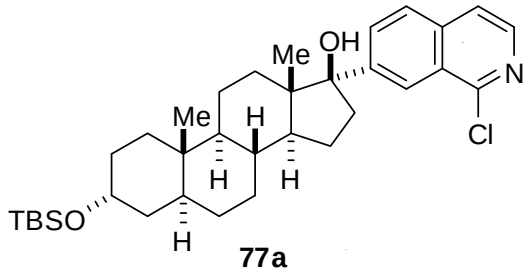
74

^{13}C-NMR
100 MHz
$CDCl_3$



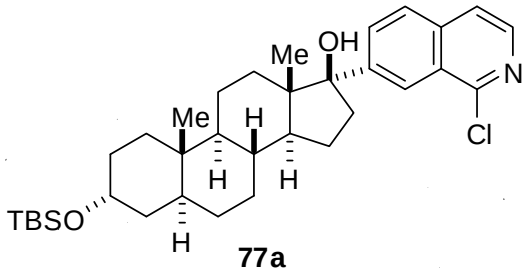
74

^{1}H-NMR
400 MHz
$CDCl_3$



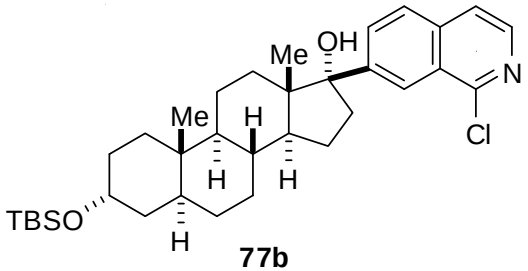
77a

^{13}C-NMR
100 MHz
$CDCl_3$



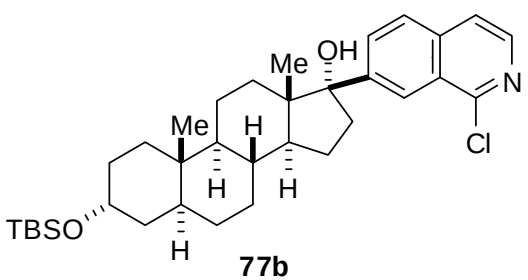
77a

^{1}H-NMR
500 MHz
$CDCl_3$



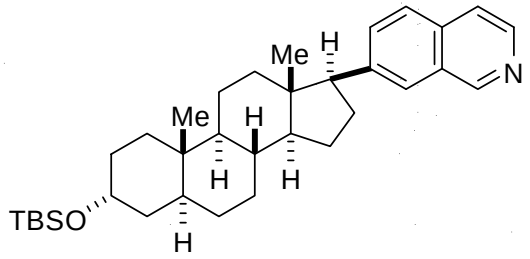
77b

^{13}C-NMR
100 MHz
$CDCl_3$



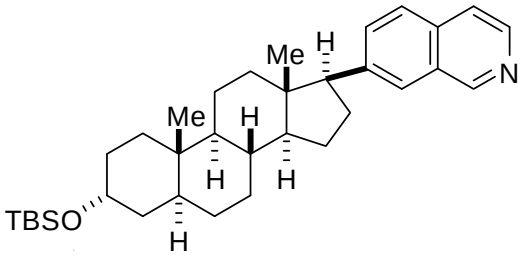
77b

^{1}H-NMR
400 MHz
$CDCl_3$



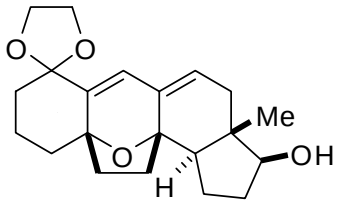
79

^{13}C-NMR
100 MHz
$CDCl_3$



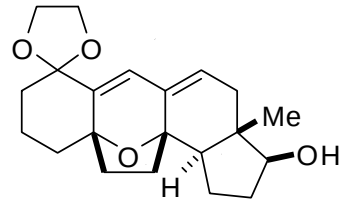
79

^{1}H-NMR
400 MHz
$CDCl_3$



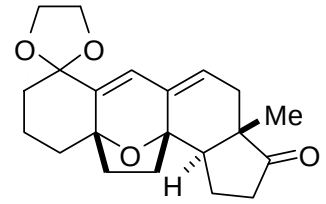
80

^{13}C-NMR
100 MHz
$CDCl_3$



