APPENDIX A

Spectra and X-Ray Crystrallographic Data: Early Efforts Toward the Synthesis of Zoanthenol

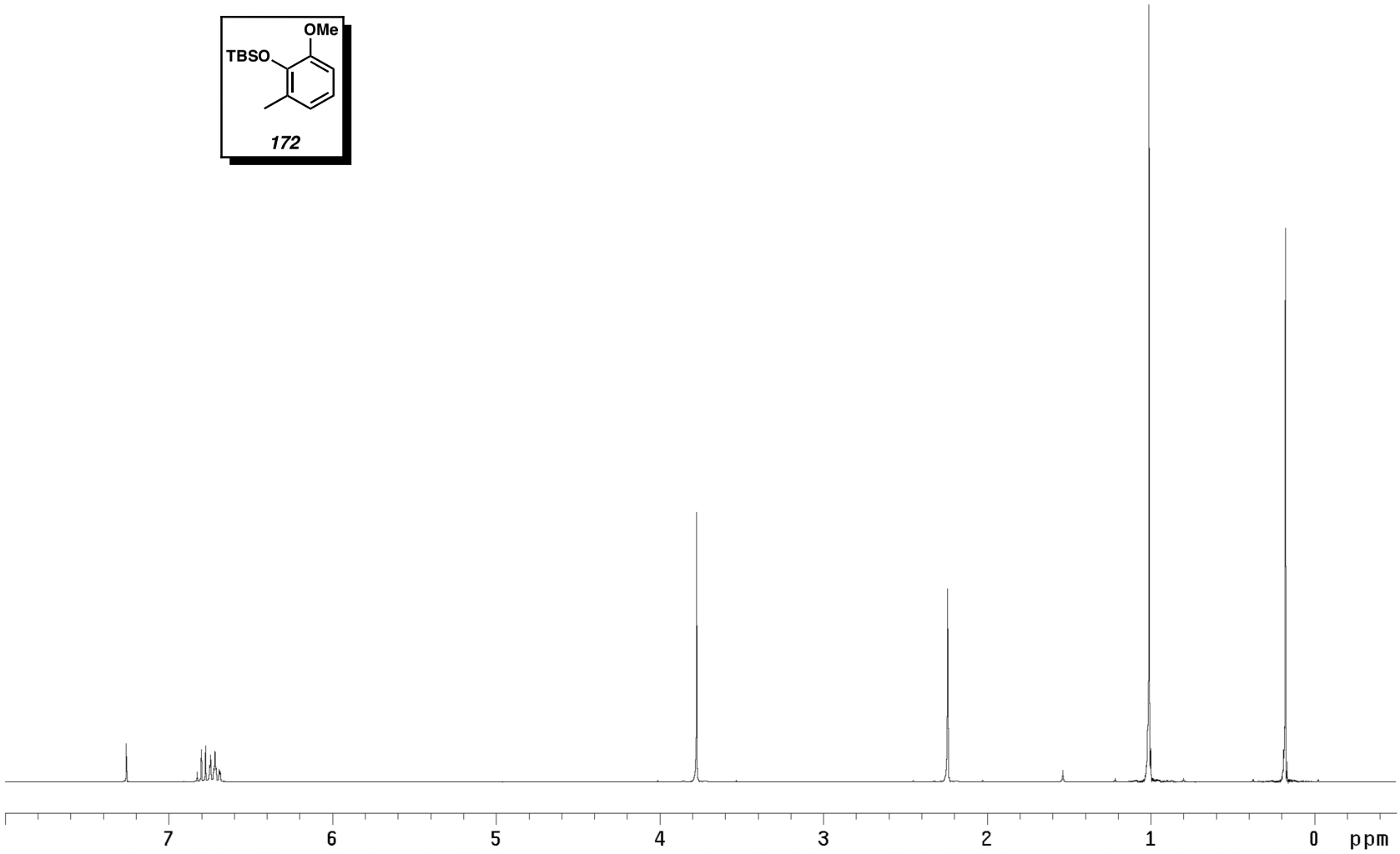

Figure A.1 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **172**.

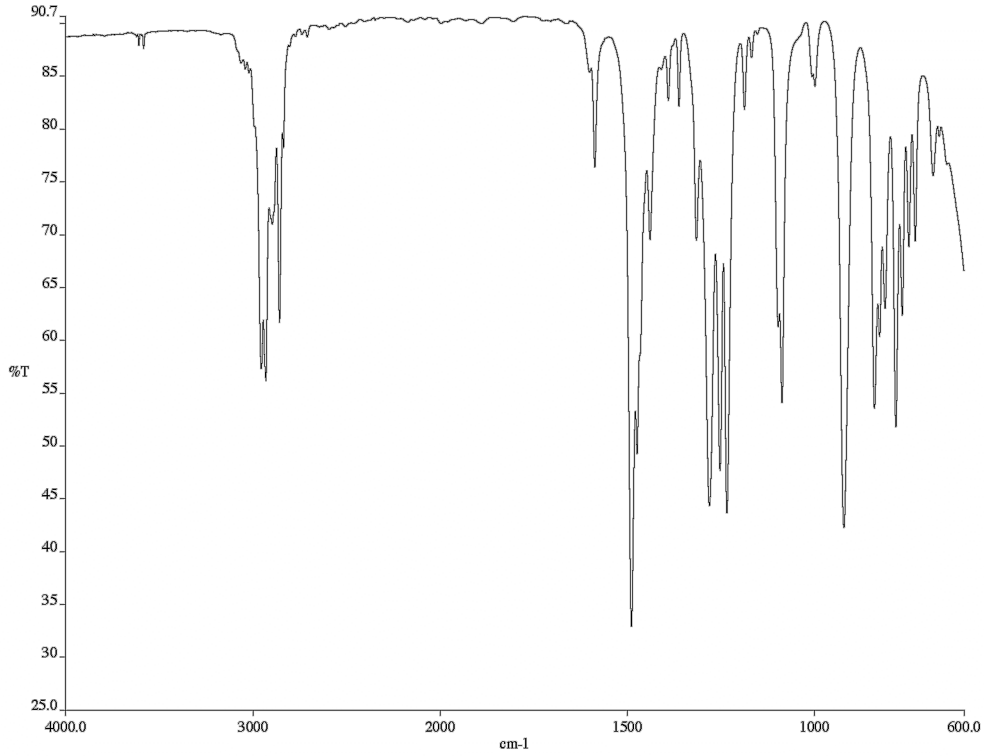

Figure A.2 Infrared spectrum (thin film/NaCl) of compound **172**.

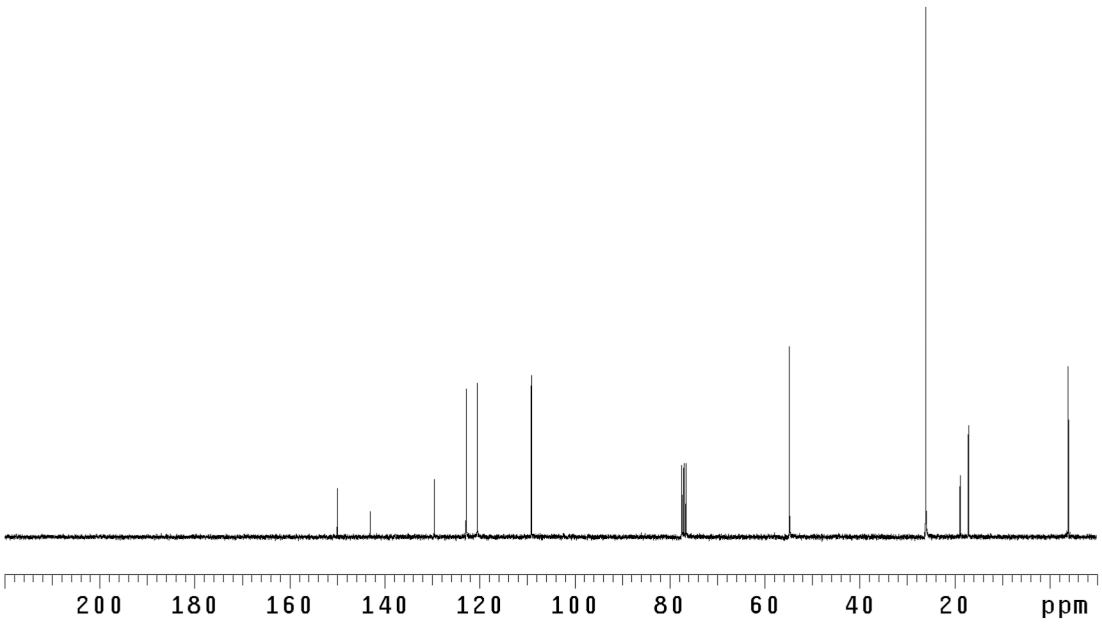

Figure A.3 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **172**.

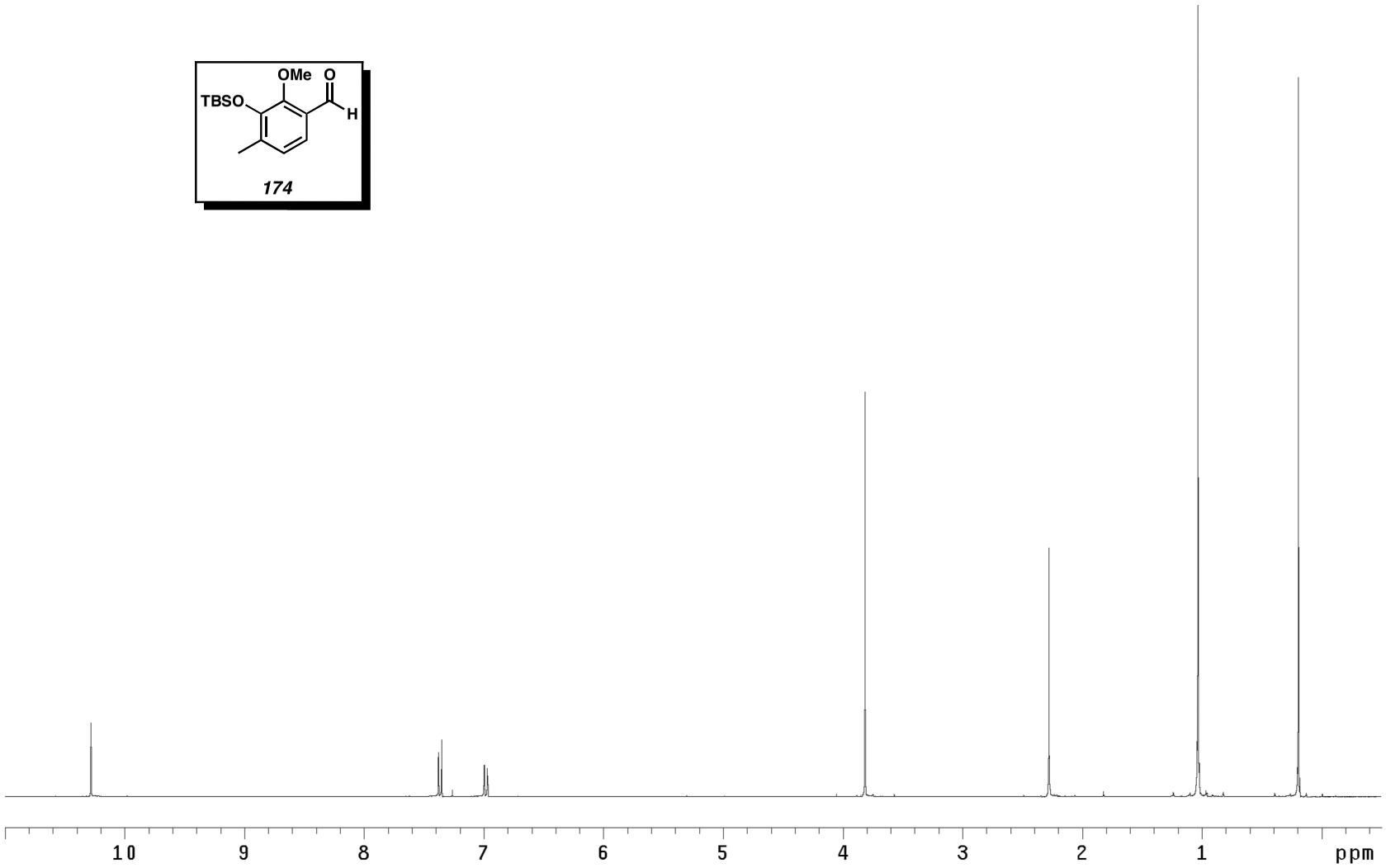

Figure A.4 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **174**.

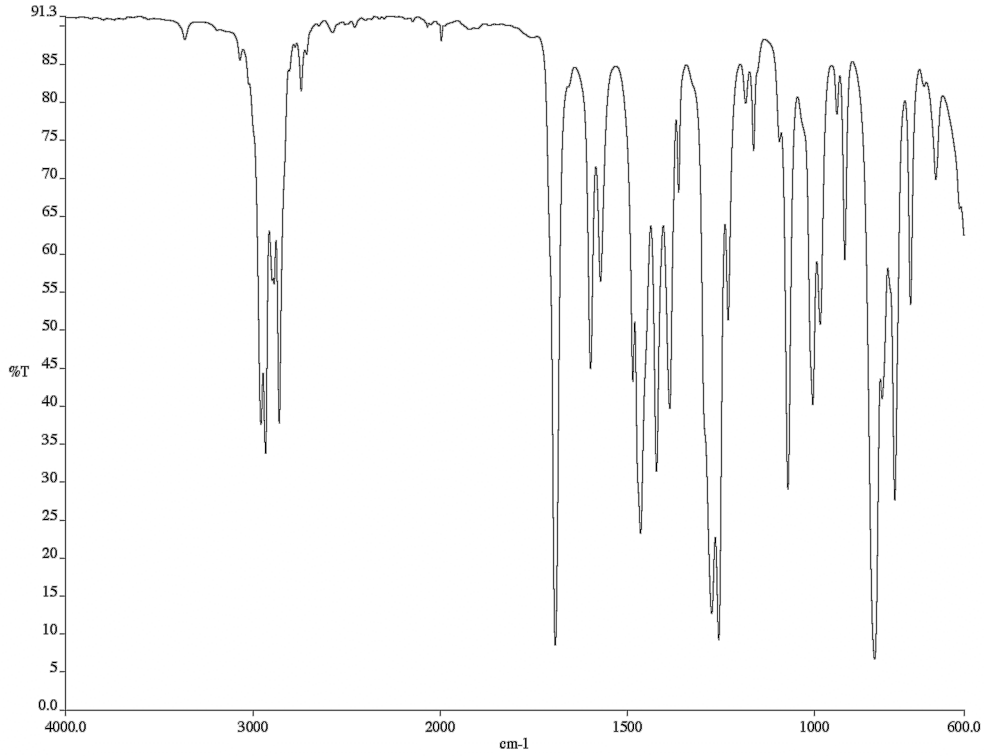

Figure A.5 Infrared spectrum (thin film/NaCl) of compound **174**.

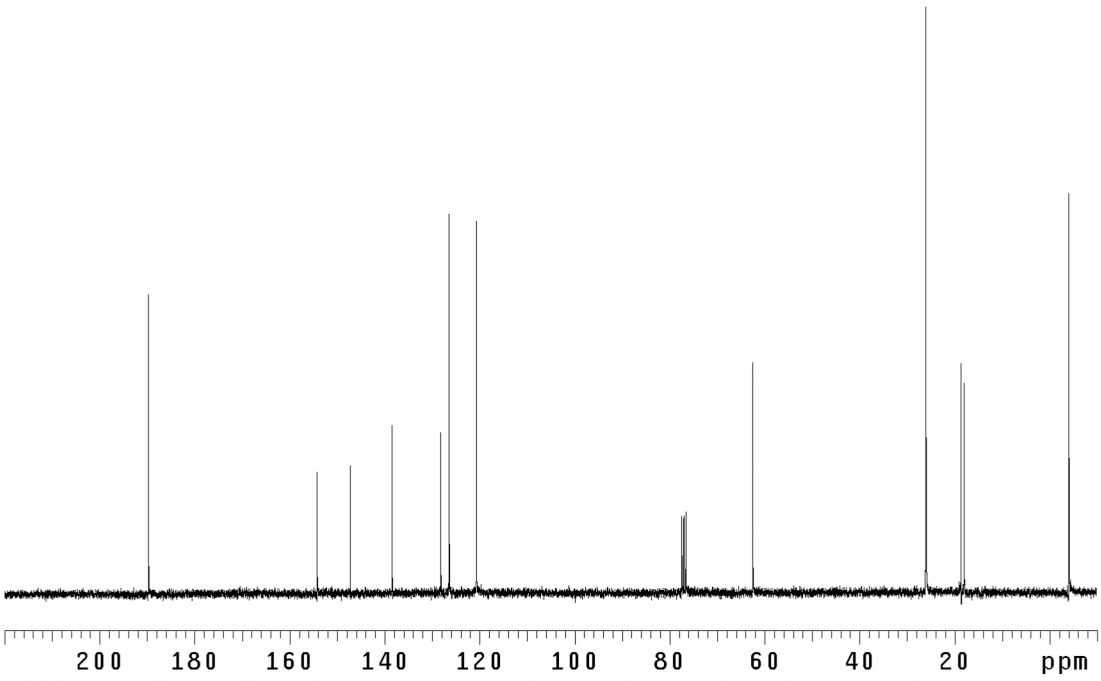

Figure A.6 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **174**.

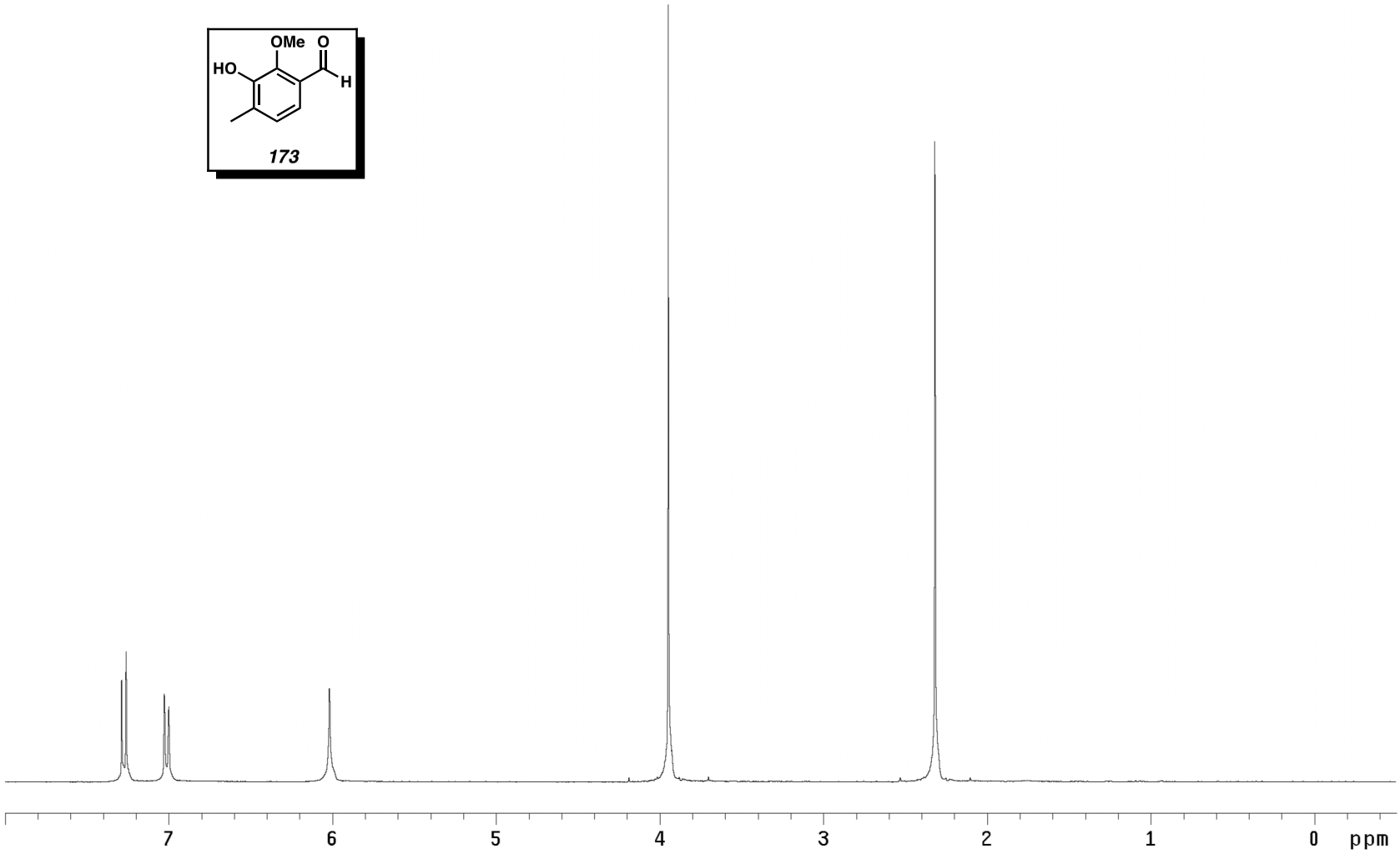

Figure A.7 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **173**.

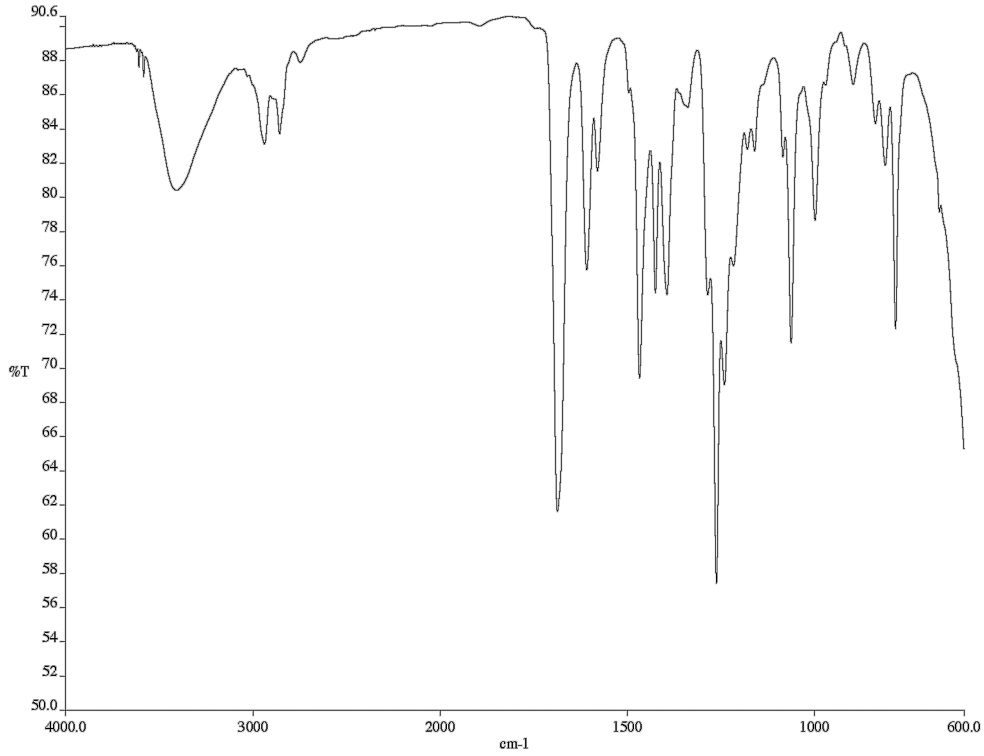

Figure A.8 Infrared spectrum (thin film/NaCl) of compound **173**.

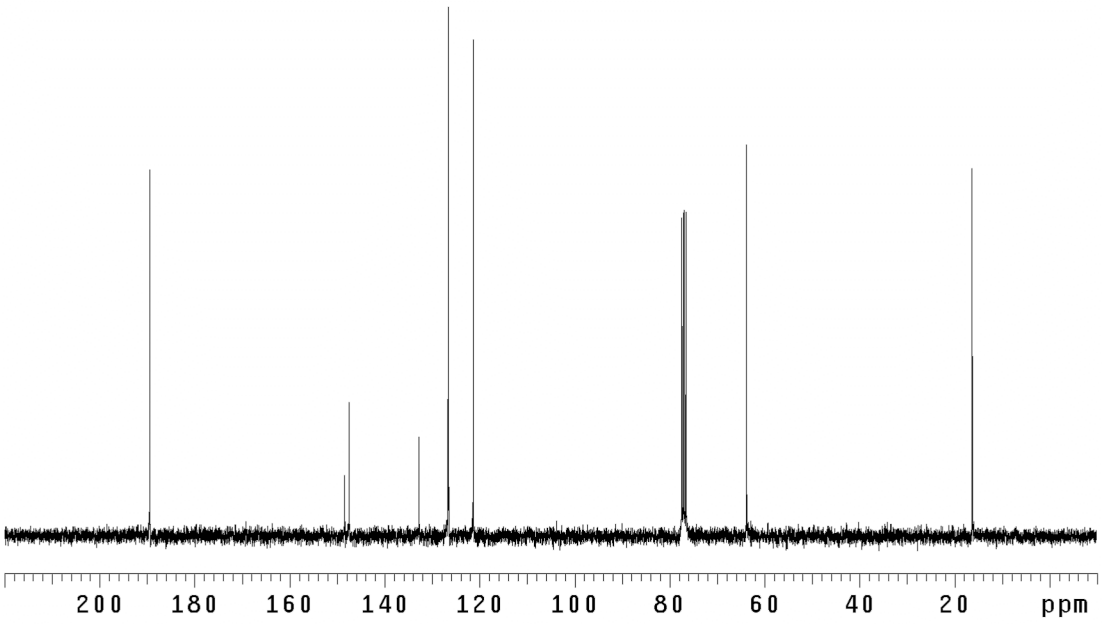

Figure A.9 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **173**.

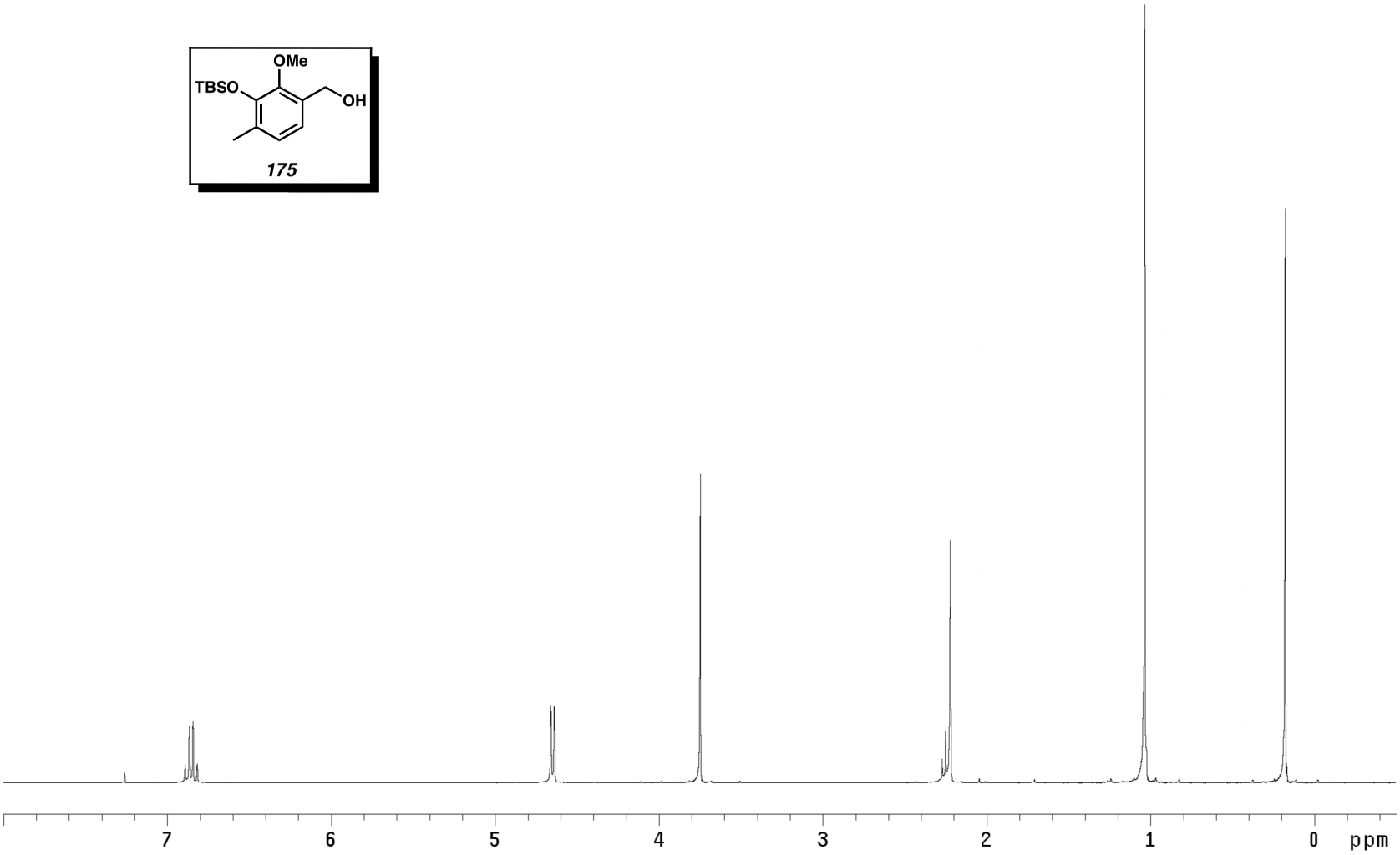

Figure A.10 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **175**.

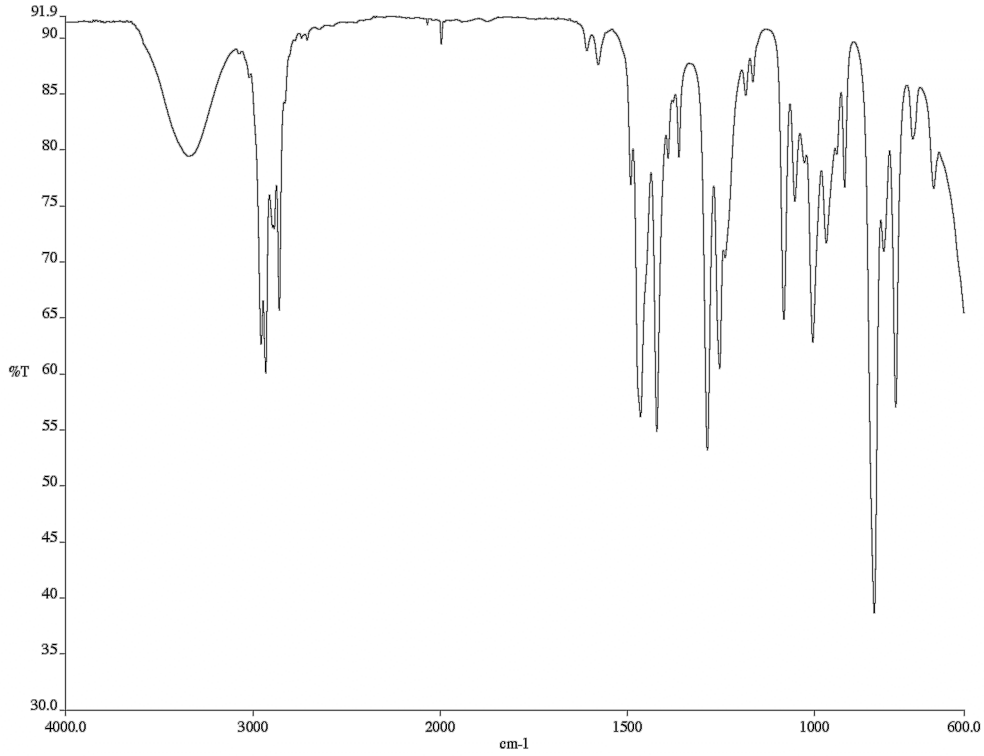

Figure A.11 Infrared spectrum (thin film/NaCl) of compound **175**.

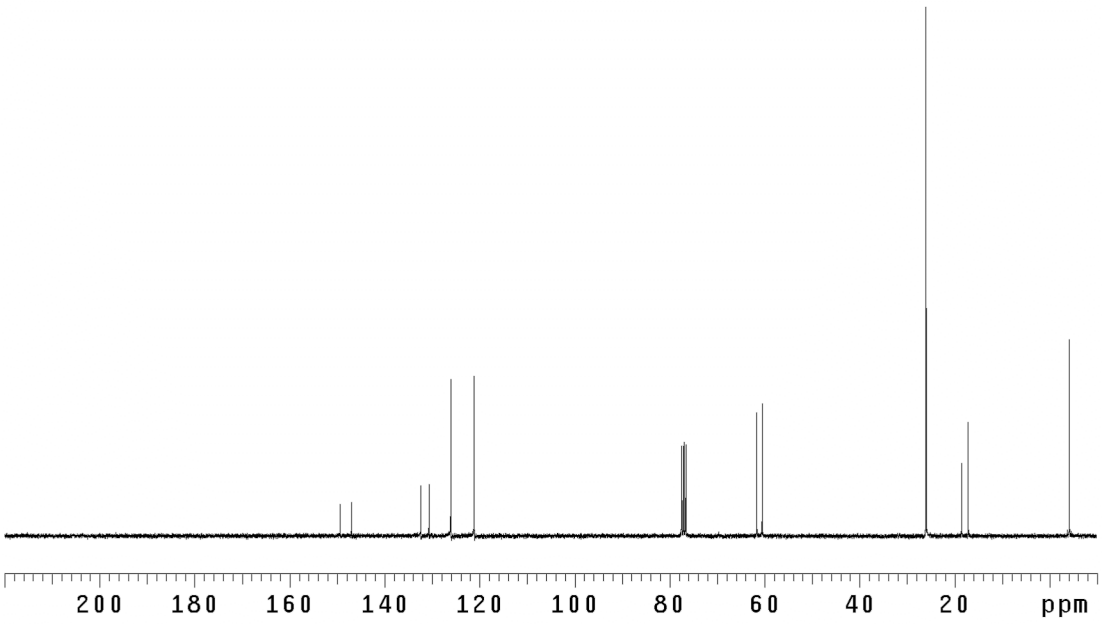

Figure A.12 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **175**.

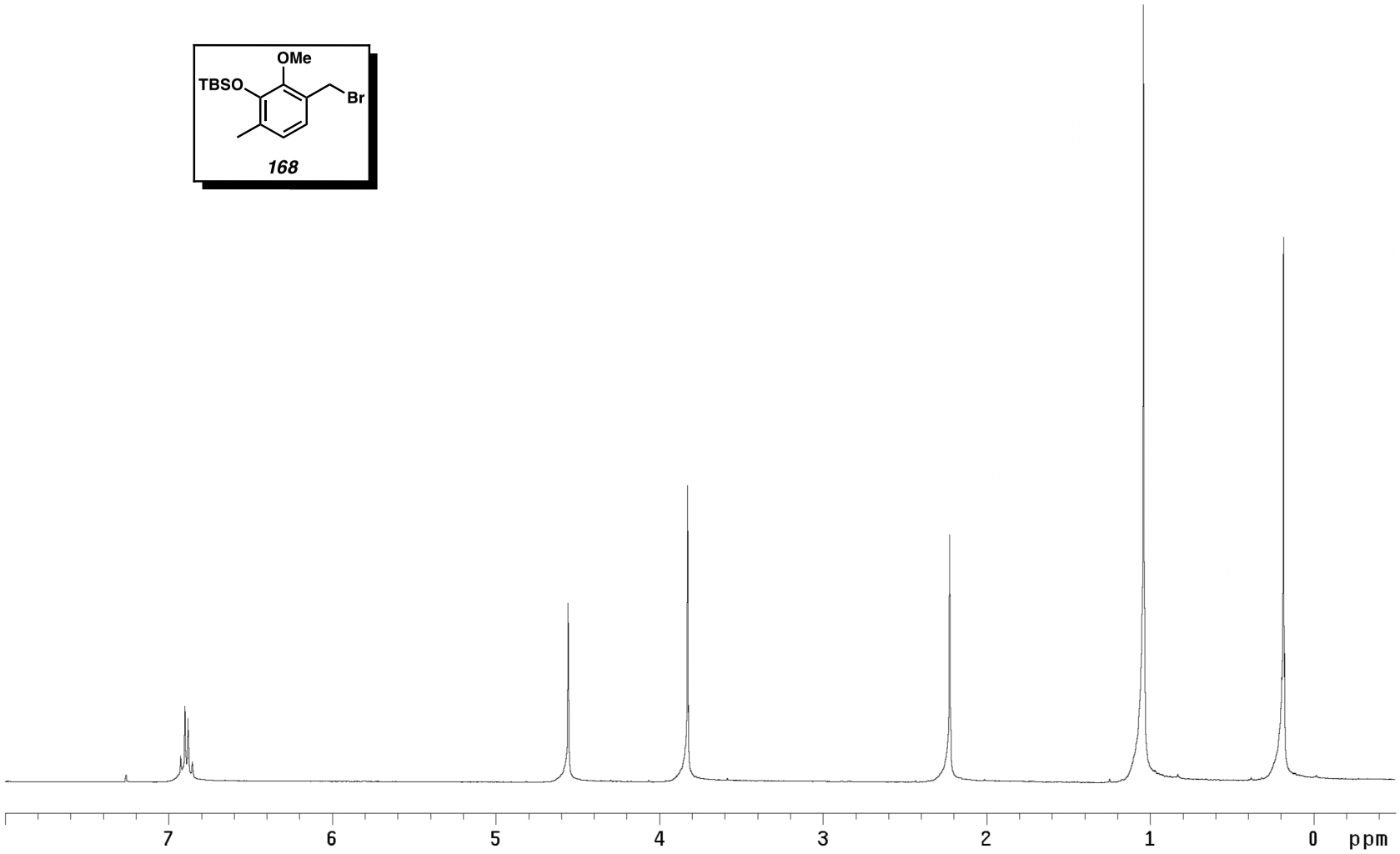

Figure A.13 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **168**.

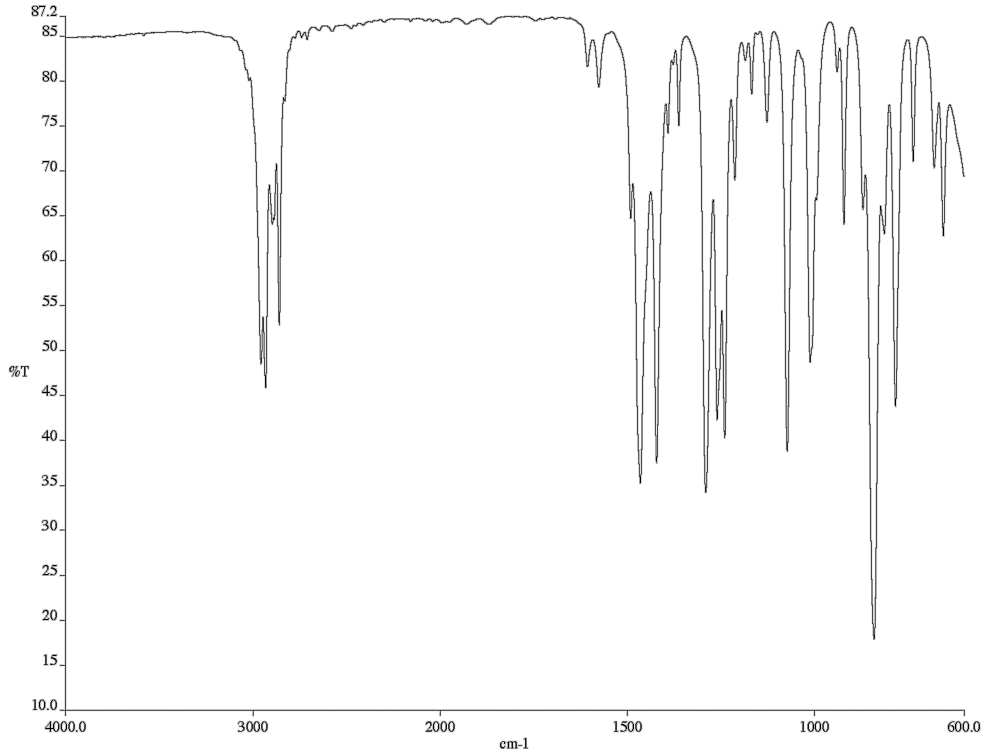

Figure A.14 Infrared spectrum (thin film/NaCl) of compound **168**.

