APPENDIX B

Spectra and X-Ray Crystrallographic Data: Acid-Mediated Cyclization Approaches to the Densely Substituted Carbocyclic Core of Zoanthenol

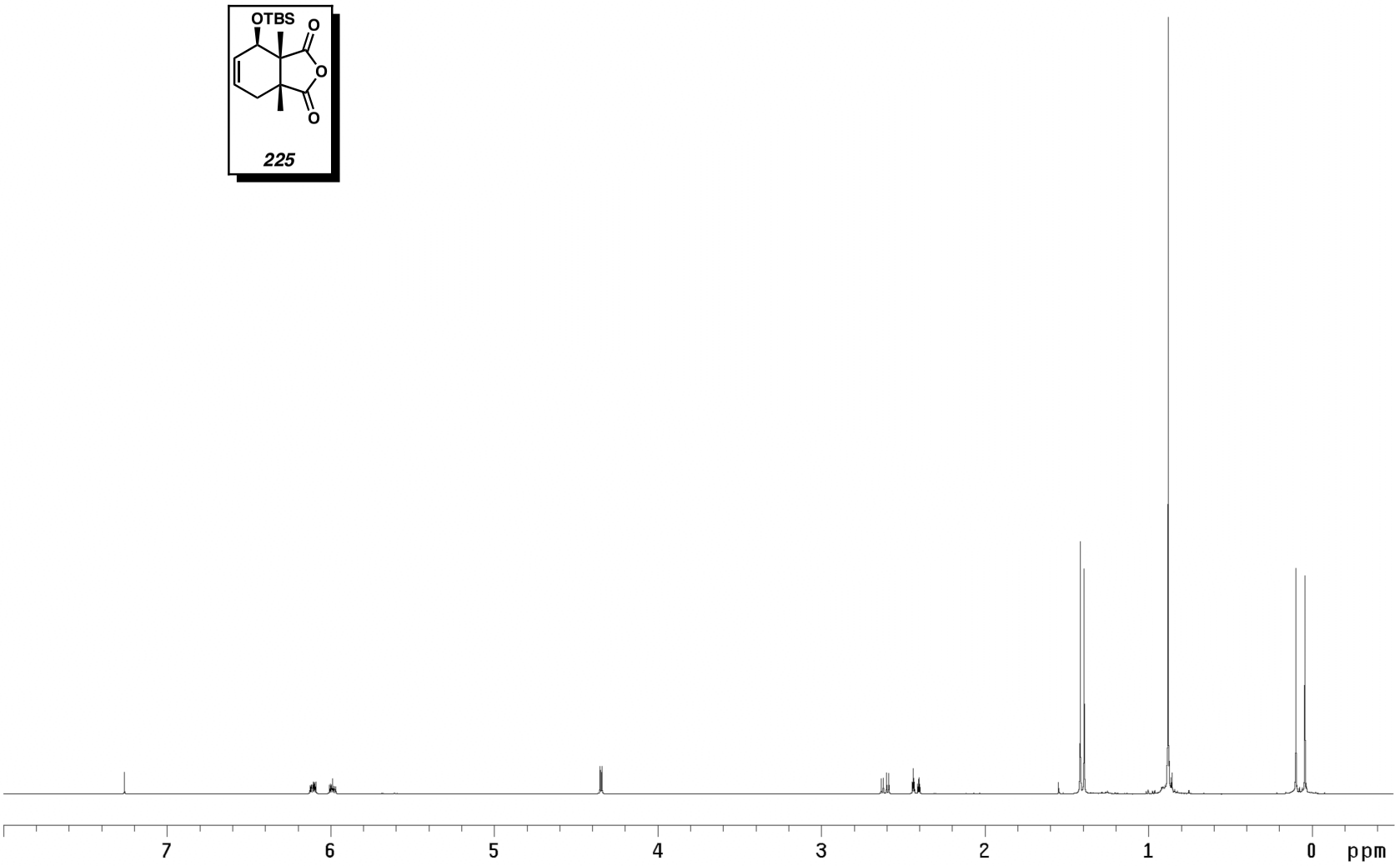

Figure B.1 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **225**.

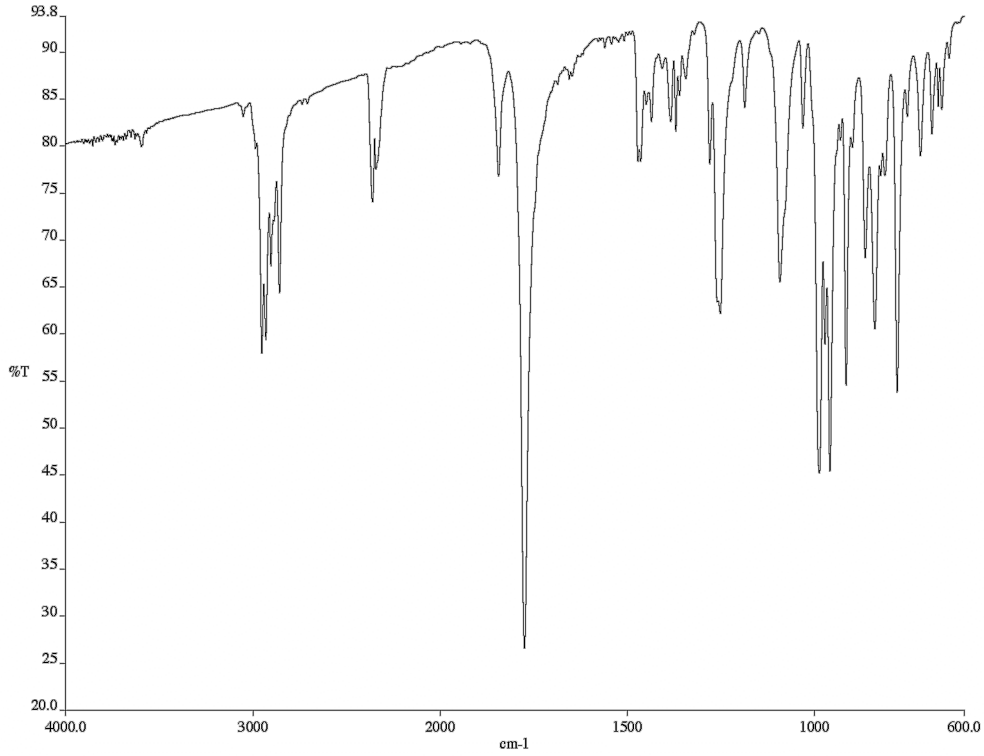

Figure B.2 Infrared spectrum (thin film/NaCl) of compound **225**.

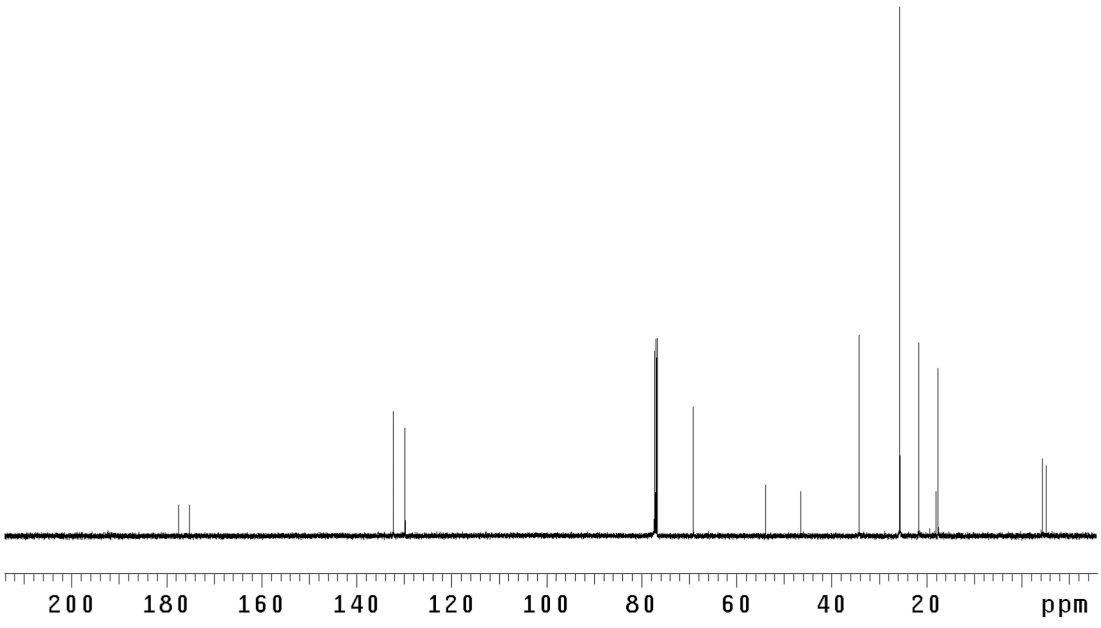

Figure B.3 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **225**.

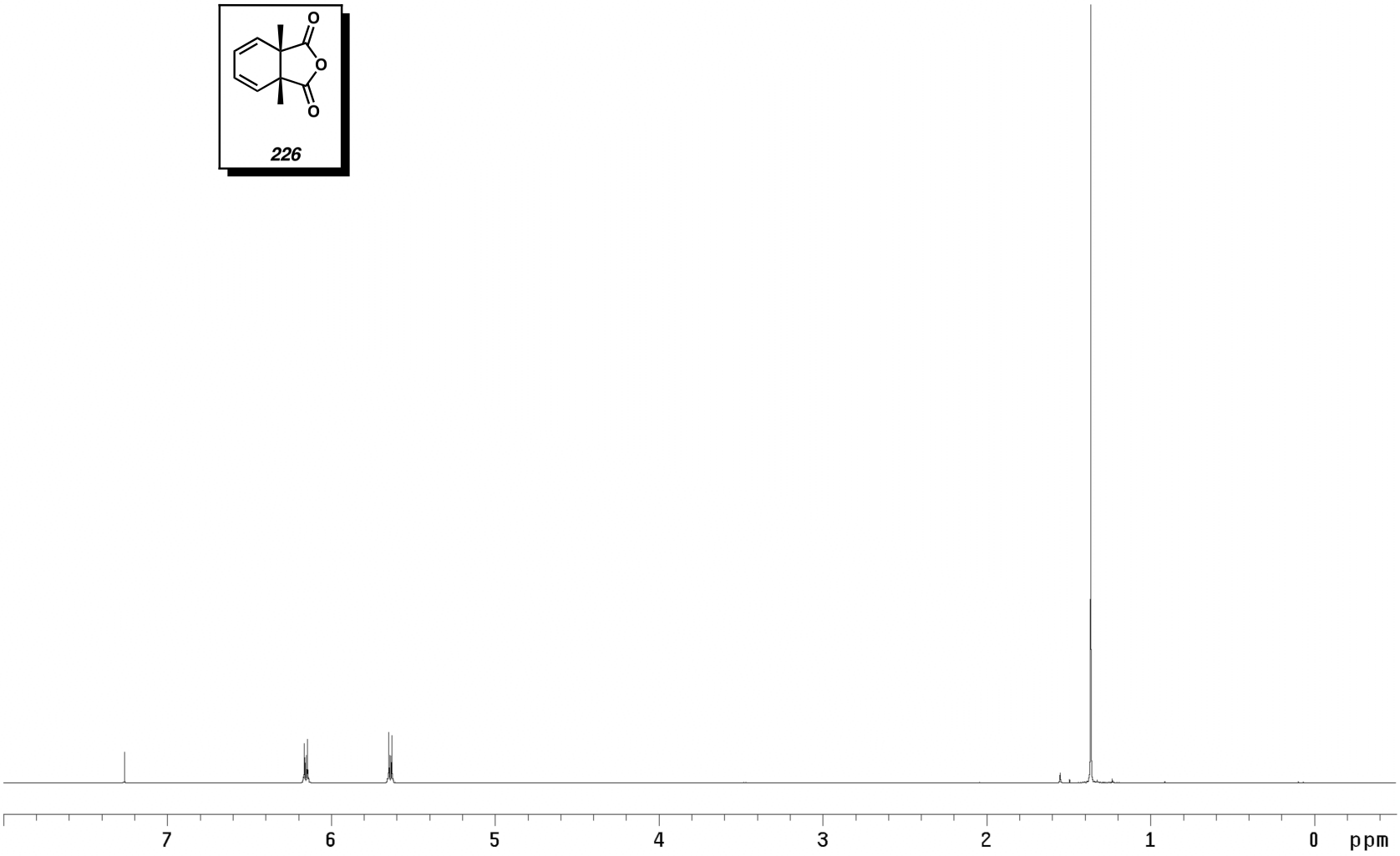

Figure B.4 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **226**.

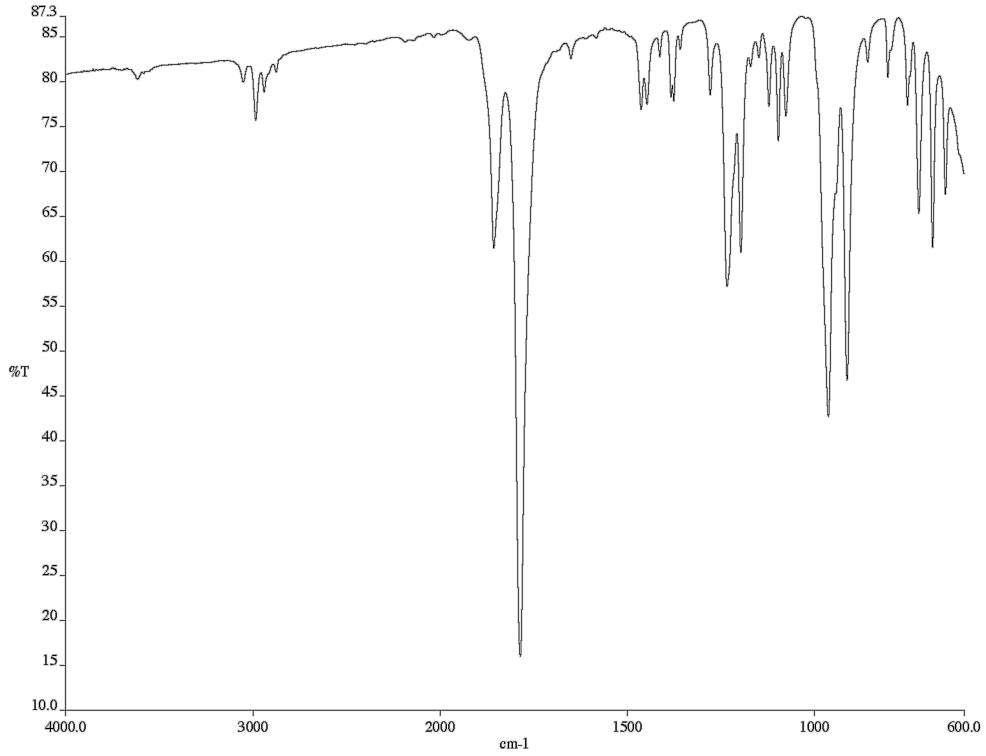

Figure B.5 Infrared spectrum (thin film/NaCl) of compound **226**.

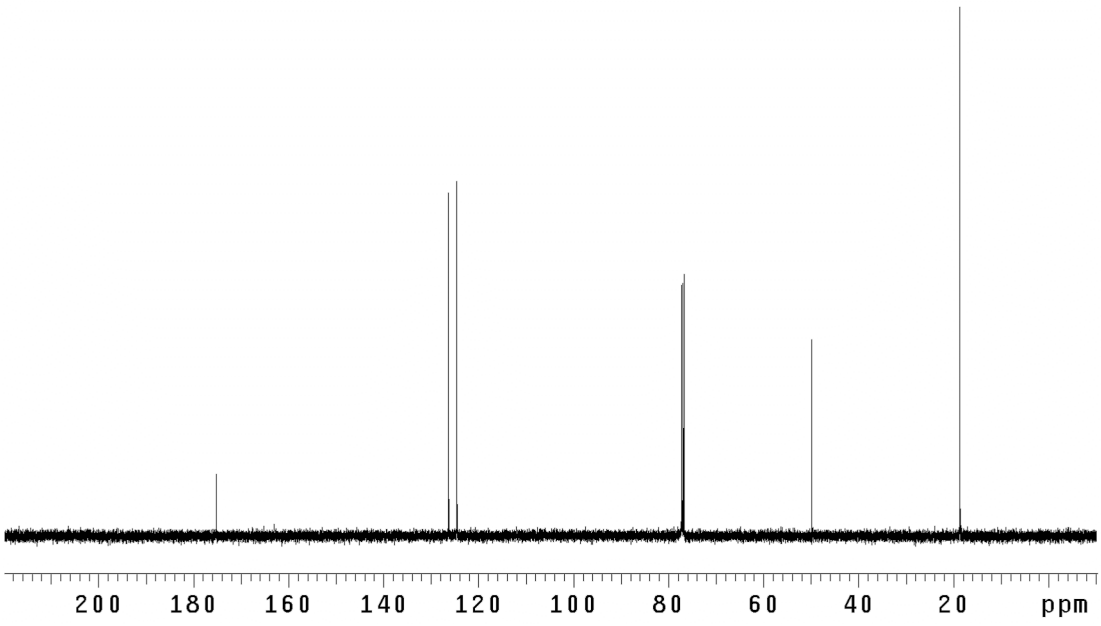

Figure B.6 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **226**.

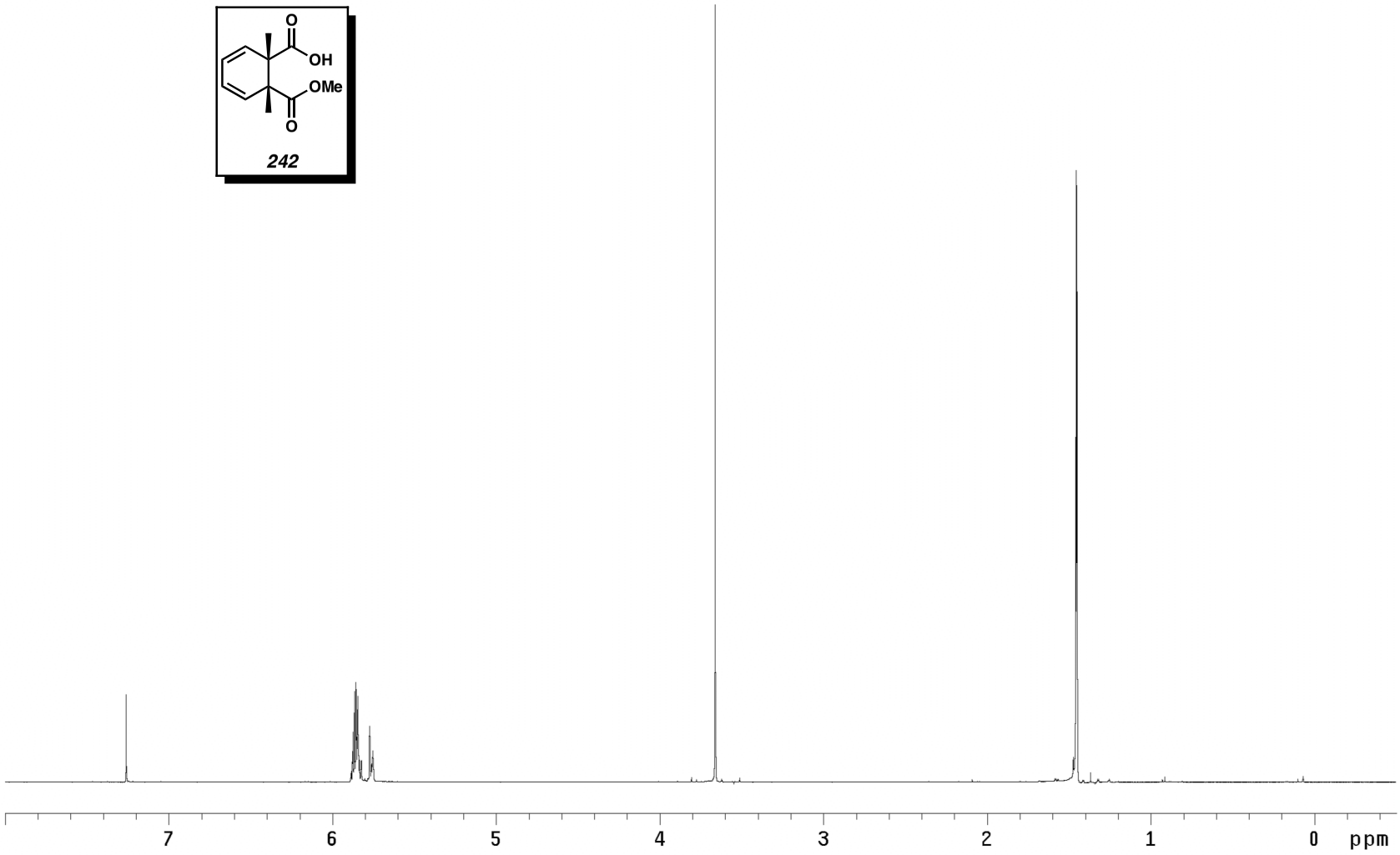

Figure B.7 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **242**.

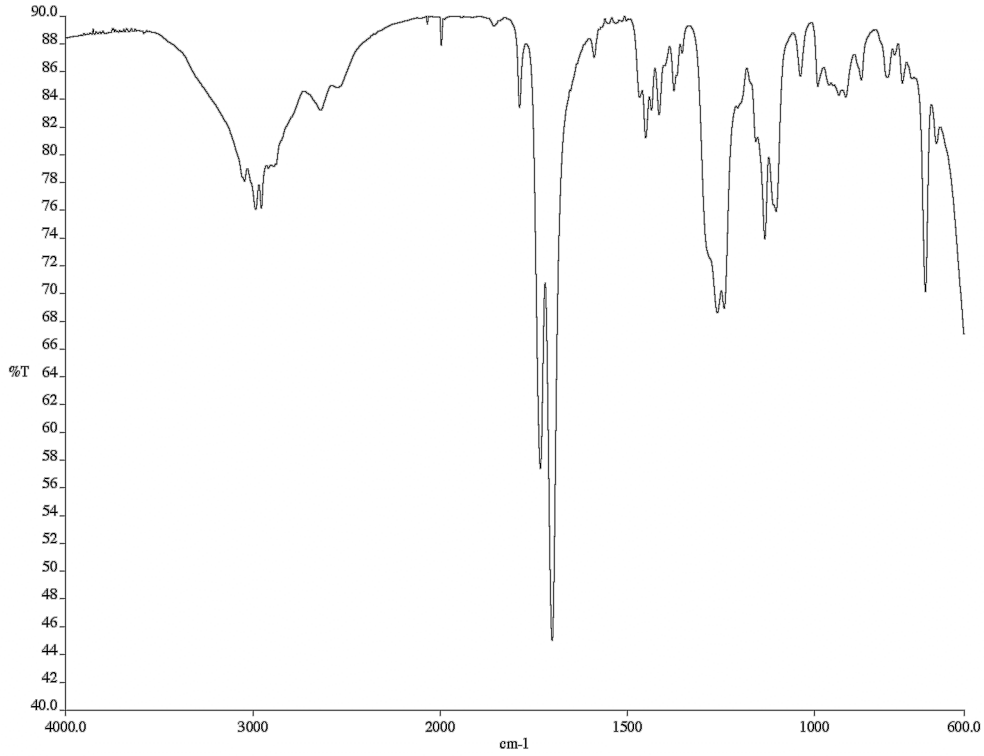

Figure B.8 Infrared spectrum (thin film/NaCl) of compound **242**.

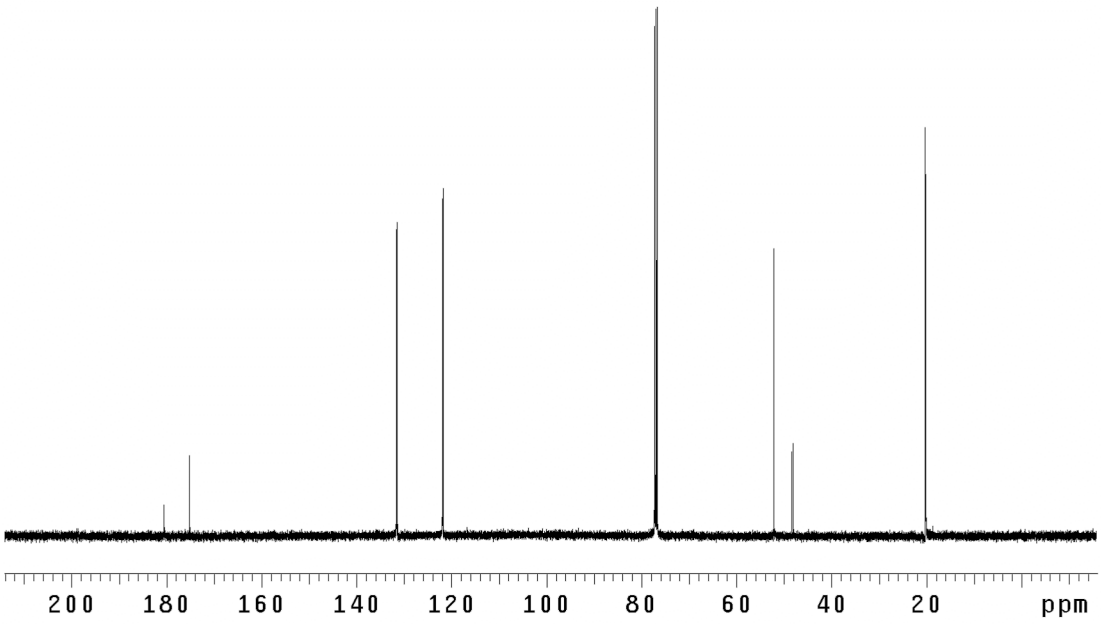

Figure B.9 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **242**.

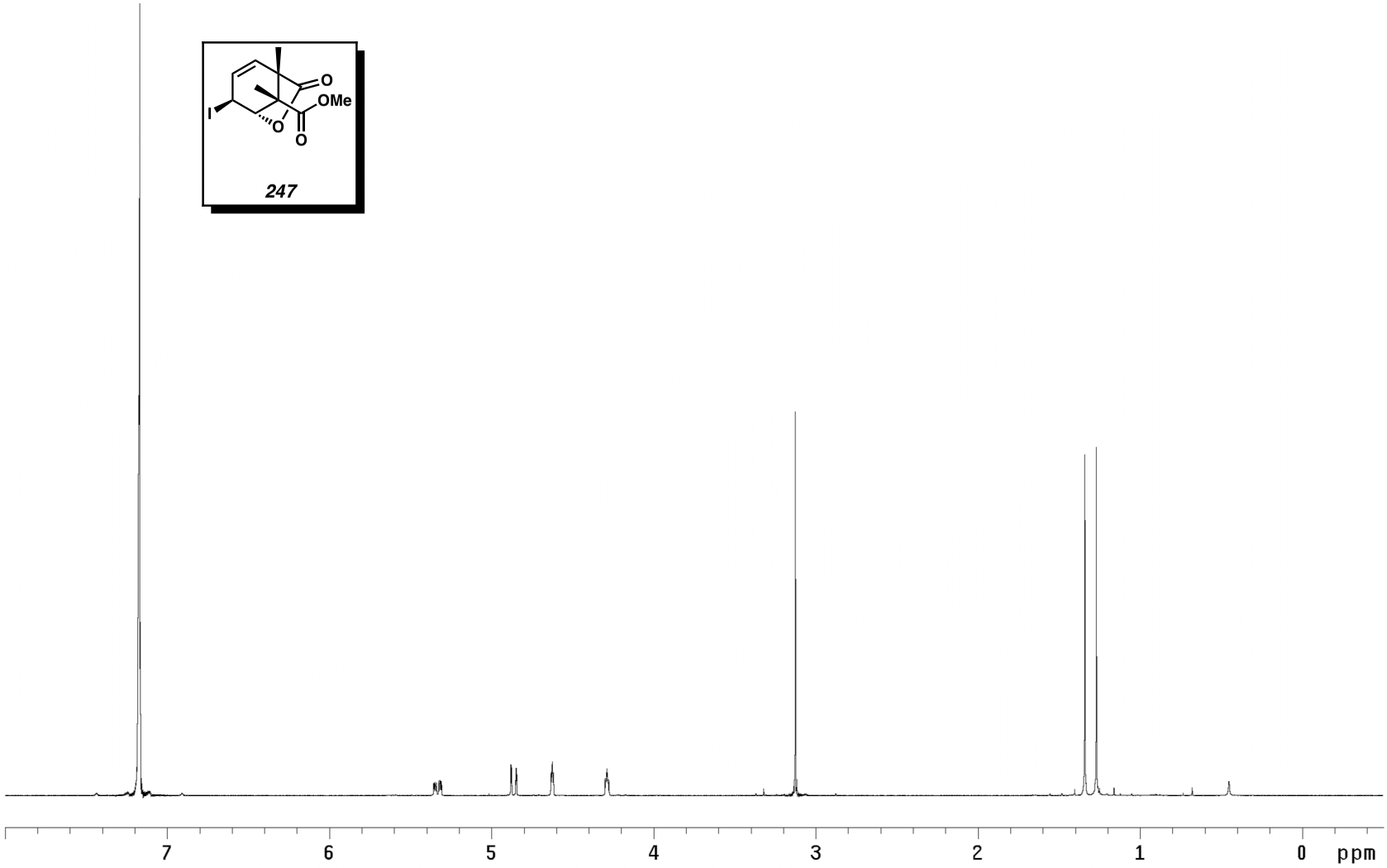

Figure B.10 ^{1}H NMR (300 MHz, C_6D_6) of compound **247**.

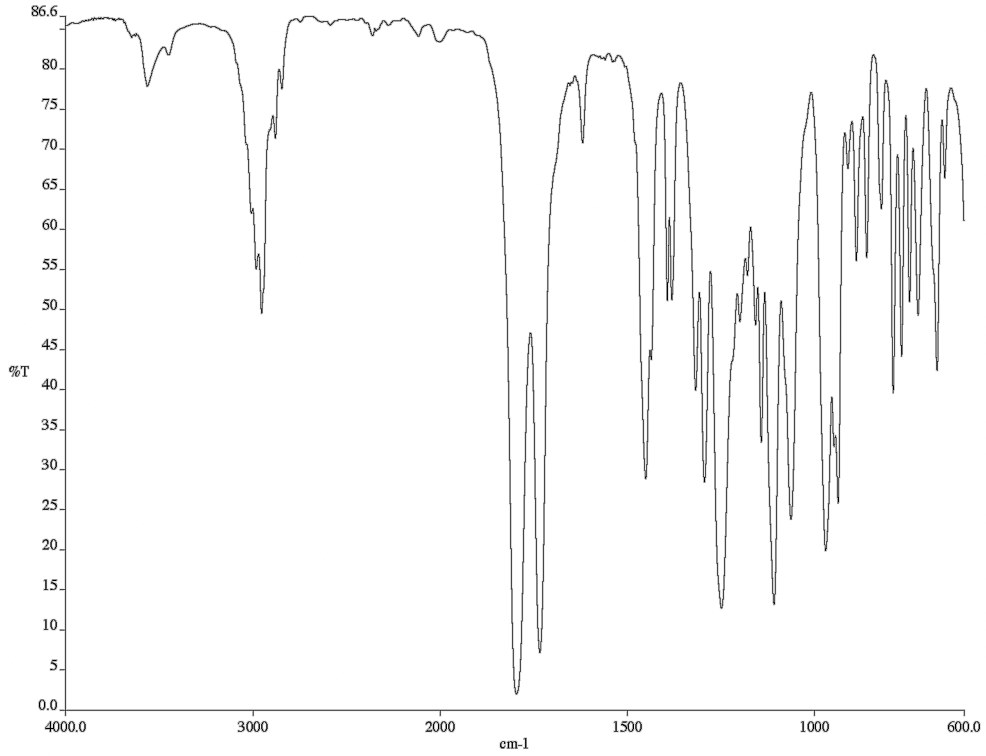

Figure B.11 Infrared spectrum (thin film/NaCl) of compound **247**.

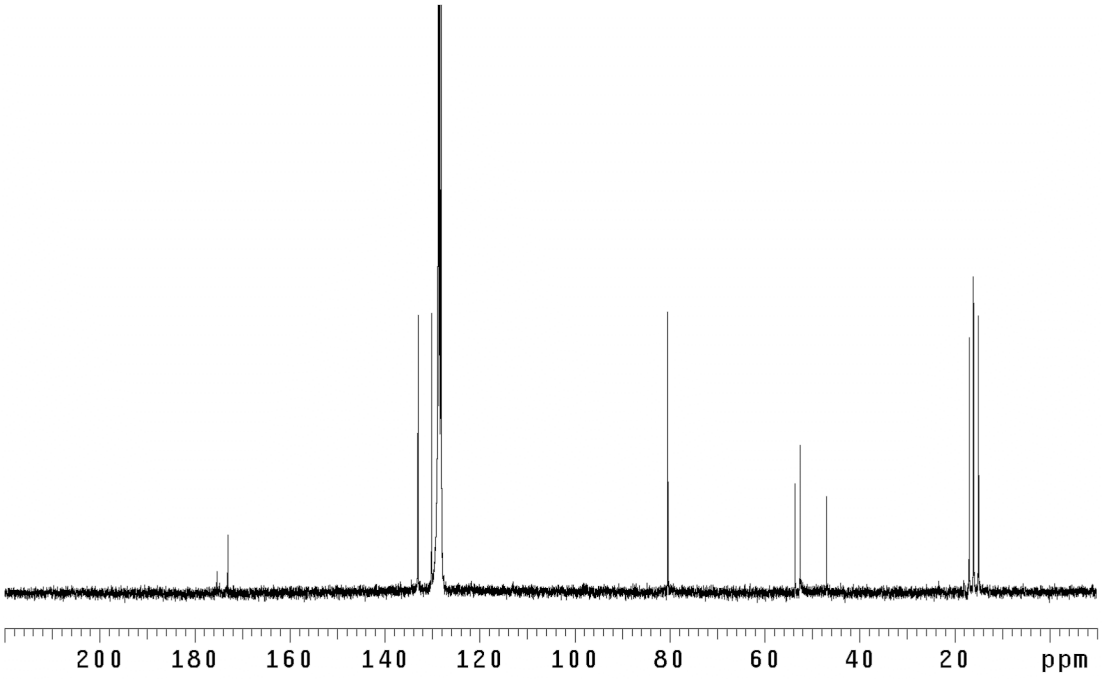

Figure B.12 ^{13}C NMR (75 MHz, C_6D_6) of compound **247**.

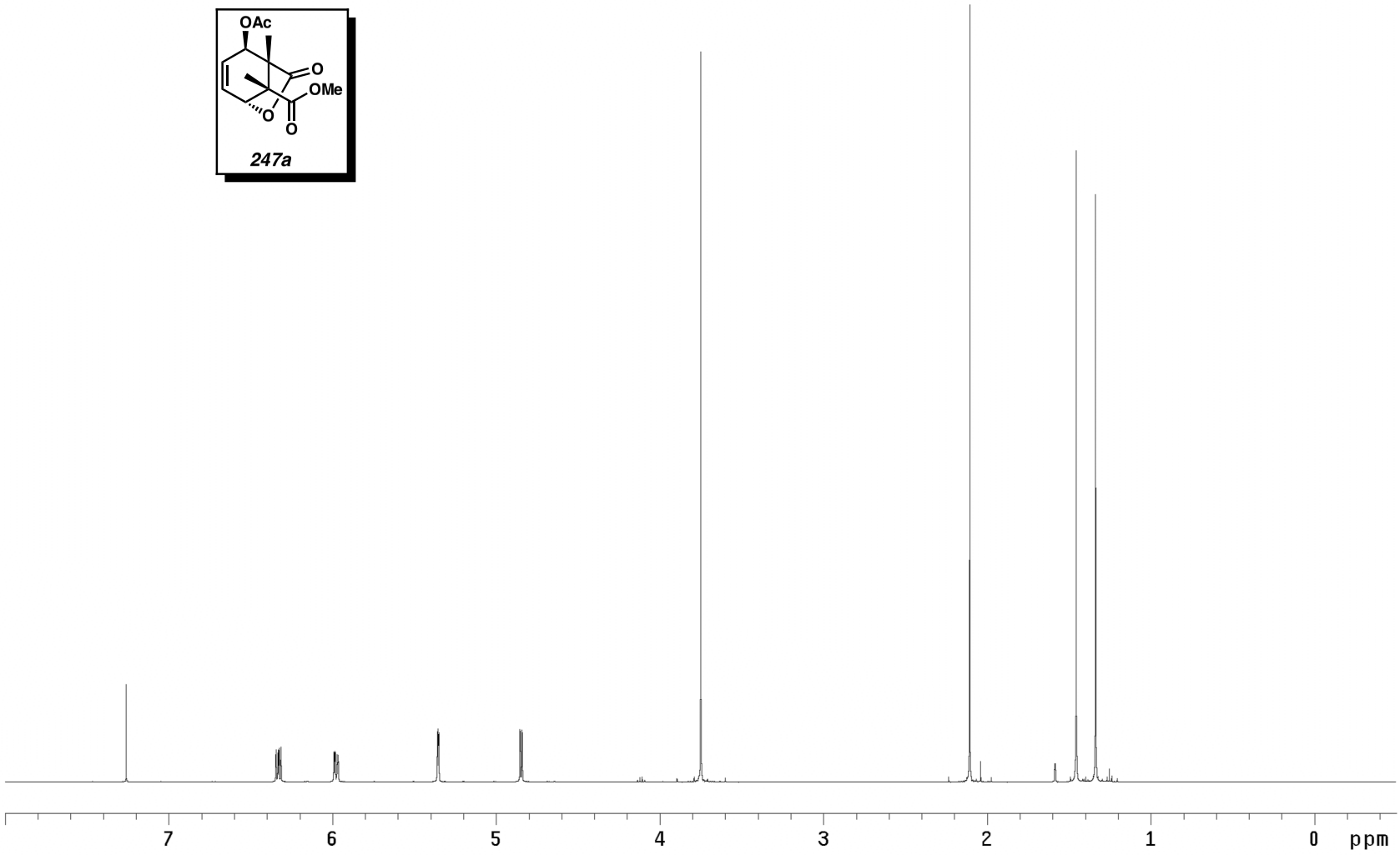

Figure B.13 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **247a.**

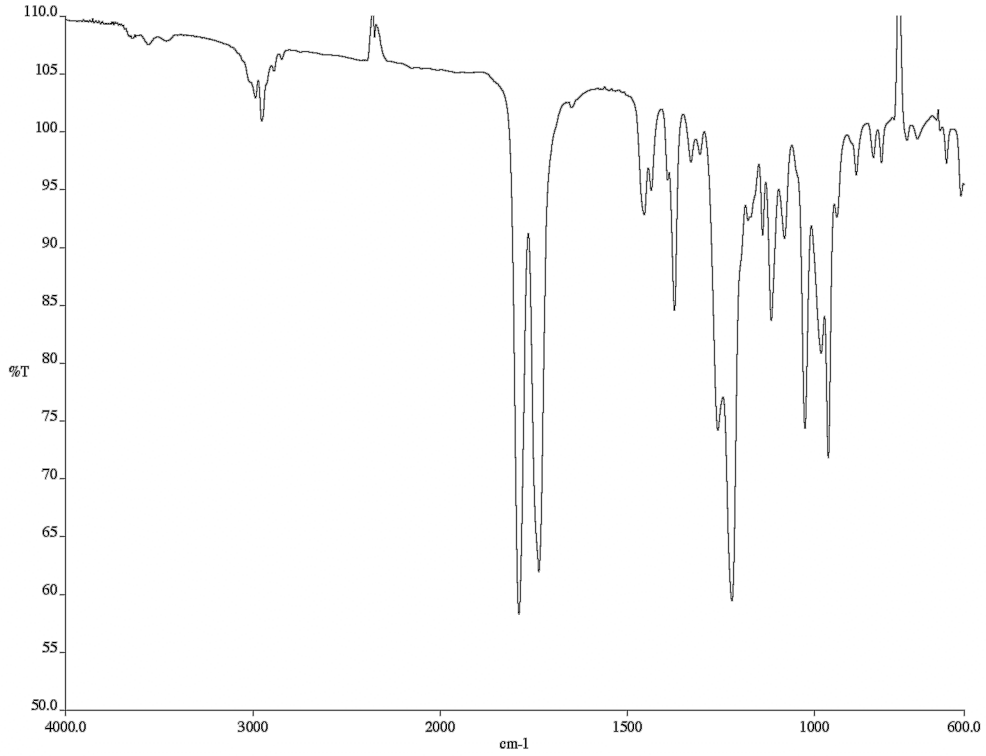

Figure B.14 Infrared spectrum (thin film/NaCl) of compound **247a**.

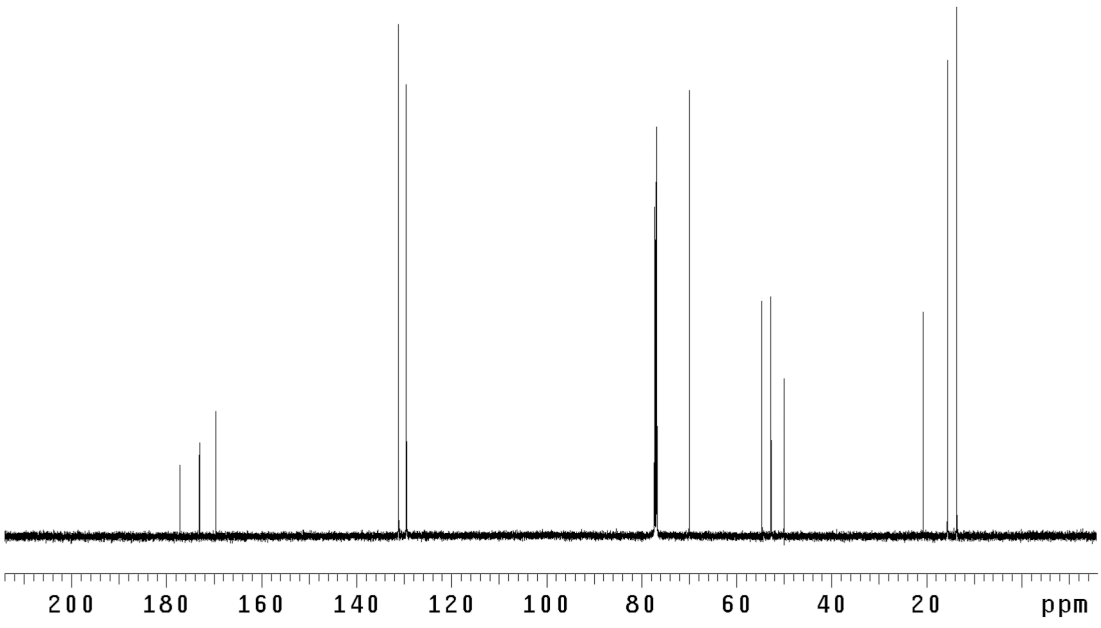

Figure B.15 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **247a**.

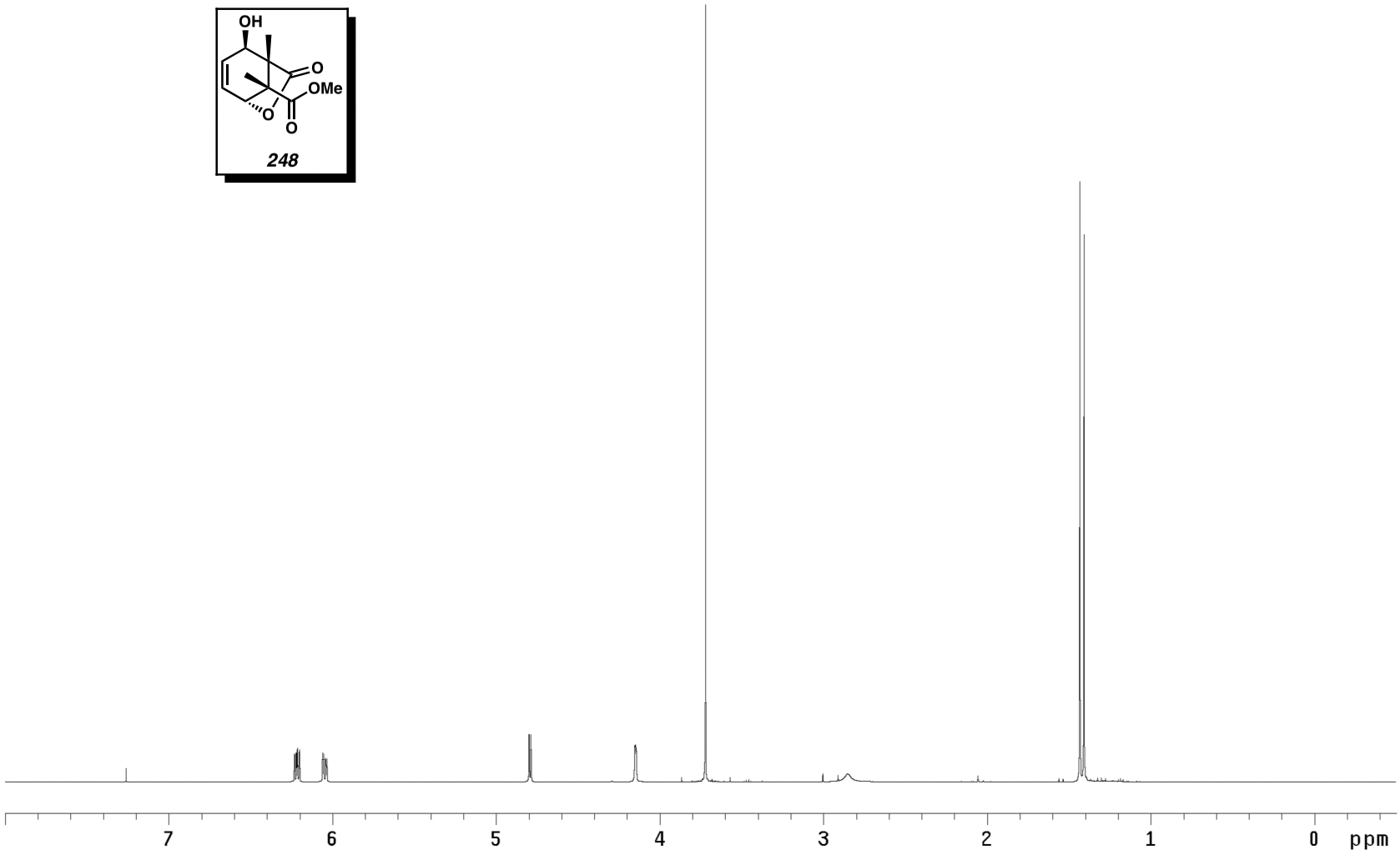

Figure B.16 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **248**.

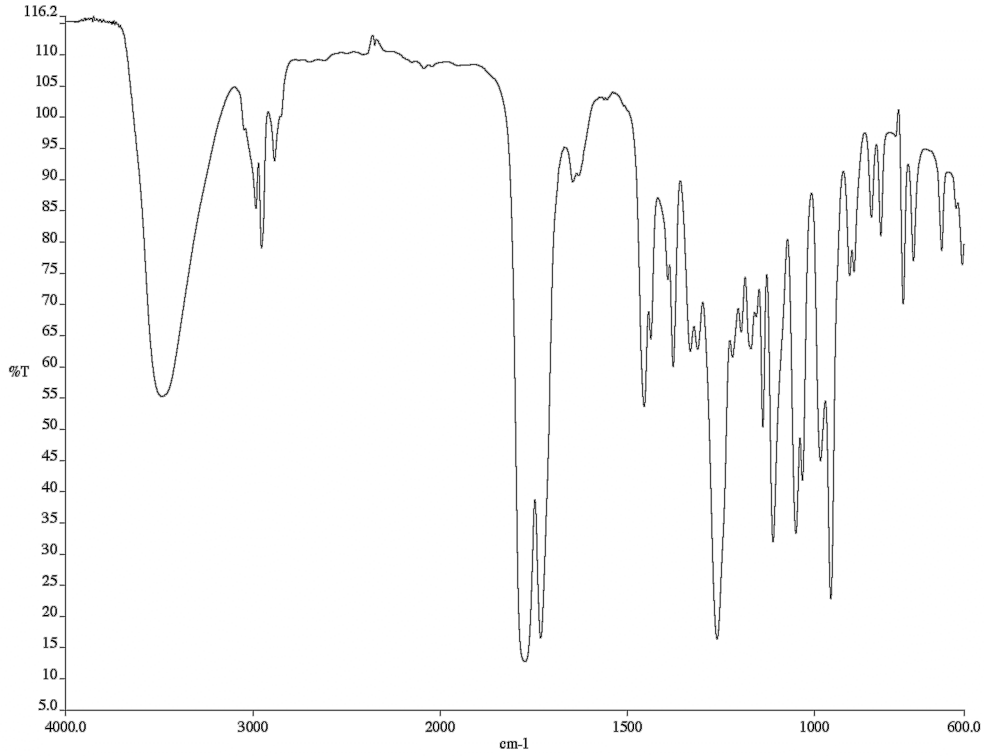

Figure B.17 Infrared spectrum (thin film/NaCl) of compound **248**.