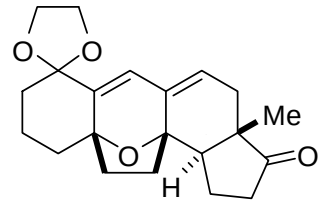
80

^{1}H-NMR
400 MHz
$CDCl_3$



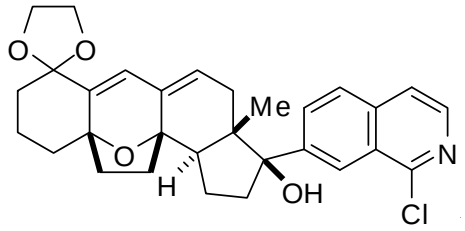
81

^{13}C-NMR
100 MHz
$CDCl_3$

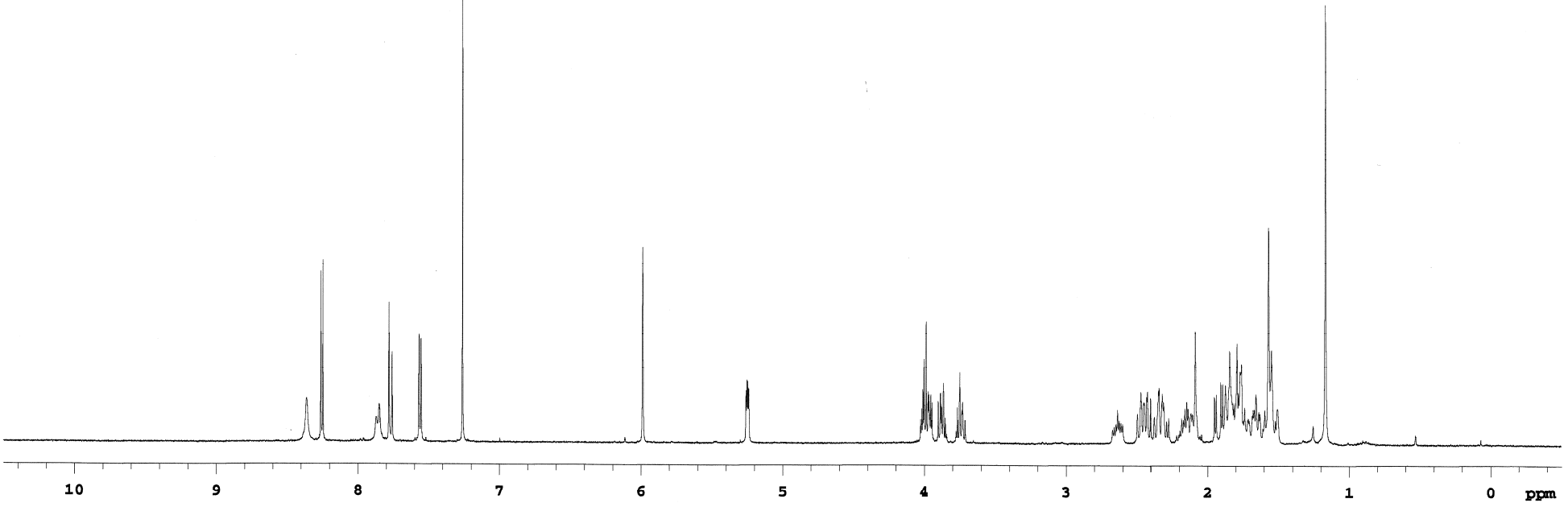


81

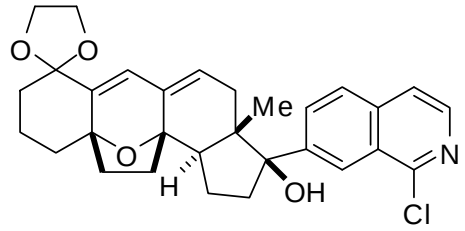
^{1}H-NMR
400 MHz
$CDCl_3$



82

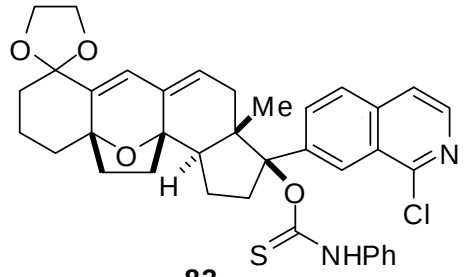


^{13}C-NMR
100 MHz
$CDCl_3$



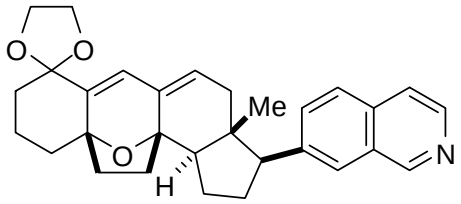
82

^{1}H-NMR
400 MHz
$CDCl_3$



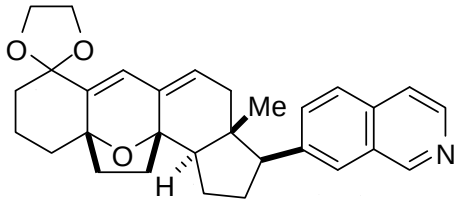
83

^{1}H-NMR
400 MHz
$CDCl_3$

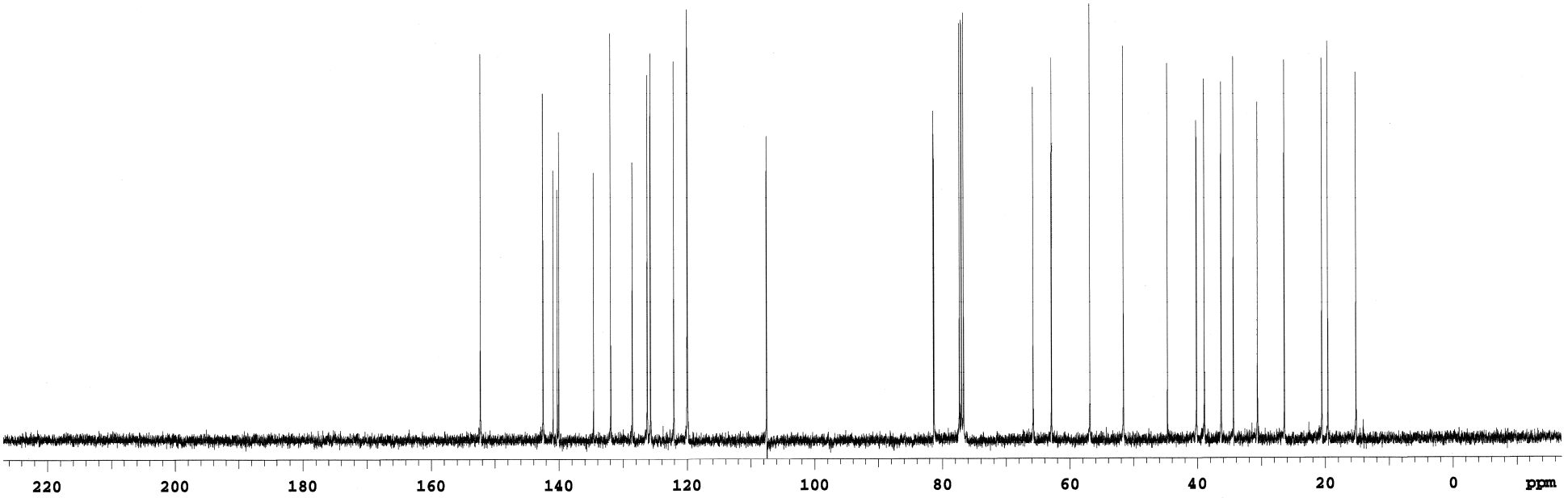


84

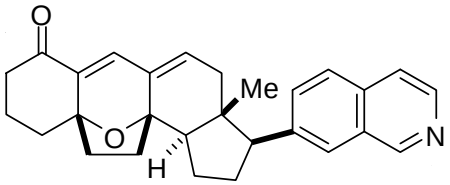
^{13}C-NMR
100 MHz
$CDCl_3$



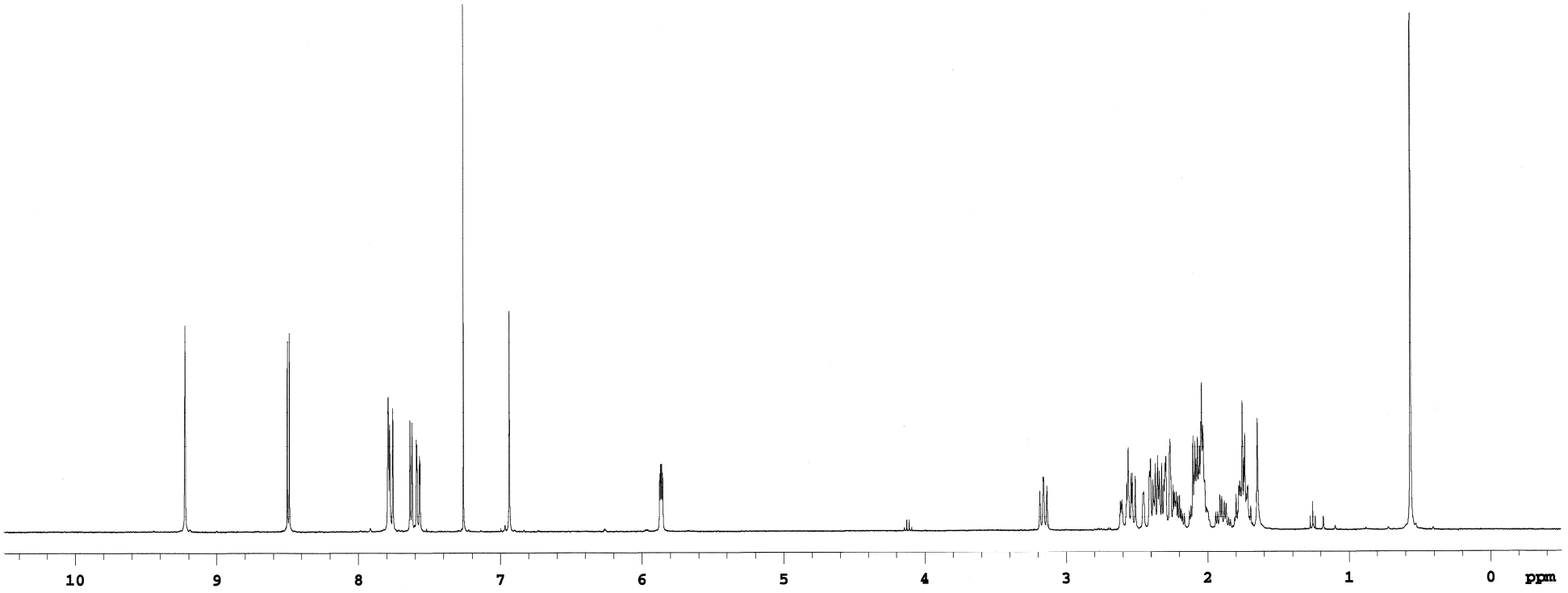
84



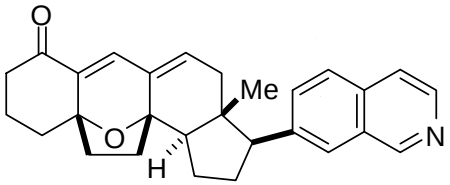
^{1}H-NMR