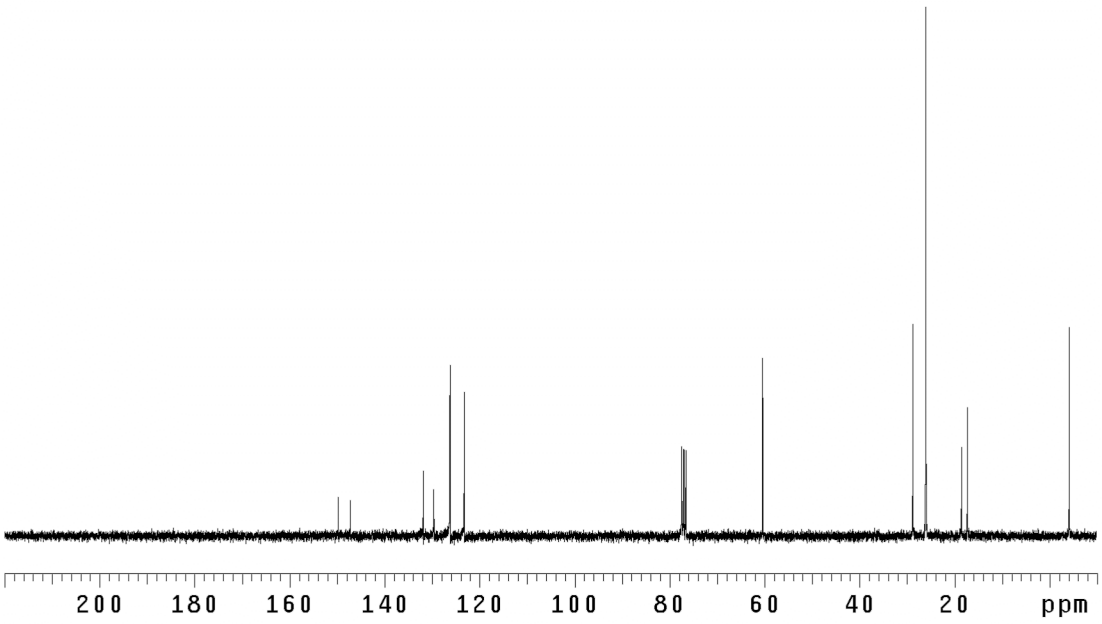

Figure A.15 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **168**.

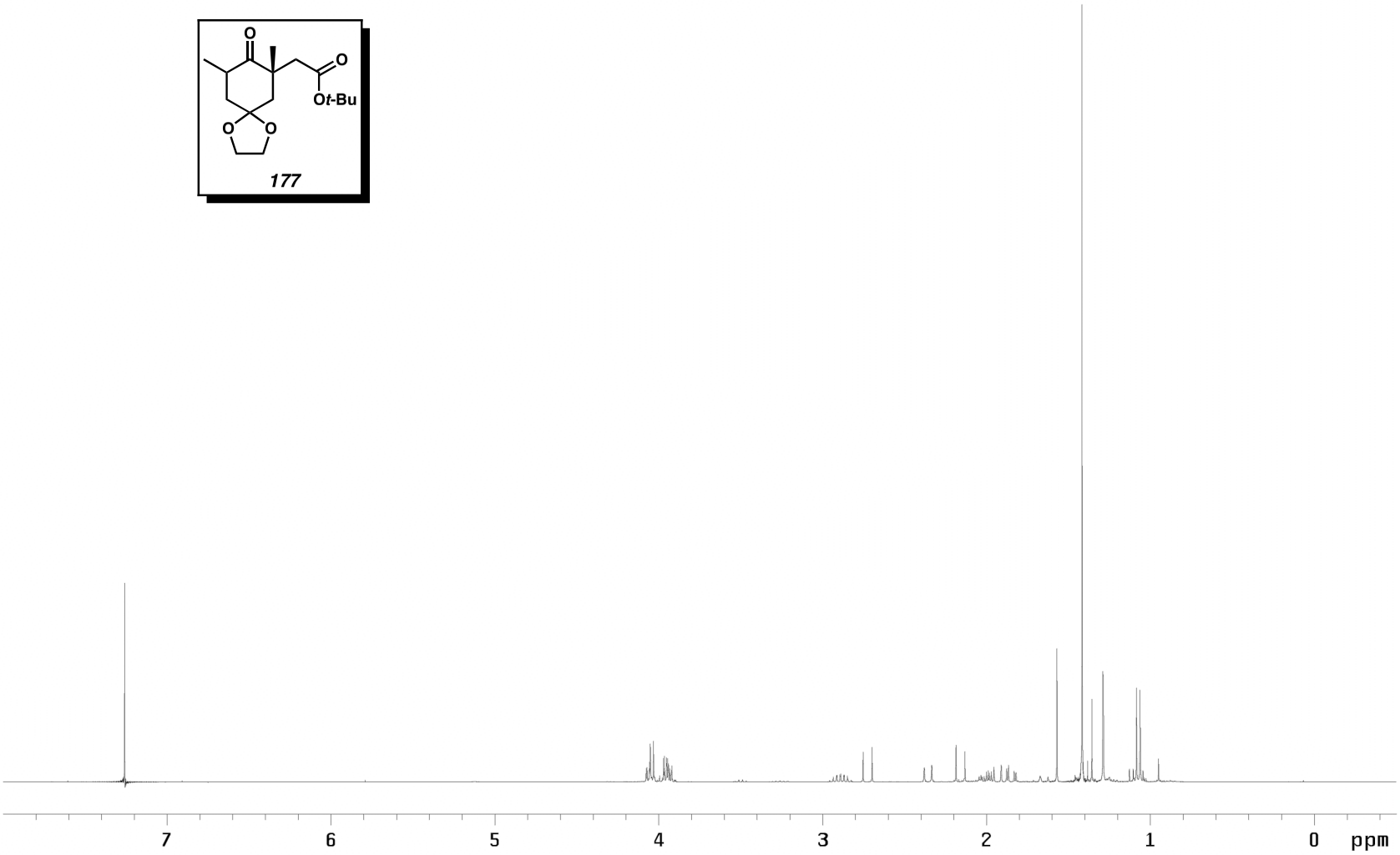

Figure A.16 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (+)-**177**.

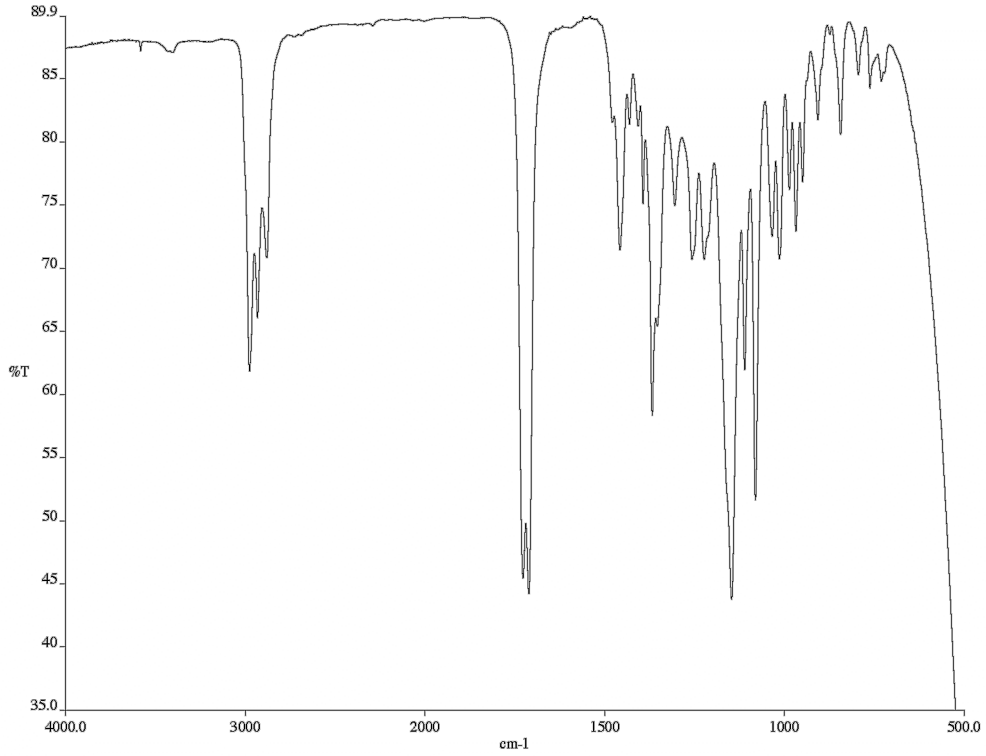

Figure A.17 Infrared spectrum (thin film/NaCl) of compound (+)-**177**.

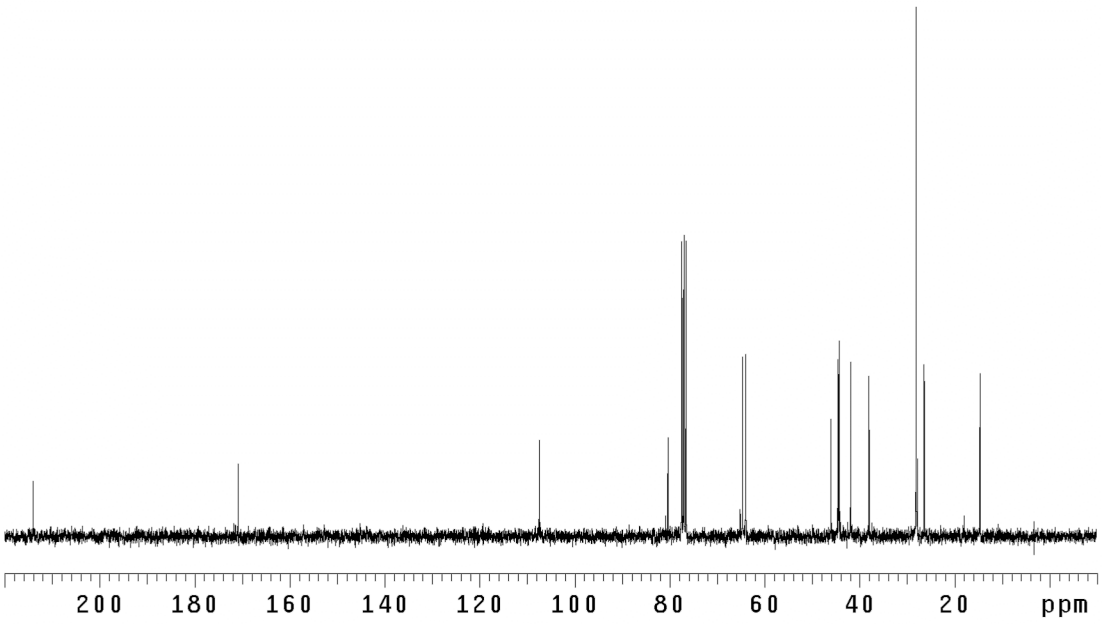

Figure A.18 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (+)-**177**.

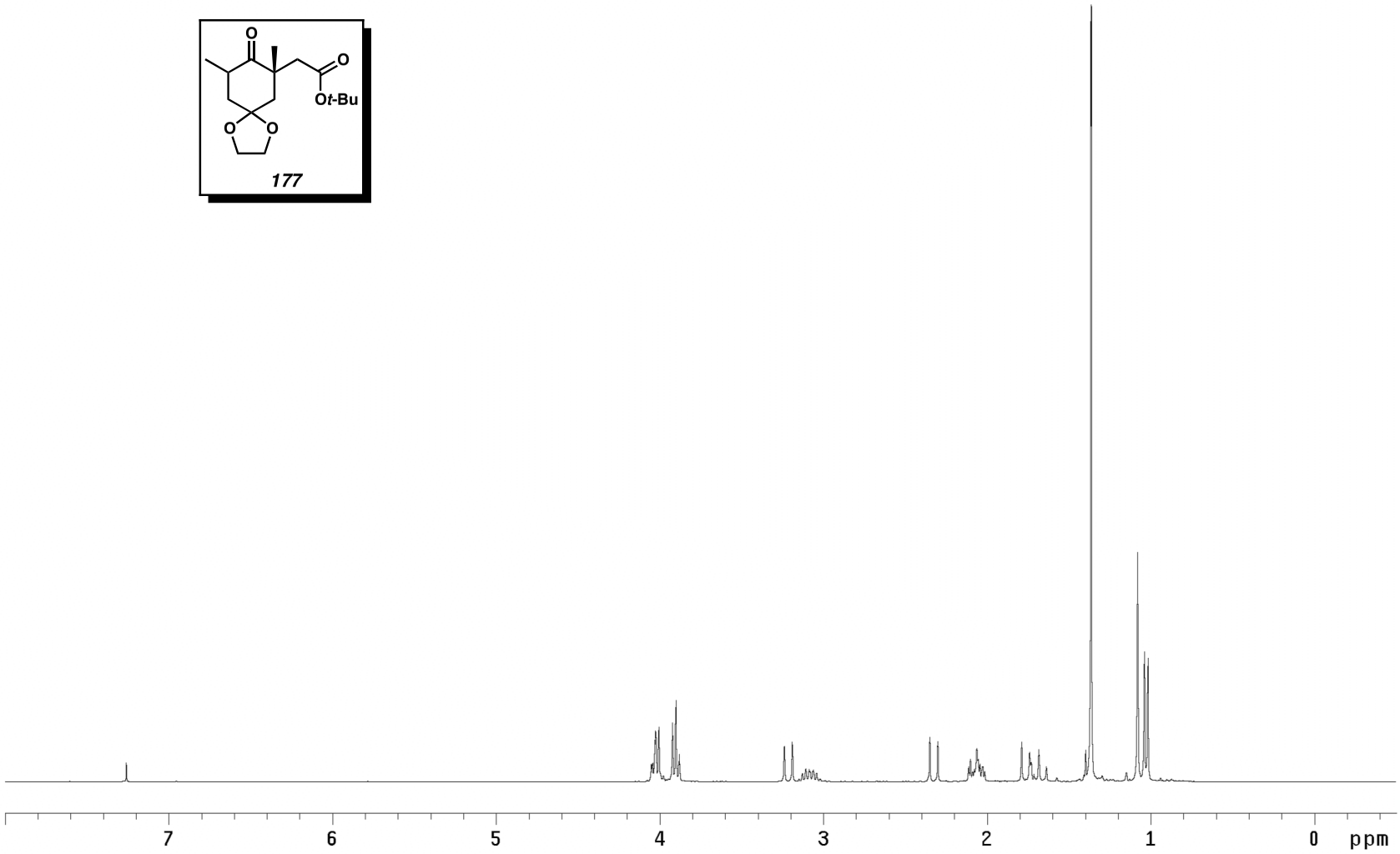

Figure A.19 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (–)-**177**.

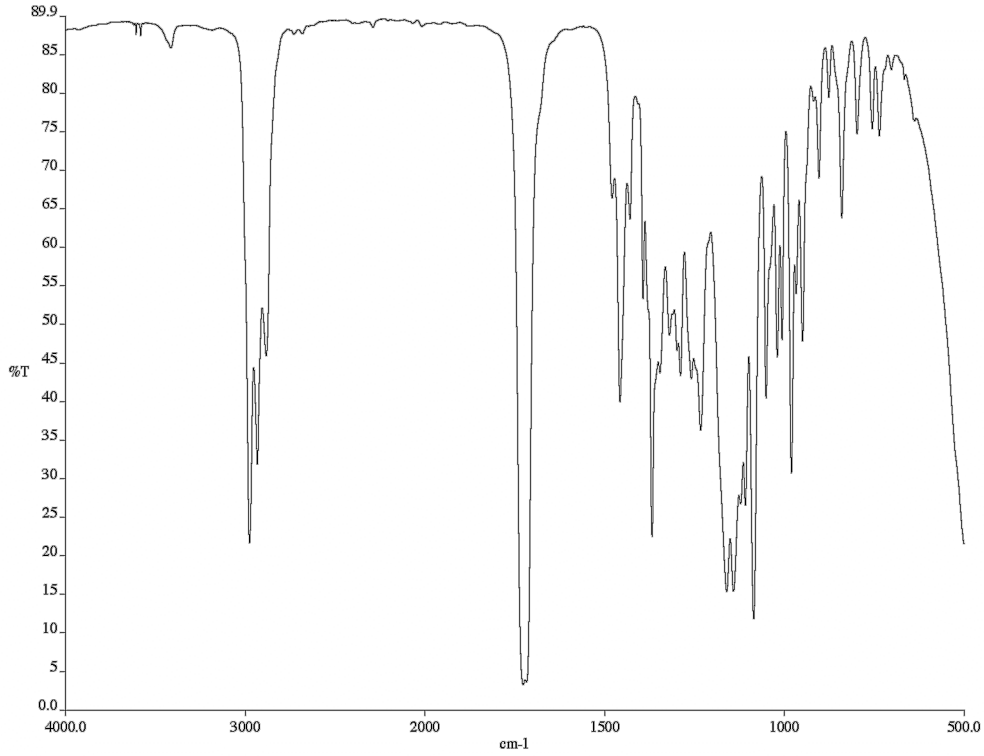

Figure A.20 Infrared spectrum (thin film/NaCl) of compound (–)-**177**.

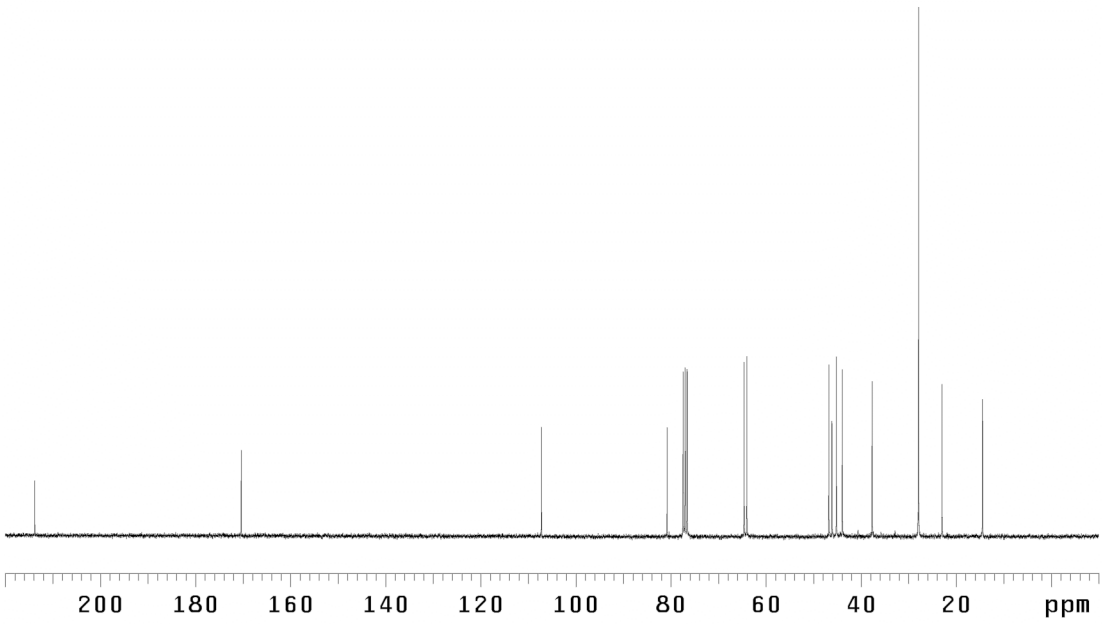

Figure A.21 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (–)-**177**.

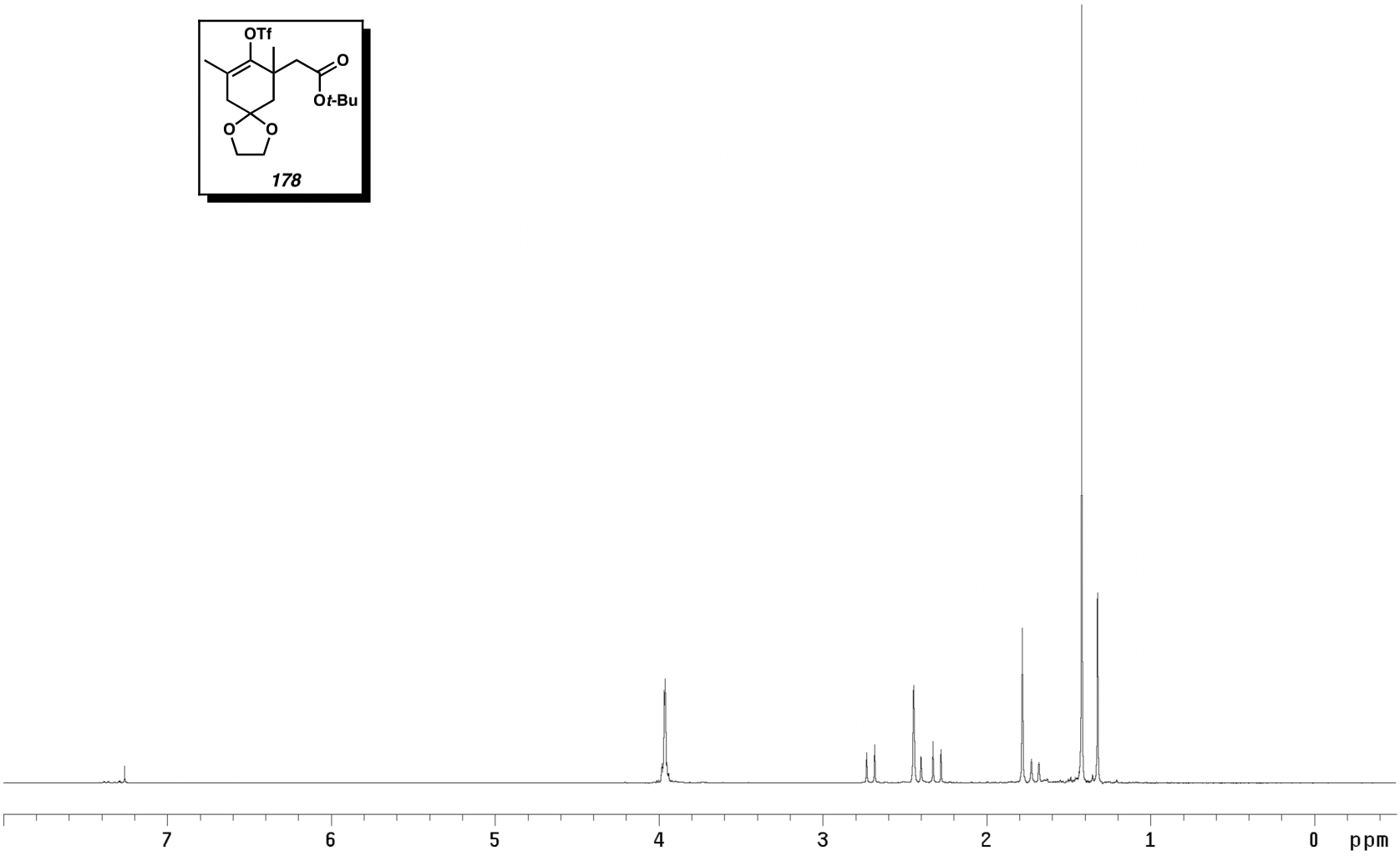

Figure A.22 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **178**.

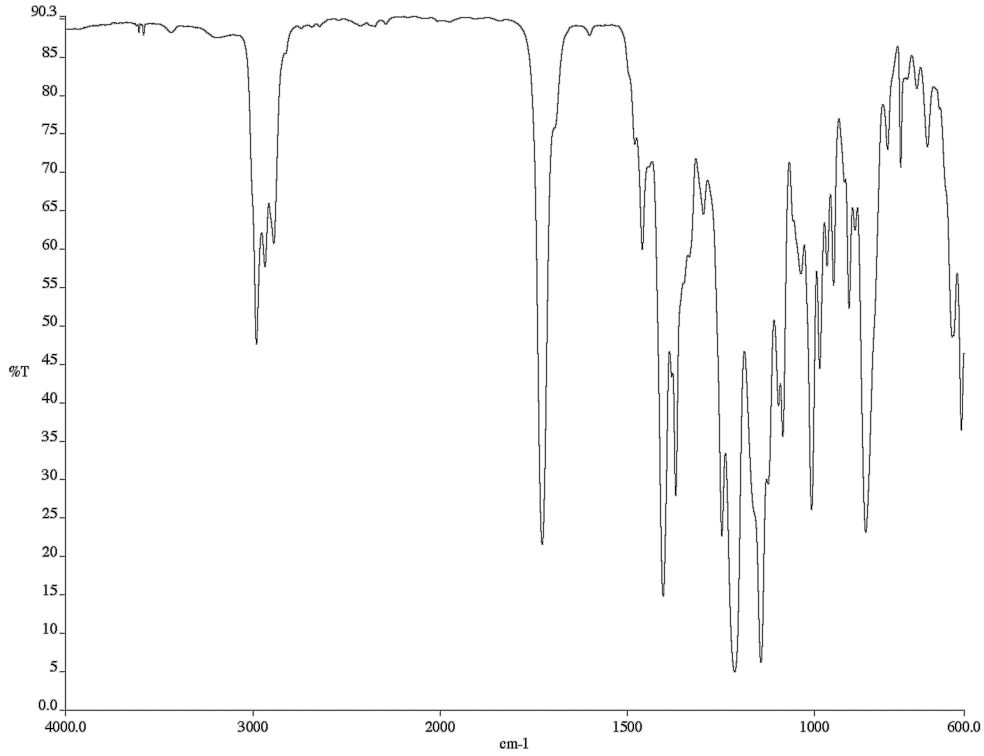

Figure A.23 Infrared spectrum (thin film/NaCl) of compound **178**.

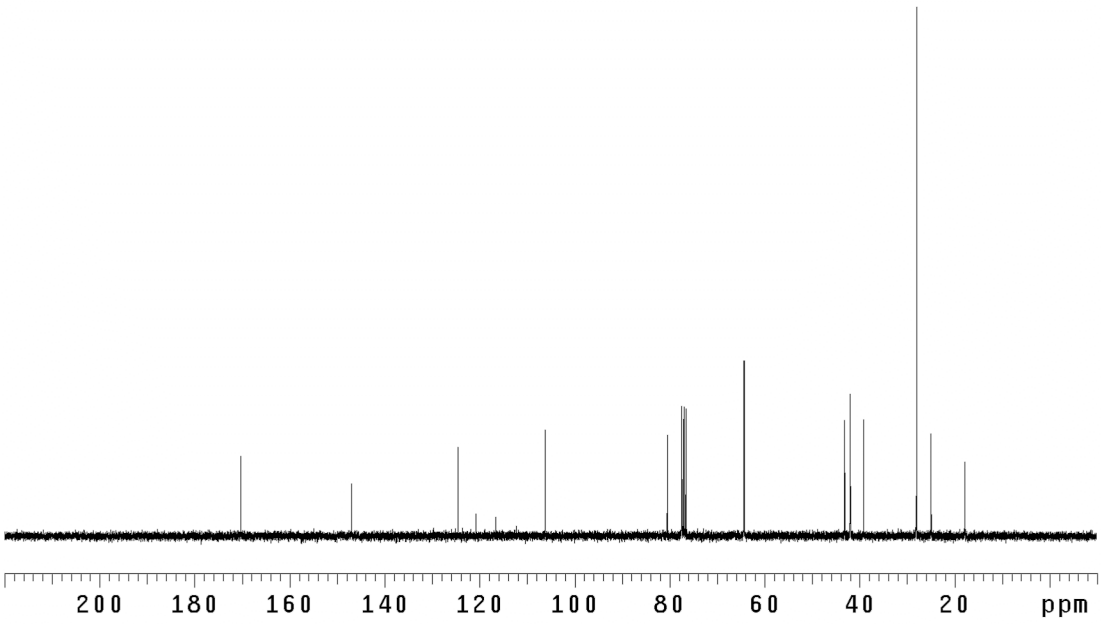

Figure A.24 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **178**.

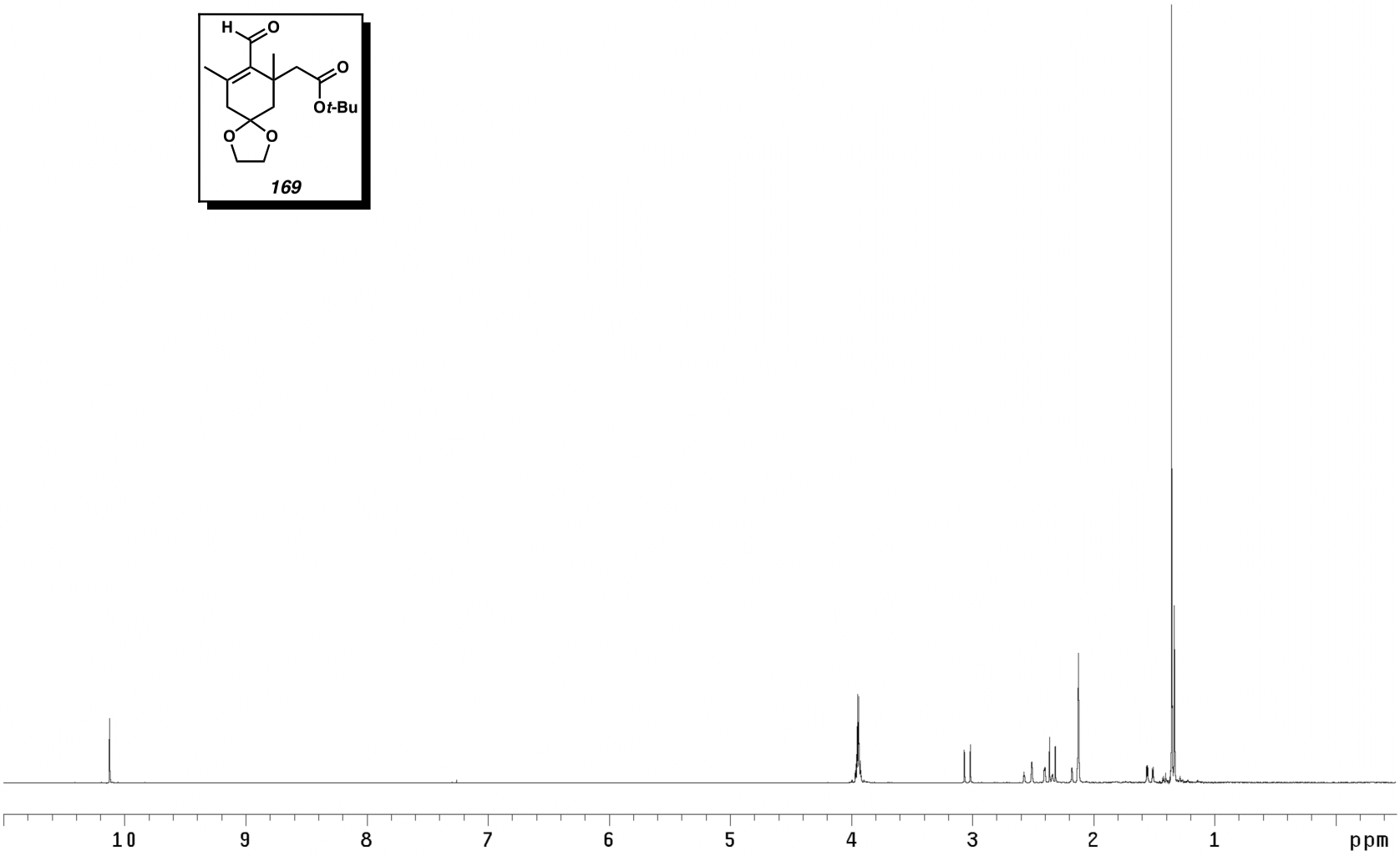

Figure A.25 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **169**.

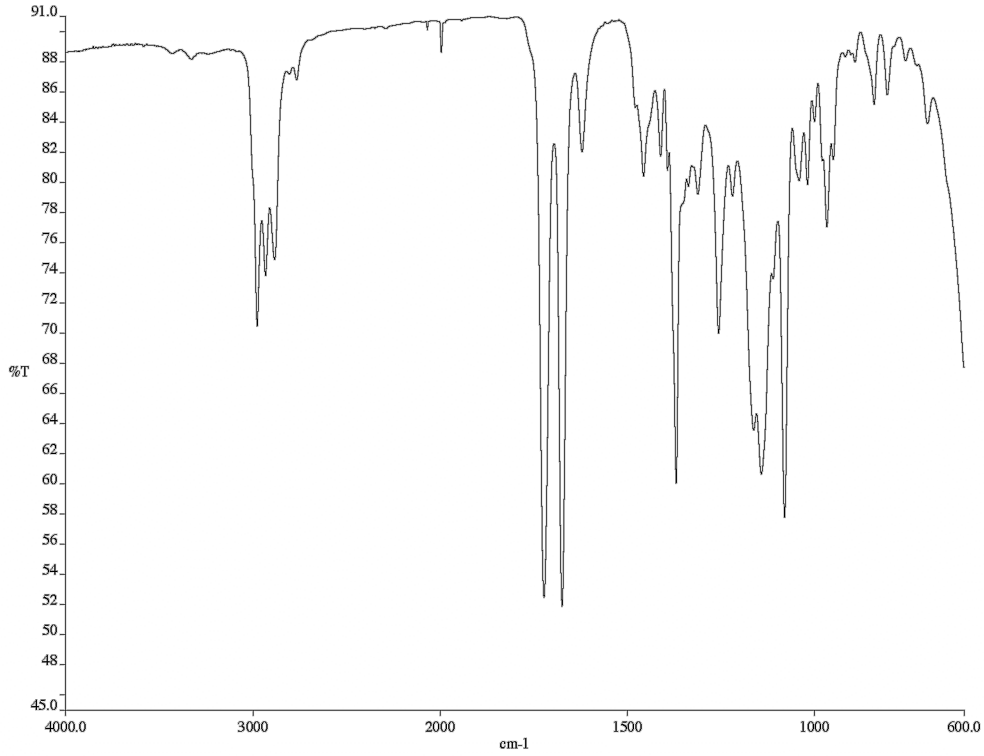

Figure A.26 Infrared spectrum (thin film/NaCl) of compound **169**.

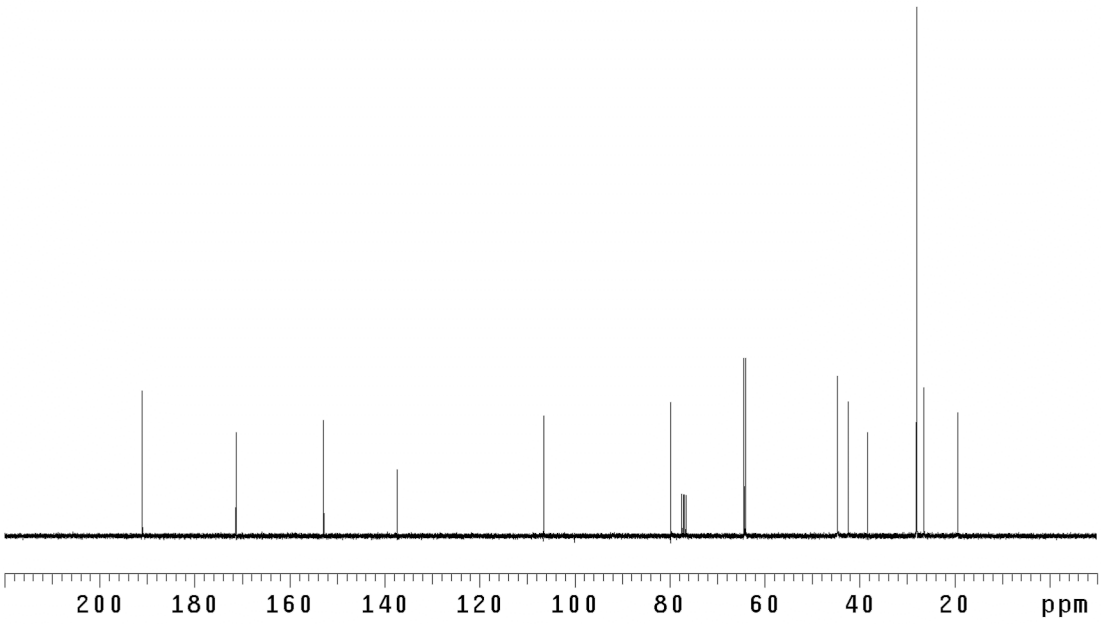

Figure A.27 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **169**.

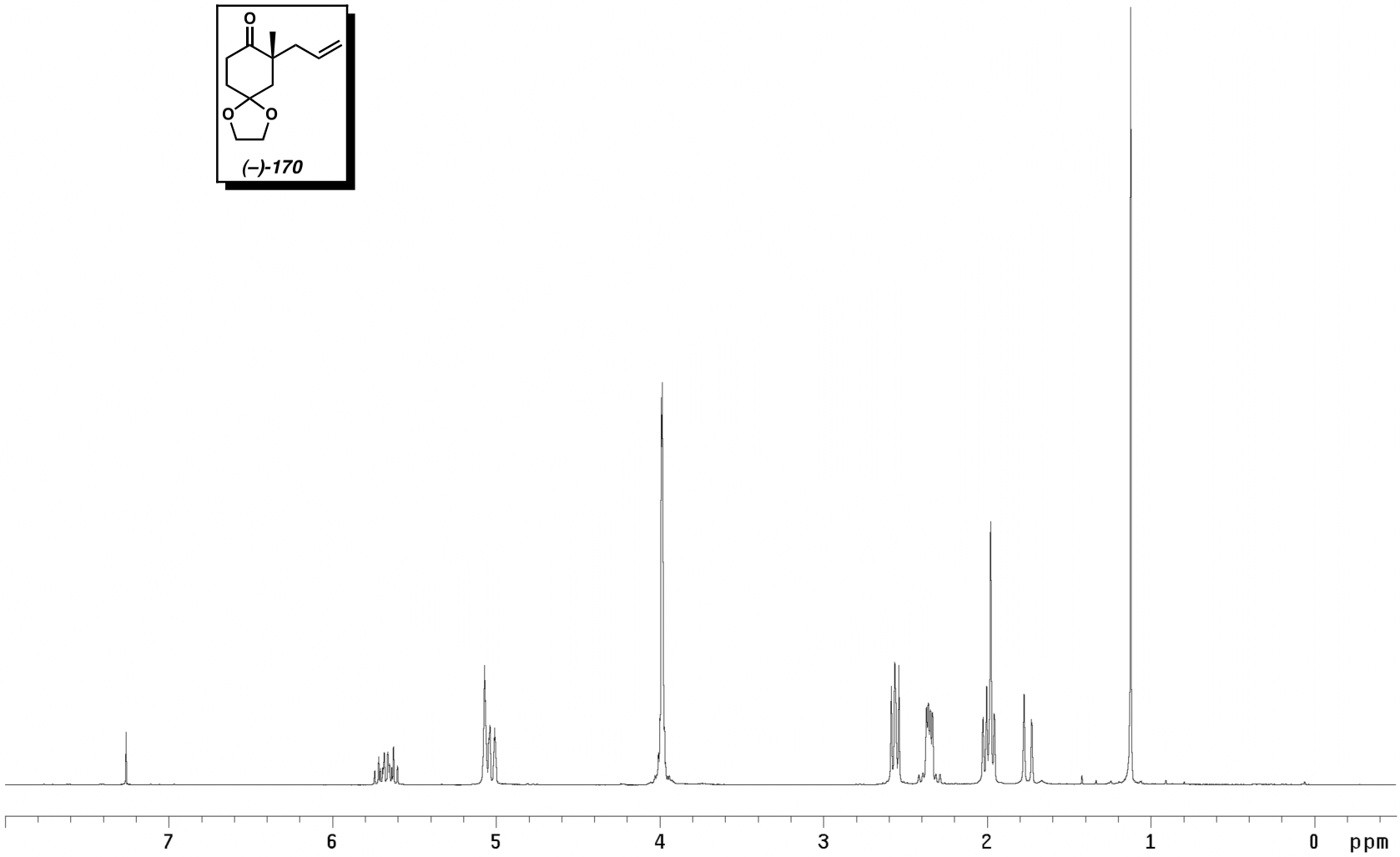

Figure A.28 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (–)-**170**.

