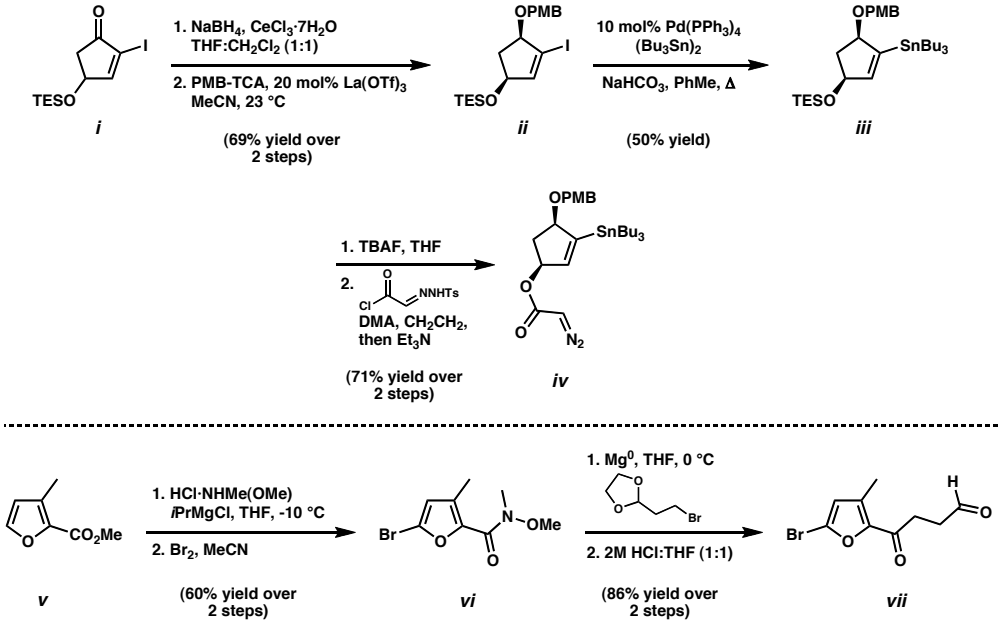
Appendix 2.1

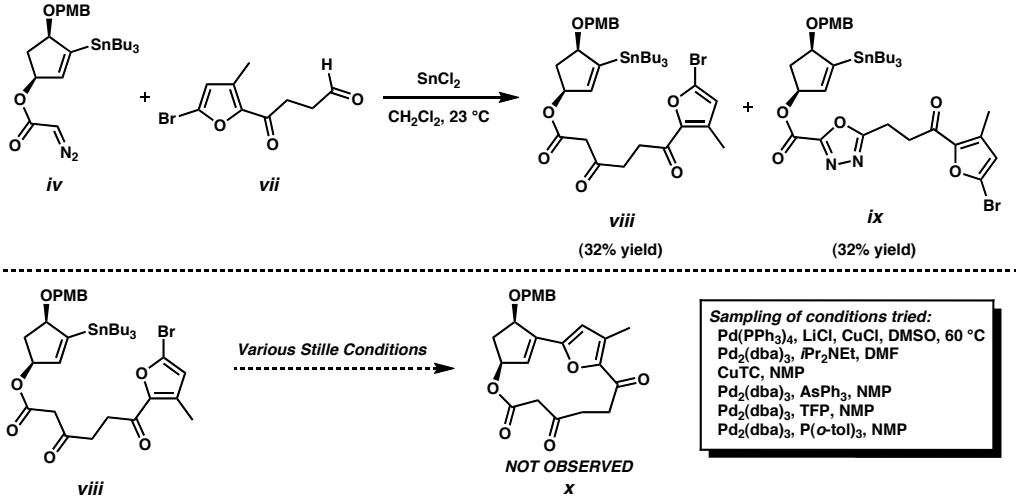
Failed Synthetic Approaches Toward a Macrocyclic Intermediate

(All Characterization Data Obtained for Compounds **i–xx** in Appendix 2.1 can be found in "JHP Characterized Compounds" binder)

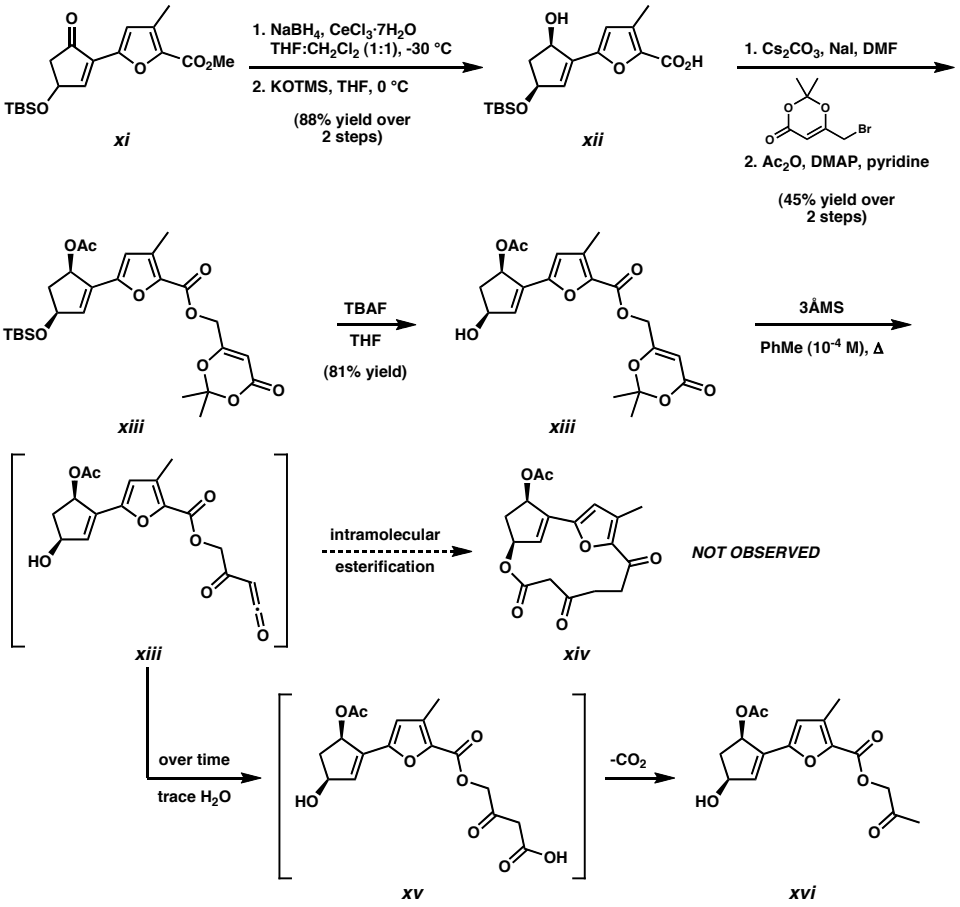
Scheme A2.1.1 Stille coupling route: formation of vinyl halide and vinyl stannane.



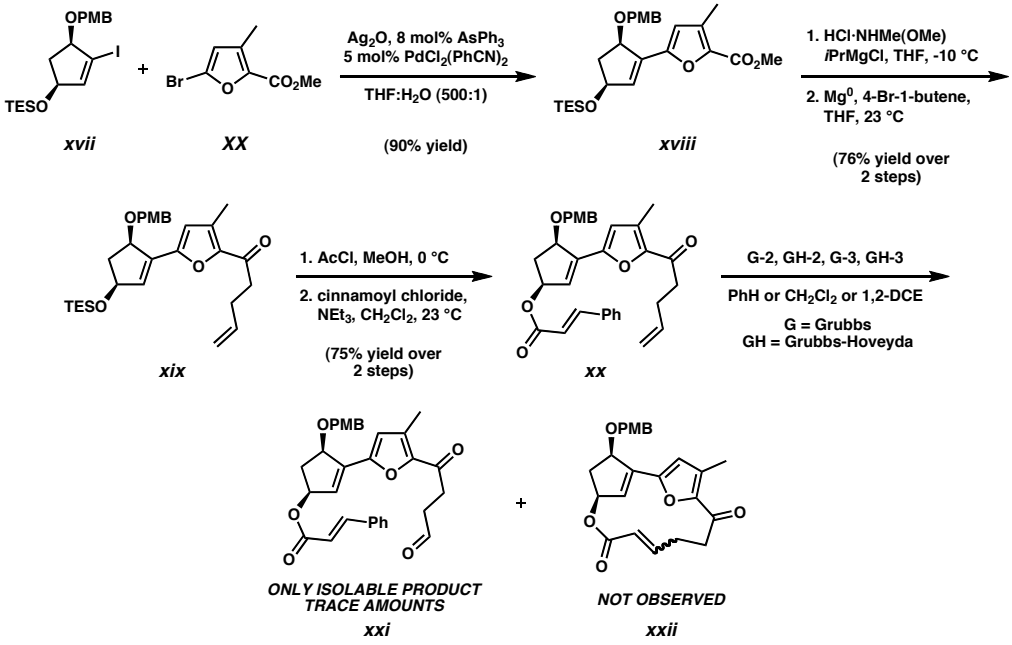
Scheme A2.1.2 Stille coupling route: Attempts at macrocyclization.



Scheme A2.1.3 Intramolecular esterification route.



Scheme A2.1.4 Ring closing metathesis route.