400 MHz
$CDCl_3$



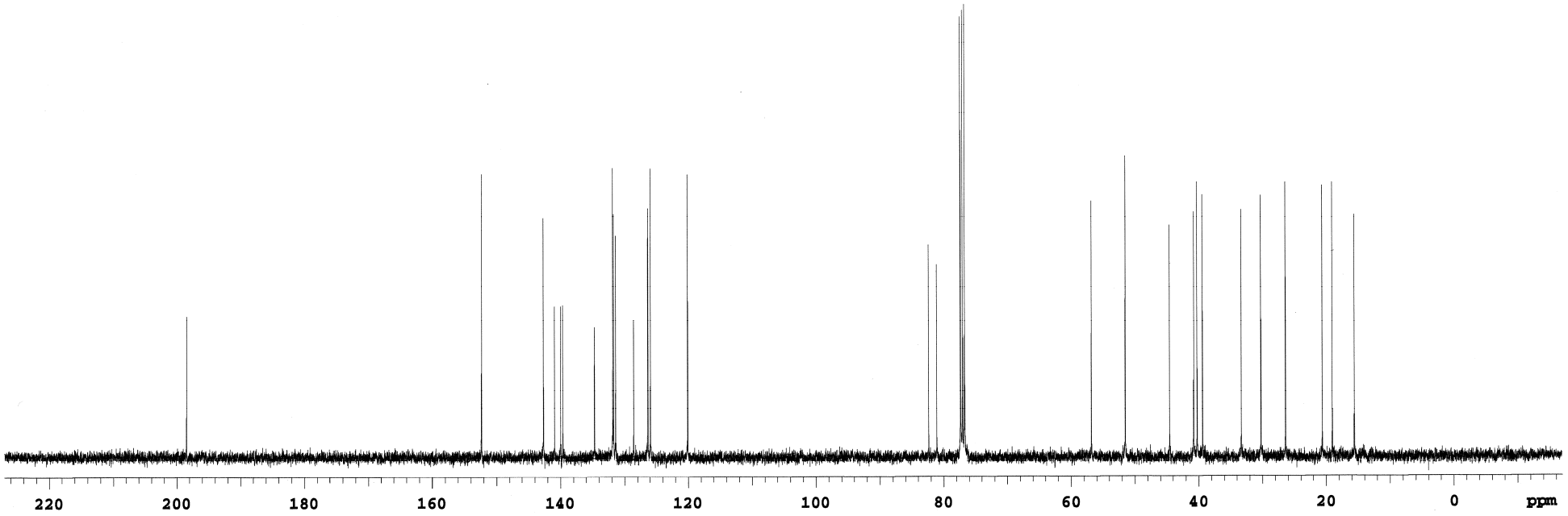
85



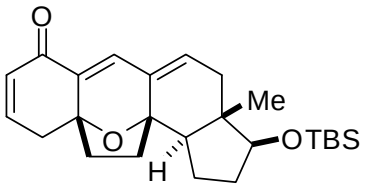
^{13}C-NMR
100 MHz
$CDCl_3$



85

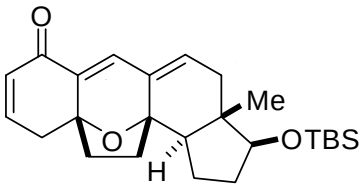


^{1}H-NMR
400 MHz
$CDCl_3$



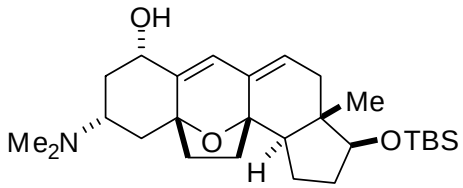
87

^{13}C-NMR
100 MHz
$CDCl_3$



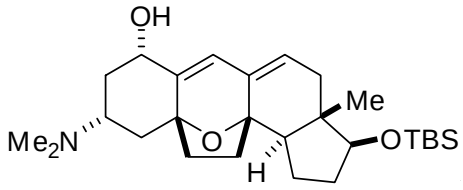
87

^{1}H-NMR
400 MHz
$CDCl_3$



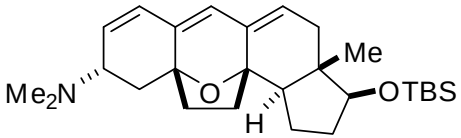
88

^{13}C-NMR
100 MHz
$CDCl_3$



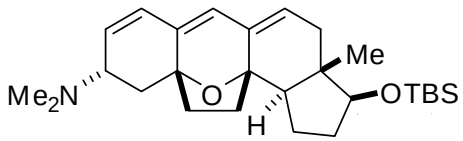
88

^{1}H-NMR
400 MHz
$CDCl_3$



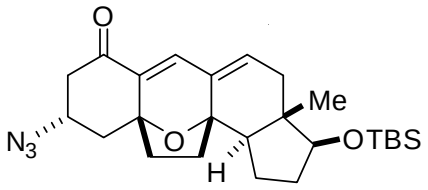
89

^{13}C-NMR
100 MHz
$CDCl_3$



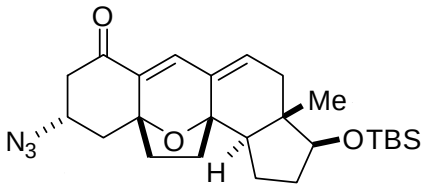
89

^{1}H-NMR
400 MHz
$CDCl_3$



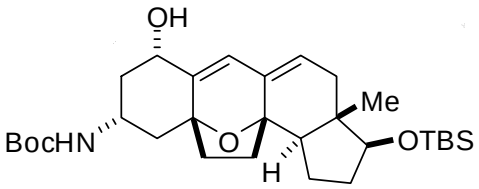