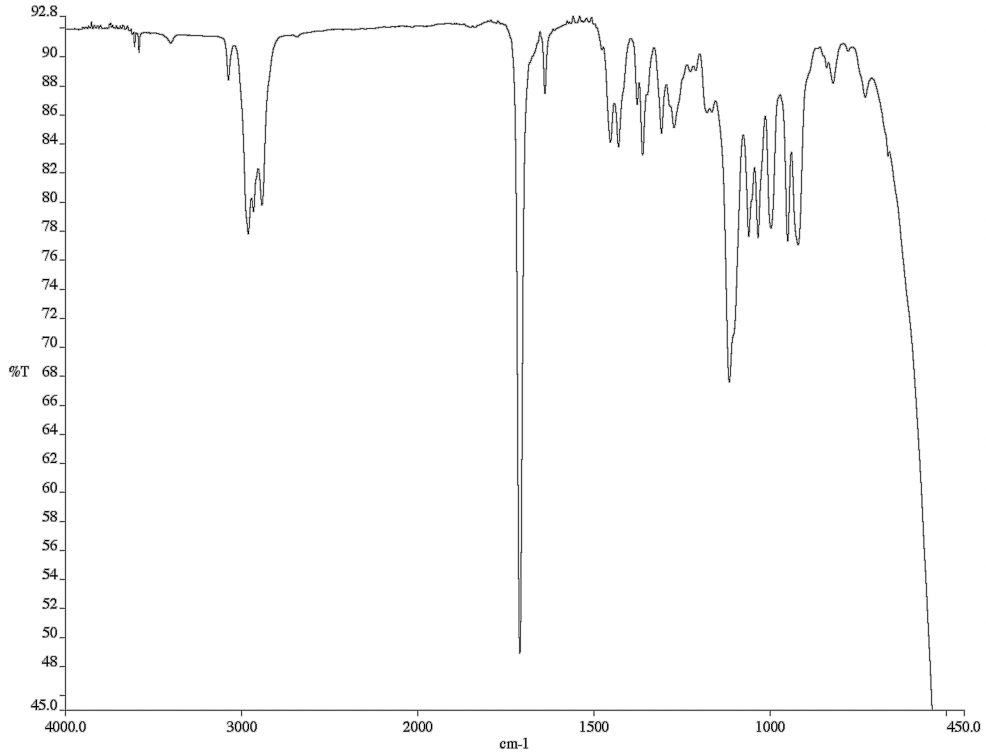

Figure A.29 Infrared spectrum (thin film/NaCl) of compound (–)-**170**.

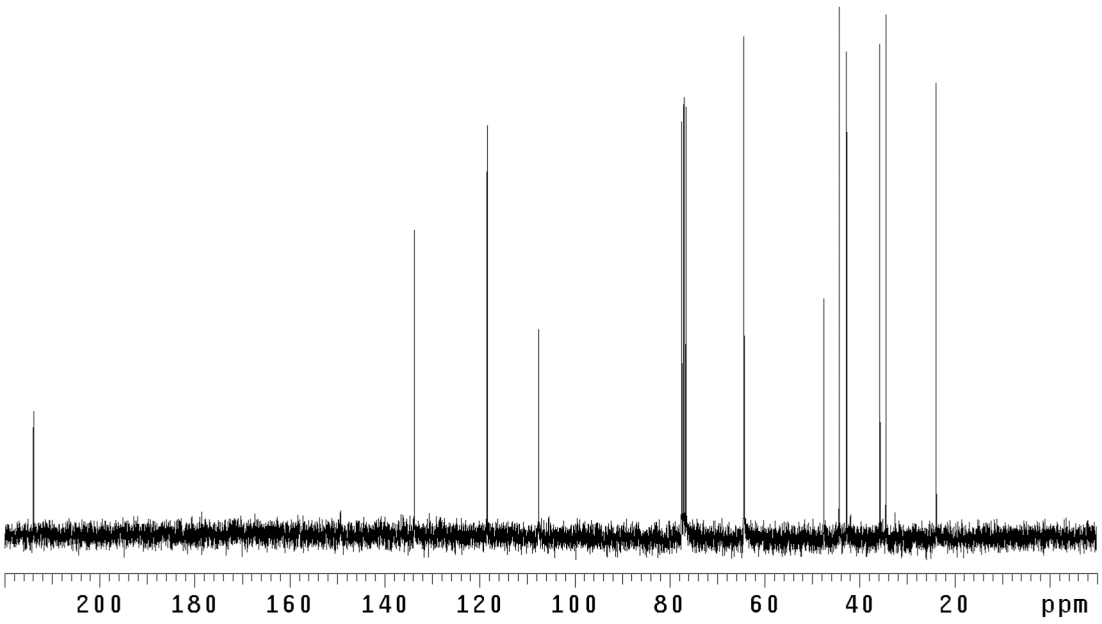

Figure A.30 ^{13}C NMR (75 MHz, XX) of compound (–)-**170**.

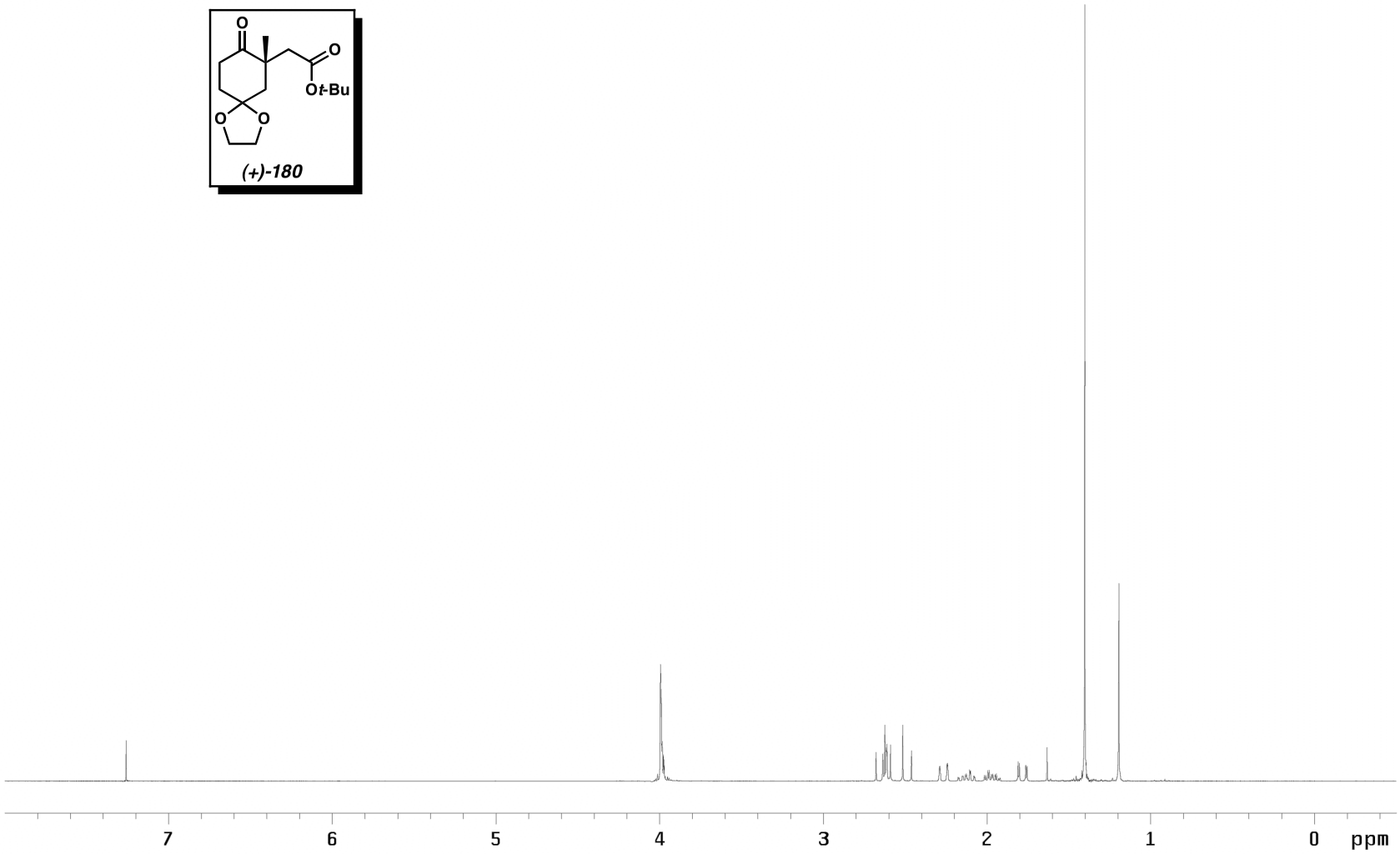

Figure A.31 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (+)-**180**.

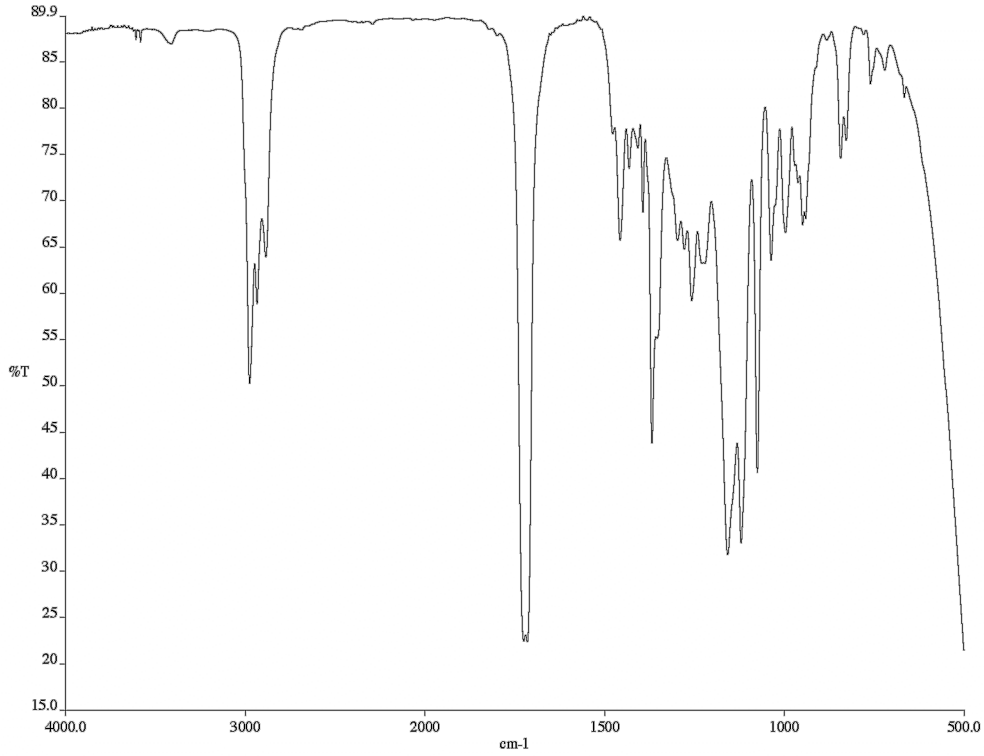

Figure A.32 Infrared spectrum (thin film/NaCl) of compound (+)-**180**.

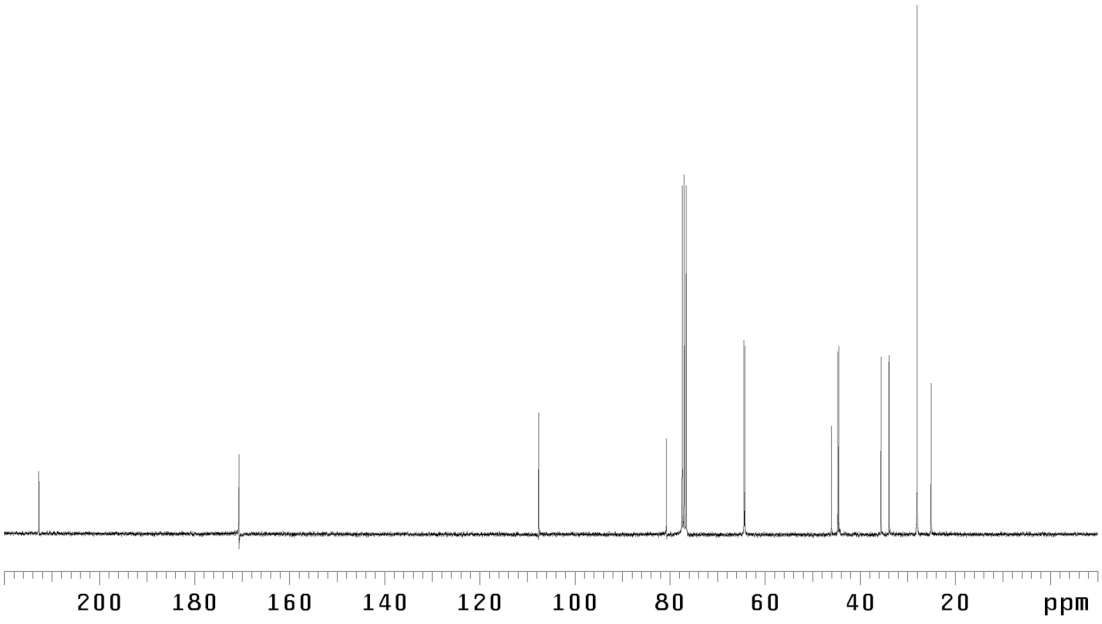

Figure A.33 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (+)-**180**.

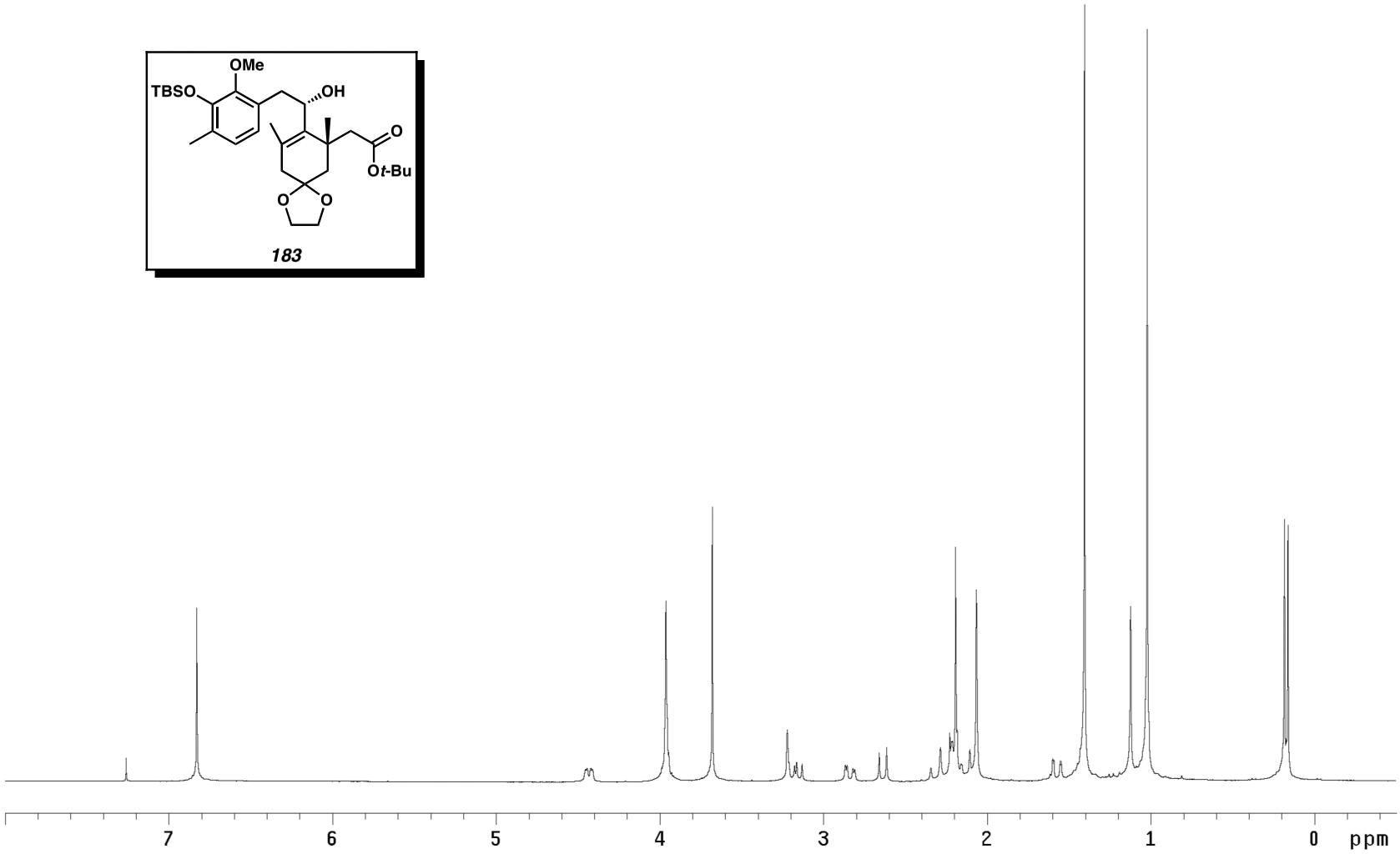

Figure A.34 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **183**.

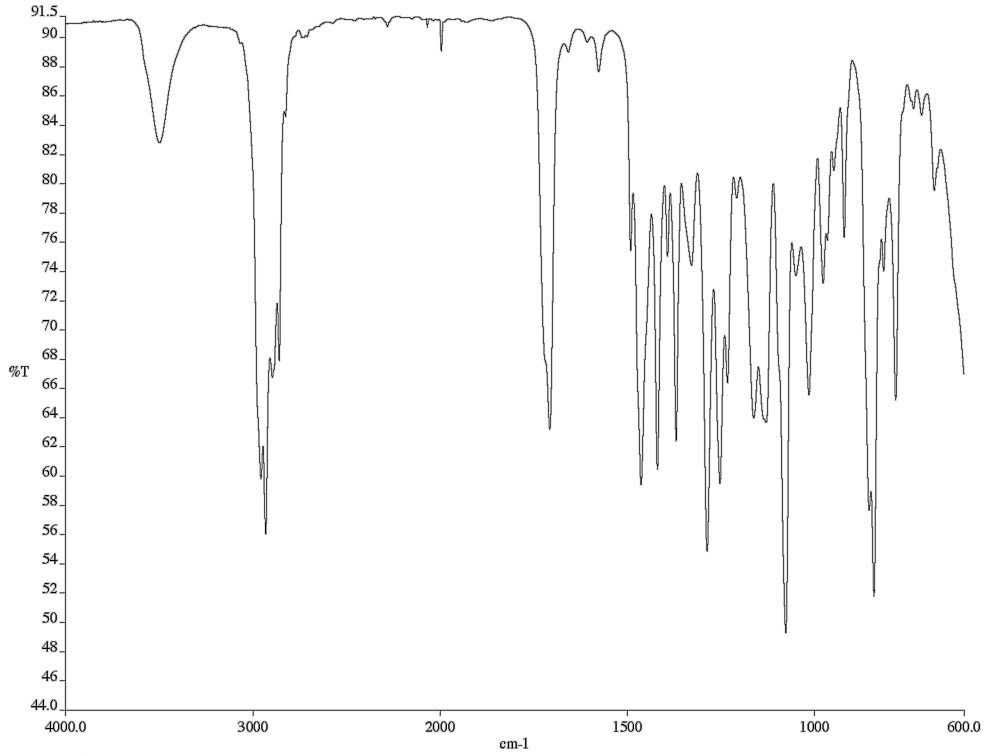

Figure A.35 Infrared spectrum (thin film/NaCl) of compound **183**.

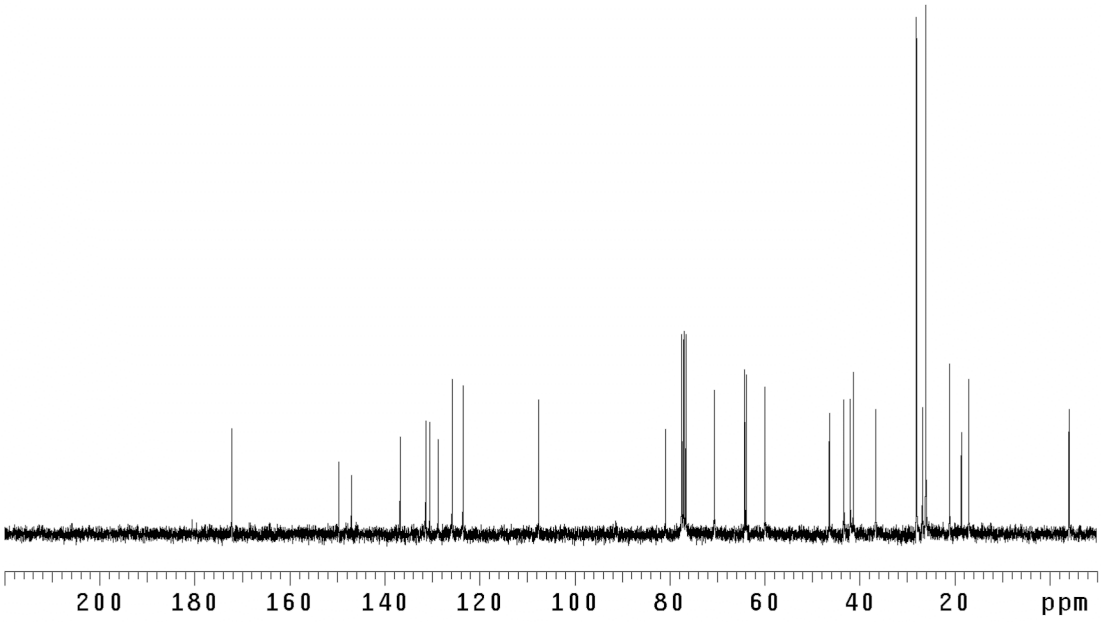

Figure A.36 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **183**.

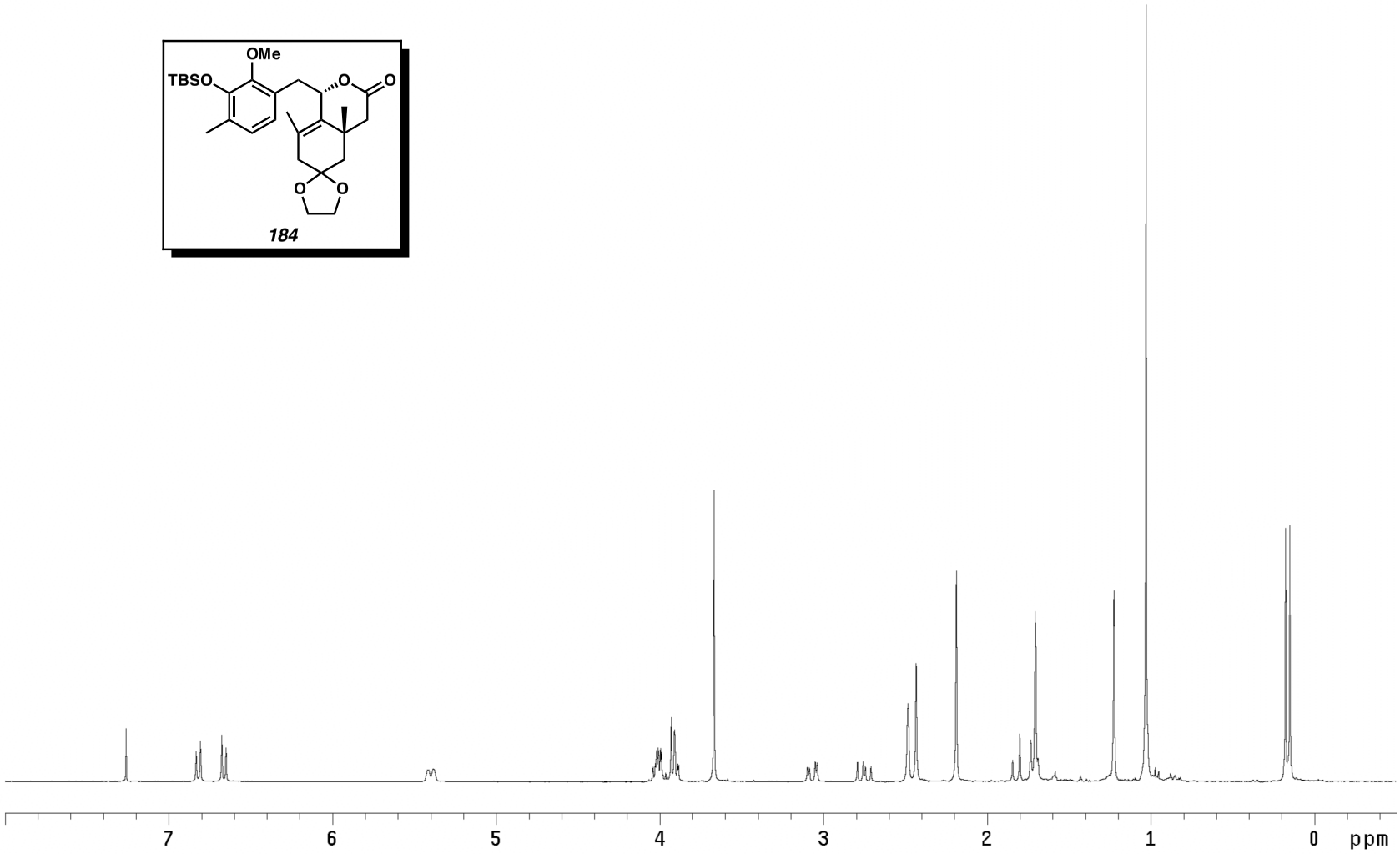

Figure A.37 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **184**.

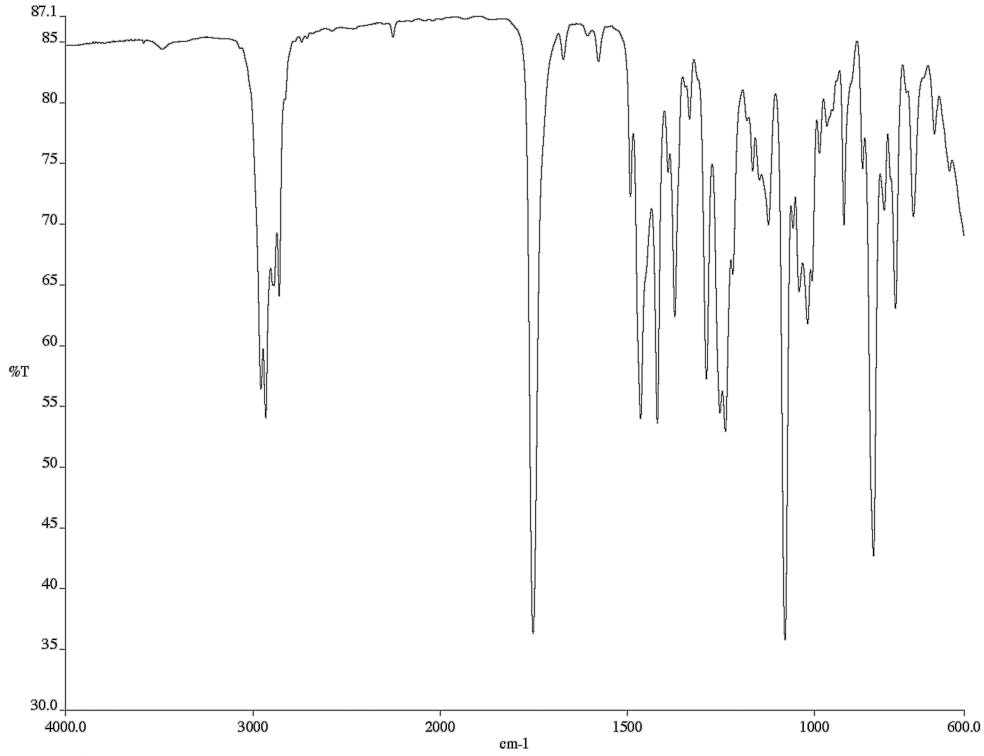

Figure A.38 Infrared spectrum (thin film/NaCl) of compound **184**.

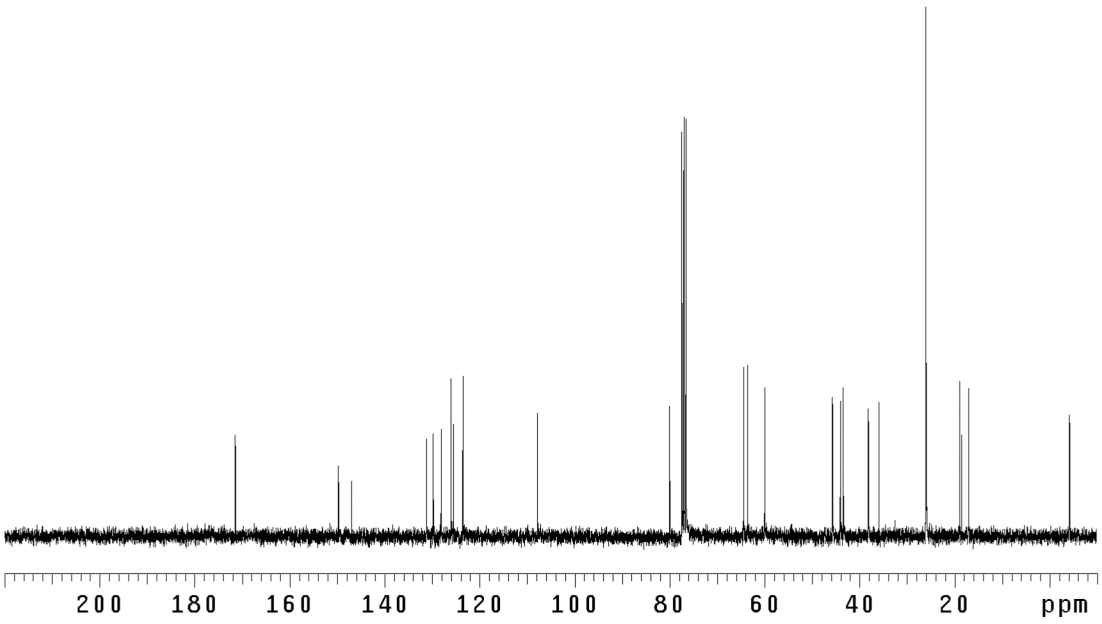

Figure A.39 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **184**.

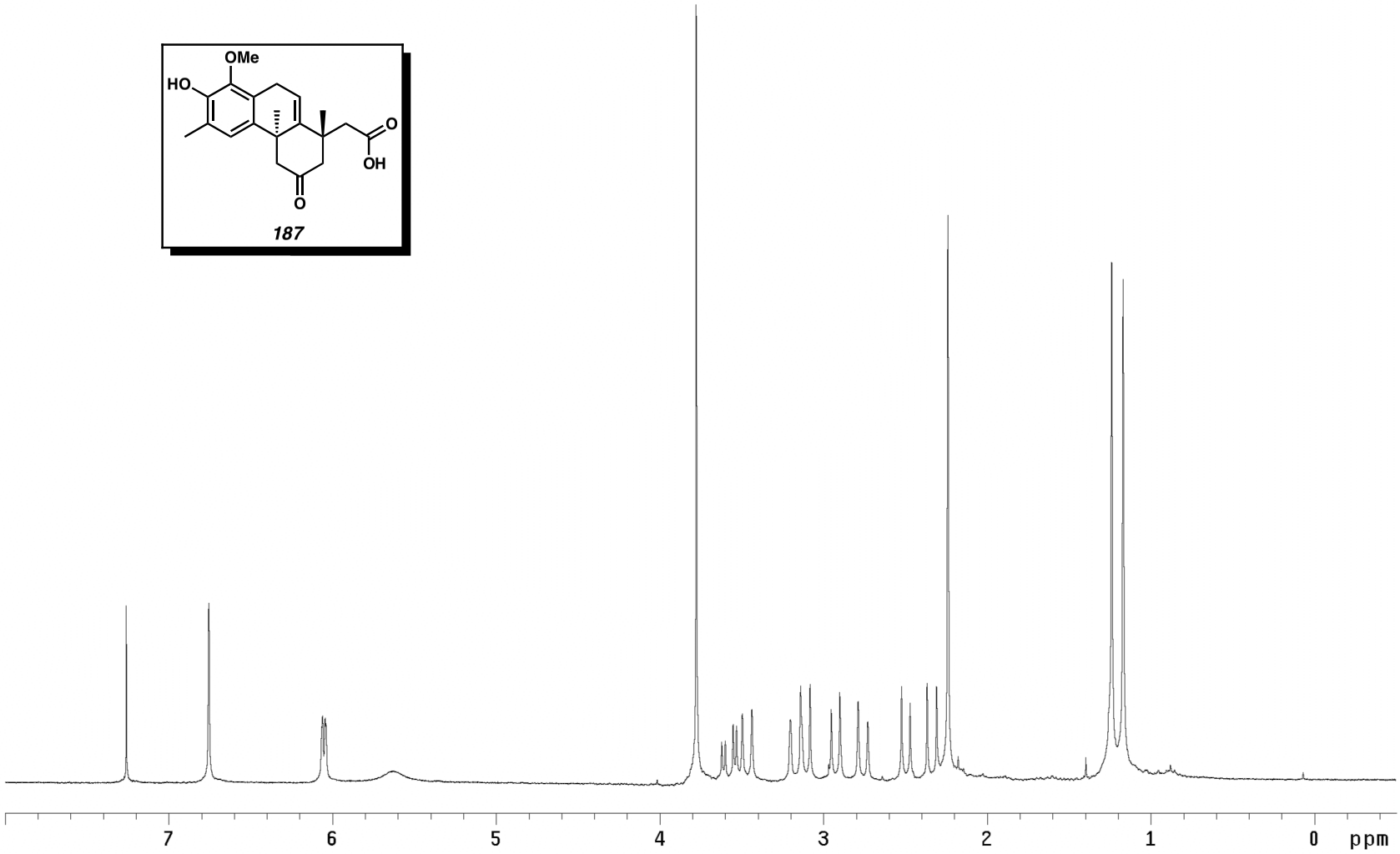

Figure A.40 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **187**.

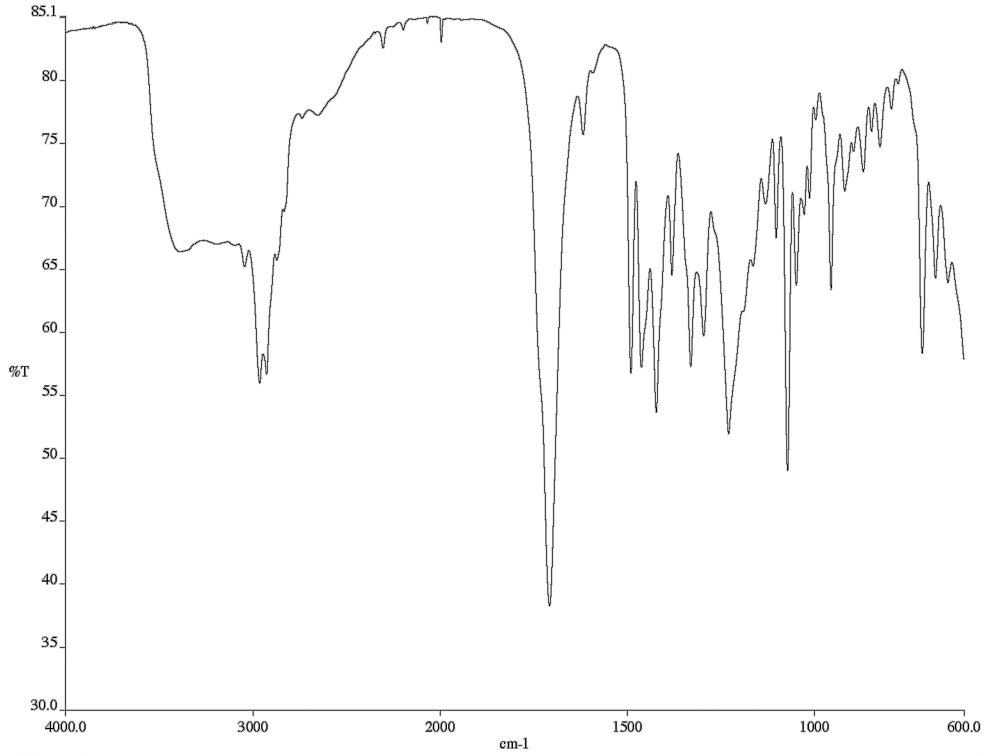

Figure A.41 Infrared spectrum (thin film/NaCl) of compound **187**.

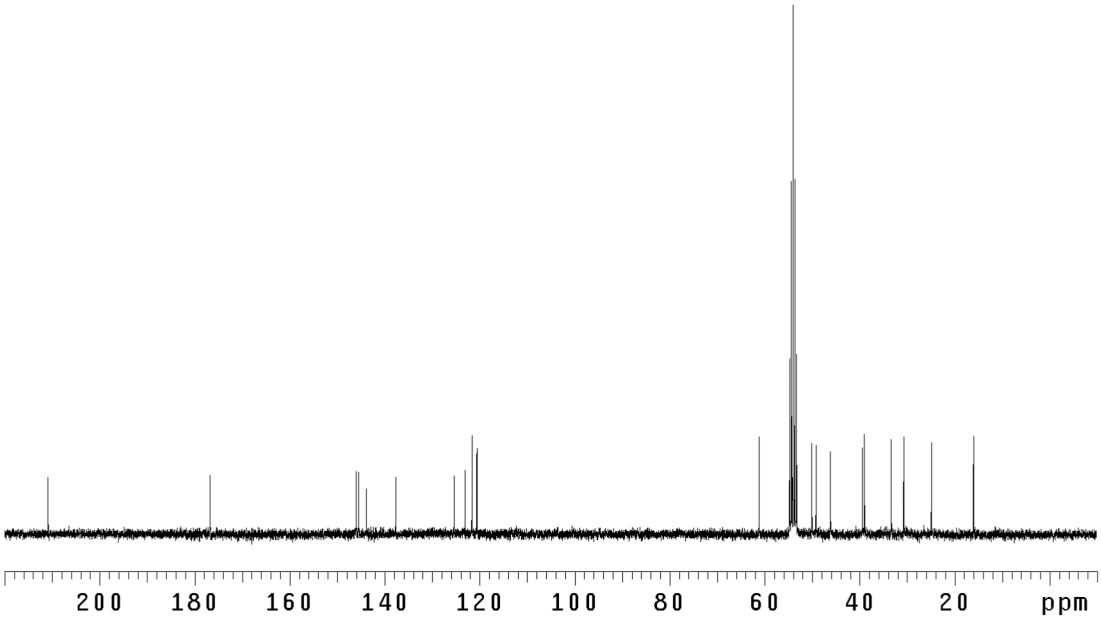

Figure A.42 ^{13}C NMR (75 MHz, CD_2Cl_2) of compound **187**.

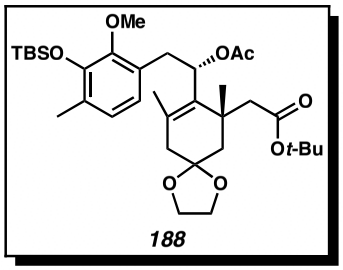

Figure A.43 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **188**.