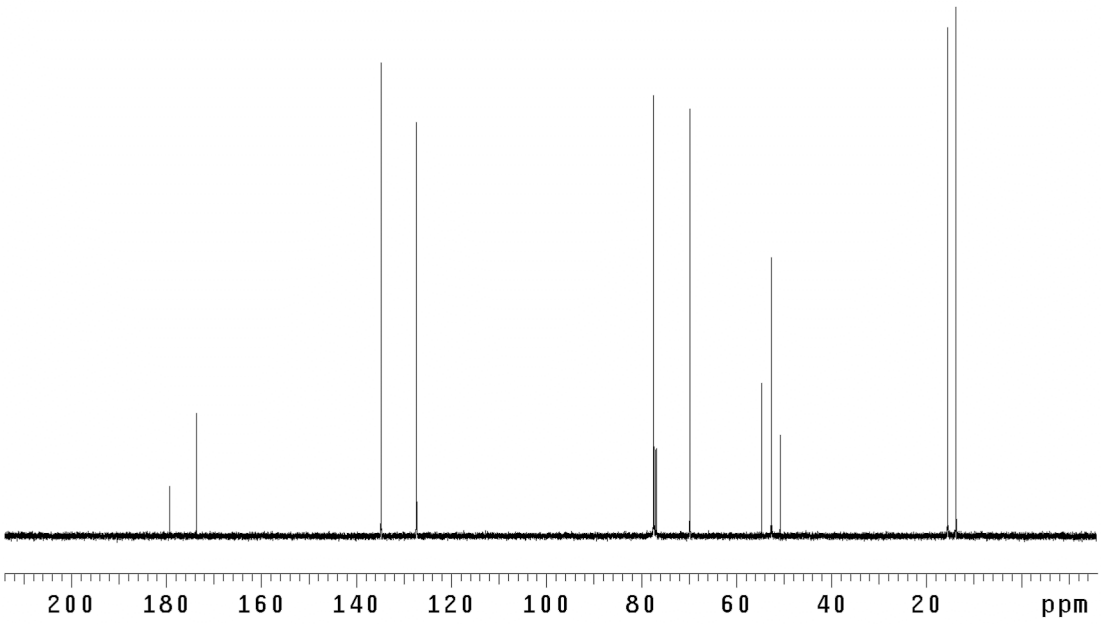

Figure B.17 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **248**.

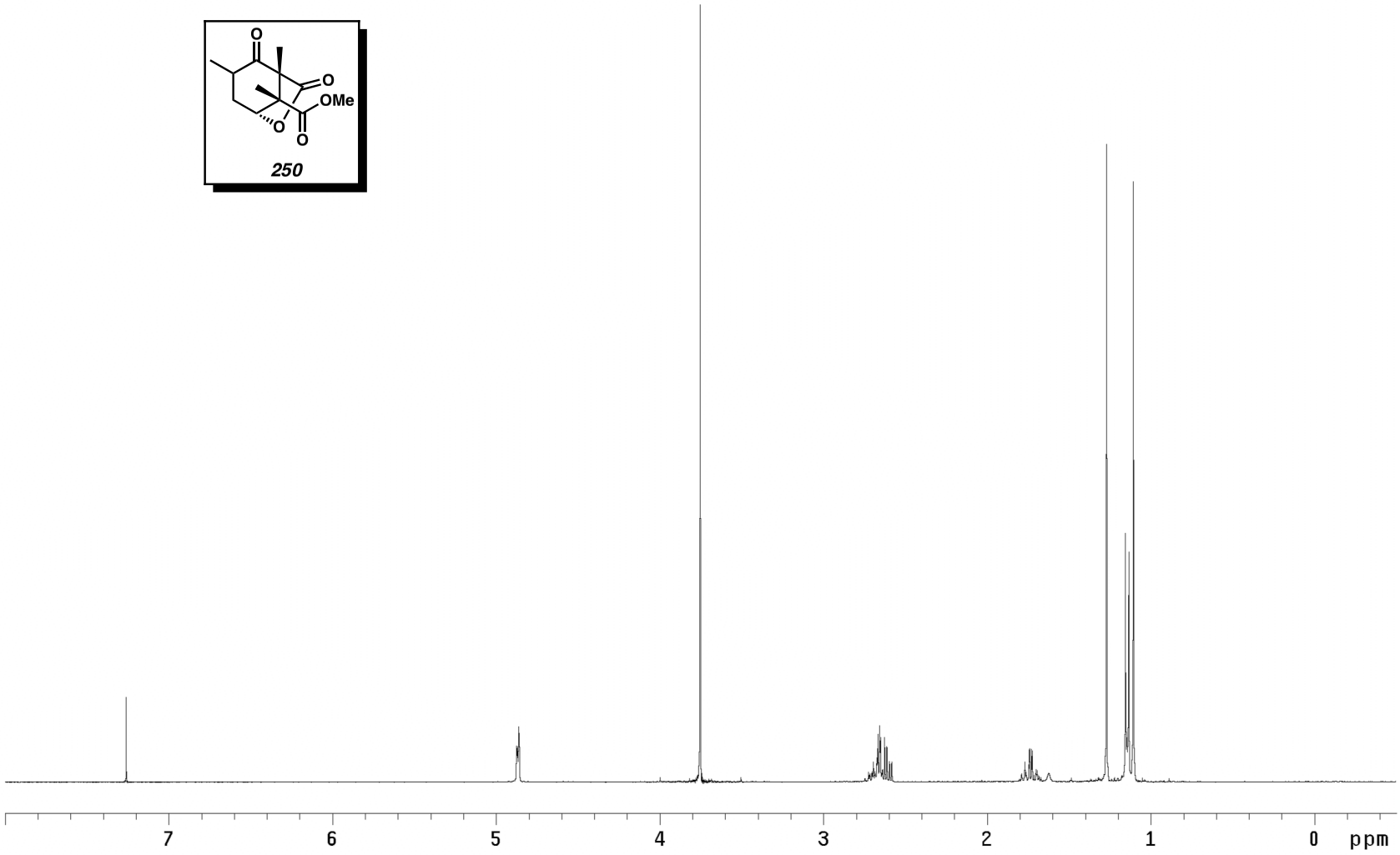

Figure B.19 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **250**.

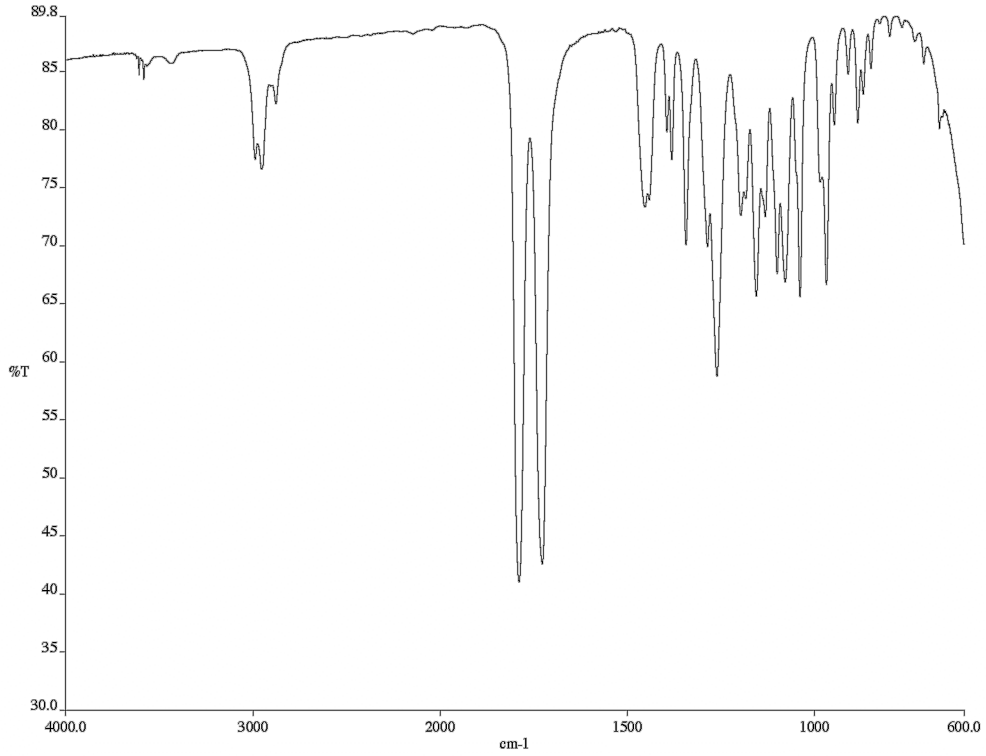

Figure B.20 Infrared spectrum (thin film/NaCl) of compound **250**.

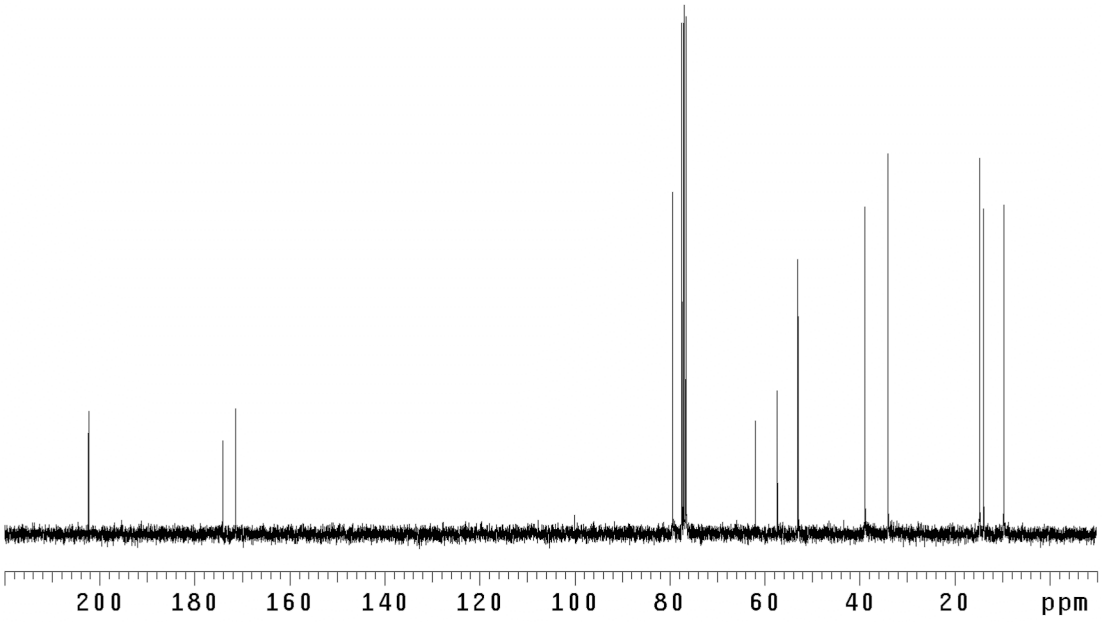

Figure B.21 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **250**.

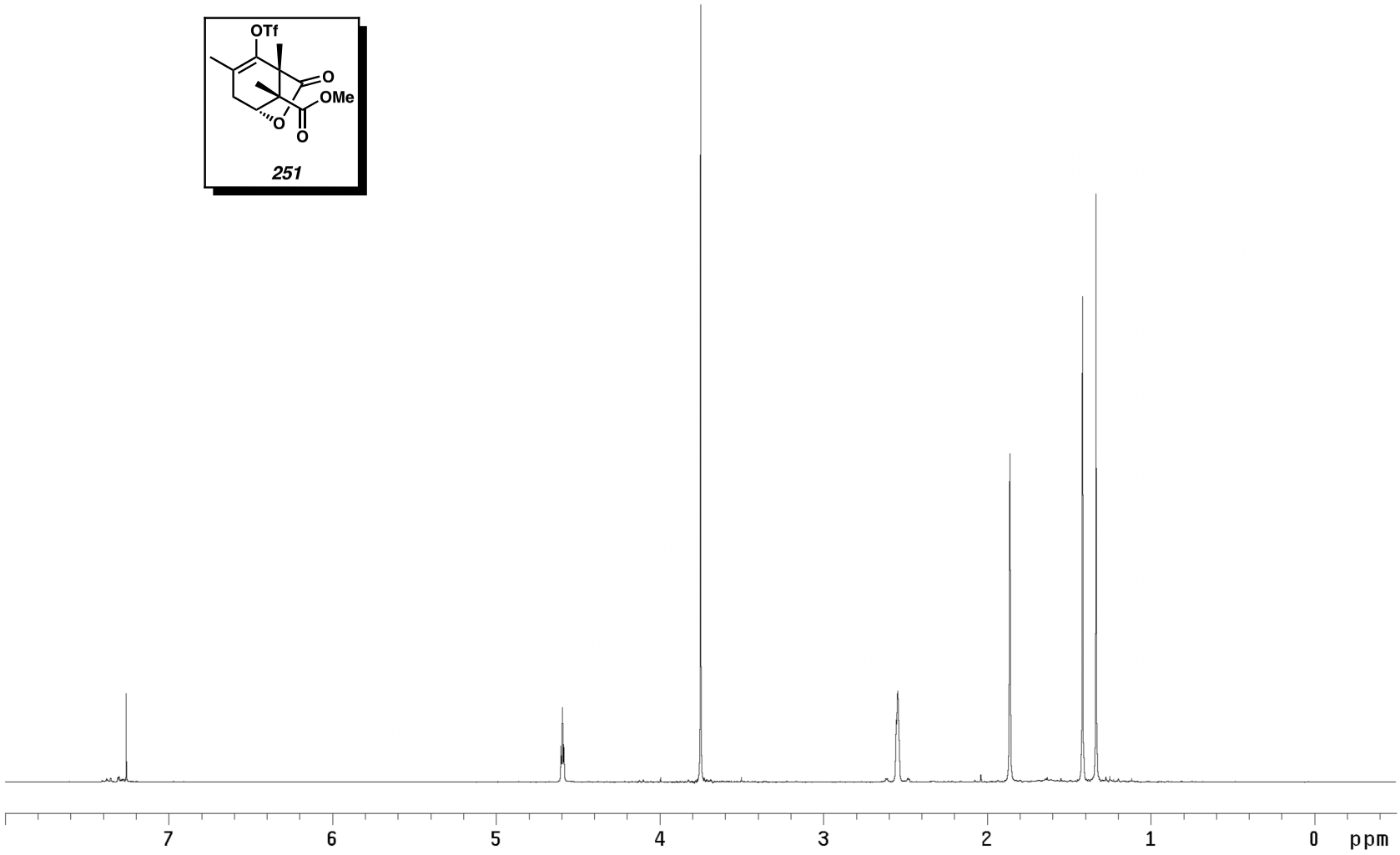

Figure B.22 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **251**.

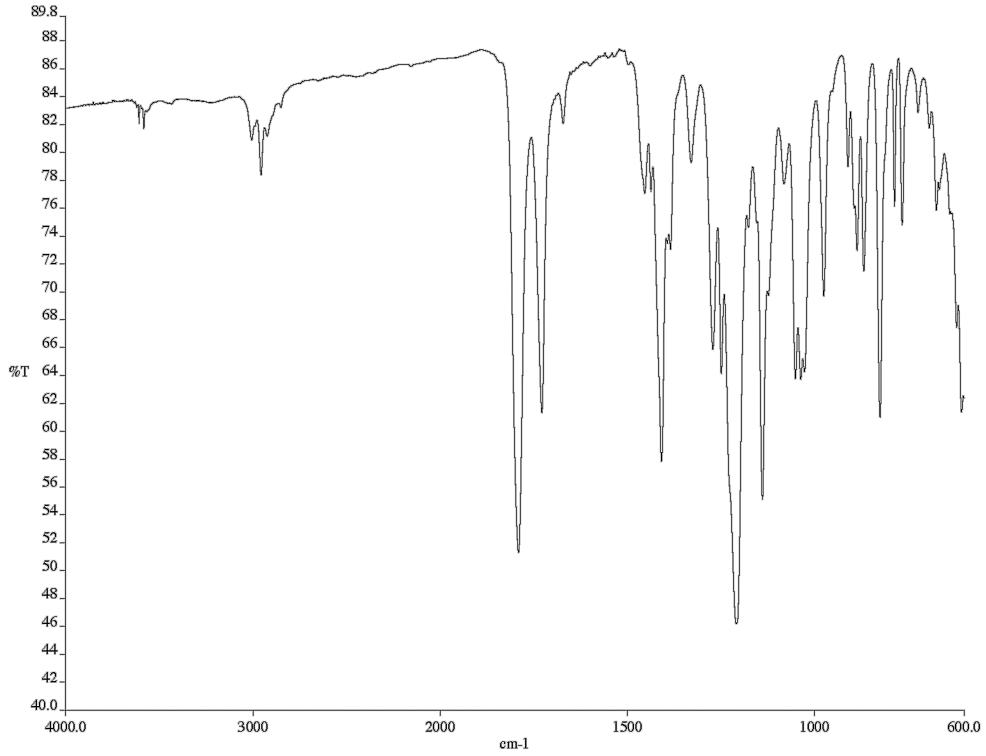

Figure B.23 Infrared spectrum (thin film/NaCl) of compound **251**.

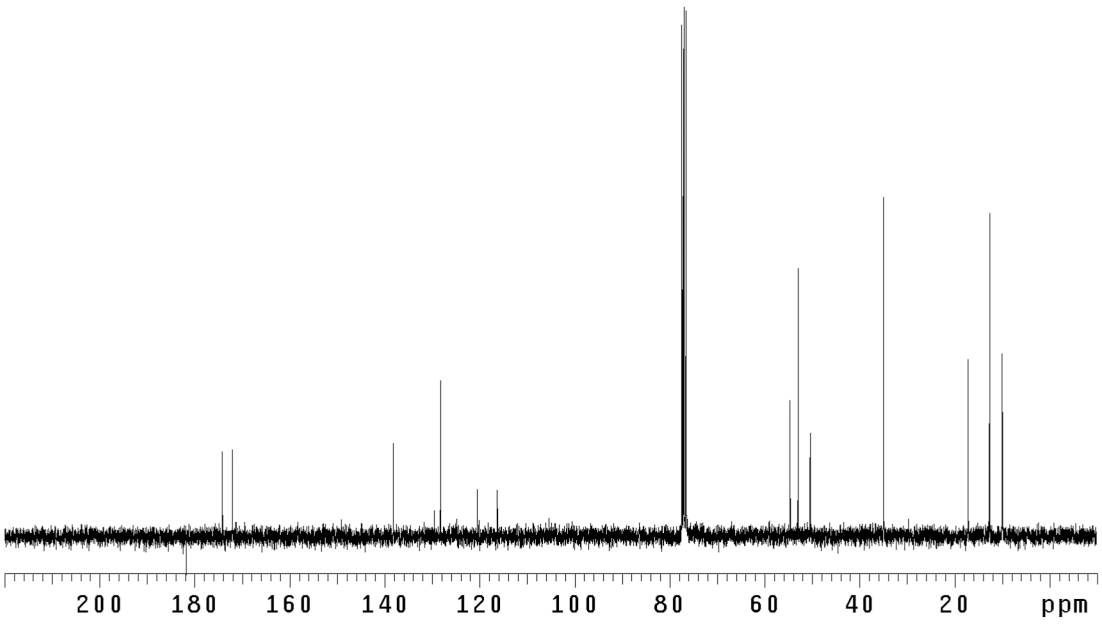

Figure B.24 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **251**.

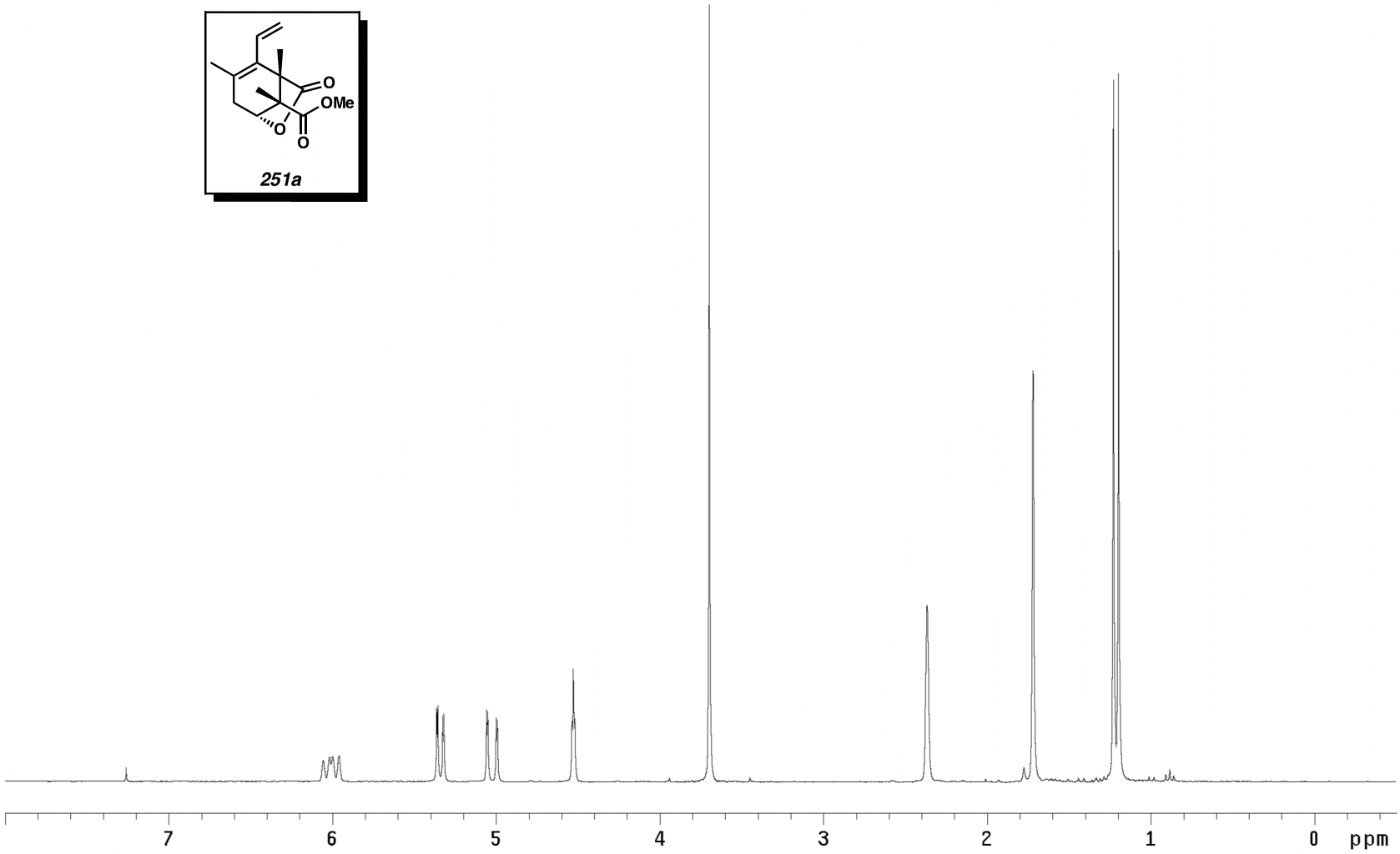

Figure B.25 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **251a**.

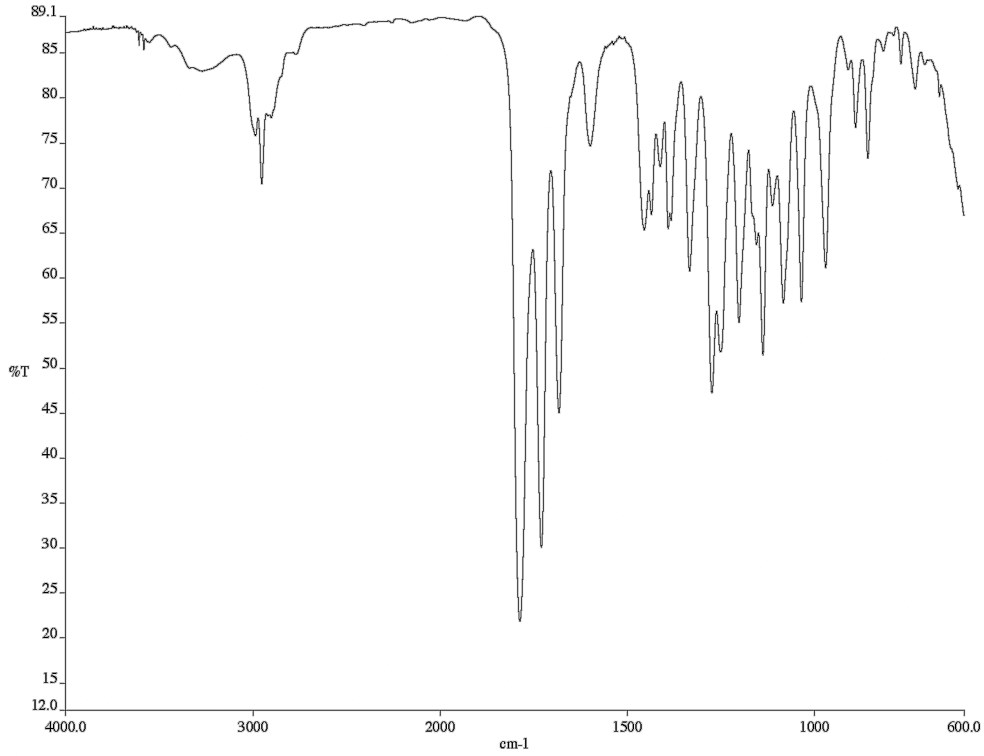

Figure B.28 Infrared spectrum (thin film/NaCl) of compound **252**.

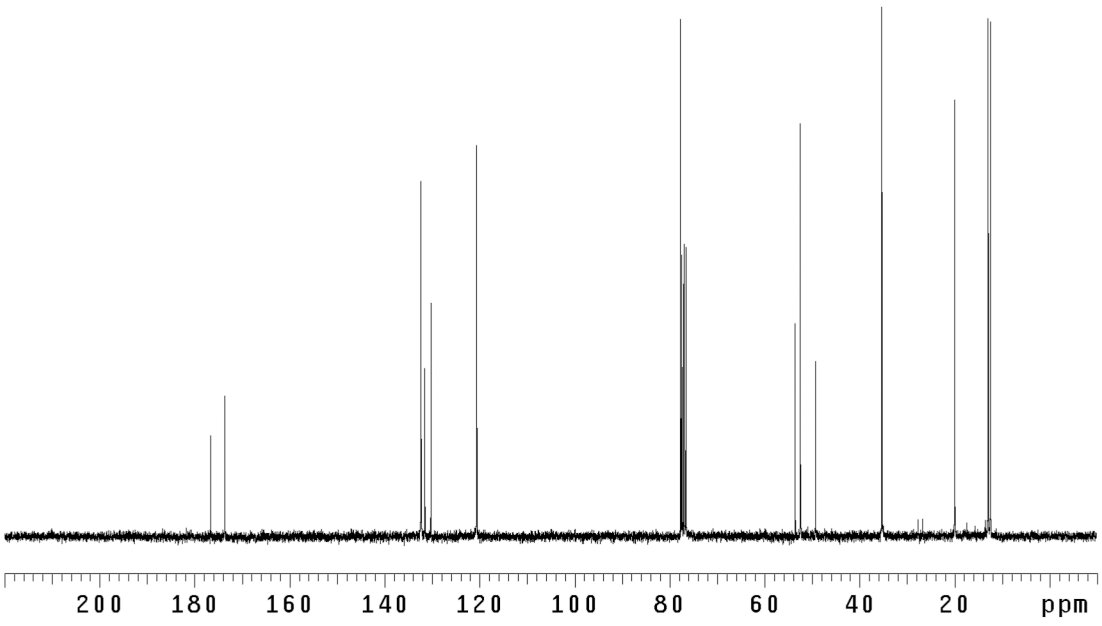

Figure B.29 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **252**.

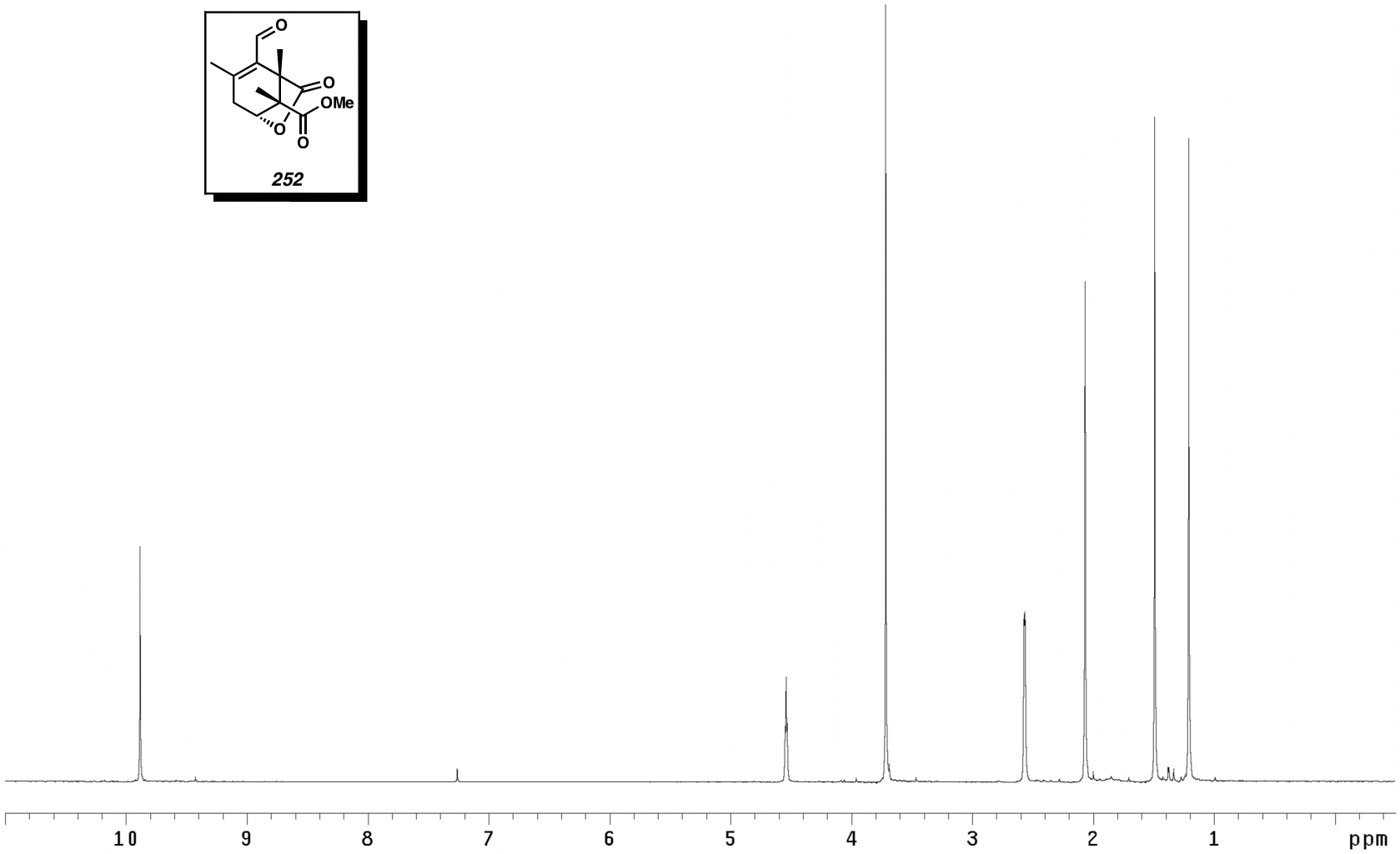

Figure B.28 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **252**.

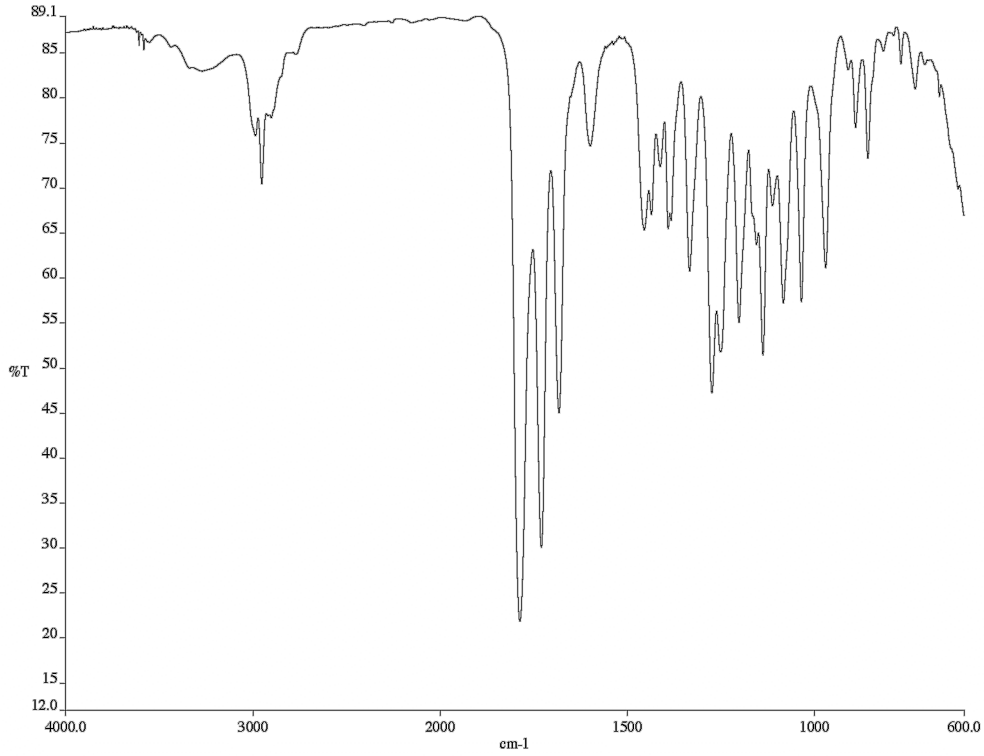

Figure B.29 Infrared spectrum (thin film/NaCl) of compound **252**.

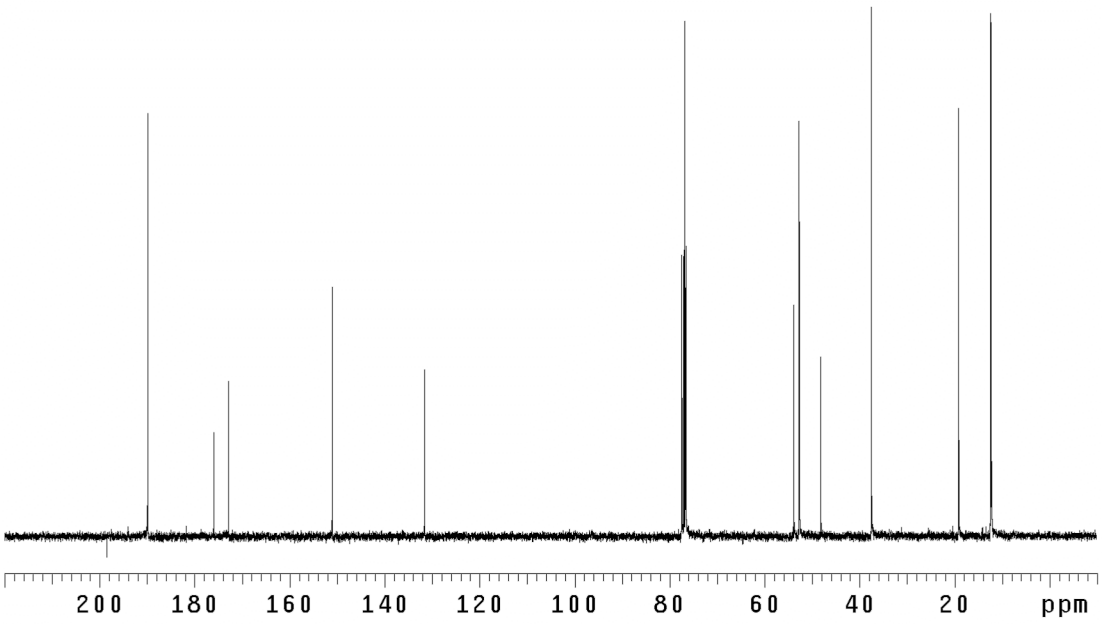

Figure B.30 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **252**.

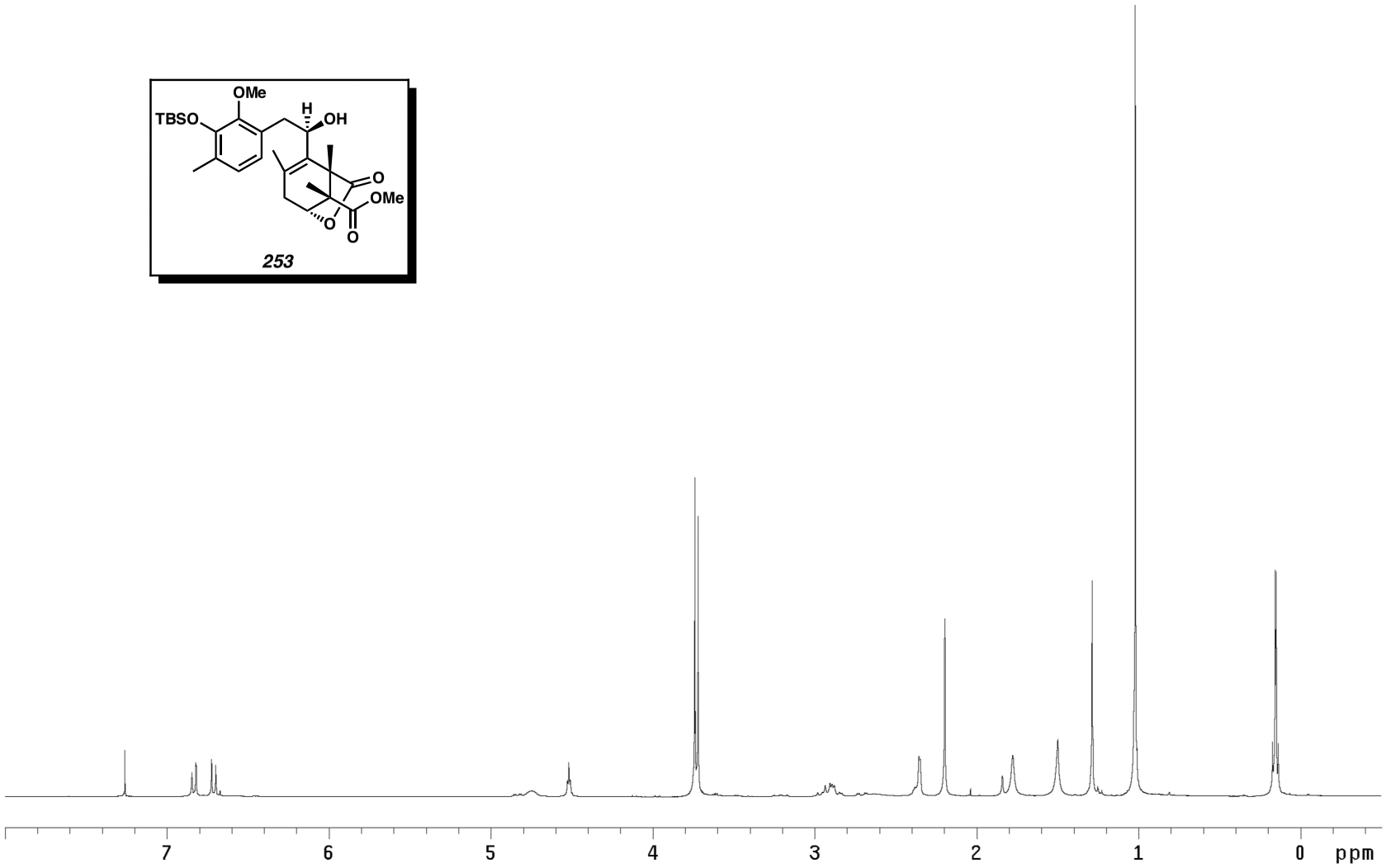

Figure B.31 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **253**.

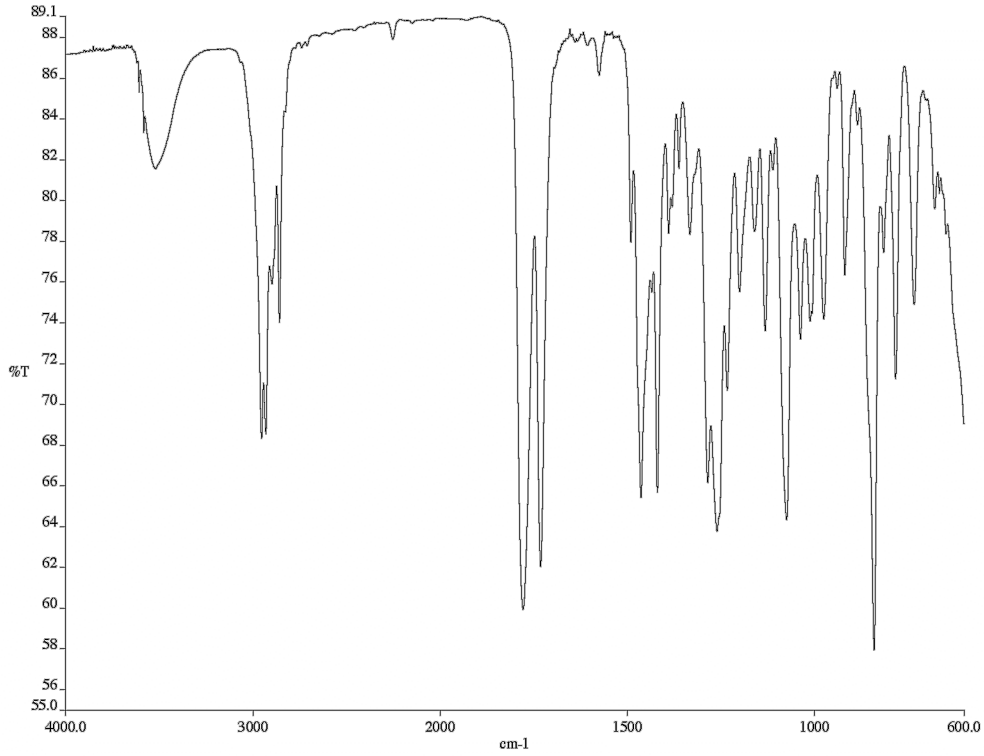

Figure B.32 Infrared spectrum (thin film/NaCl) of compound **253**.

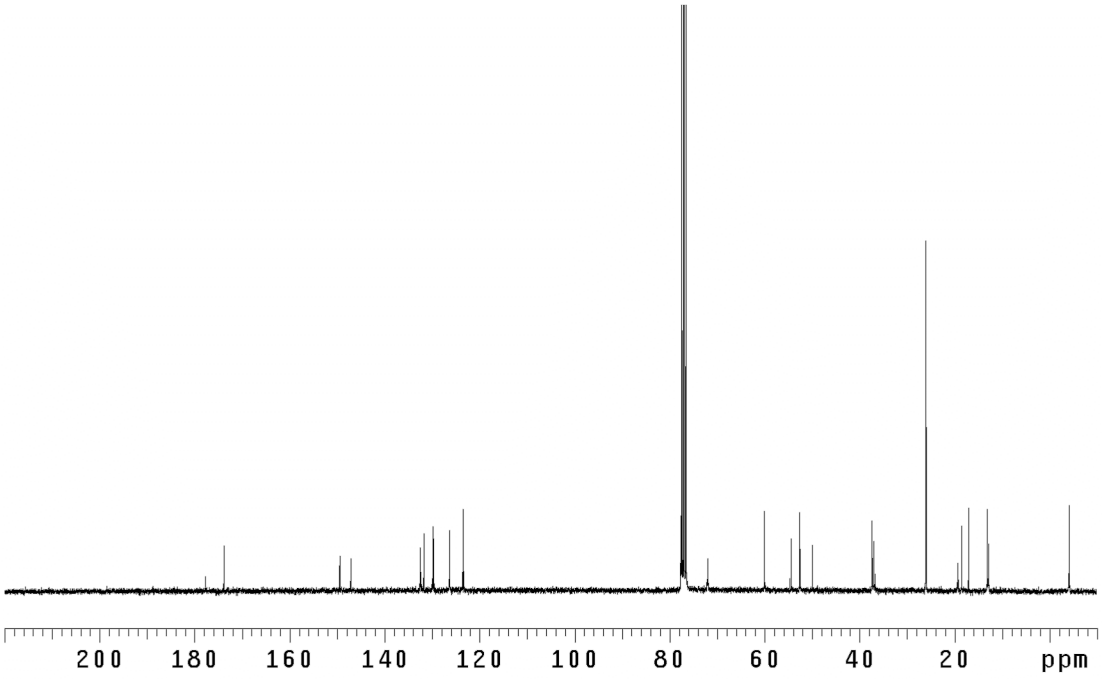

Figure B.33 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **253**.

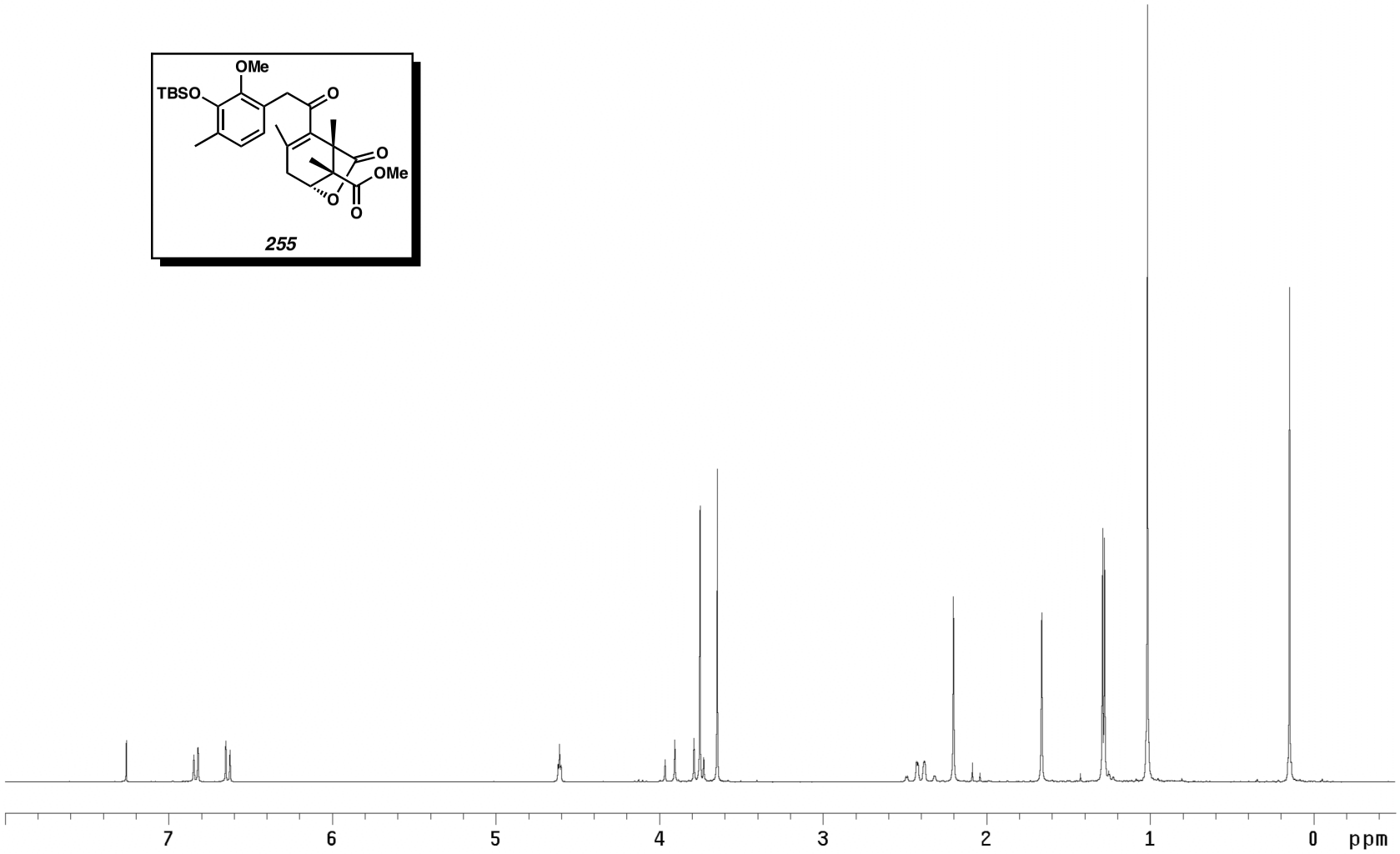

Figure B.34 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **255**.

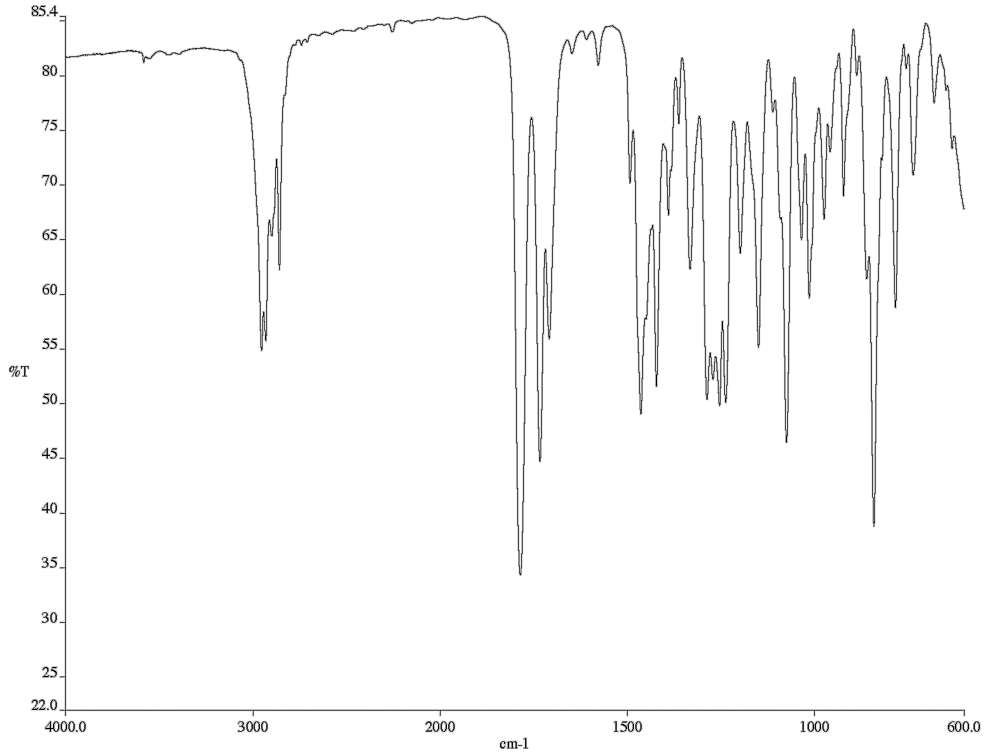

Figure B.35 Infrared spectrum (thin film/NaCl) of compound **255**.