92

^{13}C-NMR
100 MHz
$CDCl_3$

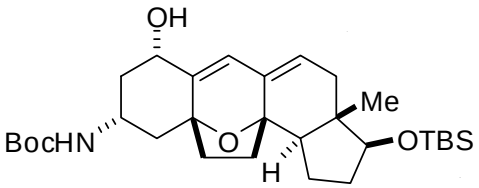


92

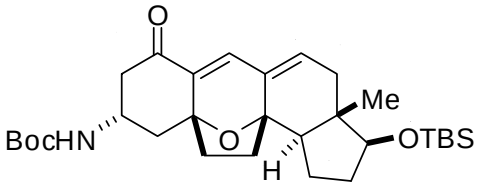
^{1}H-NMR
400 MHz
$CDCl_3$



^{13}C-NMR
100 MHz
$CDCl_3$

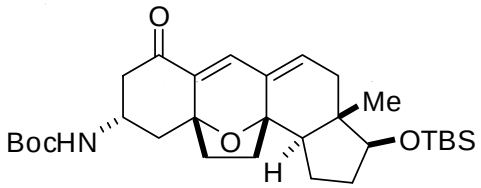


^{1}H-NMR
400 MHz
$CDCl_3$



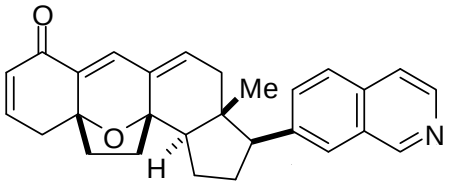
94

^{13}C-NMR
100 MHz
$CDCl_3$



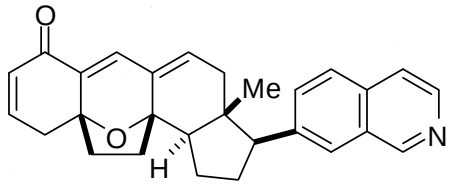
94

^{1}H-NMR
500 MHz
$CDCl_3$



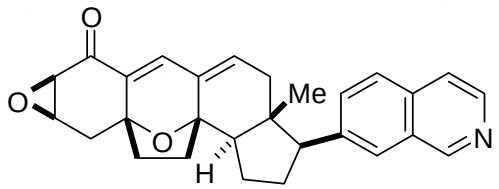
96

^{13}C-NMR
100 MHz
$CDCl_3$

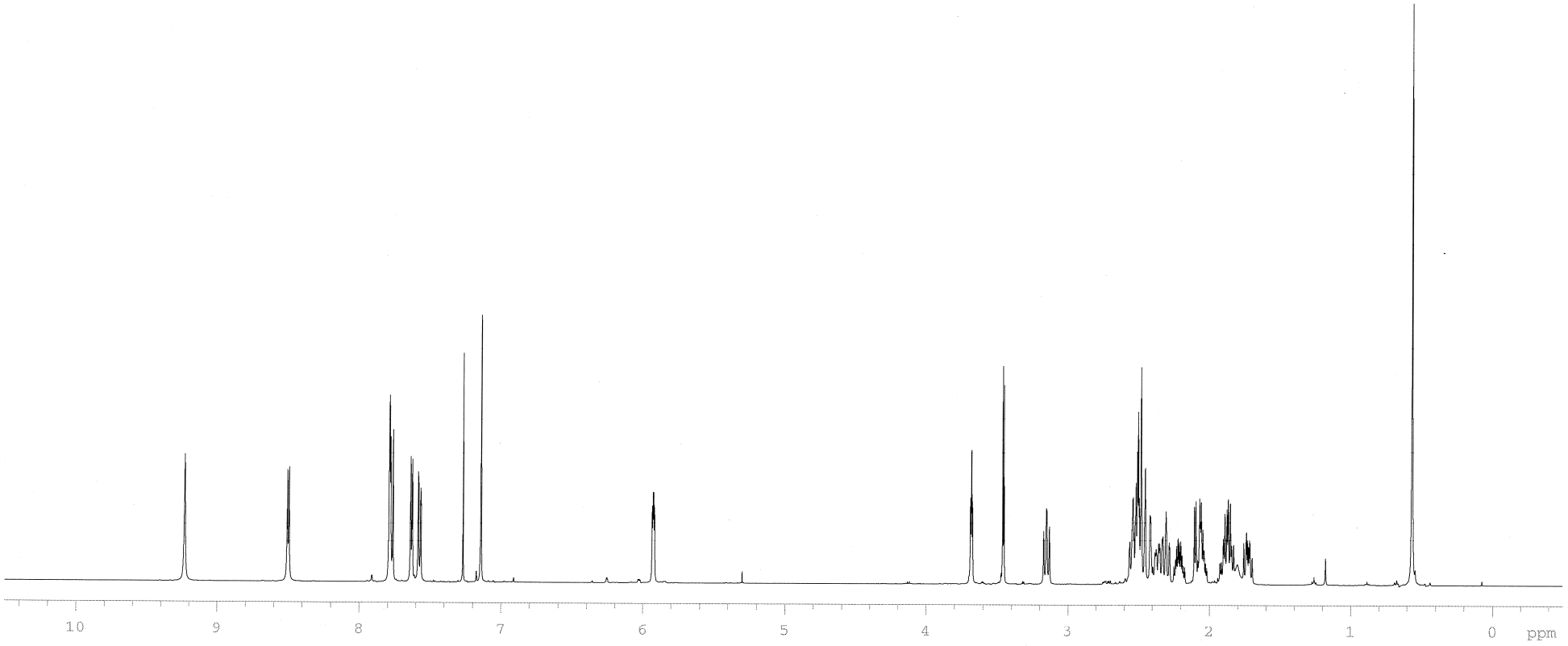


96

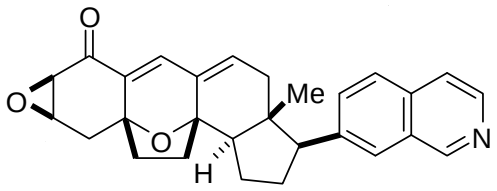
^{1}H-NMR
500 MHz
$CDCl_3$



97

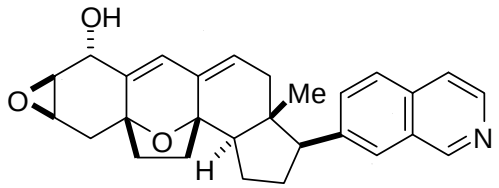


^{13}C-NMR
100 MHz
$CDCl_3$