Appendix 2.2

Spectra of Compounds Relevant to Chapter Two

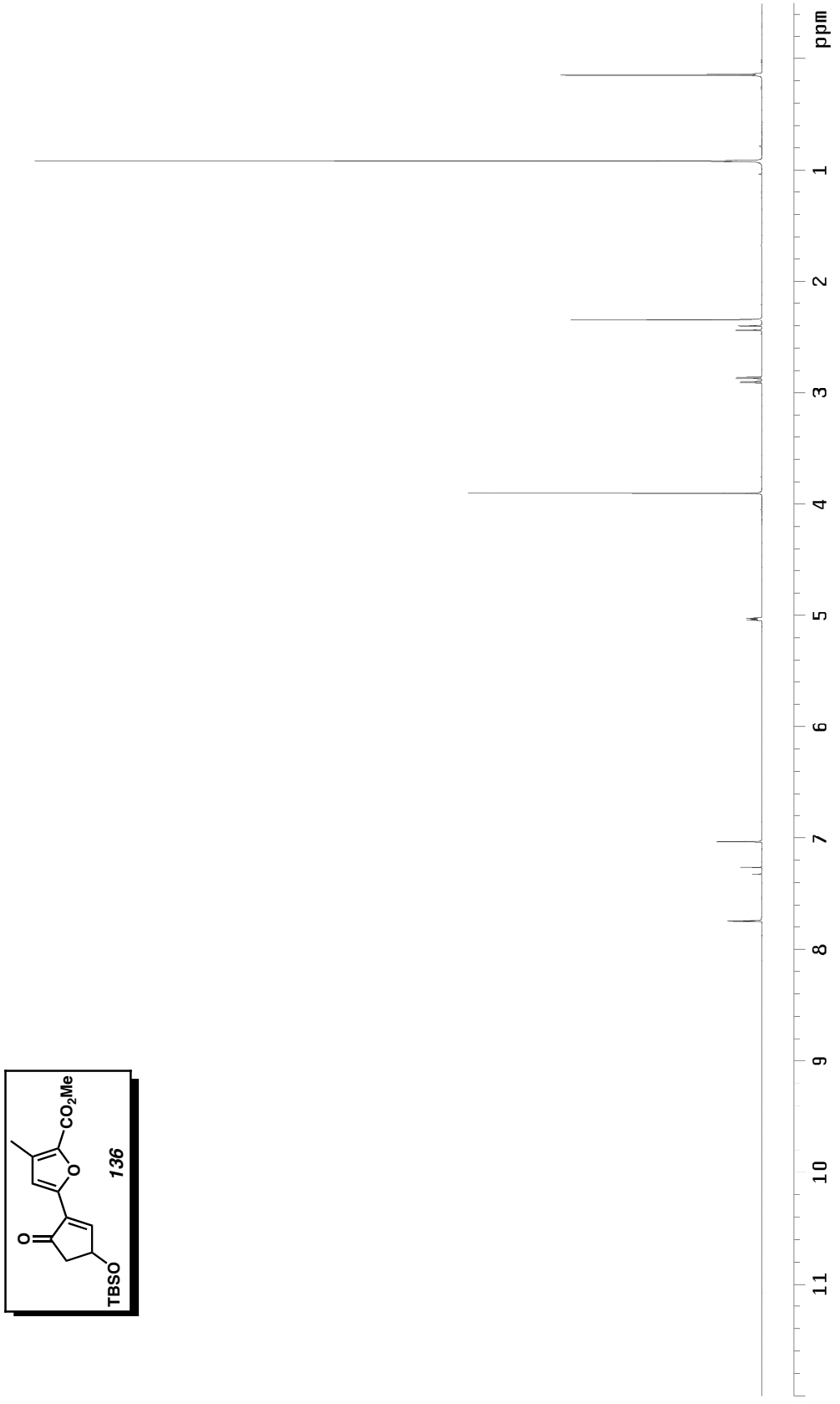


Figure A2.2.1 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **136**

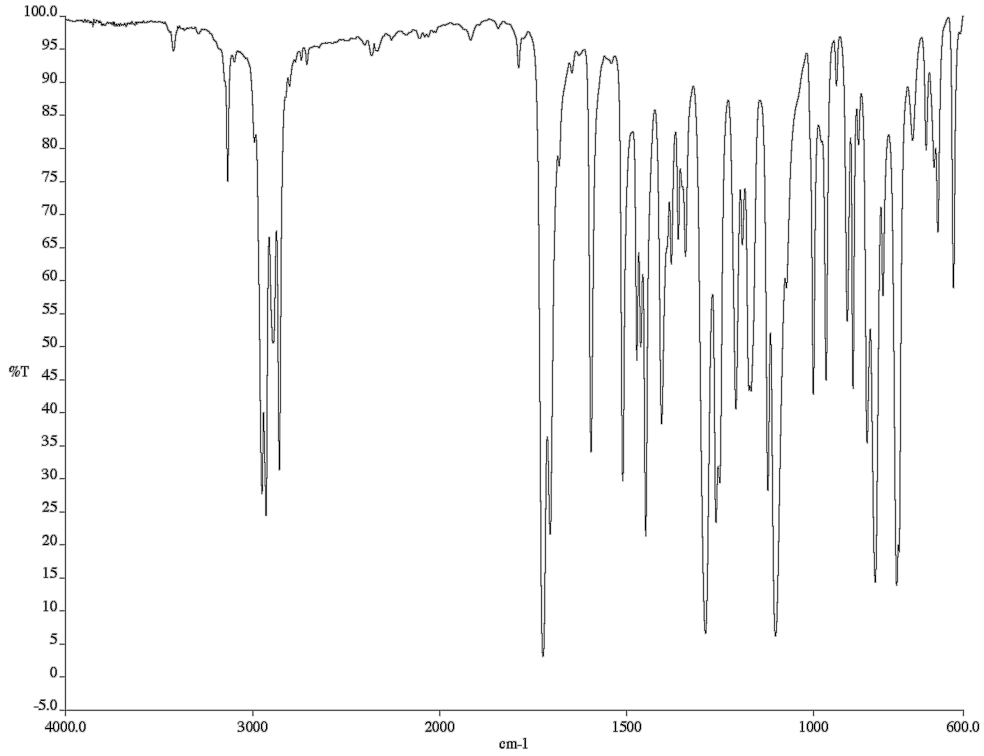


Figure A2.2.2 Infrared spectrum (film/NaCl) of compound **136**

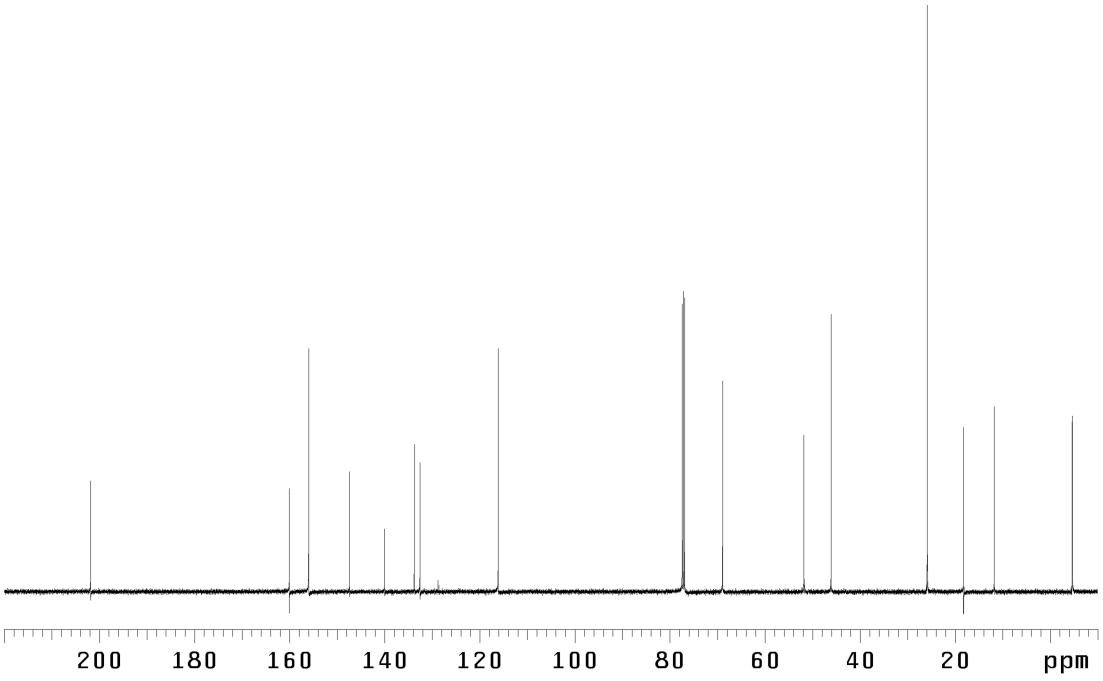


Figure A2.2.3 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **136**

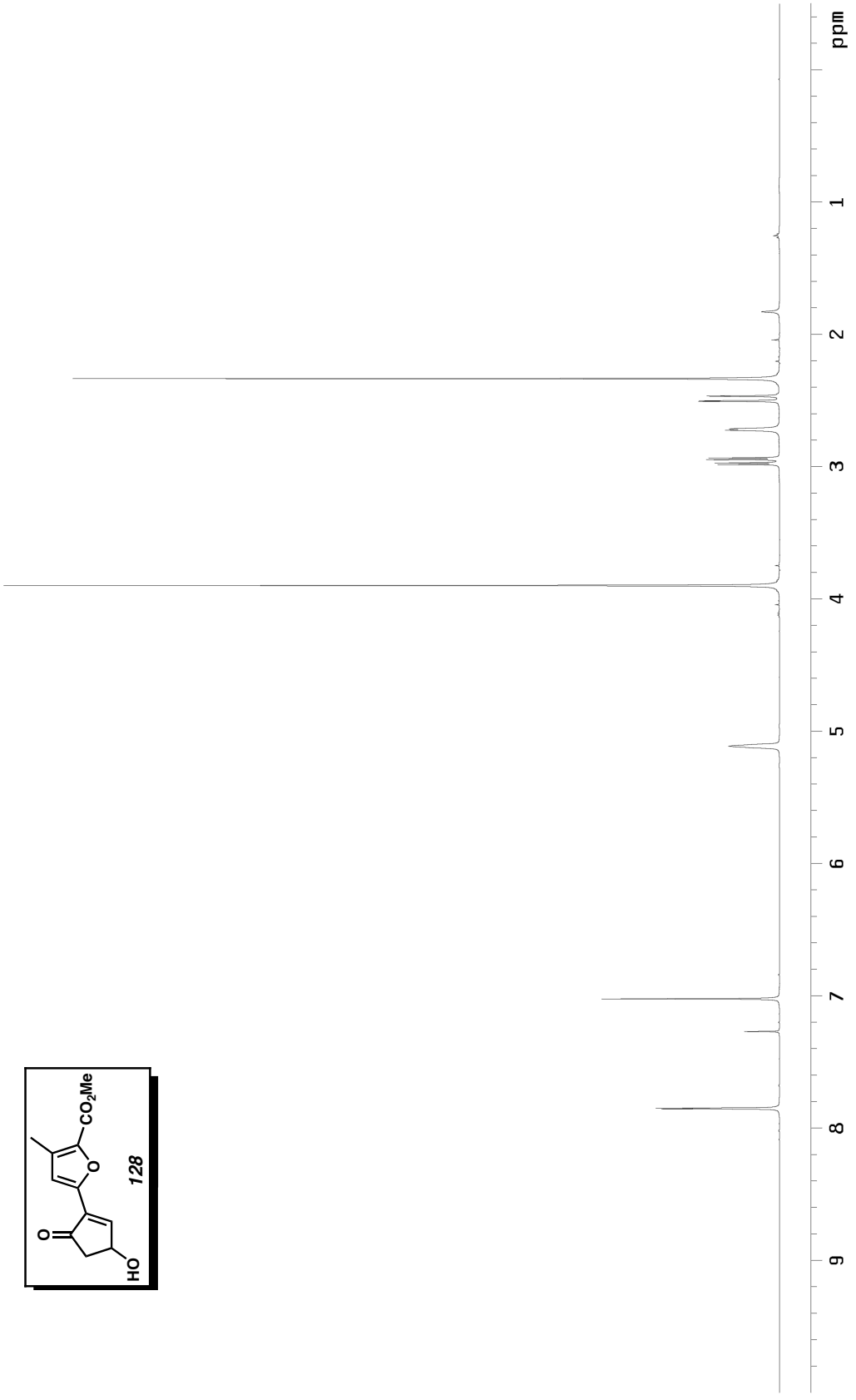