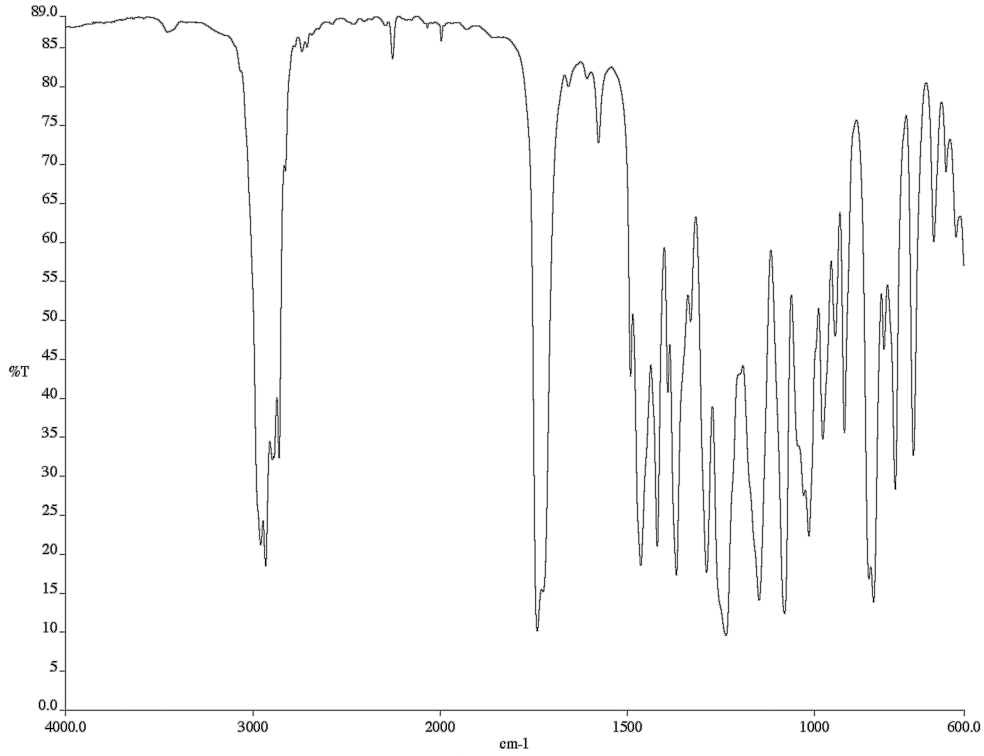

Figure A.44 Infrared spectrum (thin film/NaCl) of compound **188**.

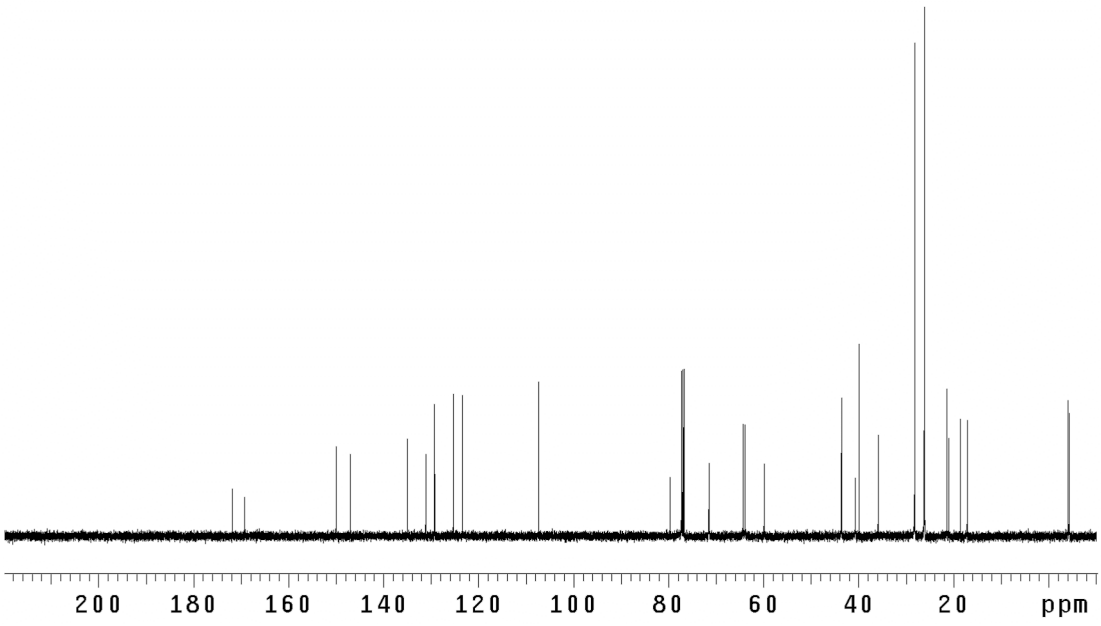

Figure A.45 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **188**.

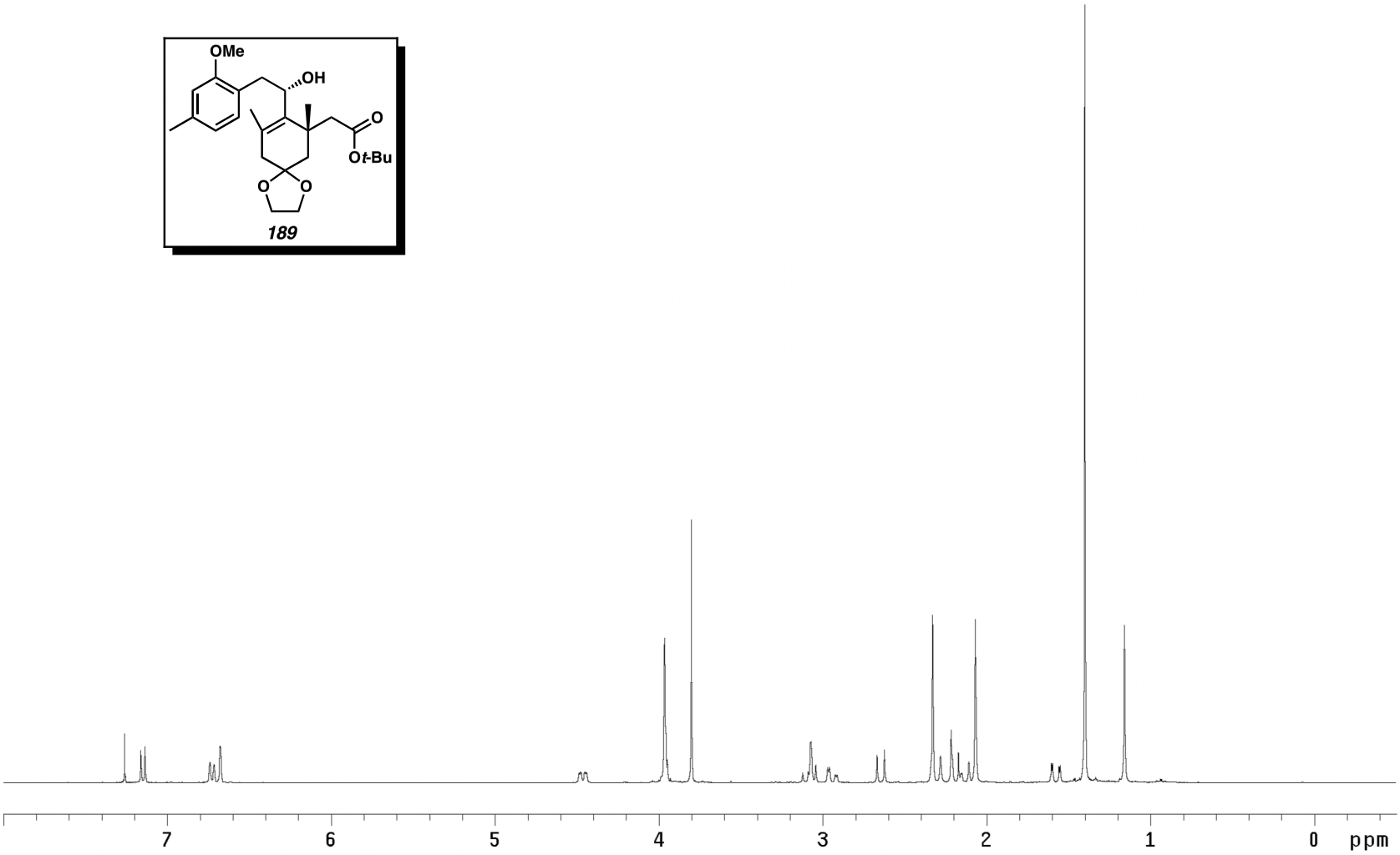

Figure A.46 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **189**.

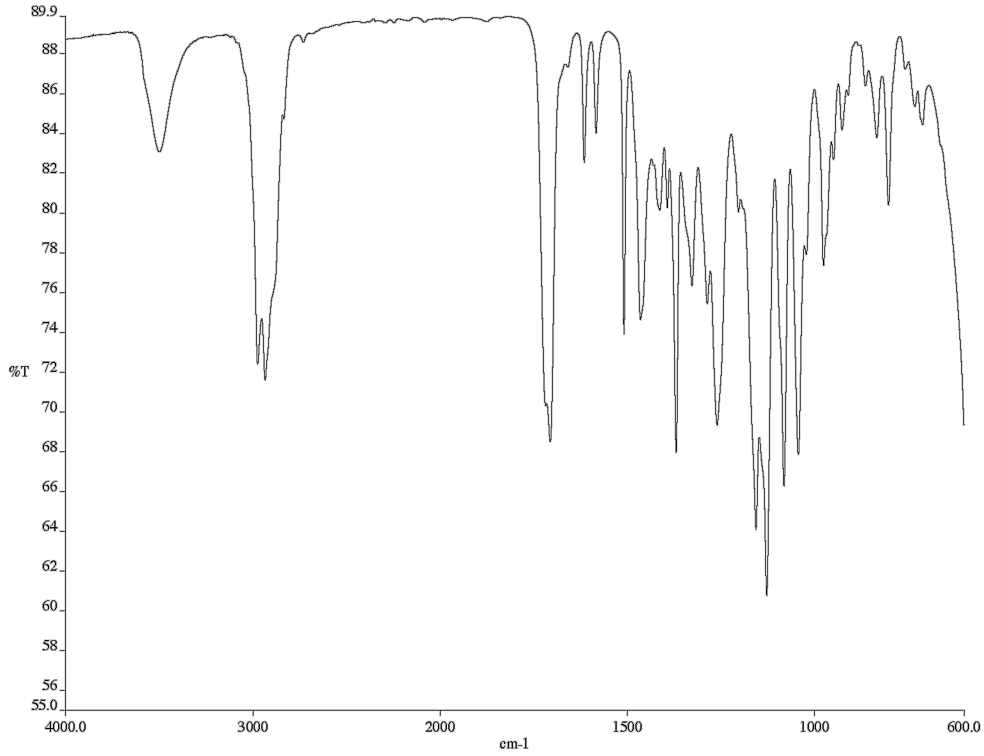

Figure A.47 Infrared spectrum (thin film/NaCl) of compound **189**.

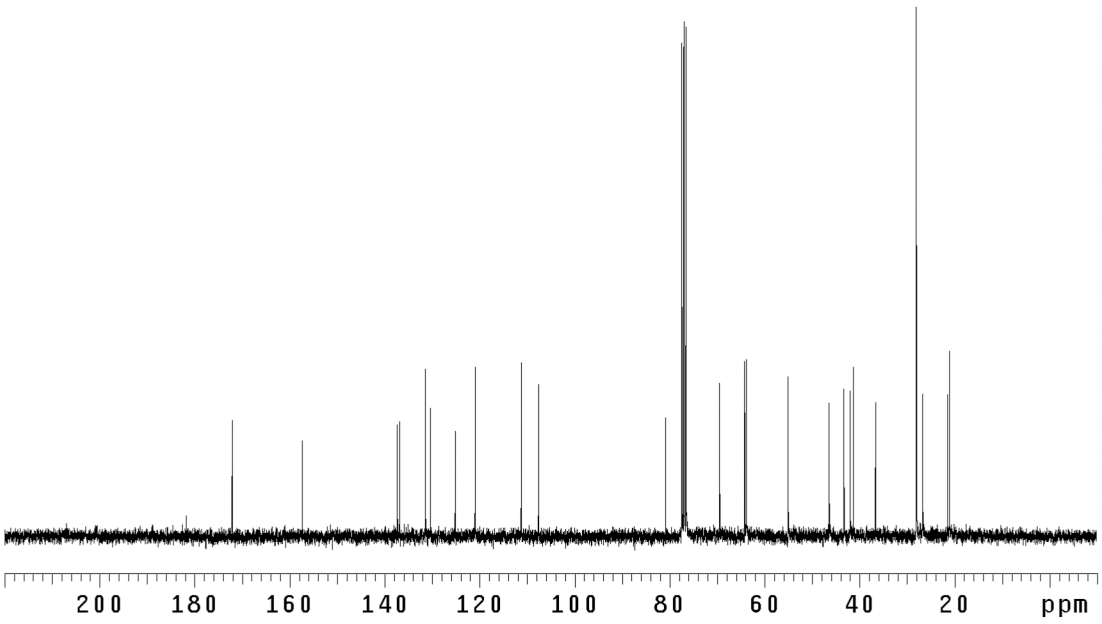

Figure A.48 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **189**.

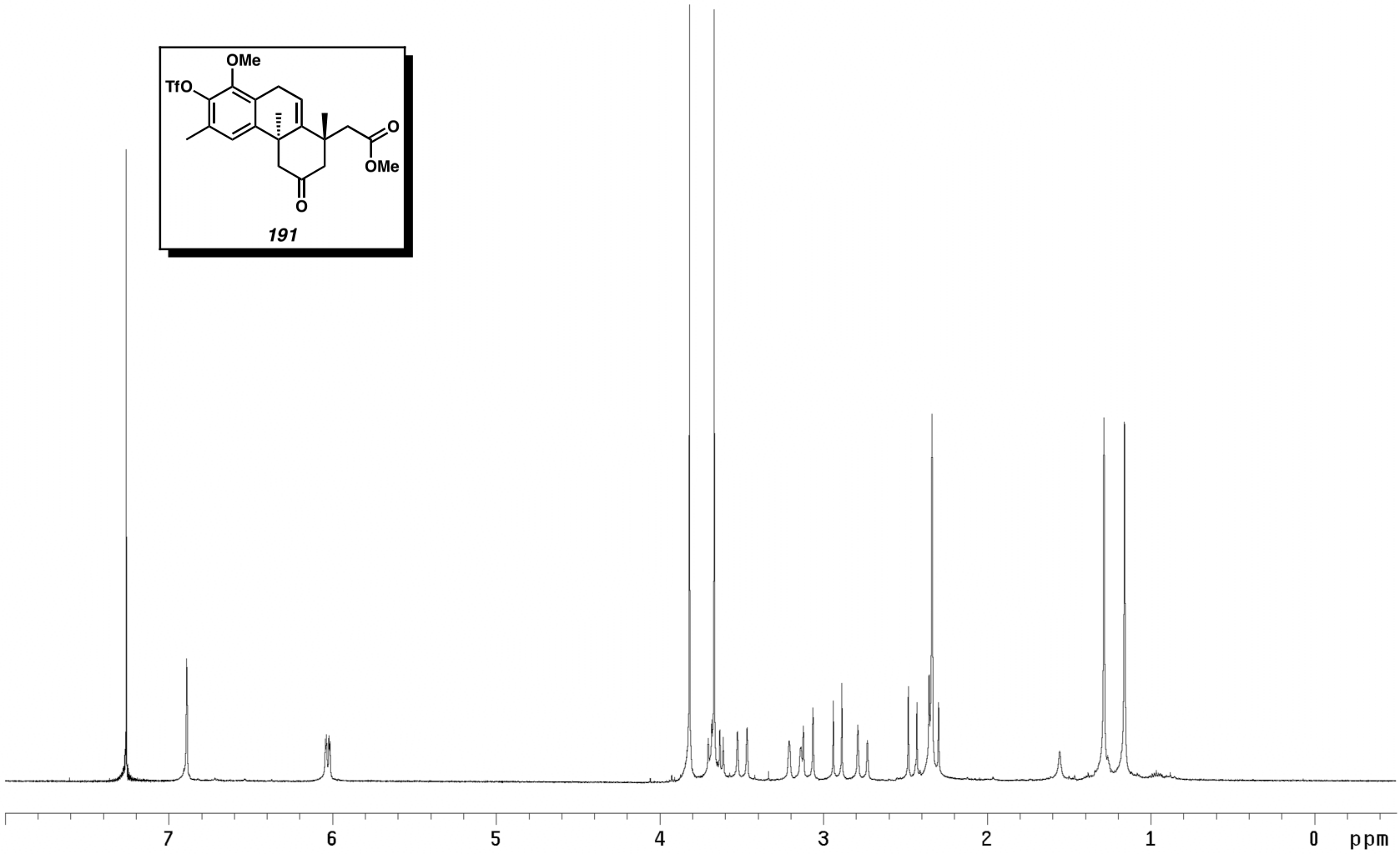

Figure A.49 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **191**.

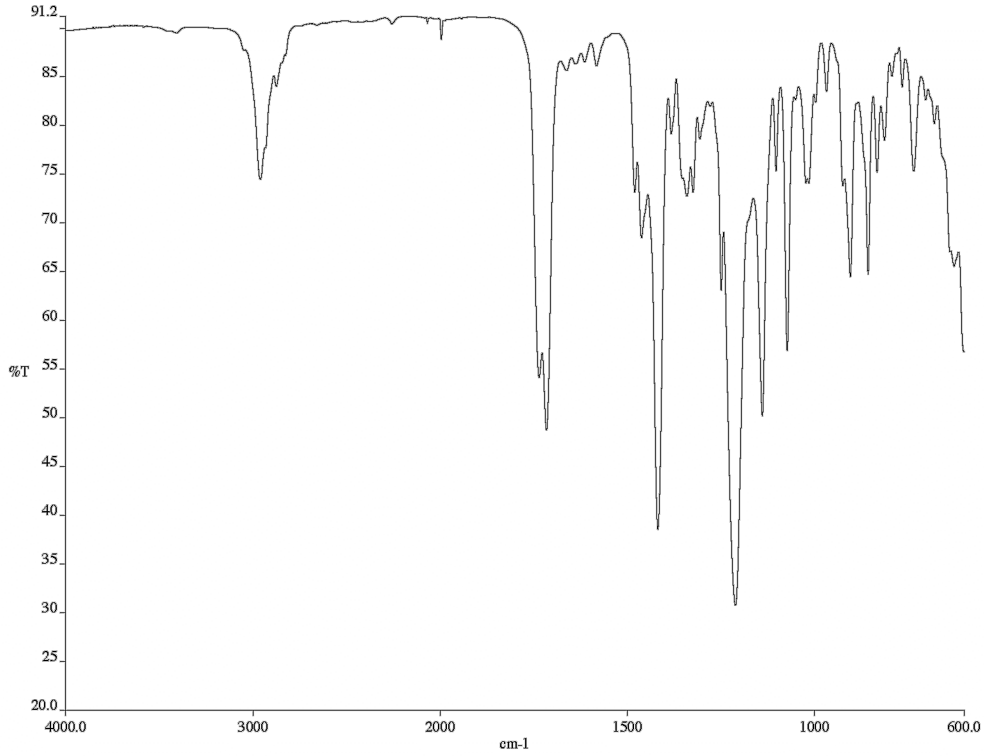

Figure A.50 Infrared spectrum (thin film/NaCl) of compound **191**.

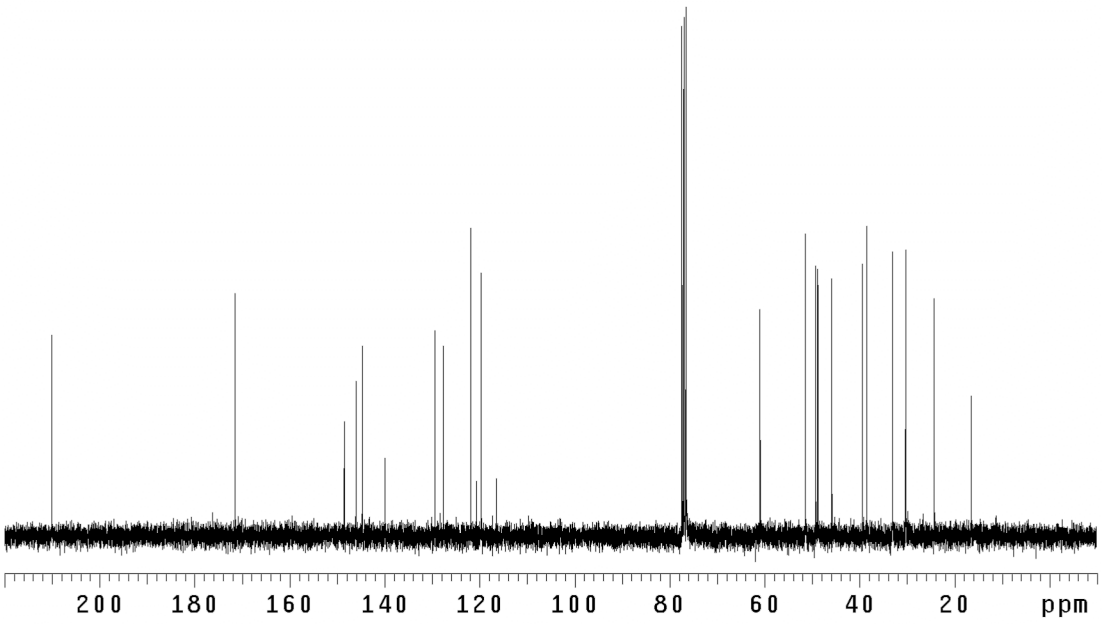

Figure A.51 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **191**.

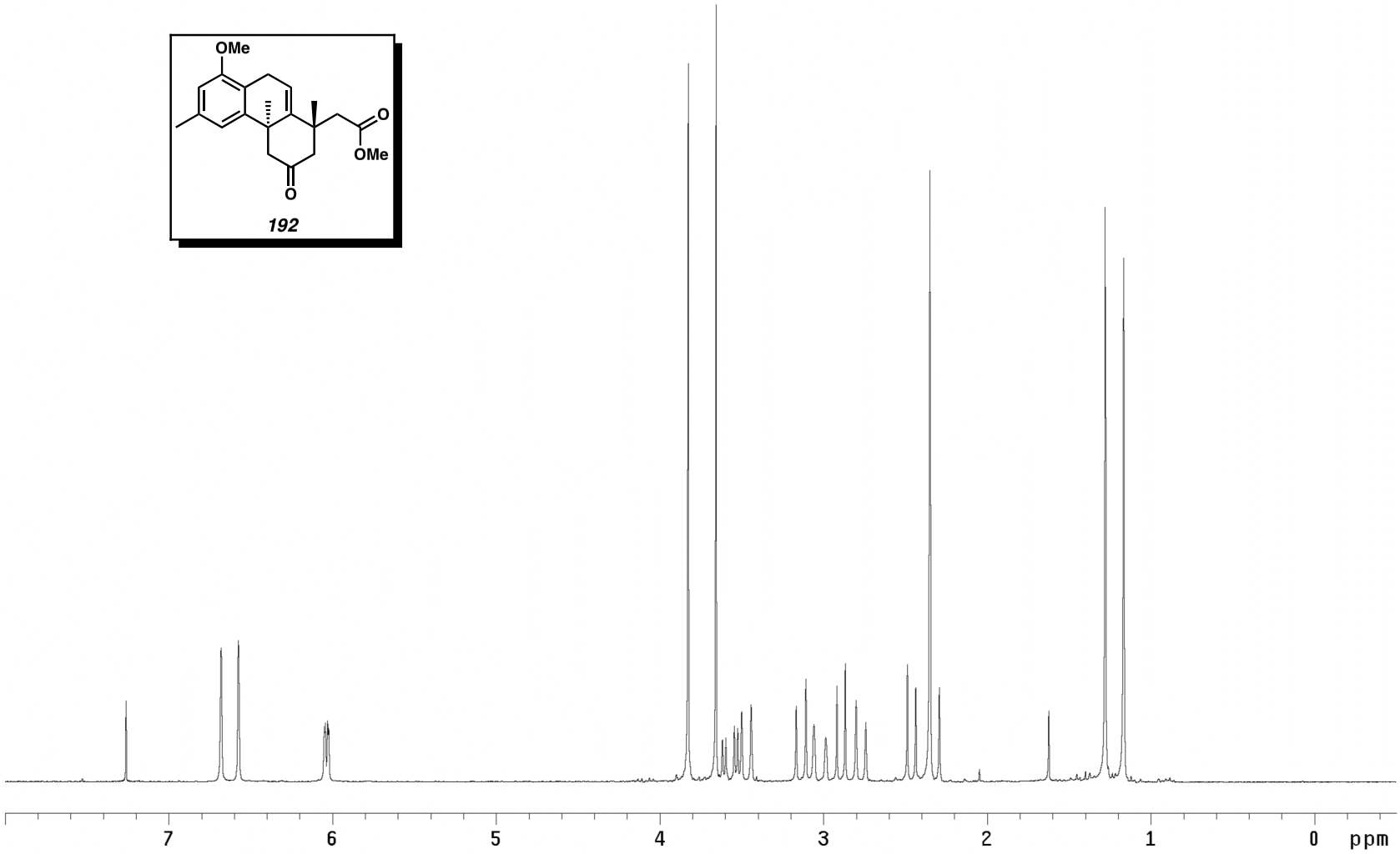

Figure A.52 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **192**.

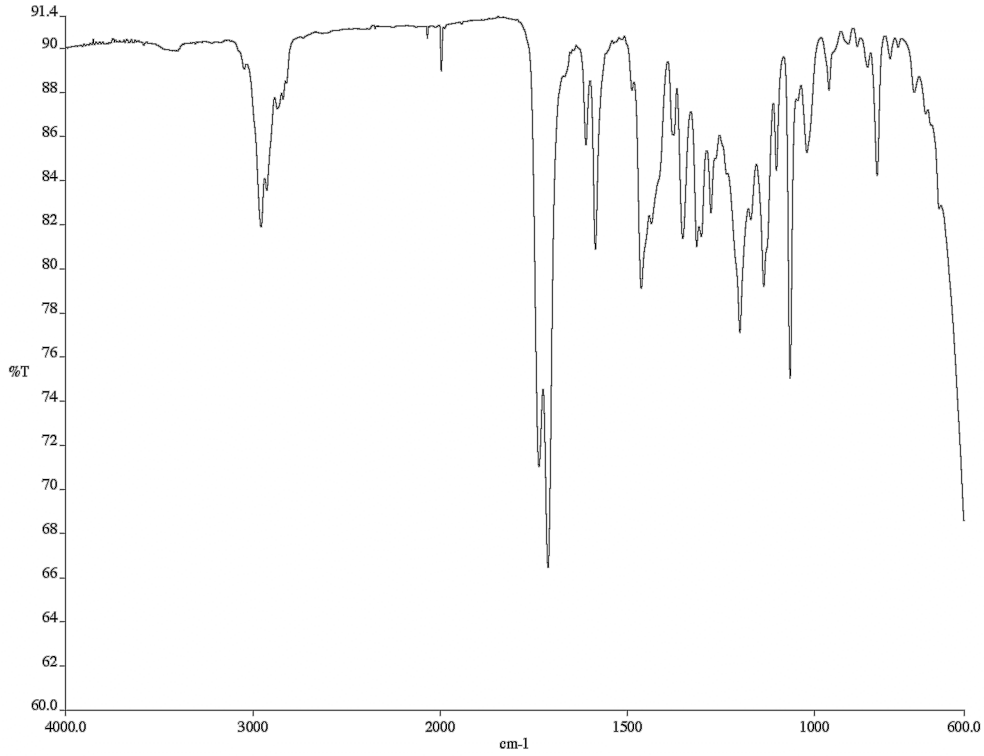

Figure A.53 Infrared spectrum (thin film/NaCl) of compound **192**.

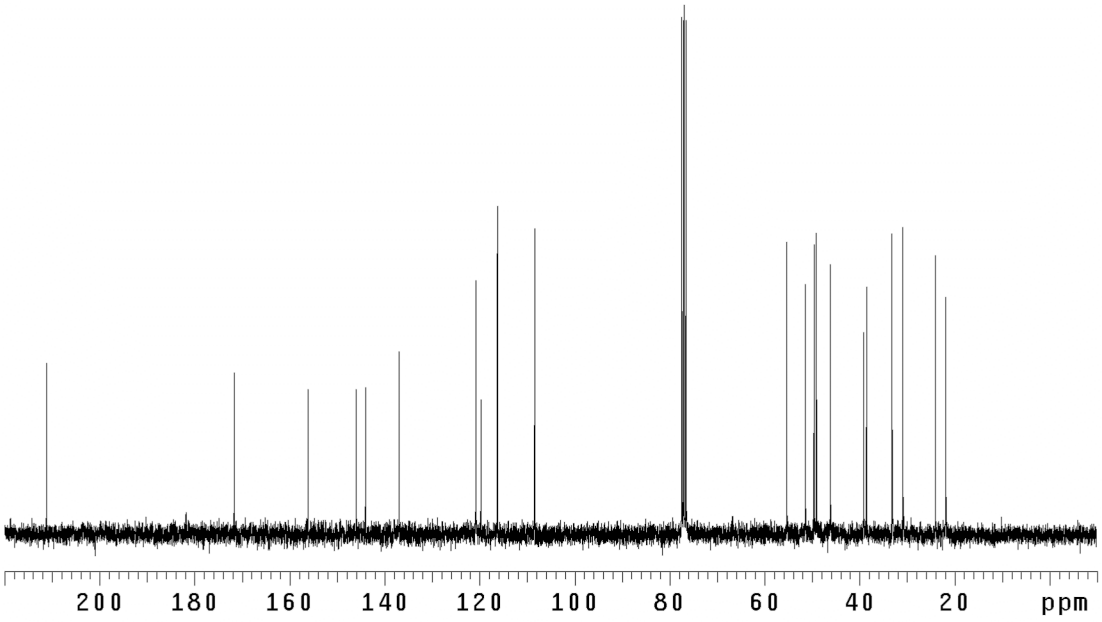

Figure A.54 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **192**.

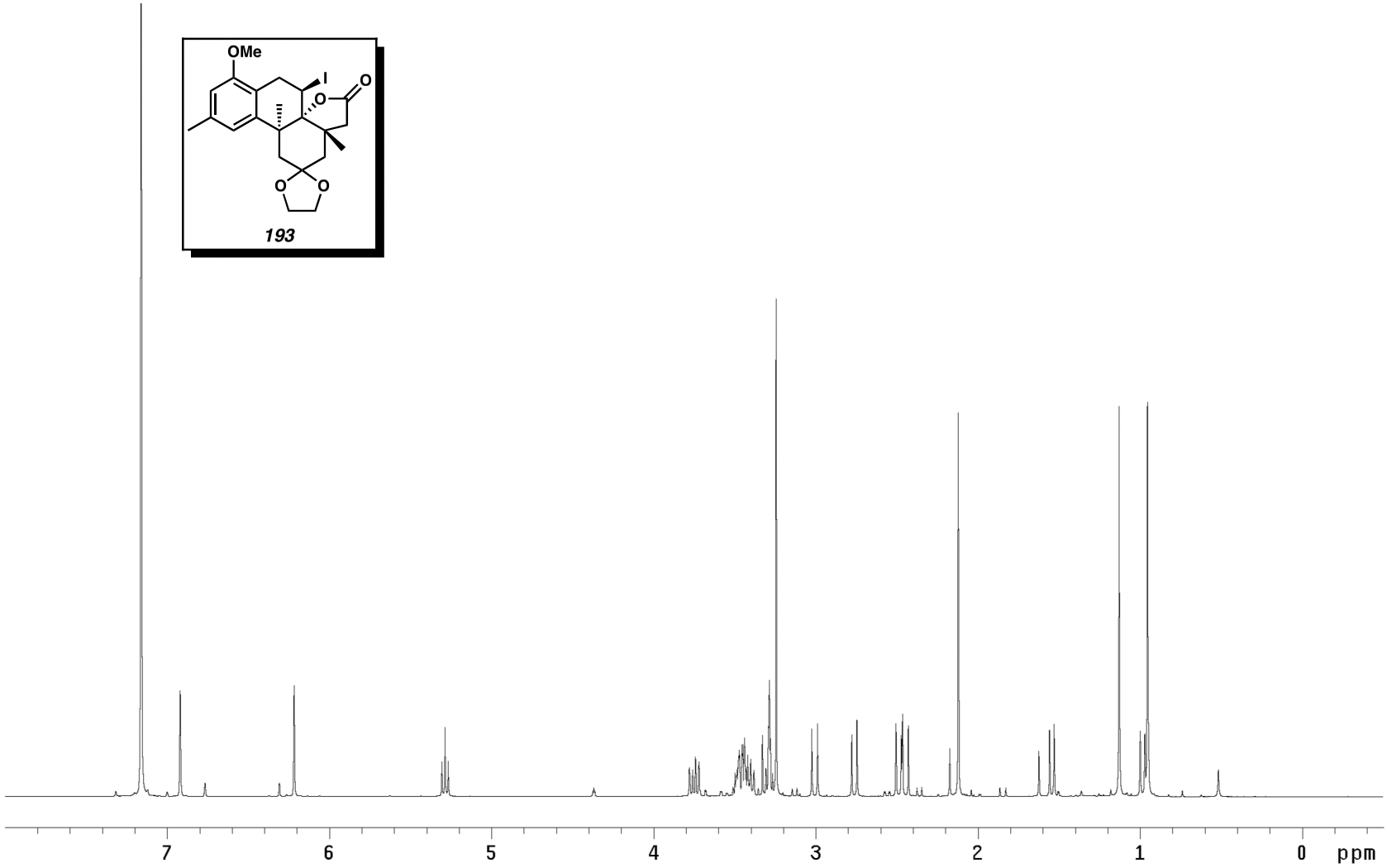

Figure A.55 ^{1}H NMR (500 MHz, C_6D_6) of compound **193**.

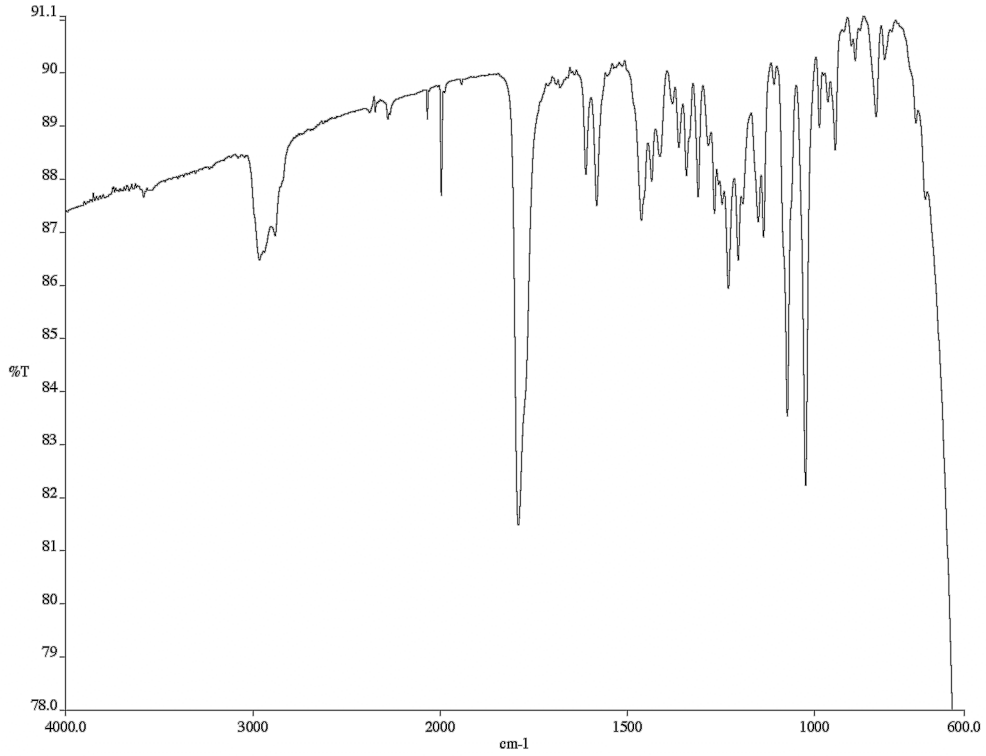

Figure A.56 Infrared spectrum (thin film/NaCl) of compound **193**.

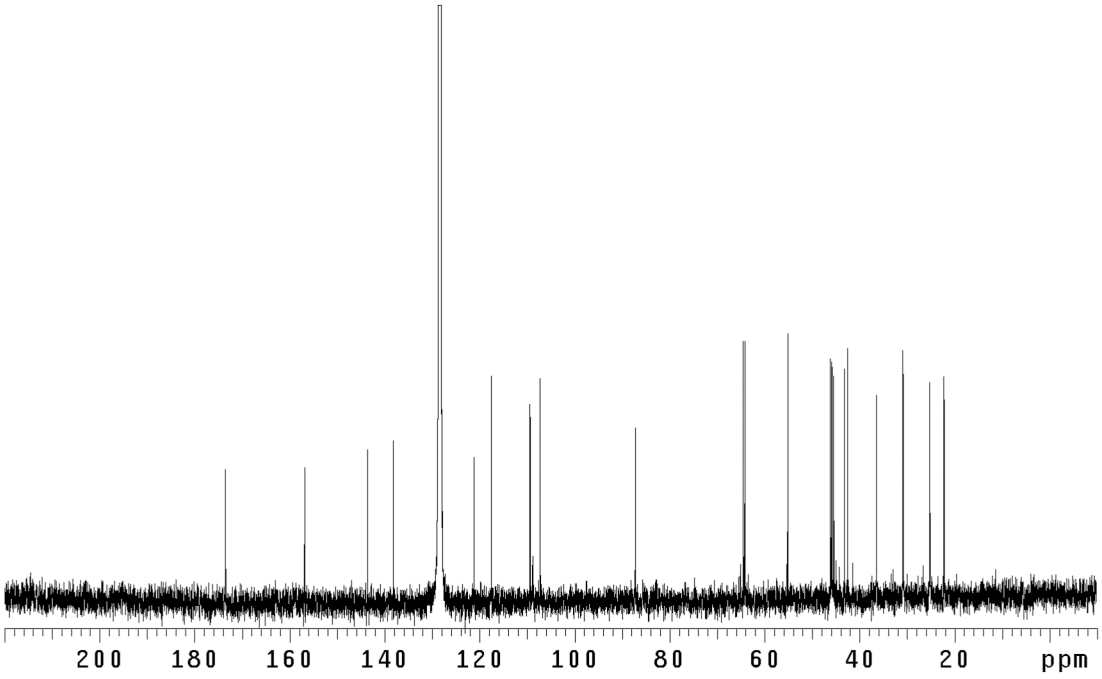

Figure A.57 ^{13}C NMR (125 MHz, C_6D_6) of compound **193**.