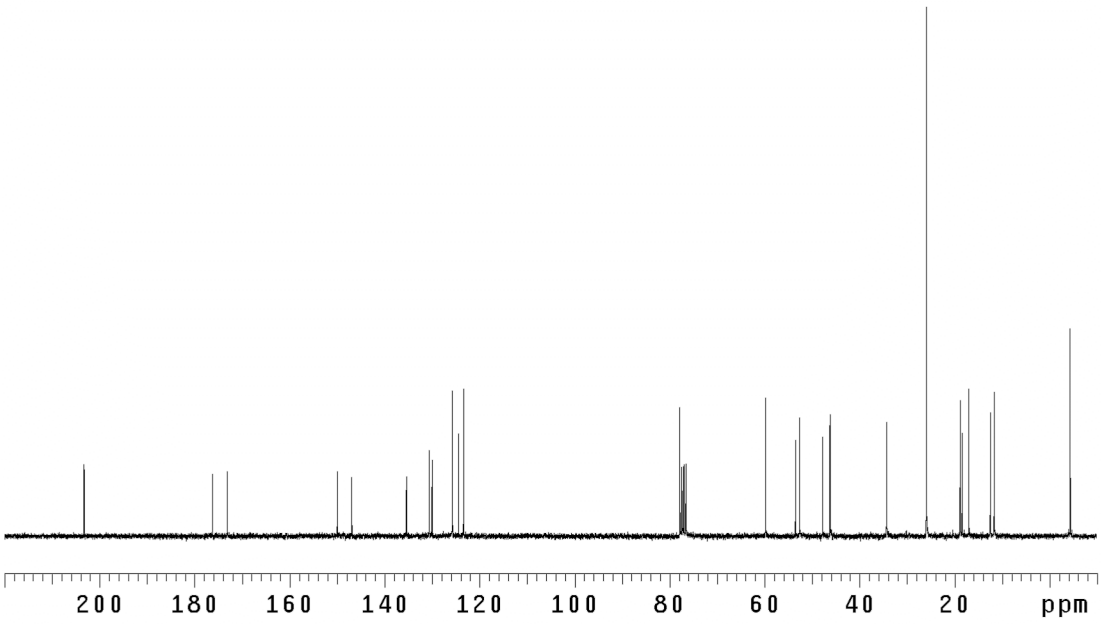

Figure B.36 ^{13}C NMR (75 MHz, 255) of compound **255**.

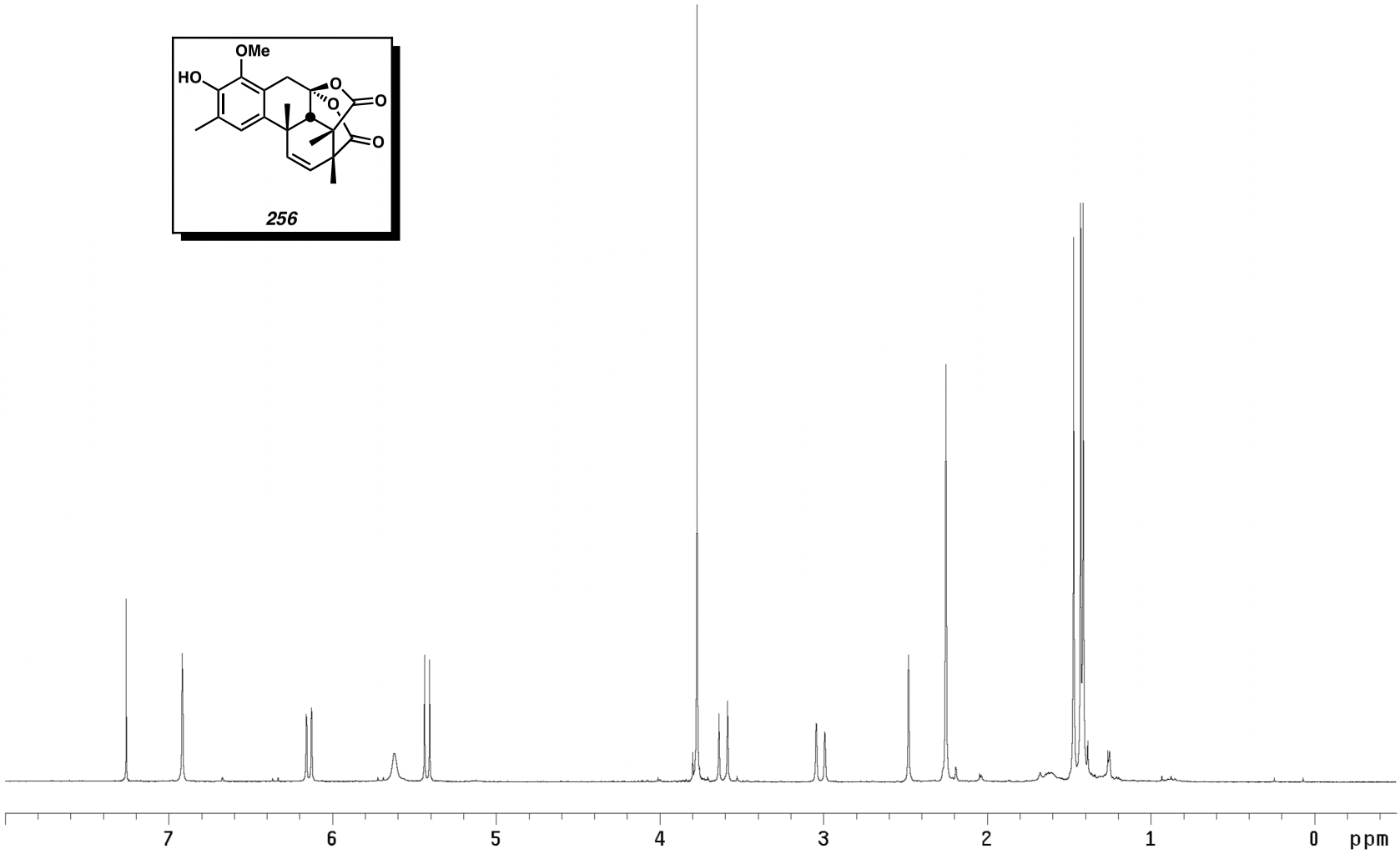

Figure B.37 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **256**.

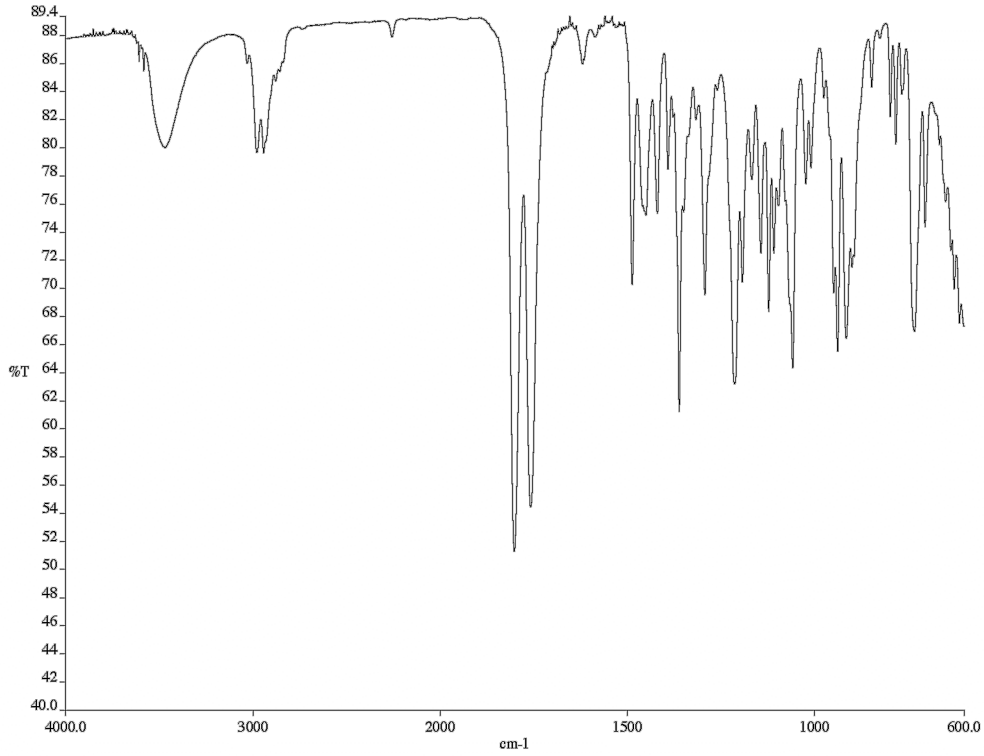

Figure B.38 Infrared spectrum (thin film/NaCl) of compound **256**.

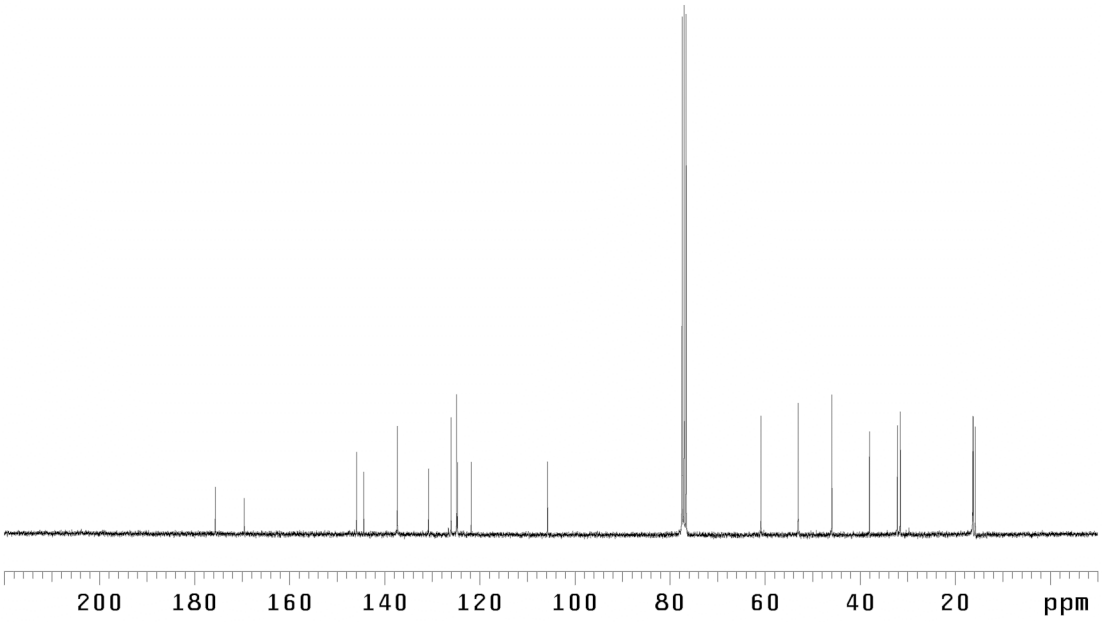

Figure B.39 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **256**.

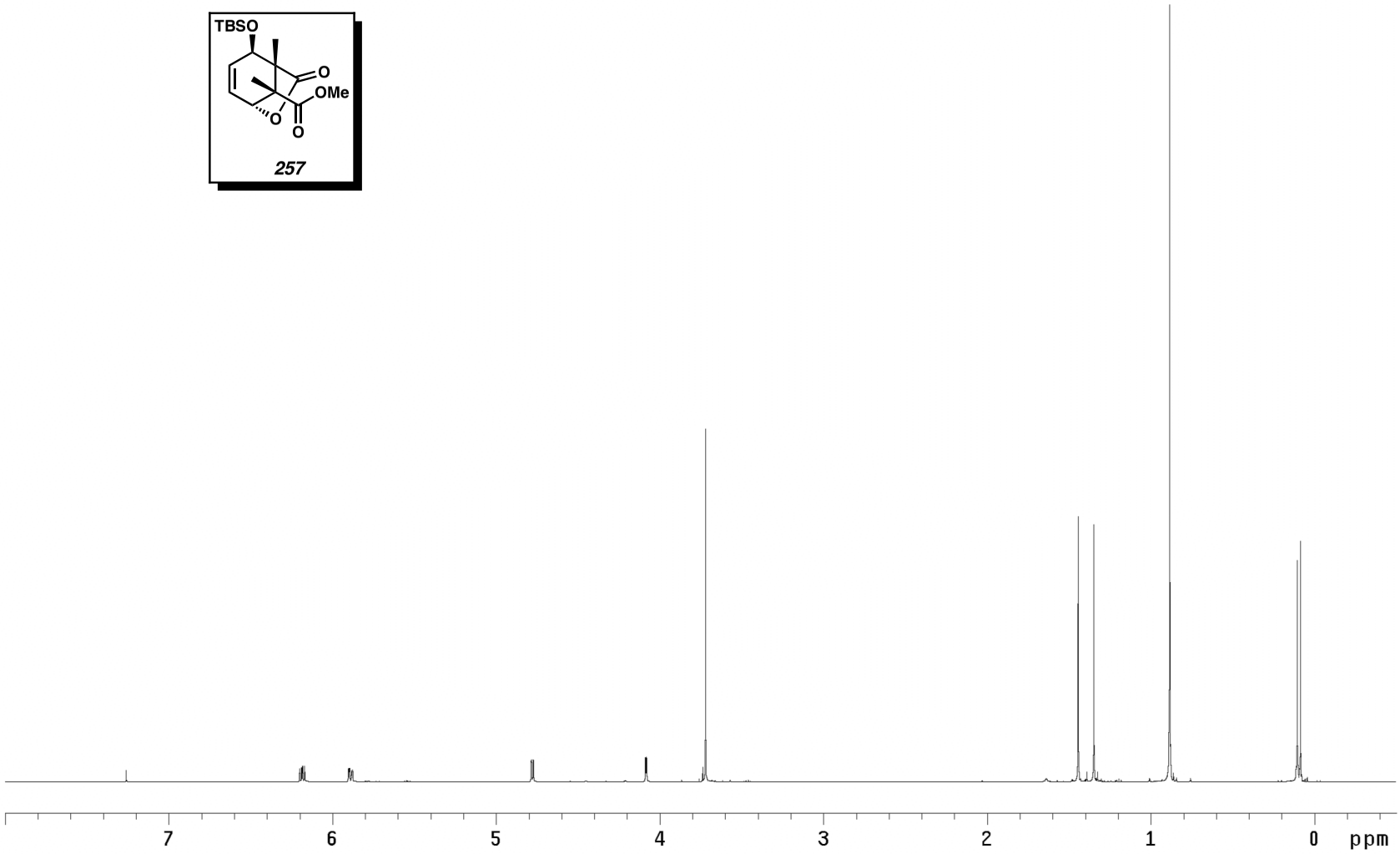

Figure B.40 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **257**.

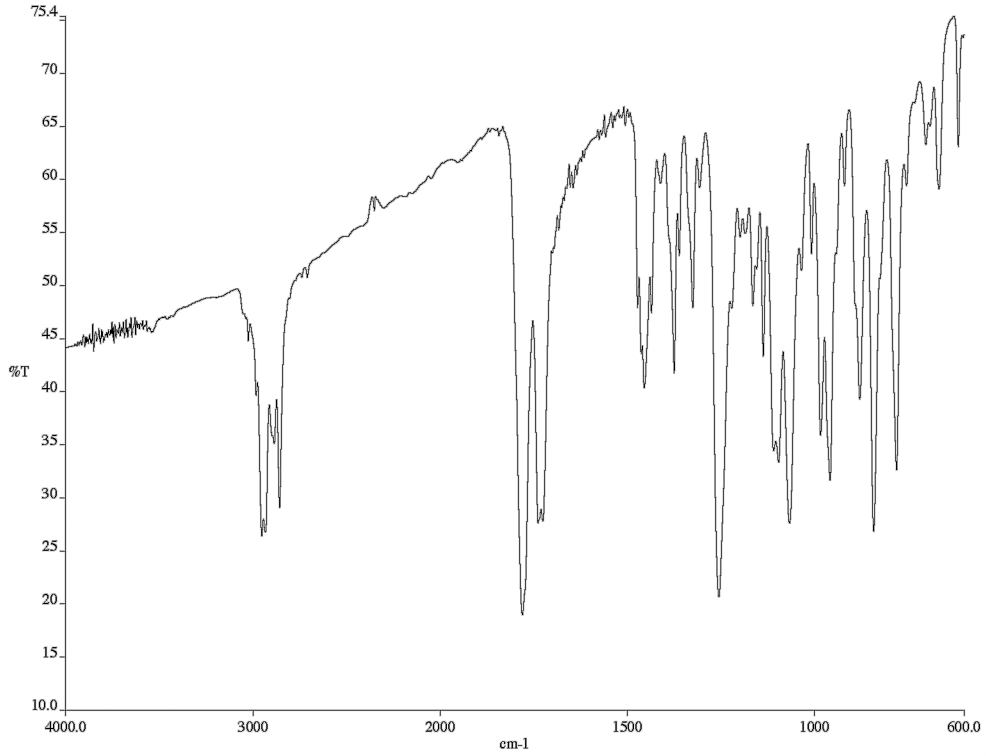

Figure B.41 Infrared spectrum (thin film/NaCl) of compound **257**.

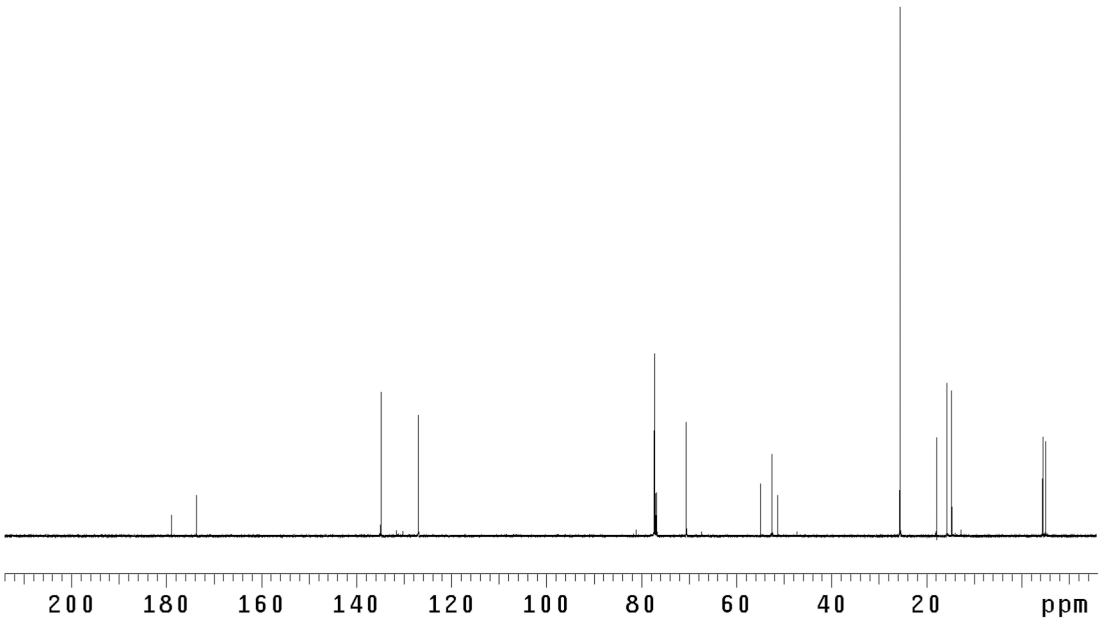

Figure B.42 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **257**.

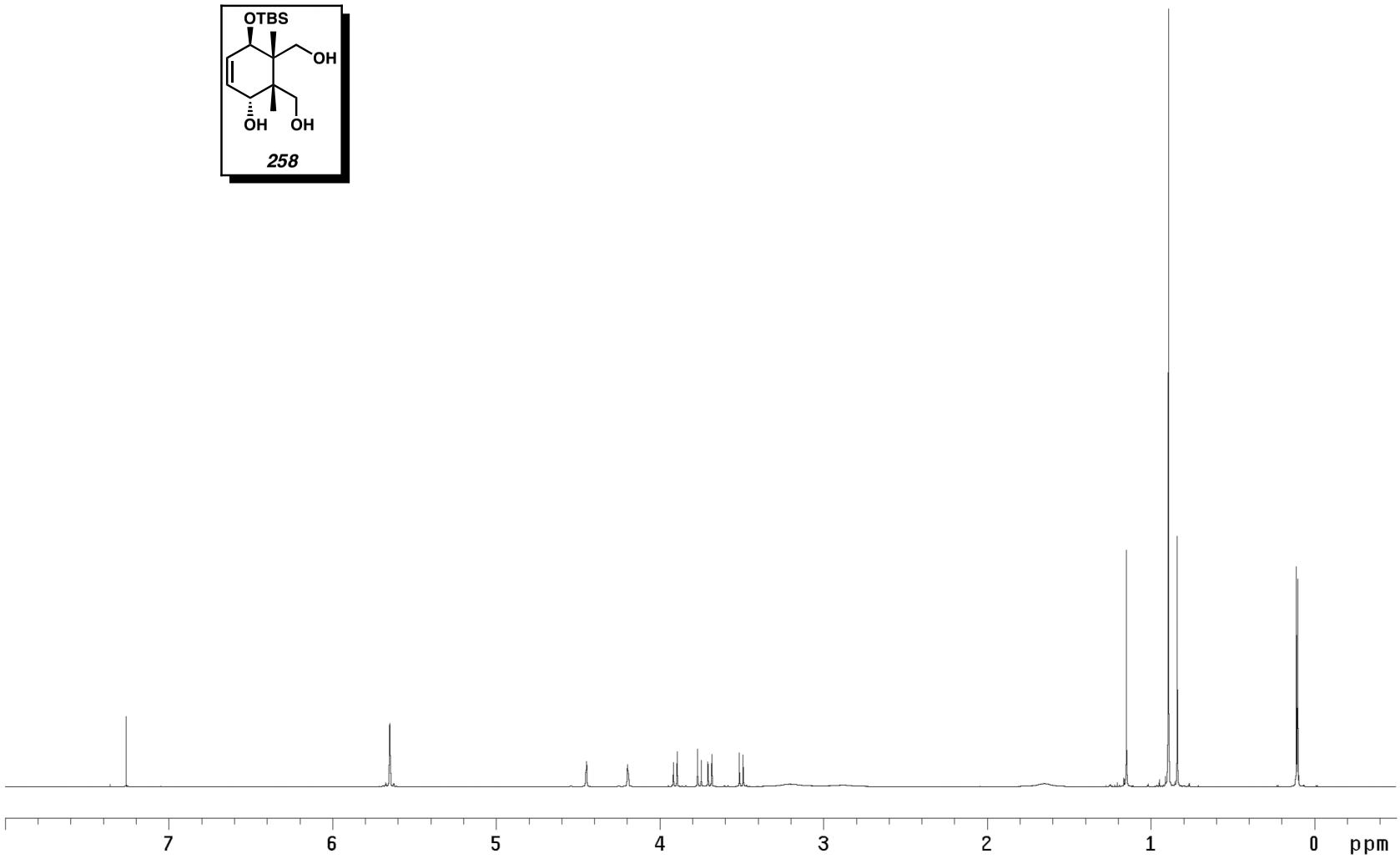

Figure B.43 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **258**.

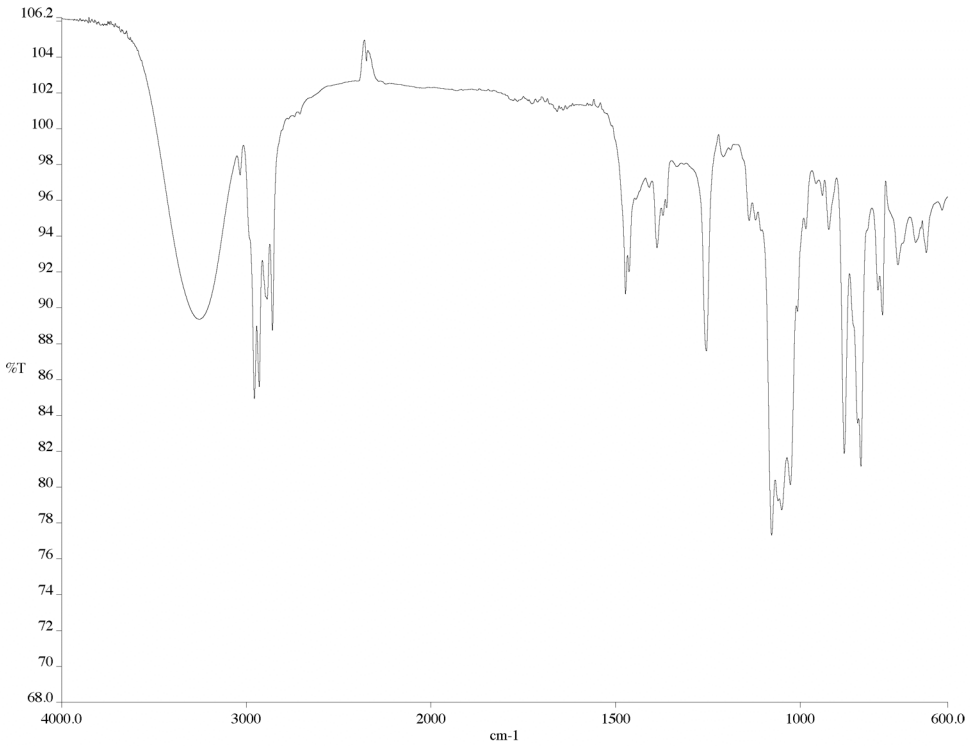

Figure B.44 Infrared spectrum (thin film/NaCl) of compound **258**.

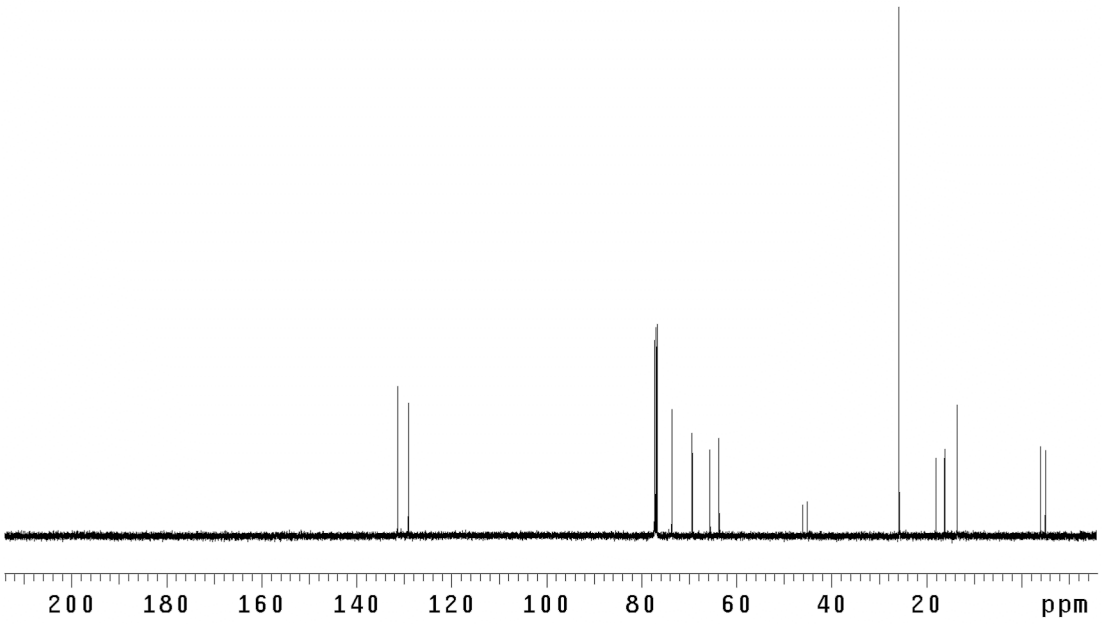

Figure B.45 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **258**.

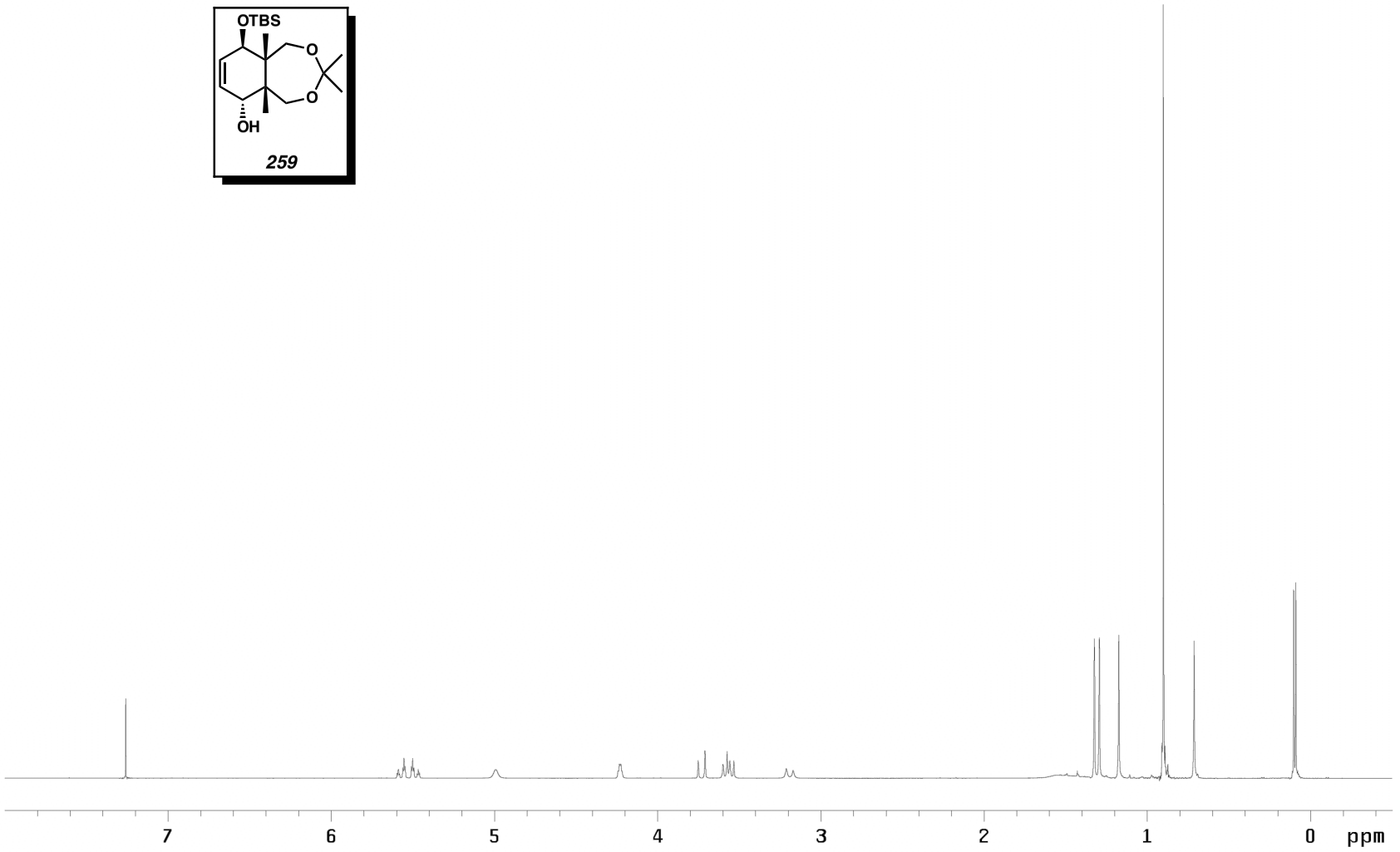

Figure B.46 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **259**.

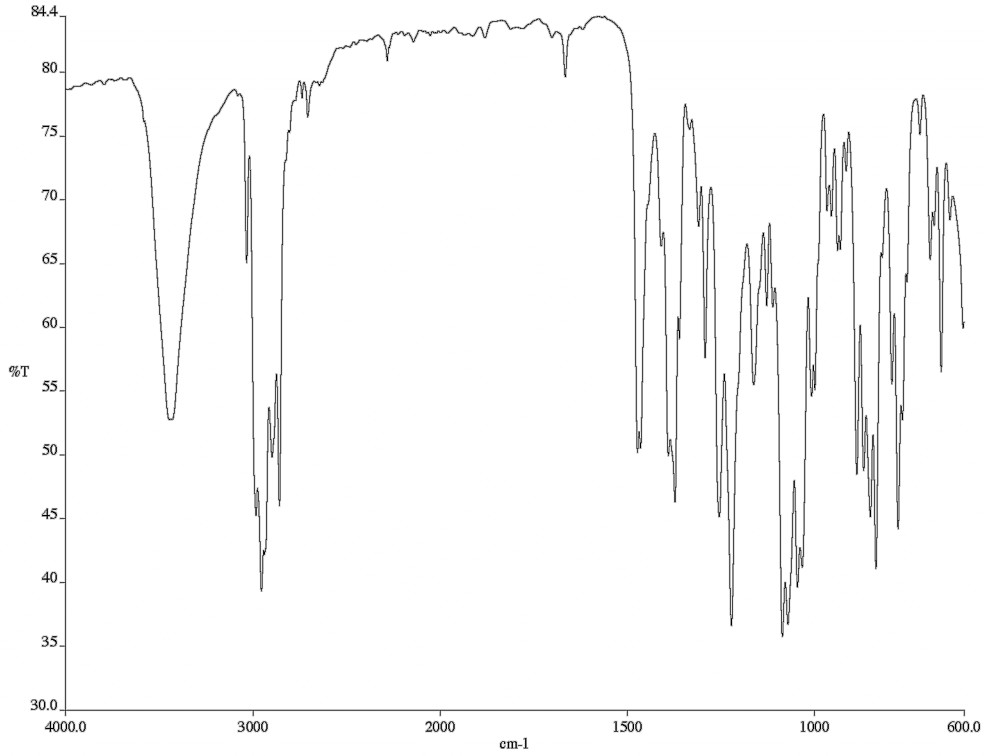

Figure B.47 Infrared spectrum (thin film/NaCl) of compound **259**.

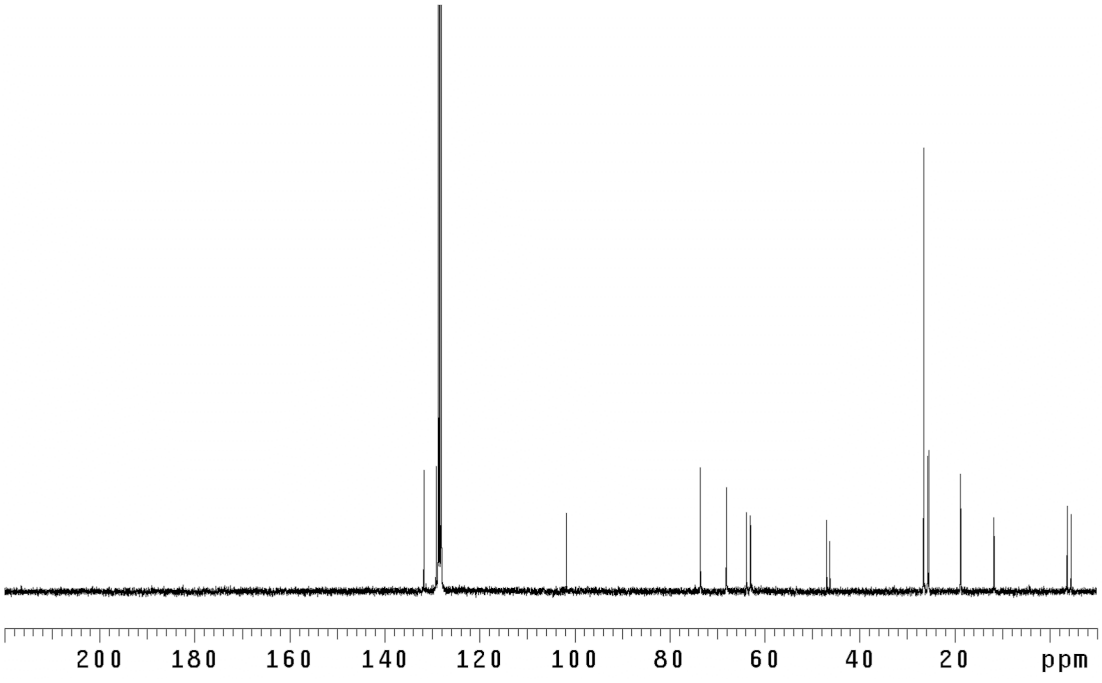

Figure B.48 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **259**.

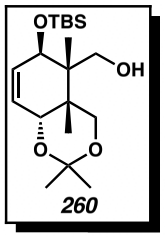

Figure B.49 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **260**.

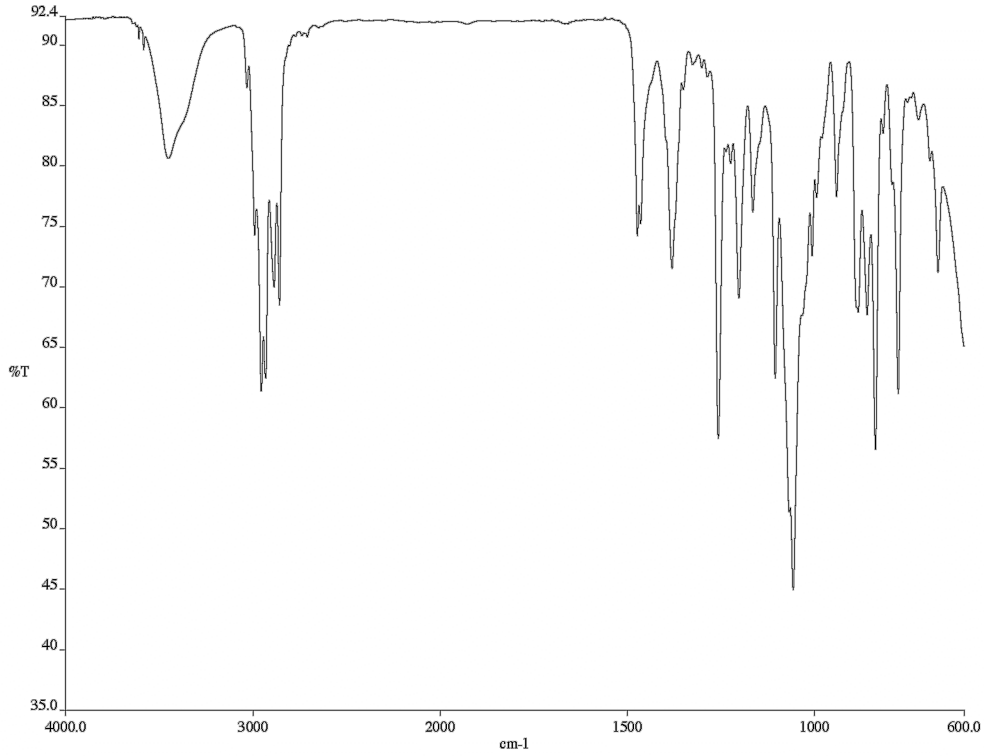

Figure B.50 Infrared spectrum (thin film/NaCl) of compound **260**.

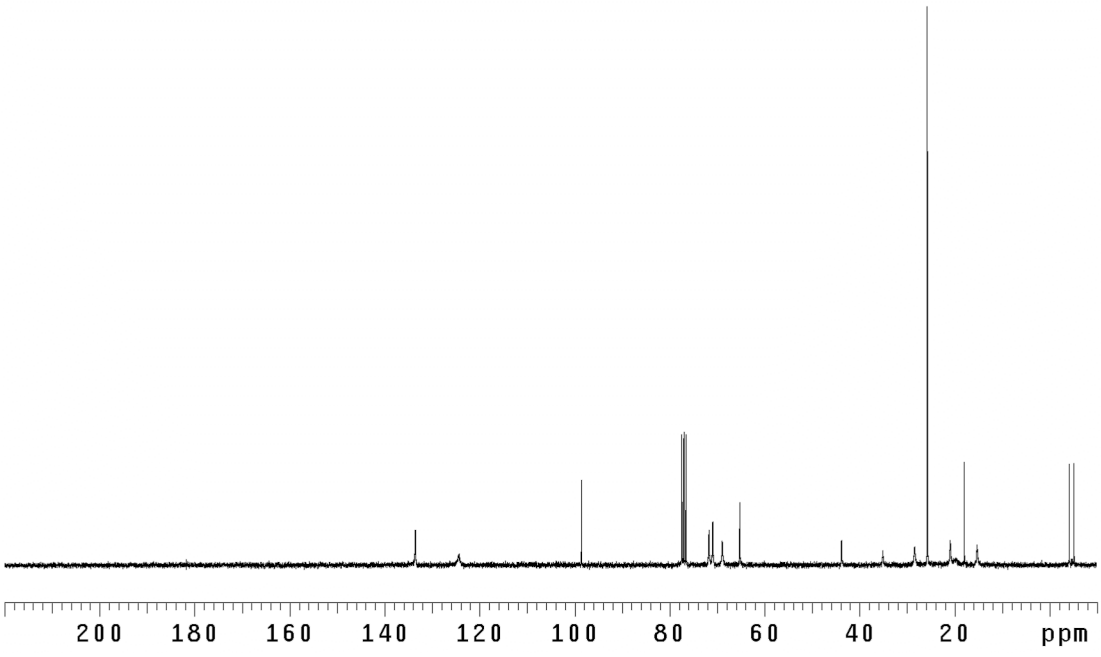

Figure B.51 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **260**.

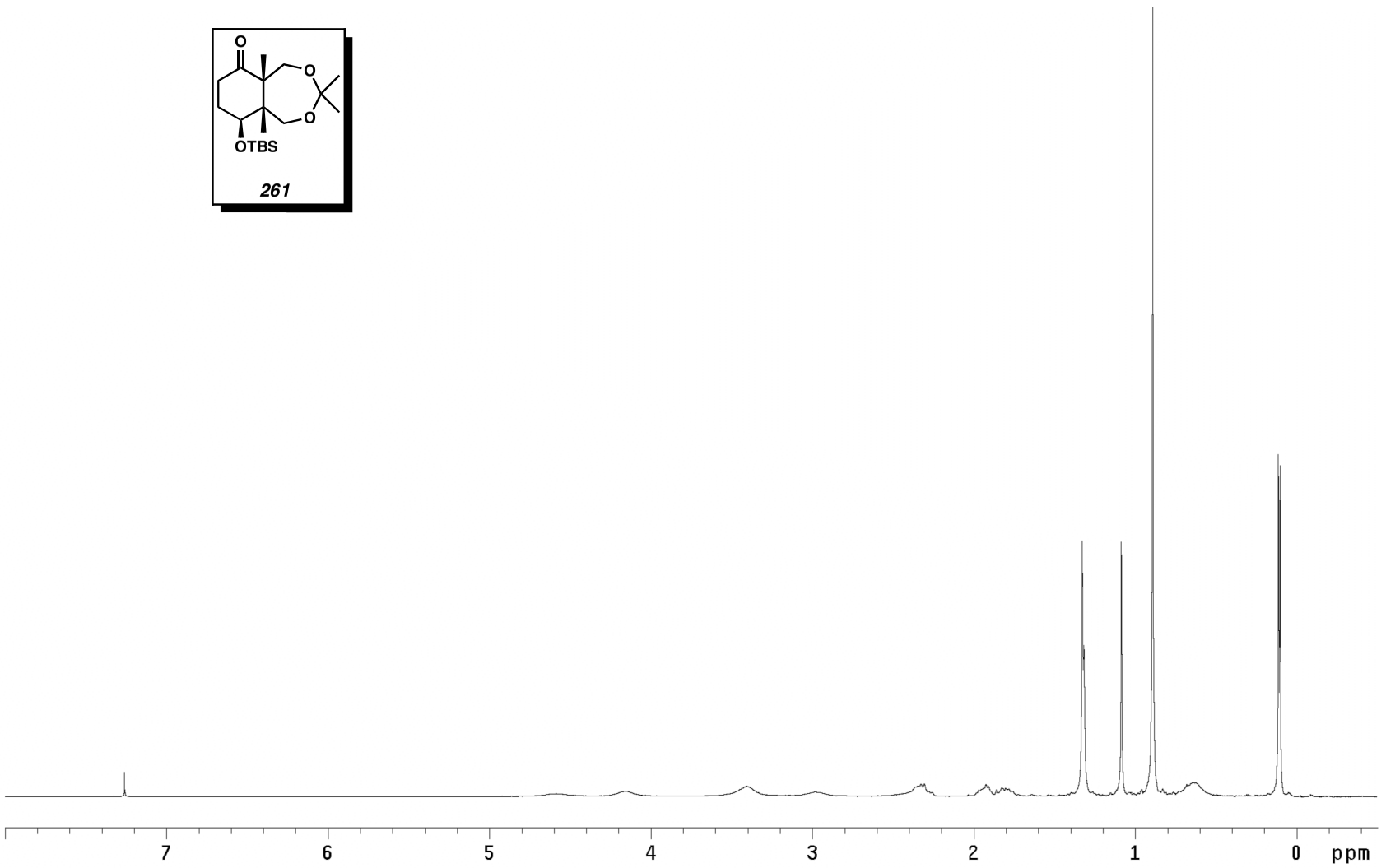

Figure B.52 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **261**.

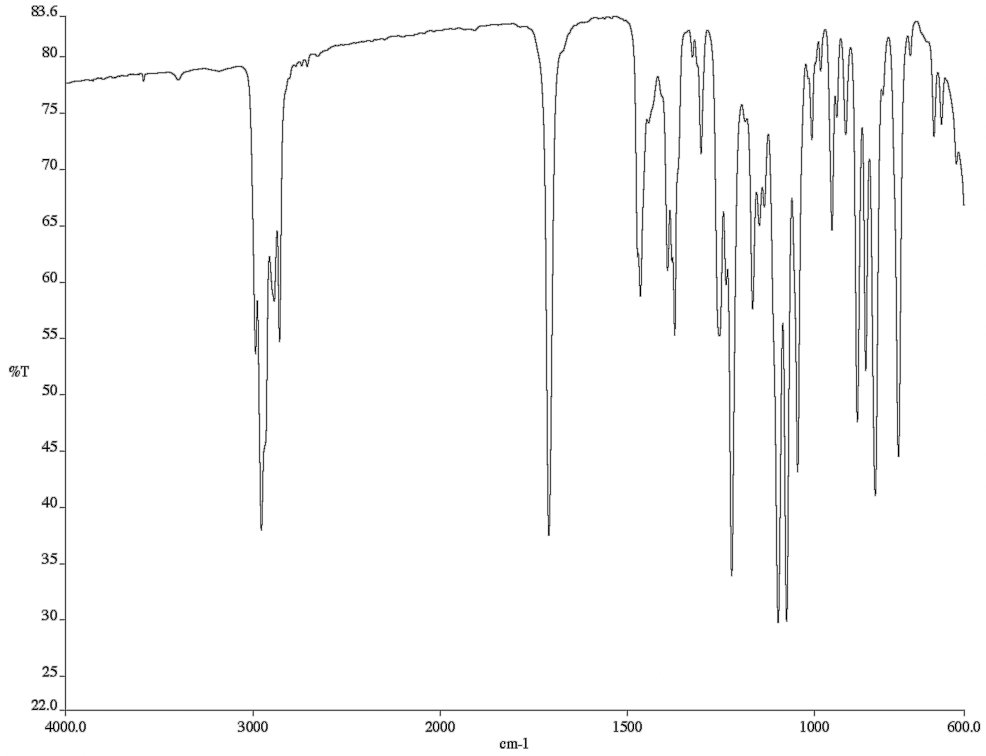

Figure B.53 Infrared spectrum (thin film/NaCl) of compound **261**.

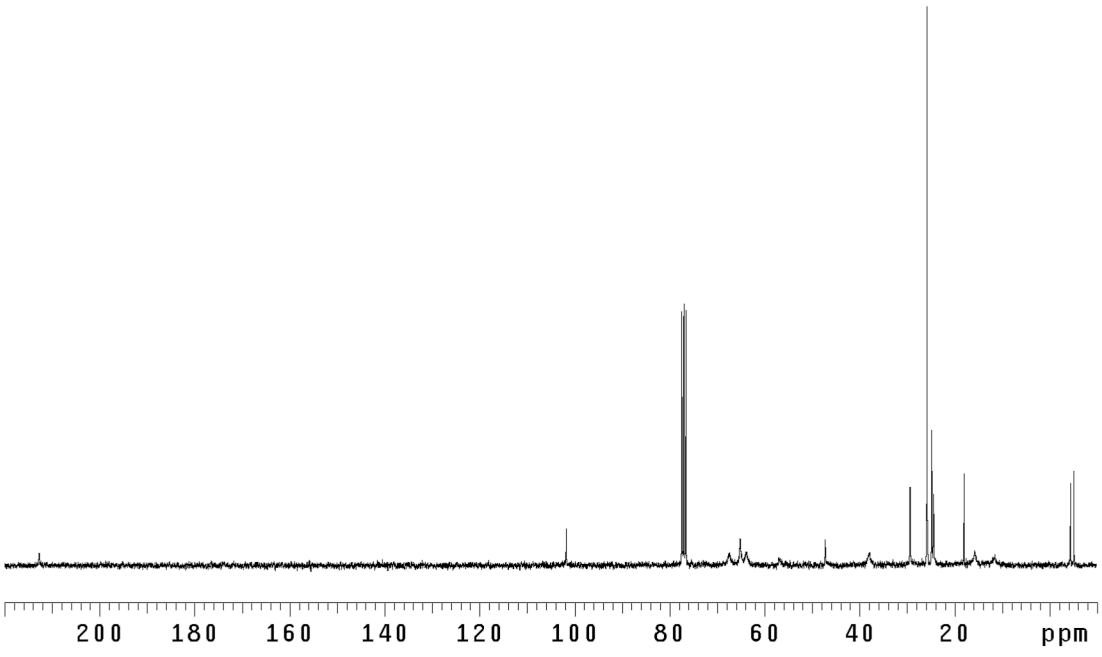

Figure B.54 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **261**.