Figure A2.2.4 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **128**

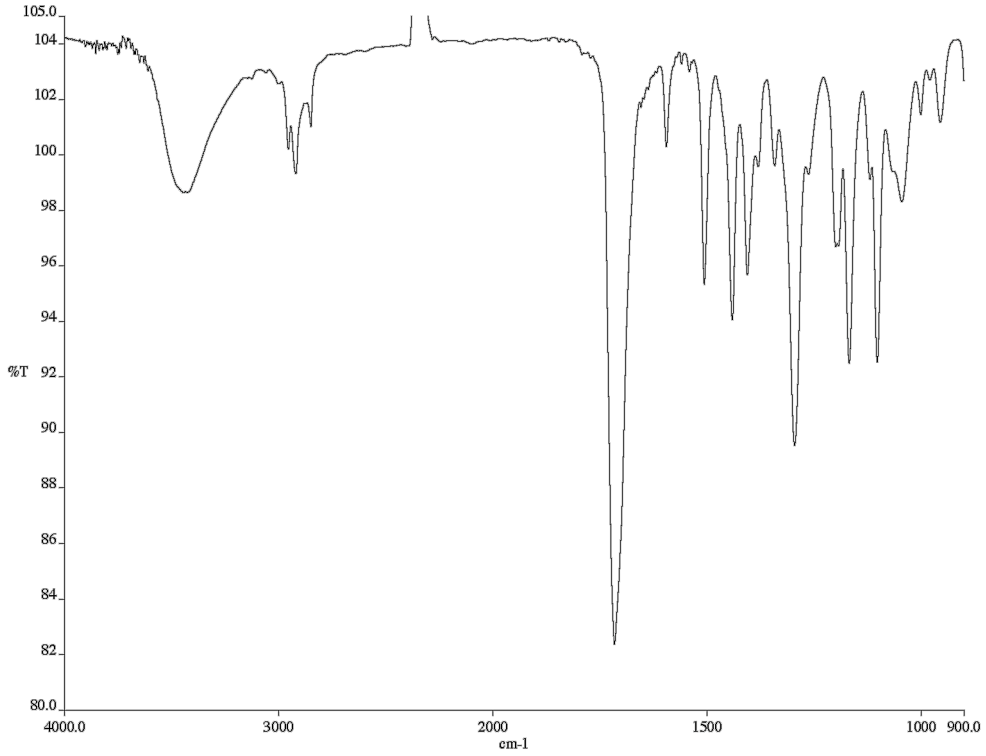


Figure A2.2.5 Infrared spectrum (film/NaCl) of compound **128**

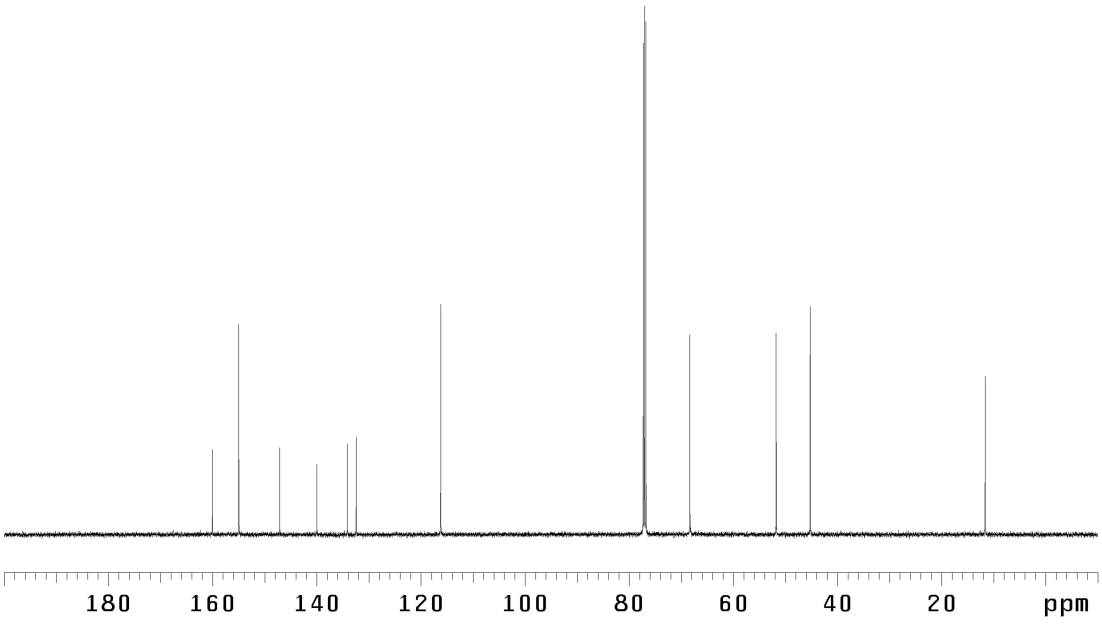


Figure A2.2.6 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **128**

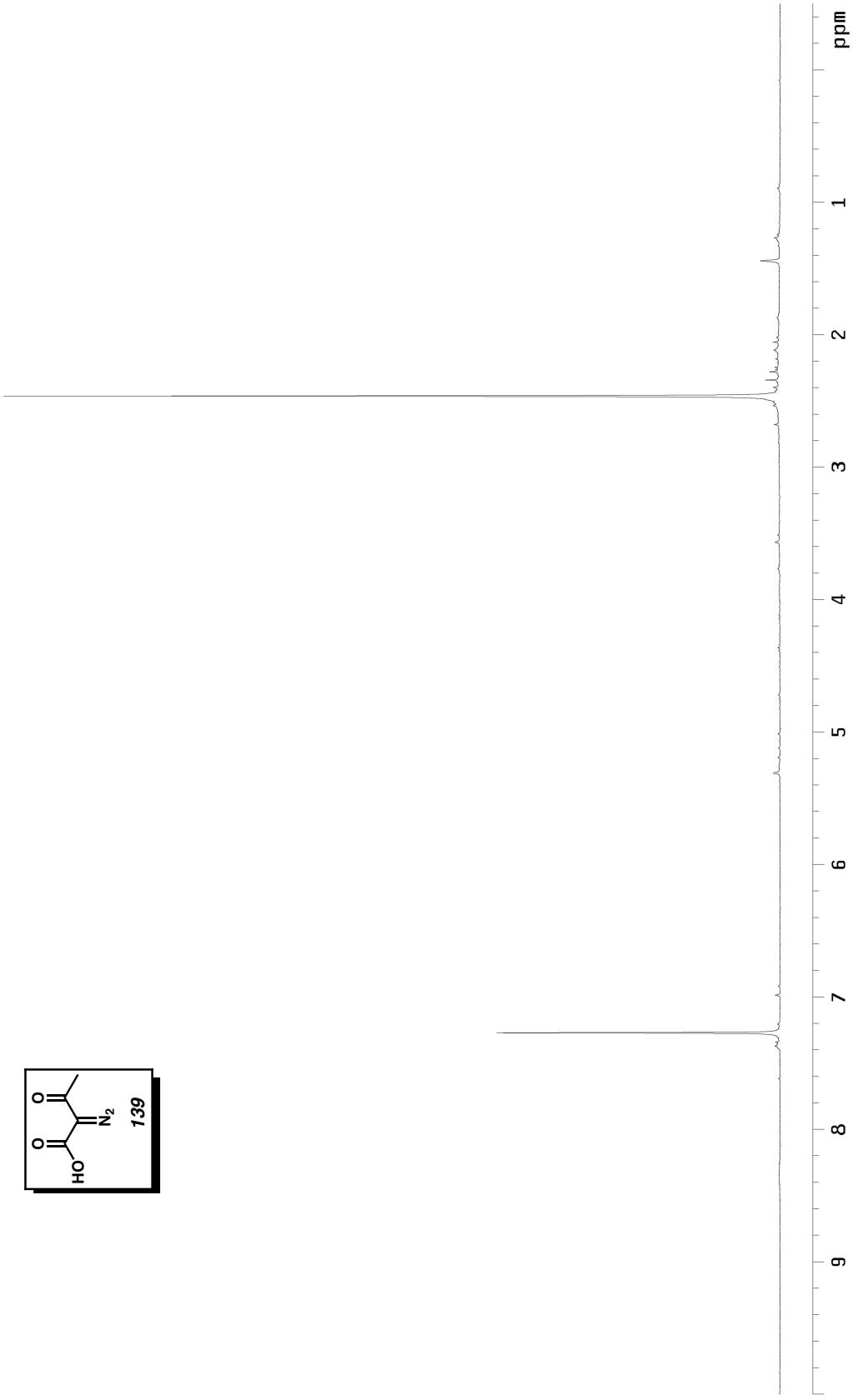


Figure A2.2.7 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **139**

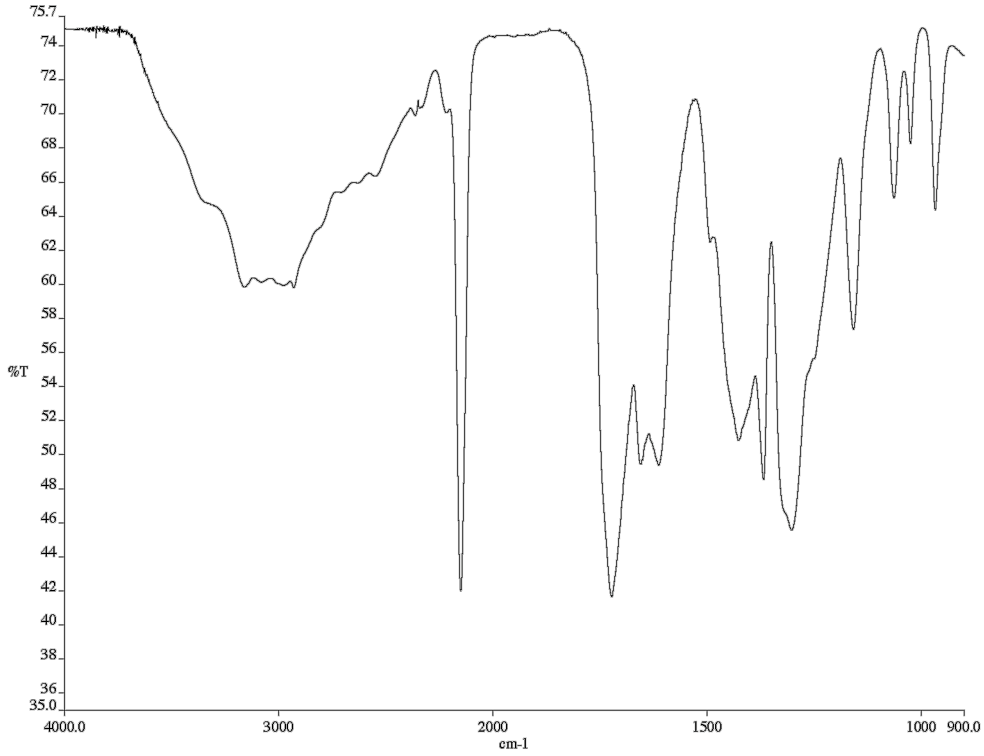