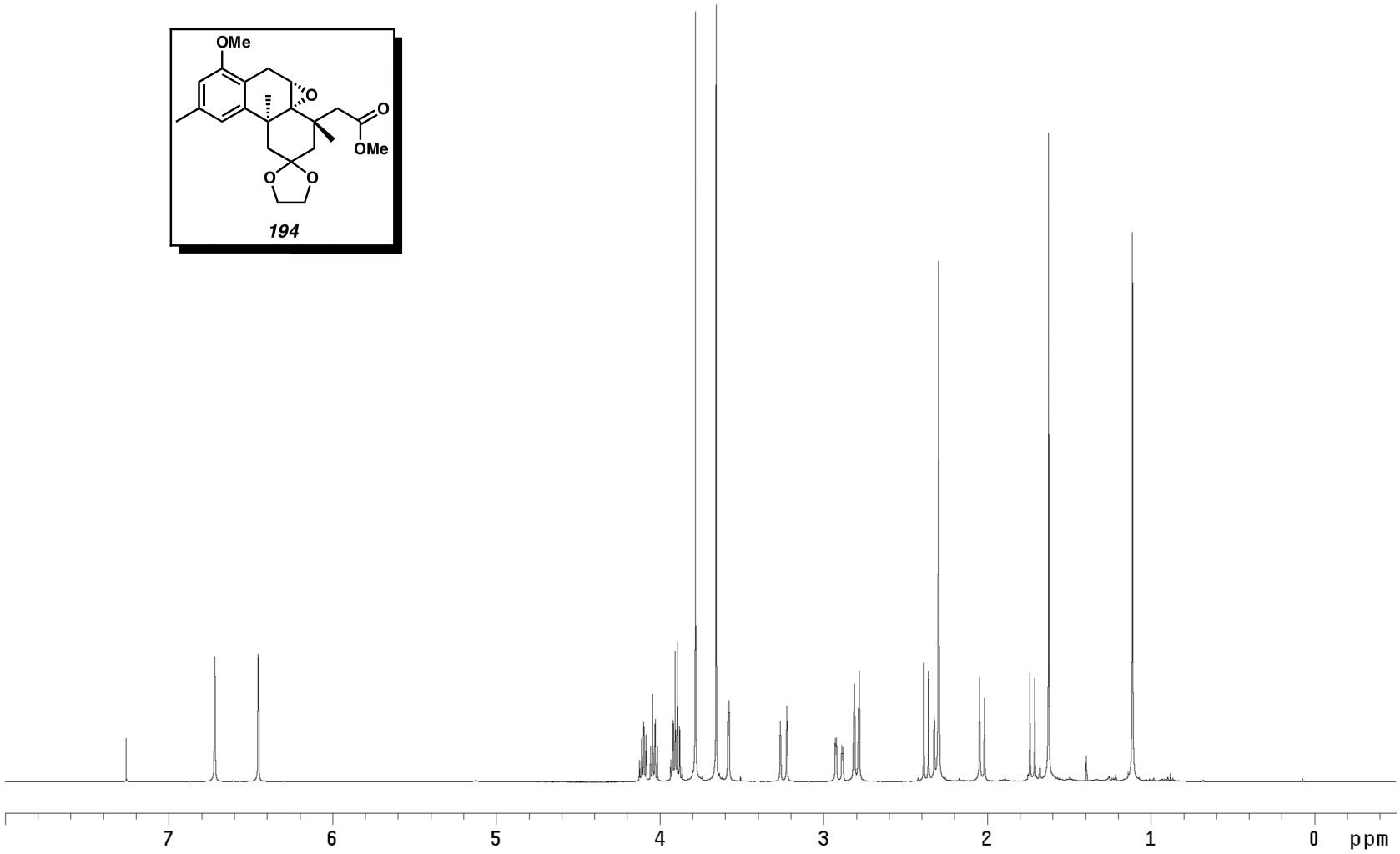

Figure A.58 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **194**.

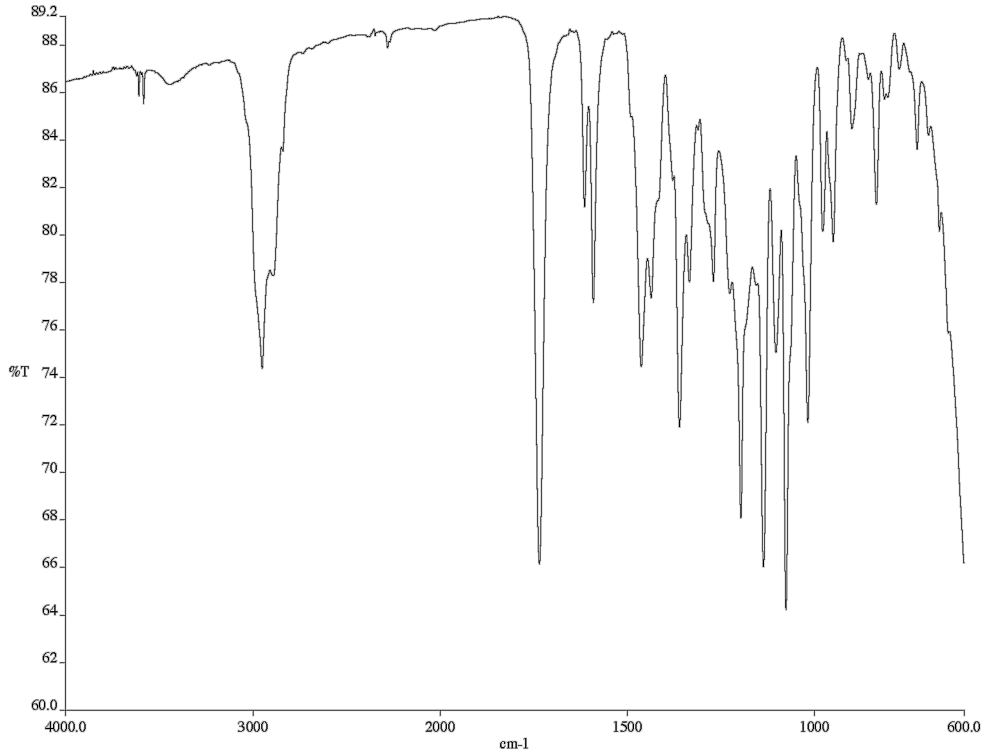

Figure A.59 Infrared spectrum (thin film/NaCl) of compound **194**.

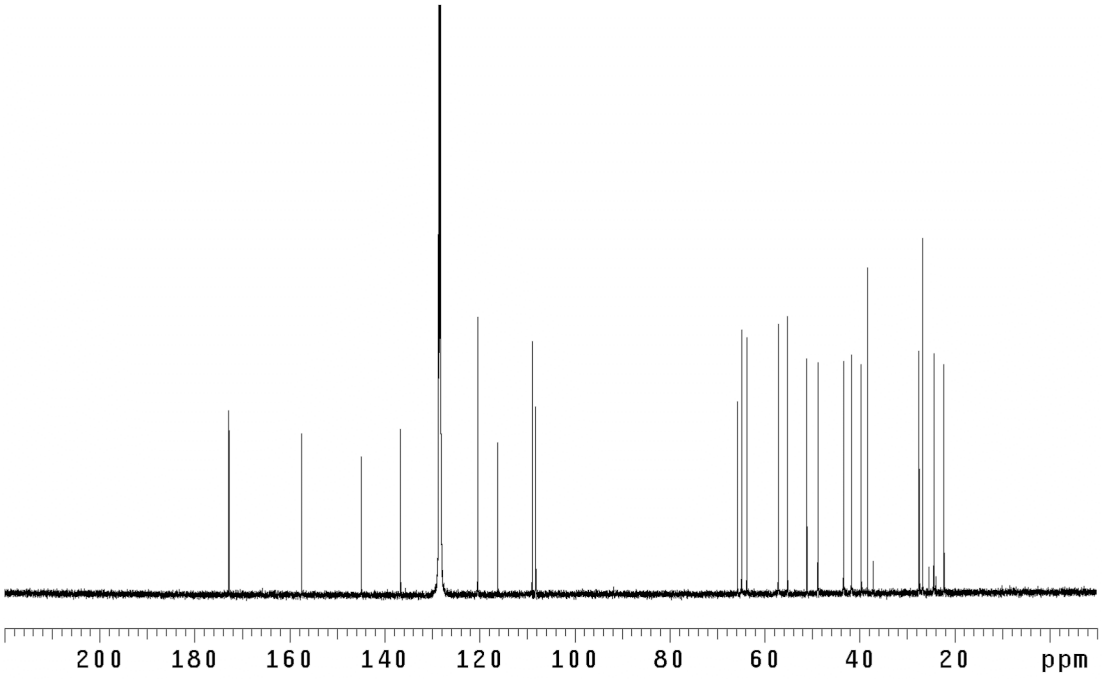

Figure A.60 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **194**.

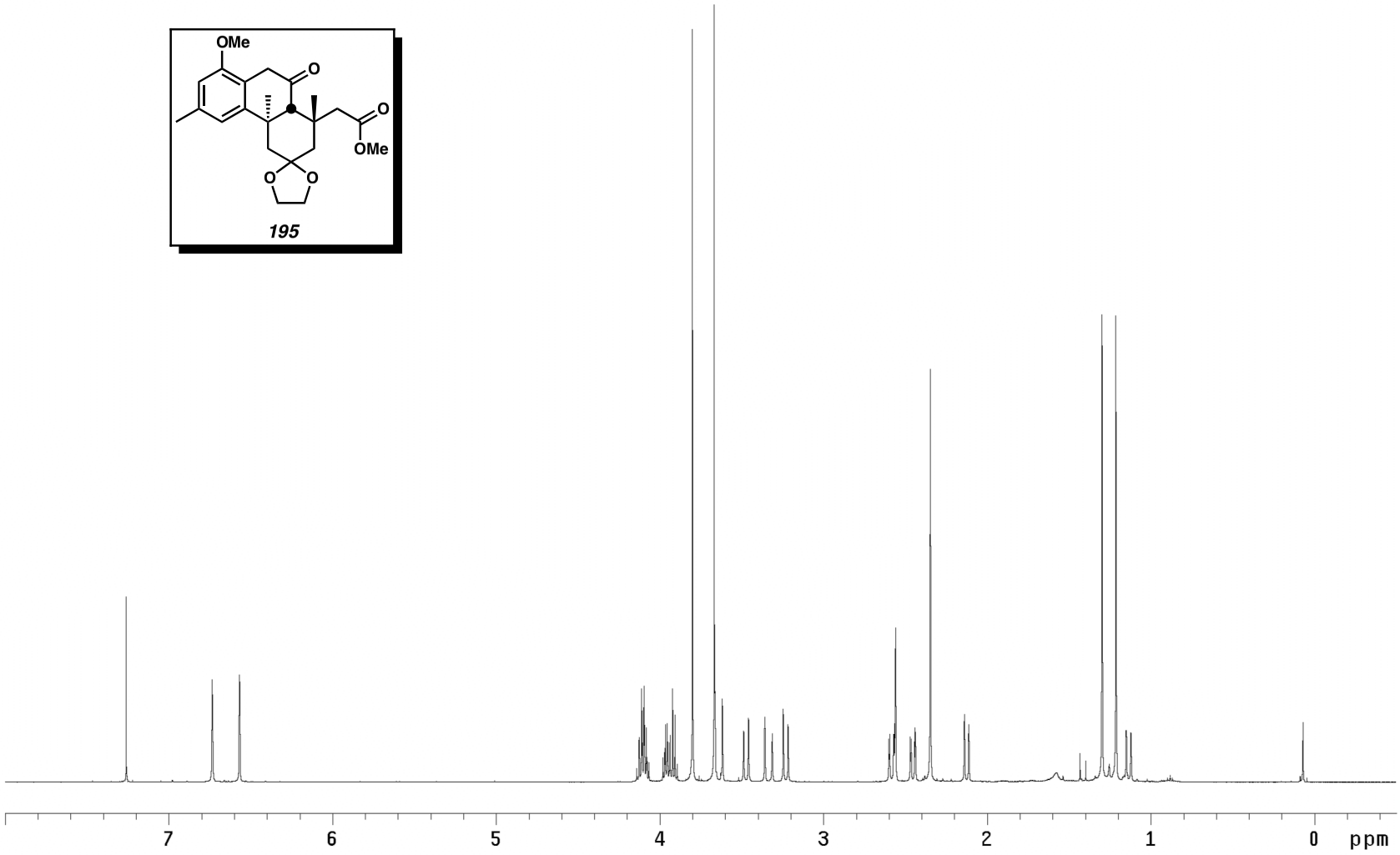

Figure A.61 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **195**.

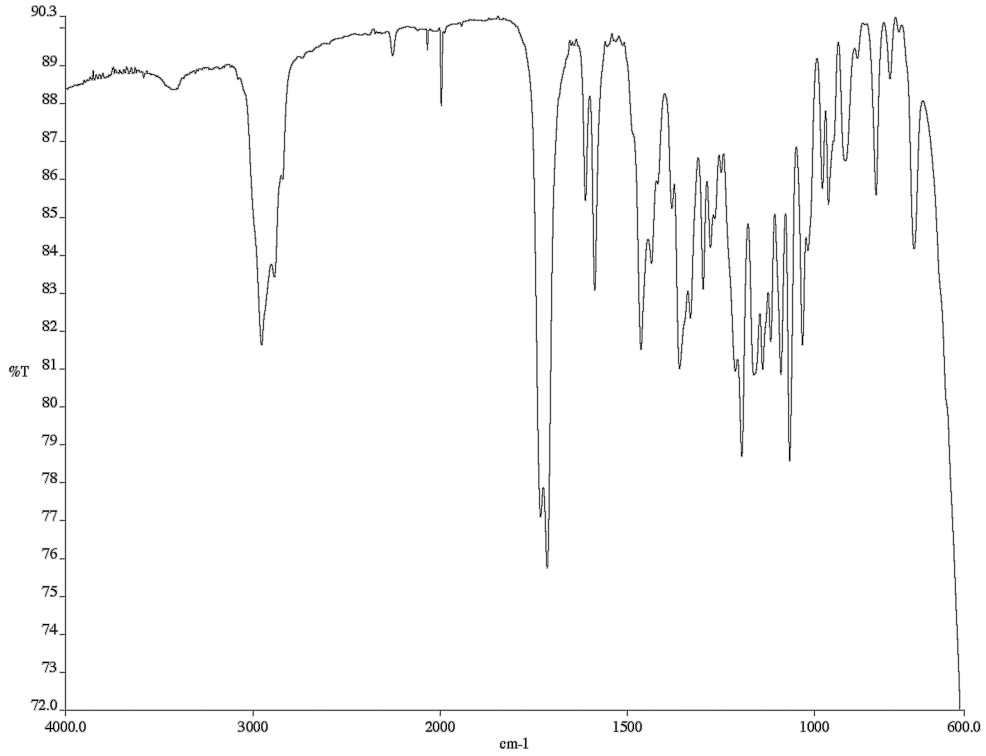

Figure A.62 Infrared spectrum (thin film/NaCl) of compound **195**.

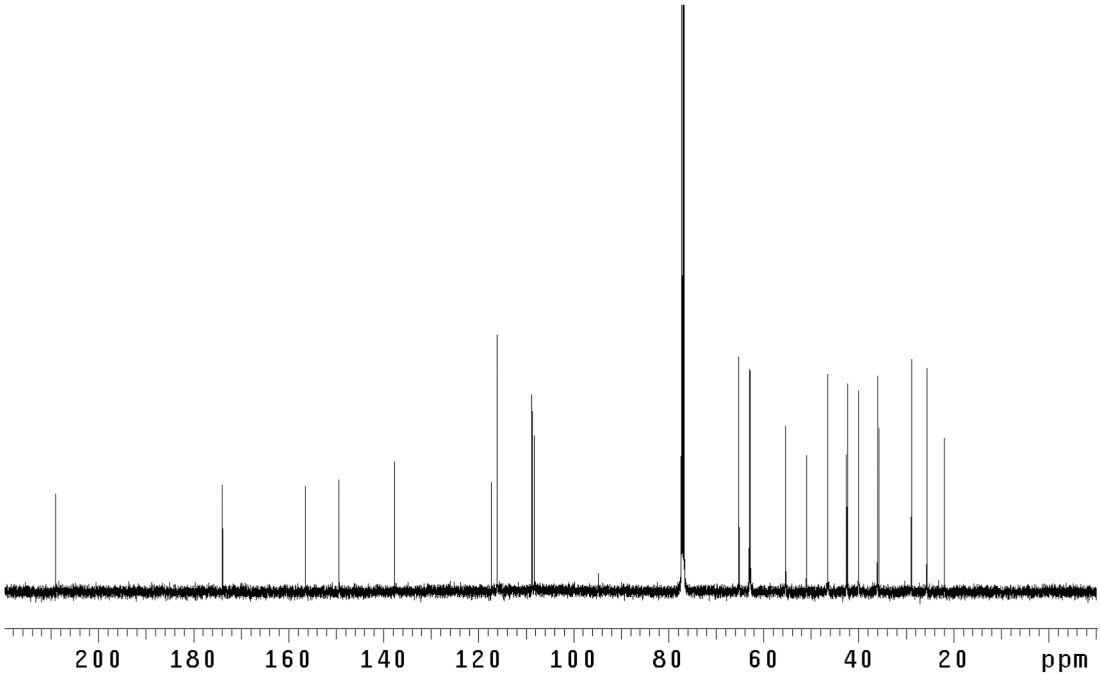

Figure A.63 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **195**.

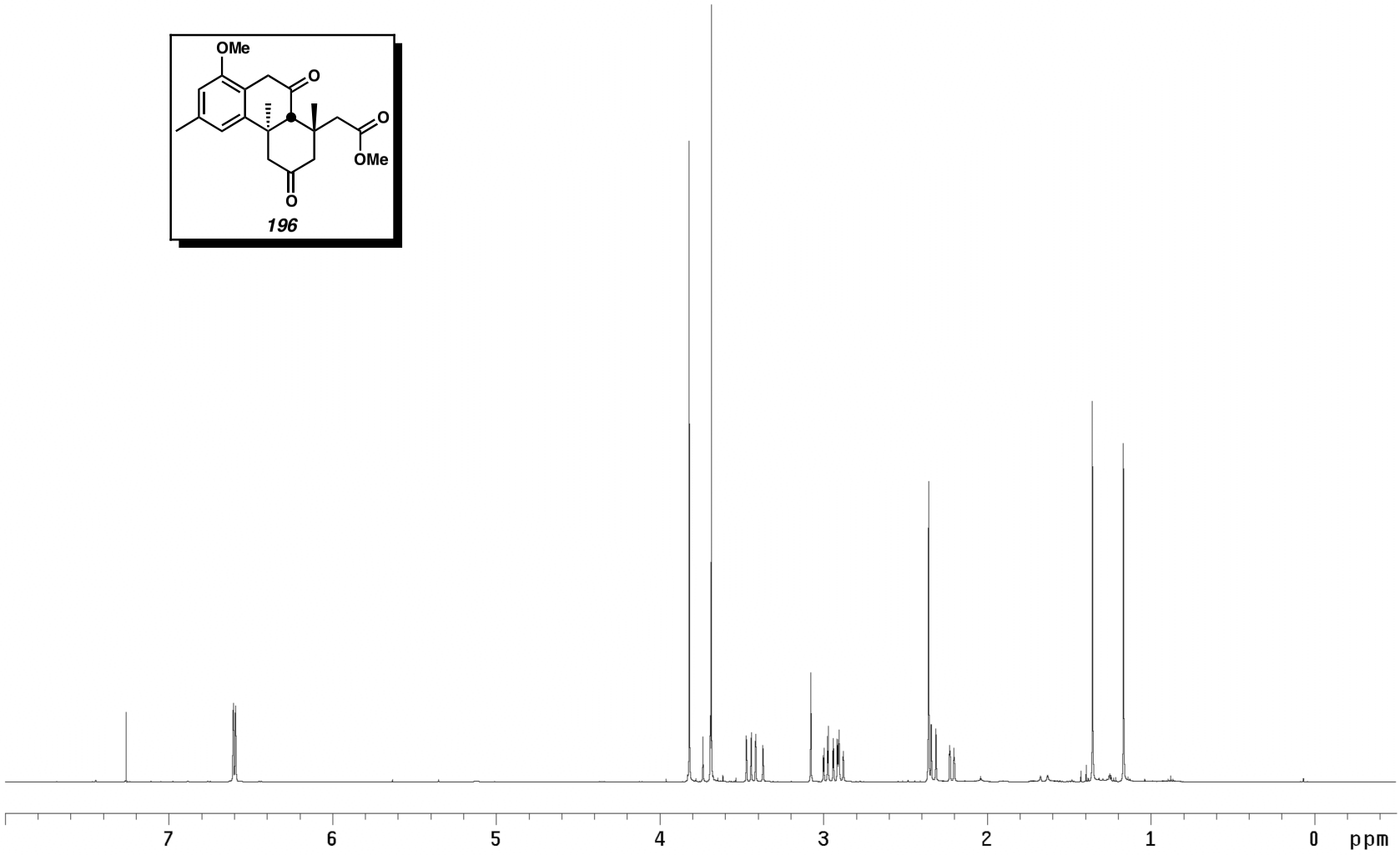

Figure A.64 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **196**.

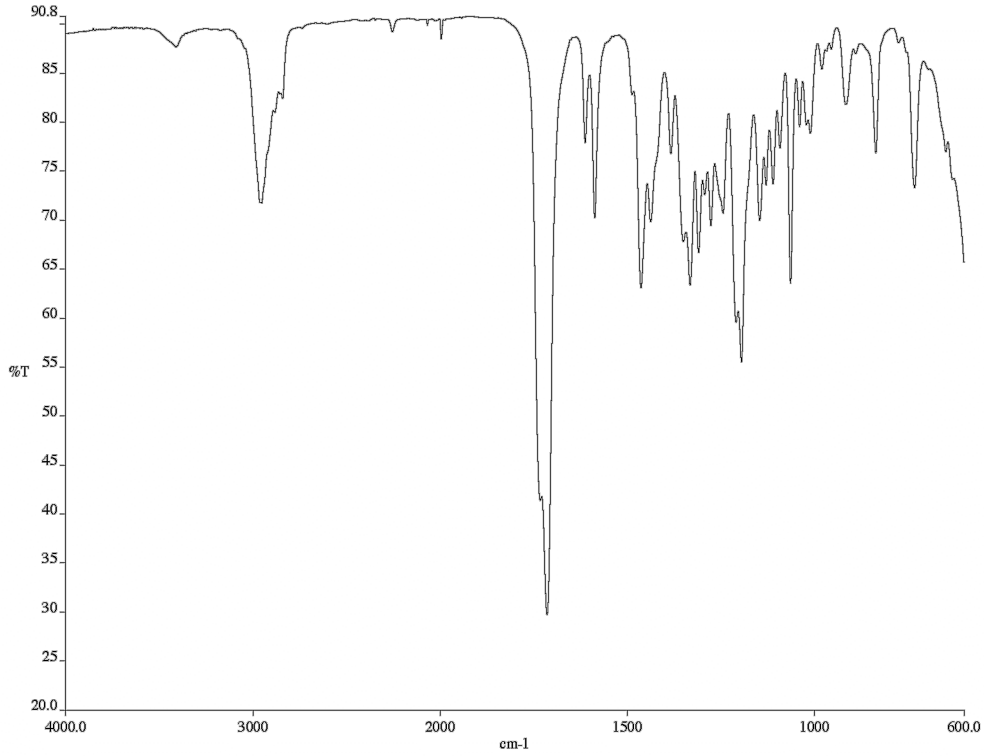

Figure A.65 Infrared spectrum (thin film/NaCl) of compound **196**.

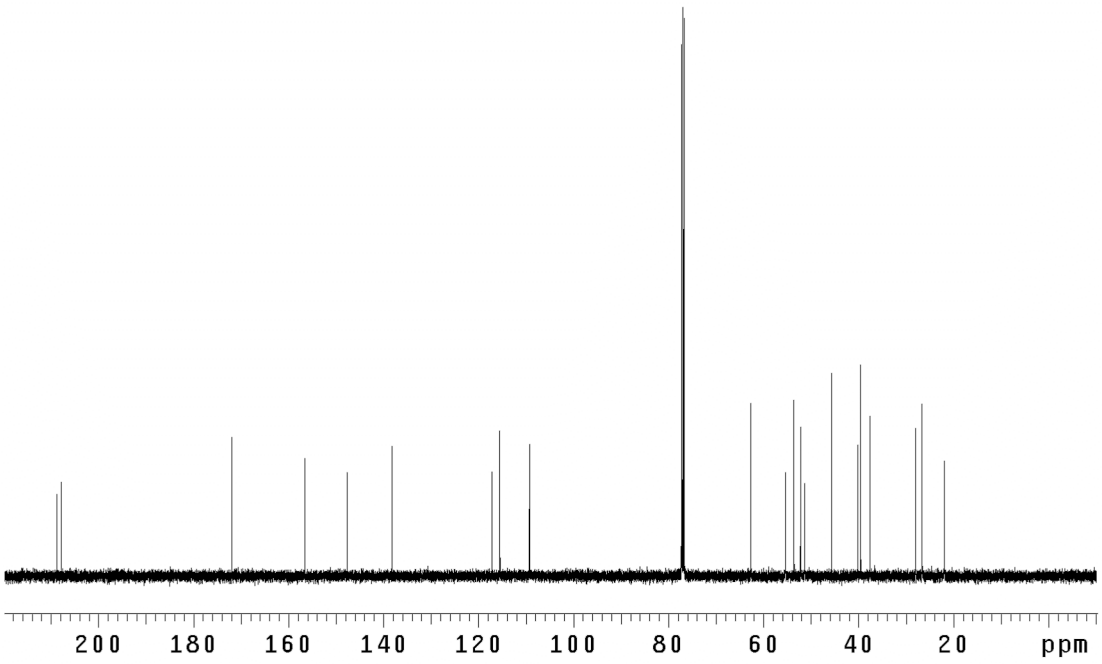

Figure A.66 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **196**.

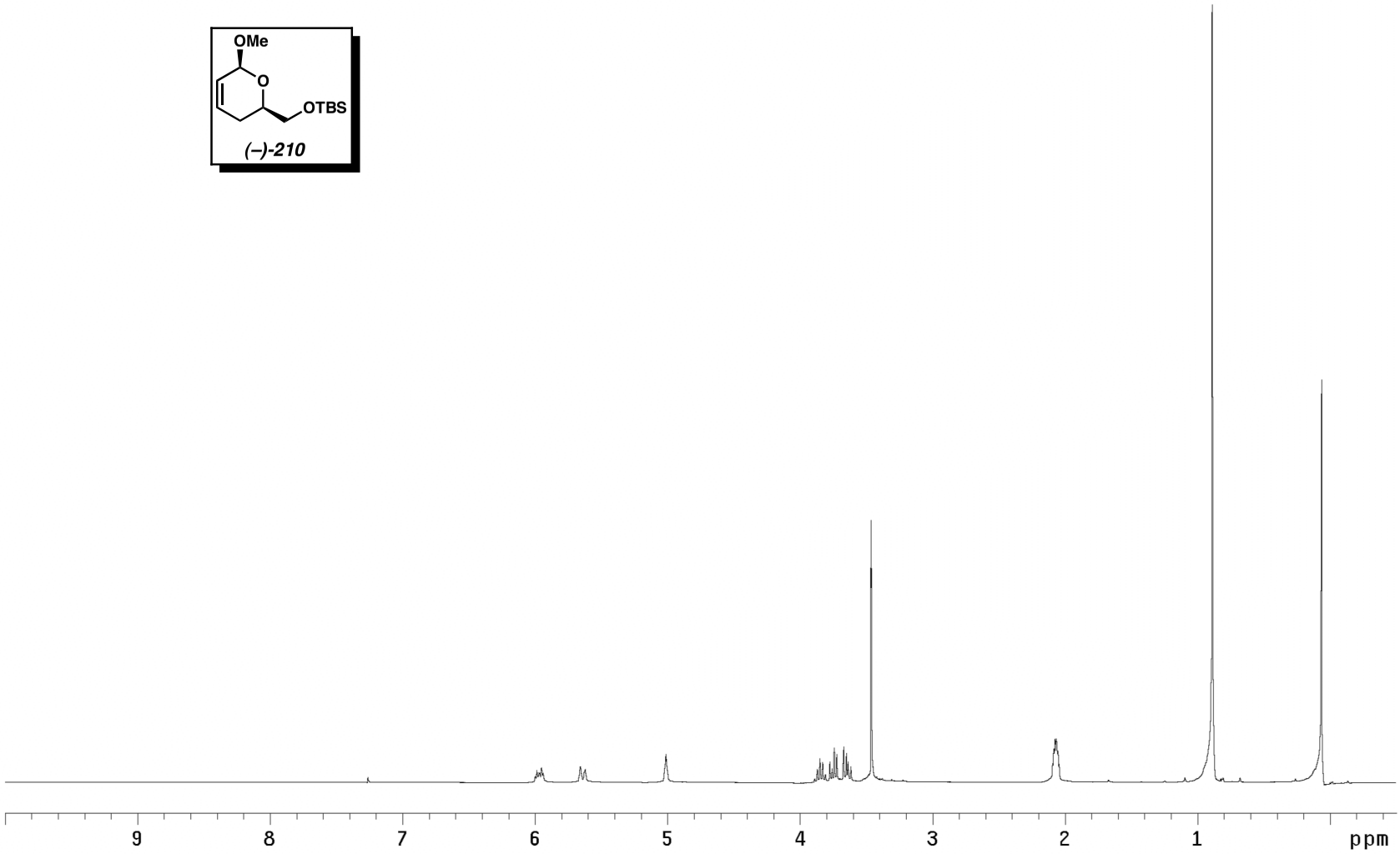

Figure A.67 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (–)-**210**.

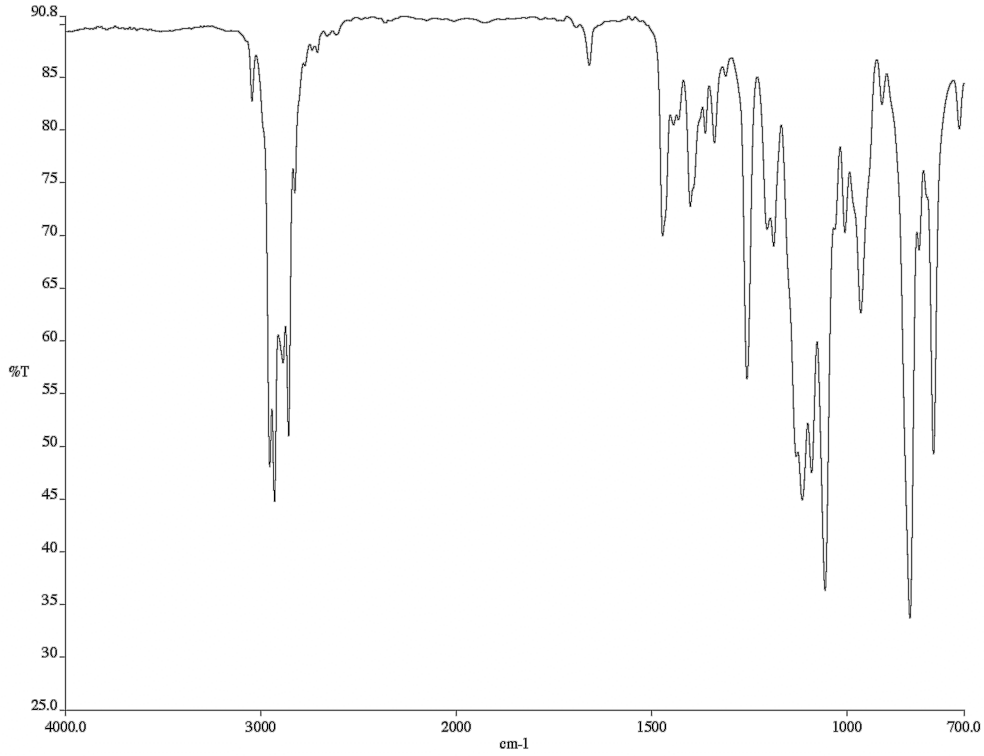

Figure A.68 Infrared spectrum (thin film/NaCl) of compound (–)-**210**.

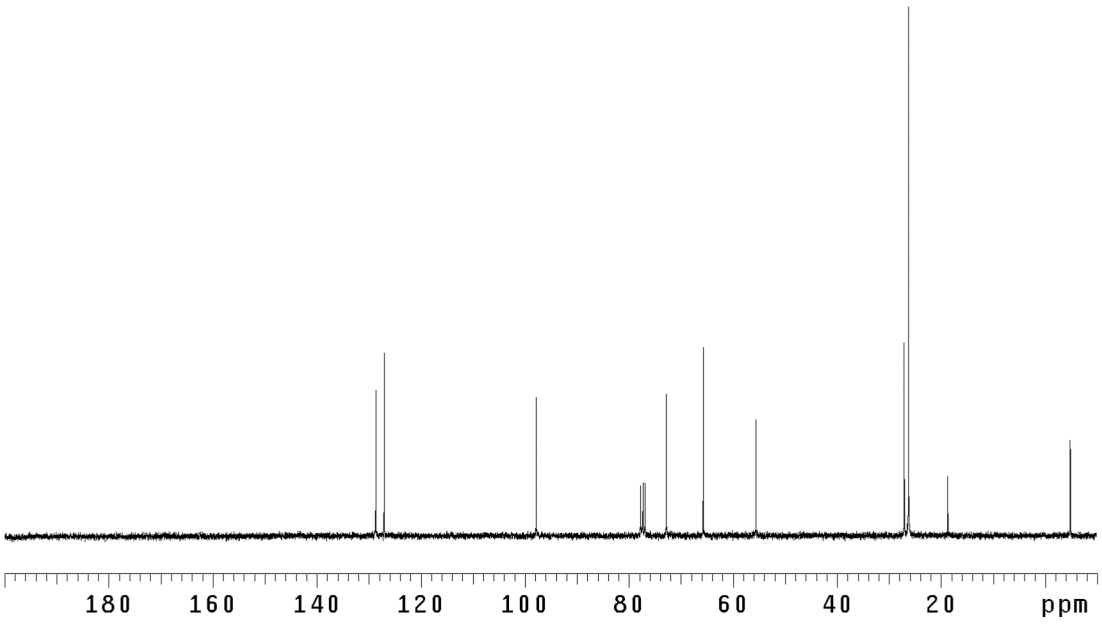

Figure A.69 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (–)-**210**.

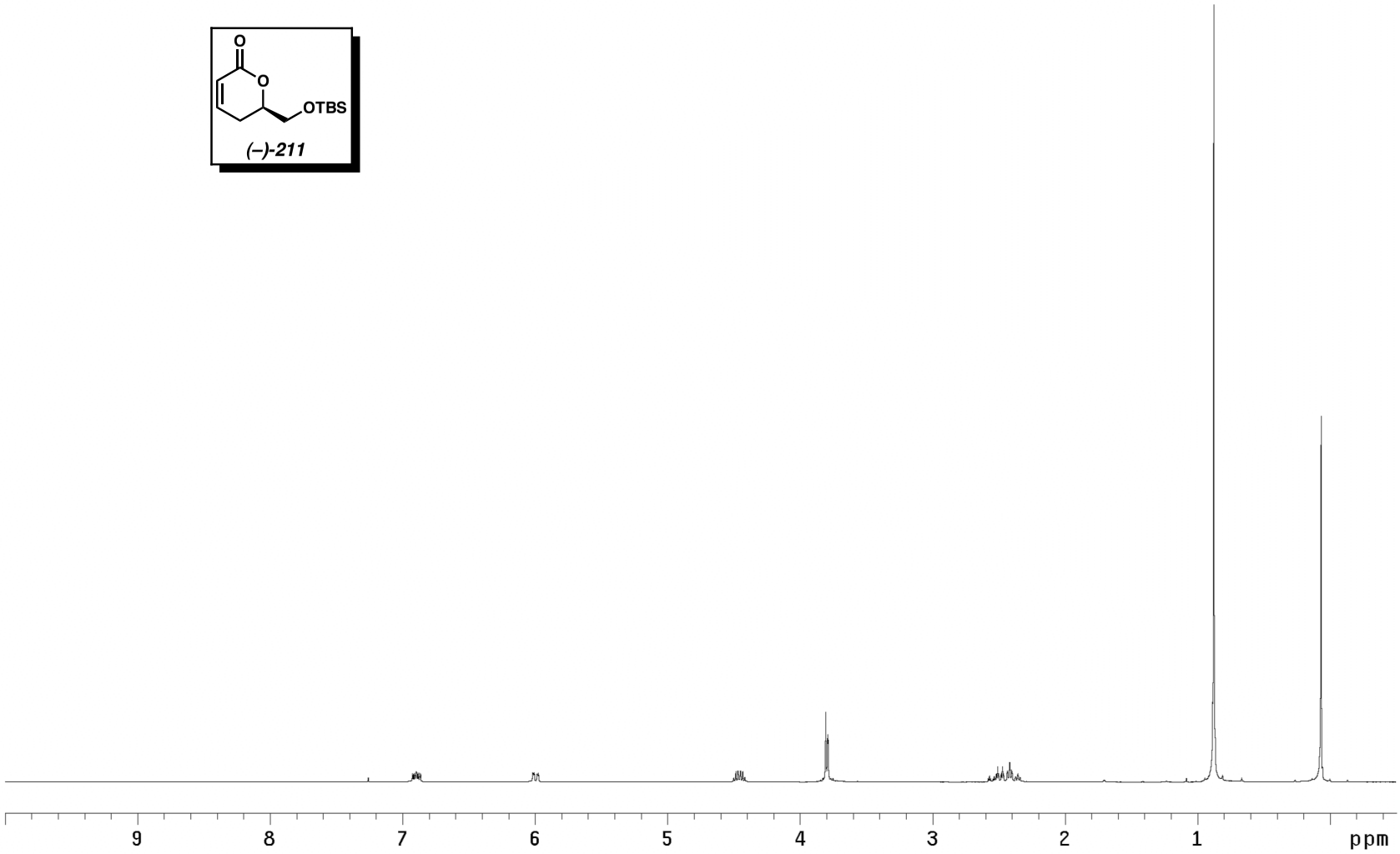

Figure A.70 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (–)-**211**.

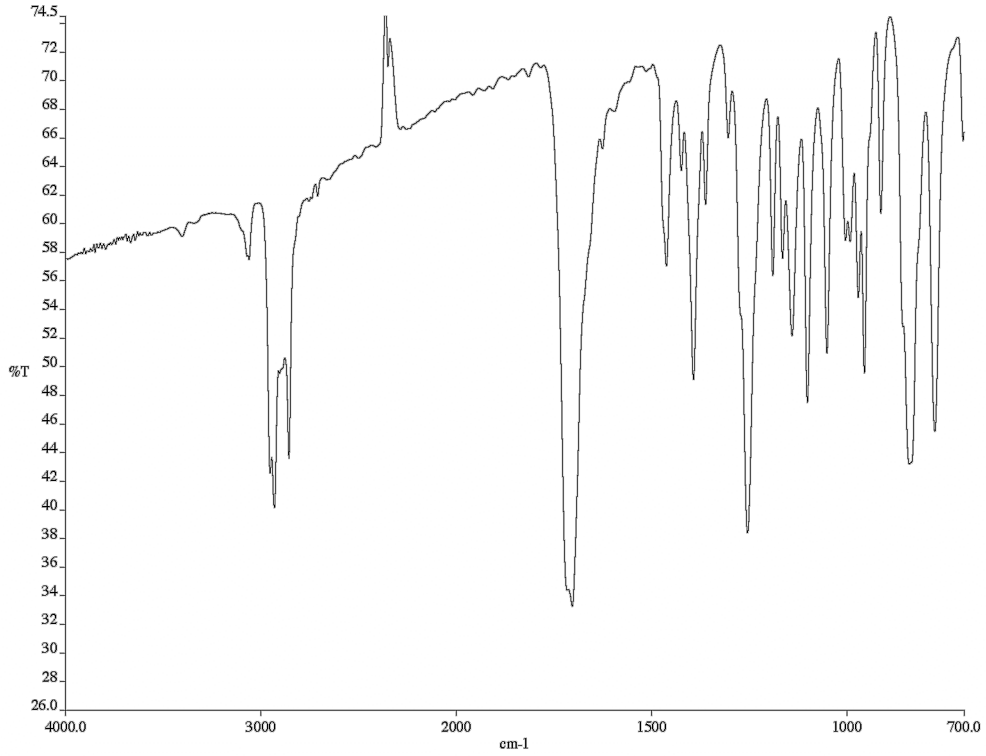

Figure A.71 Infrared spectrum (thin film/NaCl) of compound (–)-**211**.