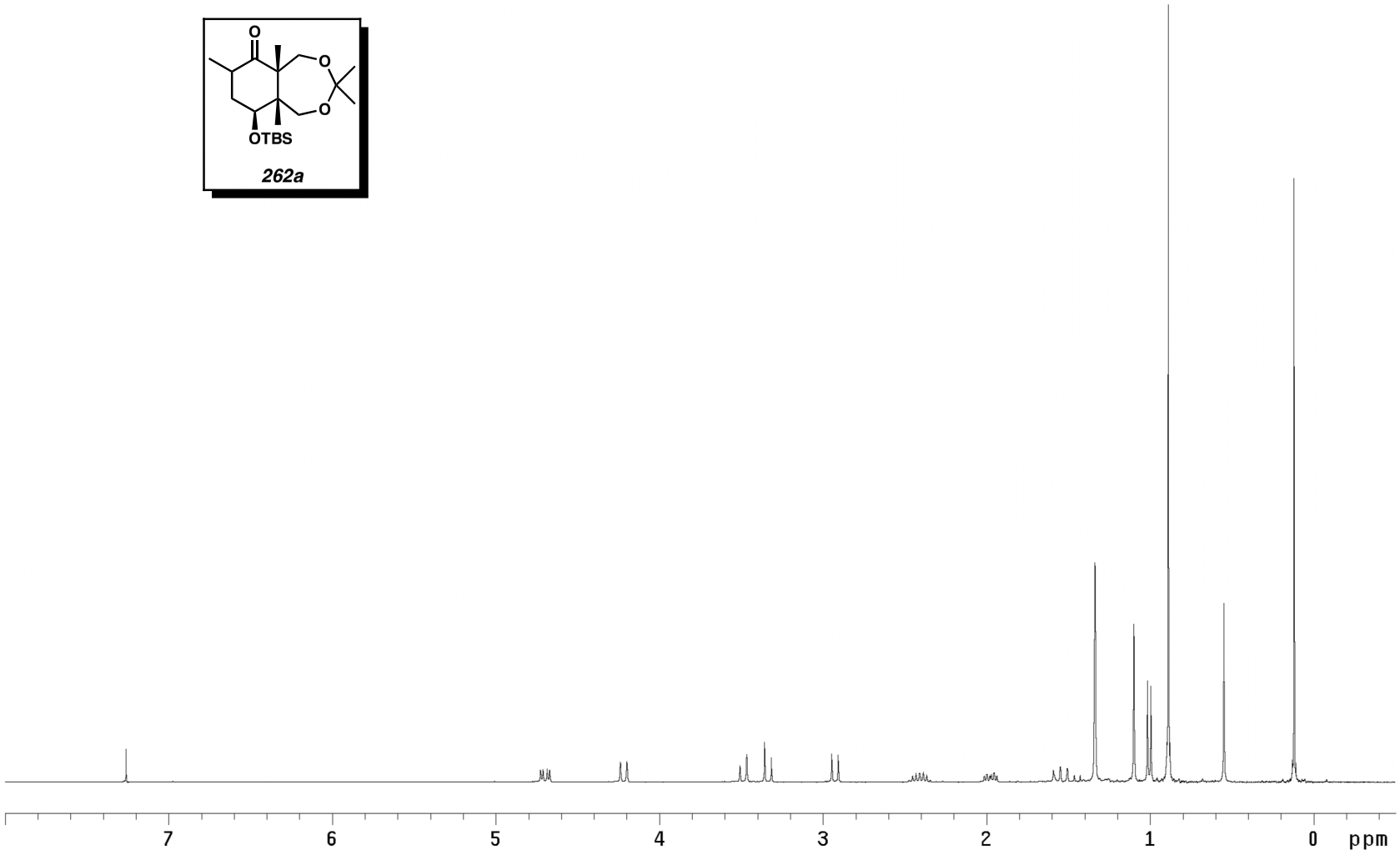

Figure B.55 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **262a**.

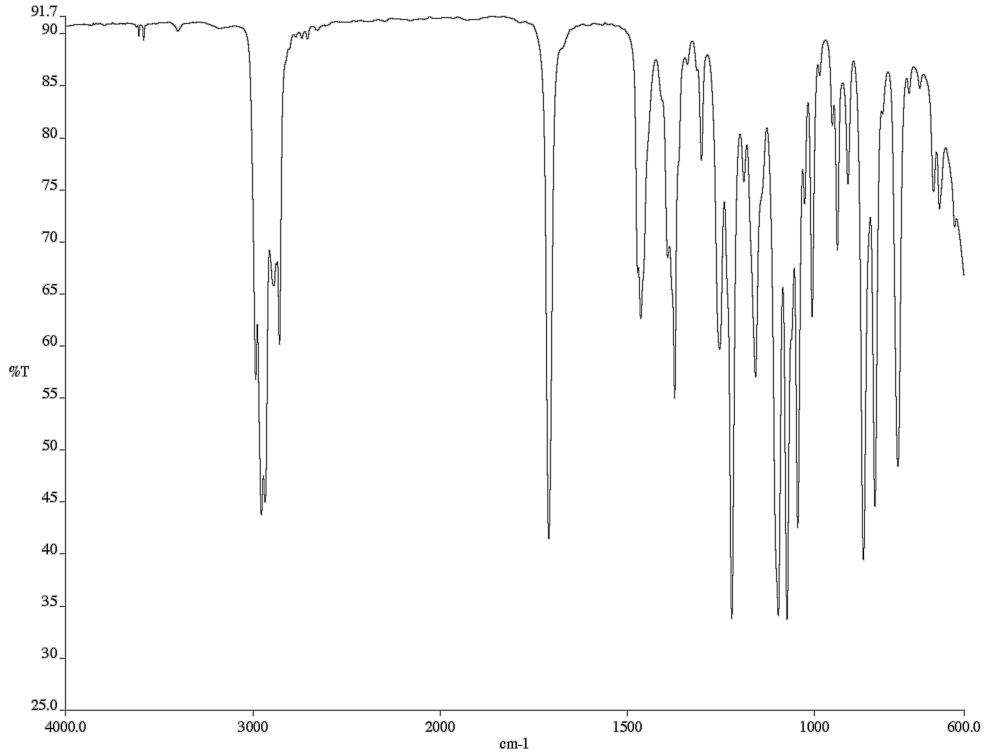

Figure B.56 Infrared spectrum (thin film/NaCl) of compound **262a**.

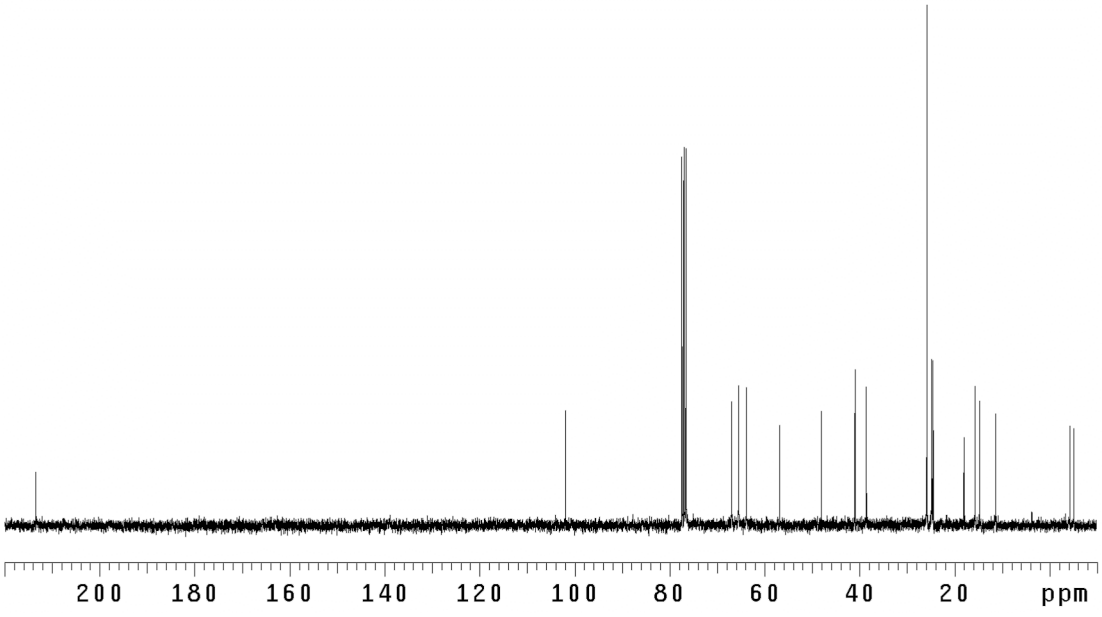

Figure B.57 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **262a**.

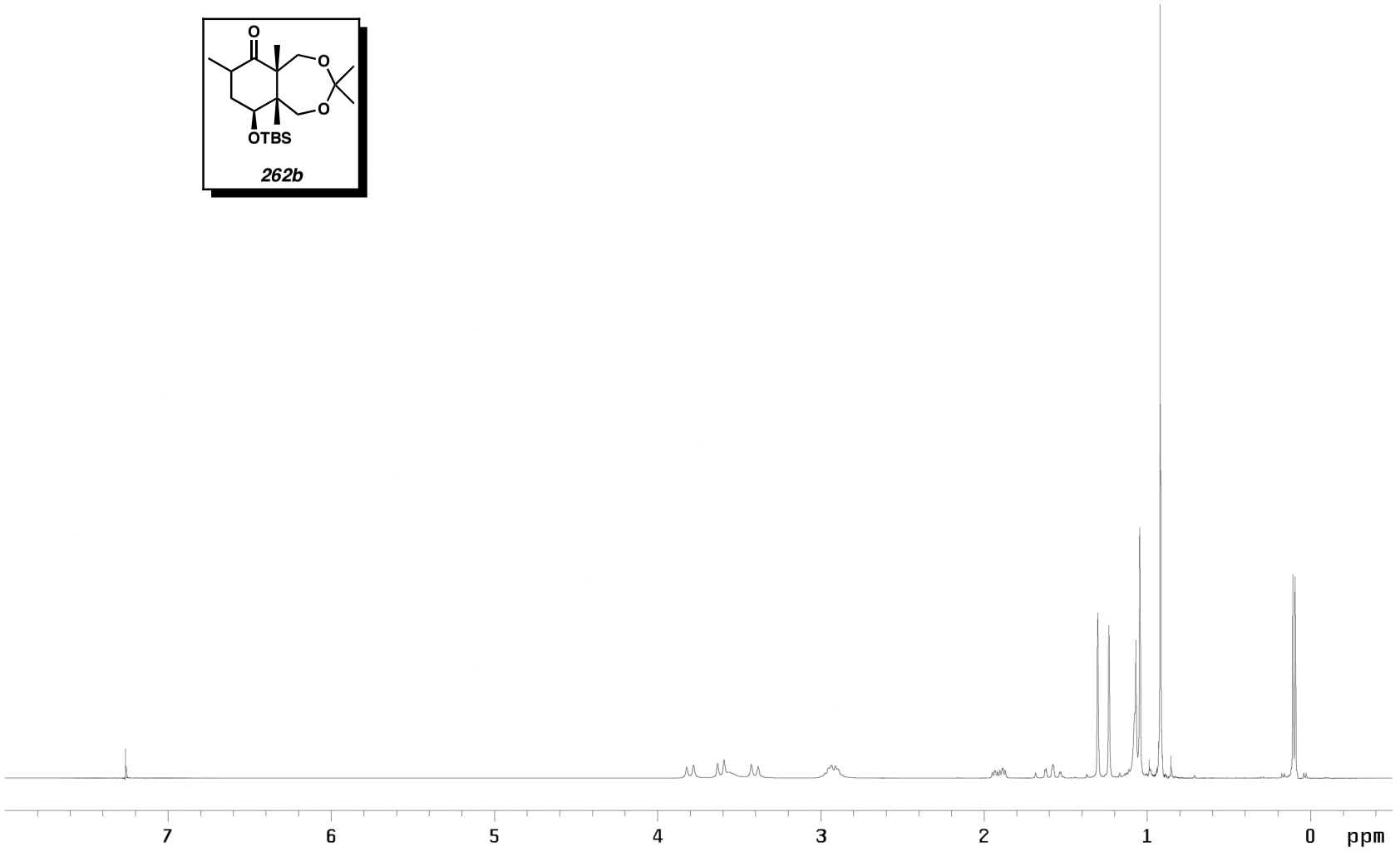

Figure B.58 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **262b**.

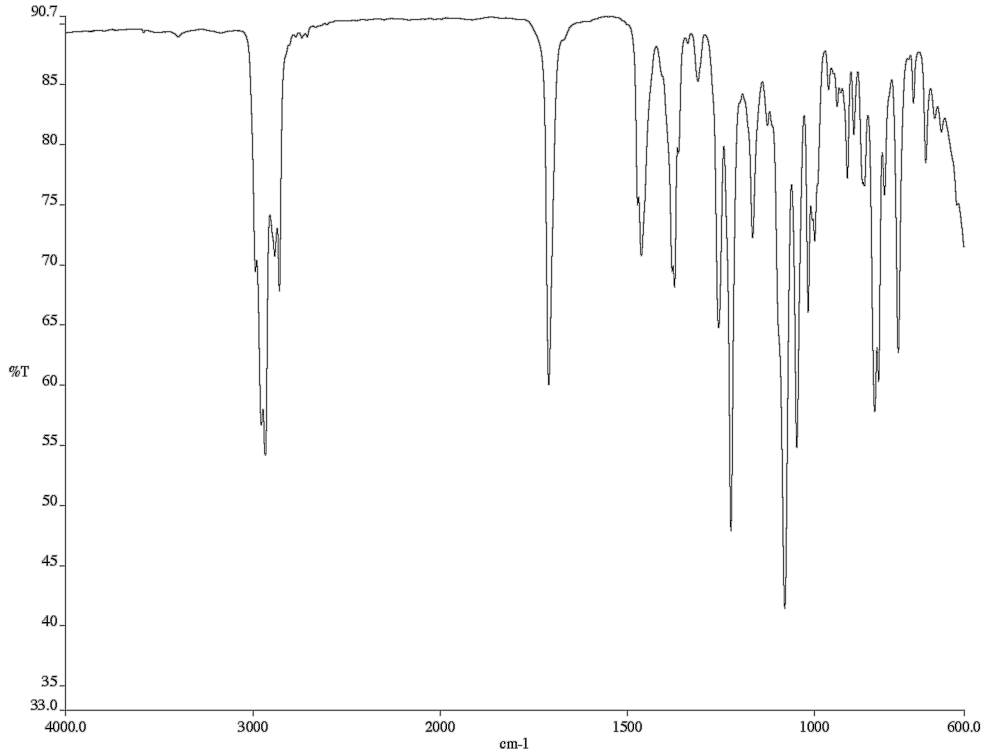

Figure B.59 Infrared spectrum (thin film/NaCl) of compound **262b**.

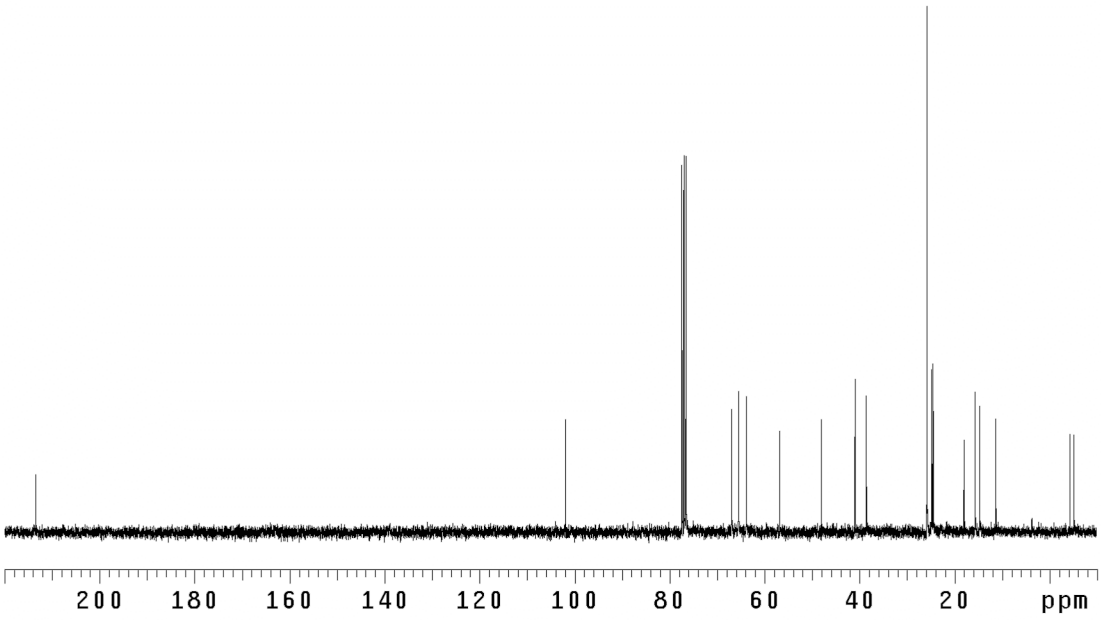

Figure B.60 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **262b**.

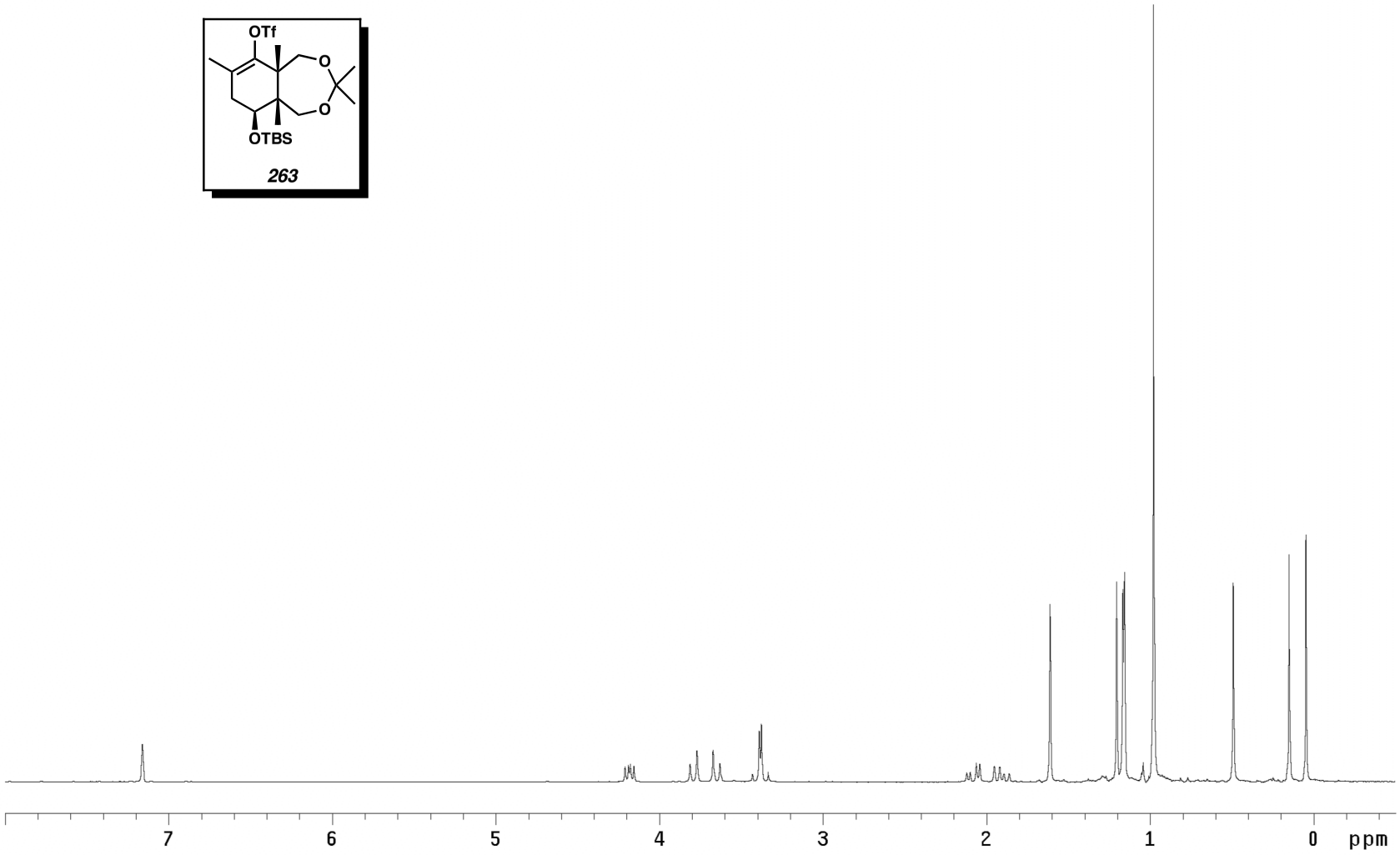

Figure B.61 ^{1}H NMR (300 MHz, C_6D_6) of compound **263**.

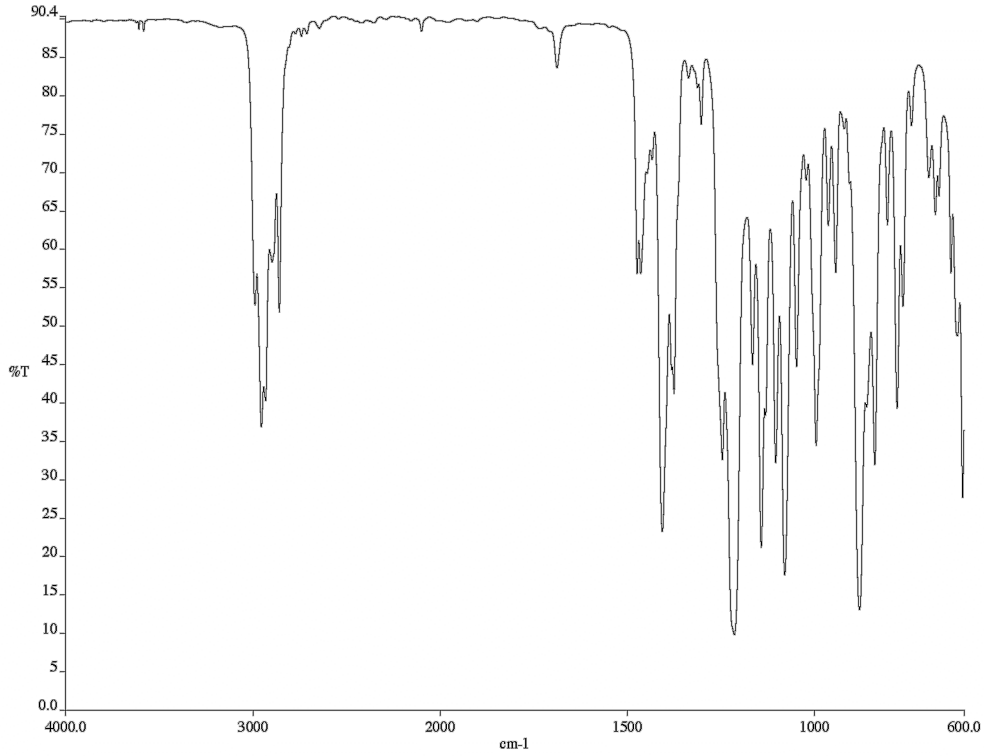

Figure B.62 Infrared spectrum (thin film/NaCl) of compound **263**.

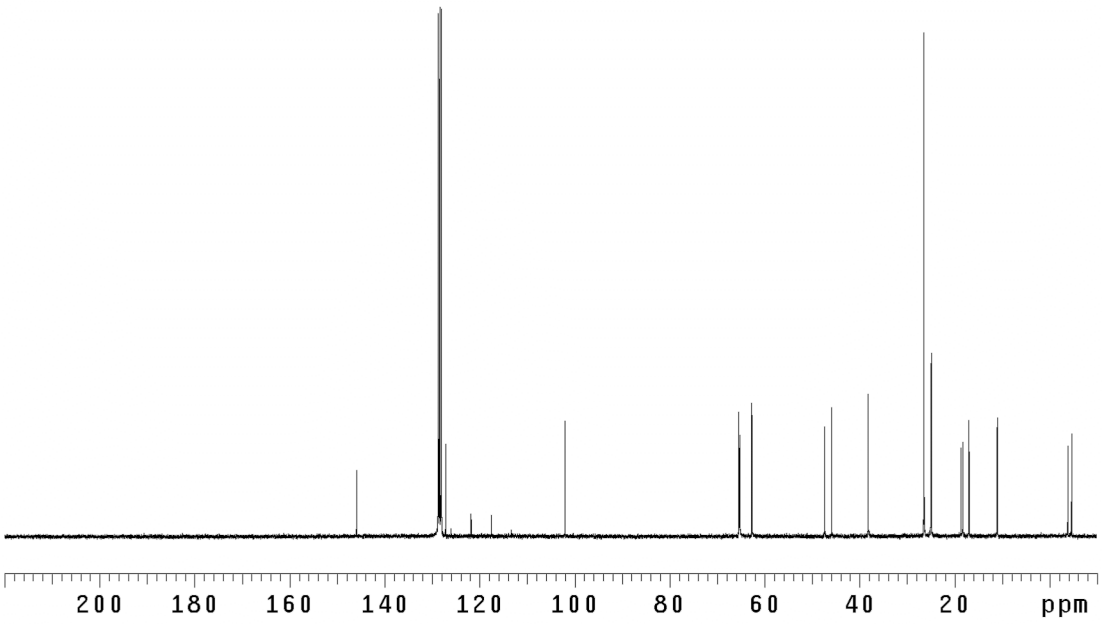

Figure B.63 ^{13}C NMR (300 MHz, C_6D_6) of compound **263**.

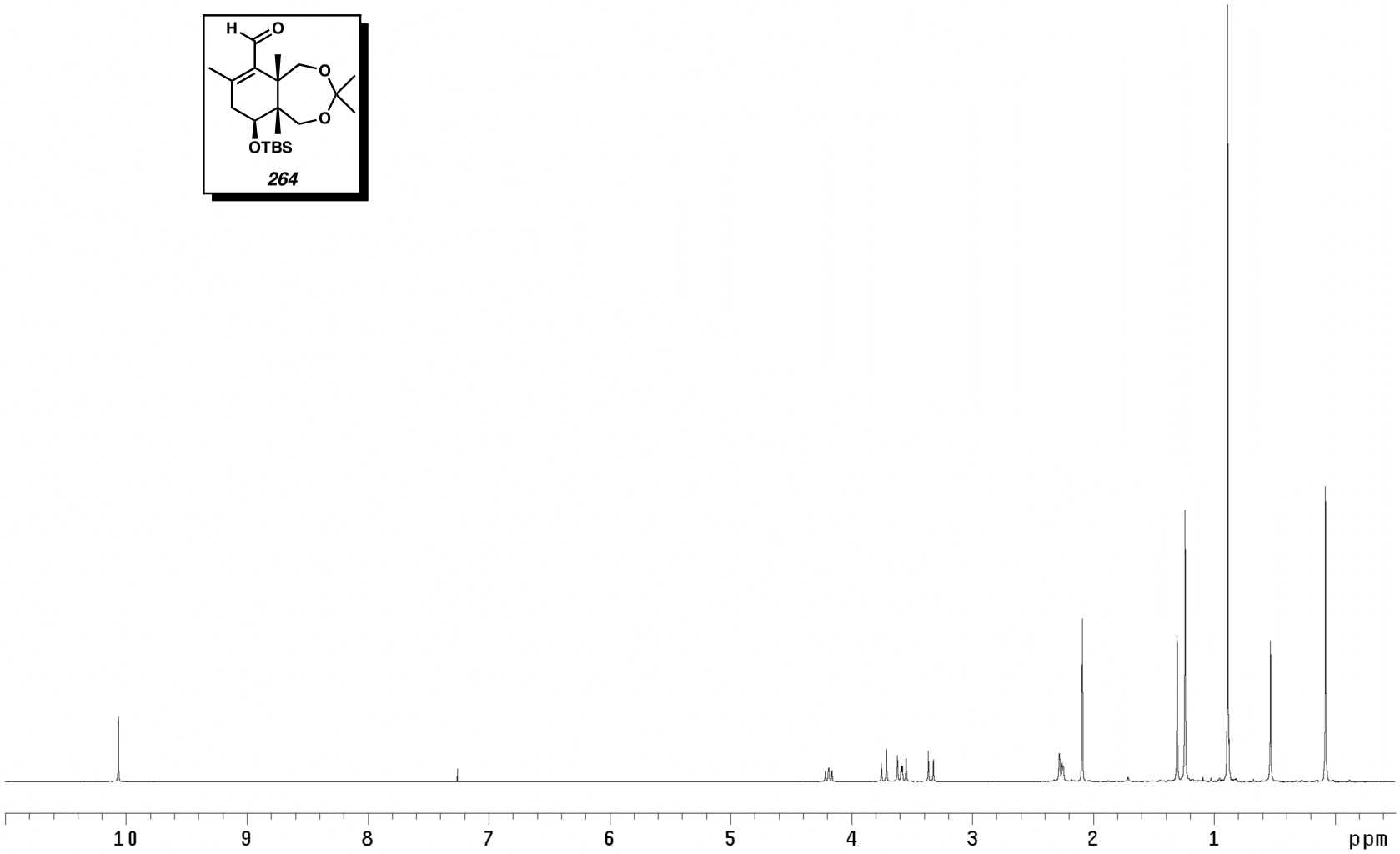

Figure B.64 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **264**.

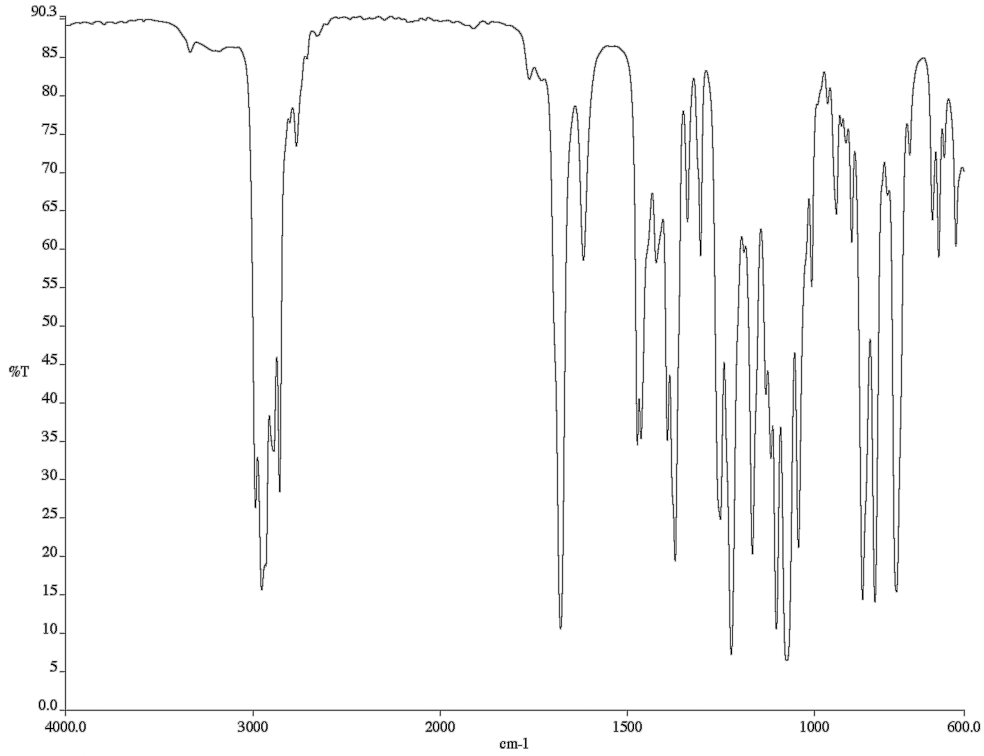

Figure B.65 Infrared spectrum (thin film/NaCl) of compound **264**.

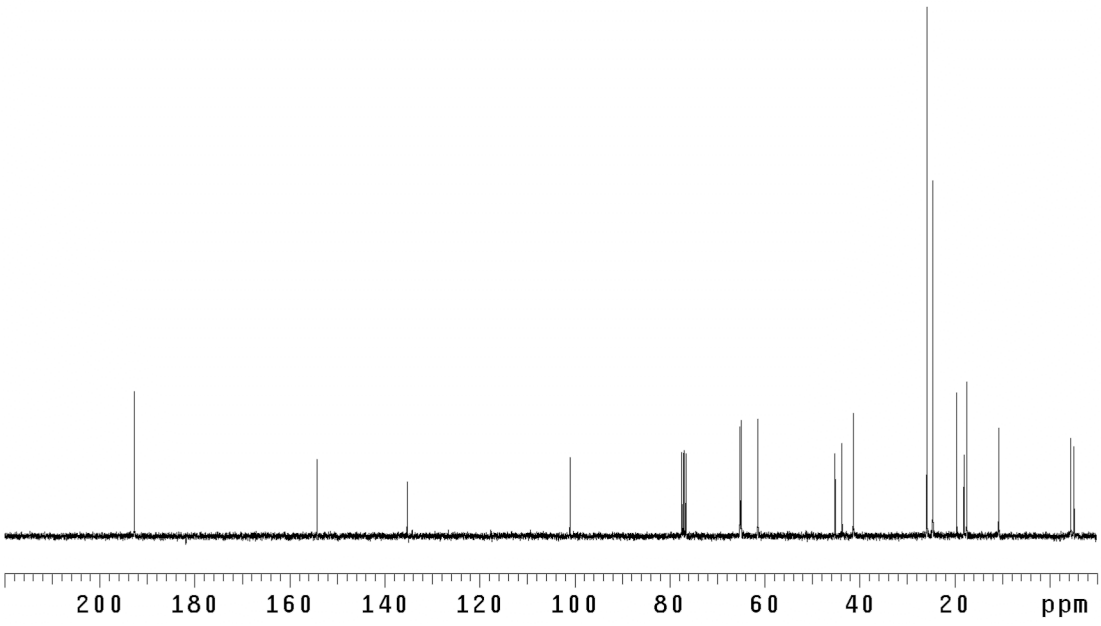

Figure B.66 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **264**.

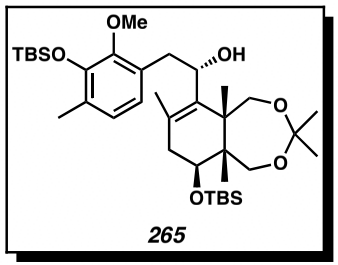

Figure B.67 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **265**.

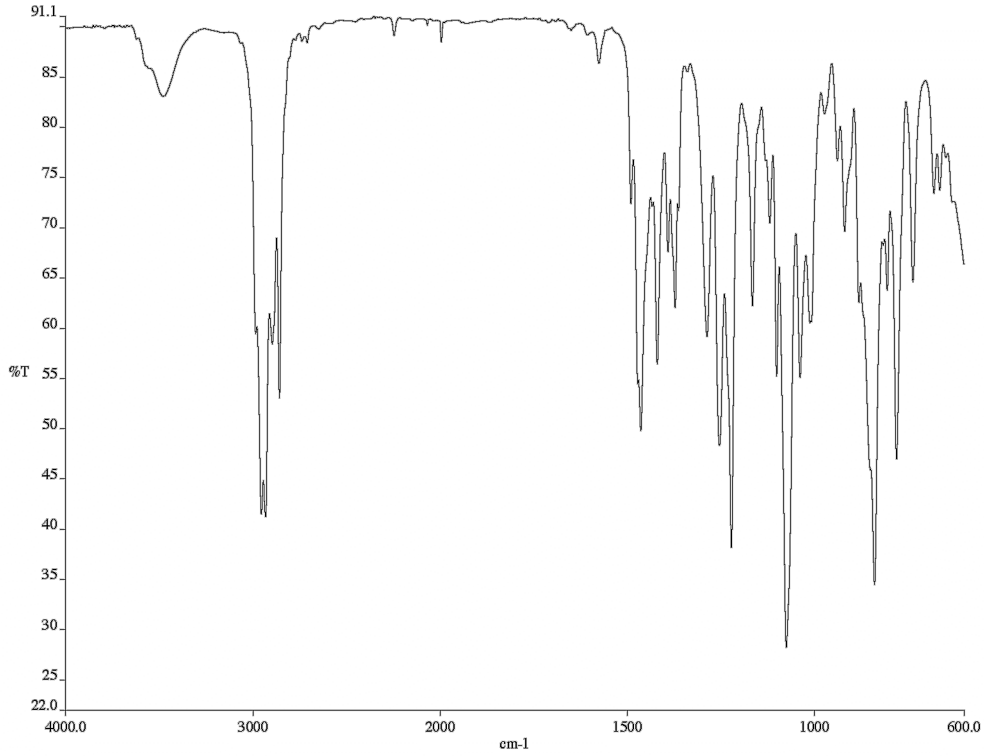

Figure B.68 Infrared spectrum (thin film/NaCl) of compound **265**.

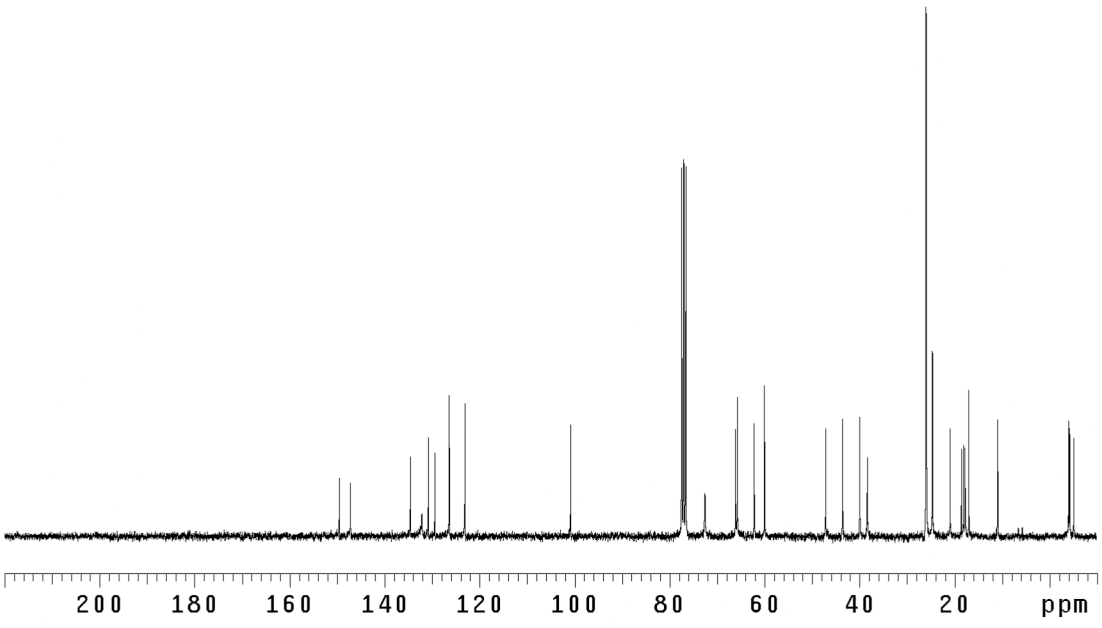

Figure B.69 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **265**.

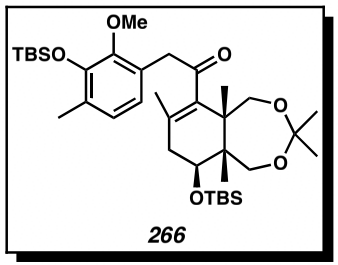

Figure B.70 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **266**.

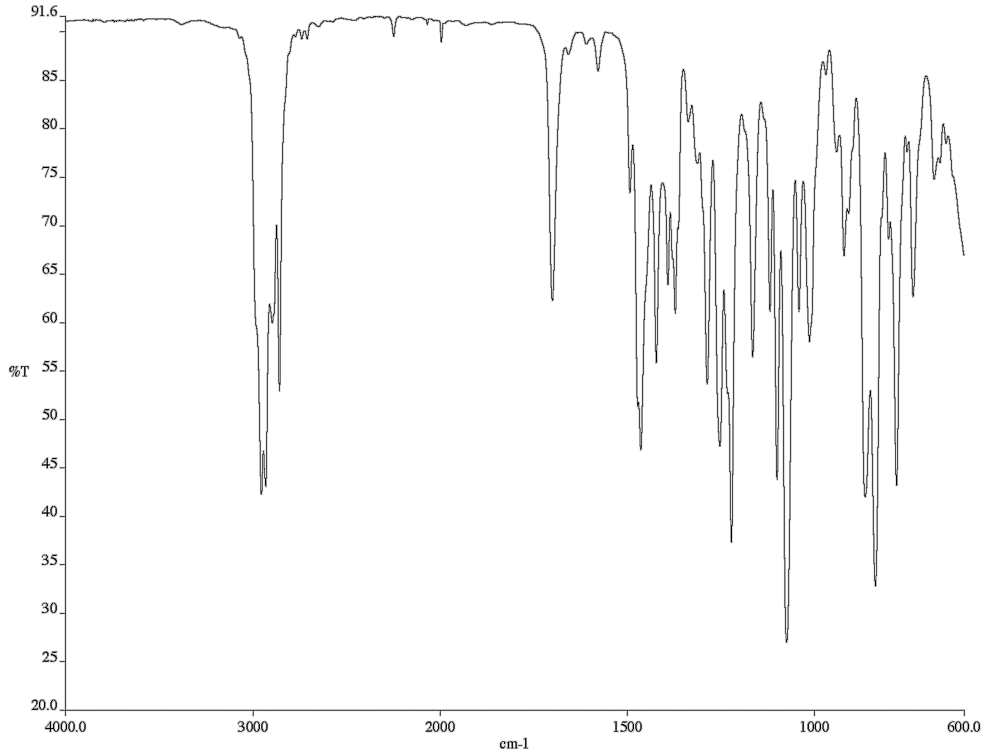

Figure B.71 Infrared spectrum (thin film/NaCl) of compound **266**.

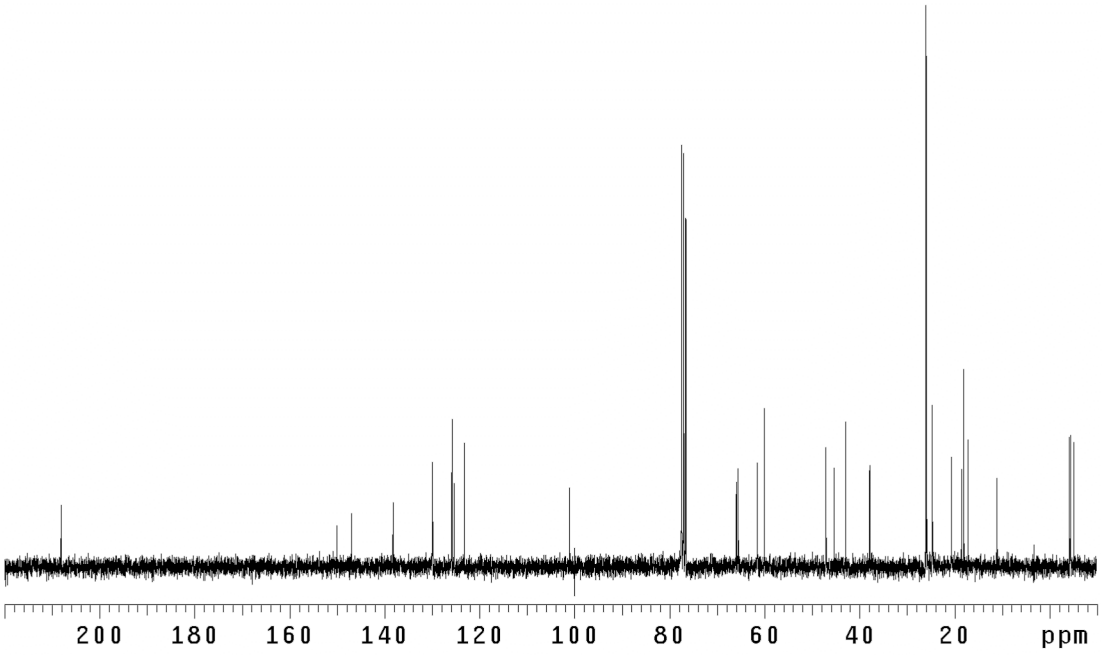

Figure B.72 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **266**.

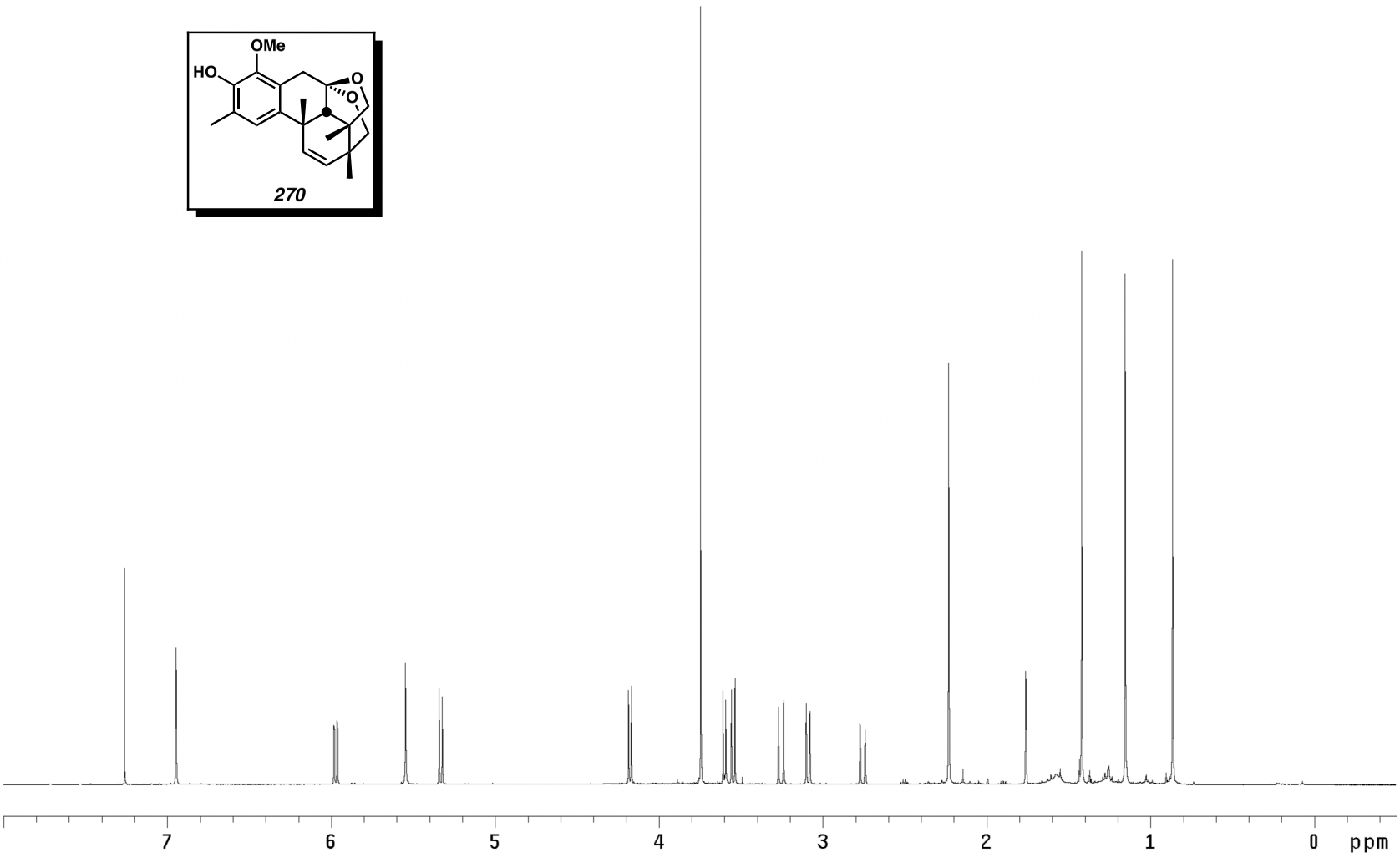

Figure B.73 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **270**.

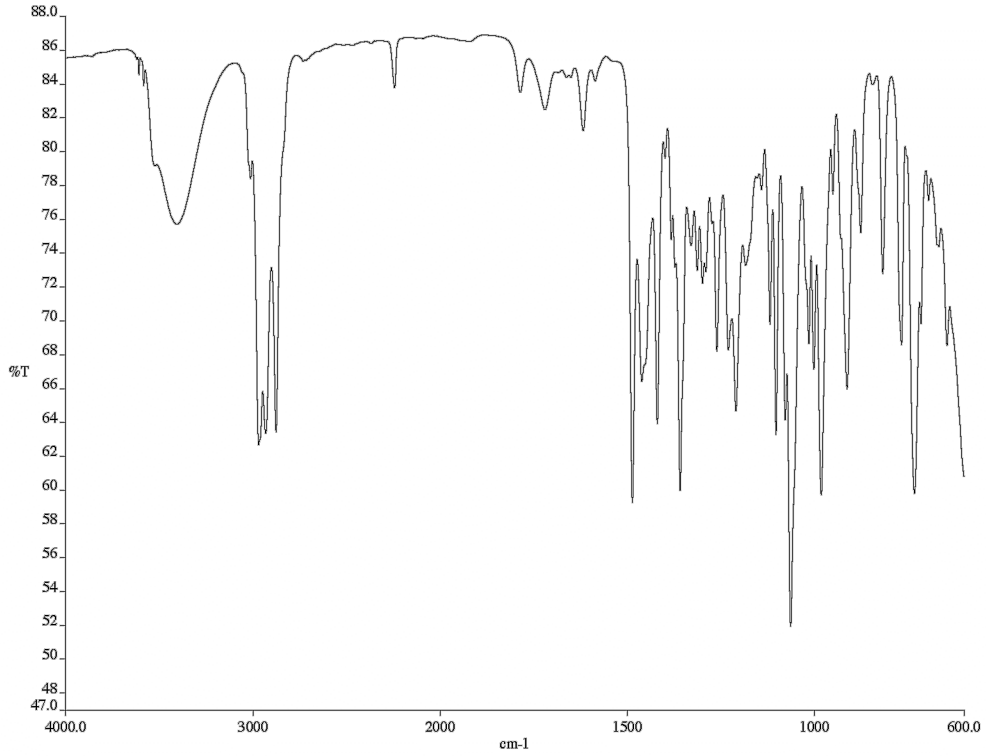

Figure B.73 Infrared spectrum (thin film/NaCl) of compound **270**.