Figure A2.2.8 Infrared spectrum (film/NaCl) of compound **139**

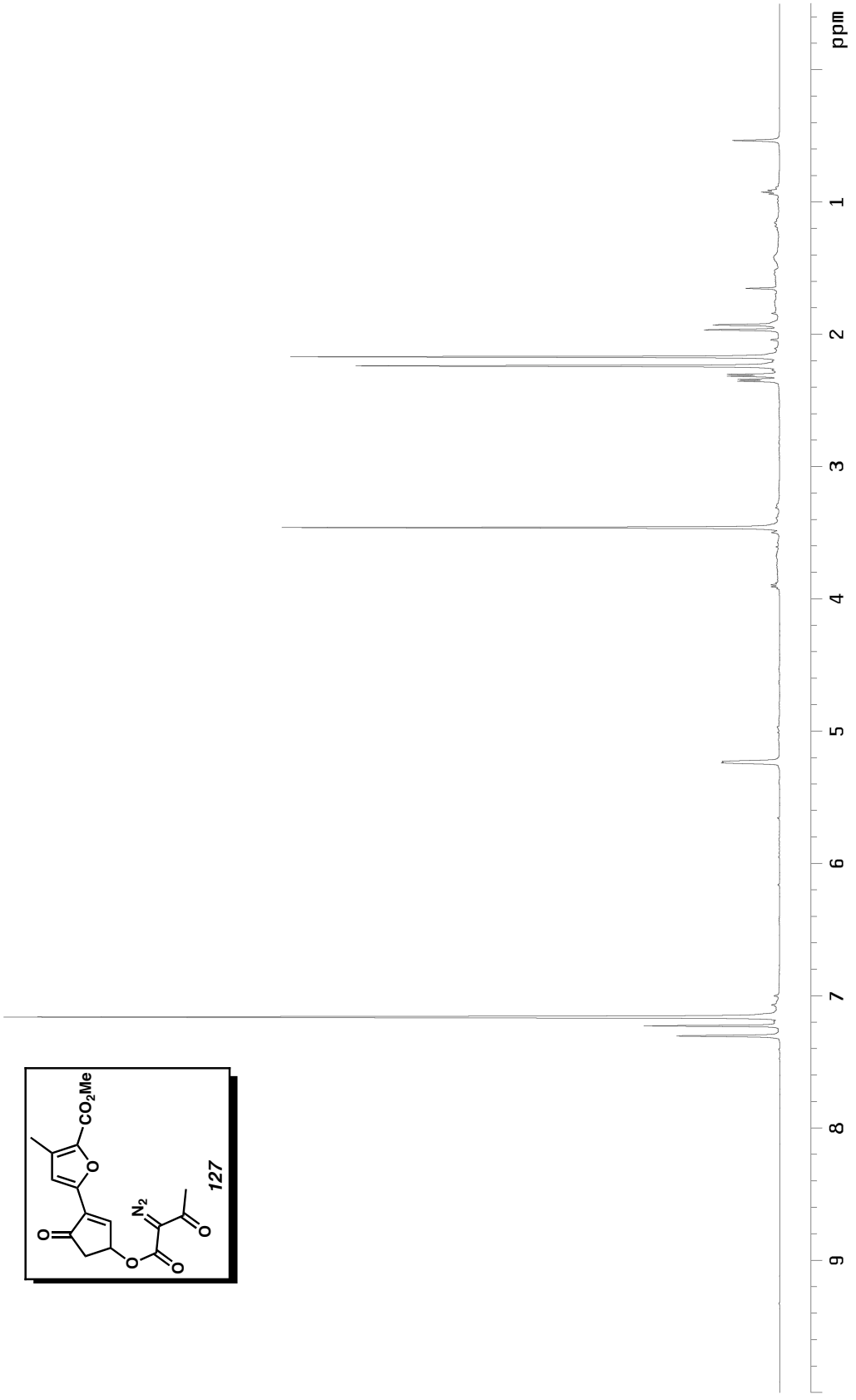


Figure A2.2.9 ^{1}H NMR (500 MHz, C_6D_6) of compound **127**

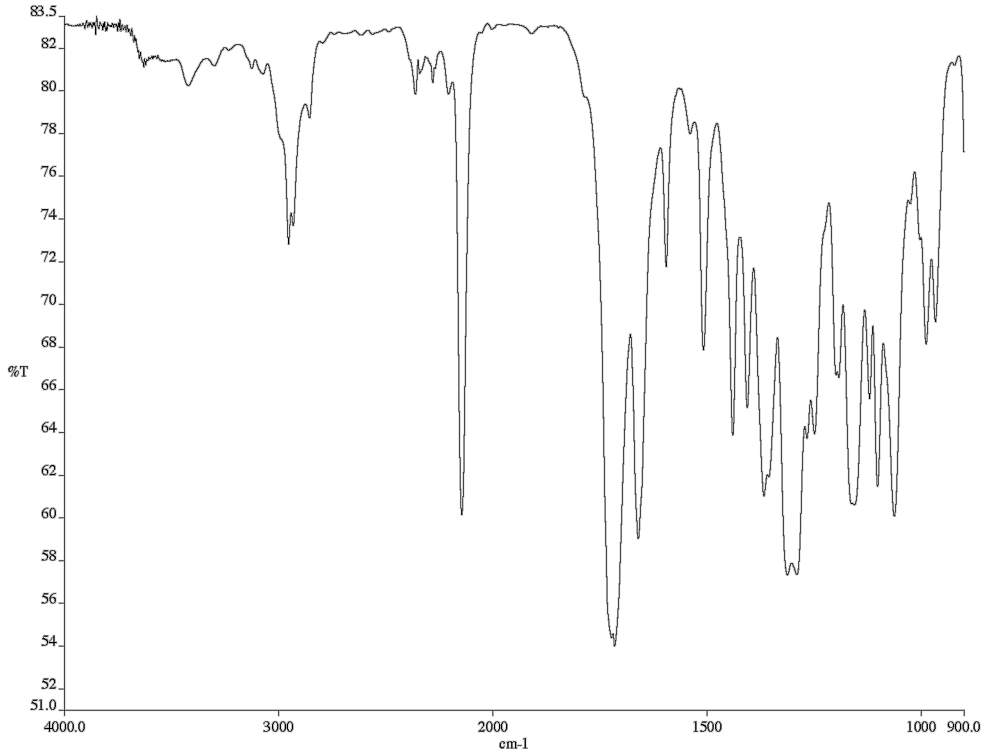


Figure A2.2.10 Infrared spectrum (film/NaCl) of compound **127**

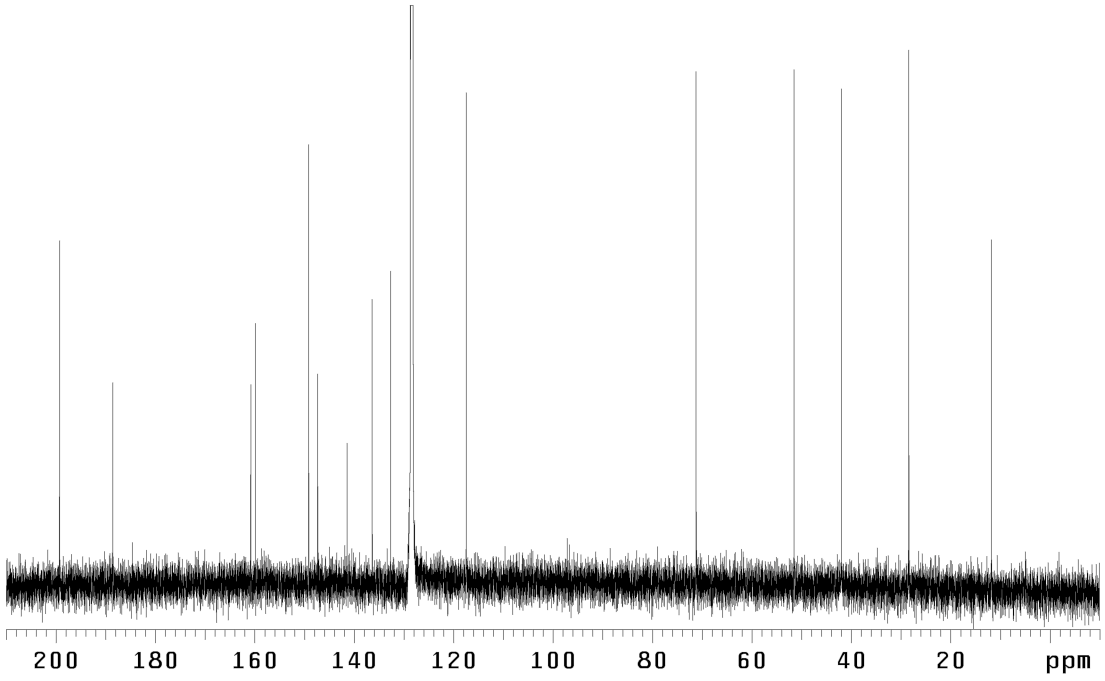


Figure A2.2.11 ^{13}C NMR (125 MHz, C_6D_6) of compound **127**

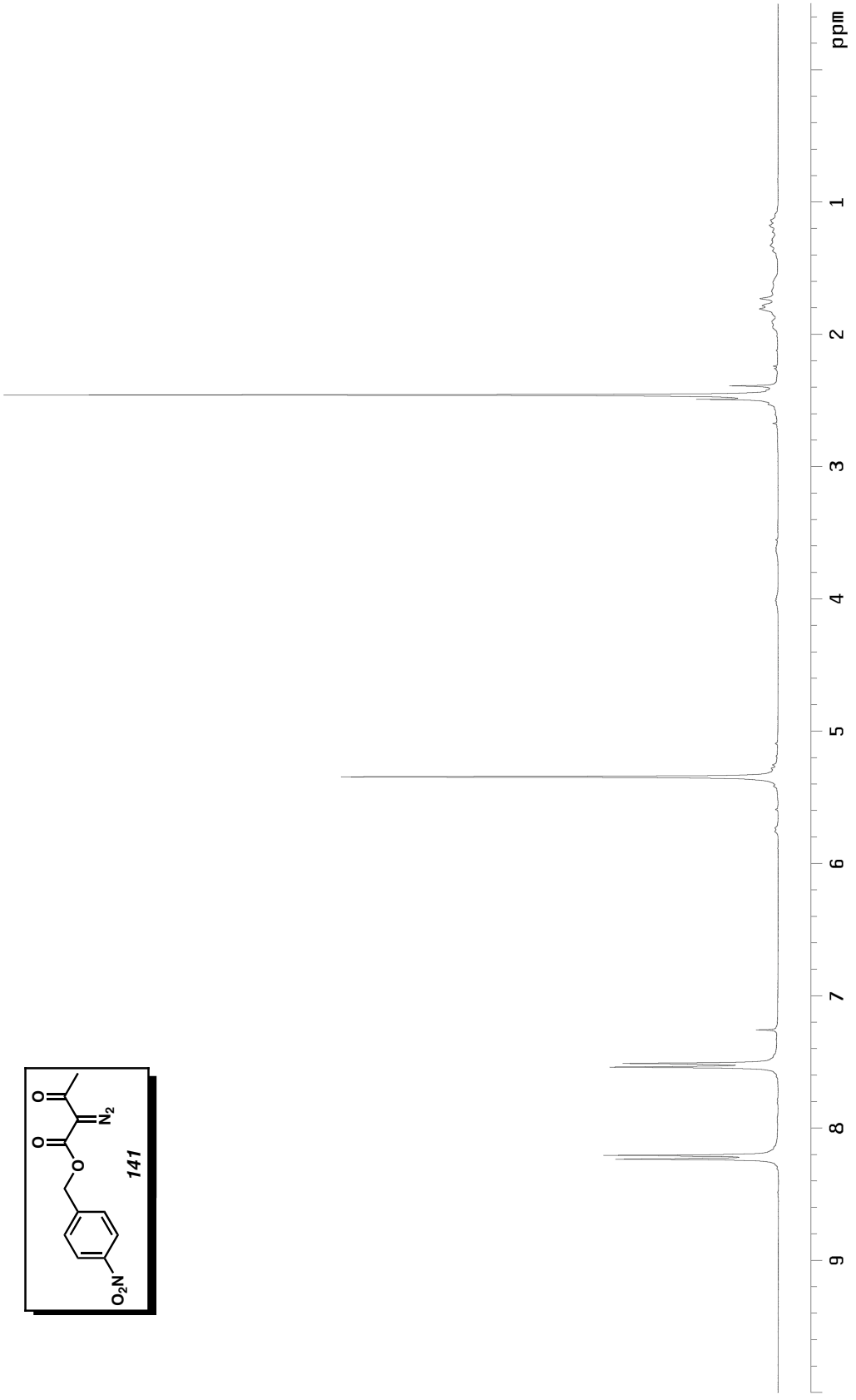