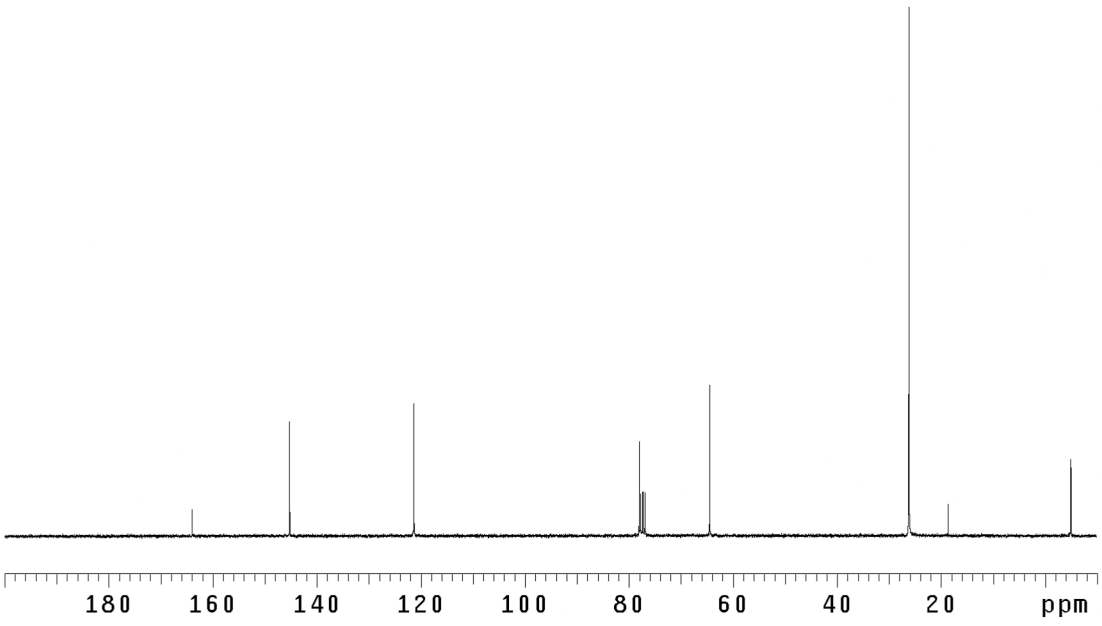

Figure A.72 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (–)-**211**.

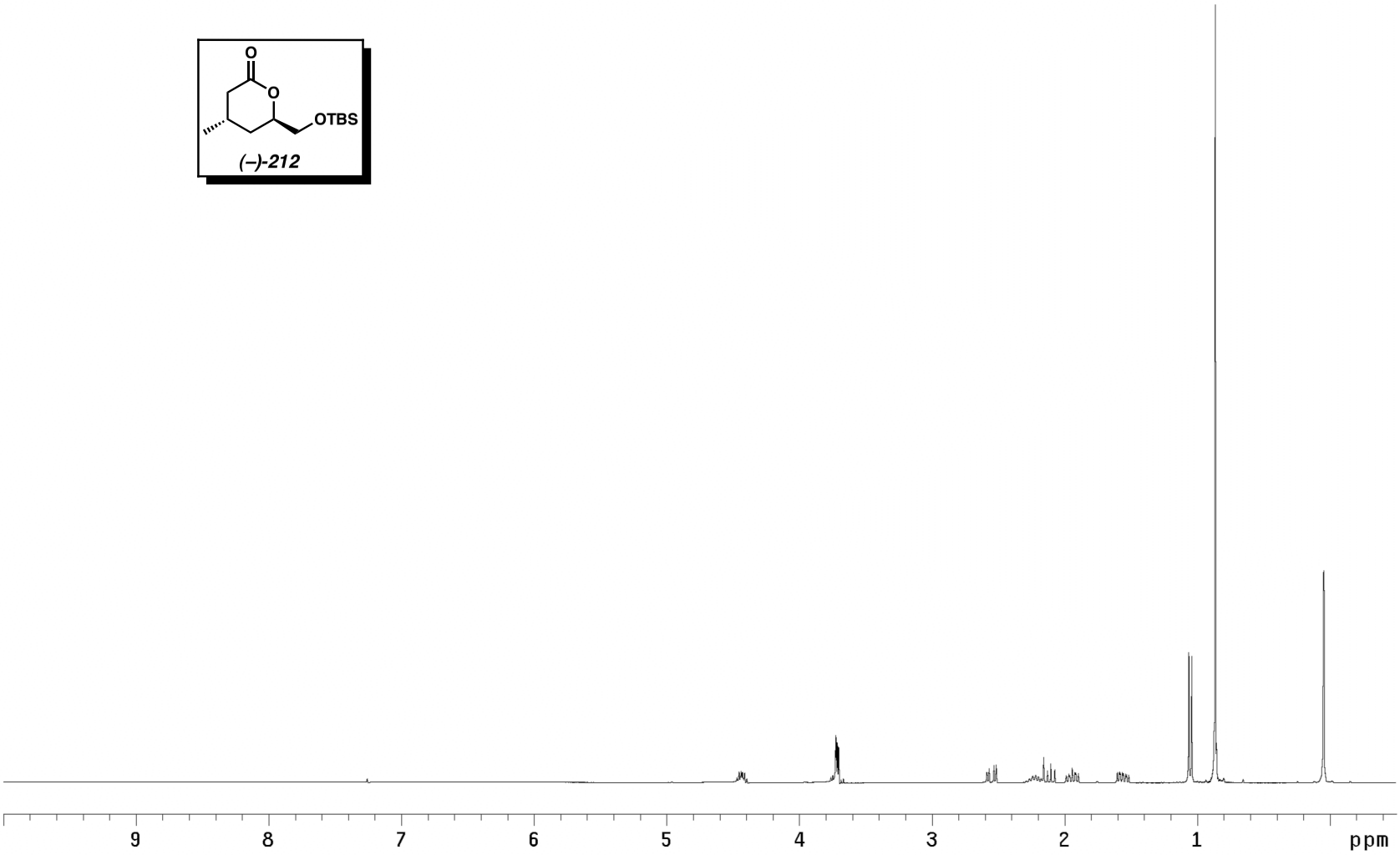

Figure A.73 ^{1}H NMR (300 MHz, $CDCl_3$) of compound (–)-**212**.

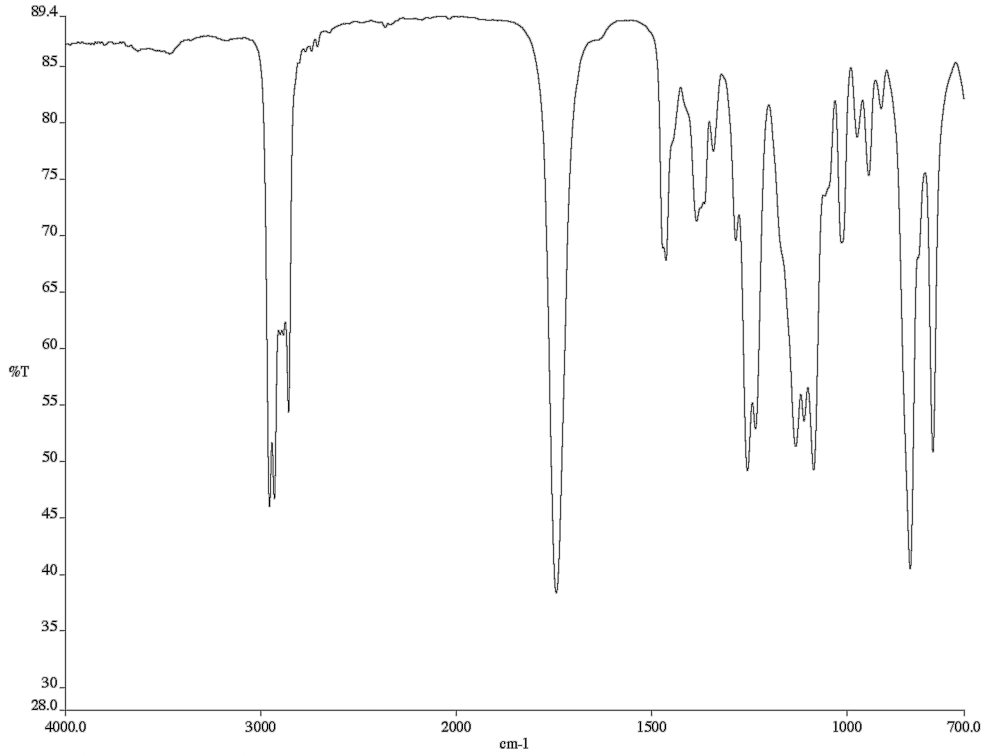

Figure A.74 Infrared spectrum (thin film/NaCl) of compound (–)-**212**.

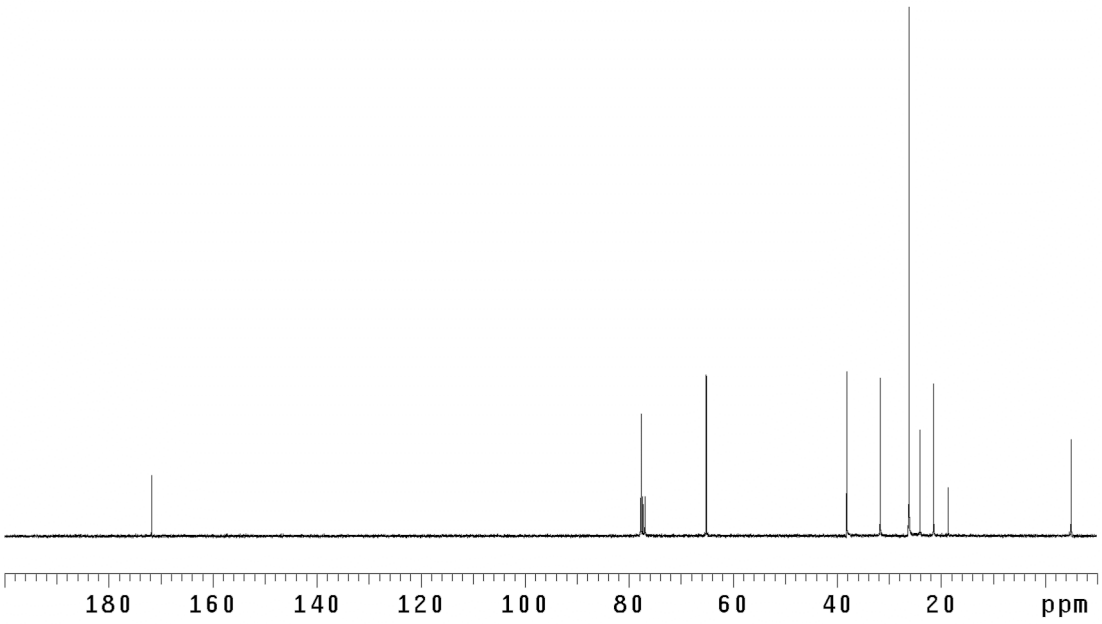

Figure A.75 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (–)-**212**.

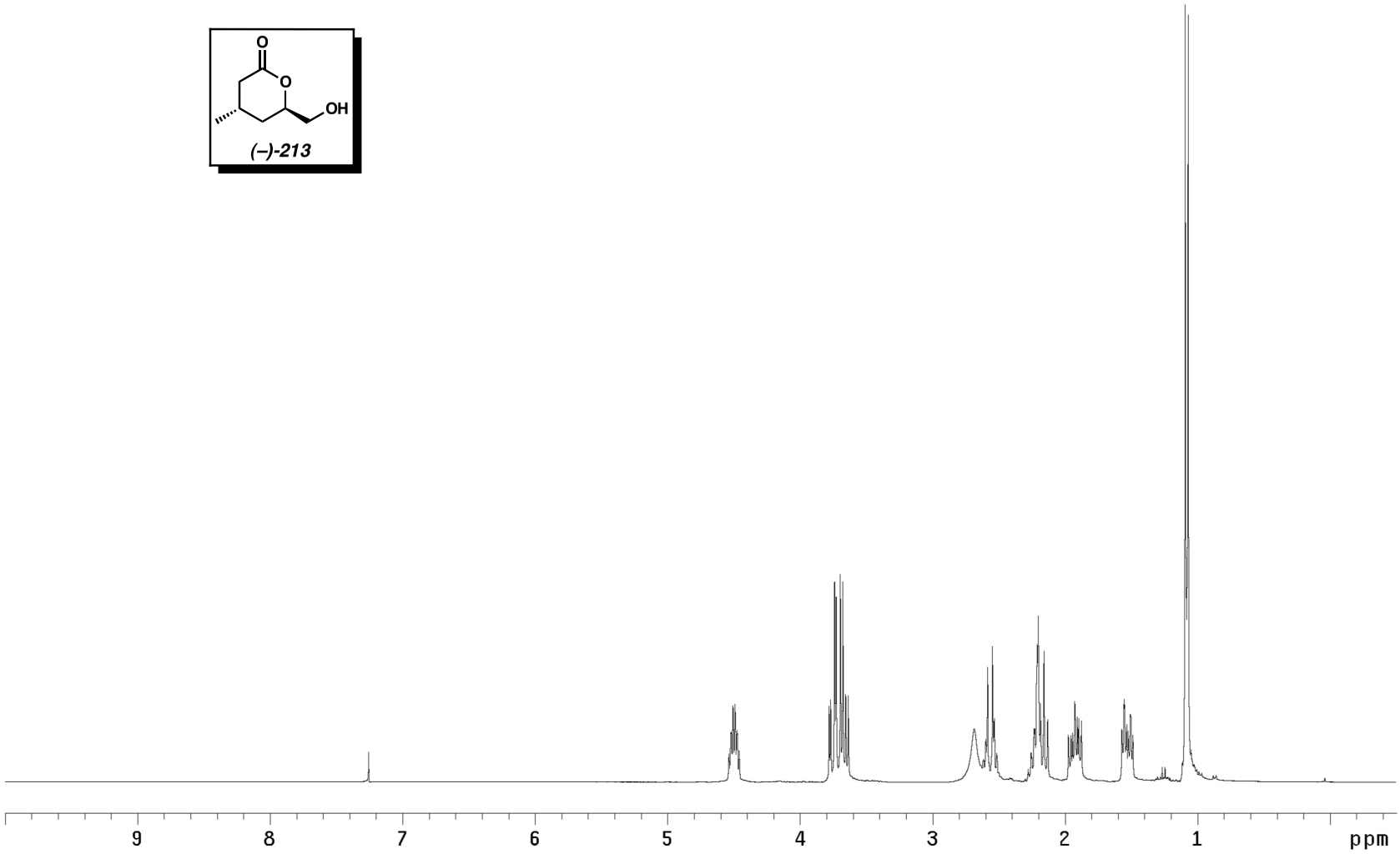

Figure A.76 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **213**.

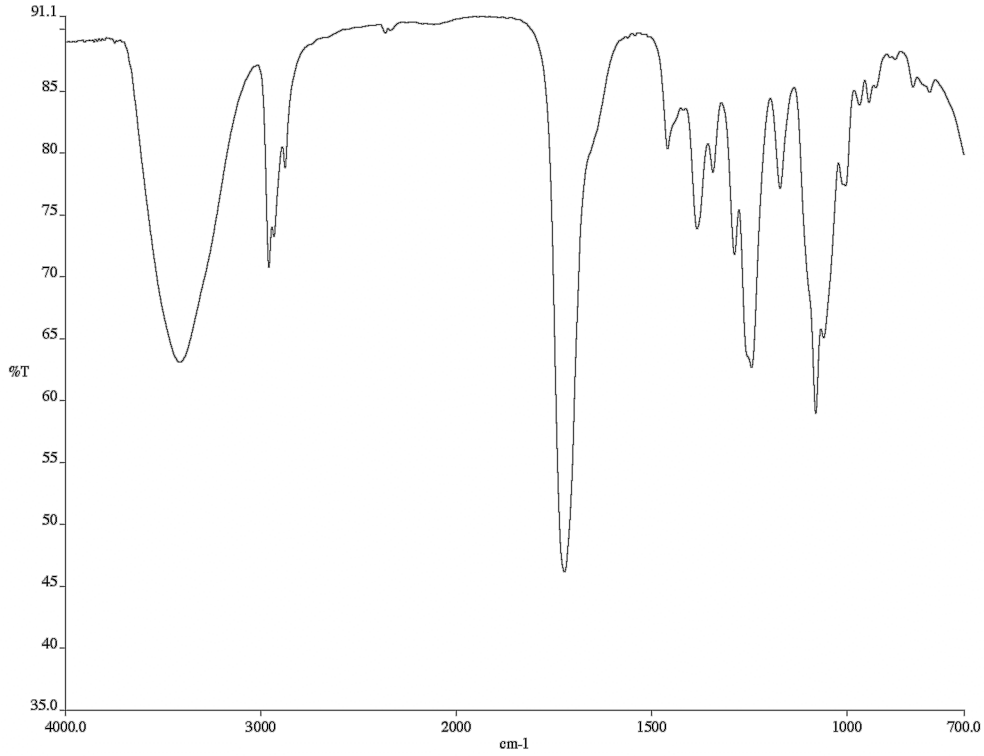

Figure A.77 Infrared spectrum (thin film/NaCl) of compound (–)-**213**.

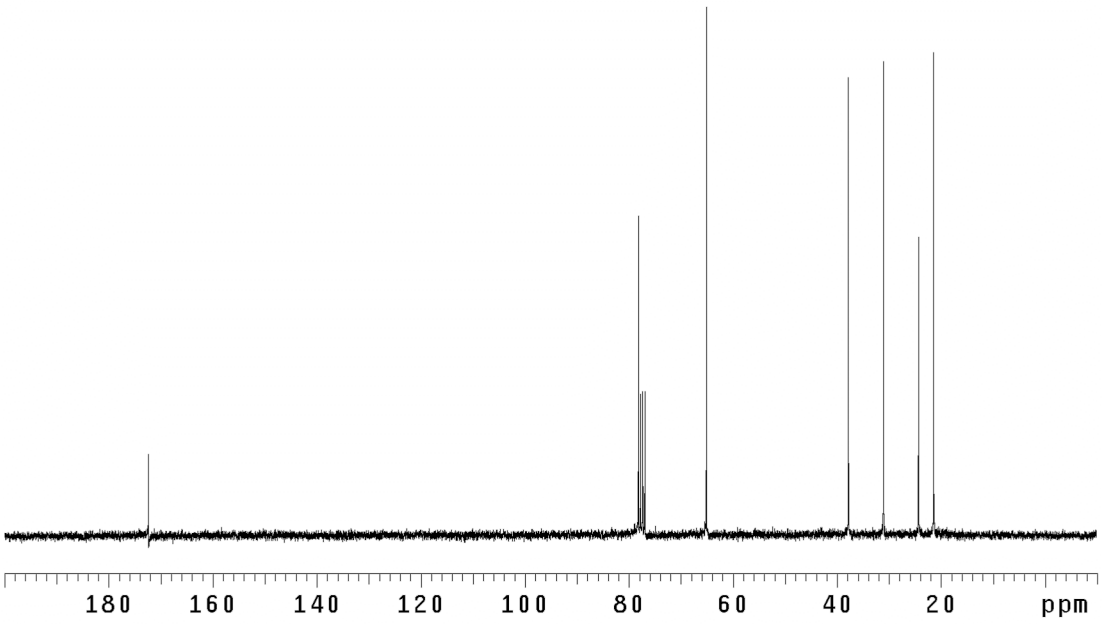

Figure A.78 ^{13}C NMR (75 MHz, $CDCl_3$) of compound (–)-**213**.

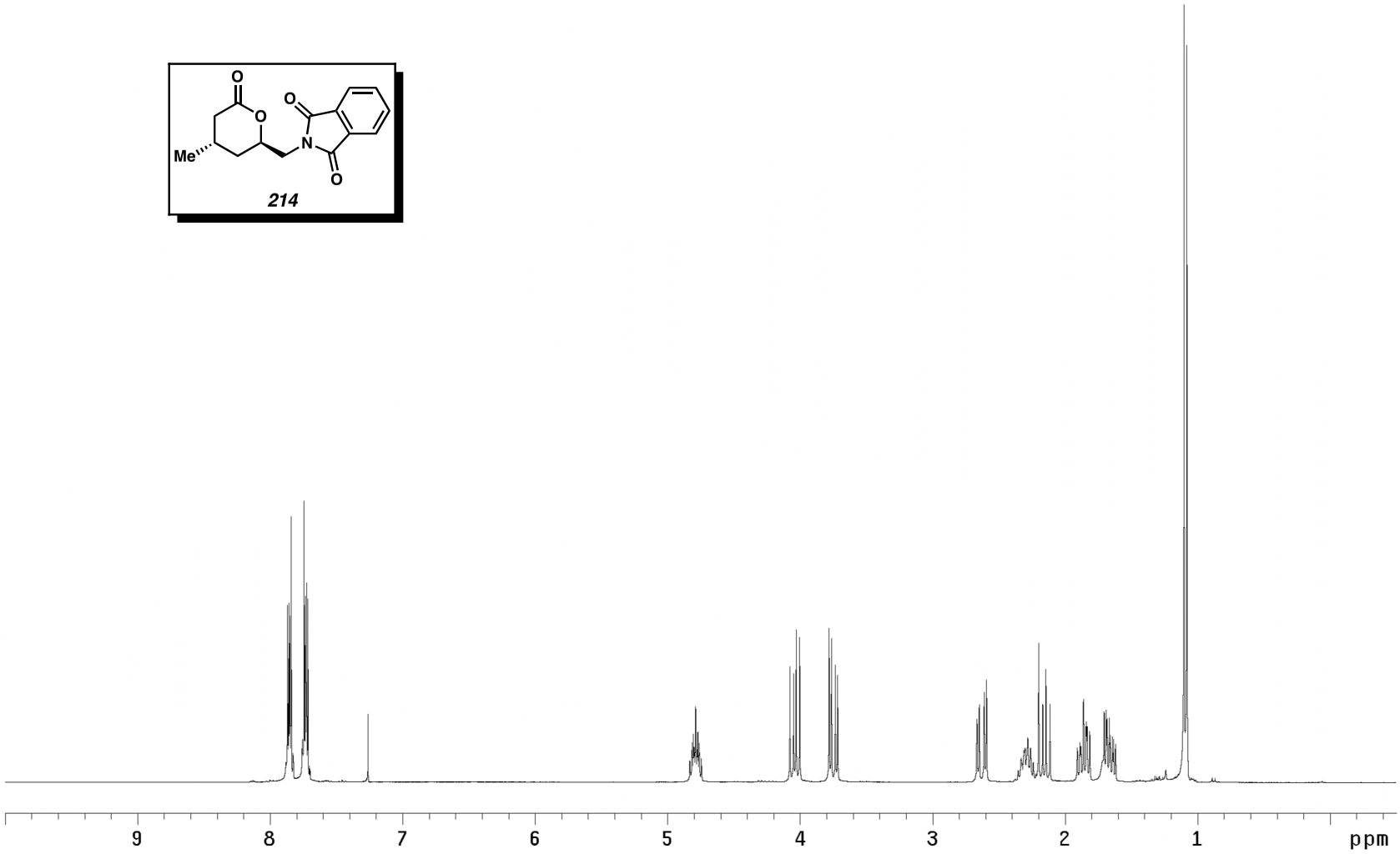

Figure A.79 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **214**.

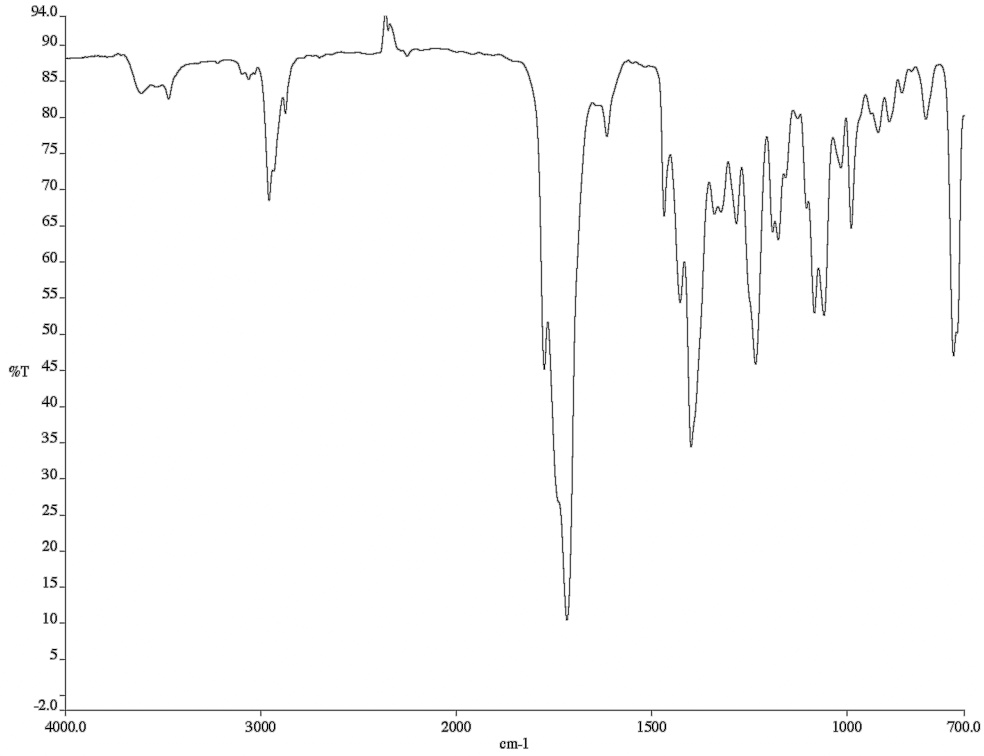

Figure A.80 Infrared spectrum (thin film/NaCl) of compound **214**.

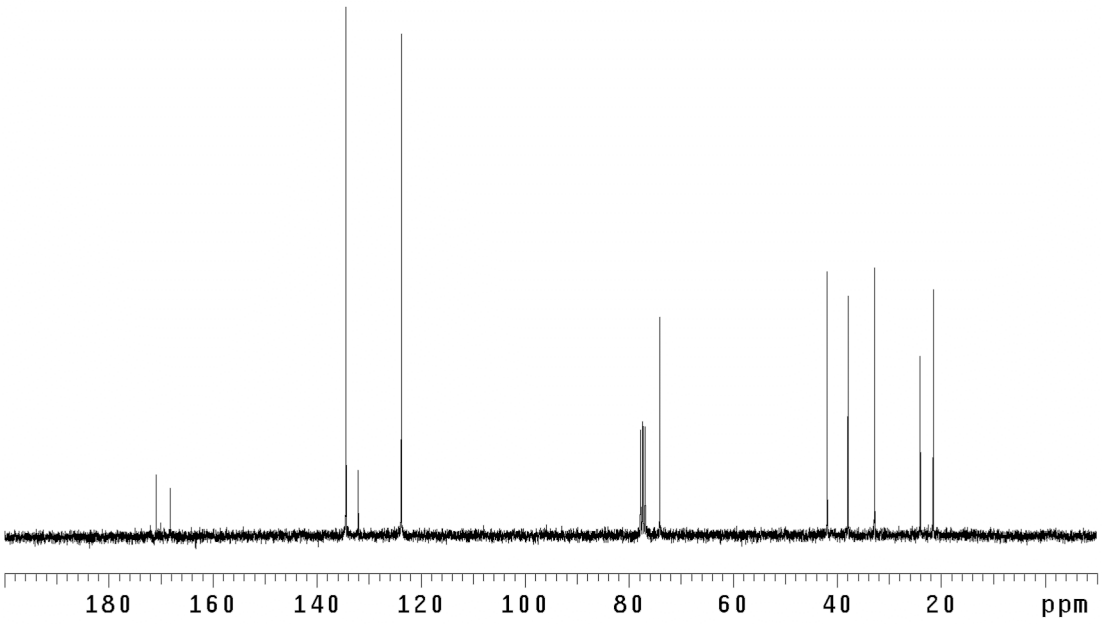

Figure A.81 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **214**.

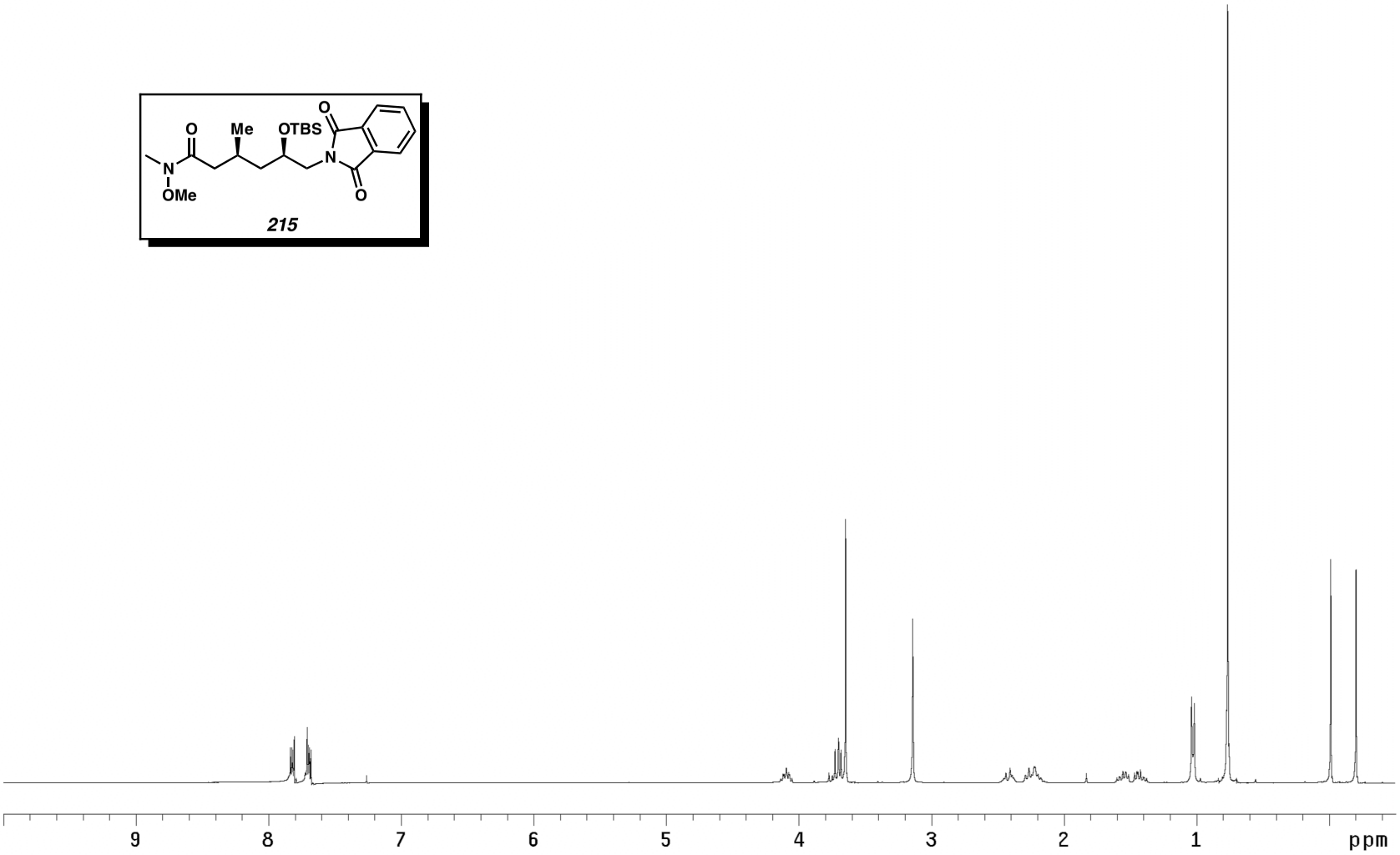

Figure A.82 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **215**.

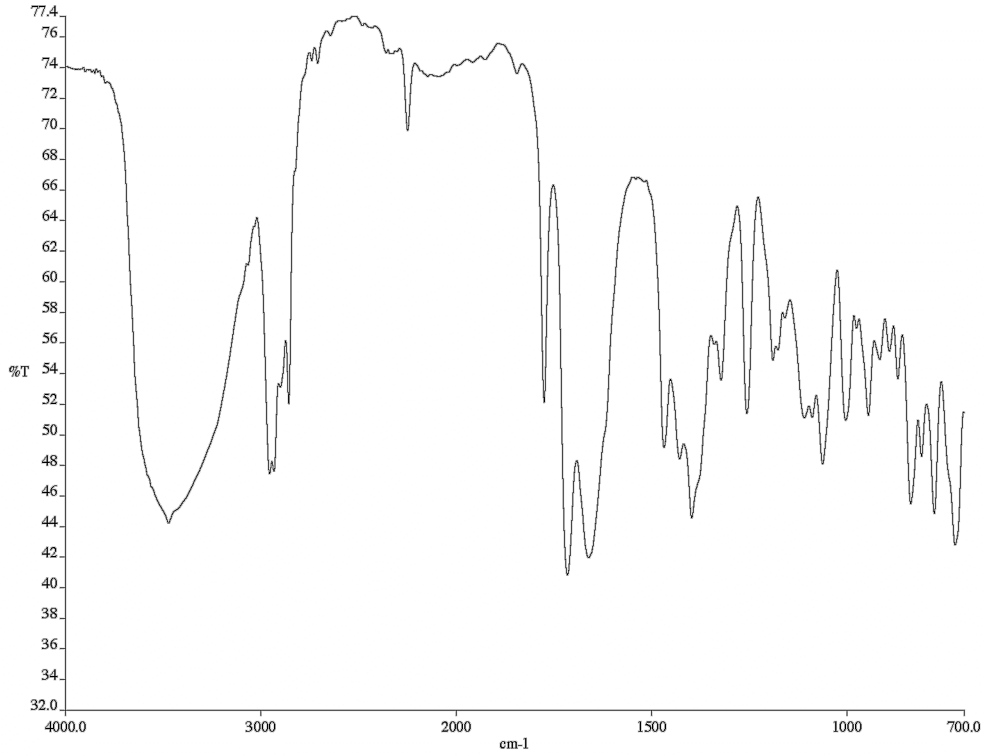

Figure A.83 Infrared spectrum (thin film/NaCl) of compound **215**.

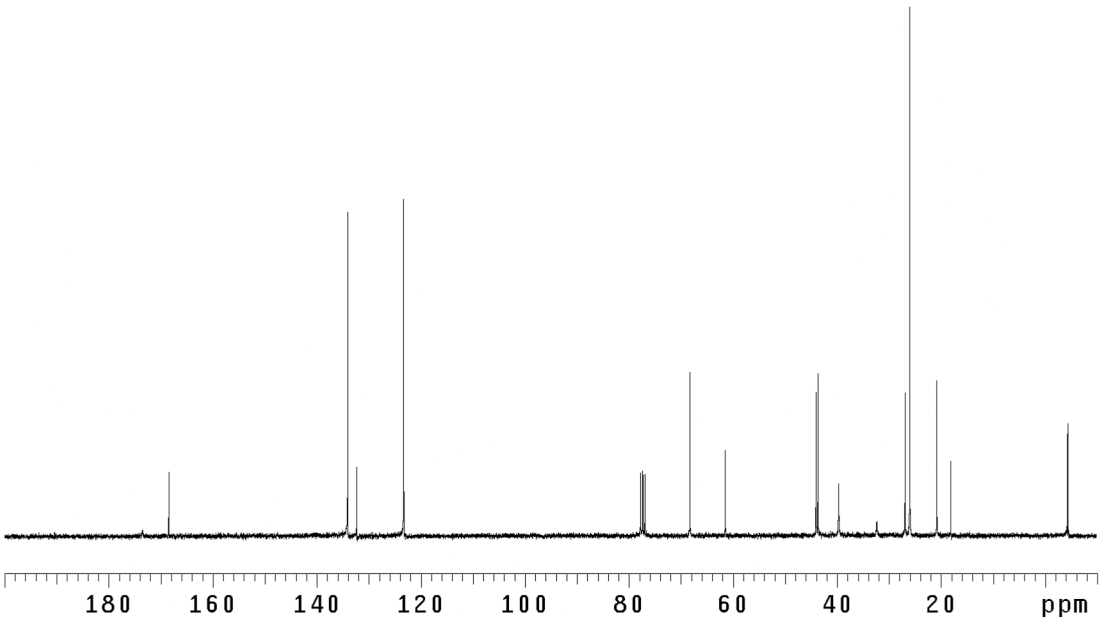

Figure A.84 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **215**.

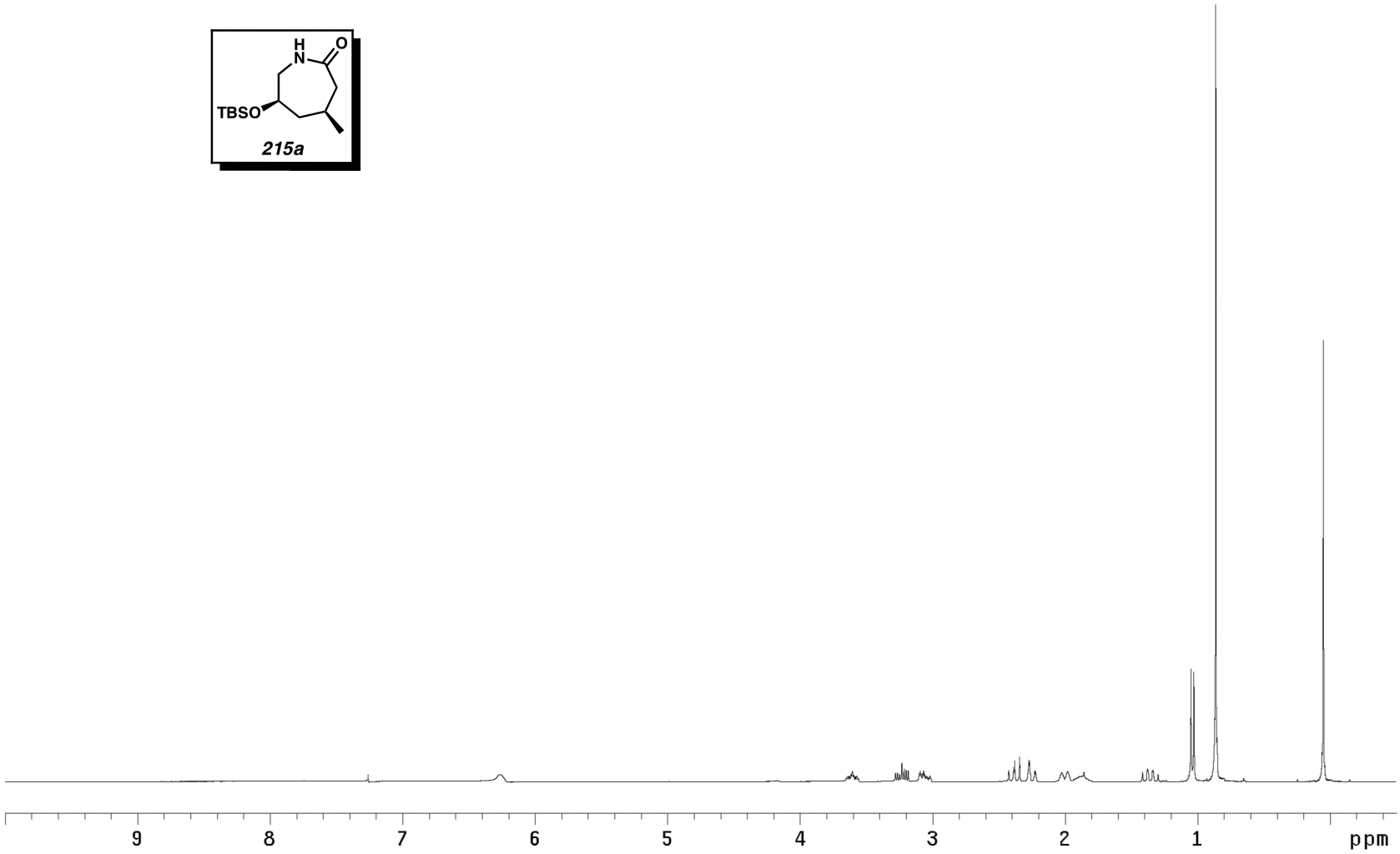

Figure A.85 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **215a**.