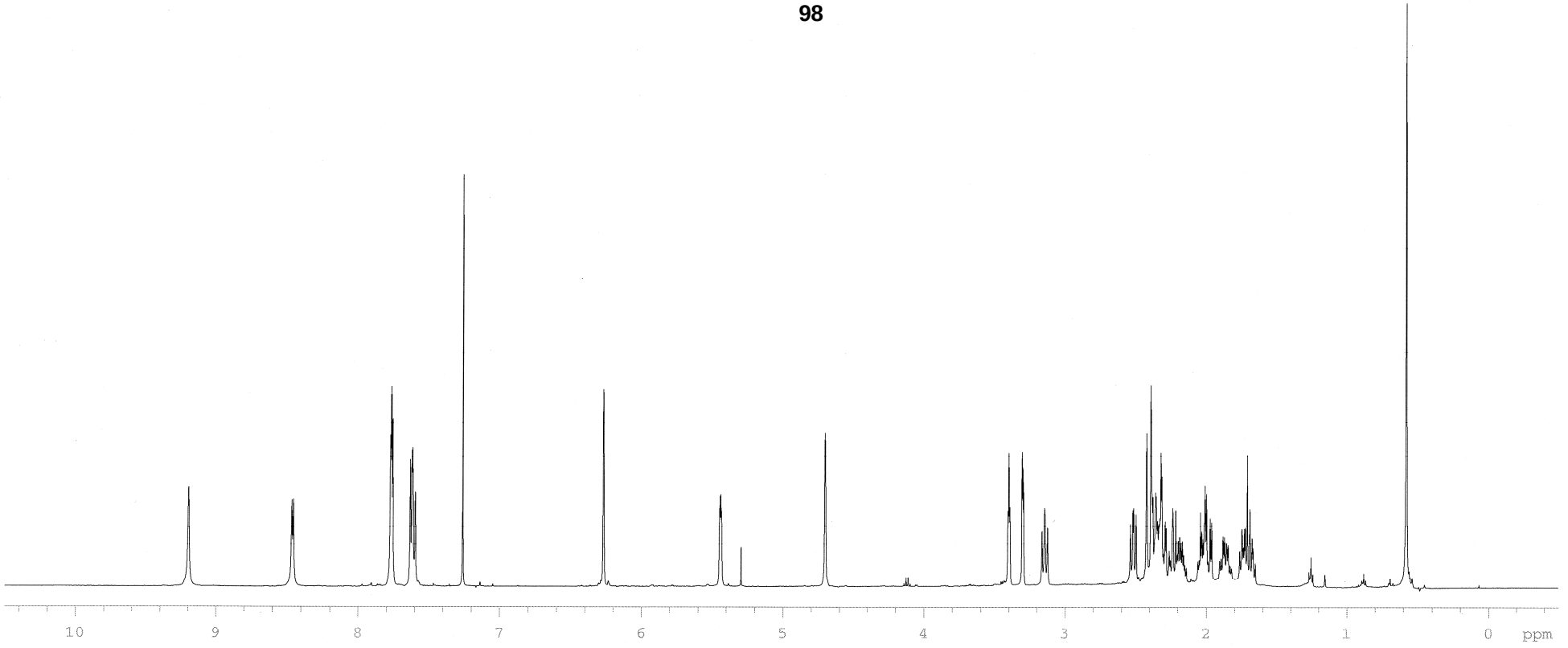


97

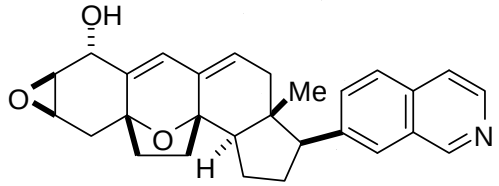
^{1}H-NMR
500 MHz
$CDCl_3$



98

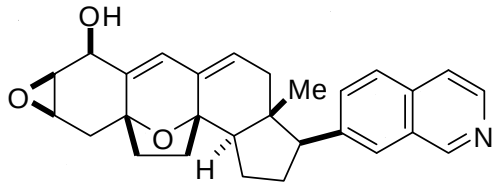


^{13}C-NMR
100 MHz
$CDCl_3$

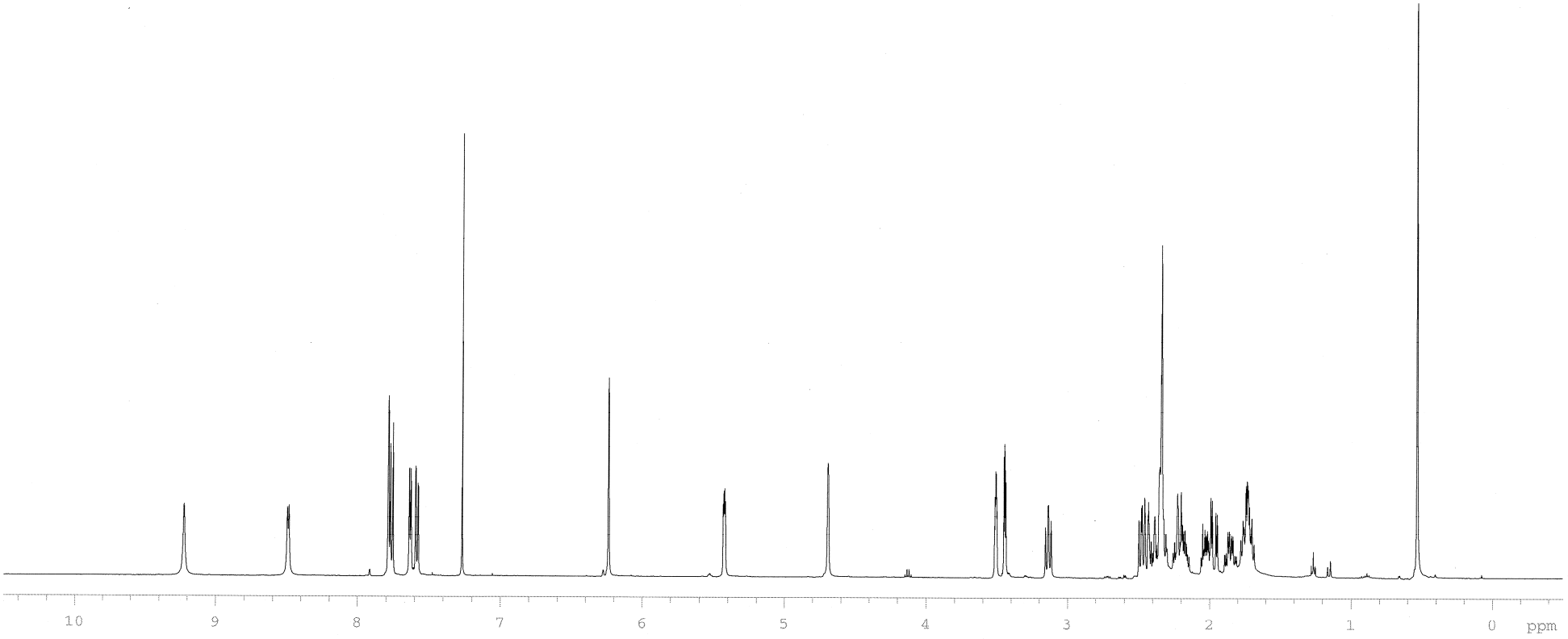


98

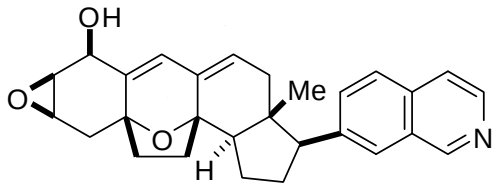
^{1}H-NMR
500 MHz
$CDCl_3$



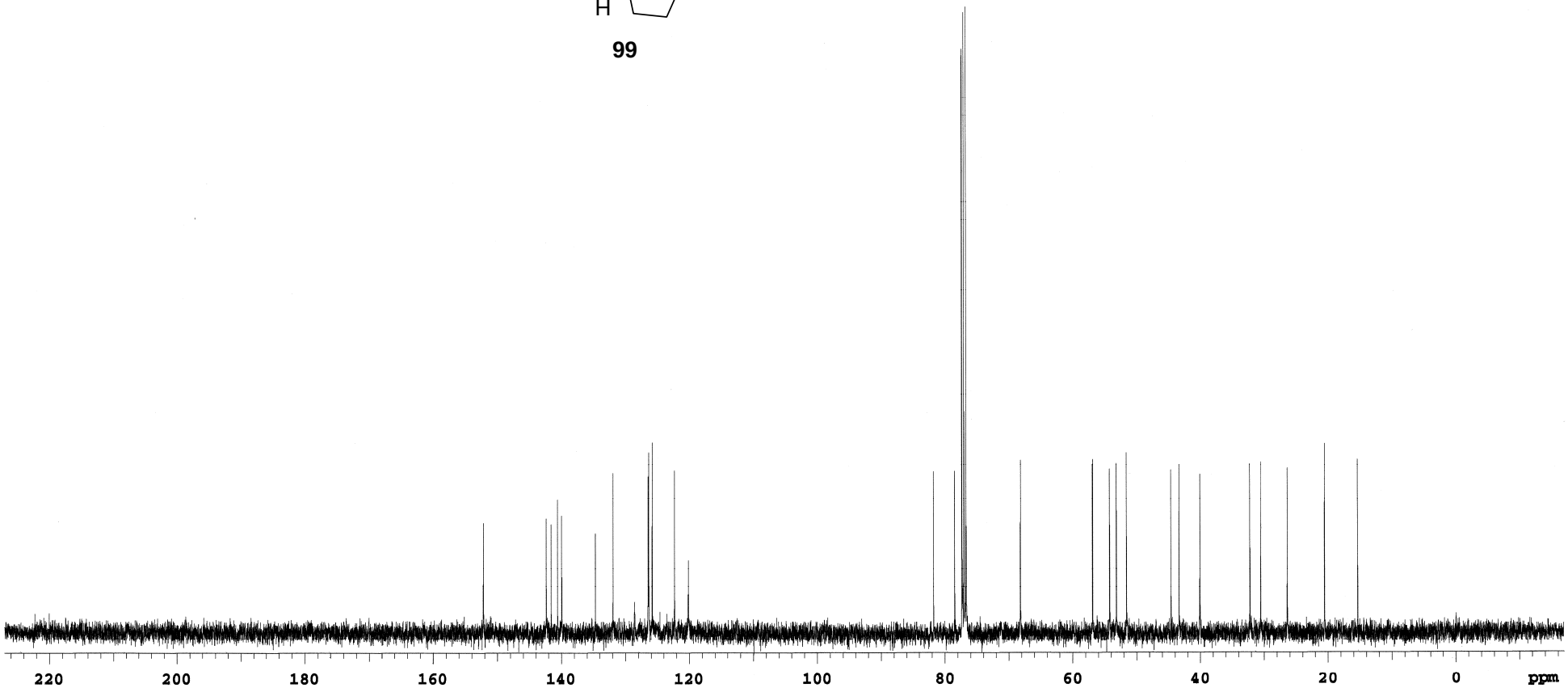
99



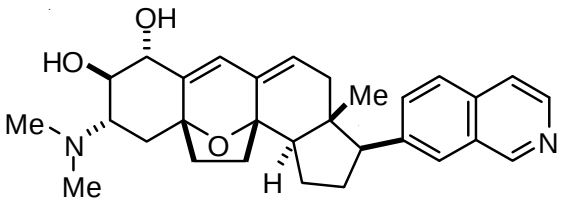
^{13}C-NMR
100 MHz
$CDCl_3$



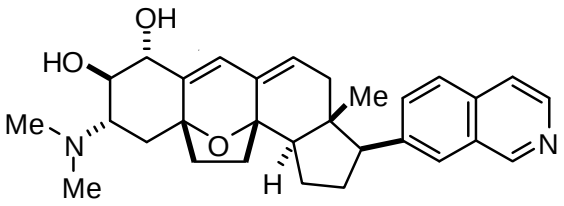
99



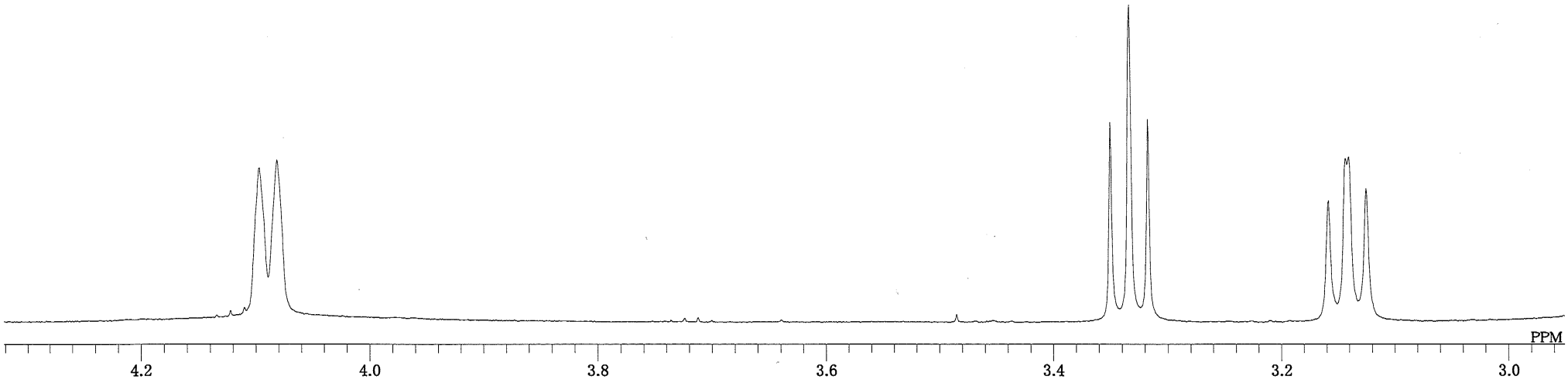
DFILE	110204_single_pulse-3.jdf
COMNT	single_pulse
DATIM	04-02-2011 11:36:33
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	594.17 MHz
OBSET	3.55 KHz
OBFIN	2.53 Hz
POINT	32768
FREQU	11140.82 Hz