Figure A2.2.12 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **141**

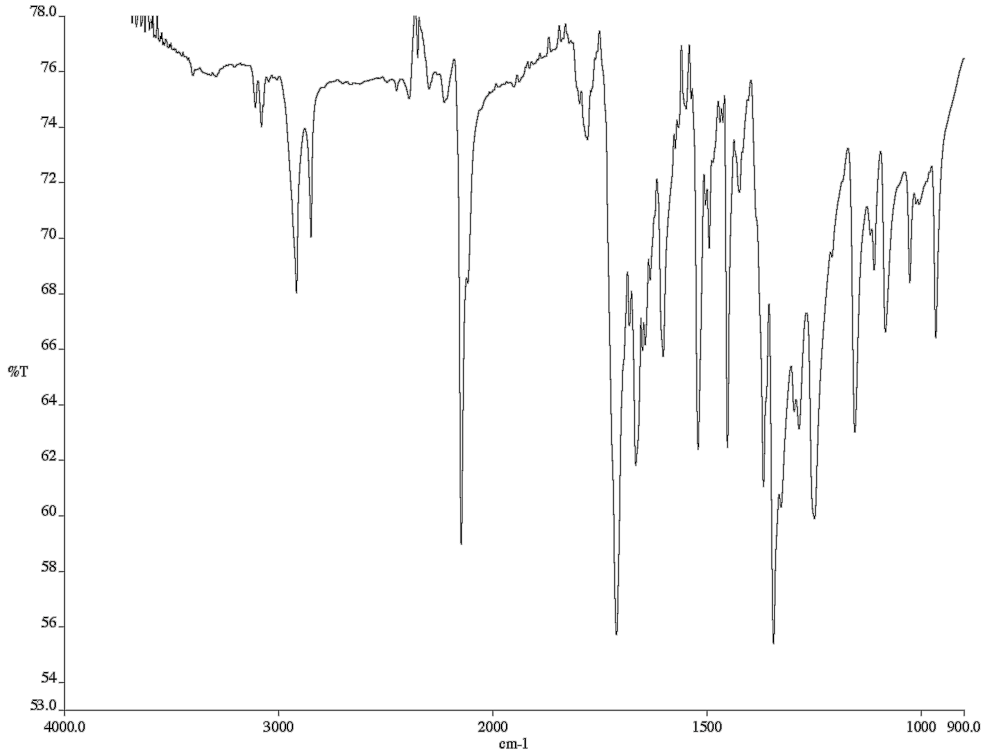


Figure A2.2.13 Infrared spectrum (film/NaCl) of compound **141**

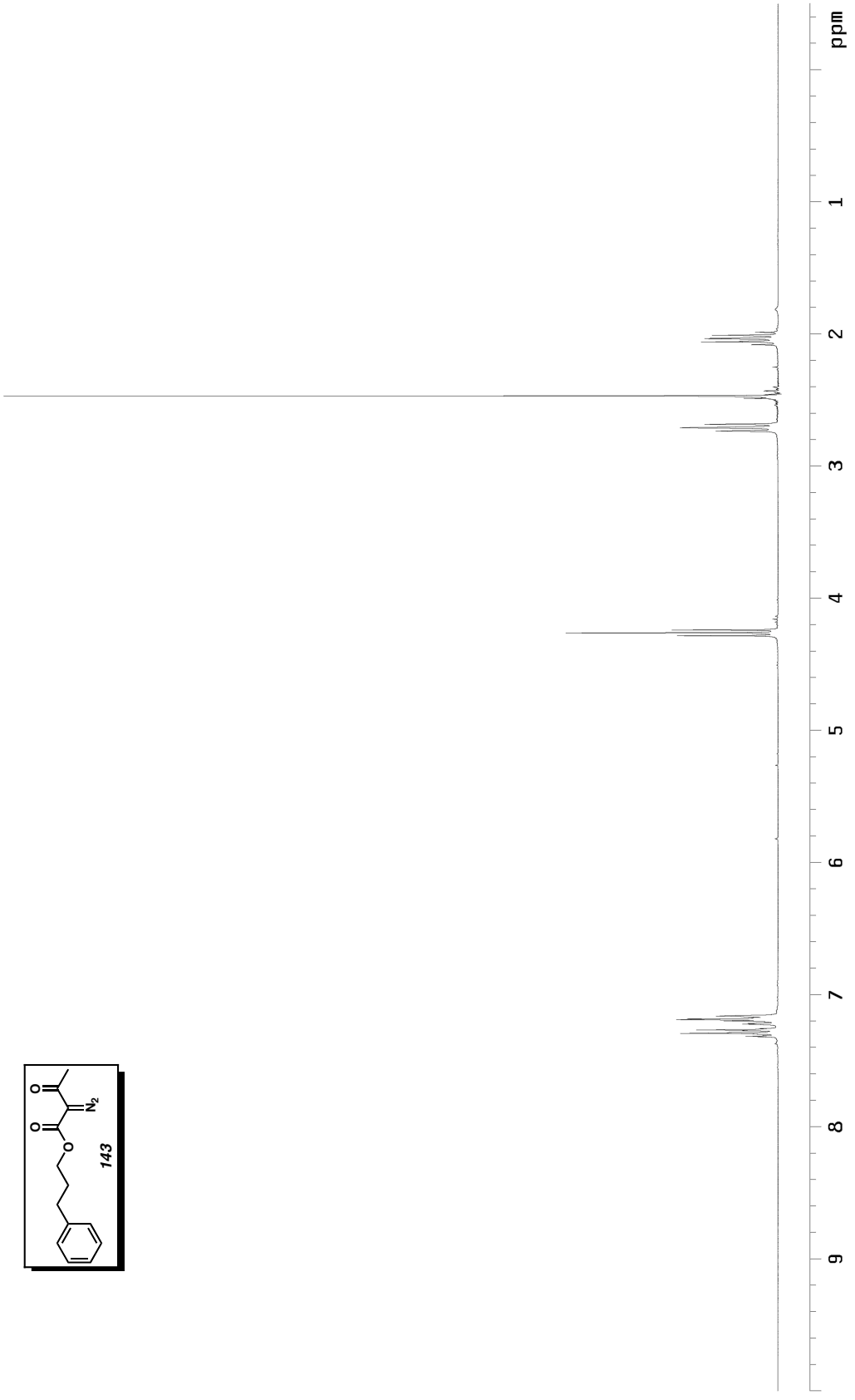


Figure A2.2.14 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **143**

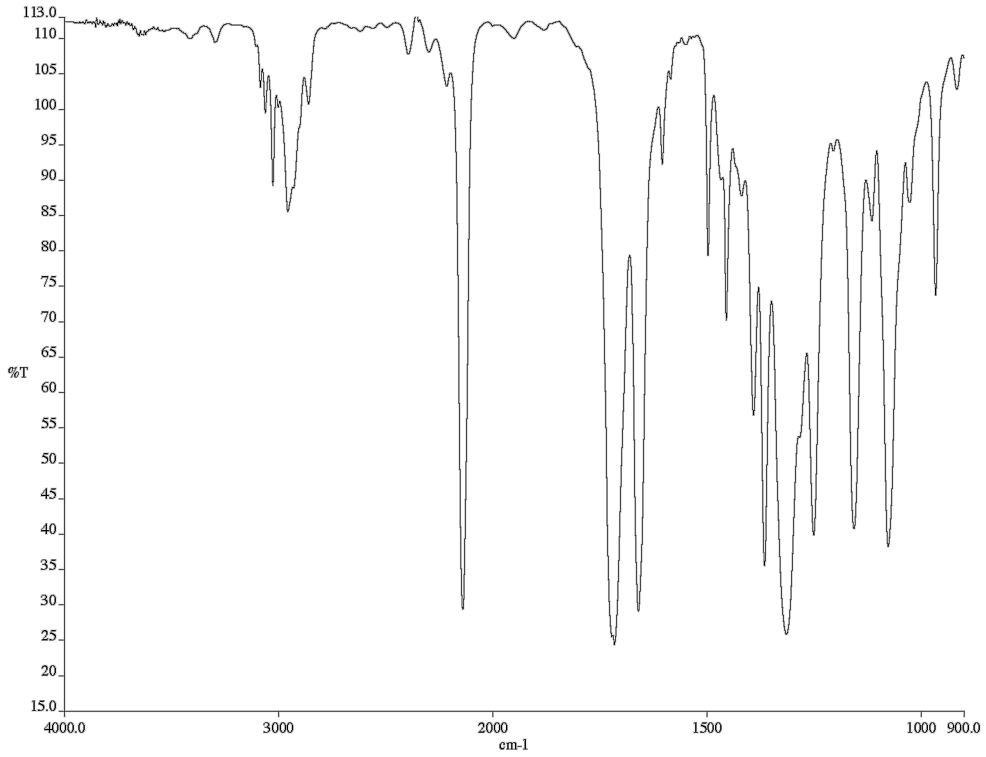


Figure A2.2.15 Infrared spectrum (film/NaCl) of compound **143**

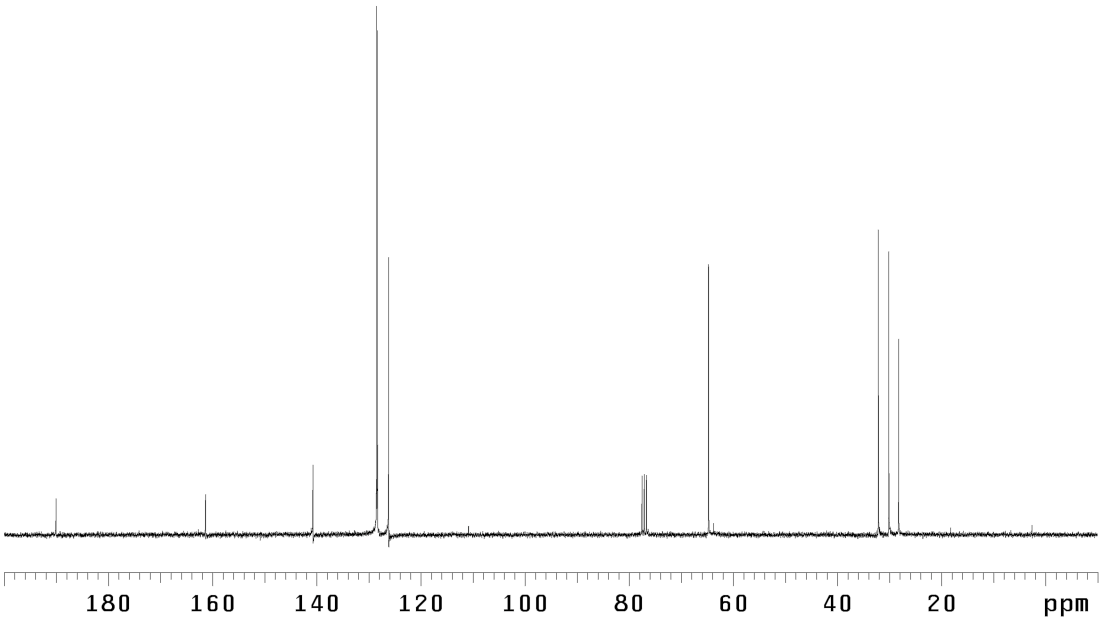