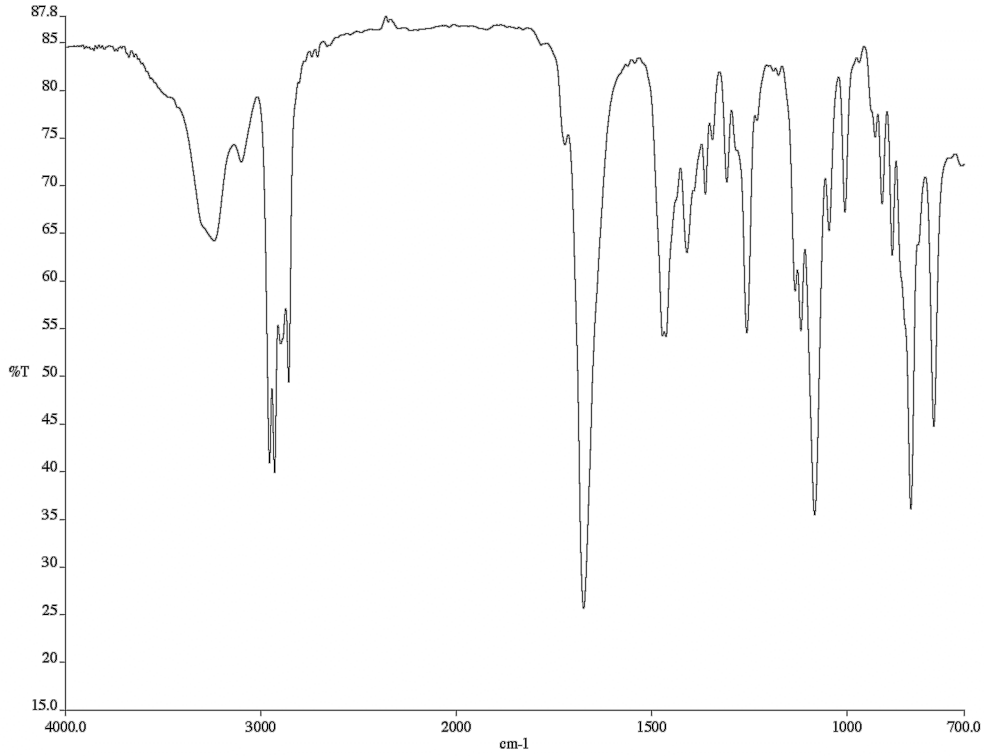

Figure A.86 Infrared spectrum (thin film/NaCl) of compound **215a**.

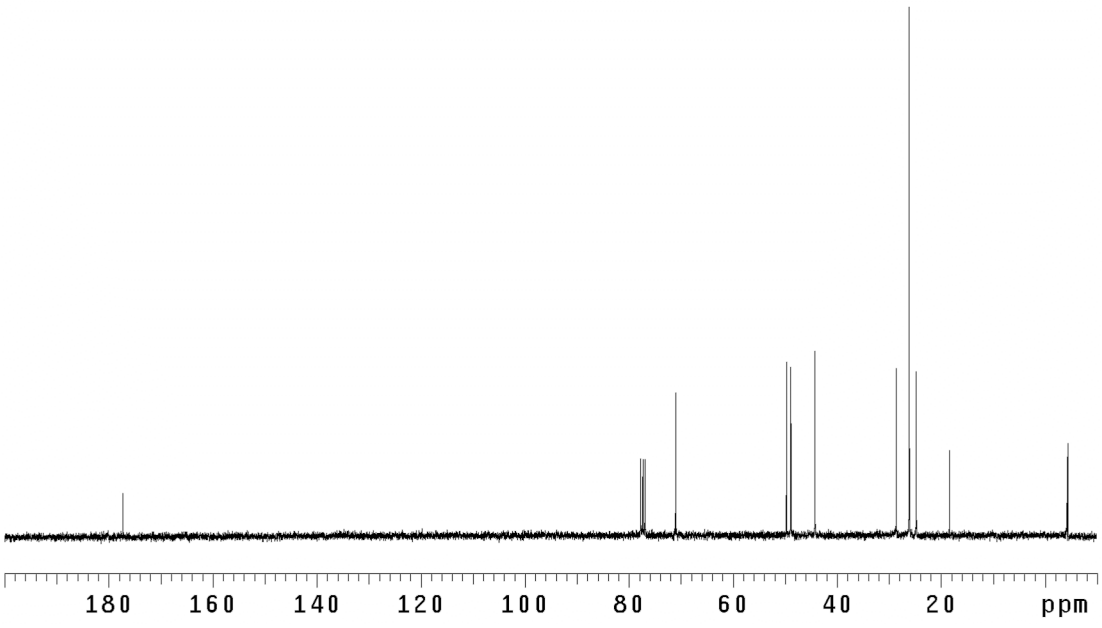

Figure A.87 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **215a**.

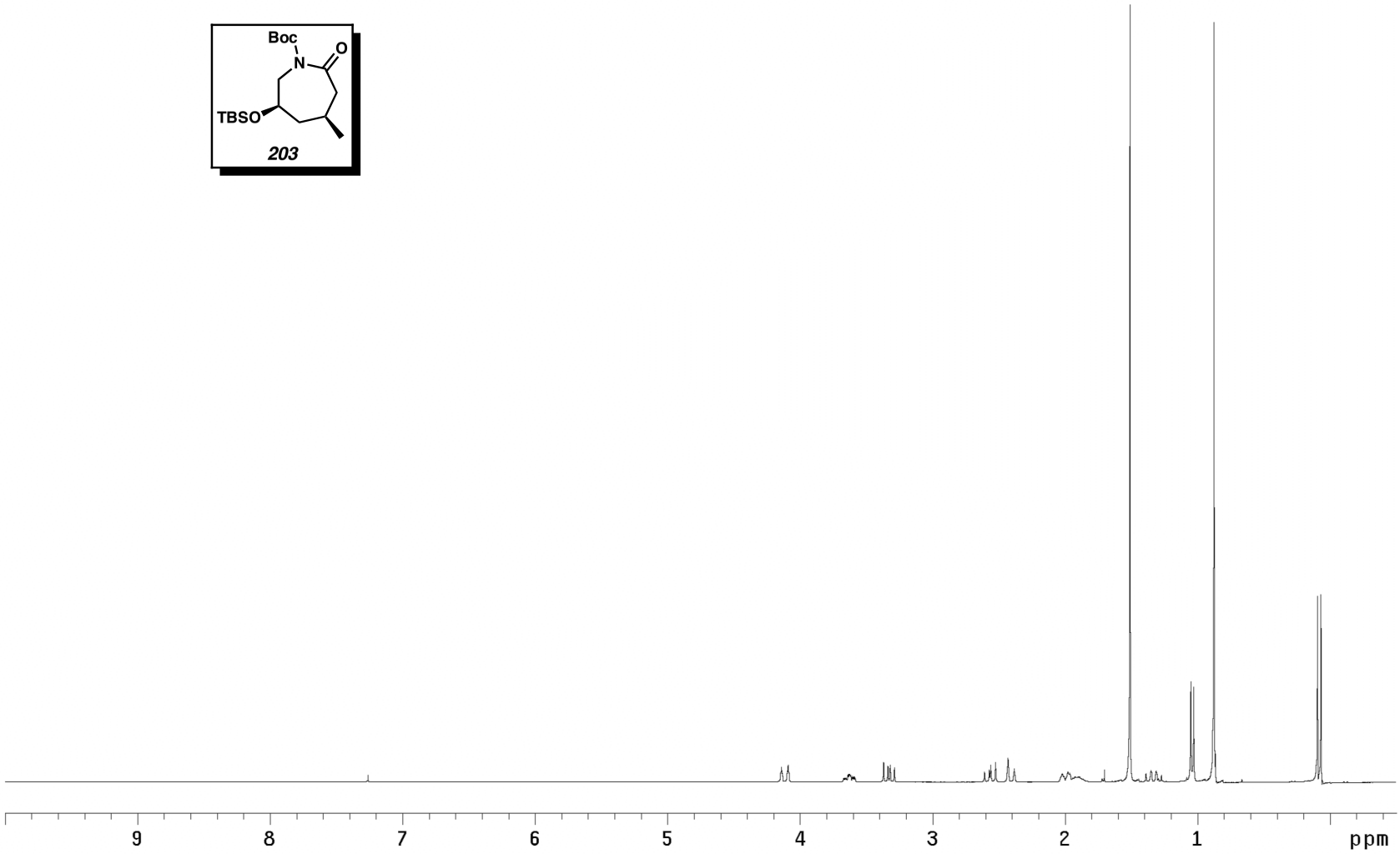

Figure A.88 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **203**.

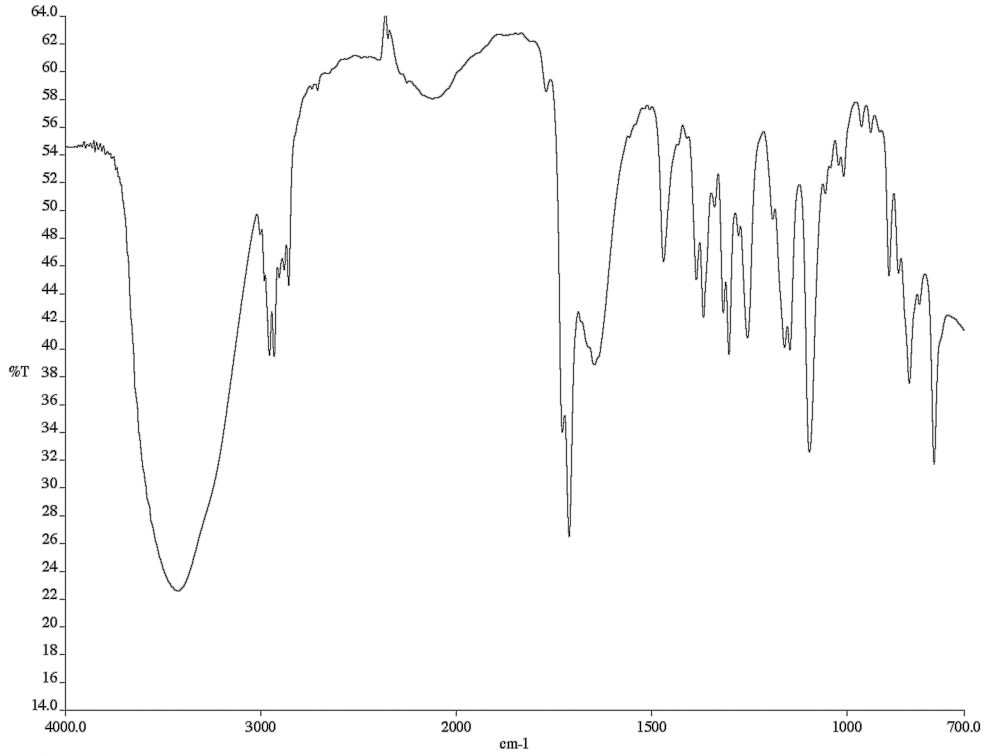

Figure A.89 Infrared spectrum (thin film/NaCl) of compound **203**.

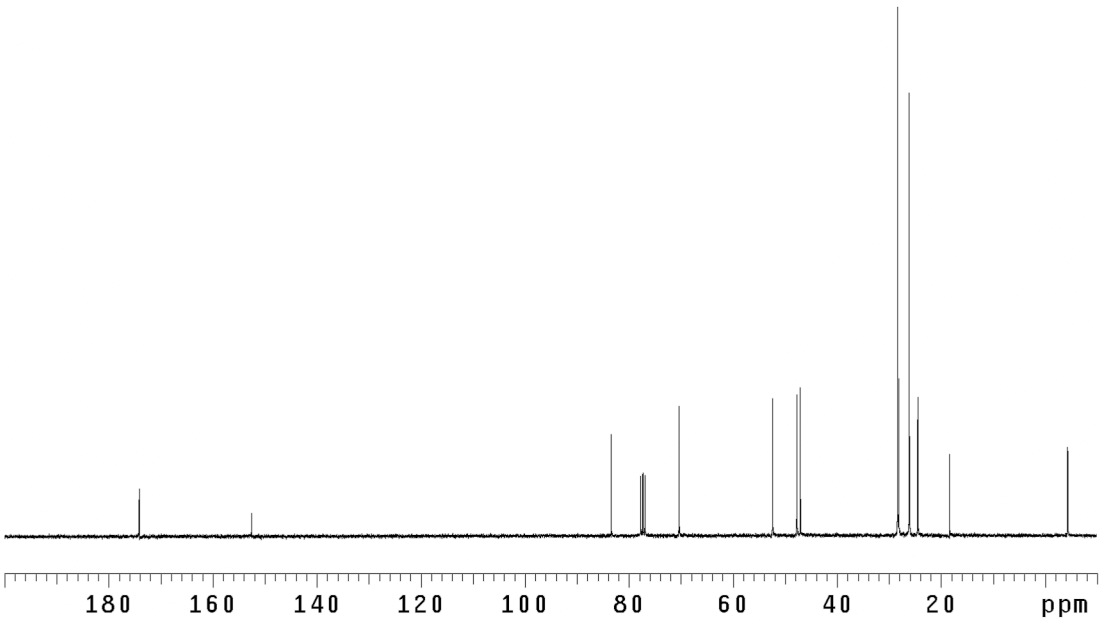

Figure A.90 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **203**.

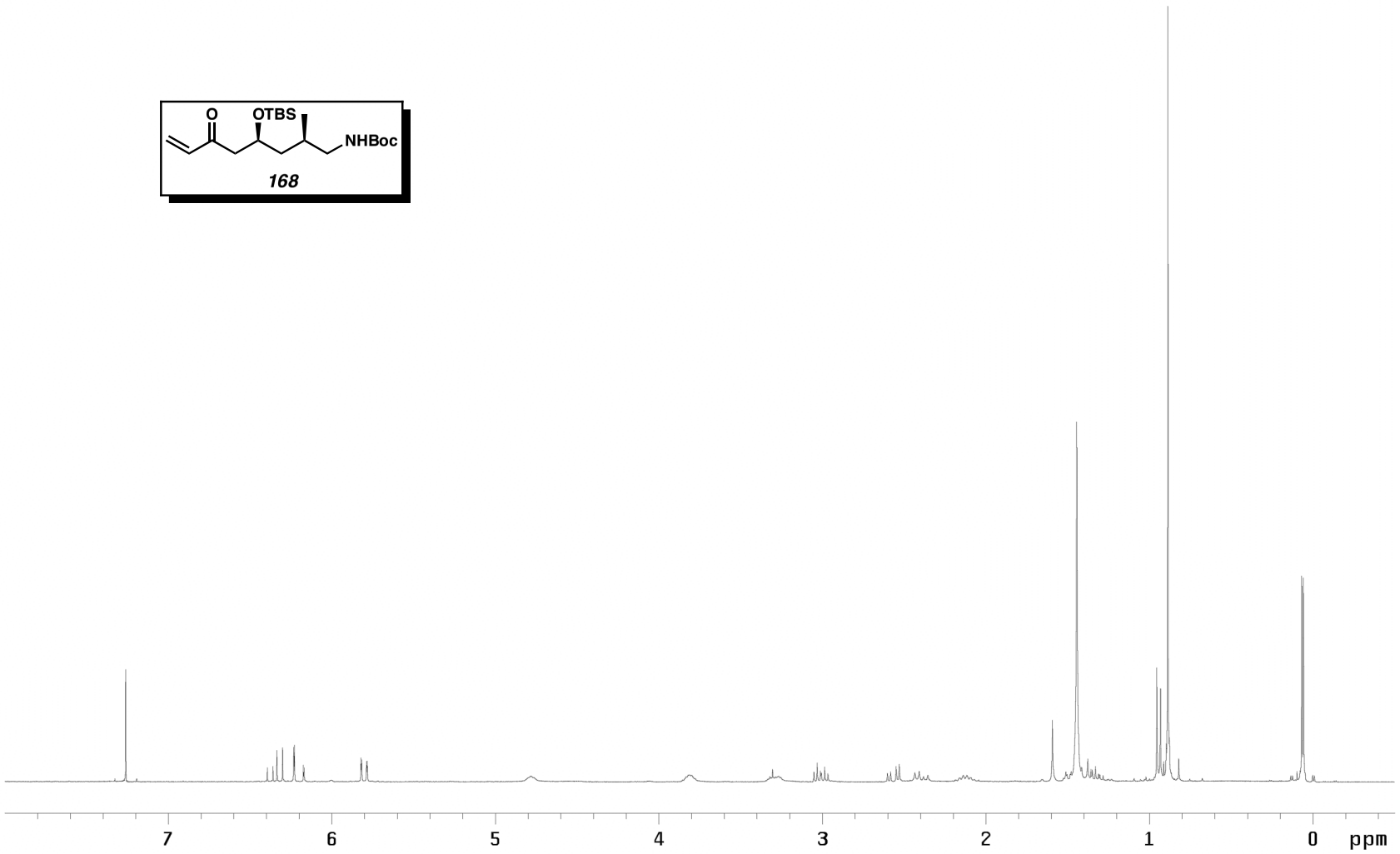

Figure A.91 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **168**.

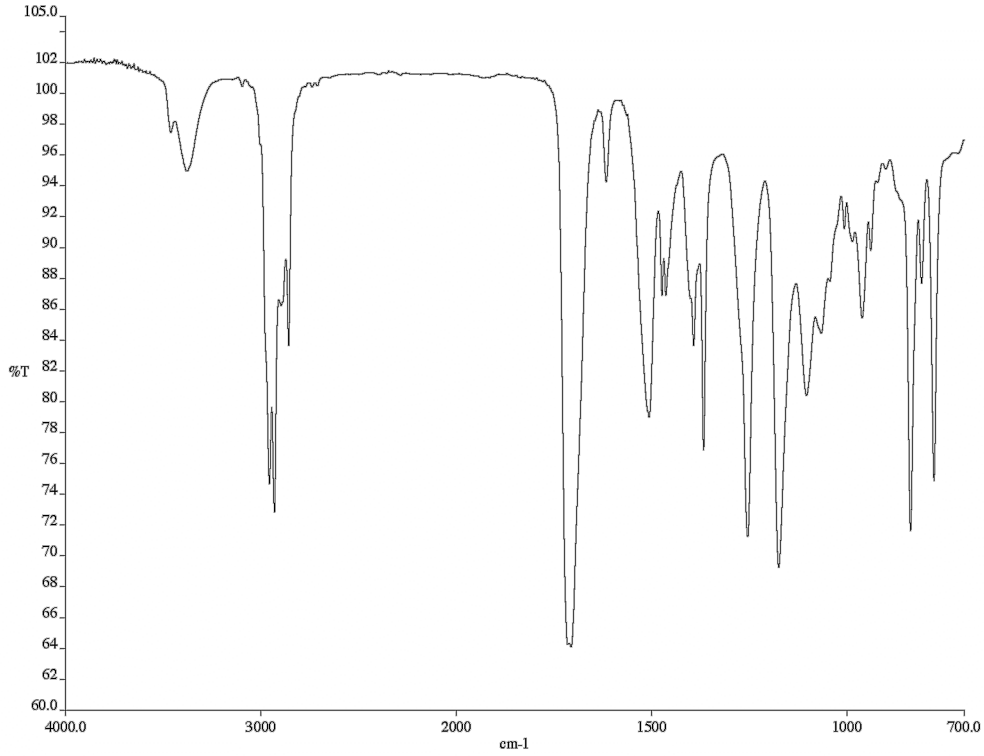

Figure A.92 Infrared spectrum (thin film/NaCl) of compound **168**.

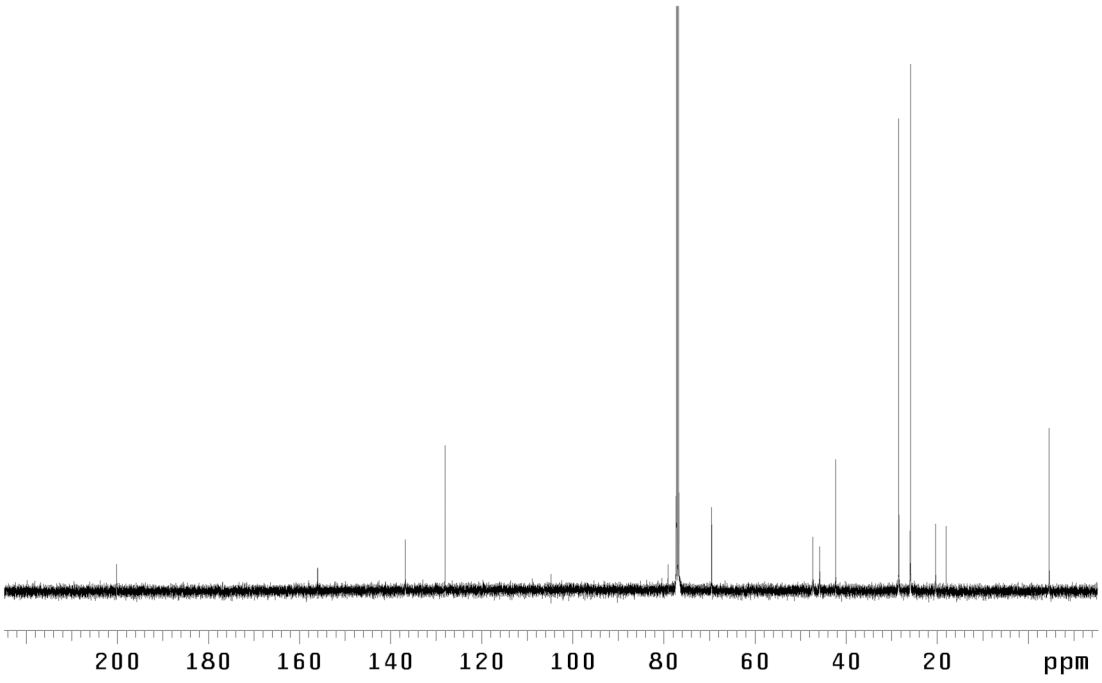

Figure A.93 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **168**.

CALIFORNIA INSTITUTE OF TECHNOLOGY

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Crystal Structure Analysis of:

Lactone **184** (DCB06)

(CCDC 175859)

Contents:

Figure A.94 Representation of Lactone **184**

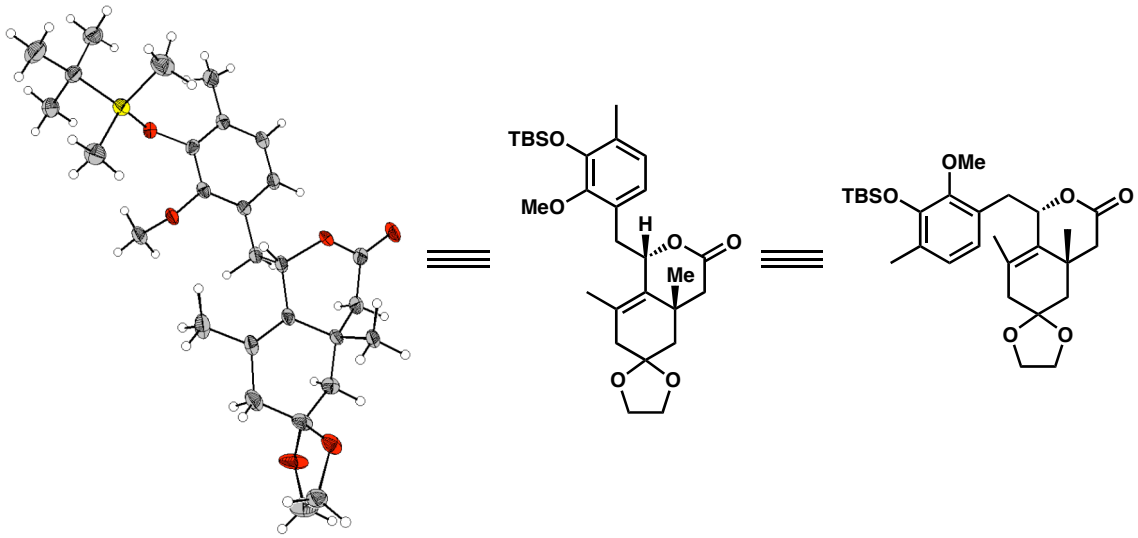

Table 1. Crystal data and structure refinement for DCB06_(CCDC_175859).

Empirical formula	$C_{28}H_{42}O_6Si$
Formula weight	502.71
Crystallization Solvent	Hexanes
Crystal Habit	Block
Crystal size	0.33 x 0.17 x 0.14 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker P4
Wavelength	0.71073 Å MoKα
Data Collection Temperature	96(2) K
θ range for 8201 reflections used in lattice determination	2.79 to 26.49°
Unit cell dimensions	a = 29.220(3) Å b = 6.7215(8) Å β= 90.035(2)° c = 14.4249(17) Å
Volume	2833.0(6) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.179 Mg/m^3
F(000)	1088
Data collection program	Bruker SMART v5.054
θ range for data collection	1.39 to 28.38°
Completeness to θ = 28.38°	93.9 %
Index ranges	$-37 \le h \le 38, -8 \le k \le 8, -19 \le l \le 19$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.22
Reflections collected	38935
Independent reflections	6656 [R_{int}= 0.0985]
Absorption coefficient	0.120 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9829 and 0.9610

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	Bruker SHELXTL
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	Bruker SHELXTL
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	6656 / 0 / 484
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.403
Final R indices [I>2σ(I), 3988 reflections]	R1 = 0.0584, *w*R2 = 0.0804
R indices (all data)	R1 = 0.1062, *w*R2 = 0.0844
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	0.333 and -0.349 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 DCB06_(CCDC_175859). U(eq) the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Si(1)	6142(1)	3545(1)	4087(1)	25(1)
O(1)	6571(1)	5169(2)	4135(1)	23(1)
O(2)	7293(1)	2606(2)	3725(1)	23(1)
O(3)	8211(1)	1108(2)	6169(1)	28(1)
O(4)	8640(1)	2202(2)	7317(1)	44(1)
O(5)	9414(1)	-4435(2)	4438(1)	30(1)
O(6)	9725(1)	-2363(2)	3364(1)	45(1)
C(1)	5665(1)	5006(3)	3527(1)	27(1)
C(2)	5841(1)	5905(4)	2608(2)	35(1)
C(3)	5260(1)	3633(5)	3329(2)	43(1)
C(4)	5503(1)	6694(4)	4154(2)	36(1)
C(5)	6282(1)	1358(4)	3362(2)	34(1)
C(6)	5995(1)	2734(5)	5291(2)	39(1)
C(7)	6962(1)	5171(3)	4672(1)	21(1)
C(8)	6990(1)	6481(3)	5429(1)	24(1)
C(9)	6595(1)	7823(4)	5660(2)	37(1)
C(10)	7397(1)	6526(4)	5928(1)	27(1)
C(11)	7762(1)	5352(3)	5700(1)	26(1)
C(12)	7740(1)	4049(3)	4942(1)	22(1)
C(13)	7334(1)	3973(3)	4446(1)	20(1)
C(14)	7349(1)	3475(4)	2815(1)	32(1)
C(15)	8145(1)	2785(3)	4670(1)	22(1)
C(16)	8169(1)	761(3)	5164(1)	22(1)
C(17)	8623(1)	1598(3)	6522(1)	28(1)
C(18)	9030(1)	1238(4)	5922(2)	26(1)
C(19)	8979(1)	-684(3)	5354(1)	19(1)
C(20)	8948(1)	-2437(4)	6036(1)	24(1)
C(21)	9394(1)	-899(3)	4715(1)	24(1)
C(22)	9349(1)	-2544(3)	4006(1)	28(1)
C(23)	9638(1)	-5668(3)	3767(2)	29(1)
C(24)	9927(1)	-4215(4)	3241(2)	39(1)
C(25)	8901(1)	-2427(4)	3480(2)	33(1)
C(26)	8502(1)	-1441(3)	3960(1)	24(1)
C(27)	8074(1)	-1401(5)	3388(2)	35(1)
C(28)	8545(1)	-548(3)	4787(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for DCB06_(CCDC_175859).

Si(1)-O(1)	1.6629(15)	C(16)-C(28)	1.510(3)
Si(1)-C(5)	1.849(3)	C(16)-H(16)	0.995(16)
Si(1)-C(6)	1.872(2)	C(17)-C(18)	1.490(3)
Si(1)-C(1)	1.887(2)	C(18)-C(19)	1.538(3)
O(1)-C(7)	1.379(2)	C(18)-H(18A)	0.96(2)
O(2)-C(13)	1.392(2)	C(18)-H(18B)	0.96(2)
O(2)-C(14)	1.446(2)	C(19)-C(28)	1.512(3)
O(3)-C(17)	1.350(2)	C(19)-C(21)	1.531(3)
O(3)-C(16)	1.473(2)	C(19)-C(20)	1.538(3)
O(4)-C(17)	1.218(2)	C(20)-H(20A)	0.987(19)
O(5)-C(22)	1.428(2)	C(20)-H(20B)	1.016(18)
O(5)-C(23)	1.433(2)	C(20)-H(20C)	1.003(19)
O(6)-C(24)	1.389(3)	C(21)-C(22)	1.512(3)
O(6)-C(22)	1.443(2)	C(21)-H(21A)	0.99(2)
C(1)-C(4)	1.527(3)	C(21)-H(21B)	0.99(2)
C(1)-C(3)	1.528(3)	C(22)-C(25)	1.516(3)
C(1)-C(2)	1.545(3)	C(23)-C(24)	1.498(3)
C(2)-H(2A)	1.05(2)	C(23)-H(23A)	0.98(2)
C(2)-H(2B)	0.99(2)	C(23)-H(23B)	0.940(19)
C(2)-H(2C)	1.00(2)	C(24)-H(24A)	0.86(3)
C(3)-H(3A)	0.98(2)	C(24)-H(24B)	0.97(3)
C(3)-H(3B)	0.95(2)	C(25)-C(26)	1.509(3)
C(3)-H(3C)	1.04(2)	C(25)-H(25A)	0.99(2)
C(4)-H(4A)	0.99(2)	C(25)-H(25B)	0.98(2)
C(4)-H(4B)	0.99(2)	C(26)-C(28)	1.340(2)
C(4)-H(4C)	0.99(2)	C(26)-C(27)	1.498(3)
C(5)-H(5A)	0.97(2)	C(27)-H(27A)	0.95(2)
C(5)-H(5B)	1.01(2)	C(27)-H(27B)	0.97(2)
C(5)-H(5C)	0.98(2)	C(27)-H(27C)	1.06(2)
C(6)-H(6A)	1.03(3)		
C(6)-H(6B)	0.98(3)	O(1)-Si(1)-C(5)	112.26(10)
C(6)-H(6C)	0.98(2)	O(1)-Si(1)-C(6)	109.05(11)
C(7)-C(13)	1.392(3)	C(5)-Si(1)-C(6)	110.16(13)
C(7)-C(8)	1.405(3)	O(1)-Si(1)-C(1)	103.44(9)
C(8)-C(10)	1.389(3)	C(5)-Si(1)-C(1)	109.60(11)
C(8)-C(9)	1.503(3)	C(6)-Si(1)-C(1)	112.21(11)
C(9)-H(9A)	1.02(2)	C(7)-O(1)-Si(1)	130.34(12)
C(9)-H(9B)	0.92(2)	C(13)-O(2)-C(14)	113.73(17)
C(9)-H(9C)	0.95(2)	C(17)-O(3)-C(16)	118.89(15)
C(10)-C(11)	1.369(3)	C(22)-O(5)-C(23)	106.28(14)
C(10)-H(10)	0.98(2)	C(24)-O(6)-C(22)	109.25(17)
C(11)-C(12)	1.403(3)	C(4)-C(1)-C(3)	108.6(2)
C(11)-H(11)	0.965(19)	C(4)-C(1)-C(2)	108.7(2)
C(12)-C(13)	1.388(3)	C(3)-C(1)-C(2)	109.5(2)
C(12)-C(15)	1.508(3)	C(4)-C(1)-Si(1)	111.25(15)
C(14)-H(14A)	0.99(2)	C(3)-C(1)-Si(1)	109.77(18)
C(14)-H(14B)	1.00(2)	C(2)-C(1)-Si(1)	108.96(15)
C(14)-H(14C)	1.05(2)	C(1)-C(2)-H(2A)	113.2(11)
C(15)-C(16)	1.538(3)	C(1)-C(2)-H(2B)	109.5(13)
C(15)-H(15A)	1.047(18)	H(2A)-C(2)-H(2B)	106.4(17)
C(15)-H(15B)	0.950(18)	C(1)-C(2)-H(2C)	108.6(12)

H(2A)-C(2)-H(2C)	108.2(18)	H(14A)-C(14)-H(14C)	108.8(17)
H(2B)-C(2)-H(2C)	110.9(18)	H(14B)-C(14)-H(14C)	114.6(17)
C(1)-C(3)-H(3A)	110.1(14)	C(12)-C(15)-C(16)	114.35(16)
C(1)-C(3)-H(3B)	110.8(14)	C(12)-C(15)-H(15A)	112.3(10)
H(3A)-C(3)-H(3B)	108.4(19)	C(16)-C(15)-H(15A)	104.7(10)
C(1)-C(3)-H(3C)	113.7(12)	C(12)-C(15)-H(15B)	109.6(11)
H(3A)-C(3)-H(3C)	108.5(18)	C(16)-C(15)-H(15B)	108.4(11)
H(3B)-C(3)-H(3C)	105.0(19)	H(15A)-C(15)-H(15B)	107.1(14)
C(1)-C(4)-H(4A)	109.9(14)	O(3)-C(16)-C(28)	112.77(15)
C(1)-C(4)-H(4B)	112.8(12)	O(3)-C(16)-C(15)	108.69(16)
H(4A)-C(4)-H(4B)	110.7(17)	C(28)-C(16)-C(15)	112.34(16)
C(1)-C(4)-H(4C)	115.2(12)	O(3)-C(16)-H(16)	101.4(9)
H(4A)-C(4)-H(4C)	104.1(17)	C(28)-C(16)-H(16)	113.5(10)
H(4B)-C(4)-H(4C)	103.8(18)	C(15)-C(16)-H(16)	107.4(10)
Si(1)-C(5)-H(5A)	113.1(14)	O(4)-C(17)-O(3)	118.11(19)
Si(1)-C(5)-H(5B)	112.4(13)	O(4)-C(17)-C(18)	124.7(2)
H(5A)-C(5)-H(5B)	108(2)	O(3)-C(17)-C(18)	117.06(17)
Si(1)-C(5)-H(5C)	110.5(14)	C(17)-C(18)-C(19)	111.65(18)
H(5A)-C(5)-H(5C)	106.6(19)	C(17)-C(18)-H(18A)	109.4(12)
H(5B)-C(5)-H(5C)	106.0(18)	C(19)-C(18)-H(18A)	112.2(12)
Si(1)-C(6)-H(6A)	110.3(13)	C(17)-C(18)-H(18B)	111.5(11)
Si(1)-C(6)-H(6B)	114.0(13)	C(19)-C(18)-H(18B)	111.1(12)
H(6A)-C(6)-H(6B)	107(2)	H(18A)-C(18)-H(18B)	100.5(16)
Si(1)-C(6)-H(6C)	107.1(13)	C(28)-C(19)-C(21)	110.20(15)
H(6A)-C(6)-H(6C)	111.0(19)	C(28)-C(19)-C(18)	108.56(17)
H(6B)-C(6)-H(6C)	108(2)	C(21)-C(19)-C(18)	108.89(16)
O(1)-C(7)-C(13)	120.97(17)	C(28)-C(19)-C(20)	110.06(16)
O(1)-C(7)-C(8)	119.01(17)	C(21)-C(19)-C(20)	111.11(17)
C(13)-C(7)-C(8)	119.93(18)	C(18)-C(19)-C(20)	107.94(16)
C(10)-C(8)-C(7)	117.79(19)	C(19)-C(20)-H(20A)	111.0(10)
C(10)-C(8)-C(9)	121.9(2)	C(19)-C(20)-H(20B)	108.3(10)
C(7)-C(8)-C(9)	120.24(19)	H(20A)-C(20)-H(20B)	107.8(14)
C(8)-C(9)-H(9A)	109.2(13)	C(19)-C(20)-H(20C)	110.5(11)
C(8)-C(9)-H(9B)	112.5(13)	H(20A)-C(20)-H(20C)	104.4(15)
H(9A)-C(9)-H(9B)	111.6(18)	H(20B)-C(20)-H(20C)	114.8(14)
C(8)-C(9)-H(9C)	112.0(13)	C(22)-C(21)-C(19)	113.99(17)
H(9A)-C(9)-H(9C)	106.5(18)	C(22)-C(21)-H(21A)	107.4(12)
H(9B)-C(9)-H(9C)	104.8(19)	C(19)-C(21)-H(21A)	112.5(11)
C(11)-C(10)-C(8)	122.0(2)	C(22)-C(21)-H(21B)	107.6(10)
C(11)-C(10)-H(10)	120.2(12)	C(19)-C(21)-H(21B)	110.2(10)
C(8)-C(10)-H(10)	117.8(12)	H(21A)-C(21)-H(21B)	104.7(16)
C(10)-C(11)-C(12)	120.8(2)	O(5)-C(22)-O(6)	104.75(15)
C(10)-C(11)-H(11)	120.4(11)	O(5)-C(22)-C(21)	110.15(16)
C(12)-C(11)-H(11)	118.8(11)	O(6)-C(22)-C(21)	107.79(17)
C(13)-C(12)-C(11)	117.63(19)	O(5)-C(22)-C(25)	112.29(19)
C(13)-C(12)-C(15)	121.07(18)	O(6)-C(22)-C(25)	109.41(17)
C(11)-C(12)-C(15)	121.30(19)	C(21)-C(22)-C(25)	112.09(19)
C(12)-C(13)-C(7)	121.79(18)	O(5)-C(23)-C(24)	102.91(18)
C(12)-C(13)-O(2)	118.78(17)	O(5)-C(23)-H(23A)	110.8(11)
C(7)-C(13)-O(2)	119.39(17)	C(24)-C(23)-H(23A)	113.5(12)
O(2)-C(14)-H(14A)	107.7(11)	O(5)-C(23)-H(23B)	109.3(12)
O(2)-C(14)-H(14B)	103.7(13)	C(24)-C(23)-H(23B)	112.7(12)
H(14A)-C(14)-H(14B)	111.7(17)	H(23A)-C(23)-H(23B)	107.6(17)
O(2)-C(14)-H(14C)	109.9(11)	O(6)-C(24)-C(23)	106.3(2)