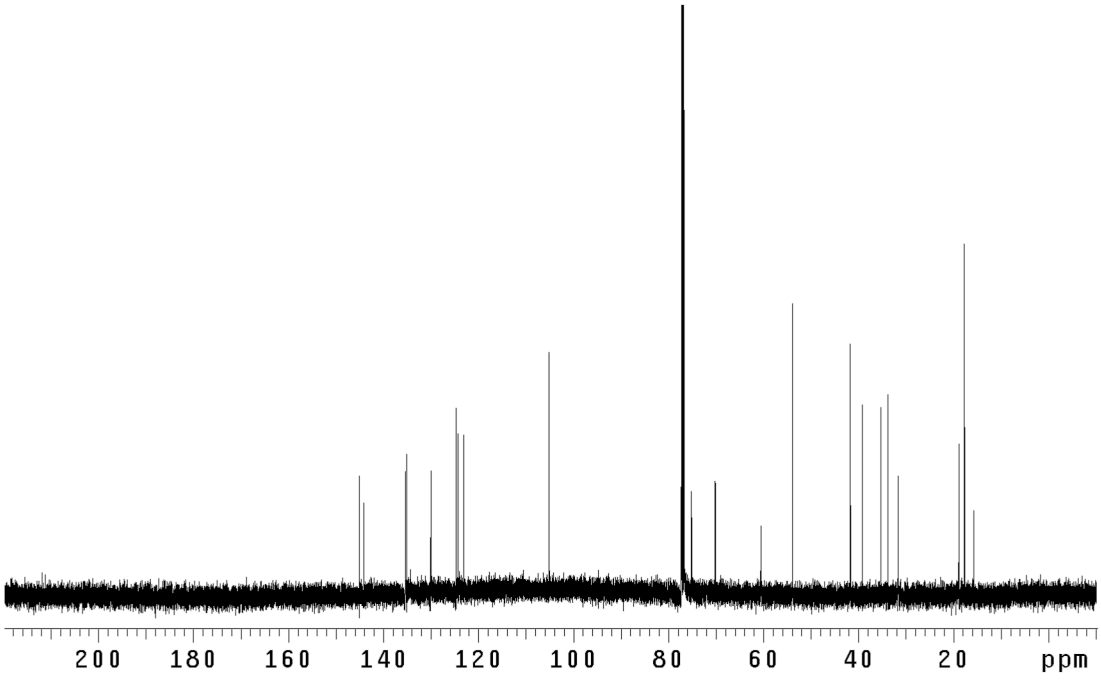

Figure B.74 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **270**.

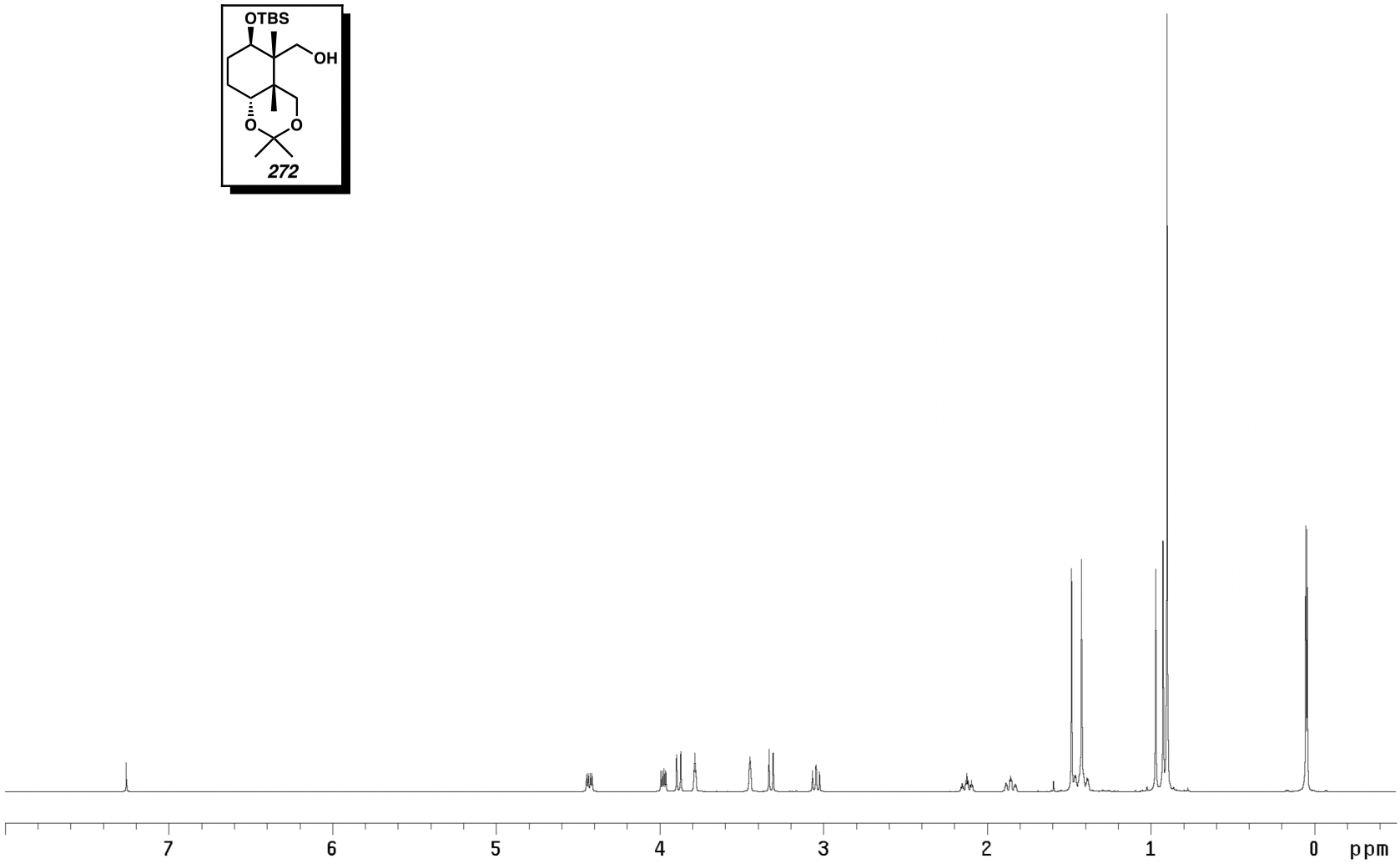

Figure B.76 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **272**.

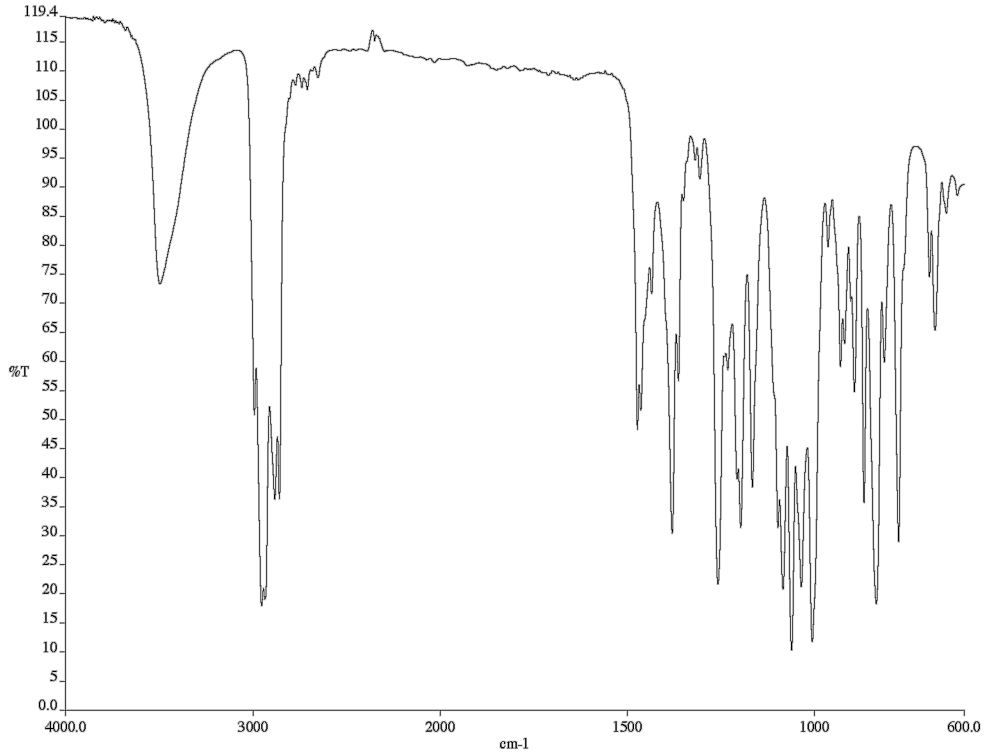

Figure B.77 Infrared spectrum (thin film/NaCl) of compound **272**.

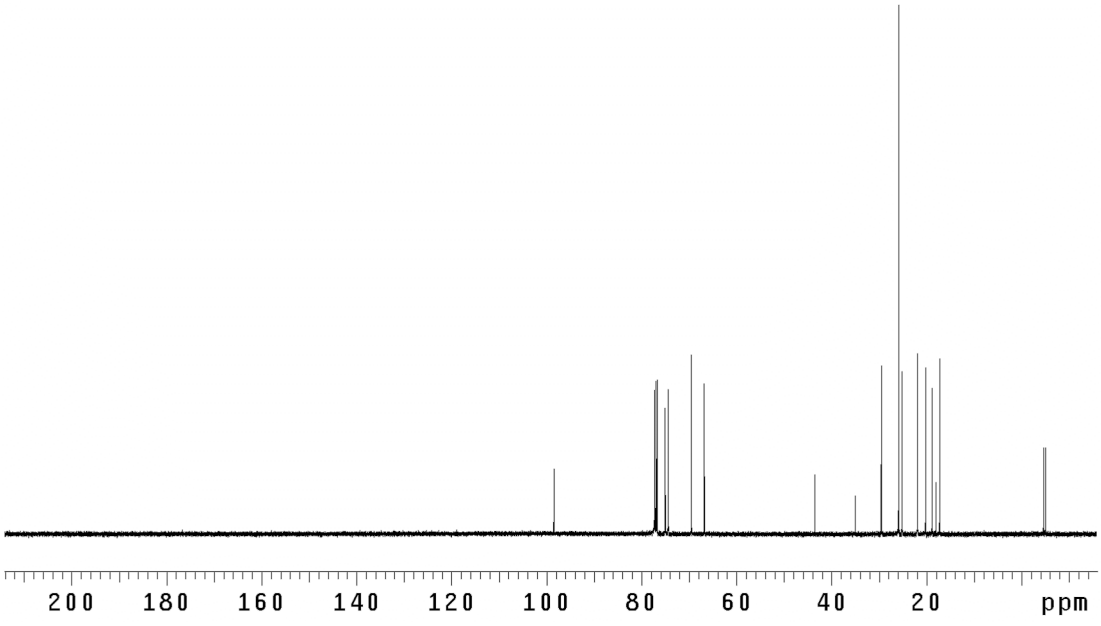

Figure B.78 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **272**.

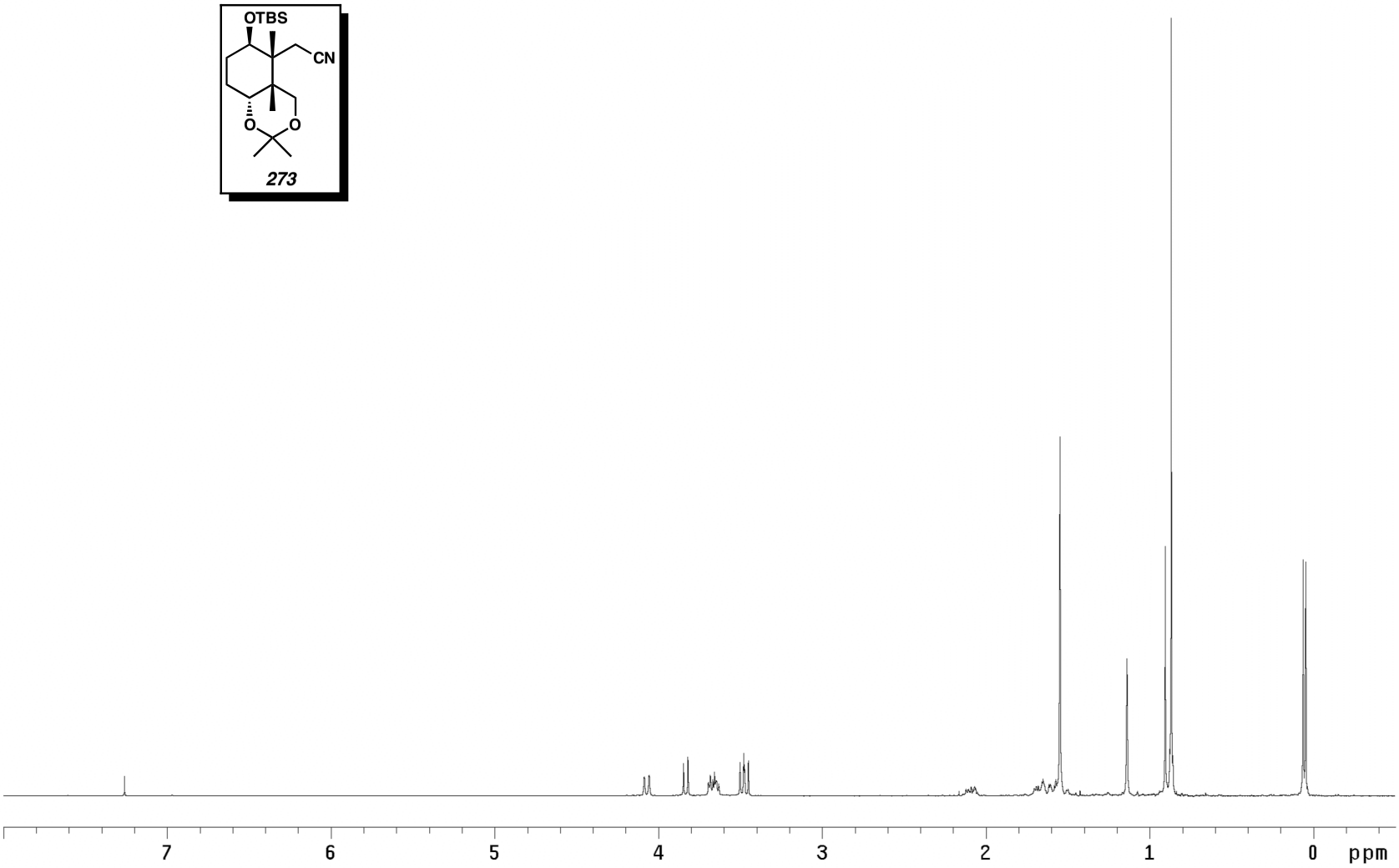

Figure B.79 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **273**.

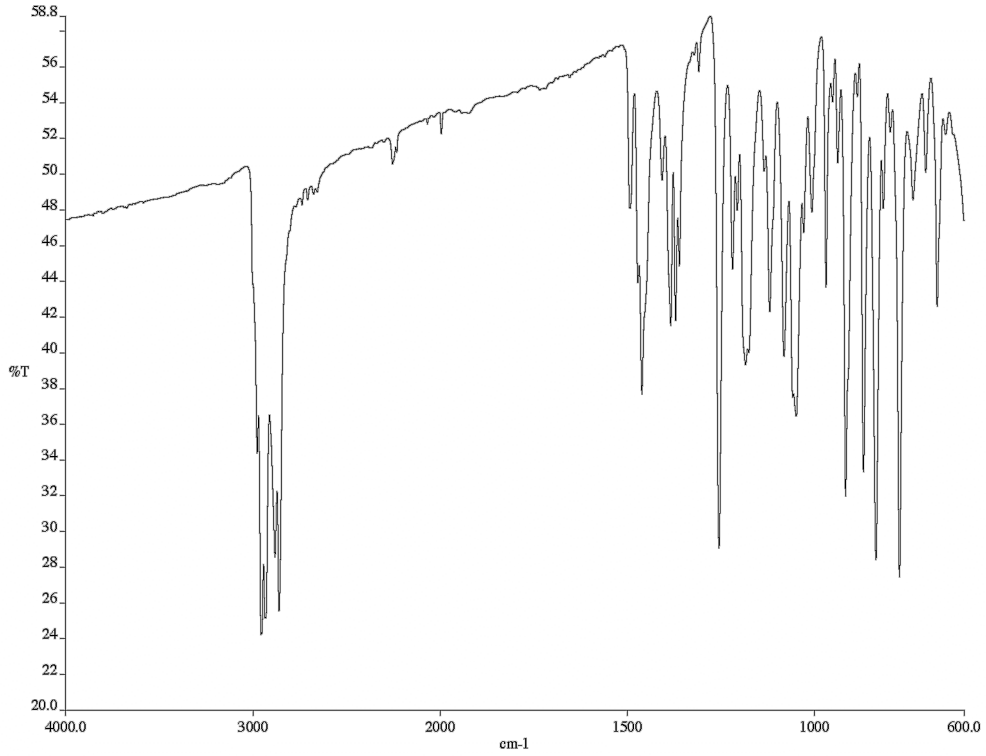

Figure B.80 Infrared spectrum (thin film/NaCl) of compound **273**.

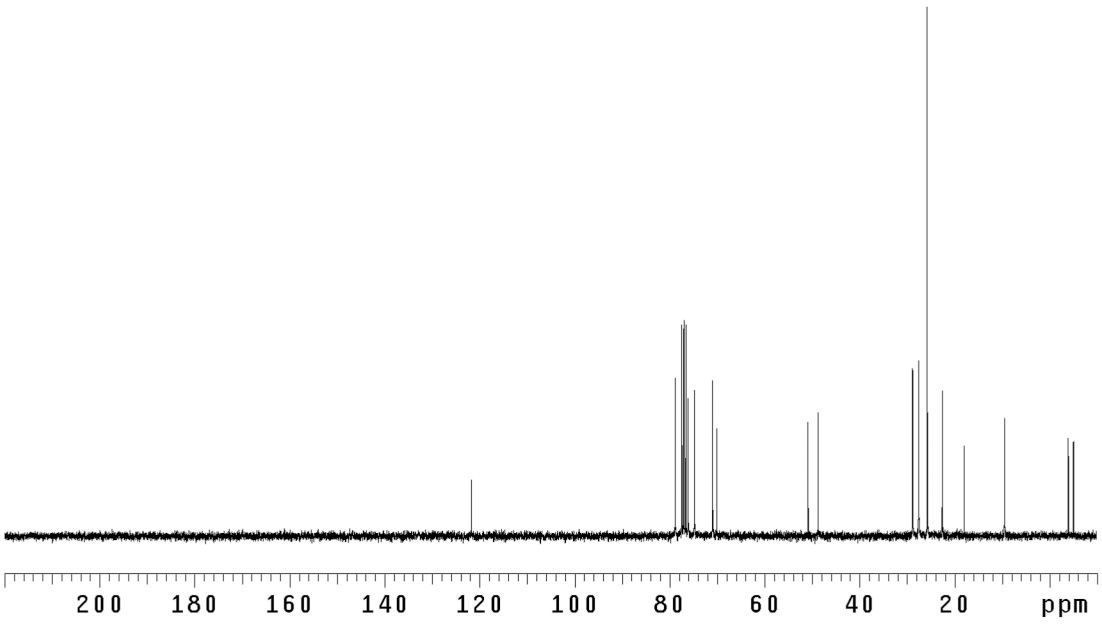

Figure B.81 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **273**.

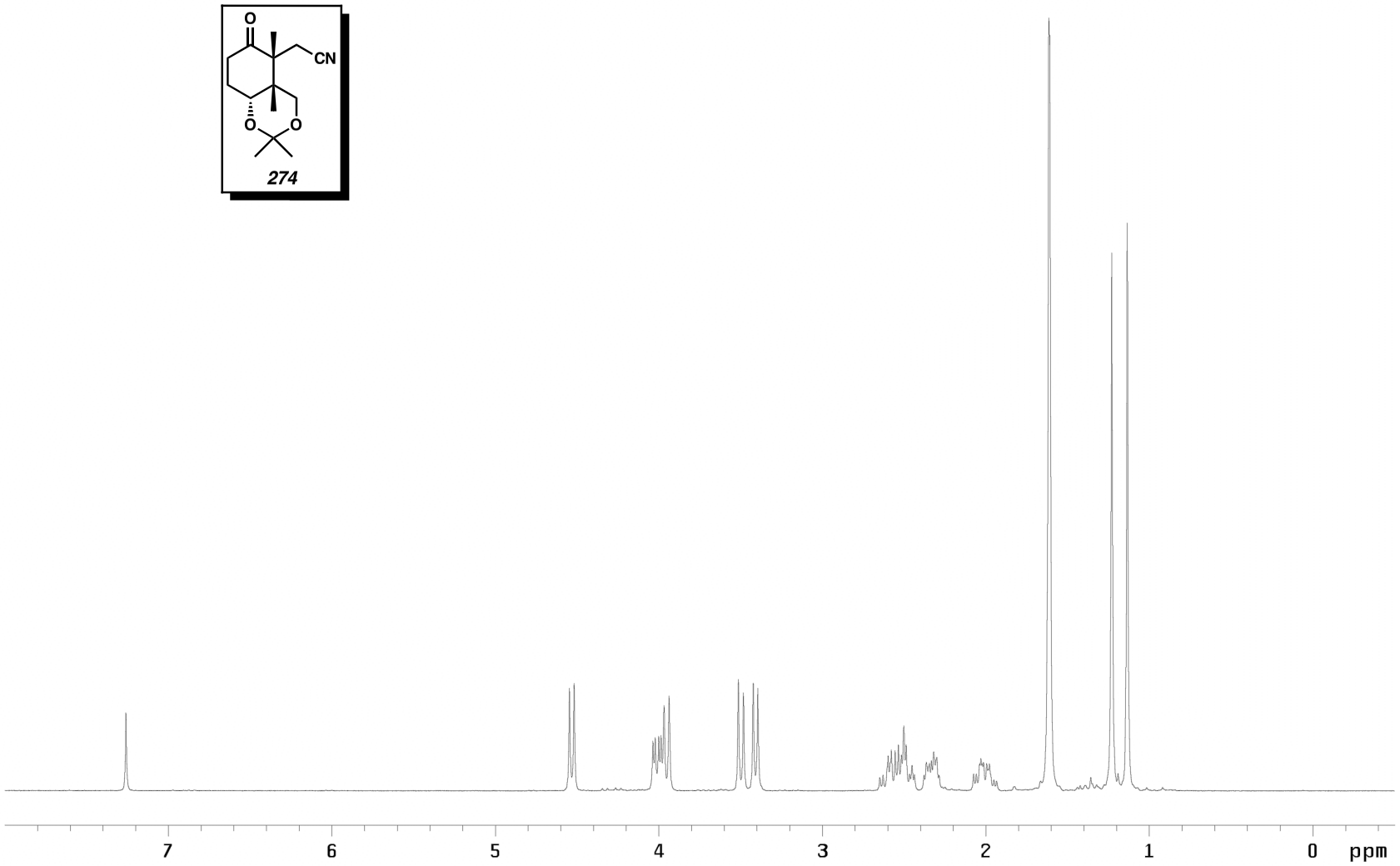

Figure B.82 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **274**.

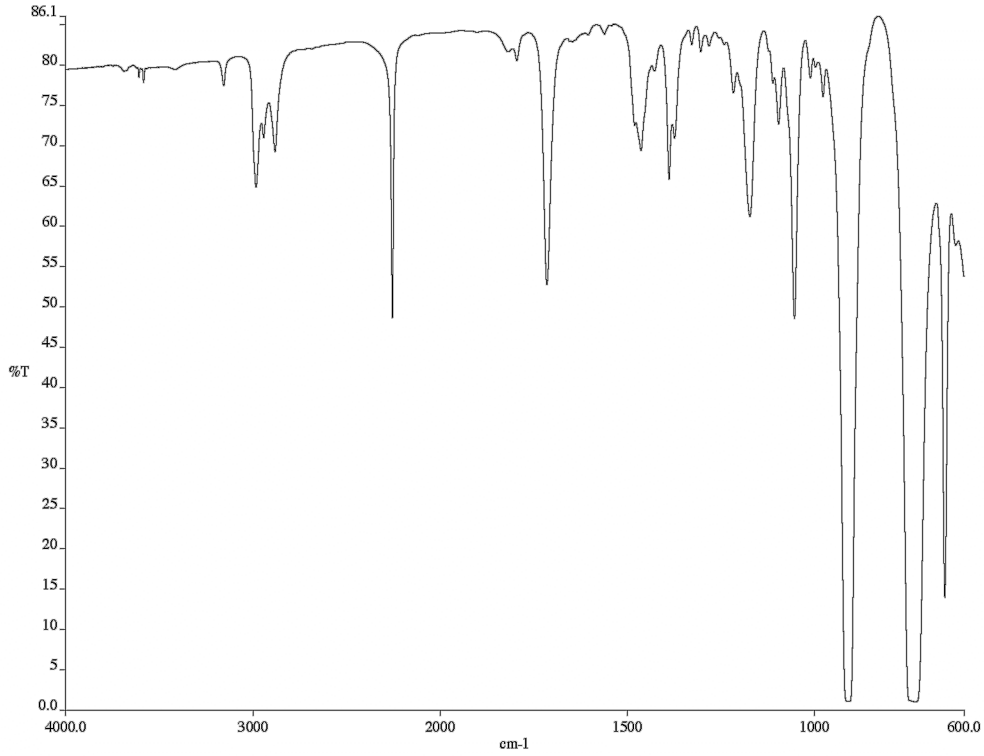

Figure B.83 Infrared spectrum (thin film/NaCl) of compound **274**.

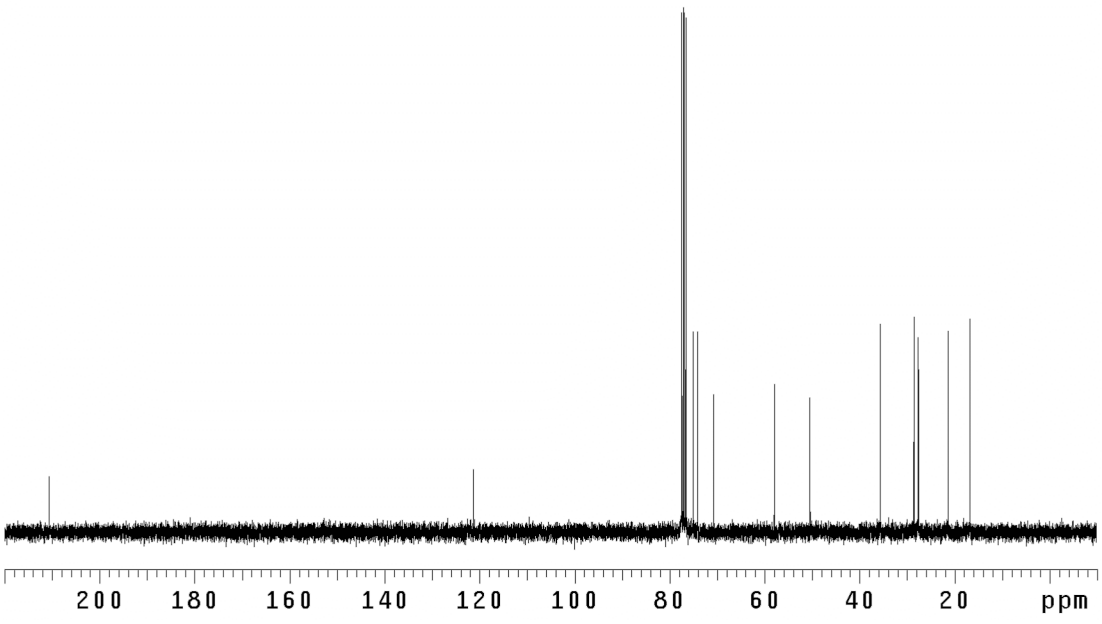

Figure B.84 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **274**.

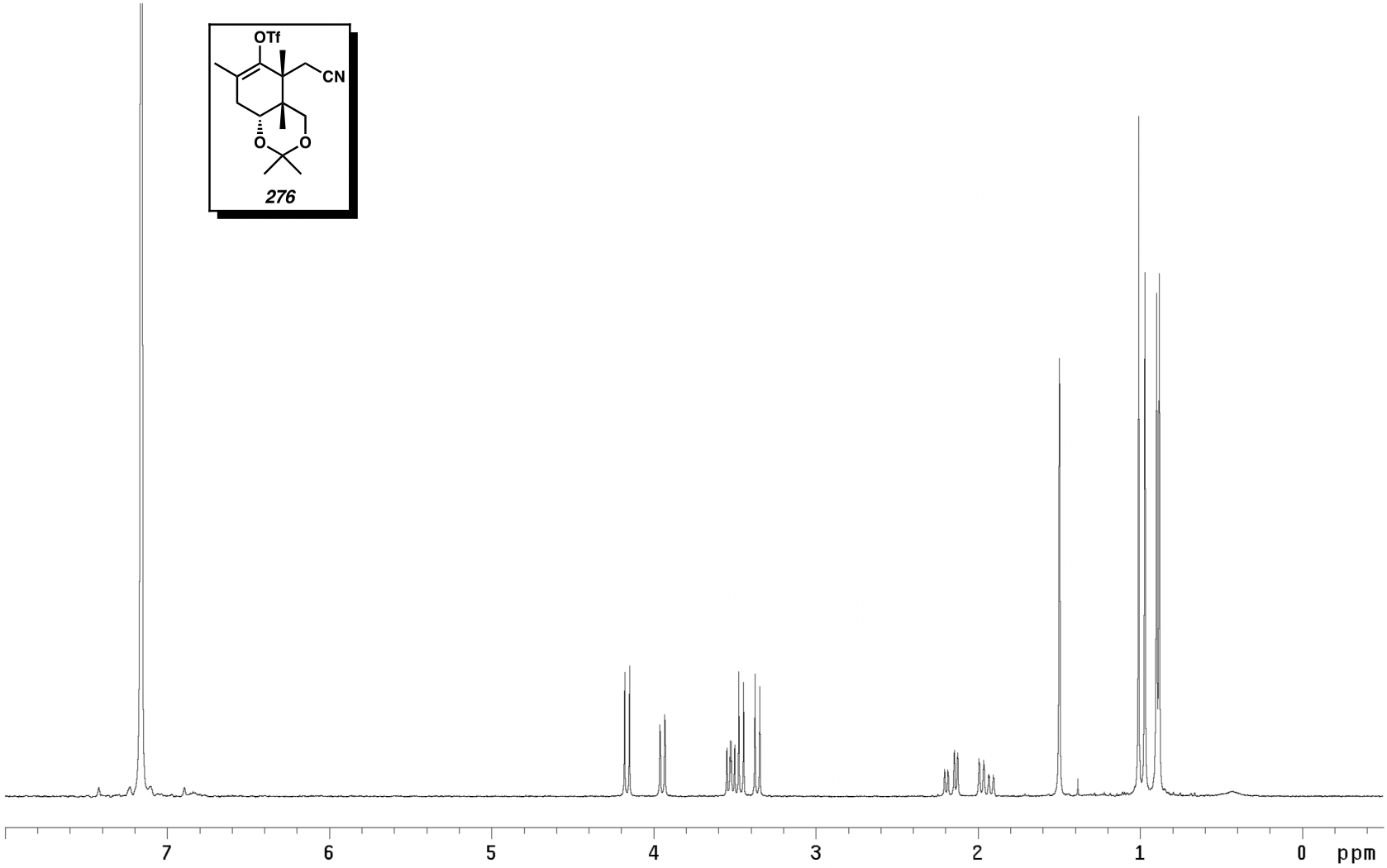

Figure B.85 ^{1}H NMR (300 MHz, C_6D_6) of compound **276**.

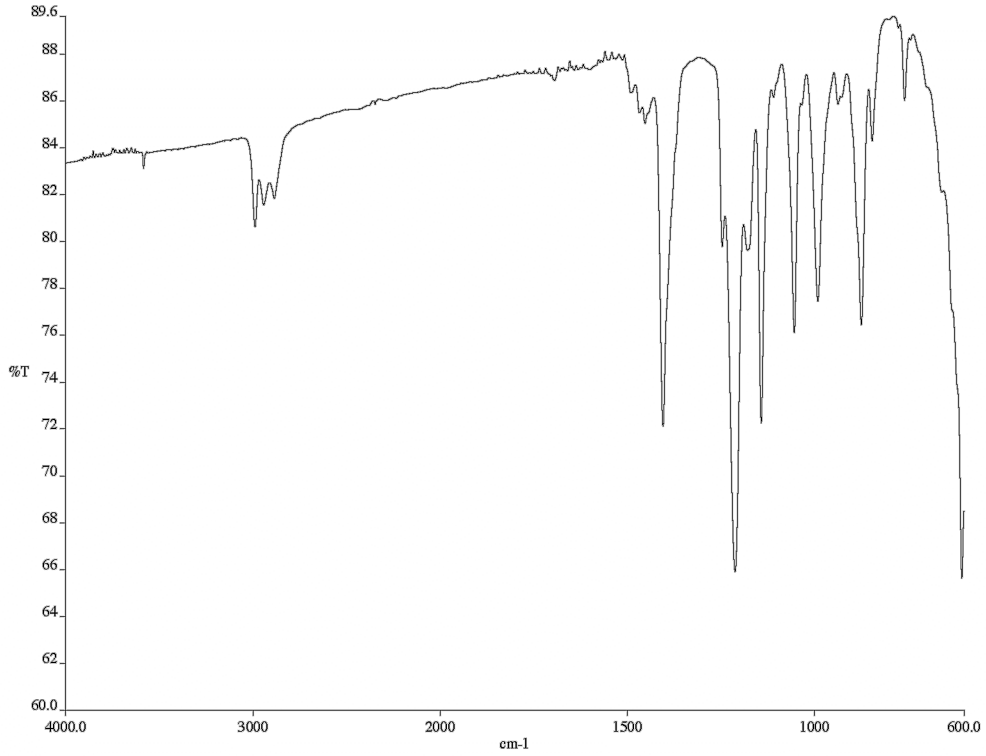

Figure B.86 Infrared spectrum (thin film/NaCl) of compound **276**.

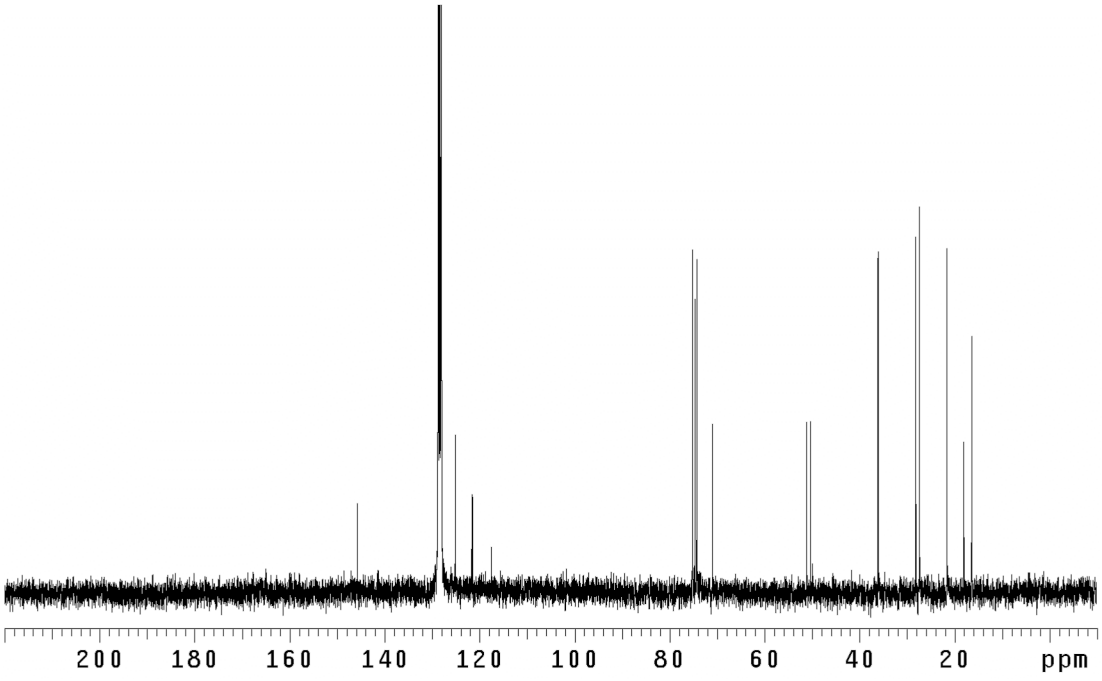

Figure B.87 ^{13}C NMR (75 MHz, C_6D_6) of compound **276**.

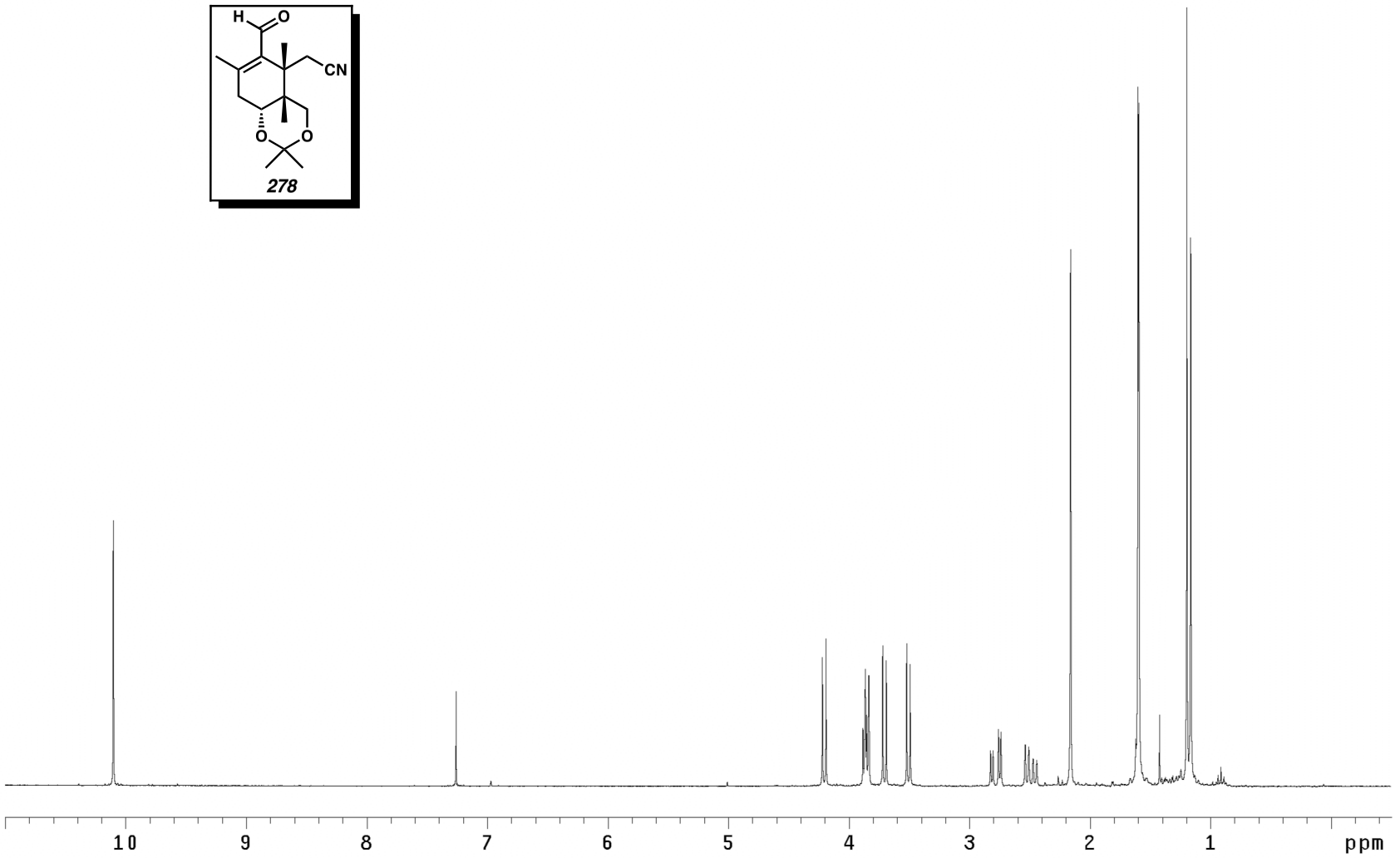

Figure B.88 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **278**.

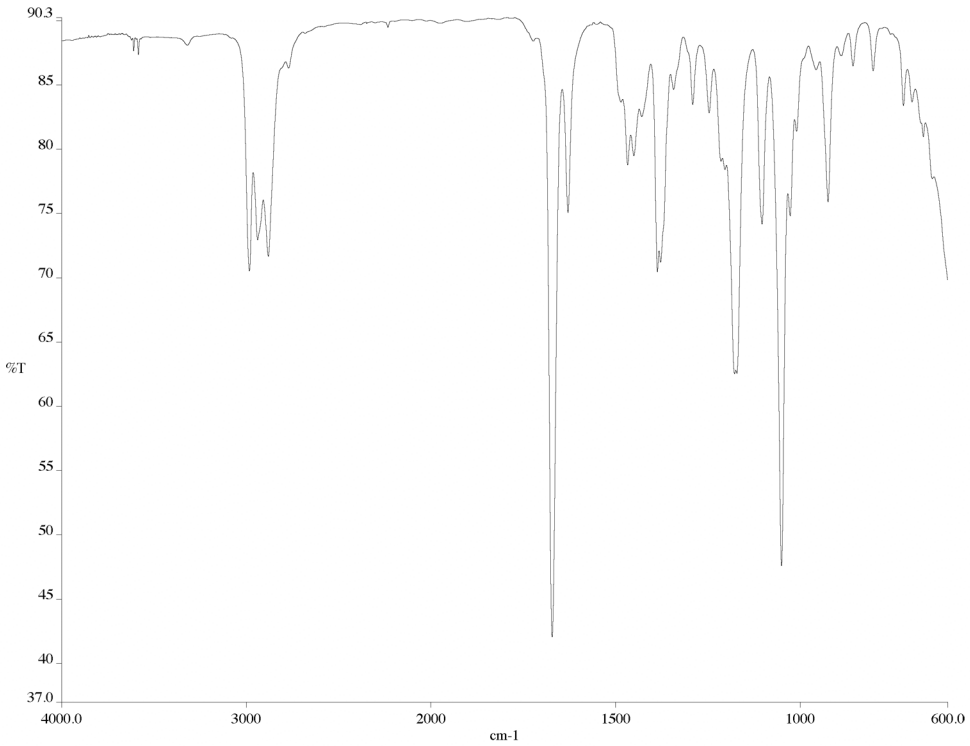

Figure B.89 Infrared spectrum (thin film/NaCl) of compound **278**.

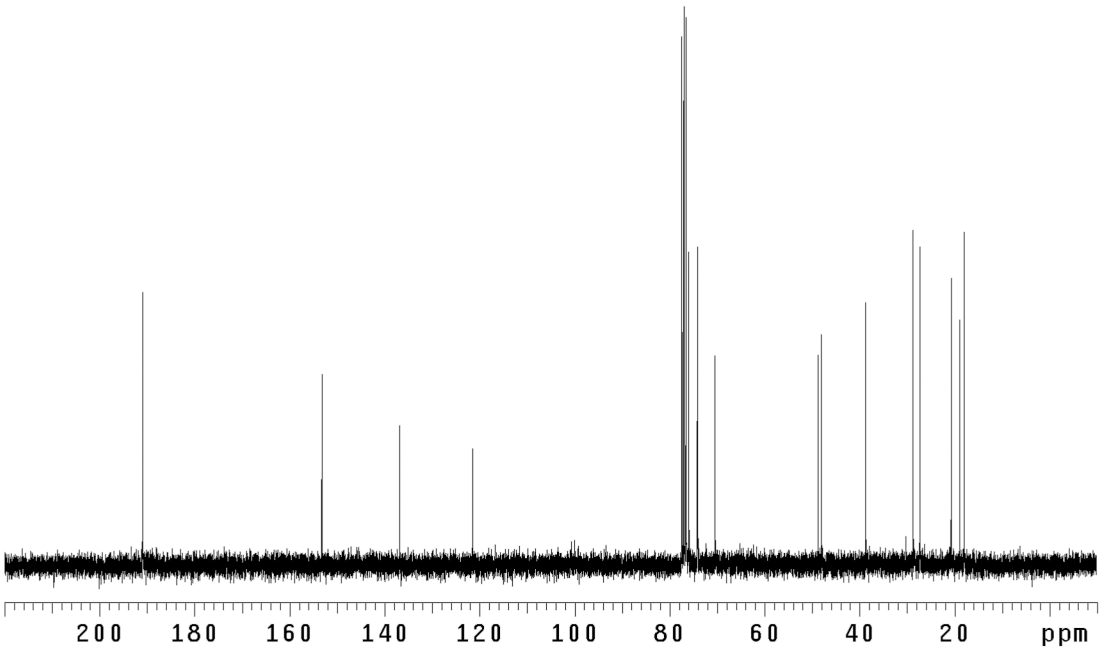

Figure B.90 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **278**.

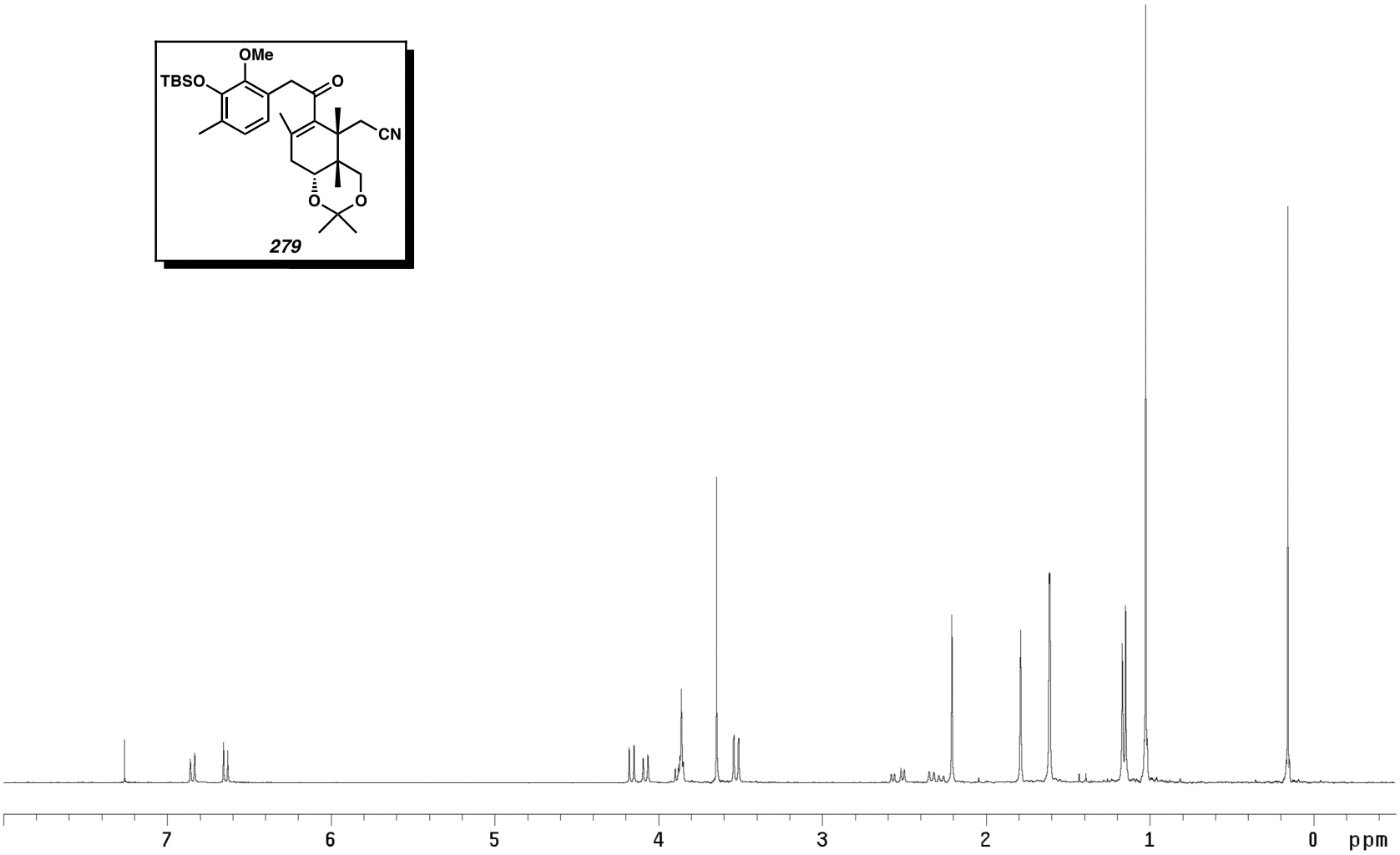

Figure B.91 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **279**.