Figure A2.2.16 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **143**

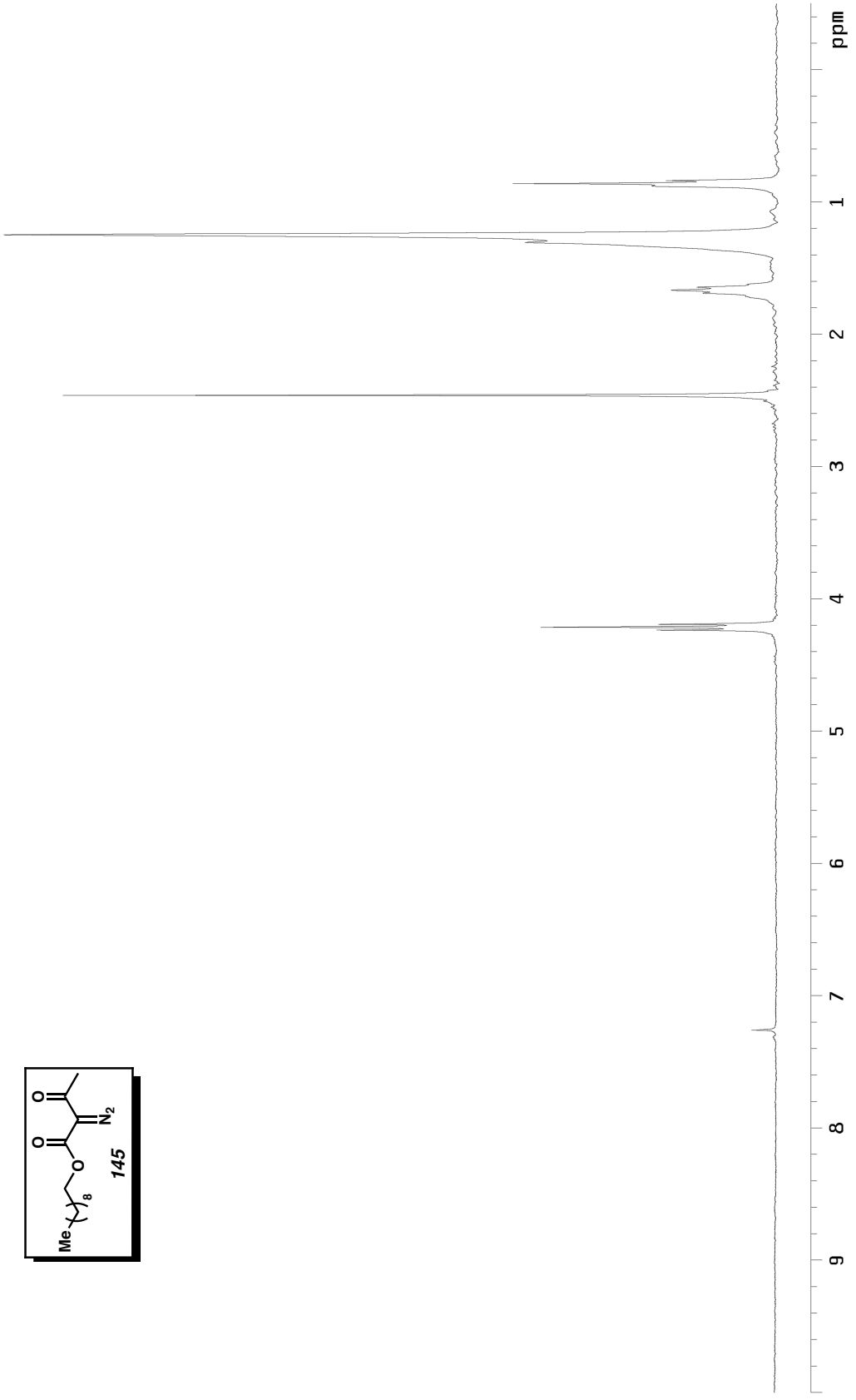


Figure A2.2.17 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **145**

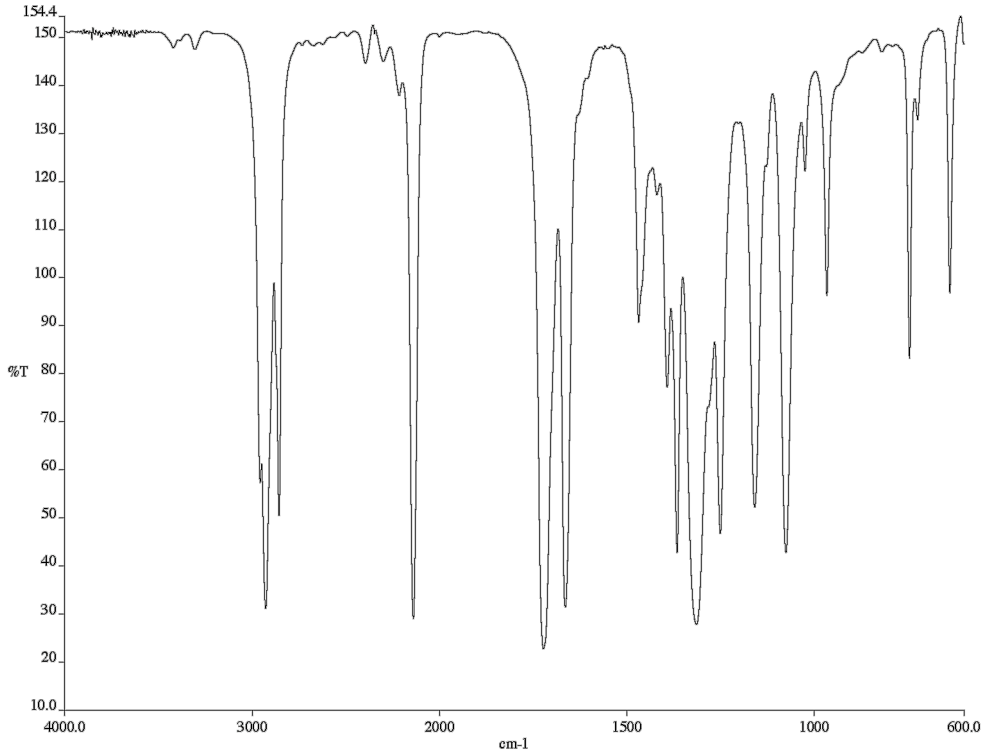


Figure A2.2.18 Infrared spectrum (film/NaCl) of compound **145**

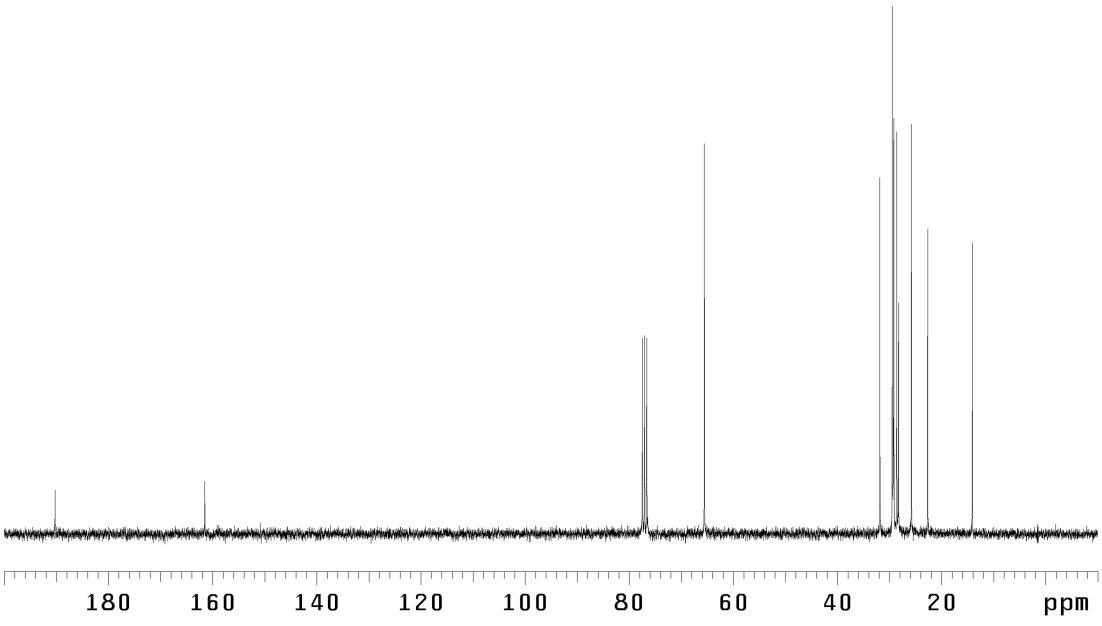


Figure A2.2.19 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **145**

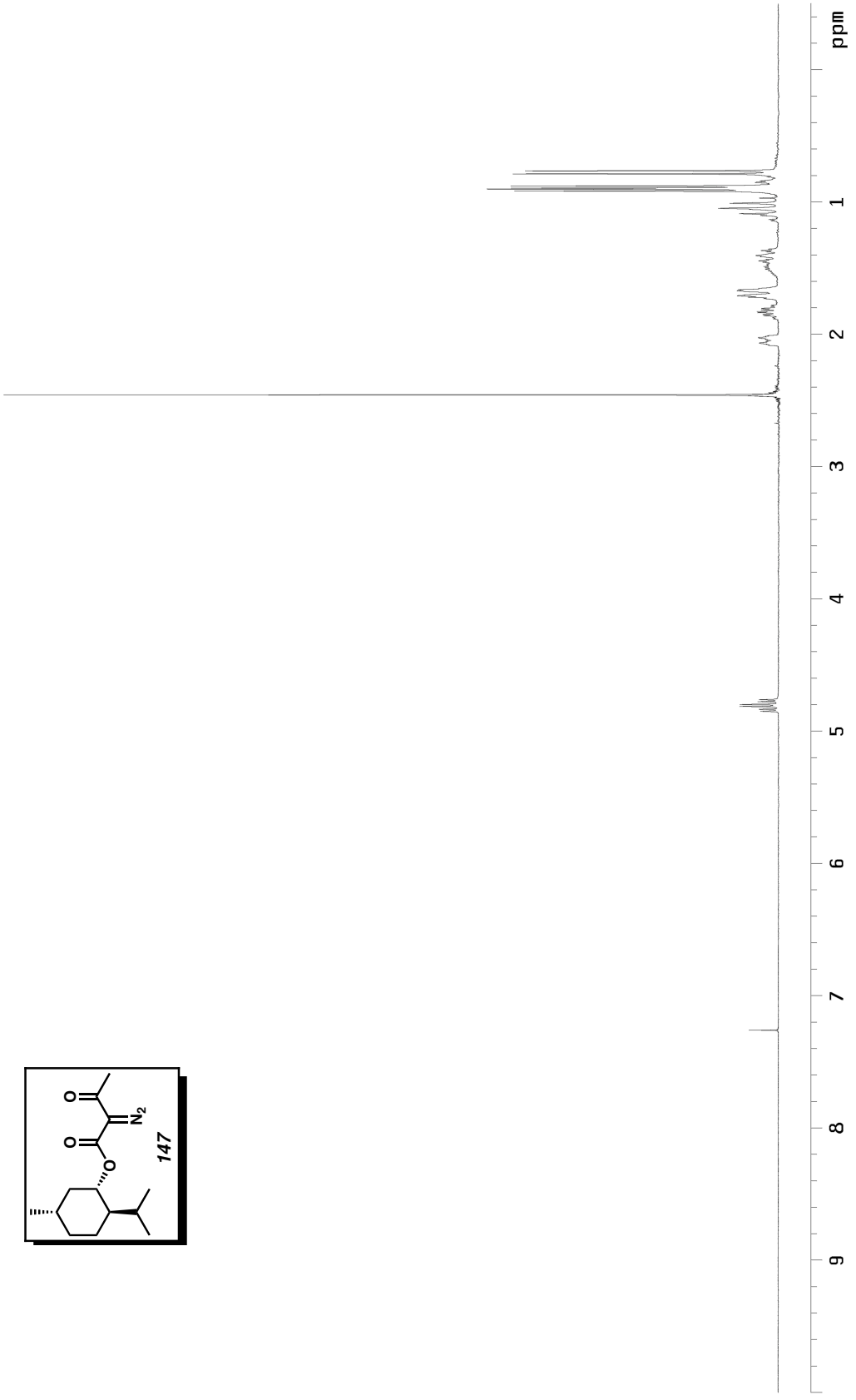