SCANS	16
ACQTM	2.9413 sec
PD	2.0000 sec
PW1	7.90 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	44

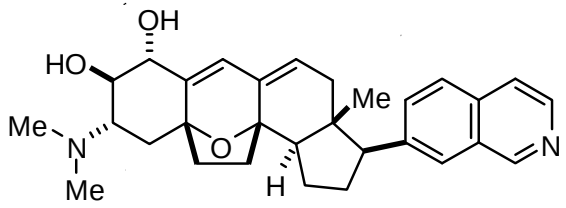


cortistatin A (1)



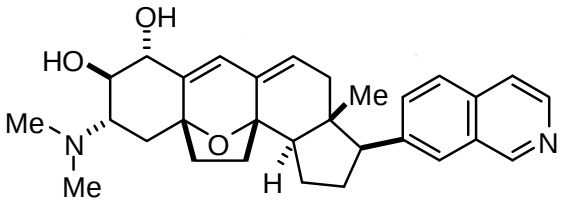
cortistatin A (1)





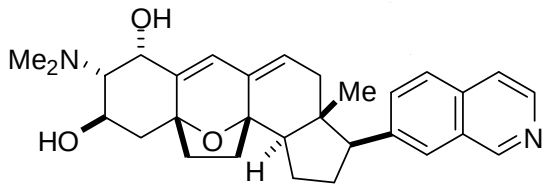
cortistatin A (1)

DFILE	110204_single_pulse_dec-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	04-02-2011 14:01:56
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	149.41 MHz
OBSET	9.23 KHz
OBFIN	6.55 Hz
POINT	32768
FREQU	46992.48 Hz
SCANS	3815
ACQTM	0.6973 sec
PD	1.5000 sec
PW1	5.07 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



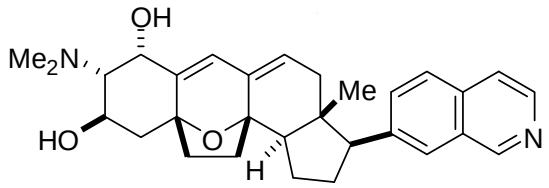
cortistatin A (1)