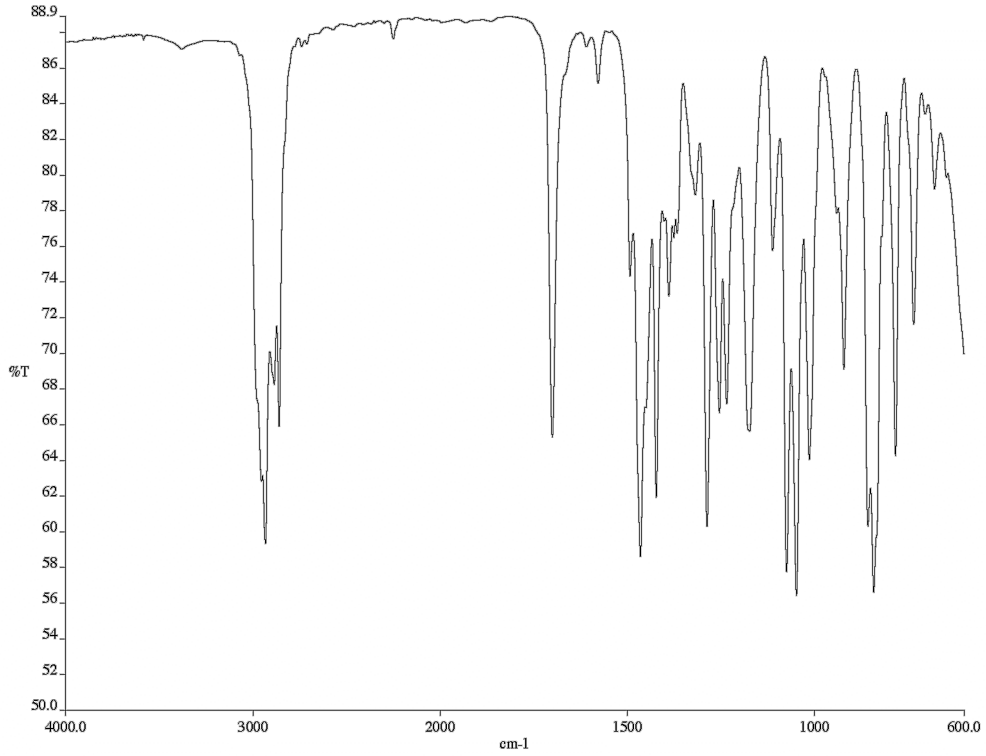

Figure B.92 Infrared spectrum (thin film/NaCl) of compound **279**.

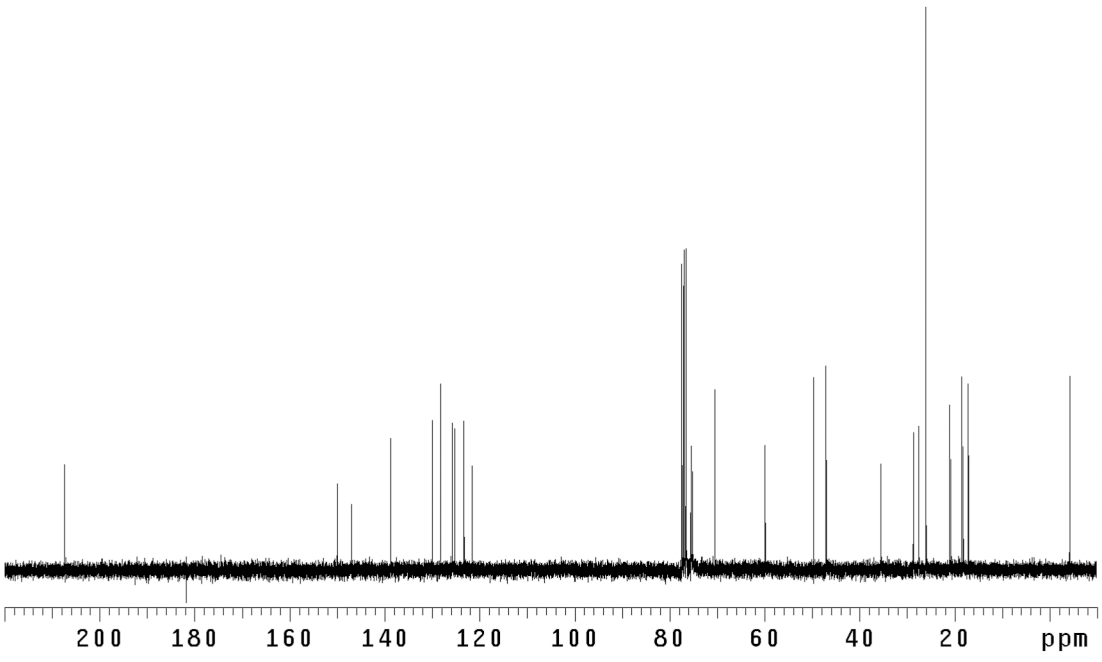

Figure B.93 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **279**.

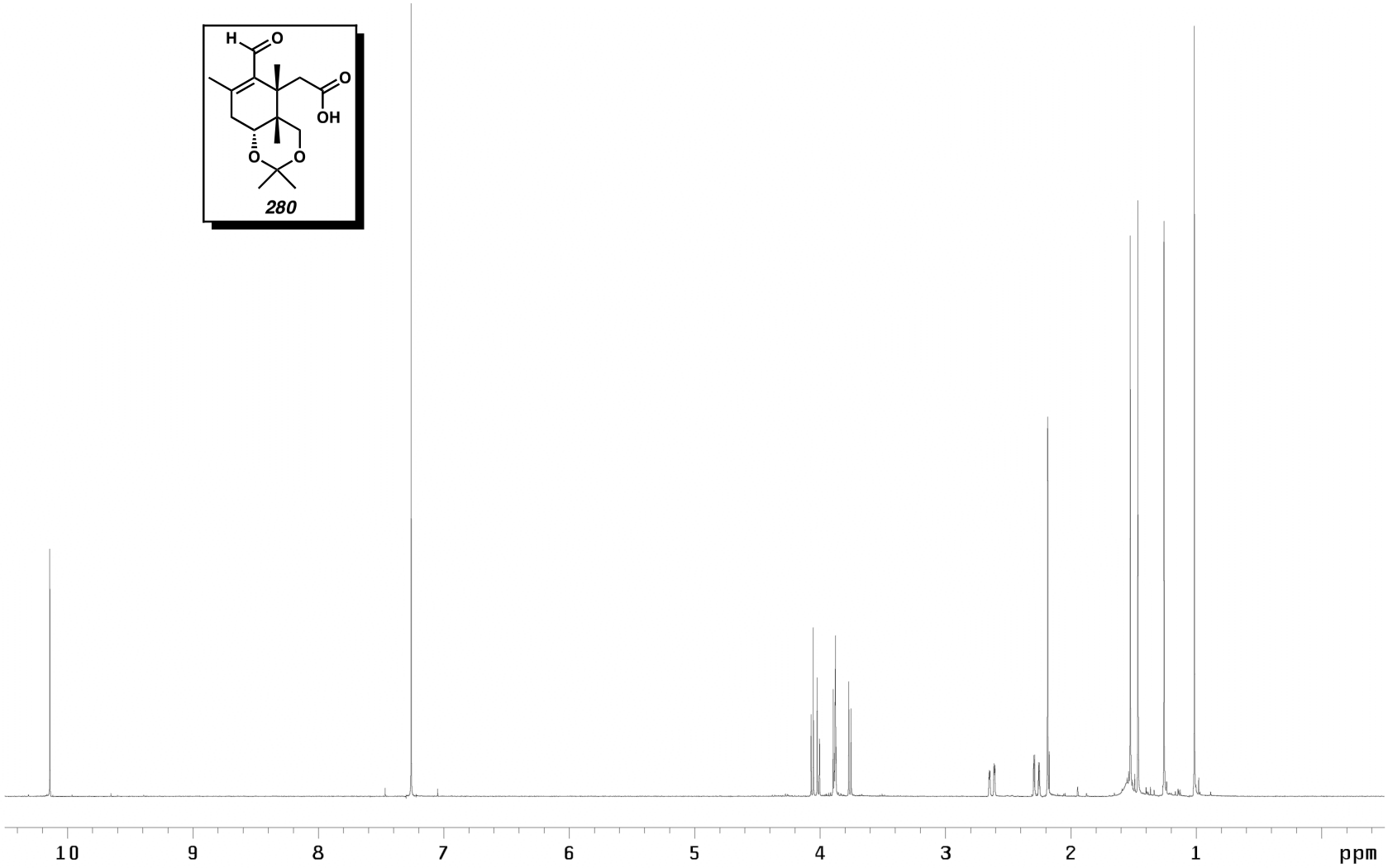

Figure B.94 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **280**.

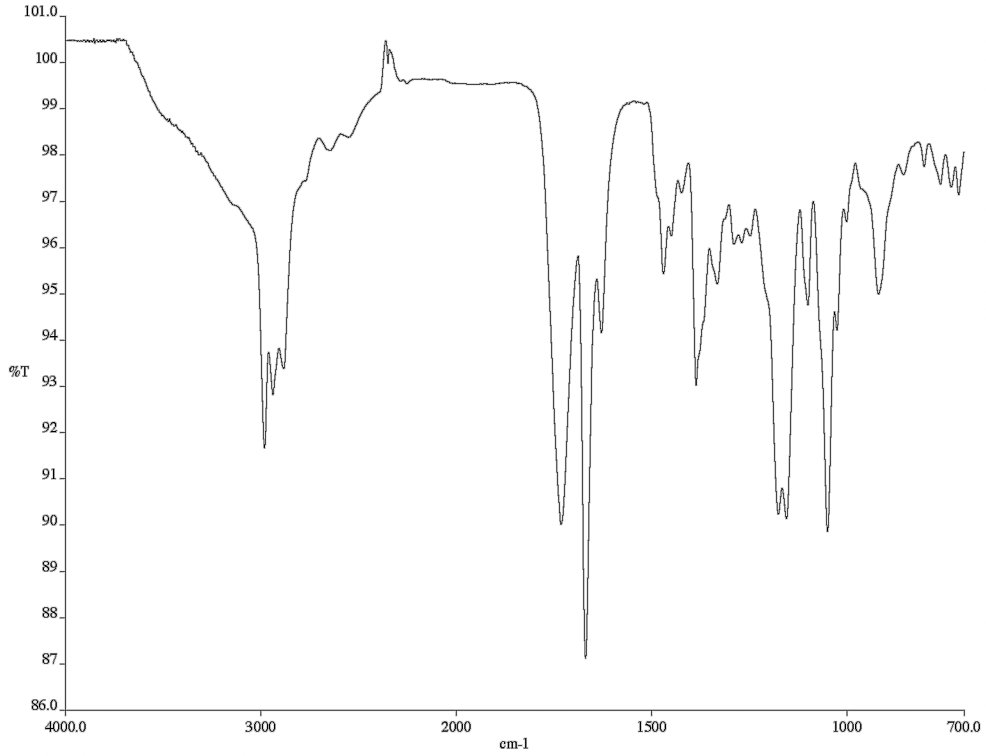

Figure B.95 Infrared spectrum (thin film/NaCl) of compound **280**.

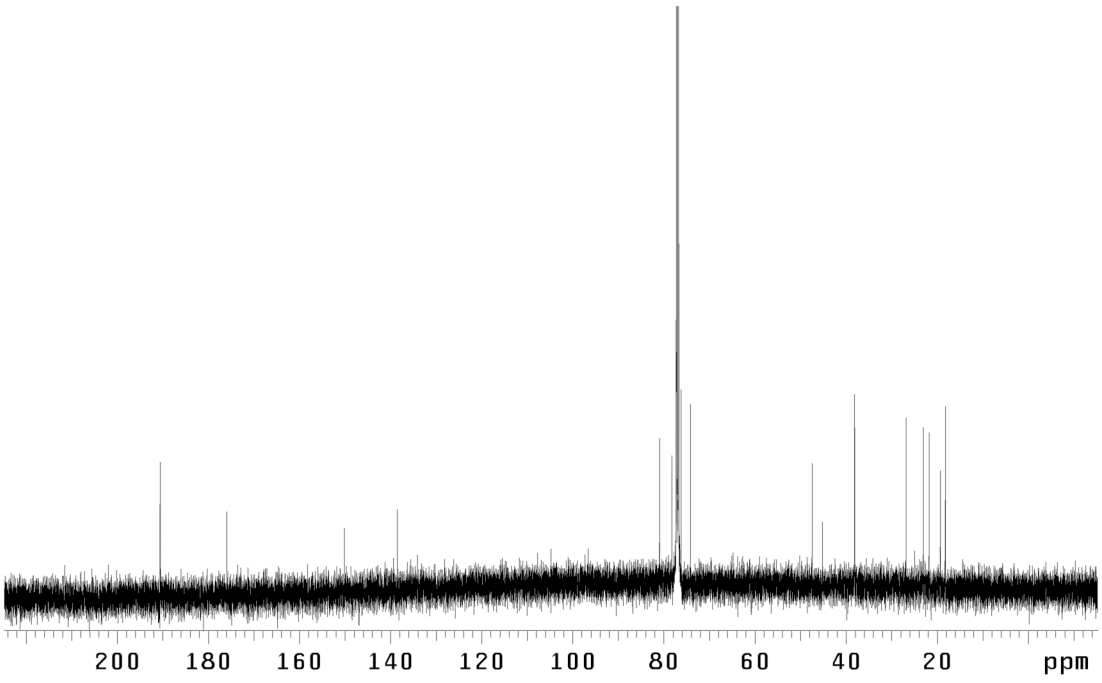

Figure B.96 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **280**.

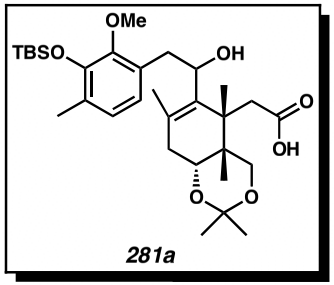

Figure B.97 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **281a**.

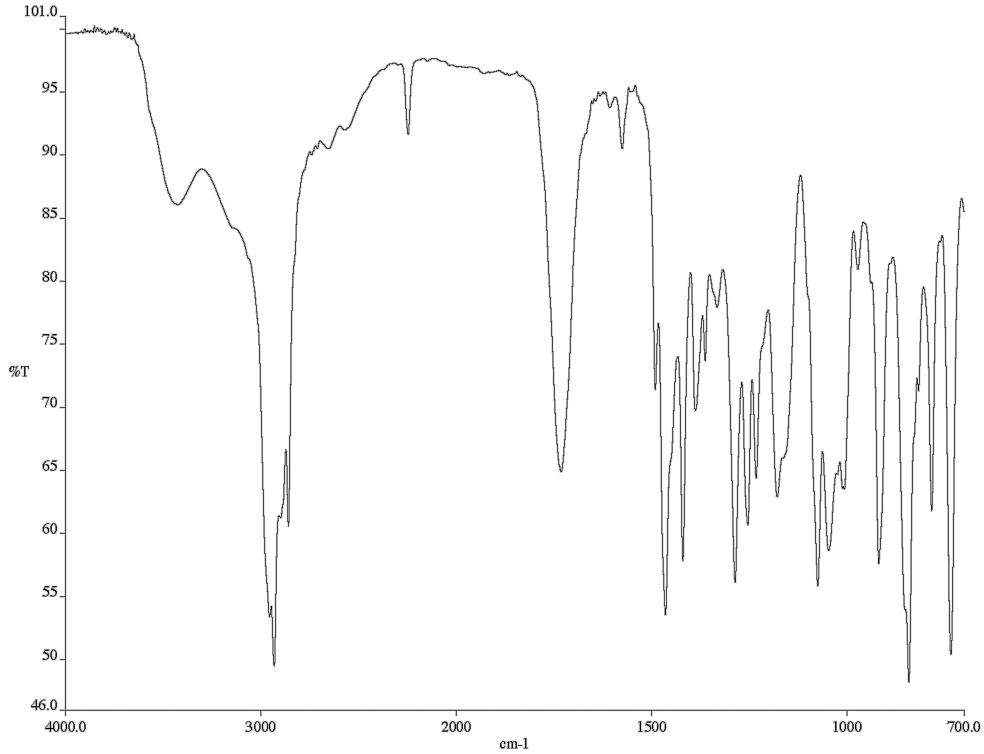

Figure B.98 Infrared spectrum (thin film/NaCl) of compound **281a**.

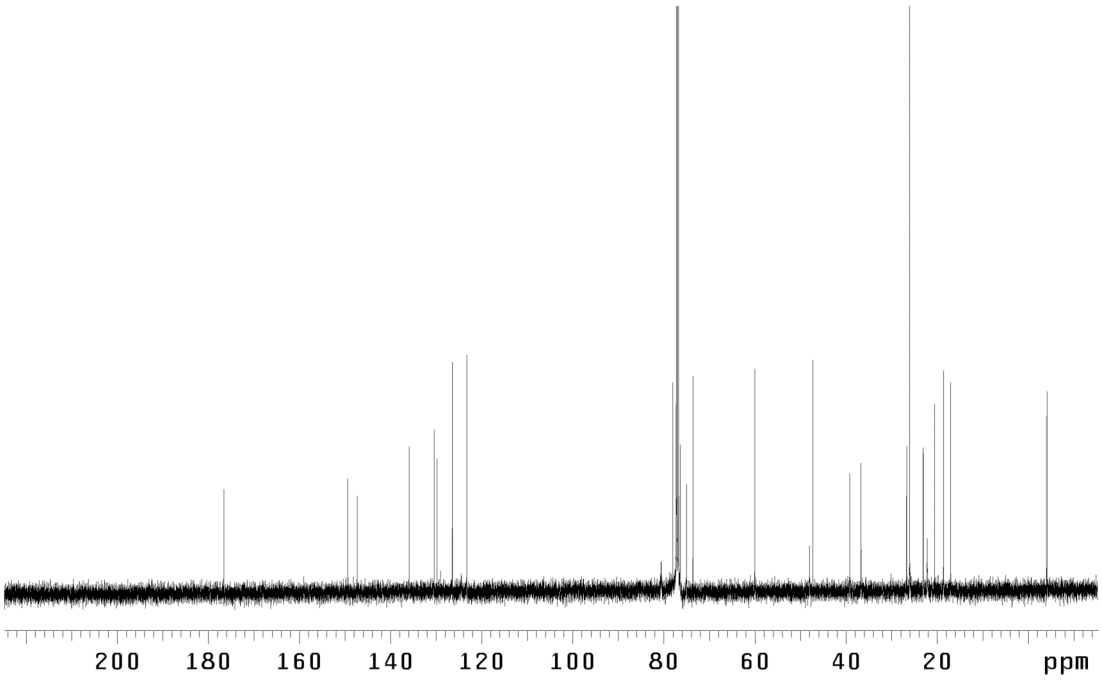

Figure B.99 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **281a**.

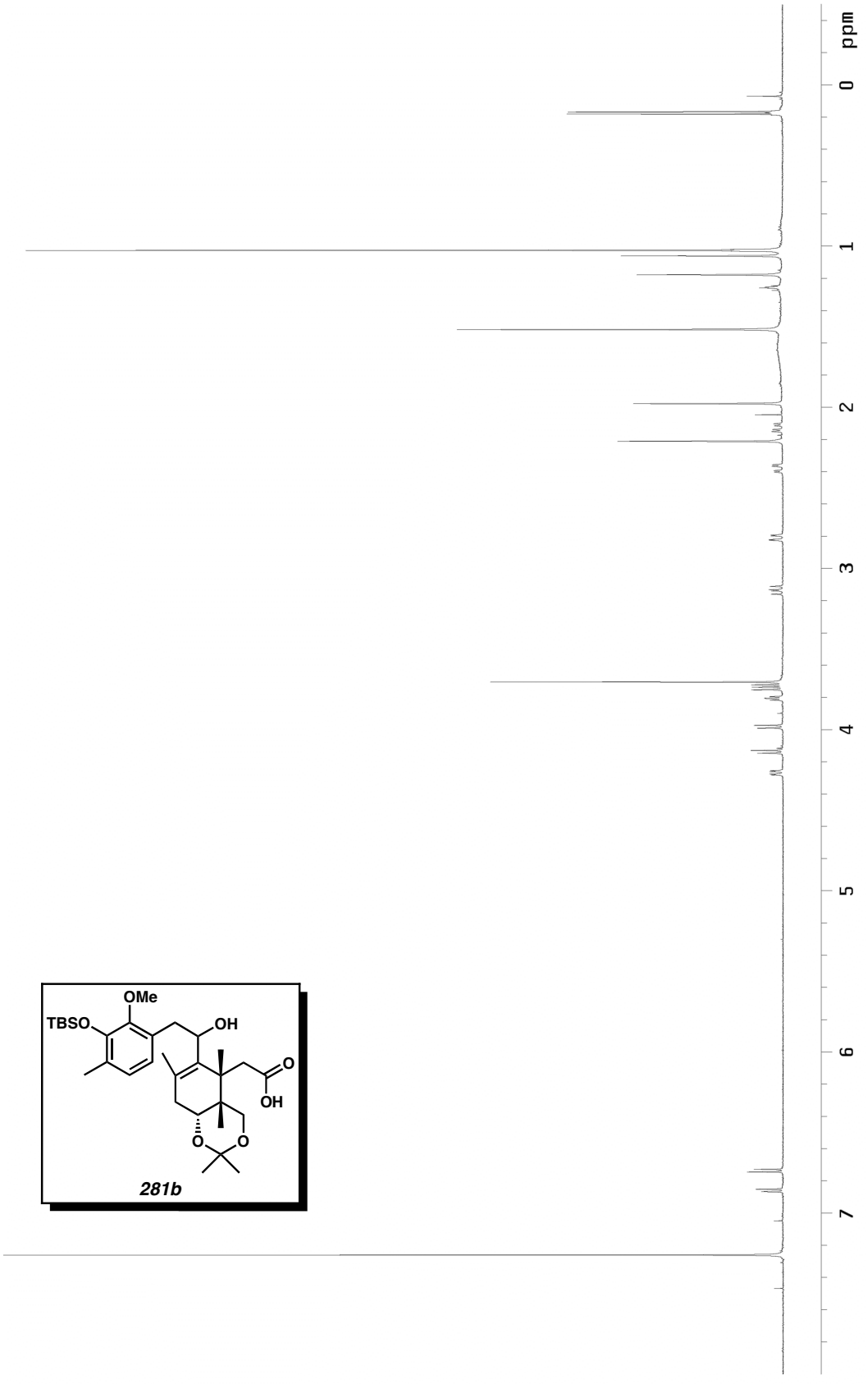

Figure B.100 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **281b**.

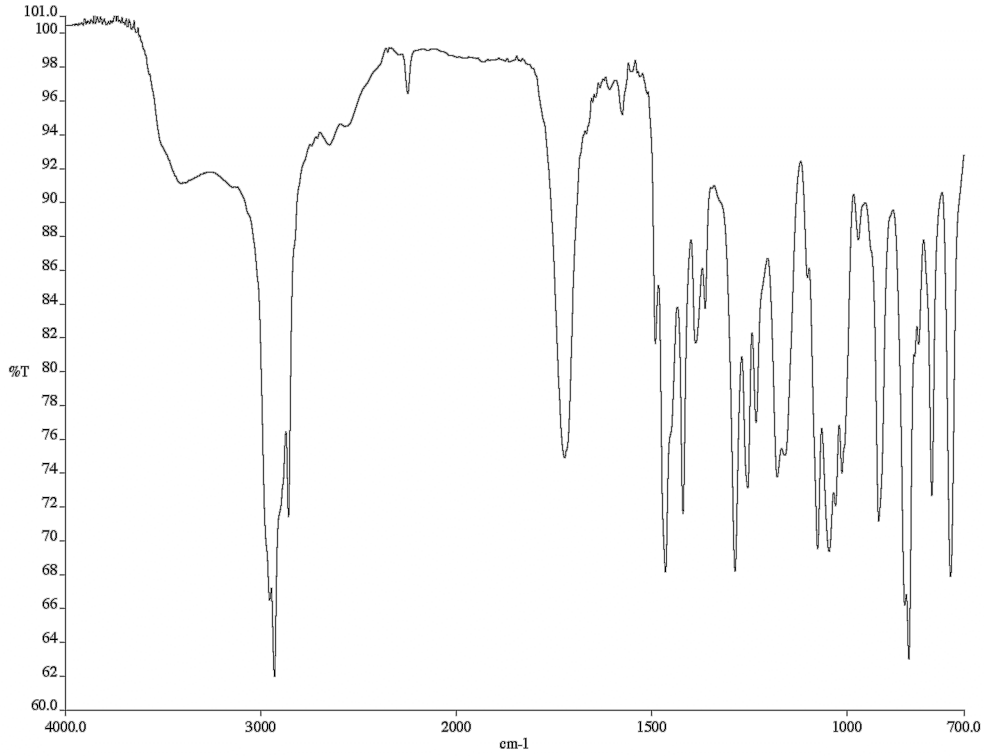

Figure B.101 Infrared spectrum (thin film/NaCl) of compound **281b**.

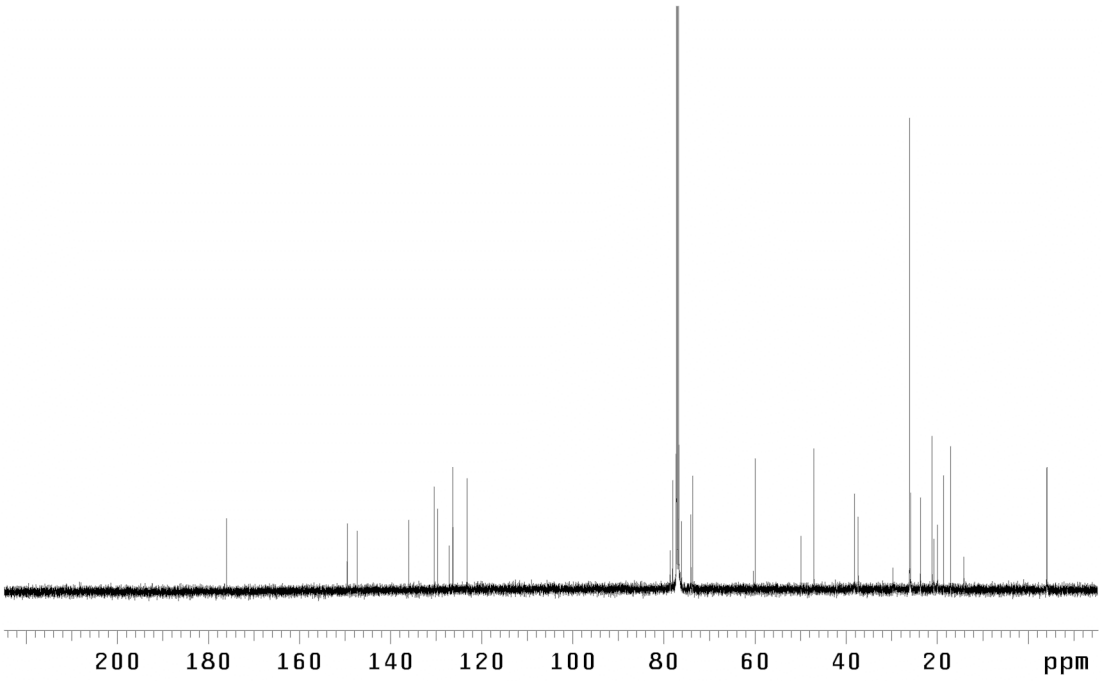

Figure B.102 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **281b**.

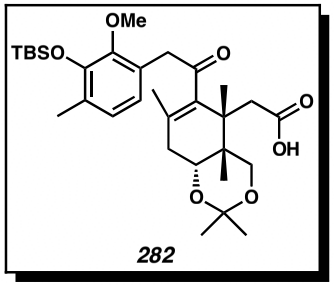

Figure B.103 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **282**.

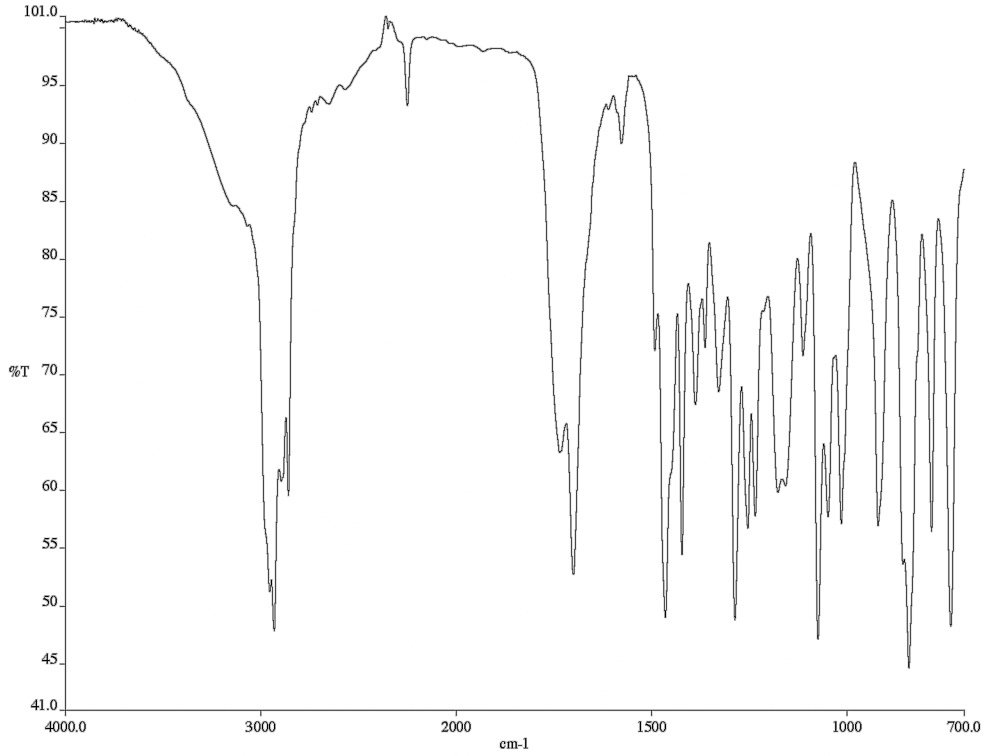

Figure B.104 Infrared spectrum (thin film/NaCl) of compound **282**.

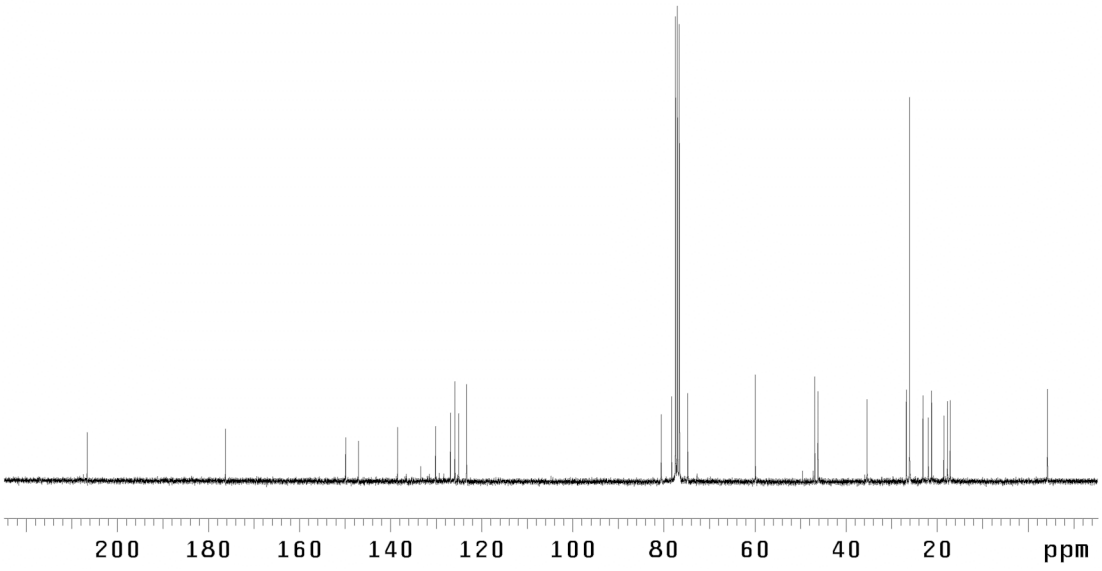

Figure B.105 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **282**.

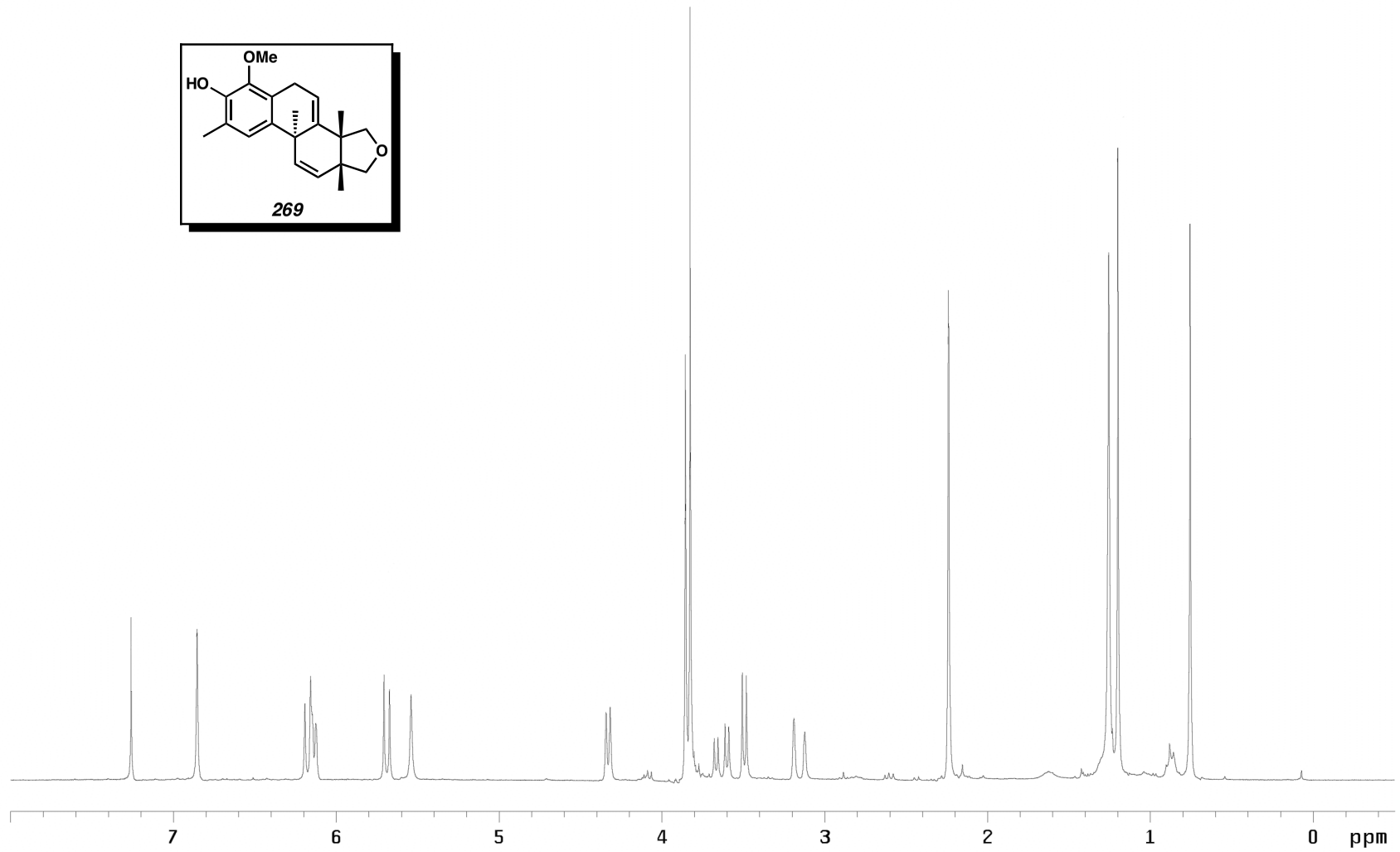

Figure B.106 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **269**.

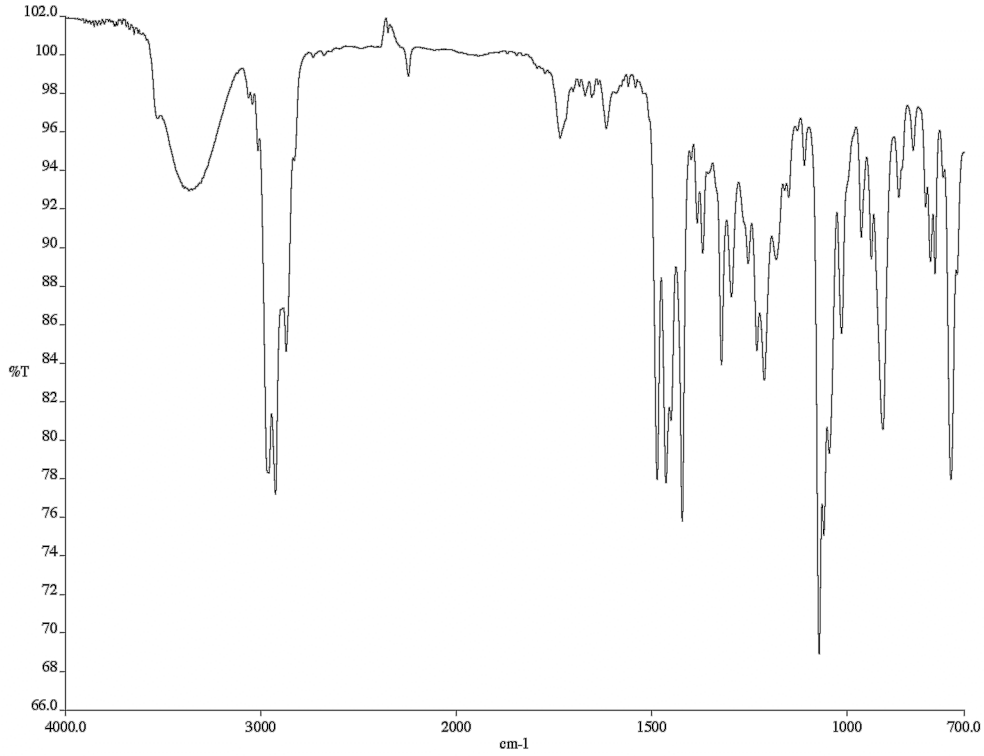

Figure B.107 Infrared spectrum (thin film/NaCl) of compound **269**.

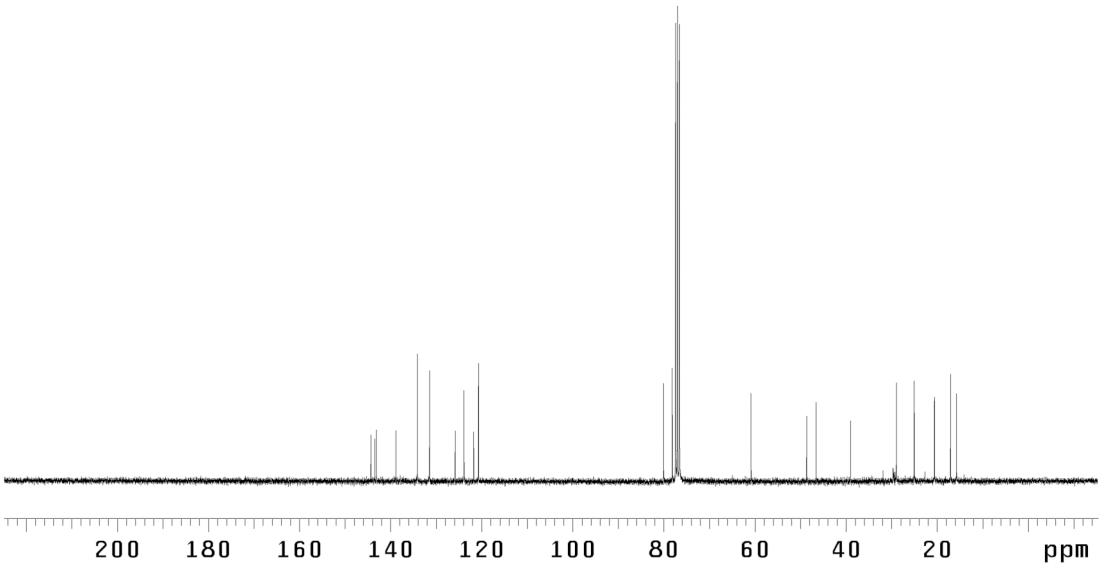

Figure B.108 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **269**.

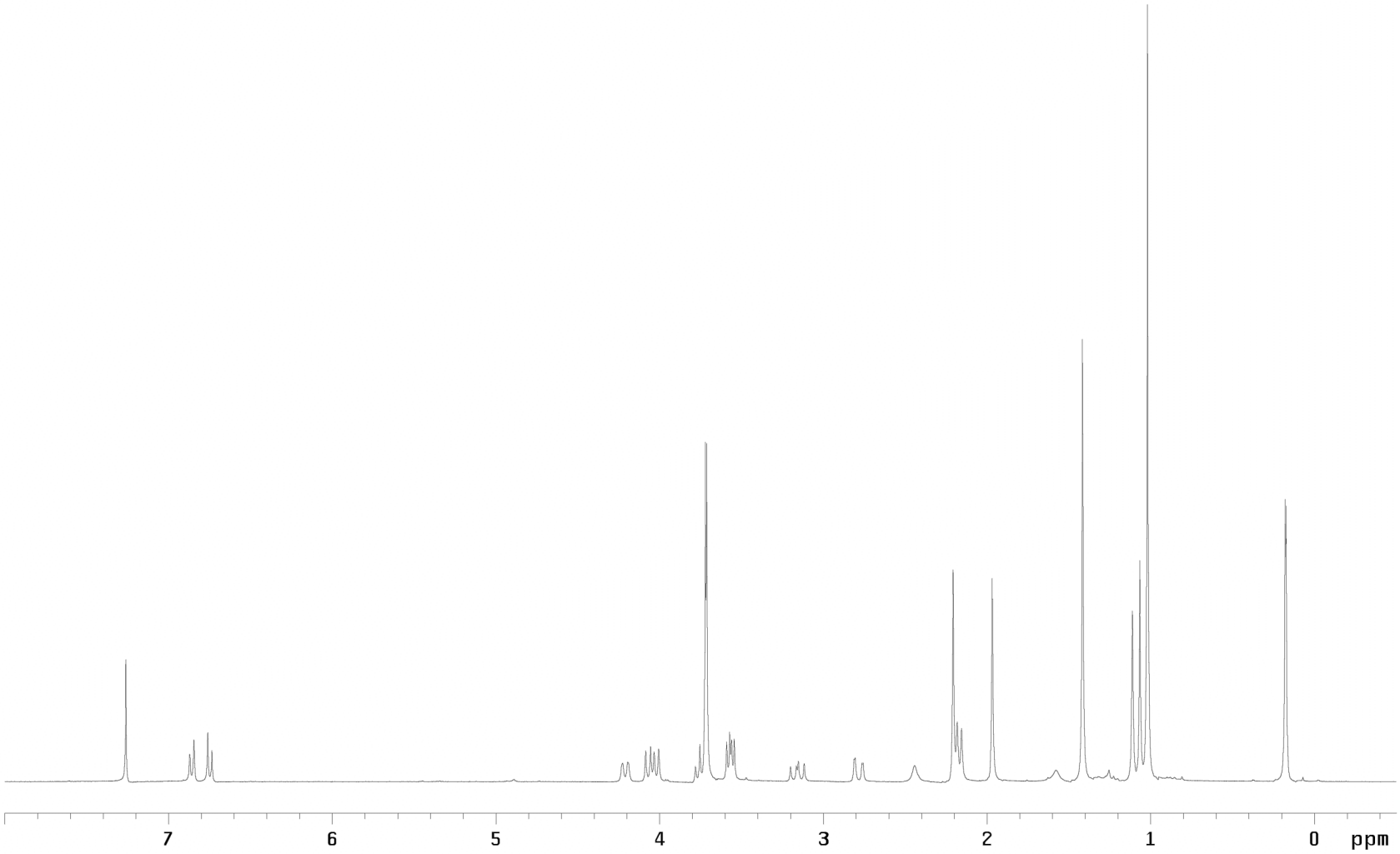

Figure B.109 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **283**.

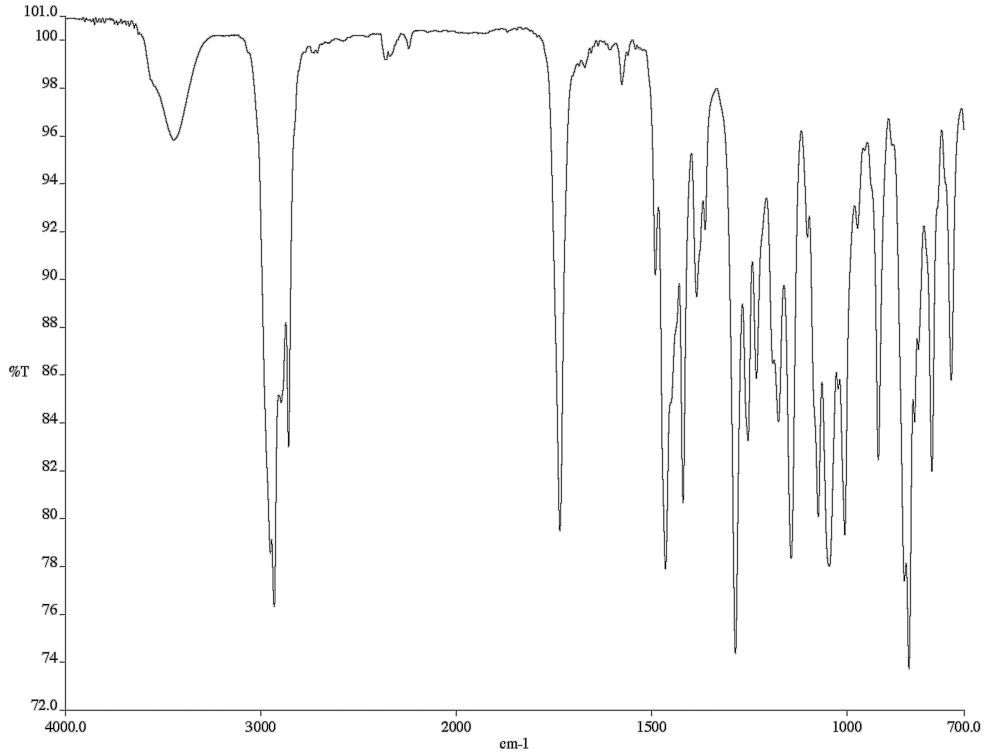

Figure B.110 Infrared spectrum (thin film/NaCl) of compound **283**.

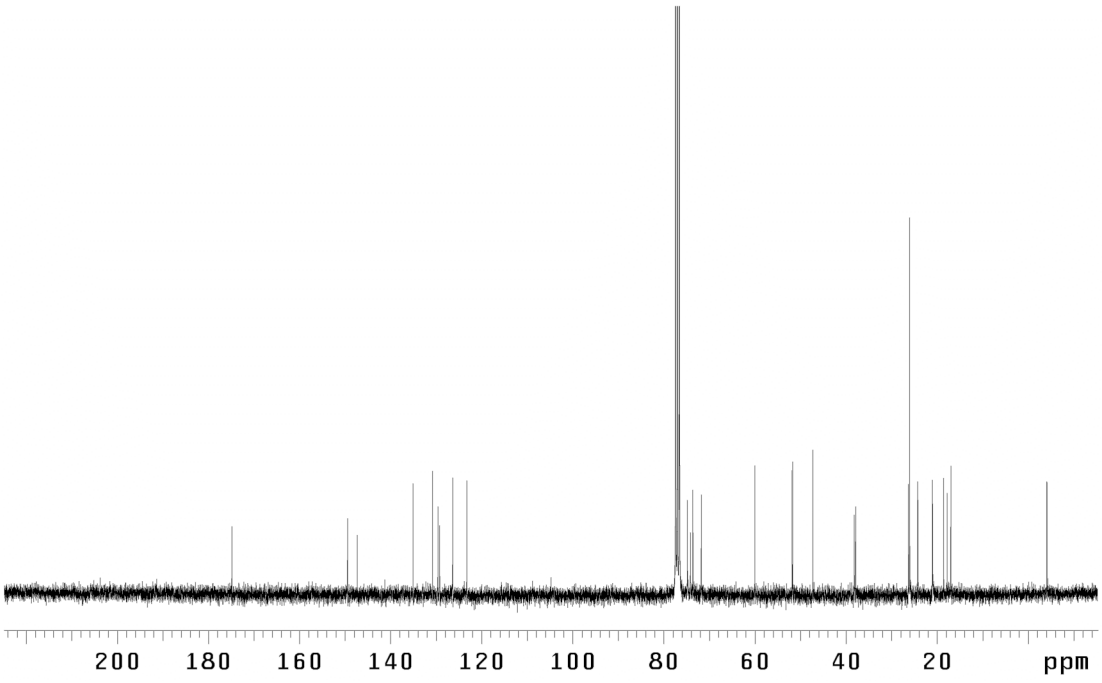

Figure B.111 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **283**.

CALIFORNIA INSTITUTE OF TECHNOLOGY

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Crystal Structure Analysis of:

Allylic Alcohol **248** (DCB30)

(CCDC 277462)

Contents:

Figure B.112 Representation of Allylic Alcohol **248**

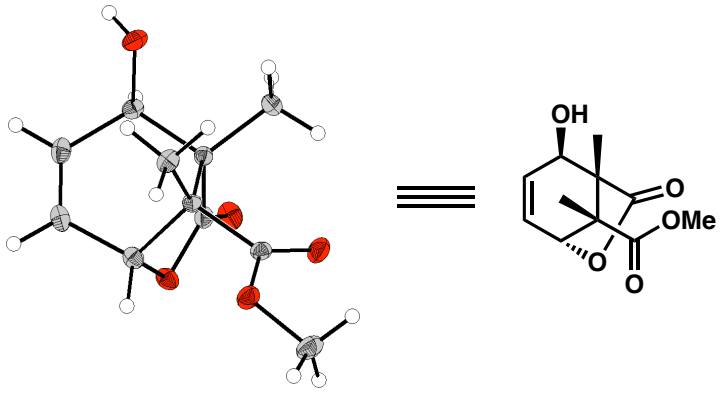

Table 1. Crystal data and structure refinement for DCB30 (CCDC 277462).