Figure A2.2.20 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **147**

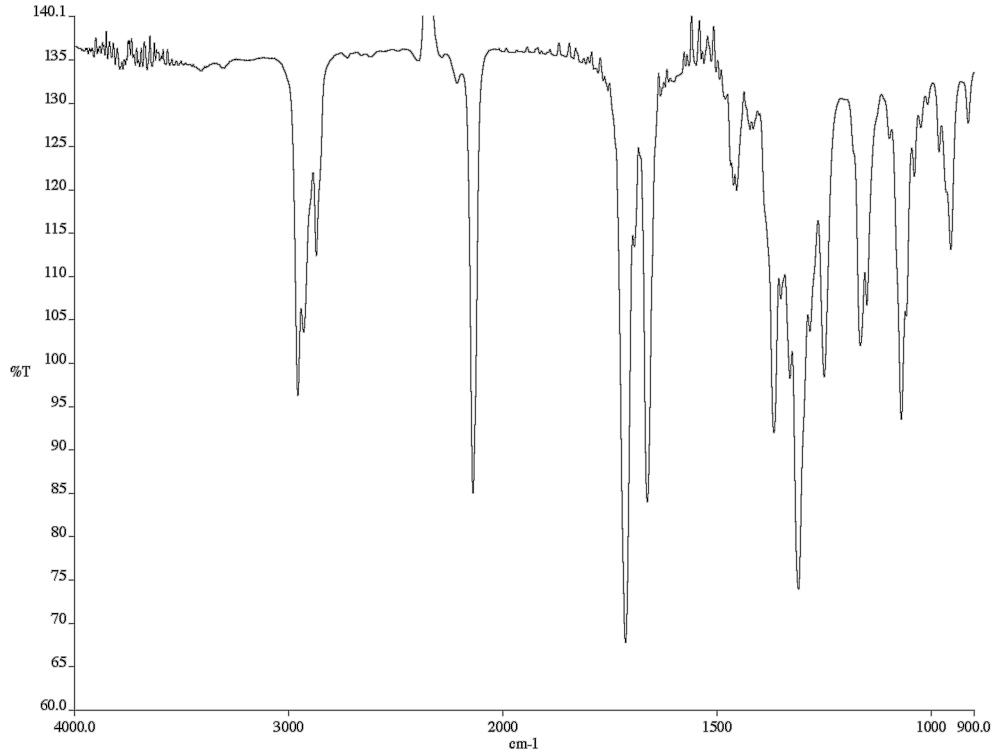


Figure A2.2.21 Infrared spectrum (film/NaCl) of compound **147**

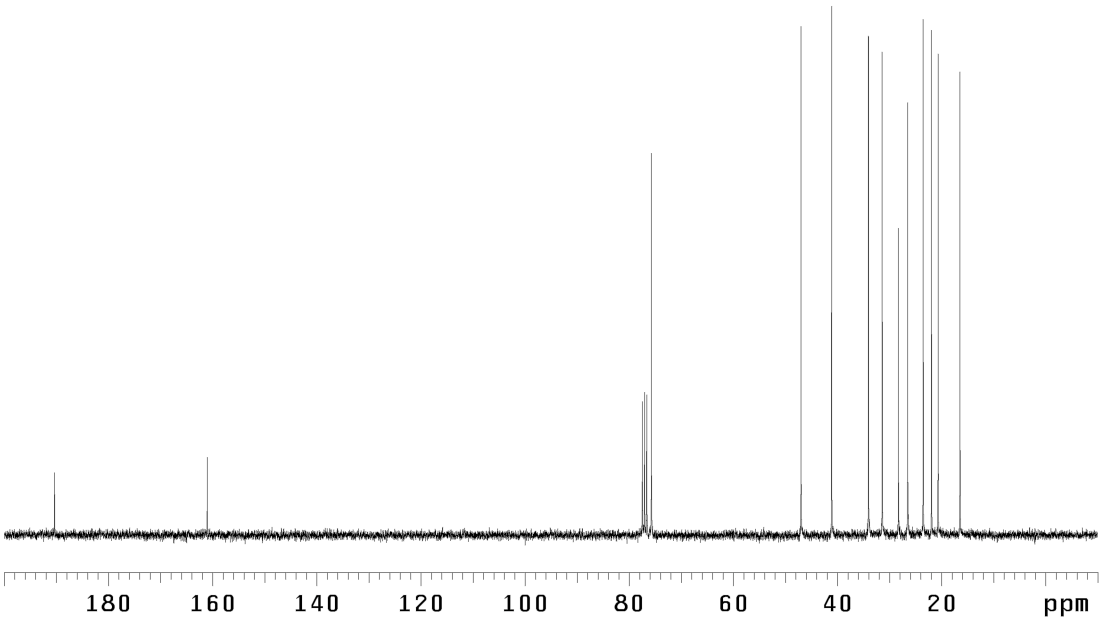


Figure A2.2.22 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **147**

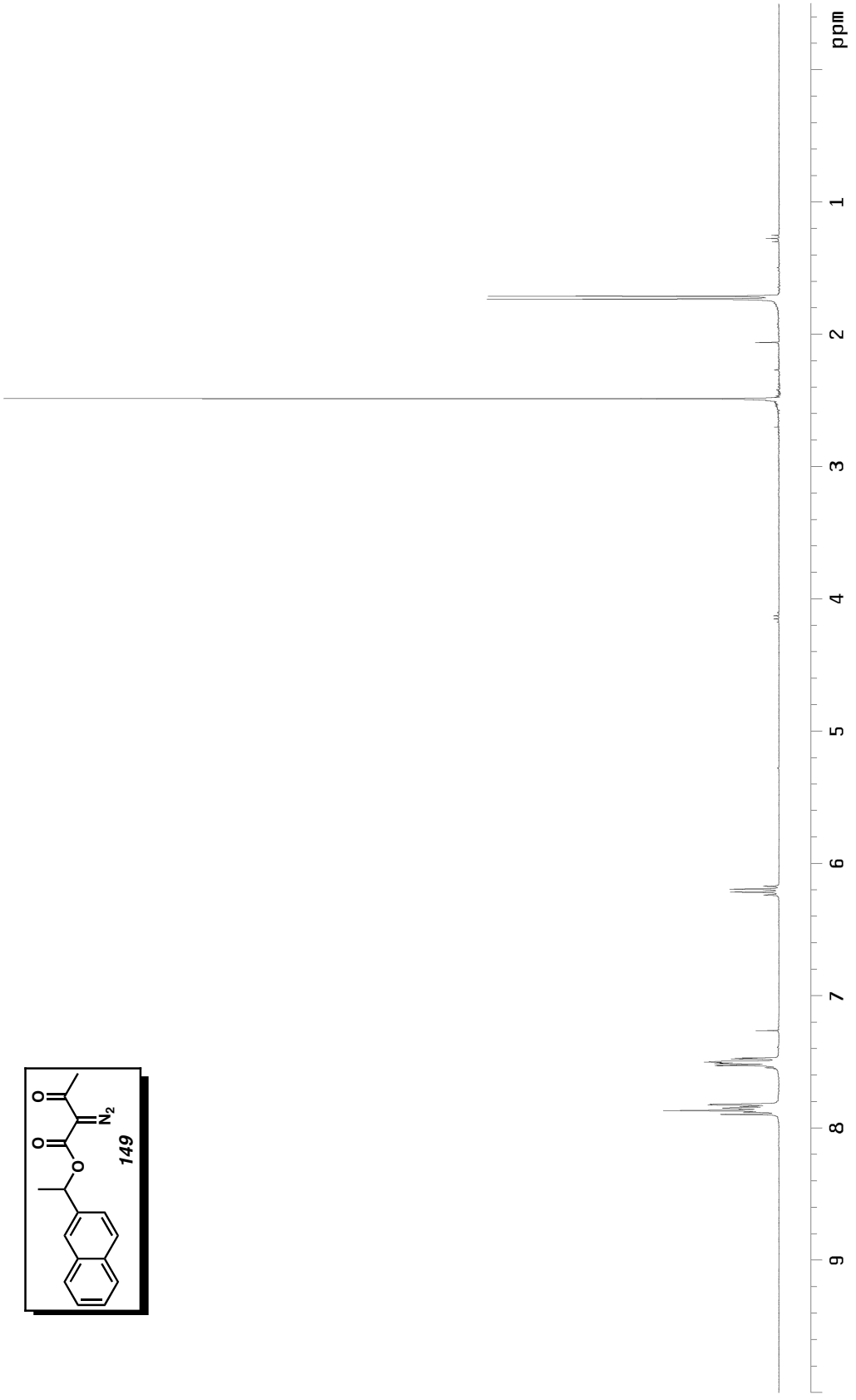


Figure A2.2.23 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **149**