^{1}H-NMR
500 MHz
$CDCl_3$

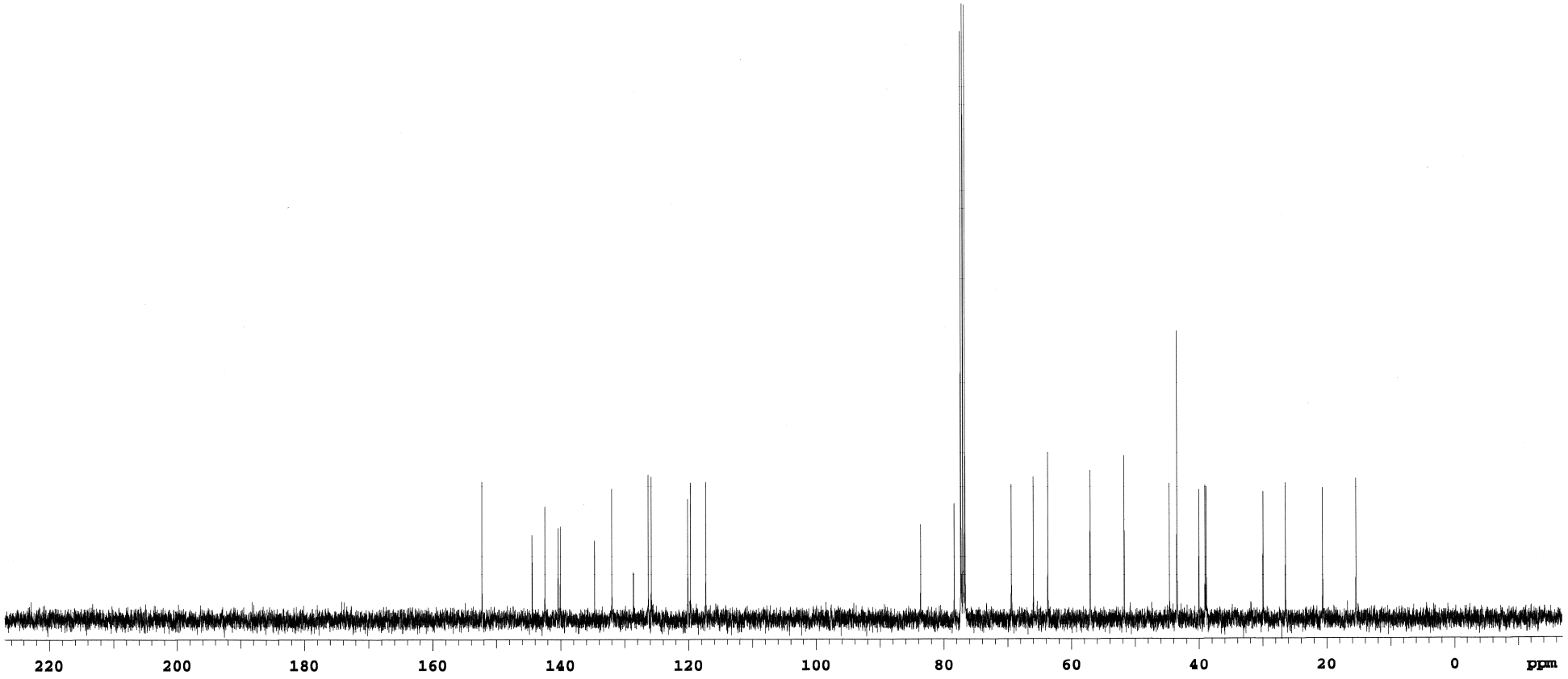


100

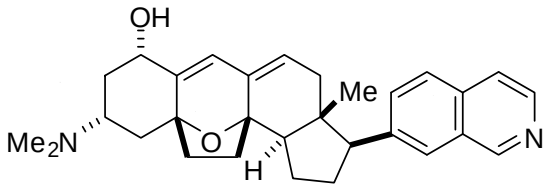
^{13}C-NMR
100 MHz
$CDCl_3$



100

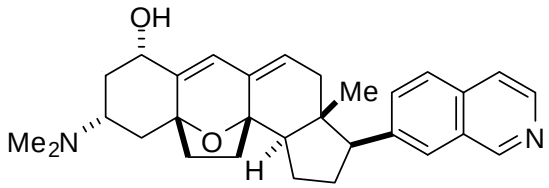


^{1}H-NMR
400 MHz
$CDCl_3$

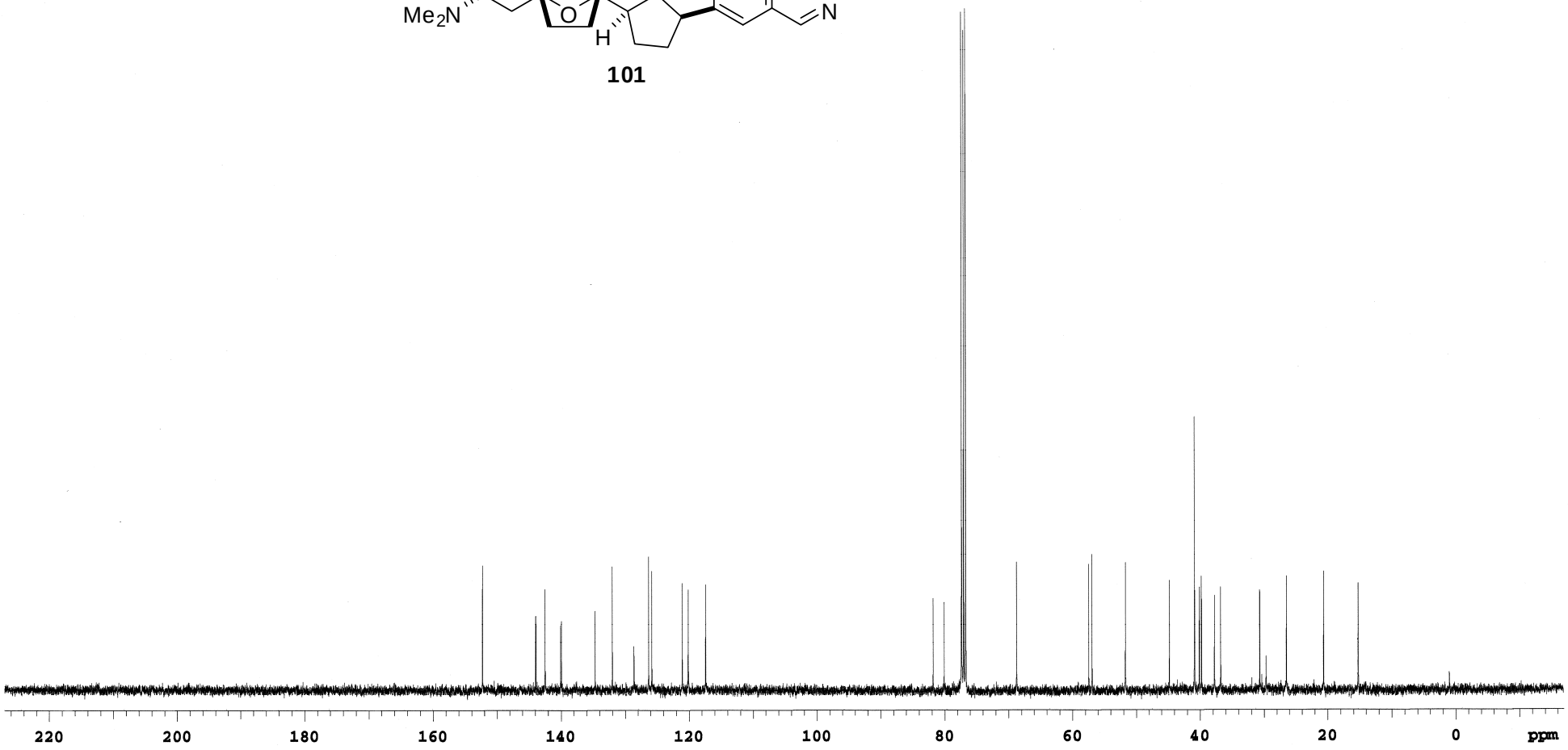


101

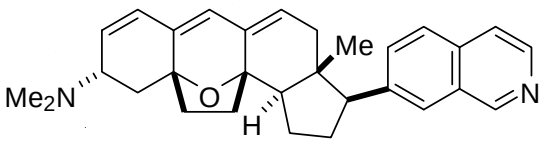
^{13}C-NMR
100 MHz
$CDCl_3$



101

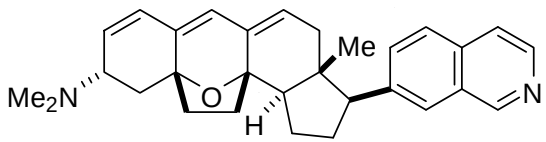


^{1}H-NMR
500 MHz
$CDCl_3$

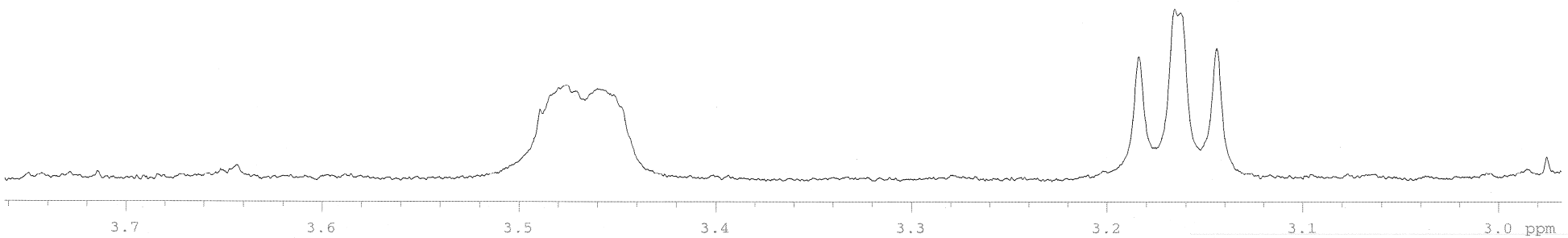


cortistatin J (**5**)

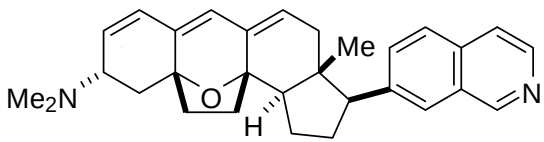
^{1}H-NMR
500 MHz
$CDCl_3$