Empirical formula	$C_{11}H_{14}O_5$
Formula weight	226.22
Crystallization Solvent	Heptane/diethylether
Crystal Habit	Fragment
Crystal size	0.41 x 0.24 x 0.16 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 8068 reflections used in lattice determination	2.74 to 39.14°
Unit cell dimensions	a = 8.5469(4) Å b = 8.7203(4) Å c = 14.1988(6) Å
Volume	1058.26(8) $Å^3$
Z	4
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Density (calculated)	1.420 Mg/m^3
F(000)	480
Data collection program	Bruker SMART v5.630
θ range for data collection	2.74 to 40.70°
Completeness to θ = 40.70°	92.3 %
Index ranges	$-15 \le h \le 13$, $-15 \le k \le 15$, $-25 \le l \le 23$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	19516
Independent reflections	6114 [R_{int}= 0.0607]
Absorption coefficient	0.113 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9822 and 0.9553

Table 1 (cont.)

Structure Solution and Refinement

Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	6114 / 0 / 201
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.304
Final R indices [I>2σ(I), 4485 reflections]	R1 = 0.0415, *w*R2 = 0.0690
R indices (all data)	R1 = 0.0621, *w*R2 = 0.0715
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Absolute structure determination	Not possible to reliably determine absolute configuration
Absolute structure parameter	-0.2(6)
Largest diff. peak and hole	0.427 and -0.273 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB30 (CCDC 277462). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	1165(1)	11191(1)	7918(1)	16(1)
O(2)	1872(1)	13004(1)	8946(1)	21(1)
O(3)	4173(1)	9617(1)	8803(1)	21(1)
O(4)	3237(1)	7678(1)	7931(1)	18(1)
O(5)	-878(1)	9680(1)	10618(1)	19(1)
C(1)	-1241(1)	9774(1)	8195(1)	17(1)
C(2)	492(1)	9626(1)	8002(1)	15(1)
C(3)	1380(1)	8989(1)	8868(1)	12(1)
C(4)	1180(1)	10414(1)	9520(1)	12(1)
C(5)	-565(1)	10628(1)	9813(1)	14(1)
C(6)	-1706(1)	10259(1)	9031(1)	17(1)
C(7)	1471(1)	11701(1)	8802(1)	14(1)
C(8)	2214(1)	10544(1)	10395(1)	16(1)
C(9)	763(1)	7465(1)	9253(1)	16(1)
C(10)	3095(1)	8815(1)	8558(1)	14(1)
C(11)	4809(1)	7460(1)	7566(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for DCB30 (CCDC 277462).

O(1)-C(7)	1.3559(11)	C(8)-C(4)-C(7)	113.05(7)
O(1)-C(2)	1.4855(11)	C(8)-C(4)-C(3)	118.58(7)
O(2)-C(7)	1.2050(11)	C(7)-C(4)-C(3)	99.84(6)
O(3)-C(10)	1.2082(11)	C(8)-C(4)-C(5)	109.06(7)
O(4)-C(10)	1.3387(11)	C(7)-C(4)-C(5)	104.16(7)
O(4)-C(11)	1.4519(12)	C(3)-C(4)-C(5)	111.03(7)
O(5)-C(5)	1.4350(11)		
O(5)-H(5A)	0.783(13)	O(5)-C(5)-C(6)	110.00(7)
C(1)-C(6)	1.3223(13)	O(5)-C(5)-C(4)	108.75(7)
C(1)-C(2)	1.5115(13)	C(6)-C(5)-C(4)	113.26(7)
C(1)-H(1)	0.973(11)	O(5)-C(5)-H(5)	109.9(6)
C(2)-C(3)	1.5486(12)	C(6)-C(5)-H(5)	108.8(6)
C(2)-H(2)	0.989(9)		
C(3)-C(9)	1.5301(12)	C(4)-C(5)-H(5)	106.0(6)
C(3)-C(10)	1.5380(13)	C(1)-C(6)-C(5)	122.26(8)
C(3)-C(4)	1.5586(12)	C(1)-C(6)-H(6)	120.8(6)
C(4)-C(8)	1.5294(12)	C(5)-C(6)-H(6)	116.9(6)
C(4)-C(7)	1.5365(12)	O(2)-C(7)-O(1)	121.44(8)
C(4)-C(5)	1.5600(12)	O(2)-C(7)-C(4)	128.47(8)
C(5)-C(6)	1.5120(13)	O(1)-C(7)-C(4)	110.07(7)
C(5)-H(5)	0.994(11)	C(4)-C(8)-H(8A)	108.8(6)
C(6)-H(6)	0.951(10)	C(4)-C(8)-H(8B)	108.5(7)
C(8)-H(8A)	1.025(12)	H(8A)-C(8)-H(8B)	109.1(9)
C(8)-H(8B)	0.977(11)	C(4)-C(8)-H(8C)	112.1(7)
C(8)-H(8C)	0.976(12)	H(8A)-C(8)-H(8C)	105.7(10)
C(9)-H(9A)	0.987(11)	H(8B)-C(8)-H(8C)	112.6(10)
C(9)-H(9B)	0.952(11)	C(3)-C(9)-H(9A)	110.7(6)
C(9)-H(9C)	0.952(12)	C(3)-C(9)-H(9B)	111.9(7)
C(11)-H(11A)	0.967(12)	H(9A)-C(9)-H(9B)	104.0(9)
C(11)-H(11B)	0.940(13)	C(3)-C(9)-H(9C)	110.2(7)
C(11)-H(11C)	0.962(12)	H(9A)-C(9)-H(9C)	108.6(9)
		H(9B)-C(9)-H(9C)	111.2(10)
C(7)-O(1)-C(2)	107.57(7)	O(3)-C(10)-O(4)	123.45(8)
C(10)-O(4)-C(11)	114.73(8)	O(3)-C(10)-C(3)	125.99(8)
C(5)-O(5)-H(5A)	104.1(10)	O(4)-C(10)-C(3)	110.48(7)
C(6)-C(1)-C(2)	119.00(8)	O(4)-C(11)-H(11A)	107.9(7)
C(6)-C(1)-H(1)	122.2(6)	O(4)-C(11)-H(11B)	106.0(8)
C(2)-C(1)-H(1)	118.8(6)	H(11A)-C(11)-H(11B)	111.7(10)
O(1)-C(2)-C(1)	108.39(7)	O(4)-C(11)-H(11C)	108.7(7)
O(1)-C(2)-C(3)	101.75(7)	H(11A)-C(11)-H(11C)	111.6(9)
C(1)-C(2)-C(3)	111.51(7)	H(11B)-C(11)-H(11C)	110.6(10)
O(1)-C(2)-H(2)	105.7(6)		
C(1)-C(2)-H(2)	112.9(6)		
C(3)-C(2)-H(2)	115.6(5)		
C(9)-C(3)-C(10)	110.19(7)		
C(9)-C(3)-C(2)	115.23(8)		
C(10)-C(3)-C(2)	105.96(7)		
C(9)-C(3)-C(4)	116.29(7)		
C(10)-C(3)-C(4)	110.65(7)		
C(2)-C(3)-C(4)	97.55(7)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB30 (CCDC 277462). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	203(3)	149(3)	142(3)	30(2)	-26(3)	-27(3)
O(2)	239(4)	146(3)	239(3)	28(3)	-42(3)	-47(3)
O(3)	154(3)	239(4)	236(3)	-25(3)	30(3)	-54(3)
O(4)	154(3)	204(3)	196(3)	-37(3)	29(3)	16(3)
O(5)	178(3)	213(3)	185(3)	47(3)	60(3)	34(3)
C(1)	159(4)	156(4)	203(4)	11(3)	-58(3)	-11(4)
C(2)	167(4)	130(4)	149(4)	-4(3)	-18(3)	-17(4)
C(3)	129(4)	116(4)	122(3)	8(3)	3(3)	-2(3)
C(4)	120(4)	119(4)	124(3)	11(3)	-4(3)	0(3)
C(5)	147(4)	127(4)	144(4)	9(3)	21(3)	14(3)
C(6)	129(4)	162(4)	230(4)	24(3)	-19(3)	10(4)
C(7)	118(4)	149(4)	157(4)	18(3)	-10(3)	3(3)
C(8)	163(4)	190(5)	138(4)	6(3)	-24(3)	-5(4)
C(9)	154(4)	117(4)	201(4)	11(3)	21(4)	-5(4)
C(10)	148(4)	151(4)	120(4)	32(3)	18(3)	10(4)
C(11)	191(5)	259(6)	230(5)	-5(4)	75(4)	52(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for DCB30 (CCDC 277462).

	x	y	z	U_{iso}
H(5A)	-1603(15)	10067(15)	10856(9)	35(4)
H(1)	-1977(13)	9484(13)	7703(7)	18(3)
H(2)	719(11)	9112(11)	7397(7)	6(2)
H(5)	-680(12)	11727(13)	9986(7)	15(3)
H(6)	-2786(12)	10404(12)	9164(7)	14(3)
H(8A)	1942(13)	11540(14)	10742(8)	23(3)
H(8B)	1993(13)	9673(14)	10807(7)	23(3)
H(8C)	3321(14)	10627(15)	10235(8)	29(3)
H(9A)	1386(12)	7128(12)	9800(8)	17(3)
H(9B)	-271(13)	7561(12)	9496(8)	17(3)
H(9C)	815(14)	6693(14)	8779(8)	27(3)
H(11A)	5491(14)	7243(13)	8093(8)	20(3)
H(11B)	4748(14)	6625(15)	7148(9)	29(3)
H(11C)	5121(13)	8376(14)	7239(8)	20(3)

Table 6. Hydrogen bonds for DCB30 (CCDC 277462) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-H(5A)...O(2)#1	0.783(13)	2.147(13)	2.8567(10)	151.0(13)

Symmetry transformations used to generate equivalent atoms:
#1 x-1/2,-y+5/2,-z+2

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Crystal Structure Analysis of:

Allylic Alcohol **253** (DCB31)

(CCDC 283708)

Contents:

Figure B.113 Representation of Allylic Alcohol **253**

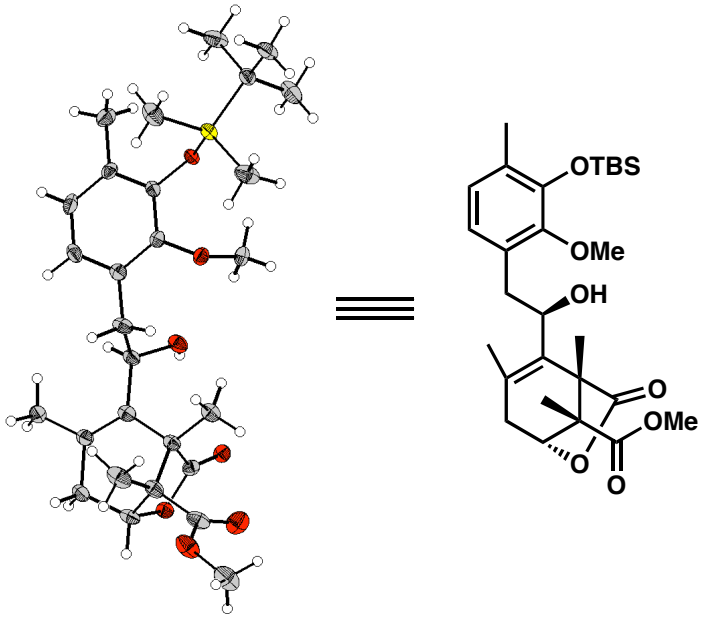

Table 1. Crystal data and structure refinement for DCB31 (CCDC 283708).

Empirical formula	$C_{28}H_{42}O_7$ Si
Formula weight	518.71
Crystallization Solvent	EtOAc/heptane
Crystal Habit	Block
Crystal size	0.32 x 0.31 x 0.22 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 15772 reflections used in lattice determination	2.32 to 28.21°	
Unit cell dimensions	a = 12.6604(8) Å b = 15.4100(10) Å c = 15.7147(10) Å	α= 81.0750(10)° β= 66.6280(10)° γ = 87.6100(10)°
Volume	2779.6(3) $Å^3$	
Z	4	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.240 Mg/m^3	
F(000)	1120	
Data collection program	Bruker SMART v5.630	
θ range for data collection	1.75 to 28.27°	
Completeness to θ = 28.27°	92.1 %	
Index ranges	$-16 \le h \le 16$, $-20 \le k \le 19$, $-20 \le l \le 20$	
Data collection scan type	ω scans at 7 ϕ settings	
Data reduction program	Bruker SAINT v6.45A	
Reflections collected	56601	
Independent reflections	12691 [R_{int}= 0.0626]	
Absorption coefficient	0.127 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9725 and 0.9604	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12691 / 0 / 985
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.502
Final R indices [I>2σ(I), 7901 reflections]	R1 = 0.0455, wR2 = 0.0721
R indices (all data)	R1 = 0.0839, wR2 = 0.0763
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Largest diff. peak and hole	0.492 and -0.382 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB31 (CCDC 283708). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Si(1)	-42(1)	510(1)	2060(1)	21(1)
O(1A)	816(1)	1084(1)	2346(1)	19(1)
O(2A)	1297(1)	2515(1)	937(1)	23(1)
O(3A)	3033(1)	4131(1)	770(1)	26(1)
O(4A)	3980(1)	6315(1)	-2542(1)	37(1)
O(5A)	2422(1)	7069(1)	-2515(1)	38(1)
O(6A)	4208(1)	6810(1)	-903(1)	21(1)
O(7A)	4924(1)	5456(1)	-917(1)	22(1)
C(1A)	646(1)	1925(1)	2572(1)	18(1)
C(2A)	899(1)	2648(1)	1864(1)	18(1)
C(3A)	719(1)	3501(1)	2073(1)	21(1)
C(4A)	306(2)	3607(1)	3011(1)	27(1)
C(5A)	107(2)	2895(1)	3709(1)	26(1)
C(6A)	276(1)	2039(1)	3508(1)	20(1)
C(7A)	81(2)	1260(1)	4263(1)	29(1)
C(8A)	2448(2)	2186(2)	597(2)	28(1)
C(9A)	166(2)	811(2)	810(2)	38(1)
C(10A)	-1553(2)	709(2)	2806(2)	35(1)
C(11A)	375(1)	-659(1)	2268(1)	22(1)
C(12A)	-338(2)	-1258(1)	1990(2)	30(1)
C(13A)	1656(2)	-771(2)	1691(2)	37(1)
C(14A)	138(2)	-938(1)	3311(1)	34(1)
C(15A)	999(2)	4291(1)	1317(1)	25(1)
C(16A)	2140(1)	4736(1)	1130(1)	21(1)
C(17A)	2349(1)	5629(1)	503(1)	20(1)
C(18A)	2866(1)	5673(1)	-583(1)	20(1)
C(19A)	2473(1)	6519(1)	-1042(1)	24(1)
C(20A)	3079(2)	7190(1)	-749(1)	24(1)
C(21A)	2454(2)	7262(1)	276(1)	24(1)
C(22A)	2158(1)	6374(1)	879(1)	22(1)
C(23A)	1640(2)	6435(1)	1910(1)	29(1)
C(24A)	2706(2)	4851(1)	-947(1)	25(1)
C(25A)	4114(2)	5923(1)	-833(1)	20(1)
C(26A)	1171(2)	6613(2)	-701(2)	35(1)
C(27A)	3055(2)	6601(1)	-2107(1)	28(1)
C(28A)	2900(3)	7207(2)	-3527(2)	44(1)
Si(2)	5149(1)	9590(1)	2638(1)	21(1)
O(1B)	4218(1)	9004(1)	2444(1)	20(1)
O(2B)	3743(1)	7599(1)	3860(1)	24(1)
O(3B)	1918(1)	5944(1)	4116(1)	26(1)
O(4B)	1344(1)	3774(1)	7408(1)	34(1)
O(5B)	2918(1)	2985(1)	7232(1)	32(1)
O(6B)	863(1)	3261(1)	5877(1)	21(1)
O(7B)	126(1)	4607(1)	5934(1)	23(1)
C(1B)	4350(1)	8159(1)	2224(1)	17(1)
C(2B)	4097(1)	7444(1)	2943(1)	18(1)

C(3B)	4225(1)	6587(1)	2749(1)	21(1)
C(4B)	4584(1)	6466(1)	1817(1)	24(1)
C(5B)	4779(1)	7171(1)	1112(1)	24(1)
C(6B)	4666(1)	8032(1)	1295(1)	19(1)
C(7B)	4854(2)	8801(1)	525(1)	26(1)
C(8B)	2574(2)	7893(1)	4228(1)	29(1)
C(9B)	5680(2)	8968(1)	3484(2)	29(1)
C(10B)	6404(2)	9919(2)	1510(2)	33(1)
C(11B)	4327(2)	10573(1)	3092(1)	25(1)
C(12B)	5122(2)	11175(1)	3282(2)	32(1)
C(13B)	3289(2)	10289(2)	4020(2)	46(1)
C(14B)	3894(2)	11088(2)	2385(2)	46(1)
C(15B)	3966(2)	5805(1)	3514(1)	25(1)
C(16B)	2821(1)	5350(1)	3733(1)	19(1)
C(17B)	2636(1)	4457(1)	4354(1)	19(1)
C(18B)	2219(1)	4416(1)	5438(1)	19(1)
C(19B)	2690(1)	3582(1)	5839(1)	22(1)
C(20B)	2018(2)	2897(1)	5624(1)	22(1)
C(21B)	2507(2)	2827(1)	4603(1)	22(1)
C(22B)	2764(1)	3713(1)	3988(1)	20(1)
C(23B)	3189(2)	3634(1)	2963(1)	26(1)
C(24B)	2419(2)	5244(1)	5771(1)	23(1)
C(25B)	955(2)	4150(1)	5785(1)	19(1)
C(26B)	3999(2)	3506(2)	5394(2)	29(1)
C(27B)	2231(2)	3484(1)	6908(1)	25(1)
C(28B)	2541(2)	2794(2)	8236(1)	35(1)

Table 3. Bond lengths [Å] and angles [°] for DCB31 (CCDC 283708).

Si(1)-O(1A)	1.6602(11)	C(17A)-C(22A)	1.342(2)
Si(1)-C(10A)	1.847(2)	C(17A)-C(18A)	1.559(2)
Si(1)-C(9A)	1.860(2)	C(18A)-C(25A)	1.520(2)
Si(1)-C(11A)	1.8719(17)	C(18A)-C(24A)	1.519(2)
O(1A)-C(1A)	1.3834(18)	C(18A)-C(19A)	1.552(2)
O(2A)-C(2A)	1.3854(18)	C(19A)-C(26A)	1.525(2)
O(2A)-C(8A)	1.440(2)	C(19A)-C(27A)	1.525(2)
O(3A)-C(16A)	1.4266(19)	C(19A)-C(20A)	1.538(2)
O(3A)-H(3A)	0.92(2)	C(20A)-C(21A)	1.506(2)
O(4A)-C(27A)	1.203(2)	C(20A)-H(20A)	1.015(16)
O(5A)-C(27A)	1.337(2)	C(21A)-C(22A)	1.505(2)
O(5A)-C(28A)	1.442(2)	C(21A)-H(21A)	0.976(15)
O(6A)-C(25A)	1.3611(19)	C(21A)-H(21B)	1.002(16)
O(6A)-C(20A)	1.4637(19)	C(22A)-C(23A)	1.505(2)
O(7A)-C(25A)	1.2020(18)	C(23A)-H(23A)	0.95(2)
C(1A)-C(2A)	1.394(2)	C(23A)-H(23B)	0.963(19)
C(1A)-C(6A)	1.393(2)	C(23A)-H(23C)	0.98(2)
C(2A)-C(3A)	1.392(2)	C(24A)-H(24A)	0.973(16)
C(3A)-C(4A)	1.389(2)	C(24A)-H(24B)	1.022(16)
C(3A)-C(15A)	1.508(2)	C(24A)-H(24C)	0.984(16)
C(4A)-C(5A)	1.378(2)	C(26A)-H(26A)	1.042(16)
C(4A)-H(4A)	0.933(16)	C(26A)-H(26B)	0.994(18)
C(5A)-C(6A)	1.392(2)	C(26A)-H(26C)	0.944(18)
C(5A)-H(5A)	0.918(15)	C(28A)-H(28A)	0.96(2)
C(6A)-C(7A)	1.504(2)	C(28A)-H(28B)	1.00(2)
C(7A)-H(7A1)	0.978(19)	C(28A)-H(28C)	0.95(2)
C(7A)-H(7A2)	0.991(17)	Si(2)-O(1B)	1.6614(12)
C(7A)-H(7A3)	0.977(17)	Si(2)-C(9B)	1.844(2)
C(8A)-H(8A1)	0.96(2)	Si(2)-C(10B)	1.864(2)
C(8A)-H(8A2)	0.96(2)	Si(2)-C(11B)	1.8712(17)
C(8A)-H(8A3)	0.980(18)	O(1B)-C(1B)	1.3842(18)
C(9A)-H(9A1)	0.96(2)	O(2B)-C(2B)	1.3868(18)
C(9A)-H(9A2)	0.99(2)	O(2B)-C(8B)	1.441(2)
C(9A)-H(9A3)	1.04(2)	O(3B)-C(16B)	1.4298(19)
C(10A)-H(10A)	1.032(18)	O(3B)-H(3B)	0.98(3)
C(10A)-H(10B)	0.95(2)	O(4B)-C(27B)	1.2025(19)
C(10A)-H(10C)	0.91(2)	O(5B)-C(27B)	1.3347(19)
C(11A)-C(13A)	1.530(2)	O(5B)-C(28B)	1.441(2)
C(11A)-C(14A)	1.537(2)	O(6B)-C(25B)	1.3590(19)
C(11A)-C(12A)	1.539(2)	O(6B)-C(20B)	1.4668(19)
C(12A)-H(12A)	0.995(16)	O(7B)-C(25B)	1.2030(18)
C(12A)-H(12B)	1.002(16)	C(1B)-C(6B)	1.396(2)
C(12A)-H(12C)	0.939(17)	C(1B)-C(2B)	1.396(2)
C(13A)-H(13A)	0.986(18)	C(2B)-C(3B)	1.389(2)
C(13A)-H(13B)	0.990(17)	C(3B)-C(4B)	1.391(2)
C(13A)-H(13C)	1.072(16)	C(3B)-C(15B)	1.509(2)
C(14A)-H(14A)	1.043(18)	C(4B)-C(5B)	1.378(2)
C(14A)-H(14B)	1.016(17)	C(4B)-H(4B)	0.941(15)
C(14A)-H(14C)	0.982(18)	C(5B)-C(6B)	1.391(2)
C(15A)-C(16A)	1.525(2)	C(5B)-H(5B)	0.956(15)
C(15A)-H(15A)	0.967(16)	C(6B)-C(7B)	1.508(2)
C(15A)-H(15B)	1.012(16)	C(7B)-H(7B1)	0.984(18)
C(16A)-C(17A)	1.529(2)	C(7B)-H(7B2)	0.973(18)
C(16A)-H(16A)	1.032(13)	C(7B)-H(7B3)	0.959(18)

C(8B)-H(8B1)	0.994(16)
C(8B)-H(8B2)	0.949(17)
C(8B)-H(8B3)	0.976(19)
C(9B)-H(9B1)	1.021(19)
C(9B)-H(9B2)	0.968(18)
C(9B)-H(9B3)	0.910(18)
C(10B)-H(10D)	0.929(19)
C(10B)-H(10E)	0.972(19)
C(10B)-H(10F)	1.041(19)
C(11B)-C(14B)	1.529(3)
C(11B)-C(12B)	1.537(3)
C(11B)-C(13B)	1.541(3)
C(12B)-H(12D)	0.961(18)
C(12B)-H(12E)	0.976(16)
C(12B)-H(12F)	1.036(18)
C(13B)-H(13D)	0.99(2)
C(13B)-H(13E)	0.97(2)
C(13B)-H(13F)	0.988(19)
C(14B)-H(14D)	1.009(19)
C(14B)-H(14E)	0.99(2)
C(14B)-H(14F)	1.010(19)
C(15B)-C(16B)	1.526(2)
C(15B)-H(15C)	0.983(16)
C(15B)-H(15D)	0.969(15)
C(16B)-C(17B)	1.527(2)
C(16B)-H(16B)	1.061(14)
C(17B)-C(22B)	1.336(2)
C(17B)-C(18B)	1.562(2)
C(18B)-C(24B)	1.519(2)
C(18B)-C(25B)	1.524(2)
C(18B)-C(19B)	1.545(2)
C(19B)-C(27B)	1.528(2)
C(19B)-C(26B)	1.530(2)
C(19B)-C(20B)	1.538(2)
C(20B)-C(21B)	1.494(2)
C(20B)-H(20B)	0.966(15)
C(21B)-C(22B)	1.511(2)
C(21B)-H(21C)	1.005(15)
C(21B)-H(21D)	0.945(15)
C(22B)-C(23B)	1.505(2)
C(23B)-H(23D)	1.002(18)
C(23B)-H(23E)	0.977(19)
C(23B)-H(23F)	0.991(17)
C(24B)-H(24D)	0.991(16)
C(24B)-H(24E)	0.985(17)
C(24B)-H(24F)	0.994(16)
C(26B)-H(26D)	1.02(2)
C(26B)-H(26E)	1.022(17)
C(26B)-H(26F)	0.902(18)
C(28B)-H(28D)	0.973(19)
C(28B)-H(28E)	1.01(2)
C(28B)-H(28F)	1.005(18)

O(1A)-Si(1)-C(10A)	108.83(9)
O(1A)-Si(1)-C(9A)	112.73(9)
C(10A)-Si(1)-C(9A)	108.59(12)
O(1A)-Si(1)-C(11A)	104.53(7)
C(10A)-Si(1)-C(11A)	112.83(9)
C(9A)-Si(1)-C(11A)	109.35(9)
C(1A)-O(1A)-Si(1)	126.87(10)
C(2A)-O(2A)-C(8A)	112.94(13)
C(16A)-O(3A)-H(3A)	109.0(14)
C(27A)-O(5A)-C(28A)	116.14(17)
C(25A)-O(6A)-C(20A)	108.79(12)
O(1A)-C(1A)-C(2A)	119.97(14)
O(1A)-C(1A)-C(6A)	119.17(14)
C(2A)-C(1A)-C(6A)	120.77(16)
O(2A)-C(2A)-C(3A)	119.27(15)
O(2A)-C(2A)-C(1A)	119.52(15)
C(3A)-C(2A)-C(1A)	121.17(15)
C(4A)-C(3A)-C(2A)	117.57(16)
C(4A)-C(3A)-C(15A)	120.43(17)
C(2A)-C(3A)-C(15A)	121.96(16)
C(5A)-C(4A)-C(3A)	121.26(18)
C(5A)-C(4A)-H(4A)	120.0(10)
C(3A)-C(4A)-H(4A)	118.7(10)
C(4A)-C(5A)-C(6A)	121.60(17)
C(4A)-C(5A)-H(5A)	120.6(10)
C(6A)-C(5A)-H(5A)	117.8(10)
C(5A)-C(6A)-C(1A)	117.47(16)
C(5A)-C(6A)-C(7A)	121.95(16)
C(1A)-C(6A)-C(7A)	120.58(16)
C(6A)-C(7A)-H(7A1)	113.2(11)
C(6A)-C(7A)-H(7A2)	110.5(10)
H(7A1)-C(7A)-H(7A2)	109.8(14)
C(6A)-C(7A)-H(7A3)	111.4(10)
H(7A1)-C(7A)-H(7A3)	105.1(15)
H(7A2)-C(7A)-H(7A3)	106.4(13)
O(2A)-C(8A)-H(8A1)	109.8(11)
O(2A)-C(8A)-H(8A2)	107.6(11)
H(8A1)-C(8A)-H(8A2)	113.2(16)
O(2A)-C(8A)-H(8A3)	111.8(10)
H(8A1)-C(8A)-H(8A3)	106.8(15)
H(8A2)-C(8A)-H(8A3)	107.7(15)
Si(1)-C(9A)-H(9A1)	110.4(11)
Si(1)-C(9A)-H(9A2)	110.6(11)
H(9A1)-C(9A)-H(9A2)	107.4(16)
Si(1)-C(9A)-H(9A3)	112.4(11)
H(9A1)-C(9A)-H(9A3)	108.1(16)
H(9A2)-C(9A)-H(9A3)	107.9(16)
Si(1)-C(10A)-H(10A)	108.2(10)
Si(1)-C(10A)-H(10B)	108.1(11)
H(10A)-C(10A)-H(10B)	104.5(15)
Si(1)-C(10A)-H(10C)	108.5(13)
H(10A)-C(10A)-H(10C)	115.9(16)
H(10B)-C(10A)-H(10C)	111.3(17)
C(13A)-C(11A)-C(14A)	108.50(17)
C(13A)-C(11A)-C(12A)	109.27(16)
C(14A)-C(11A)-C(12A)	108.78(16)
C(13A)-C(11A)-Si(1)	110.65(13)
C(14A)-C(11A)-Si(1)	110.17(12)
C(12A)-C(11A)-Si(1)	109.44(13)
C(11A)-C(12A)-H(12A)	110.6(9)
C(11A)-C(12A)-H(12B)	110.9(9)

H(12A)-C(12A)-H(12B)	109.3(13)	C(21A)-C(22A)-C(23A)	112.67(16)
C(11A)-C(12A)-H(12C)	109.0(11)	C(22A)-C(23A)-H(23A)	115.5(12)
H(12A)-C(12A)-H(12C)	110.9(14)	C(22A)-C(23A)-H(23B)	108.7(11)
H(12B)-C(12A)-H(12C)	106.0(14)	H(23A)-C(23A)-H(23B)	102.7(15)
C(11A)-C(13A)-H(13A)	109.7(10)	C(22A)-C(23A)-H(23C)	113.8(11)
C(11A)-C(13A)-H(13B)	111.0(9)	H(23A)-C(23A)-H(23C)	108.7(17)
H(13A)-C(13A)-H(13B)	106.6(14)	H(23B)-C(23A)-H(23C)	106.5(16)
C(11A)-C(13A)-H(13C)	107.1(9)	C(18A)-C(24A)-H(24A)	112.1(10)
H(13A)-C(13A)-H(13C)	113.9(14)	C(18A)-C(24A)-H(24B)	110.3(9)
H(13B)-C(13A)-H(13C)	108.6(13)	H(24A)-C(24A)-H(24B)	107.2(13)
C(11A)-C(14A)-H(14A)	111.0(10)	C(18A)-C(24A)-H(24C)	111.6(9)
C(11A)-C(14A)-H(14B)	108.8(9)	H(24A)-C(24A)-H(24C)	107.9(13)
H(14A)-C(14A)-H(14B)	107.3(14)	H(24B)-C(24A)-H(24C)	107.4(13)
C(11A)-C(14A)-H(14C)	110.1(10)	O(7A)-C(25A)-O(6A)	121.67(15)
H(14A)-C(14A)-H(14C)	111.2(14)	O(7A)-C(25A)-C(18A)	129.29(16)
H(14B)-C(14A)-H(14C)	108.3(14)	O(6A)-C(25A)-C(18A)	108.93(14)
C(3A)-C(15A)-C(16A)	112.34(15)	C(19A)-C(26A)-H(26A)	110.5(9)
C(3A)-C(15A)-H(15A)	110.7(9)	C(19A)-C(26A)-H(26B)	107.7(10)
C(16A)-C(15A)-H(15A)	105.5(9)	H(26A)-C(26A)-H(26B)	107.1(13)
C(3A)-C(15A)-H(15B)	110.2(9)	C(19A)-C(26A)-H(26C)	109.7(11)
C(16A)-C(15A)-H(15B)	110.8(9)	H(26A)-C(26A)-H(26C)	109.4(14)
H(15A)-C(15A)-H(15B)	107.2(13)	H(26B)-C(26A)-H(26C)	112.4(15)
O(3A)-C(16A)-C(15A)	107.75(15)	O(4A)-C(27A)-O(5A)	123.21(17)
O(3A)-C(16A)-C(17A)	112.97(13)	O(4A)-C(27A)-C(19A)	125.58(16)
C(15A)-C(16A)-C(17A)	113.83(14)	O(5A)-C(27A)-C(19A)	111.16(16)
O(3A)-C(16A)-H(16A)	99.6(7)	O(5A)-C(28A)-H(28A)	110.6(12)
C(15A)-C(16A)-H(16A)	112.7(7)	O(5A)-C(28A)-H(28B)	103.5(13)
C(17A)-C(16A)-H(16A)	109.1(7)	H(28A)-C(28A)-H(28B)	109.6(18)
C(22A)-C(17A)-C(16A)	120.51(15)	O(5A)-C(28A)-H(28C)	110.8(11)
C(22A)-C(17A)-C(18A)	119.78(15)	H(28A)-C(28A)-H(28C)	112.4(17)
C(16A)-C(17A)-C(18A)	119.58(15)	H(28B)-C(28A)-H(28C)	109.6(17)
C(25A)-C(18A)-C(24A)	114.37(15)	O(1B)-Si(2)-C(9B)	112.15(8)
C(25A)-C(18A)-C(19A)	100.28(13)	O(1B)-Si(2)-C(10B)	109.07(9)
C(24A)-C(18A)-C(19A)	113.17(14)	C(9B)-Si(2)-C(10B)	108.51(10)
C(25A)-C(18A)-C(17A)	101.39(12)	O(1B)-Si(2)-C(11B)	104.44(7)
C(24A)-C(18A)-C(17A)	116.05(14)	C(9B)-Si(2)-C(11B)	111.62(9)
C(19A)-C(18A)-C(17A)	109.92(14)	C(10B)-Si(2)-C(11B)	111.00(9)
C(26A)-C(19A)-C(27A)	112.09(16)	C(1B)-O(1B)-Si(2)	127.35(10)
C(26A)-C(19A)-C(20A)	114.62(16)	C(2B)-O(2B)-C(8B)	112.18(13)
C(27A)-C(19A)-C(20A)	106.69(14)	C(16B)-O(3B)-H(3B)	107.7(14)
C(26A)-C(19A)-C(18A)	114.66(15)	C(27B)-O(5B)-C(28B)	116.43(15)
C(27A)-C(19A)-C(18A)	109.97(14)	C(25B)-O(6B)-C(20B)	108.58(13)
C(20A)-C(19A)-C(18A)	97.74(13)	O(1B)-C(1B)-C(6B)	119.68(14)
O(6A)-C(20A)-C(21A)	109.08(14)	O(1B)-C(1B)-C(2B)	119.45(14)
O(6A)-C(20A)-C(19A)	103.63(13)	C(6B)-C(1B)-C(2B)	120.76(16)
C(21A)-C(20A)-C(19A)	110.94(15)	O(2B)-C(2B)-C(3B)	119.92(14)
O(6A)-C(20A)-H(20A)	107.1(9)	O(2B)-C(2B)-C(1B)	118.93(15)
C(21A)-C(20A)-H(20A)	112.5(9)	C(3B)-C(2B)-C(1B)	121.12(15)
C(19A)-C(20A)-H(20A)	113.0(9)	C(2B)-C(3B)-C(4B)	117.72(16)
C(22A)-C(21A)-C(20A)	112.11(15)	C(2B)-C(3B)-C(15B)	121.99(16)
C(22A)-C(21A)-H(21A)	108.0(9)	C(4B)-C(3B)-C(15B)	120.27(17)
C(20A)-C(21A)-H(21A)	111.1(9)	C(5B)-C(4B)-C(3B)	121.15(17)
C(22A)-C(21A)-H(21B)	111.3(9)	C(5B)-C(4B)-H(4B)	119.8(9)
C(20A)-C(21A)-H(21B)	107.9(9)	C(3B)-C(4B)-H(4B)	119.0(9)
H(21A)-C(21A)-H(21B)	106.3(13)	C(4B)-C(5B)-C(6B)	121.71(17)
C(17A)-C(22A)-C(21A)	121.63(16)	C(4B)-C(5B)-H(5B)	120.8(10)
C(17A)-C(22A)-C(23A)	125.70(16)	C(6B)-C(5B)-H(5B)	117.5(10)

C(5B)-C(6B)-C(1B)	117.39(16)	O(3B)-C(16B)-C(17B)	112.54(13)
C(5B)-C(6B)-C(7B)	121.52(16)	O(3B)-C(16B)-C(15B)	108.21(14)
C(1B)-C(6B)-C(7B)	121.08(16)	C(17B)-C(16B)-C(15B)	114.24(14)
C(6B)-C(7B)-H(7B1)	113.2(10)	O(3B)-C(16B)-H(16B)	98.2(7)
C(6B)-C(7B)-H(7B2)	112.6(10)	C(17B)-C(16B)-H(16B)	110.5(8)
H(7B1)-C(7B)-H(7B2)	104.5(15)	C(15B)-C(16B)-H(16B)	112.1(7)
C(6B)-C(7B)-H(7B3)	111.5(11)	C(22B)-C(17B)-C(16B)	120.84(15)
H(7B1)-C(7B)-H(7B3)	109.7(14)	C(22B)-C(17B)-C(18B)	119.74(14)
H(7B2)-C(7B)-H(7B3)	104.8(14)	C(16B)-C(17B)-C(18B)	119.31(14)
O(2B)-C(8B)-H(8B1)	110.8(9)	C(24B)-C(18B)-C(25B)	113.84(15)
O(2B)-C(8B)-H(8B2)	107.1(10)	C(24B)-C(18B)-C(19B)	113.46(14)
H(8B1)-C(8B)-H(8B2)	110.8(14)	C(25B)-C(18B)-C(19B)	100.43(13)
O(2B)-C(8B)-H(8B3)	109.1(11)	C(24B)-C(18B)-C(17B)	116.12(14)
H(8B1)-C(8B)-H(8B3)	107.0(14)	C(25B)-C(18B)-C(17B)	102.22(12)
H(8B2)-C(8B)-H(8B3)	112.0(14)	C(19B)-C(18B)-C(17B)	109.14(13)
Si(2)-C(9B)-H(9B1)	110.4(11)	C(27B)-C(19B)-C(26B)	112.00(15)
Si(2)-C(9B)-H(9B2)	109.6(10)	C(27B)-C(19B)-C(20B)	105.71(14)
H(9B1)-C(9B)-H(9B2)	106.8(15)	C(26B)-C(19B)-C(20B)	114.79(16)
Si(2)-C(9B)-H(9B3)	109.9(11)	C(27B)-C(19B)-C(18B)	110.73(14)
H(9B1)-C(9B)-H(9B3)	110.6(15)	C(26B)-C(19B)-C(18B)	114.56(14)
H(9B2)-C(9B)-H(9B3)	109.6(15)	C(20B)-C(19B)-C(18B)	97.96(13)
Si(2)-C(10B)-H(10D)	109.2(11)	O(6B)-C(20B)-C(21B)	108.63(14)
Si(2)-C(10B)-H(10E)	110.5(10)	O(6B)-C(20B)-C(19B)	103.56(13)
H(10D)-C(10B)-H(10E)	107.8(15)	C(21B)-C(20B)-C(19B)	111.22(14)
Si(2)-C(10B)-H(10F)	113.3(10)	O(6B)-C(20B)-H(20B)	107.2(9)
H(10D)-C(10B)-H(10F)	108.2(15)	C(21B)-C(20B)-H(20B)	112.0(9)
H(10E)-C(10B)-H(10F)	107.7(14)	C(19B)-C(20B)-H(20B)	113.7(9)
C(14B)-C(11B)-C(12B)	109.06(17)	C(20B)-C(21B)-C(22B)	112.67(15)
C(14B)-C(11B)-C(13B)	108.9(2)	C(20B)-C(21B)-H(21C)	110.0(8)
C(12B)-C(11B)-C(13B)	108.43(17)	C(22B)-C(21B)-H(21C)	110.7(8)
C(14B)-C(11B)-Si(2)	111.31(14)	C(20B)-C(21B)-H(21D)	108.8(9)
C(12B)-C(11B)-Si(2)	108.80(13)	C(22B)-C(21B)-H(21D)	110.2(9)
C(13B)-C(11B)-Si(2)	110.29(13)	H(21C)-C(21B)-H(21D)	104.2(12)
C(11B)-C(12B)-H(12D)	110.8(11)	C(17B)-C(22B)-C(23B)	126.52(16)
C(11B)-C(12B)-H(12E)	111.3(10)	C(17B)-C(22B)-C(21B)	121.36(15)
H(12D)-C(12B)-H(12E)	105.1(14)	C(23B)-C(22B)-C(21B)	112.11(15)
C(11B)-C(12B)-H(12F)	112.8(10)	C(22B)-C(23B)-H(23D)	115.8(10)
H(12D)-C(12B)-H(12F)	105.8(14)	C(22B)-C(23B)-H(23E)	113.2(10)
H(12E)-C(12B)-H(12F)	110.6(14)	H(23D)-C(23B)-H(23E)	108.1(14)
C(11B)-C(13B)-H(13D)	109.3(12)	C(22B)-C(23B)-H(23F)	108.3(9)
C(11B)-C(13B)-H(13E)	110.6(12)	H(23D)-C(23B)-H(23F)	104.7(13)
H(13D)-C(13B)-H(13E)	108.2(17)	H(23E)-C(23B)-H(23F)	105.9(14)
C(11B)-C(13B)-H(13F)	108.0(10)	C(18B)-C(24B)-H(24D)	113.2(9)
H(13D)-C(13B)-H(13F)	110.4(16)	C(18B)-C(24B)-H(24E)	110.4(10)
H(13E)-C(13B)-H(13F)	110.3(16)	H(24D)-C(24B)-H(24E)	108.6(13)
C(11B)-C(14B)-H(14D)	109.6(10)	C(18B)-C(24B)-H(24F)	110.5(9)
C(11B)-C(14B)-H(14E)	110.8(12)	H(24D)-C(24B)-H(24F)	108.5(13)
H(14D)-C(14B)-H(14E)	105.5(15)	H(24E)-C(24B)-H(24F)	105.3(13)
C(11B)-C(14B)-H(14F)	108.6(12)	O(7B)-C(25B)-O(6B)	121.65(16)
H(14D)-C(14B)-H(14F)	115.0(16)	O(7B)-C(25B)-C(18B)	129.16(16)
H(14E)-C(14B)-H(14F)	107.3(17)	O(6B)-C(25B)-C(18B)	109.13(14)
C(3B)-C(15B)-C(16B)	112.59(15)	C(19B)-C(26B)-H(26D)	109.0(11)
C(3B)-C(15B)-H(15C)	108.3(10)	C(19B)-C(26B)-H(26E)	109.2(9)
C(16B)-C(15B)-H(15C)	110.0(9)	H(26D)-C(26B)-H(26E)	111.8(14)
C(3B)-C(15B)-H(15D)	110.1(9)	C(19B)-C(26B)-H(26F)	110.8(11)
C(16B)-C(15B)-H(15D)	107.5(9)	H(26D)-C(26B)-H(26F)	105.9(15)
H(15C)-C(15B)-H(15D)	108.3(13)	H(26E)-C(26B)-H(26F)	110.2(15)

O(4B)-C(27B)-O(5B)	123.36(17)
O(4B)-C(27B)-C(19B)	125.61(16)
O(5B)-C(27B)-C(19B)	110.94(15)
O(5B)-C(28B)-H(28D)	112.0(11)
O(5B)-C(28B)-H(28E)	107.3(11)
H(28D)-C(28B)-H(28E)	117.1(16)
O(5B)-C(28B)-H(28F)	112.6(10)
H(28D)-C(28B)-H(28F)	96.4(14)
H(28E)-C(28B)-H(28F)	111.3(15)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB31 (CCDC 283708). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	246(3)	166(3)	247(3)	-46(2)	-122(2)	20(2)
O(1A)	218(7)	140(6)	209(6)	-42(5)	-89(5)	25(5)
O(2A)	235(7)	253(7)	173(6)	-30(5)	-67(5)	39(6)
O(3A)	198(7)	203(7)	373(8)	-42(6)	-118(6)	22(6)
O(4A)	350(8)	465(9)	301(8)	4(7)	-165(6)	64(7)
O(5A)	562(9)	293(8)	437(9)	-82(7)	-372(7)	140(7)
O(6A)	189(7)	196(7)	260(7)	-20(5)	-99(5)	-15(5)
O(7A)	170(7)	244(7)	230(7)	-32(5)	-82(5)	31(6)
C(1A)	140(9)	163(10)	236(10)	-59(8)	-64(8)	2(8)
C(2A)	125(9)	219(10)	200(10)	-34(8)	-58(7)	3(8)
C(3A)	99(9)	196(10)	304(11)	-37(8)	-44(8)	0(8)
C(4A)	175(10)	184(11)	378(12)	-115(10)	-14(9)	-18(8)
C(5A)	205(10)	327(12)	211(11)	-121(9)	0(8)	-51(9)
C(6A)	156(9)	238(11)	176(10)	-32(8)	-27(8)	-46(8)
C(7A)	308(13)	319(13)	205(11)	-27(9)	-78(9)	-47(11)
C(8A)	227(12)	296(13)	235(12)	-65(10)	4(9)	37(10)
C(9A)	642(18)	241(13)	365(13)	-23(10)	-322(13)	-22(12)
C(10A)	281(12)	289(13)	538(16)	-137(12)	-195(11)	40(10)
C(11A)	254(10)	188(10)	231(10)	-56(8)	-103(8)	11(8)
C(12A)	390(14)	169(12)	358(13)	-41(10)	-182(11)	1(10)
C(13A)	351(13)	250(13)	550(16)	-136(11)	-187(12)	57(10)
C(14A)	506(15)	206(12)	379(13)	22(10)	-264(12)	-19(11)
C(15A)	157(10)	174(11)	395(13)	2(9)	-100(9)	-11(8)
C(16A)	163(10)	150(10)	287(11)	-20(8)	-78(8)	13(8)
C(17A)	116(9)	190(10)	297(10)	-27(8)	-78(8)	-11(8)
C(18A)	178(10)	160(10)	270(10)	-26(8)	-103(8)	10(8)
C(19A)	222(10)	189(10)	341(11)	-43(8)	-145(9)	34(8)
C(20A)	242(11)	139(10)	339(11)	8(9)	-143(9)	4(8)
C(21A)	213(11)	162(10)	324(11)	-52(9)	-93(9)	17(9)
C(22A)	140(9)	178(10)	300(11)	-25(8)	-48(8)	10(8)
C(23A)	272(12)	208(12)	307(12)	-50(10)	-27(10)	10(10)
C(24A)	238(12)	217(11)	321(12)	-52(9)	-141(10)	12(9)
C(25A)	220(10)	213(11)	163(9)	-8(8)	-94(8)	-19(8)
C(26A)	263(12)	263(13)	599(16)	-47(12)	-252(12)	52(10)
C(27A)	365(12)	184(11)	394(12)	-14(9)	-269(10)	-28(9)
C(28A)	750(20)	329(15)	444(15)	-103(12)	-428(14)	111(14)
Si(2)	224(3)	166(3)	247(3)	-30(2)	-96(2)	14(2)
O(1B)	223(7)	140(7)	237(7)	-21(5)	-98(5)	13(5)
O(2B)	255(7)	269(7)	176(7)	0(6)	-64(5)	-3(6)
O(3B)	237(8)	179(7)	385(8)	-45(6)	-133(6)	34(6)
O(4B)	300(8)	425(9)	267(7)	-10(6)	-100(6)	72(7)
O(5B)	431(8)	303(8)	273(7)	-35(6)	-207(6)	95(6)
O(6B)	175(7)	173(7)	257(7)	-21(5)	-58(5)	-15(5)
O(7B)	179(7)	243(7)	246(7)	-58(6)	-74(5)	33(6)
C(1B)	149(9)	135(9)	218(10)	-21(8)	-63(8)	1(7)
C(2B)	141(9)	212(10)	167(9)	1(8)	-48(7)	7(8)

C(3B)	121(9)	188(10)	273(10)	15(8)	-54(8)	-24(8)
C(4B)	182(10)	150(11)	346(12)	-63(9)	-58(9)	-11(8)
C(5B)	171(10)	289(12)	213(11)	-96(9)	-15(8)	-23(8)
C(6B)	149(9)	212(10)	178(9)	-1(8)	-37(7)	-24(8)
C(7B)	294(12)	270(12)	189(11)	31(9)	-89(9)	-29(10)
C(8B)	303(13)	269(13)	222(12)	-62(10)	-26(9)	15(10)
C(9B)	321(13)	249(12)	356(13)	-55(10)	-177(11)	25(10)
C(10B)	290(12)	329(13)	334(13)	-78(11)	-82(10)	-43(11)
C(11B)	242(10)	183(10)	341(11)	-73(8)	-111(9)	19(8)
C(12B)	332(13)	219(12)	412(14)	-126(10)	-134(11)	19(10)
C(13B)	329(14)	304(14)	624(18)	-215(13)	7(12)	17(12)
C(14B)	586(17)	253(13)	755(19)	-156(13)	-461(16)	154(13)
C(15B)	181(11)	171(11)	348(12)	48(9)	-92(9)	-14(9)
C(16B)	171(10)	135(10)	264(10)	2(8)	-92(8)	-13(8)
C(17B)	128(9)	179(10)	227(10)	0(8)	-58(8)	-16(8)
C(18B)	186(10)	141(10)	236(10)	-8(8)	-88(8)	-2(8)
C(19B)	231(10)	186(10)	232(10)	-4(8)	-93(8)	9(8)
C(20B)	213(10)	139(10)	267(11)	27(8)	-74(8)	0(8)
C(21B)	215(11)	154(10)	265(11)	-55(8)	-66(9)	22(9)
C(22B)	169(10)	174(10)	218(10)	-17(8)	-50(8)	-14(8)
C(23B)	279(12)	219(12)	218(11)	-32(9)	-31(9)	14(10)
C(24B)	243(12)	193(11)	284(12)	-45(9)	-124(9)	-5(9)
C(25B)	231(10)	207(10)	142(9)	-28(8)	-79(8)	-4(9)
C(26B)	214(11)	284(13)	350(13)	20(10)	-119(10)	53(10)
C(27B)	272(11)	193(10)	312(11)	-10(9)	-156(9)	-17(9)
C(28B)	468(15)	337(13)	264(12)	-40(10)	-188(11)	62(12)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for DCB31 (CCDC 283708).