O(6)-C(24)-H(24A)	111(2)
C(23)-C(24)-H(24A)	121(2)
O(6)-C(24)-H(24B)	107.4(15)
C(23)-C(24)-H(24B)	109.6(15)
H(24A)-C(24)-H(24B)	101(2)
C(26)-C(25)-C(22)	117.35(18)
C(26)-C(25)-H(25A)	105.2(13)
C(22)-C(25)-H(25A)	103.6(13)
C(26)-C(25)-H(25B)	109.0(12)
C(22)-C(25)-H(25B)	108.3(12)
H(25A)-C(25)-H(25B)	113.4(18)
C(28)-C(26)-C(27)	124.0(2)
C(28)-C(26)-C(25)	122.27(19)
C(27)-C(26)-C(25)	113.50(18)
C(26)-C(27)-H(27A)	111.8(14)
C(26)-C(27)-H(27B)	110.0(13)
H(27A)-C(27)-H(27B)	108.2(19)
C(26)-C(27)-H(27C)	118.2(11)
H(27A)-C(27)-H(27C)	102.5(18)
H(27B)-C(27)-H(27C)	105.5(18)
C(26)-C(28)-C(16)	120.96(18)
C(26)-C(28)-C(19)	122.09(18)
C(16)-C(28)-C(19)	116.83(16)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB06_(CCDC_175859). 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)	252(3)	279(4)	224(3)	4(3)	10(3)	-5(3)
O(1)	223(8)	280(9)	195(7)	-17(6)	-35(6)	7(6)
O(2)	277(8)	286(9)	134(7)	-31(6)	-19(6)	-16(7)
O(3)	292(9)	444(10)	110(7)	15(7)	31(6)	67(7)
O(4)	623(12)	535(12)	148(8)	-121(7)	-86(7)	211(9)
O(5)	483(10)	221(9)	186(7)	34(7)	67(7)	24(7)
O(6)	607(11)	324(10)	426(9)	130(8)	373(8)	165(8)
C(1)	206(12)	327(14)	273(12)	19(10)	-8(10)	-14(10)
C(2)	339(16)	452(18)	257(13)	51(12)	-44(12)	55(14)
C(3)	243(15)	500(19)	547(18)	-31(17)	-58(13)	-29(14)
C(4)	287(15)	436(17)	370(15)	43(13)	11(12)	69(14)
C(5)	344(16)	311(15)	366(15)	-48(12)	17(12)	-74(14)
C(6)	465(18)	396(17)	313(14)	73(13)	77(13)	51(15)
C(7)	190(12)	250(13)	199(11)	50(9)	-14(9)	-7(10)
C(8)	254(12)	283(13)	190(10)	6(10)	-4(9)	29(10)
C(9)	400(17)	389(17)	309(15)	-133(14)	-63(12)	122(14)
C(10)	315(14)	336(14)	170(11)	-59(11)	-37(10)	-4(11)
C(11)	274(14)	329(14)	165(11)	3(10)	-41(10)	-10(11)
C(12)	225(12)	279(13)	143(10)	36(9)	23(9)	1(9)
C(13)	237(12)	217(13)	151(10)	13(9)	9(9)	-59(10)
C(14)	336(16)	453(16)	160(11)	-4(12)	-1(10)	-19(14)
C(15)	208(13)	273(13)	170(11)	13(10)	-15(9)	-33(10)
C(16)	202(12)	323(14)	128(10)	10(9)	-18(9)	-19(10)
C(17)	328(14)	308(14)	207(11)	3(10)	-45(10)	114(11)
C(18)	266(14)	297(15)	202(11)	-55(11)	-68(10)	5(11)
C(19)	191(11)	245(12)	126(10)	-11(9)	3(8)	-18(9)
C(20)	228(14)	356(15)	125(11)	21(11)	-11(10)	15(11)
C(21)	253(13)	218(14)	240(12)	33(10)	54(10)	-15(10)
C(22)	378(14)	250(13)	209(11)	52(10)	128(10)	45(11)
C(23)	364(15)	201(13)	292(13)	-18(11)	14(12)	51(12)
C(24)	351(17)	247(15)	576(18)	-68(13)	127(15)	-24(12)
C(25)	501(16)	330(16)	164(12)	6(12)	1(11)	53(13)
C(26)	318(13)	239(12)	150(10)	21(10)	-44(9)	-16(10)
C(27)	458(17)	310(16)	269(13)	-22(13)	-157(12)	-2(13)
C(28)	208(12)	244(13)	125(10)	42(9)	2(9)	-23(9)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB06_(CCDC_175859).

	x	y	z	U_{iso}
H(2A)	6133(7)	6800(30)	2693(13)	38(6)
H(2B)	5928(7)	4820(40)	2180(15)	49(7)
H(2C)	5594(8)	6750(40)	2334(15)	49(7)
H(3A)	5005(8)	4400(40)	3059(15)	54(8)
H(3B)	5343(7)	2620(40)	2906(15)	47(8)
H(3C)	5141(7)	2890(30)	3909(15)	48(7)
H(4A)	5371(7)	6140(40)	4734(15)	53(7)
H(4B)	5749(7)	7660(30)	4298(13)	29(6)
H(4C)	5256(7)	7520(30)	3897(14)	38(6)
H(5A)	6544(8)	620(40)	3588(15)	55(8)
H(5B)	6018(8)	410(40)	3306(15)	53(7)
H(5C)	6356(8)	1770(40)	2728(16)	53(7)
H(6A)	5864(8)	3910(40)	5662(16)	68(8)
H(6B)	5767(8)	1660(40)	5317(15)	60(8)
H(6C)	6278(8)	2230(30)	5577(15)	50(7)
H(9A)	6319(8)	6970(30)	5851(14)	47(7)
H(9B)	6667(7)	8740(30)	6106(14)	36(7)
H(9C)	6499(7)	8590(30)	5140(16)	48(7)
H(10)	7416(7)	7440(30)	6454(14)	36(6)
H(11)	8045(6)	5450(30)	6044(12)	27(6)
H(14A)	7663(7)	4000(30)	2774(12)	28(6)
H(14B)	7302(7)	2330(40)	2385(15)	49(7)
H(14C)	7118(7)	4650(30)	2727(13)	39(6)
H(15A)	8144(6)	2430(30)	3962(13)	27(5)
H(15B)	8421(6)	3490(30)	4792(11)	16(5)
H(16)	7859(6)	150(20)	5125(10)	8(4)
H(18A)	9081(6)	2380(30)	5535(13)	31(6)
H(18B)	9308(7)	1230(30)	6276(13)	31(6)
H(20A)	8897(6)	-3700(30)	5704(12)	18(5)
H(20B)	9252(6)	-2550(30)	6377(12)	21(5)
H(20C)	8673(7)	-2290(30)	6446(12)	26(5)
H(21A)	9683(7)	-1130(30)	5063(13)	34(6)
H(21B)	9448(6)	360(30)	4373(12)	24(5)
H(23A)	9815(7)	-6710(30)	4072(13)	34(6)
H(23B)	9417(6)	-6290(30)	3393(13)	24(6)
H(24A)	9995(11)	-4420(50)	2670(20)	112(14)
H(24B)	10229(9)	-4160(40)	3514(16)	64(9)
H(25A)	8972(7)	-1560(30)	2948(15)	51(7)
H(25B)	8812(7)	-3770(30)	3298(14)	39(7)
H(27A)	8088(8)	-420(40)	2917(16)	56(8)
H(27B)	8027(7)	-2690(40)	3096(15)	48(7)
H(27C)	7762(7)	-1060(30)	3722(13)	43(7)

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Crystal Structure Analysis of:

Acid **187**•$CHCl_3$ (DCB05)

(CCDC 175588)

Contents:

Figure A.95 Representation of Acid **187•$CHCl_3$**

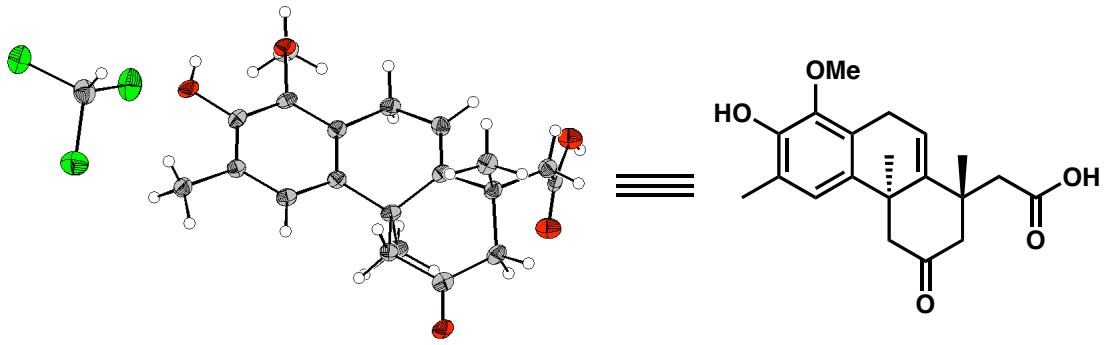

Table 1. Crystal data and structure refinement for DCB05 (CCDC 175588).

Empirical formula	$C_{20}H_{24}O_5 \cdot CHCl_3$
Formula weight	463.76
Crystallization Solvent	Chloroform
Crystal Habit	Fragment
Crystal size	0.22 x 0.15 x 0.15 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 4336 reflections used in lattice determination	2.47 to 25.80°
Unit cell dimensions	a = 11.137(3) Å b = 13.282(3) Å β= 98.762(4)° c = 15.008(4) Å
Volume	2194.3(10) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.404 Mg/m^3
F(000)	968
Data collection program	Bruker SMART v5.054
θ range for data collection	2.06 to 28.36°
Completeness to θ = 28.36°	93.7 %
Index ranges	$-14 \le h \le 14, -17 \le k \le 17, -19 \le l \le 19$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.22
Reflections collected	32070
Independent reflections	5144 [R_{int}= 0.1503]
Absorption coefficient	0.447 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9368 and 0.9072

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	5144 / 0 / 362
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.064
Final R indices [I>2σ(I), 2718 reflections]	R1 = 0.0468, *w*R2 = 0.0744
R indices (all data)	R1 = 0.1218, *w*R2 = 0.0862
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	0.402 and -0.348 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 DCB05 (CCDC 175588). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	2996(2)	1246(2)	424(1)	23(1)
O(2)	3667(2)	289(1)	2011(1)	23(1)
O(3)	10816(2)	767(1)	809(1)	24(1)
O(4)	10340(2)	21(1)	3988(1)	28(1)
O(5)	9289(2)	-1153(1)	4583(1)	27(1)
C(1)	4196(2)	1102(2)	735(2)	20(1)
C(2)	4579(2)	649(2)	1547(2)	19(1)
C(3)	5789(2)	489(2)	1874(2)	19(1)
C(4)	6178(2)	-65(2)	2727(2)	22(1)
C(5)	7352(2)	-608(2)	2734(2)	20(1)
C(6)	8184(2)	-314(2)	2243(2)	18(1)
C(7)	9343(2)	-929(2)	2231(2)	20(1)
C(8)	9093(3)	-1750(2)	1504(2)	25(1)
C(9)	9748(3)	-1452(2)	3131(2)	22(1)
C(10)	9824(2)	-789(2)	3926(2)	21(1)
C(11)	10377(2)	-254(2)	2003(2)	23(1)
C(12)	10036(2)	410(2)	1214(2)	21(1)
C(13)	8738(2)	652(2)	926(2)	19(1)
C(14)	7993(2)	658(2)	1710(2)	18(1)
C(15)	8424(3)	1530(2)	2337(2)	22(1)
C(16)	6654(2)	805(2)	1345(2)	18(1)
C(17)	6244(2)	1260(2)	531(2)	19(1)
C(18)	5037(2)	1421(2)	199(2)	19(1)
C(19)	4615(3)	1892(3)	-688(2)	24(1)
C(20)	3502(3)	888(3)	2774(2)	28(1)
C(21)	1118(3)	3109(2)	603(2)	31(1)
Cl(1)	1475(1)	3147(1)	1778(1)	41(1)
Cl(2)	2089(1)	3910(1)	117(1)	45(1)
Cl(3)	-397(1)	3458(1)	263(1)	39(1)

Table 3. Bond lengths [Å] and angles [°] for DCB05 (CCDC 175588).

O(1)-C(1)	1.361(3)	C(21)-Cl(3)	1.749(3)
O(1)-H(1)	0.72(3)	C(21)-Cl(1)	1.749(3)
O(2)-C(2)	1.401(3)	C(21)-Cl(2)	1.753(3)
O(2)-C(20)	1.428(3)	C(21)-H(21)	0.91(3)
O(3)-C(12)	1.230(3)		
O(4)-C(10)	1.216(3)	C(1)-O(1)-H(1)	107(3)
O(5)-C(10)	1.319(3)	C(2)-O(2)-C(20)	113.6(2)
O(5)-H(5A)	0.97(4)	C(10)-O(5)-H(5A)	111.0(18)
C(1)-C(2)	1.368(4)	O(1)-C(1)-C(2)	121.6(2)
C(1)-C(18)	1.390(3)	O(1)-C(1)-C(18)	118.2(2)
C(2)-C(3)	1.378(3)	C(2)-C(1)-C(18)	120.3(2)
C(3)-C(16)	1.403(3)	C(1)-C(2)-C(3)	122.7(2)
C(3)-C(4)	1.483(4)	C(1)-C(2)-O(2)	116.2(2)
C(4)-C(5)	1.492(4)	C(3)-C(2)-O(2)	120.9(2)
C(4)-H(4A)	0.94(3)	C(2)-C(3)-C(16)	118.1(2)
C(4)-H(4B)	0.96(3)	C(2)-C(3)-C(4)	121.7(2)
C(5)-C(6)	1.328(3)	C(16)-C(3)-C(4)	120.1(2)
C(5)-H(5)	0.97(3)	C(3)-C(4)-C(5)	112.8(2)
C(6)-C(14)	1.516(4)	C(3)-C(4)-H(4A)	108.3(16)
C(6)-C(7)	1.529(3)	C(5)-C(4)-H(4A)	109.2(15)
C(7)-C(9)	1.525(4)	C(3)-C(4)-H(4B)	108.8(16)
C(7)-C(8)	1.538(4)	C(5)-C(4)-H(4B)	107.1(16)
C(7)-C(11)	1.539(3)	H(4A)-C(4)-H(4B)	111(2)
C(8)-H(8A)	0.95(3)	C(6)-C(5)-C(4)	122.6(3)
C(8)-H(8B)	1.02(3)	C(6)-C(5)-H(5)	119.0(14)
C(8)-H(8C)	1.03(3)	C(4)-C(5)-H(5)	118.4(14)
C(9)-C(10)	1.475(4)	C(5)-C(6)-C(14)	119.3(2)
C(9)-H(9A)	0.92(3)	C(5)-C(6)-C(7)	120.7(2)
C(9)-H(9B)	0.93(2)	C(14)-C(6)-C(7)	119.97(19)
C(11)-C(12)	1.478(4)	C(9)-C(7)-C(6)	111.69(19)
C(11)-H(11A)	0.99(3)	C(9)-C(7)-C(8)	107.6(2)
C(11)-H(11B)	1.00(3)	C(6)-C(7)-C(8)	109.0(2)
C(12)-C(13)	1.480(4)	C(9)-C(7)-C(11)	109.4(2)
C(13)-C(14)	1.541(3)	C(6)-C(7)-C(11)	110.7(2)
C(13)-H(13A)	0.94(2)	C(8)-C(7)-C(11)	108.4(2)
C(13)-H(13B)	1.00(2)	C(7)-C(8)-H(8A)	108.3(15)
C(14)-C(16)	1.520(4)	C(7)-C(8)-H(8B)	107.9(16)
C(14)-C(15)	1.523(4)	H(8A)-C(8)-H(8B)	109(2)
C(15)-H(15A)	1.00(2)	C(7)-C(8)-H(8C)	113.1(14)
C(15)-H(15B)	1.00(2)	H(8A)-C(8)-H(8C)	106(2)
C(15)-H(15C)	1.01(3)	H(8B)-C(8)-H(8C)	113(2)
C(16)-C(17)	1.378(4)	C(10)-C(9)-C(7)	114.7(2)
C(17)-C(18)	1.377(4)	C(10)-C(9)-H(9A)	111.5(15)
C(17)-H(17)	0.97(2)	C(7)-C(9)-H(9A)	114.9(16)
C(18)-C(19)	1.481(4)	C(10)-C(9)-H(9B)	105.6(15)
C(19)-H(19A)	0.94(3)	C(7)-C(9)-H(9B)	111.0(15)
C(19)-H(19B)	0.92(3)	H(9A)-C(9)-H(9B)	97(2)
C(19)-H(19C)	1.03(2)	O(4)-C(10)-O(5)	122.0(2)
C(20)-H(20A)	1.00(3)	O(4)-C(10)-C(9)	123.9(2)
C(20)-H(20B)	0.95(2)	O(5)-C(10)-C(9)	114.2(2)
C(20)-H(20C)	0.99(3)	C(12)-C(11)-C(7)	114.6(2)

C(12)-C(11)-H(11A)	106.0(16)
C(7)-C(11)-H(11A)	112.5(15)
C(12)-C(11)-H(11B)	113.7(14)
C(7)-C(11)-H(11B)	108.6(15)
H(11A)-C(11)-H(11B)	101(2)
O(3)-C(12)-C(11)	120.7(2)
O(3)-C(12)-C(13)	120.3(2)
C(11)-C(12)-C(13)	119.0(2)
C(12)-C(13)-C(14)	113.2(2)
C(12)-C(13)-H(13A)	109.8(13)
C(14)-C(13)-H(13A)	108.8(12)
C(12)-C(13)-H(13B)	110.1(13)
C(14)-C(13)-H(13B)	109.3(12)
H(13A)-C(13)-H(13B)	105.3(19)
C(6)-C(14)-C(16)	110.5(2)
C(6)-C(14)-C(15)	108.5(2)
C(16)-C(14)-C(15)	108.9(2)
C(6)-C(14)-C(13)	110.5(2)
C(16)-C(14)-C(13)	109.8(2)
C(15)-C(14)-C(13)	108.6(2)
C(14)-C(15)-H(15A)	109.5(14)
C(14)-C(15)-H(15B)	111.6(14)
H(15A)-C(15)-H(15B)	107.9(18)
C(14)-C(15)-H(15C)	116.1(16)
H(15A)-C(15)-H(15C)	111(2)
H(15B)-C(15)-H(15C)	100(2)
C(17)-C(16)-C(3)	118.0(2)
C(17)-C(16)-C(14)	123.4(2)
C(3)-C(16)-C(14)	118.6(2)
C(18)-C(17)-C(16)	124.3(2)
C(18)-C(17)-H(17)	117.2(15)
C(16)-C(17)-H(17)	118.6(15)
C(17)-C(18)-C(1)	116.7(2)
C(17)-C(18)-C(19)	123.3(2)
C(1)-C(18)-C(19)	120.0(2)
C(18)-C(19)-H(19A)	114.1(17)
C(18)-C(19)-H(19B)	111.4(17)
H(19A)-C(19)-H(19B)	109(2)
C(18)-C(19)-H(19C)	109.9(15)
H(19A)-C(19)-H(19C)	104(2)
H(19B)-C(19)-H(19C)	107(2)
O(2)-C(20)-H(20A)	111.9(14)
O(2)-C(20)-H(20B)	104.7(14)
H(20A)-C(20)-H(20B)	108(2)
O(2)-C(20)-H(20C)	112.1(16)
H(20A)-C(20)-H(20C)	108(2)
H(20B)-C(20)-H(20C)	111(2)
Cl(3)-C(21)-Cl(1)	110.32(16)
Cl(3)-C(21)-Cl(2)	110.32(17)
Cl(1)-C(21)-Cl(2)	109.98(17)
Cl(3)-C(21)-H(21)	109.2(19)
Cl(1)-C(21)-H(21)	105.5(19)
Cl(2)-C(21)-H(21)	111.4(16)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB05 (CCDC 175588). 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)	149(10)	334(13)	235(11)	33(9)	80(9)	-25(9)
O(2)	182(10)	311(11)	210(10)	7(9)	113(8)	-10(8)
O(3)	154(10)	348(11)	253(11)	24(9)	102(8)	-2(8)
O(4)	324(11)	318(12)	214(10)	-30(9)	98(9)	-60(9)
O(5)	325(11)	325(12)	182(11)	-13(10)	95(9)	-42(9)
C(1)	142(14)	230(15)	221(15)	-30(12)	23(11)	8(12)
C(2)	142(14)	248(15)	205(15)	-29(12)	82(11)	-27(11)
C(3)	174(15)	228(15)	167(14)	-8(12)	58(11)	4(11)
C(4)	183(15)	279(17)	222(16)	16(14)	92(12)	-9(13)
C(5)	223(15)	228(16)	165(15)	17(13)	41(12)	4(12)
C(6)	155(14)	222(15)	159(14)	-23(11)	31(11)	-4(11)
C(7)	164(14)	230(15)	198(15)	8(12)	36(11)	1(11)
C(8)	240(17)	298(18)	208(16)	-20(14)	47(13)	18(14)
C(9)	190(16)	242(16)	242(16)	20(13)	49(12)	24(14)
C(10)	148(14)	267(17)	208(15)	27(13)	-21(12)	41(12)
C(11)	150(15)	294(17)	243(16)	-13(14)	53(12)	15(13)
C(12)	216(15)	196(15)	230(16)	-72(12)	56(12)	-7(12)
C(13)	175(15)	235(16)	172(15)	24(13)	71(12)	9(12)
C(14)	158(14)	222(15)	186(14)	-19(12)	92(11)	-10(11)
C(15)	192(15)	254(16)	217(16)	-18(13)	85(12)	-7(13)
C(16)	169(14)	203(14)	180(14)	-3(11)	79(11)	13(11)
C(17)	176(14)	229(15)	194(15)	-22(12)	92(12)	-2(12)
C(18)	184(14)	216(15)	191(14)	-5(12)	67(11)	22(11)
C(19)	181(16)	298(18)	228(16)	44(15)	29(13)	-2(14)
C(20)	237(18)	400(20)	250(17)	-48(15)	140(15)	-19(15)
C(21)	345(18)	274(18)	296(17)	-15(15)	10(14)	23(15)
Cl(1)	443(5)	519(5)	245(4)	-55(4)	17(3)	122(4)
Cl(2)	415(5)	447(5)	491(5)	63(4)	117(4)	-38(4)
Cl(3)	331(4)	475(5)	339(4)	-10(4)	21(3)	69(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB05 (CCDC 175588).

	x	y	z	U_{iso}
H(1)	2670(30)	960(20)	710(20)	37(11)
H(4A)	6260(20)	400(20)	3203(17)	23(7)
H(4B)	5570(20)	-560(20)	2799(17)	29(7)
H(5)	7500(20)	-1210(20)	3098(17)	29(8)
H(5A)	9470(30)	-750(30)	5120(20)	60(10)
H(8A)	8480(20)	-2180(20)	1656(18)	30(8)
H(8B)	9870(30)	-2160(20)	1514(18)	40(8)
H(8C)	8770(20)	-1467(19)	875(19)	30(7)
H(9A)	9340(20)	-2040(20)	3214(16)	19(7)
H(9B)	10520(20)	-1729(19)	3152(16)	22(7)
H(11A)	10710(20)	190(20)	2508(19)	39(8)
H(11B)	11100(20)	-680(20)	1959(16)	29(7)
H(13A)	8666(18)	1287(17)	642(14)	3(6)
H(13B)	8370(20)	165(18)	456(16)	16(7)
H(15A)	8294(19)	2176(18)	1999(15)	11(6)
H(15B)	7970(20)	1565(18)	2856(17)	22(7)
H(15C)	9280(30)	1480(20)	2670(19)	39(8)
H(17)	6830(20)	1466(18)	154(16)	23(7)
H(19A)	4240(20)	2520(20)	-652(18)	31(8)
H(19B)	4110(20)	1470(20)	-1056(19)	33(8)
H(19C)	5350(20)	2042(19)	-1011(17)	32(7)
H(20A)	4260(30)	910(20)	3234(18)	29(8)
H(20B)	2880(20)	553(17)	3031(15)	14(6)
H(20C)	3270(20)	1590(20)	2602(19)	38(9)
H(21)	1220(20)	2450(20)	447(19)	39(9)

Table 6. Hydrogen bonds for DCB05 (CCDC 175588) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(3)#1	0.72(3)	2.10(3)	2.656(2)	135(3)
O(1)-H(1)...O(2)	0.72(3)	2.28(3)	2.703(3)	119(3)
O(5)-H(5A)...O(4)#2	0.97(4)	1.64(4)	2.600(3)	175(3)

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

CALIFORNIA INSTITUTE OF TECHNOLOGY

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X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Diketone **196** (DCB11)

(CCDC 201187)

Contents:

Figure A.96 Representation of Diketone **196**.

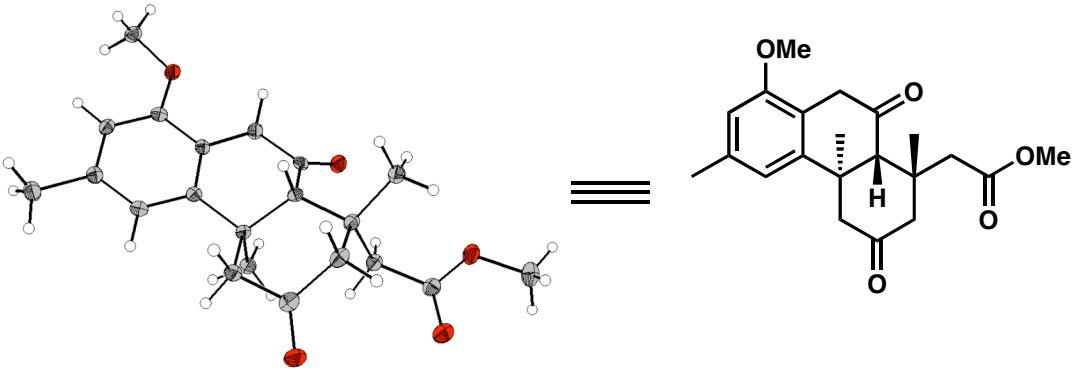

Table 1. Crystal data and structure refinement for DCB11 (CCDC 201187).

Empirical formula	$C_{21}H_{26}O_5$
Formula weight	358.42
Crystallization Solvent	Acetone/heptane
Crystal Habit	Fragment
Crystal size	0.26 x 0.22 x 0.17 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 11980 reflections used in lattice determination	2.28 to 28.32°
Unit cell dimensions	a = 9.0211(6) Å b = 11.3617(7) Å β= 97.5510(10)° c = 17.9596(12) Å
Volume	1824.8(2) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.305 Mg/m^3
F(000)	768
Data collection program	Bruker SMART v5.054
θ range for data collection	2.13 to 28.32°
Completeness to θ = 28.32°	93.0 %
Index ranges	$-11 \le h \le 11$, $-14 \le k \le 14$, $-23 \le l \le 23$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.022
Reflections collected	25862
Independent reflections	4226 [R_{int}= 0.0517]
Absorption coefficient	0.092 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9845 and 0.9764

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	4226 / 0 / 339
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	2.153
Final R indices [I>2σ(I), 3426 reflections]	R1 = 0.0404, *w*R2 = 0.0704
R indices (all data)	R1 = 0.0511, *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
Largest diff. peak and hole	0.326 and -0.254 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 DCB11 (CCDC 201187). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	7731(1)	4507(1)	529(1)	19(1)
O(2)	4749(1)	8200(1)	472(1)	21(1)
O(3)	9216(1)	11345(1)	2166(1)	25(1)
O(4)	3377(1)	11397(1)	1288(1)	24(1)
O(5)	5418(1)	12446(1)	1710(1)	29(1)
C(1)	10631(1)	7158(1)	1330(1)	16(1)
C(2)	11220(1)	6047(1)	1256(1)	16(1)
C(3)	12869(2)	5814(1)	1455(1)	23(1)
C(4)	10262(1)	5133(1)	986(1)	16(1)
C(5)	8757(1)	5346(1)	789(1)	15(1)
C(6)	8217(2)	3305(1)	562(1)	20(1)
C(7)	8162(1)	6478(1)	851(1)	14(1)
C(8)	6510(1)	6650(1)	623(1)	18(1)
C(9)	5914(1)	7834(1)	811(1)	15(1)
C(10)	6851(1)	8484(1)	1442(1)	14(1)
C(11)	6098(1)	9579(1)	1751(1)	15(1)
C(12)	4682(1)	9175(1)	2071(1)	19(1)
C(13)	5708(1)	10548(1)	1148(1)	17(1)
C(14)	4865(1)	11569(1)	1417(1)	18(1)
C(15)	2478(2)	12330(1)	1540(1)	31(1)
C(16)	7204(1)	10084(1)	2400(1)	18(1)
C(17)	8678(1)	10359(1)	2139(1)	18(1)
C(18)	9450(1)	9319(1)	1844(1)	18(1)
C(19)	8466(1)	8626(1)	1220(1)	14(1)
C(20)	8500(2)	9259(1)	463(1)	19(1)
C(21)	9104(1)	7387(1)	1138(1)	14(1)

Table 3. Bond lengths [Å] and angles [°] for DCB11 (CCDC 201187).

O(1)-C(5)	1.3673(13)	C(20)-H(20A)	0.996(14)
O(1)-C(6)	1.4332(14)	C(20)-H(20B)	0.987(13)
O(2)-C(9)	1.2162(13)	C(20)-H(20C)	0.985(13)
O(3)-C(17)	1.2186(13)		
O(4)-C(14)	1.3457(14)	C(5)-O(1)-C(6)	117.41(9)
O(4)-C(15)	1.4438(16)	C(14)-O(4)-C(15)	115.32(10)
O(5)-C(14)	1.2036(14)	C(2)-C(1)-C(21)	121.54(11)
C(1)-C(2)	1.3828(16)	C(2)-C(1)-H(1)	118.7(7)
C(1)-C(21)	1.3997(16)	C(21)-C(1)-H(1)	119.8(7)
C(1)-H(1)	1.006(12)	C(1)-C(2)-C(4)	118.97(11)
C(2)-C(4)	1.3963(16)	C(1)-C(2)-C(3)	121.17(11)
C(2)-C(3)	1.5069(17)	C(4)-C(2)-C(3)	119.86(11)
C(3)-H(3A)	0.967(14)	C(2)-C(3)-H(3A)	109.9(8)
C(3)-H(3B)	0.963(16)	C(2)-C(3)-H(3B)	111.4(9)
C(3)-H(3C)	0.980(15)	H(3A)-C(3)-H(3B)	109.7(12)
C(4)-C(5)	1.3783(16)	C(2)-C(3)-H(3C)	111.9(9)
C(4)-H(4)	0.958(11)	H(3A)-C(3)-H(3C)	108.7(12)
C(5)-C(7)	1.4037(15)	H(3B)-C(3)-H(3C)	105.1(12)
C(6)-H(6A)	0.978(12)	C(5)-C(4)-C(2)	120.19(11)
C(6)-H(6B)	0.973(12)	C(5)-C(4)-H(4)	119.0(7)
C(6)-H(6C)	0.999(13)	C(2)-C(4)-H(4)	120.8(7)
C(7)-C(21)	1.3926(16)	O(1)-C(5)-C(4)	124.44(10)
C(7)-C(8)	1.5051(16)	O(1)-C(5)-C(7)	114.64(10)
C(8)-C(9)	1.5034(16)	C(4)-C(5)-C(7)	120.92(11)
C(8)-H(8A)	0.993(13)	O(1)-C(6)-H(6A)	105.1(7)
C(8)-H(8B)	0.996(13)	O(1)-C(6)-H(6B)	110.4(7)
C(9)-C(10)	1.5144(16)	H(6A)-C(6)-H(6B)	110.7(10)
C(10)-C(11)	1.5546(16)	O(1)-C(6)-H(6C)	111.8(7)
C(10)-C(19)	1.5679(16)	H(6A)-C(6)-H(6C)	110.0(10)
C(10)-H(10)	0.982(11)	H(6B)-C(6)-H(6C)	108.8(10)
C(11)-C(12)	1.5377(16)	C(5)-C(7)-C(21)	119.20(11)
C(11)-C(13)	1.5525(16)	C(5)-C(7)-C(8)	118.20(10)
C(11)-C(16)	1.5420(16)	C(21)-C(7)-C(8)	122.58(10)
C(12)-H(12A)	0.986(12)	C(7)-C(8)-C(9)	115.21(10)
C(12)-H(12B)	1.025(12)	C(7)-C(8)-H(8A)	112.9(8)
C(12)-H(12C)	0.993(12)	C(9)-C(8)-H(8A)	106.4(7)
C(13)-C(14)	1.5018(16)	C(7)-C(8)-H(8B)	109.6(8)
C(13)-H(13A)	0.986(12)	C(9)-C(8)-H(8B)	105.9(8)
C(13)-H(13B)	0.957(11)	H(8A)-C(8)-H(8B)	106.3(11)
C(15)-H(15A)	0.969(14)	O(2)-C(9)-C(10)	124.61(11)
C(15)-H(15B)	0.980(15)	O(2)-C(9)-C(8)	120.35(11)
C(15)-H(15C)	0.989(15)	C(10)-C(9)-C(8)	115.03(10)
C(16)-C(17)	1.4999(17)	C(9)-C(10)-C(11)	115.52(10)
C(16)-H(16A)	0.980(12)	C(9)-C(10)-C(19)	107.82(9)
C(16)-H(16B)	0.982(12)	C(11)-C(10)-C(19)	118.21(9)
C(17)-C(18)	1.5039(17)	C(9)-C(10)-H(10)	104.1(6)
C(18)-C(19)	1.5492(16)	C(11)-C(10)-H(10)	105.7(6)
C(18)-H(18A)	0.966(12)	C(19)-C(10)-H(10)	103.8(6)
C(18)-H(18B)	0.996(12)	C(12)-C(11)-C(10)	108.53(9)
C(19)-C(21)	1.5357(15)	C(12)-C(11)-C(13)	110.46(10)
C(19)-C(20)	1.5424(16)	C(10)-C(11)-C(13)	112.81(9)

C(12)-C(11)-C(16)	108.40(10)
C(10)-C(11)-C(16)	107.36(9)
C(13)-C(11)-C(16)	109.15(10)
C(11)-C(12)-H(12A)	109.5(7)
C(11)-C(12)-H(12B)	110.2(7)
H(12A)-C(12)-H(12B)	109.2(9)
C(11)-C(12)-H(12C)	109.5(7)
H(12A)-C(12)-H(12C)	108.7(10)
H(12B)-C(12)-H(12C)	109.7(9)
C(14)-C(13)-C(11)	113.64(10)
C(14)-C(13)-H(13A)	104.7(7)
C(11)-C(13)-H(13A)	110.9(7)
C(14)-C(13)-H(13B)	108.1(7)
C(11)-C(13)-H(13B)	109.4(7)
H(13A)-C(13)-H(13B)	110.0(10)
O(5)-C(14)-O(4)	122.87(11)
O(5)-C(14)-C(13)	125.55(12)
O(4)-C(14)-C(13)	111.58(10)
O(4)-C(15)-H(15A)	107.3(8)
O(4)-C(15)-H(15B)	109.3(9)
H(15A)-C(15)-H(15B)	110.0(12)
O(4)-C(15)-H(15C)	110.6(8)
H(15A)-C(15)-H(15C)	111.5(12)
H(15B)-C(15)-H(15C)	108.1(12)
C(17)-C(16)-C(11)	110.67(10)
C(17)-C(16)-H(16A)	108.5(7)
C(11)-C(16)-H(16A)	111.5(7)
C(17)-C(16)-H(16B)	108.4(7)
C(11)-C(16)-H(16B)	107.5(7)
H(16A)-C(16)-H(16B)	110.2(9)
O(3)-C(17)-C(18)	122.40(11)
O(3)-C(17)-C(16)	122.92(11)
C(18)-C(17)-C(16)	114.67(11)
C(17)-C(18)-C(19)	113.93(10)
C(17)-C(18)-H(18A)	109.0(7)
C(19)-C(18)-H(18A)	110.2(7)
C(17)-C(18)-H(18B)	105.8(7)
C(19)-C(18)-H(18B)	109.9(7)
H(18A)-C(18)-H(18B)	107.8(9)
C(21)-C(19)-C(20)	106.90(9)
C(21)-C(19)-C(18)	110.43(9)
C(20)-C(19)-C(18)	108.92(10)
C(21)-C(19)-C(10)	107.65(9)
C(20)-C(19)-C(10)	113.48(10)
C(18)-C(19)-C(10)	109.42(9)
C(19)-C(20)-H(20A)	108.7(7)
C(19)-C(20)-H(20B)	111.1(7)
H(20A)-C(20)-H(20B)	107.8(10)
C(19)-C(20)-H(20C)	112.4(7)
H(20A)-C(20)-H(20C)	107.0(10)
H(20B)-C(20)-H(20C)	109.7(10)
C(1)-C(21)-C(7)	119.14(11)
C(1)-C(21)-C(19)	121.02(10)
C(7)-C(21)-C(19)	119.82(10)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB11 (CCDC 201187). 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)	179(5)	129(4)	246(5)	-34(4)	5(4)	-8(3)
O(2)	175(5)	198(5)	252(5)	-10(4)	-23(4)	19(4)
O(3)	239(5)	164(5)	340(5)	-50(4)	-12(4)	-28(4)
O(4)	181(5)	202(5)	316(5)	-22(4)	1(4)	57(4)
O(5)	270(5)	197(5)	396(6)	-85(4)	44(4)	-6(4)
C(1)	170(6)	162(6)	152(6)	-5(5)	26(5)	-28(5)
C(2)	160(6)	188(7)	140(6)	12(5)	29(5)	5(5)
C(3)	165(7)	220(8)	302(8)	-28(7)	7(6)	13(6)
C(4)	189(7)	140(6)	158(6)	10(5)	43(5)	31(5)
C(5)	176(6)	159(6)	118(6)	-7(5)	29(5)	-31(5)
C(6)	223(8)	142(7)	237(7)	-9(6)	13(6)	-2(6)
C(7)	145(6)	157(6)	123(6)	7(5)	31(5)	4(5)
C(8)	162(7)	168(7)	209(7)	-33(6)	8(5)	0(5)
C(9)	142(6)	161(6)	161(6)	30(5)	48(5)	-18(5)
C(10)	151(6)	128(6)	143(6)	26(5)	21(5)	7(5)
C(11)	173(6)	128(6)	153(6)	1(5)	32(5)	14(5)
C(12)	202(7)	167(7)	216(7)	13(6)	73(6)	27(6)
C(13)	185(7)	159(7)	171(7)	4(5)	29(5)	13(5)
C(14)	209(7)	164(7)	163(6)	30(5)	19(5)	19(5)
C(15)	218(8)	287(9)	419(10)	-13(7)	64(7)	94(7)
C(16)	241(7)	145(7)	153(7)	-7(5)	26(5)	29(5)
C(17)	203(7)	172(7)	130(6)	-11(5)	-53(5)	9(5)
C(18)	158(7)	159(7)	206(7)	-1(5)	4(5)	-17(5)
C(19)	139(6)	125(6)	166(6)	0(5)	15(5)	-6(5)
C(20)	196(7)	182(7)	201(7)	18(5)	58(6)	0(6)
C(21)	162(6)	150(6)	114(6)	8(5)	37(5)	3(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB11 (CCDC 201187).

	x	y	z	U_{iso}
H(1)	11324(13)	7813(10)	1530(6)	16(3)
H(3A)	13385(15)	6538(12)	1604(7)	35(4)
H(3B)	13289(17)	5469(13)	1039(9)	50(5)
H(3C)	13068(16)	5243(13)	1865(9)	48(5)
H(4)	10635(12)	4351(10)	941(6)	13(3)
H(6A)	7338(14)	2849(10)	357(7)	21(3)
H(6B)	8556(13)	3077(10)	1078(7)	18(3)
H(6C)	9048(14)	3171(10)	254(7)	19(3)
H(8A)	6202(14)	6529(11)	77(8)	29(4)
H(8B)	5944(15)	6062(11)	886(7)	33(4)
H(10)	6980(11)	7914(9)	1856(6)	9(3)
H(12A)	4310(12)	9821(10)	2363(6)	17(3)
H(12B)	3867(14)	8942(10)	1643(7)	22(3)
H(12C)	4927(13)	8491(11)	2410(7)	20(3)
H(13A)	6623(14)	10898(10)	997(6)	18(3)
H(13B)	5112(12)	10214(10)	719(6)	12(3)
H(15A)	1440(17)	12099(12)	1419(8)	38(4)
H(15B)	2664(16)	13060(13)	1277(8)	43(4)
H(15C)	2744(16)	12466(12)	2086(9)	44(4)
H(16A)	6816(13)	10804(10)	2604(6)	16(3)
H(16B)	7366(13)	9478(10)	2792(7)	18(3)
H(18A)	10357(14)	9583(10)	1663(6)	16(3)
H(18B)	9741(13)	8799(10)	2285(7)	22(3)
H(20A)	9509(15)	9153(10)	304(7)	26(3)
H(20B)	7759(14)	8917(10)	69(7)	22(3)
H(20C)	8331(13)	10112(11)	498(6)	21(3)