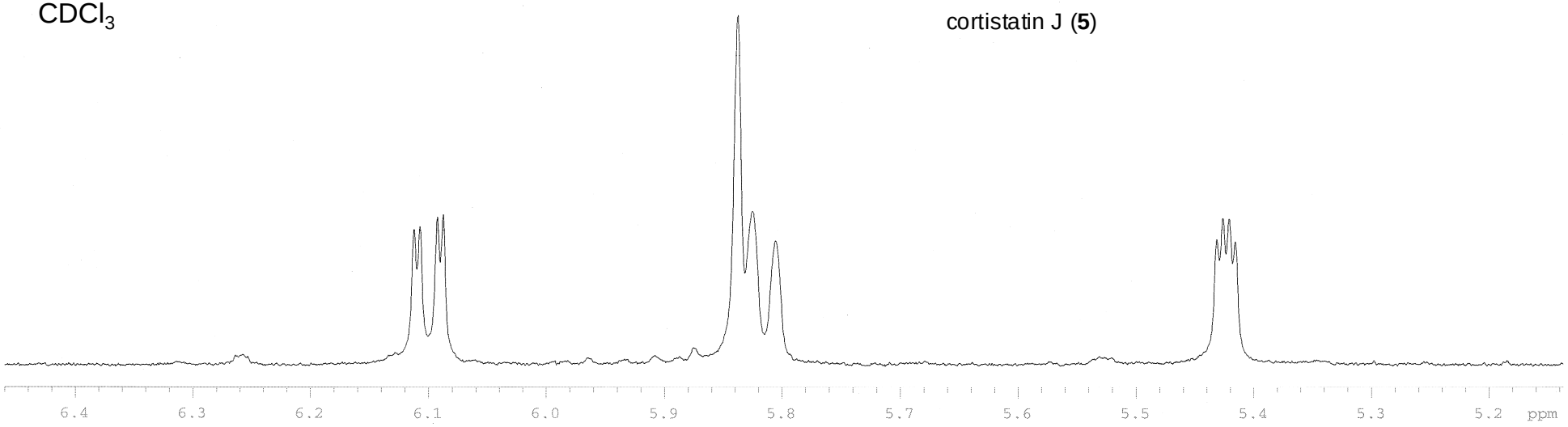
cortistatin J (**5**)

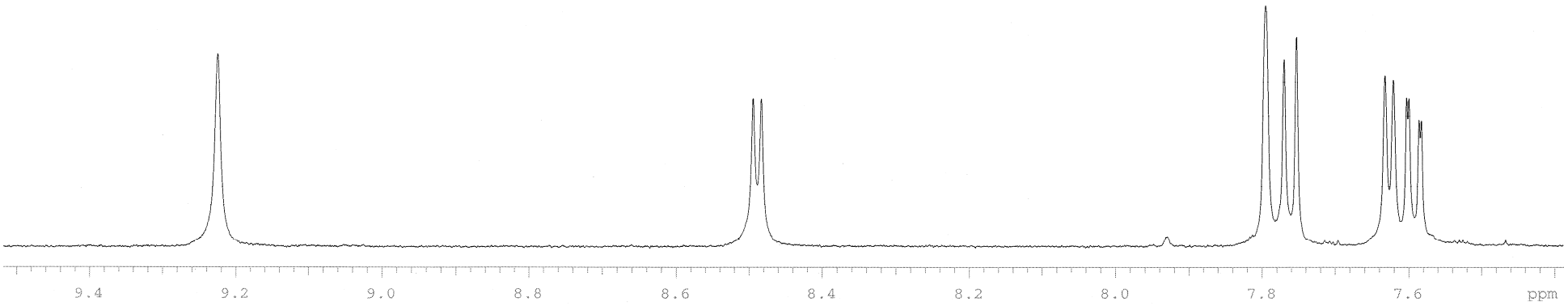


^{1}H-NMR
500 MHz
$CDCl_3$

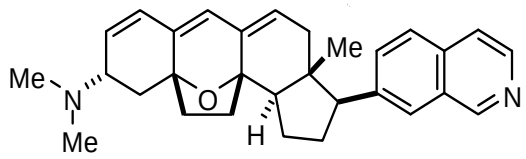


cortistatin J (**5**)





DFILE	110203_single_pulse_dec-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	03-02-2011 10:02:22
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	149.41 MHz
OBSET	9.23 KHz
OBFIN	6.55 Hz
POINT	32768
FREQU	46992.48 Hz
SCANS	38661
ACQTM	0.6973 sec
PD	1.5000 sec
PW1	5.07 usec
IRNUC	1H
CTEMP	24.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



cortistatin J (2)