	x	y	z	U_{iso}
H(3A)	3688(19)	4314(14)	814(14)	78(8)
H(4A)	201(13)	4175(11)	3162(11)	30(5)
H(5A)	-132(13)	2974(10)	4325(11)	25(5)
H(7A1)	-544(16)	867(12)	4330(12)	46(6)
H(7A2)	-69(13)	1455(11)	4872(12)	32(5)
H(7A3)	760(15)	894(11)	4127(11)	40(6)
H(8A1)	2967(16)	2594(13)	644(12)	53(7)
H(8A2)	2642(16)	2081(13)	-37(14)	60(7)
H(8A3)	2505(14)	1627(12)	968(12)	41(6)
H(9A1)	45(16)	1427(14)	673(13)	59(7)
H(9A2)	-397(16)	488(13)	676(13)	58(7)
H(9A3)	985(18)	670(14)	351(14)	71(8)
H(10A)	-1676(14)	505(12)	3497(13)	46(6)
H(10B)	-2032(17)	324(13)	2692(13)	55(7)
H(10C)	-1708(17)	1284(14)	2659(14)	66(8)
H(12A)	-171(13)	-1113(10)	1309(12)	28(5)
H(12B)	-1181(15)	-1199(10)	2360(11)	29(5)
H(12C)	-170(14)	-1847(12)	2143(11)	33(5)
H(13A)	1874(15)	-1379(12)	1840(12)	45(6)
H(13B)	2135(14)	-385(11)	1849(11)	29(5)
H(13C)	1796(13)	-581(11)	968(12)	34(5)
H(14A)	-721(16)	-845(12)	3734(12)	49(6)
H(14B)	627(14)	-554(11)	3490(11)	31(5)
H(14C)	355(14)	-1552(12)	3421(12)	43(6)
H(15A)	425(13)	4736(10)	1503(10)	25(5)
H(15B)	998(13)	4120(11)	721(11)	30(5)
H(16A)	2236(11)	4803(8)	1738(9)	3(4)
H(20A)	3219(13)	7783(11)	-1165(11)	34(5)
H(21A)	2915(13)	7589(10)	494(10)	21(5)
H(21B)	1745(13)	7611(10)	350(10)	26(5)
H(23A)	1387(17)	5894(14)	2315(14)	64(7)
H(23B)	941(17)	6760(13)	2048(12)	54(7)
H(23C)	2132(17)	6743(13)	2117(13)	60(7)
H(24A)	3128(14)	4891(11)	-1621(12)	33(5)
H(24B)	1857(15)	4754(10)	-810(11)	32(5)
H(24C)	2960(13)	4324(11)	-645(11)	28(5)
H(26A)	782(14)	6455(11)	27(12)	33(5)
H(26B)	880(14)	6179(12)	-967(11)	39(6)
H(26C)	997(14)	7196(12)	-883(12)	40(6)
H(28A)	3459(16)	7683(14)	-3771(13)	55(7)
H(28B)	2220(20)	7377(15)	-3685(15)	87(9)
H(28C)	3208(16)	6680(13)	-3761(13)	53(7)
H(3B)	1220(20)	5723(16)	4087(17)	114(10)
H(4B)	4665(12)	5890(10)	1672(10)	19(5)
H(5B)	5010(13)	7083(10)	475(11)	26(5)
H(7B1)	5590(16)	9117(12)	338(12)	44(6)
H(7B2)	4273(15)	9247(12)	709(12)	45(6)

H(7B3)	4798(14)	8624(11)	-15(12)	39(6)
H(8B1)	2458(13)	8392(11)	3792(11)	29(5)
H(8B2)	2434(13)	8067(11)	4816(12)	32(5)
H(8B3)	2056(16)	7416(13)	4285(12)	51(6)
H(9B1)	5015(17)	8795(12)	4117(14)	58(7)
H(9B2)	6204(15)	9337(12)	3590(11)	43(6)
H(9B3)	6052(15)	8483(12)	3257(12)	41(6)
H(10D)	6756(15)	9418(13)	1273(12)	47(6)
H(10E)	6969(15)	10252(12)	1610(12)	45(6)
H(10F)	6179(15)	10302(12)	1002(13)	52(6)
H(12D)	4742(15)	11708(12)	3470(12)	43(6)
H(12E)	5801(14)	11364(11)	2713(12)	30(5)
H(12F)	5359(15)	10891(12)	3820(13)	47(6)
H(13D)	3564(17)	9949(14)	4480(14)	66(8)
H(13E)	2761(17)	9917(13)	3923(13)	60(7)
H(13F)	2901(15)	10825(12)	4254(12)	44(6)
H(14D)	3408(15)	11588(12)	2666(12)	48(6)
H(14E)	3379(17)	10716(13)	2251(13)	62(7)
H(14F)	4577(18)	11267(13)	1777(14)	62(8)
H(15C)	3961(13)	6006(11)	4080(11)	29(5)
H(15D)	4563(13)	5374(10)	3328(10)	20(5)
H(16B)	2690(11)	5298(9)	3116(10)	13(4)
H(20B)	1938(12)	2330(10)	6009(10)	21(5)
H(21C)	1972(13)	2468(10)	4457(10)	21(5)
H(21D)	3183(13)	2494(10)	4462(10)	19(5)
H(23D)	3419(14)	4199(12)	2518(12)	43(6)
H(23E)	2648(16)	3318(12)	2812(12)	47(6)
H(23F)	3899(15)	3285(11)	2794(11)	34(5)
H(24D)	2117(13)	5778(11)	5512(10)	26(5)
H(24E)	3246(15)	5340(11)	5603(11)	34(5)
H(24F)	2058(13)	5180(10)	6467(12)	29(5)
H(26D)	4364(16)	3949(13)	5623(12)	55(7)
H(26E)	4271(14)	3613(11)	4680(12)	42(6)
H(26F)	4218(15)	2973(12)	5585(12)	39(6)
H(28D)	2178(16)	3296(13)	8539(13)	51(6)
H(28E)	3210(17)	2533(13)	8372(13)	60(7)
H(28F)	1846(15)	2391(12)	8536(12)	41(6)

Table 6. Hydrogen bonds for DCB31 (CCDC 283708) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3A)-H(3A)...O(7A)#1	0.92(2)	1.88(2)	2.7946(17)	173(2)
O(3B)-H(3B)...O(7B)#2	0.98(3)	1.81(3)	2.7910(17)	176(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z
#2 -x,-y+1,-z+1

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X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Bisacetoxyacetal **256** (DCB32)

(CCDC 289914)

Contents:

Figure B.114 Representation of Bisacetoxyacetal **256**

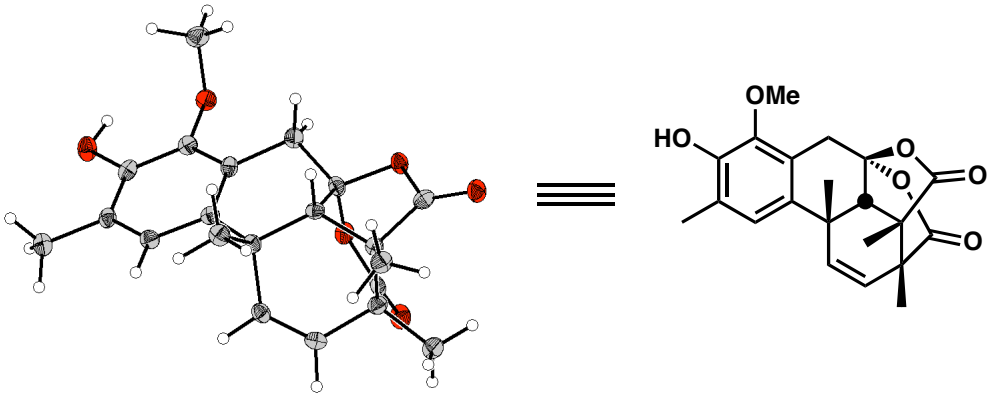

Table 1. Crystal data and structure refinement for DCB32 (CCDC 289914).

Empirical formula	$C_{21}H_{22}O_6$
Formula weight	370.39
Crystallization Solvent	Et_2O/hexanes
Crystal Habit	Needle
Crystal size	0.39 x 0.22 x 0.19 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 13215 reflections used in lattice determination	2.27 to 28.03°
Unit cell dimensions	a = 21.9617(16) Å b = 8.5236(6) Å c = 19.6358(14) Å
Volume	3675.7(5) $Å^3$
Z	8
Crystal system	Orthorhombic
Space group	Pbcn
Density (calculated)	1.339 Mg/m^3
F(000)	1568
Data collection program	Bruker SMART v5.630
θ range for data collection	1.85 to 28.38°
Completeness to θ = 28.38°	94.2 %
Index ranges	$-28 \le h \le 28$, $-11 \le k \le 11$, $-24 \le l \le 26$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	50823
Independent reflections	4344 [R_{int}= 0.0809]
Absorption coefficient	0.098 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9816 and 0.9628

Table 1 (cont.)

Structure Solution and Refinement

Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	4344 / 0 / 332
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.880
Final R indices [I>2σ(I), 3001 reflections]	R1 = 0.0466, *w*R2 = 0.0611
R indices (all data)	R1 = 0.0778, *w*R2 = 0.0633
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	0.331 and -0.276 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB32 (CCDC 289914). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	2750(1)	8498(1)	11657(1)	20(1)
O(2)	3463(1)	6194(1)	12216(1)	22(1)
O(3)	4347(1)	12923(1)	9814(1)	24(1)
O(4)	3652(1)	11114(1)	10020(1)	19(1)
O(5)	2849(1)	12317(1)	8276(1)	22(1)
O(6)	2812(1)	11335(1)	9329(1)	18(1)
C(1)	3243(1)	8168(2)	10565(1)	16(1)
C(2)	3171(1)	7737(2)	11243(1)	16(1)
C(3)	3539(1)	6609(2)	11548(1)	17(1)
C(4)	3993(1)	5863(2)	11174(1)	17(1)
C(5)	4067(1)	6316(2)	10500(1)	17(1)
C(6)	3705(1)	7443(2)	10182(1)	16(1)
C(7)	3834(1)	7839(2)	9427(1)	15(1)
C(8)	3386(1)	9079(2)	9173(1)	15(1)
C(9)	3172(1)	10217(2)	9710(1)	17(1)
C(10)	2832(1)	9437(2)	10272(1)	18(1)
C(11)	2149(1)	7822(2)	11619(1)	25(1)
C(12)	4388(1)	4633(2)	11498(1)	24(1)
C(13)	3759(1)	6327(2)	8998(1)	20(1)
C(14)	4480(1)	8405(2)	9339(1)	18(1)
C(15)	4634(1)	9789(2)	9089(1)	19(1)
C(16)	4175(1)	11058(2)	8904(1)	18(1)
C(17)	3586(1)	10261(2)	8630(1)	15(1)
C(18)	4452(1)	12247(2)	8416(1)	23(1)
C(19)	3652(1)	9638(2)	7907(1)	21(1)
C(20)	4062(1)	11828(2)	9598(1)	19(1)
C(21)	3057(1)	11422(2)	8689(1)	17(1)

Table 3. Bond lengths [Å] and angles [°] for DCB32 (CCDC 289914).

O(1)-C(2)	1.3917(16)	C(2)-O(1)-C(11)	113.13(12)
O(1)-C(11)	1.4405(18)	C(3)-O(2)-H(2)	108.0(13)
O(2)-C(3)	1.3695(17)	C(20)-O(4)-C(9)	117.64(11)
O(2)-H(2)	0.887(19)	C(21)-O(6)-C(9)	107.19(11)
O(3)-C(20)	1.2002(16)	C(2)-C(1)-C(6)	118.61(13)
O(4)-C(20)	1.3677(17)	C(2)-C(1)-C(10)	118.79(13)
O(4)-C(9)	1.4373(16)	C(6)-C(1)-C(10)	122.58(13)
O(5)-C(21)	1.2031(16)	C(1)-C(2)-C(3)	121.92(13)
O(6)-C(21)	1.3697(16)	C(1)-C(2)-O(1)	120.79(13)
O(6)-C(9)	1.4462(16)	C(3)-C(2)-O(1)	117.17(13)
C(1)-C(2)	1.3899(19)	O(2)-C(3)-C(2)	121.36(13)
C(1)-C(6)	1.4059(19)	O(2)-C(3)-C(4)	118.33(13)
C(1)-C(10)	1.521(2)	C(2)-C(3)-C(4)	120.30(13)
C(2)-C(3)	1.3915(19)	C(5)-C(4)-C(3)	117.36(14)
C(3)-C(4)	1.3917(19)	C(5)-C(4)-C(12)	122.04(14)
C(4)-C(5)	1.388(2)	C(3)-C(4)-C(12)	120.60(14)
C(4)-C(12)	1.503(2)	C(4)-C(5)-C(6)	123.54(14)
C(5)-C(6)	1.3954(19)	C(4)-C(5)-H(5)	118.2(7)
C(5)-H(5)	0.960(12)	C(6)-C(5)-H(5)	118.2(7)
C(6)-C(7)	1.546(2)	C(5)-C(6)-C(1)	118.25(14)
C(7)-C(14)	1.5080(19)	C(5)-C(6)-C(7)	118.43(13)
C(7)-C(8)	1.5268(19)	C(1)-C(6)-C(7)	123.32(13)
C(7)-C(13)	1.548(2)	C(14)-C(7)-C(8)	110.30(12)
C(8)-C(9)	1.5086(19)	C(14)-C(7)-C(6)	110.57(12)
C(8)-C(17)	1.5300(19)	C(8)-C(7)-C(6)	110.27(12)
C(8)-H(8)	0.966(13)	C(14)-C(7)-C(13)	107.68(12)
C(9)-C(10)	1.490(2)	C(8)-C(7)-C(13)	109.33(12)
C(10)-H(10A)	1.027(14)	C(6)-C(7)-C(13)	108.64(12)
C(10)-H(10B)	0.976(14)	C(9)-C(8)-C(7)	114.69(12)
C(11)-H(11A)	1.018(15)	C(9)-C(8)-C(17)	98.80(11)
C(11)-H(11B)	0.941(14)	C(7)-C(8)-C(17)	119.94(12)
C(11)-H(11C)	1.017(15)	C(9)-C(8)-H(8)	106.2(8)
C(12)-H(12A)	0.965(18)	C(7)-C(8)-H(8)	107.8(8)
C(12)-H(12B)	0.994(18)	C(17)-C(8)-H(8)	108.4(8)
C(12)-H(12C)	0.989(17)	O(4)-C(9)-O(6)	105.61(11)
C(13)-H(13A)	1.003(14)	O(4)-C(9)-C(10)	106.90(12)
C(13)-H(13B)	0.988(15)	O(6)-C(9)-C(10)	113.83(12)
C(13)-H(13C)	0.996(14)	O(4)-C(9)-C(8)	114.14(12)
C(14)-C(15)	1.323(2)	O(6)-C(9)-C(8)	103.46(11)
C(14)-H(14)	0.975(13)	C(10)-C(9)-C(8)	112.83(13)
C(15)-C(16)	1.523(2)	C(9)-C(10)-C(1)	107.47(13)
C(15)-H(15)	0.987(11)	C(9)-C(10)-H(10A)	107.7(7)
C(16)-C(18)	1.520(2)	C(1)-C(10)-H(10A)	113.2(8)
C(16)-C(20)	1.533(2)	C(9)-C(10)-H(10B)	111.0(8)
C(16)-C(17)	1.5582(19)	C(1)-C(10)-H(10B)	110.6(8)
C(17)-C(19)	1.523(2)	H(10A)-C(10)-H(10B)	106.9(11)
C(17)-C(21)	1.530(2)	O(1)-C(11)-H(11A)	111.2(8)
C(18)-H(18A)	0.989(14)	O(1)-C(11)-H(11B)	104.9(8)
C(18)-H(18B)	0.977(15)	H(11A)-C(11)-H(11B)	109.5(11)
C(18)-H(18C)	1.001(16)	O(1)-C(11)-H(11C)	111.2(8)
C(19)-H(19A)	0.956(14)	H(11A)-C(11)-H(11C)	110.5(12)
C(19)-H(19B)	1.006(15)	H(11B)-C(11)-H(11C)	109.4(12)
C(19)-H(19C)	0.974(17)	C(4)-C(12)-H(12A)	112.6(11)
		C(4)-C(12)-H(12B)	111.6(10)

H(12A)-C(12)-H(12B)	104.8(14)
C(4)-C(12)-H(12C)	112.2(10)
H(12A)-C(12)-H(12C)	108.5(14)
H(12B)-C(12)-H(12C)	106.7(14)
C(7)-C(13)-H(13A)	109.3(8)
C(7)-C(13)-H(13B)	111.2(8)
H(13A)-C(13)-H(13B)	109.6(11)
C(7)-C(13)-H(13C)	108.8(8)
H(13A)-C(13)-H(13C)	110.4(11)
H(13B)-C(13)-H(13C)	107.5(11)
C(15)-C(14)-C(7)	124.68(14)
C(15)-C(14)-H(14)	118.8(8)
C(7)-C(14)-H(14)	116.5(7)
C(14)-C(15)-C(16)	123.55(14)
C(14)-C(15)-H(15)	119.1(7)
C(16)-C(15)-H(15)	117.2(7)
C(18)-C(16)-C(15)	111.10(13)
C(18)-C(16)-C(20)	109.81(13)
C(15)-C(16)-C(20)	101.49(11)
C(18)-C(16)-C(17)	113.92(13)
C(15)-C(16)-C(17)	108.83(12)
C(20)-C(16)-C(17)	111.00(12)
C(19)-C(17)-C(21)	111.62(12)
C(19)-C(17)-C(8)	116.54(13)
C(21)-C(17)-C(8)	99.01(11)
C(19)-C(17)-C(16)	113.21(12)
C(21)-C(17)-C(16)	108.81(12)
C(8)-C(17)-C(16)	106.55(11)
C(16)-C(18)-H(18A)	107.5(8)
C(16)-C(18)-H(18B)	111.6(8)
H(18A)-C(18)-H(18B)	110.3(12)
C(16)-C(18)-H(18C)	108.8(9)
H(18A)-C(18)-H(18C)	111.5(12)
H(18B)-C(18)-H(18C)	107.2(12)
C(17)-C(19)-H(19A)	107.7(8)
C(17)-C(19)-H(19B)	110.6(8)
H(19A)-C(19)-H(19B)	111.7(12)
C(17)-C(19)-H(19C)	113.5(9)
H(19A)-C(19)-H(19C)	104.7(12)
H(19B)-C(19)-H(19C)	108.6(12)
O(3)-C(20)-O(4)	118.39(14)
O(3)-C(20)-C(16)	124.22(14)
O(4)-C(20)-C(16)	117.06(13)
O(5)-C(21)-O(6)	120.27(13)
O(5)-C(21)-C(17)	130.33(14)
O(6)-C(21)-C(17)	109.40(12)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB32 (CCDC 289914). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	219(6)	189(6)	191(6)	-17(5)	52(5)	31(5)
O(2)	300(7)	204(6)	154(6)	14(5)	13(5)	46(5)
O(3)	273(6)	157(6)	288(7)	-25(5)	-80(5)	-20(5)
O(4)	233(6)	155(6)	172(6)	-15(5)	-14(5)	-8(5)
O(5)	248(6)	236(6)	179(6)	56(5)	-2(5)	32(5)
O(6)	215(6)	187(6)	141(6)	24(5)	18(5)	52(5)
C(1)	198(9)	125(8)	164(8)	-5(7)	-11(7)	-4(7)
C(2)	186(8)	133(8)	168(9)	-39(7)	23(7)	0(7)
C(3)	227(9)	148(9)	130(9)	-8(7)	-8(7)	-46(7)
C(4)	201(9)	112(8)	200(9)	-4(7)	-20(7)	-16(7)
C(5)	172(9)	147(8)	204(9)	-35(7)	31(7)	20(7)
C(6)	172(8)	139(8)	166(8)	-26(7)	1(7)	-13(7)
C(7)	156(8)	139(8)	161(8)	-18(7)	3(7)	18(7)
C(8)	136(8)	152(8)	162(9)	-18(7)	-2(7)	-18(7)
C(9)	166(8)	156(8)	183(8)	15(7)	-40(7)	20(7)
C(10)	211(9)	175(9)	157(9)	1(7)	36(8)	24(7)
C(11)	232(10)	239(10)	269(11)	-6(9)	92(9)	14(8)
C(12)	245(10)	208(10)	272(11)	45(8)	2(8)	35(8)
C(13)	221(10)	182(9)	205(10)	-34(8)	4(8)	11(8)
C(14)	179(9)	198(9)	159(9)	-11(7)	3(7)	40(7)
C(15)	141(8)	234(9)	180(9)	-23(7)	3(7)	0(7)
C(16)	168(8)	170(8)	188(9)	-1(7)	3(7)	-14(7)
C(17)	167(8)	146(8)	148(8)	-11(7)	10(7)	7(7)
C(18)	228(10)	218(10)	249(10)	30(8)	2(8)	-26(8)
C(19)	239(10)	241(10)	159(9)	7(8)	3(8)	-8(9)
C(20)	178(9)	154(9)	228(9)	32(7)	-44(7)	41(7)
C(21)	188(9)	169(8)	159(9)	-30(7)	-2(7)	-52(7)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for DCB32 (CCDC 289914).

	x	y	z	U_{iso}
H(2)	3183(9)	6820(20)	12395(10)	66(7)
H(5)	4383(6)	5822(14)	10239(6)	7(3)
H(8)	3027(6)	8542(15)	9011(6)	11(4)
H(10A)	2434(6)	9003(15)	10073(7)	16(4)
H(10B)	2719(6)	10191(17)	10624(7)	21(4)
H(11A)	1959(6)	8003(17)	11152(8)	27(4)
H(11B)	1921(6)	8358(16)	11952(7)	15(4)
H(11C)	2158(6)	6658(19)	11731(7)	26(4)
H(12A)	4187(8)	3630(20)	11532(9)	59(6)
H(12B)	4495(7)	4910(20)	11975(9)	56(6)
H(12C)	4777(8)	4488(19)	11252(8)	49(5)
H(13A)	4066(6)	5529(17)	9151(7)	26(4)
H(13B)	3815(6)	6542(16)	8508(8)	22(4)
H(13C)	3338(7)	5917(15)	9061(7)	22(4)
H(14)	4801(6)	7679(15)	9475(6)	12(4)
H(15)	5070(5)	10055(15)	9044(6)	9(4)
H(18A)	4804(6)	12732(16)	8649(7)	23(4)
H(18B)	4157(7)	13051(17)	8286(7)	29(4)
H(18C)	4578(6)	11692(17)	7990(8)	32(5)
H(19A)	3300(6)	9017(16)	7809(7)	20(4)
H(19B)	4038(7)	9014(17)	7859(7)	30(5)
H(19C)	3650(6)	10458(19)	7561(8)	36(5)

Table 6. Hydrogen bonds for DCB32 (CCDC 289914) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(2)...O(5)#1	0.887(19)	2.02(2)	2.7848(15)	144.2(17)
O(2)-H(2)...O(1)	0.887(19)	2.248(19)	2.7418(14)	114.8(15)

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+2,z+1/2

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE
X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Tetracycle **269** (JLS03)

(CCDC 701799)

Contents:

Figure B.115 Representation of Tetracycle **269**

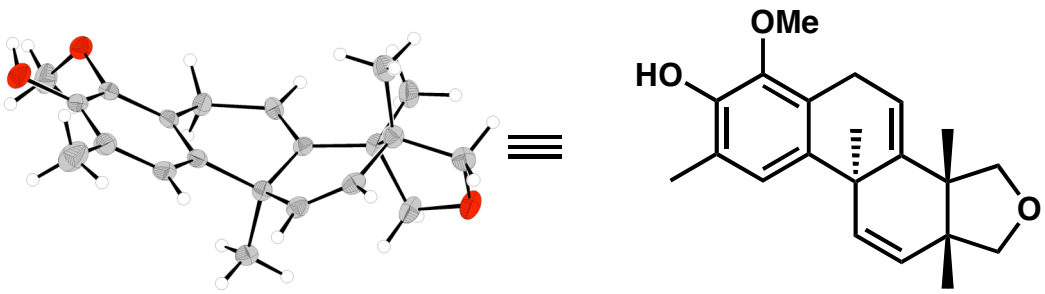

Table 1. Crystal data and structure refinement for JLS03 (CCDC 701799).

Empirical formula	$C_{21}H_{26}O_3$
Formula weight	326.42
Crystallization Solvent	Acetone/heptane
Crystal Habit	Fragment
Crystal size	0.26 x 0.24 x 0.14 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 11684 reflections used in lattice determination	2.24 to 28.20°
Unit cell dimensions	a = 14.7511(11) Å b = 20.2786(15) Å β= 102.6820(10)° c = 18.3704(14) Å
Volume	5361.1(7) $Å^3$
Z	12
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.213 Mg/m^3
F(000)	2112
Data collection program	Bruker SMART v5.630
θ range for data collection	1.41 to 28.49°
Completeness to θ = 28.49°	92.3 %
Index ranges	$-19 \le h \le 18, -26 \le k \le 26, -24 \le l \le 23$
Data collection scan type	ω scans at 4 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	59115
Independent reflections	12523 [R_{int}= 0.0934]
Absorption coefficient	0.080 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9890 and 0.9796

Table 1 (cont.)

Structure Solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12523 / 0 / 667
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.353
Final R indices [I>2σ(I), 7169 reflections]	R1 = 0.0549, wR2 = 0.0886
R indices (all data)	R1 = 0.1055, wR2 = 0.0944
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	0.822 and -0.258 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

There are three molecules in the asymmetric unit. Molecules B and C have the same stereochemistry and are the enantiomer of molecule A.

Refinement of F^2 against ALL reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for JLS03 (CCDC 701799). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1A)	7781(1)	4925(1)	6261(1)	33(1)
O(2A)	3817(1)	3682(1)	9705(1)	26(1)
O(3A)	3532(1)	4840(1)	8912(1)	22(1)
C(1A)	7309(1)	4309(1)	6048(1)	33(1)
C(2A)	6651(1)	4205(1)	6577(1)	27(1)
C(3A)	7175(1)	3880(1)	7279(1)	27(1)
C(4A)	7116(1)	4047(1)	7961(1)	26(1)
C(5A)	6523(1)	4595(1)	8155(1)	22(1)
C(6A)	5787(1)	4327(1)	8556(1)	20(1)
C(7A)	5871(1)	3722(1)	8924(1)	21(1)
C(8A)	5218(1)	3497(1)	9307(1)	21(1)
C(9A)	4454(1)	3898(1)	9322(1)	20(1)
C(10A)	4350(1)	4496(1)	8943(1)	20(1)
C(11A)	5016(1)	4720(1)	8573(1)	20(1)
C(12A)	4887(1)	5369(1)	8160(1)	27(1)
C(13A)	5309(1)	5343(1)	7494(1)	26(1)
C(14A)	6046(1)	4976(1)	7457(1)	23(1)
C(15A)	6414(1)	4931(1)	6749(1)	26(1)
C(16A)	7376(1)	5241(1)	6813(1)	30(1)
C(17A)	5815(1)	3774(1)	6218(1)	39(1)
C(18A)	7169(1)	5056(1)	8716(1)	29(1)
C(19A)	5309(1)	2835(1)	9693(1)	27(1)
C(20A)	3616(1)	5376(1)	9436(1)	30(1)
C(21A)	5758(1)	5245(1)	6074(1)	37(1)
O(1B)	12026(1)	934(1)	4456(1)	28(1)
O(2B)	6313(1)	3047(1)	1644(1)	32(1)
O(3B)	6616(1)	2908(1)	3181(1)	25(1)
C(1B)	11457(1)	485(1)	3938(1)	26(1)
C(2B)	10545(1)	848(1)	3619(1)	21(1)
C(3B)	10667(1)	1254(1)	2963(1)	22(1)
C(4B)	10390(1)	1869(1)	2834(1)	22(1)
C(5B)	9884(1)	2269(1)	3311(1)	19(1)
C(6B)	8916(1)	2466(1)	2862(1)	19(1)
C(7B)	8730(1)	2542(1)	2092(1)	24(1)
C(8B)	7869(1)	2738(1)	1681(1)	26(1)
C(9B)	7166(1)	2865(1)	2056(1)	24(1)
C(10B)	7346(1)	2803(1)	2827(1)	21(1)
C(11B)	8217(1)	2606(1)	3236(1)	20(1)
C(12B)	8385(1)	2533(1)	4068(1)	23(1)
C(13B)	9119(1)	2026(1)	4345(1)	21(1)
C(14B)	9799(1)	1877(1)	4008(1)	19(1)
C(15B)	10478(1)	1318(1)	4276(1)	21(1)
C(16B)	11504(1)	1527(1)	4509(1)	25(1)
C(17B)	9724(1)	376(1)	3380(1)	28(1)
C(18B)	10430(1)	2920(1)	3536(1)	25(1)
C(19B)	7689(2)	2784(1)	845(1)	42(1)
C(20B)	6617(1)	3554(1)	3491(1)	34(1)
C(21B)	10244(1)	942(1)	4932(1)	29(1)

O(1C)	5389(1)	7929(1)	3000(1)	32(1)
O(2C)	10400(1)	4690(1)	3474(1)	29(1)
O(3C)	10726(1)	5860(1)	4260(1)	25(1)
C(1C)	5614(1)	7760(1)	2303(1)	27(1)
C(2C)	6458(1)	7307(1)	2485(1)	22(1)
C(3C)	6132(1)	6619(1)	2589(1)	24(1)
C(4C)	6524(1)	6214(1)	3128(1)	24(1)
C(5C)	7376(1)	6367(1)	3715(1)	24(1)
C(6C)	8184(1)	5918(1)	3627(1)	21(1)
C(7C)	8054(1)	5326(1)	3225(1)	24(1)
C(8C)	8788(1)	4917(1)	3158(1)	23(1)
C(9C)	9682(1)	5097(1)	3526(1)	22(1)
C(10C)	9818(1)	5682(1)	3931(1)	21(1)
C(11C)	9084(1)	6096(1)	3975(1)	22(1)
C(12C)	9262(1)	6763(1)	4346(1)	29(1)
C(13C)	8545(1)	7252(1)	3983(1)	26(1)
C(14C)	7671(1)	7088(1)	3671(1)	23(1)
C(15C)	6974(1)	7584(1)	3257(1)	24(1)
C(16C)	6134(1)	7713(1)	3605(1)	29(1)
C(17C)	7024(1)	7318(1)	1881(1)	27(1)
C(18C)	7178(2)	6225(1)	4497(1)	37(1)
C(19C)	8632(1)	4304(1)	2687(1)	32(1)
C(20C)	11013(1)	5618(1)	5010(1)	34(1)
C(21C)	7424(1)	8253(1)	3168(1)	32(1)

Table 3. Bond lengths [Å] and angles [°] for JLS03 (CCDC 701799).

O(1A)-C(16A)	1.435(2)	C(14B)-C(15B)	1.521(2)
O(1A)-C(1A)	1.441(2)	C(15B)-C(21B)	1.529(3)
O(2A)-C(9A)	1.365(2)	C(15B)-C(16B)	1.538(2)
O(3A)-C(10A)	1.383(2)	O(1C)-C(1C)	1.433(2)
O(3A)-C(20A)	1.440(2)	O(1C)-C(16C)	1.449(2)
C(1A)-C(2A)	1.532(3)	O(2C)-C(9C)	1.362(2)
C(2A)-C(3A)	1.502(3)	O(3C)-C(10C)	1.391(2)
C(2A)-C(17A)	1.537(3)	O(3C)-C(20C)	1.435(2)
C(2A)-C(15A)	1.560(3)	C(1C)-C(2C)	1.525(3)
C(3A)-C(4A)	1.318(3)	C(2C)-C(3C)	1.501(3)
C(4A)-C(5A)	1.504(3)	C(2C)-C(17C)	1.527(3)
C(5A)-C(14A)	1.530(3)	C(2C)-C(15C)	1.560(3)
C(5A)-C(6A)	1.540(3)	C(3C)-C(4C)	1.319(3)
C(5A)-C(18A)	1.554(3)	C(4C)-C(5C)	1.497(3)
C(6A)-C(7A)	1.392(2)	C(5C)-C(14C)	1.533(3)
C(6A)-C(11A)	1.395(2)	C(5C)-C(6C)	1.536(3)
C(7A)-C(8A)	1.388(2)	C(5C)-C(18C)	1.554(3)
C(8A)-C(9A)	1.395(3)	C(6C)-C(11C)	1.388(2)
C(8A)-C(19A)	1.510(2)	C(6C)-C(7C)	1.401(3)
C(9A)-C(10A)	1.389(2)	C(7C)-C(8C)	1.390(3)
C(10A)-C(11A)	1.389(2)	C(8C)-C(9C)	1.392(3)
C(11A)-C(12A)	1.509(2)	C(8C)-C(19C)	1.502(3)
C(12A)-C(13A)	1.491(3)	C(9C)-C(10C)	1.392(3)
C(13A)-C(14A)	1.331(2)	C(10C)-C(11C)	1.386(3)
C(14A)-C(15A)	1.519(3)	C(11C)-C(12C)	1.512(3)
C(15A)-C(16A)	1.533(3)	C(12C)-C(13C)	1.496(3)
C(15A)-C(21A)	1.534(3)	C(13C)-C(14C)	1.332(3)
O(1B)-C(16B)	1.442(2)	C(14C)-C(15C)	1.518(3)
O(1B)-C(1B)	1.445(2)	C(15C)-C(21C)	1.535(3)
O(2B)-C(9B)	1.369(2)	C(15C)-C(16C)	1.537(3)
O(3B)-C(10B)	1.391(2)	C(16A)-O(1A)-C(1A)	109.14(15)
O(3B)-C(20B)	1.428(2)	C(10A)-O(3A)-C(20A)	114.02(14)
C(1B)-C(2B)	1.533(2)	O(1A)-C(1A)-C(2A)	106.45(16)
C(2B)-C(3B)	1.502(3)	C(3A)-C(2A)-C(1A)	109.07(16)
C(2B)-C(17B)	1.531(3)	C(3A)-C(2A)-C(17A)	109.39(17)
C(2B)-C(15B)	1.558(2)	C(1A)-C(2A)-C(17A)	111.47(17)
C(3B)-C(4B)	1.318(2)	C(3A)-C(2A)-C(15A)	109.67(17)
C(4B)-C(5B)	1.506(2)	C(1A)-C(2A)-C(15A)	101.46(16)
C(5B)-C(14B)	1.535(3)	C(17A)-C(2A)-C(15A)	115.44(17)
C(5B)-C(6B)	1.537(2)	C(4A)-C(3A)-C(2A)	124.98(19)
C(5B)-C(18B)	1.556(2)	C(3A)-C(4A)-C(5A)	125.32(19)
C(6B)-C(7B)	1.389(2)	C(4A)-C(5A)-C(14A)	111.02(17)
C(6B)-C(11B)	1.389(2)	C(4A)-C(5A)-C(6A)	111.13(15)
C(7B)-C(8B)	1.386(3)	C(14A)-C(5A)-C(6A)	109.66(15)
C(8B)-C(9B)	1.389(3)	C(4A)-C(5A)-C(18A)	107.35(16)
C(8B)-C(19B)	1.504(3)	C(14A)-C(5A)-C(18A)	110.74(15)
C(9B)-C(10B)	1.388(3)	C(6A)-C(5A)-C(18A)	106.84(15)
C(10B)-C(11B)	1.396(2)	C(7A)-C(6A)-C(11A)	118.66(18)
C(11B)-C(12B)	1.500(2)	C(7A)-C(6A)-C(5A)	123.12(17)
C(12B)-C(13B)	1.499(2)	C(11A)-C(6A)-C(5A)	118.18(17)
C(13B)-C(14B)	1.325(2)	C(8A)-C(7A)-C(6A)	122.53(18)

C(7A)-C(8A)-C(9A)	118.11(18)	C(6B)-C(11B)-C(10B)	119.17(18)
C(7A)-C(8A)-C(19A)	122.05(17)	C(6B)-C(11B)-C(12B)	120.61(17)
C(9A)-C(8A)-C(19A)	119.84(17)	C(10B)-C(11B)-C(12B)	120.21(17)
O(2A)-C(9A)-C(10A)	121.73(17)	C(13B)-C(12B)-C(11B)	111.08(16)
O(2A)-C(9A)-C(8A)	118.26(17)	C(14B)-C(13B)-C(12B)	124.23(18)
C(10A)-C(9A)-C(8A)	120.00(18)	C(13B)-C(14B)-C(15B)	122.10(17)
O(3A)-C(10A)-C(9A)	117.39(17)	C(13B)-C(14B)-C(5B)	118.75(17)
O(3A)-C(10A)-C(11A)	121.25(17)	C(15B)-C(14B)-C(5B)	119.13(16)
C(9A)-C(10A)-C(11A)	121.24(17)	C(14B)-C(15B)-C(21B)	112.37(16)
C(10A)-C(11A)-C(6A)	119.40(17)	C(14B)-C(15B)-C(16B)	115.01(15)
C(10A)-C(11A)-C(12A)	120.44(17)	C(21B)-C(15B)-C(16B)	107.24(16)
C(6A)-C(11A)-C(12A)	120.10(17)	C(14B)-C(15B)-C(2B)	110.76(15)
C(13A)-C(12A)-C(11A)	110.63(16)	C(21B)-C(15B)-C(2B)	111.63(15)
C(14A)-C(13A)-C(12A)	123.67(19)	C(16B)-C(15B)-C(2B)	99.10(14)
C(13A)-C(14A)-C(15A)	121.84(18)	O(1B)-C(16B)-C(15B)	105.02(15)
C(13A)-C(14A)-C(5A)	118.05(18)	C(1C)-O(1C)-C(16C)	109.10(14)
C(15A)-C(14A)-C(5A)	120.11(16)	C(10C)-O(3C)-C(20C)	112.42(14)
C(14A)-C(15A)-C(16A)	114.14(17)	O(1C)-C(1C)-C(2C)	106.71(15)
C(14A)-C(15A)-C(21A)	112.67(16)	C(3C)-C(2C)-C(1C)	108.54(16)
C(16A)-C(15A)-C(21A)	107.48(17)	C(3C)-C(2C)-C(17C)	110.42(15)
C(14A)-C(15A)-C(2A)	111.58(16)	C(1C)-C(2C)-C(17C)	112.50(16)
C(16A)-C(15A)-C(2A)	99.14(15)	C(3C)-C(2C)-C(15C)	108.92(16)
C(21A)-C(15A)-C(2A)	111.01(17)	C(1C)-C(2C)-C(15C)	101.13(15)
O(1A)-C(16A)-C(15A)	106.47(16)	C(17C)-C(2C)-C(15C)	114.85(16)
C(16B)-O(1B)-C(1B)	109.14(13)	C(4C)-C(3C)-C(2C)	125.08(18)
C(10B)-O(3B)-C(20B)	113.18(14)	C(3C)-C(4C)-C(5C)	124.87(19)
O(1B)-C(1B)-C(2B)	106.45(14)	C(4C)-C(5C)-C(14C)	111.29(16)
C(3B)-C(2B)-C(17B)	109.82(16)	C(4C)-C(5C)-C(6C)	110.47(16)
C(3B)-C(2B)-C(1B)	108.71(16)	C(14C)-C(5C)-C(6C)	108.89(16)
C(17B)-C(2B)-C(1B)	112.45(16)	C(4C)-C(5C)-C(18C)	109.14(16)
C(3B)-C(2B)-C(15B)	109.02(15)	C(14C)-C(5C)-C(18C)	109.82(16)
C(17B)-C(2B)-C(15B)	115.07(16)	C(6C)-C(5C)-C(18C)	107.13(15)
C(1B)-C(2B)-C(15B)	101.36(15)	C(11C)-C(6C)-C(7C)	118.14(18)
C(4B)-C(3B)-C(2B)	125.12(18)	C(11C)-C(6C)-C(5C)	118.87(17)
C(3B)-C(4B)-C(5B)	125.37(18)	C(7C)-C(6C)-C(5C)	122.97(17)
C(4B)-C(5B)-C(14B)	110.38(16)	C(8C)-C(7C)-C(6C)	122.59(18)
C(4B)-C(5B)-C(6B)	110.43(15)	C(7C)-C(8C)-C(9C)	118.30(18)
C(14B)-C(5B)-C(6B)	110.24(15)	C(7C)-C(8C)-C(19C)	121.33(18)
C(4B)-C(5B)-C(18B)	108.57(15)	C(9C)-C(8C)-C(19C)	120.36(18)
C(14B)-C(5B)-C(18B)	110.42(15)	O(2C)-C(9C)-C(8C)	118.38(18)
C(6B)-C(5B)-C(18B)	106.73(15)	O(2C)-C(9C)-C(10C)	122.08(18)
C(7B)-C(6B)-C(11B)	118.64(17)	C(8C)-C(9C)-C(10C)	119.54(18)
C(7B)-C(6B)-C(5B)	121.87(17)	C(11C)-C(10C)-O(3C)	120.57(17)
C(11B)-C(6B)-C(5B)	119.41(17)	C(11C)-C(10C)-C(9C)	121.64(18)
C(8B)-C(7B)-C(6B)	122.58(19)	O(3C)-C(10C)-C(9C)	117.69(17)
C(7B)-C(8B)-C(9B)	118.58(19)	C(10C)-C(11C)-C(6C)	119.74(18)
C(7B)-C(8B)-C(19B)	120.58(19)	C(10C)-C(11C)-C(12C)	120.45(17)
C(9B)-C(8B)-C(19B)	120.79(18)	C(6C)-C(11C)-C(12C)	119.63(17)
O(2B)-C(9B)-C(10B)	122.40(18)	C(13C)-C(12C)-C(11C)	110.87(16)
O(2B)-C(9B)-C(8B)	118.09(18)	C(14C)-C(13C)-C(12C)	123.30(19)
C(10B)-C(9B)-C(8B)	119.51(18)	C(13C)-C(14C)-C(15C)	122.37(18)
C(9B)-C(10B)-O(3B)	117.92(17)	C(13C)-C(14C)-C(5C)	118.35(17)
C(9B)-C(10B)-C(11B)	121.49(18)	C(15C)-C(14C)-C(5C)	119.26(17)
O(3B)-C(10B)-C(11B)	120.50(17)	C(14C)-C(15C)-C(21C)	112.13(16)

C(14C)-C(15C)-C(16C)	115.08(16)
C(21C)-C(15C)-C(16C)	107.55(16)
C(14C)-C(15C)-C(2C)	111.01(15)
C(21C)-C(15C)-C(2C)	110.98(16)
C(16C)-C(15C)-C(2C)	99.40(15)
O(1C)-C(16C)-C(15C)	106.10(15)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for JLS03 (CCDC 701799). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1A)	307(9)	288(8)	454(10)	-52(7)	217(7)	-26(7)
O(2A)	231(8)	299(8)	262(8)	46(7)	69(7)	0(6)
O(3A)	206(8)	206(7)	243(8)	-56(6)	54(6)	12(6)
C(1A)	290(13)	321(13)	414(15)	-52(11)	124(11)	-40(10)
C(2A)	197(12)	307(12)	342(13)	-44(10)	125(10)	-21(10)
C(3A)	207(12)	197(11)	445(15)	-19(10)	132(11)	1(9)
C(4A)	201(12)	195(11)	386(14)	33(10)	70(10)	18(9)
C(5A)	170(11)	209(11)	284(13)	6(9)	51(9)	3(9)
C(6A)	189(11)	186(10)	213(12)	-34(9)	25(9)	-37(9)
C(7A)	159(11)	208(11)	253(12)	-55(9)	-5(9)	9(9)
C(8A)	217(12)	207(11)	178(11)	-23(9)	1(9)	-34(9)
C(9A)	179(11)	236(11)	173(11)	-39(9)	35(9)	-64(9)
C(10A)	171(11)	204(11)	210(12)	-49(9)	29(9)	-15(9)
C(11A)	187(11)	170(10)	239(12)	-16(9)	43(9)	-15(9)
C(12A)	242(12)	235(11)	352(14)	70(10)	112(10)	42(9)
C(13A)	233(12)	231(11)	336(13)	82(10)	86(10)	18(9)
C(14A)	187(11)	199(11)	315(13)	14(9)	76(10)	-24(9)
C(15A)	217(12)	280(12)	305(13)	26(10)	105(10)	41(9)
C(16A)	309(13)	236(12)	401(14)	-1(10)	188(11)	21(10)
C(17A)	334(14)	408(14)	450(15)	-97(12)	145(12)	-83(11)
C(18A)	232(12)	261(12)	382(14)	12(10)	88(10)	-28(9)
C(19A)	252(12)	269(12)	267(12)	42(10)	1(10)	-15(9)
C(20A)	282(13)	302(12)	327(13)	-132(10)	89(10)	0(10)
C(21A)	332(14)	480(15)	337(14)	53(12)	164(11)	63(11)
O(1B)	233(8)	282(8)	309(9)	-63(7)	0(7)	71(6)
O(2B)	204(8)	471(9)	279(9)	128(7)	39(7)	32(7)
O(3B)	217(8)	266(8)	284(8)	-18(6)	81(7)	26(6)
C(1B)	257(12)	244(12)	264(12)	-35(9)	40(10)	14(9)
C(2B)	183(11)	217(11)	232(12)	-26(9)	62(9)	25(9)
C(3B)	187(11)	280(12)	213(12)	-75(9)	56(9)	-14(9)
C(4B)	211(12)	273(12)	191(11)	9(9)	62(9)	3(9)
C(5B)	186(11)	217(11)	185(11)	19(9)	53(9)	9(9)
C(6B)	189(11)	169(10)	216(12)	12(9)	44(9)	-25(8)
C(7B)	224(12)	281(12)	234(12)	42(10)	83(10)	-28(9)
C(8B)	234(12)	309(12)	224(12)	60(10)	45(10)	-18(10)
C(9B)	186(11)	233(11)	269(13)	66(9)	0(10)	-19(9)
C(10B)	185(11)	184(10)	263(12)	15(9)	73(9)	-24(9)
C(11B)	215(12)	158(10)	213(12)	5(9)	37(9)	-24(8)
C(12B)	191(11)	271(12)	220(12)	16(9)	51(9)	25(9)
C(13B)	233(12)	235(11)	170(11)	32(9)	38(9)	31(9)
C(14B)	178(11)	193(11)	204(11)	-31(9)	26(9)	-15(9)
C(15B)	197(11)	220(11)	216(12)	-2(9)	53(9)	33(9)
C(16B)	206(12)	278(12)	248(12)	-64(9)	33(9)	70(9)
C(17B)	288(13)	266(12)	307(13)	-19(10)	85(10)	12(10)
C(18B)	207(11)	245(11)	278(12)	31(9)	25(9)	-21(9)
C(19B)	296(13)	685(17)	274(14)	90(12)	69(11)	-5(12)
C(20B)	301(13)	327(13)	399(15)	-37(11)	77(11)	101(10)

C(21B)	307(13)	272(12)	302(13)	39(10)	94(10)	89(10)
O(1C)	242(8)	444(9)	254(9)	-78(7)	31(7)	90(7)
O(2C)	236(8)	248(8)	376(9)	6(7)	71(7)	32(6)
O(3C)	210(8)	319(8)	207(8)	35(6)	24(6)	24(6)
C(1C)	240(12)	306(12)	263(13)	-48(10)	58(10)	8(10)
C(2C)	188(11)	247(11)	214(12)	-46(9)	32(9)	20(9)
C(3C)	183(11)	276(12)	240(12)	-72(10)	35(9)	-15(9)
C(4C)	195(11)	252(11)	304(13)	-14(10)	87(10)	-16(9)
C(5C)	217(12)	304(12)	216(12)	23(10)	84(9)	34(9)
C(6C)	232(12)	236(11)	178(11)	43(9)	80(9)	18(9)
C(7C)	232(12)	250(11)	231(12)	79(9)	60(9)	-37(9)
C(8C)	255(12)	202(11)	242(12)	53(9)	84(10)	2(9)
C(9C)	256(12)	209(11)	217(12)	81(9)	100(10)	63(9)
C(10C)	197(11)	286(12)	156(11)	52(9)	40(9)	-4(9)
C(11C)	227(12)	260(11)	172(11)	19(9)	61(9)	11(9)
C(12C)	241(12)	362(13)	243(12)	-86(10)	15(10)	70(10)
C(13C)	278(13)	268(12)	230(12)	-91(10)	23(10)	67(10)
C(14C)	247(12)	276(12)	185(11)	-75(9)	68(9)	49(9)
C(15C)	209(11)	261(12)	243(12)	-66(9)	32(9)	32(9)
C(16C)	252(12)	346(13)	257(12)	-89(10)	20(10)	90(10)
C(17C)	227(12)	295(12)	268(13)	-38(10)	31(10)	2(9)
C(18C)	339(14)	501(15)	309(14)	92(11)	161(11)	111(11)
C(19C)	307(13)	245(12)	410(15)	12(10)	87(11)	-16(10)
C(20C)	282(13)	435(14)	257(13)	35(11)	-11(10)	23(11)
C(21C)	297(13)	271(12)	349(14)	-78(10)	-16(11)	54(10)

Table 5. Hydrogen bonds for JLS03 (CCDC 701799) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2A)-H(2A)...O(1B)#1	0.84	1.91	2.6954(18)	155.0
O(2A)-H(2A)...O(3A)	0.84	2.31	2.7465(17)	112.3
O(2B)-H(2B)...O(1C)#2	0.84	1.97	2.7397(19)	152.2
O(2B)-H(2B)...O(3B)	0.84	2.37	2.7747(18)	110.5
O(2C)-H(2C)...O(1A)#3	0.84	1.94	2.7345(19)	158.5
O(2C)-H(2C)...O(3C)	0.84	2.34	2.7633(18)	111.5

Symmetry transformations used to generate equivalent atoms:
#1 x-1,-y+1/2,z+1/2
#2 -x+1,y-1/2,-z+1/2
#3 -x+2,-y+1,-z+1