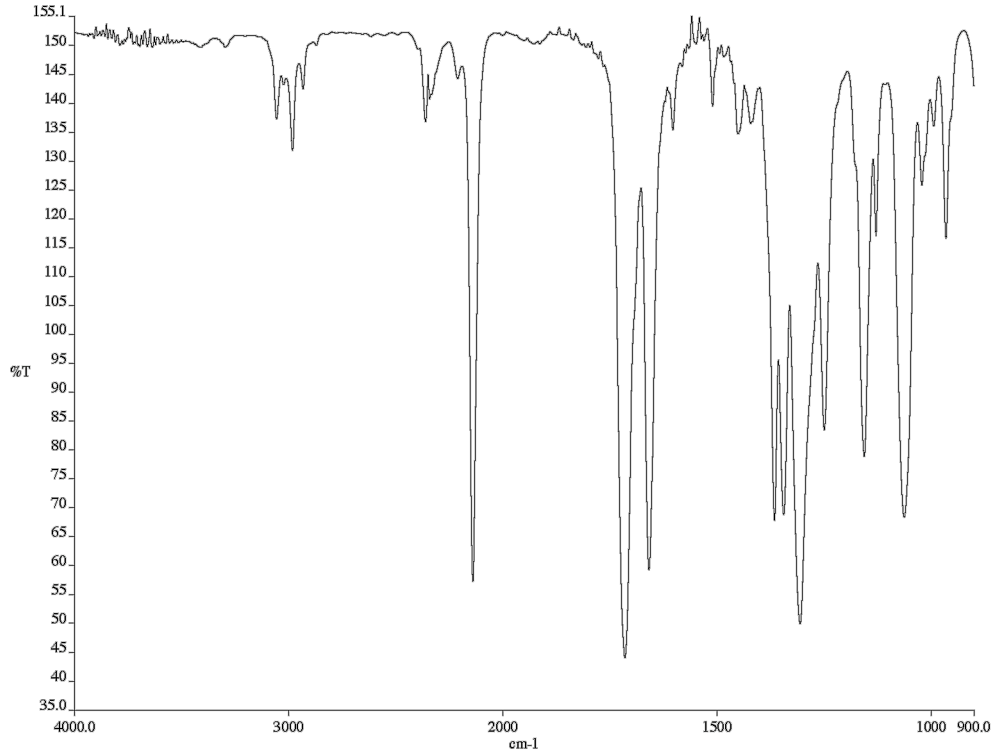


Figure A2.2.24 Infrared spectrum (film/NaCl) of compound **149**

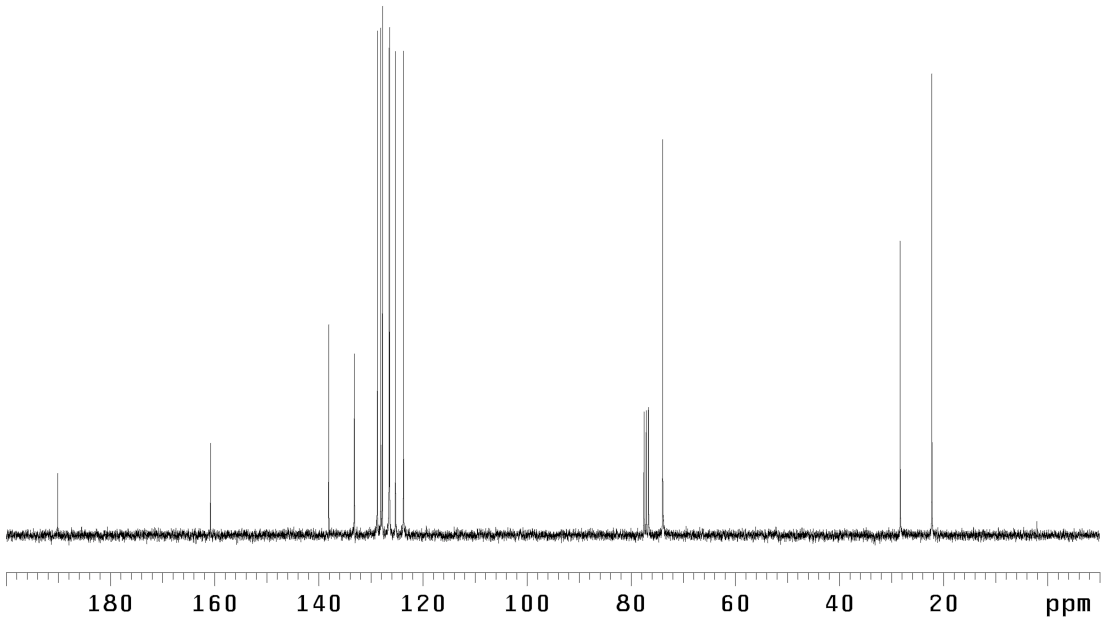


Figure A2.2.25 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **149**

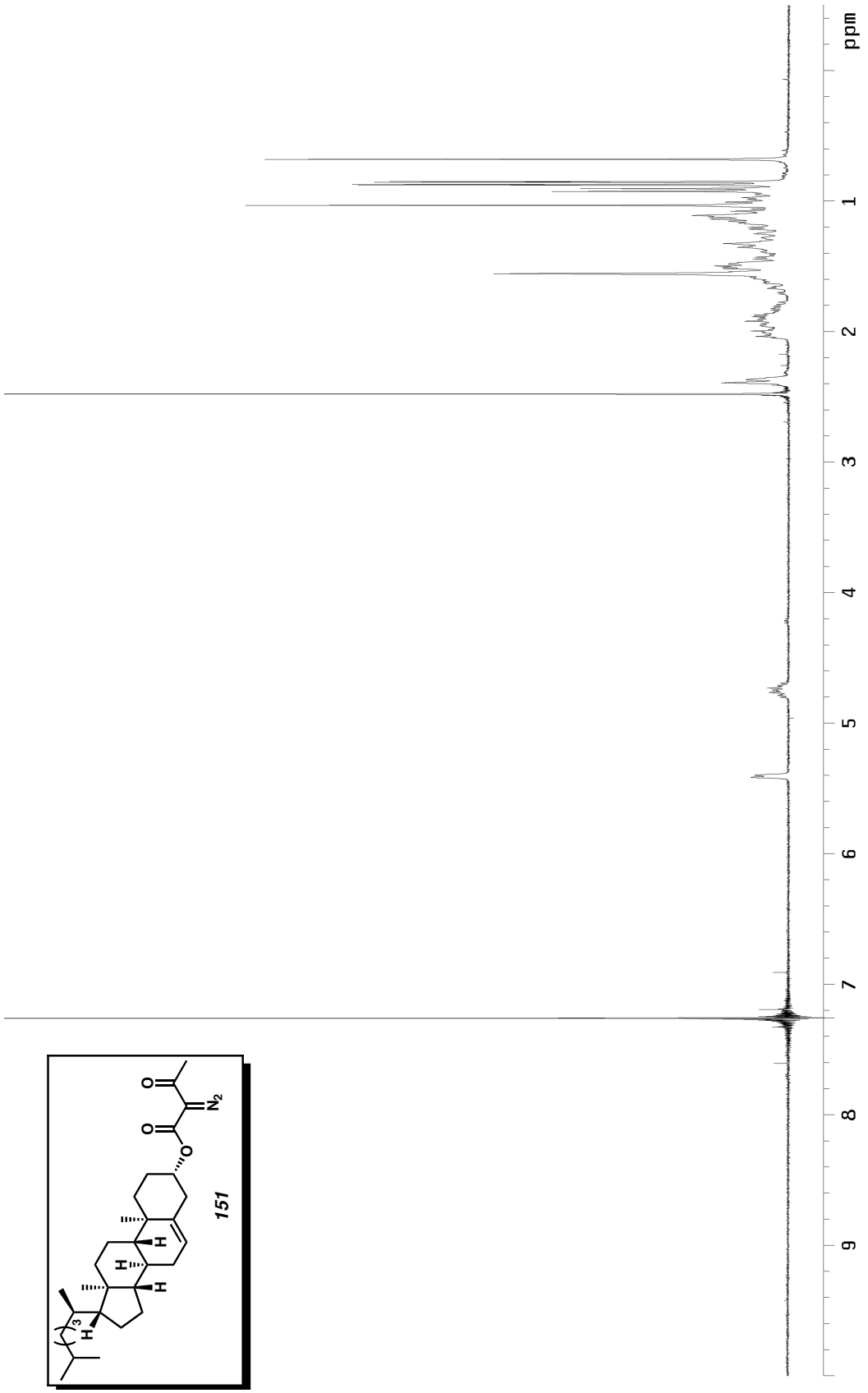


Figure A2.2.26 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **151**

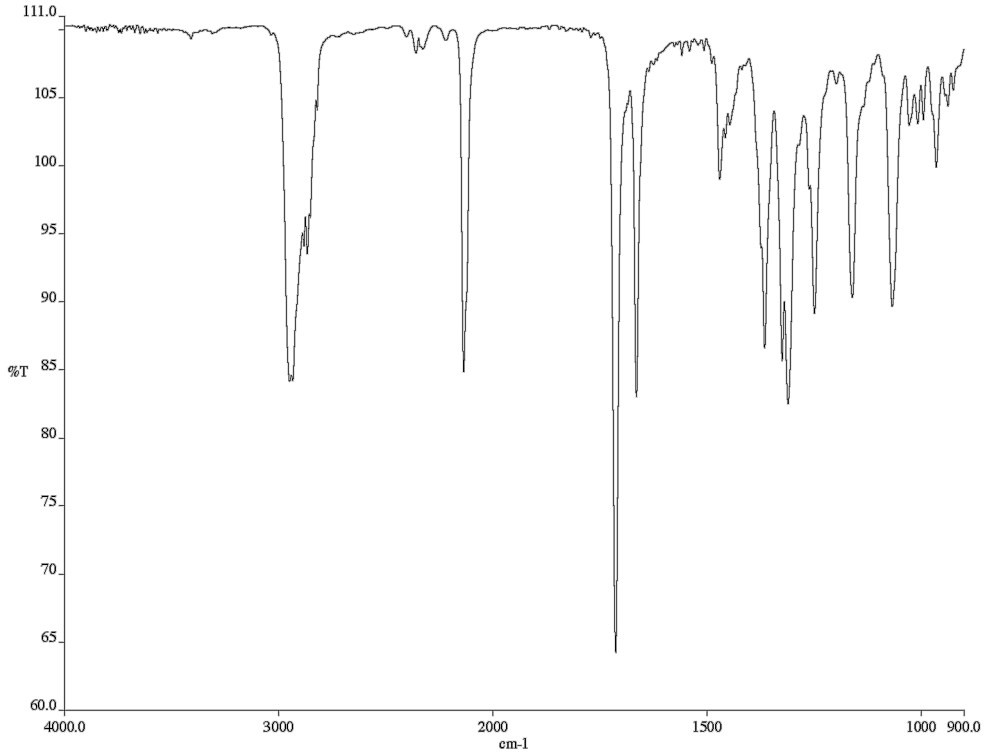


Figure A2.2.27 Infrared spectrum (film/NaCl) of compound **151**

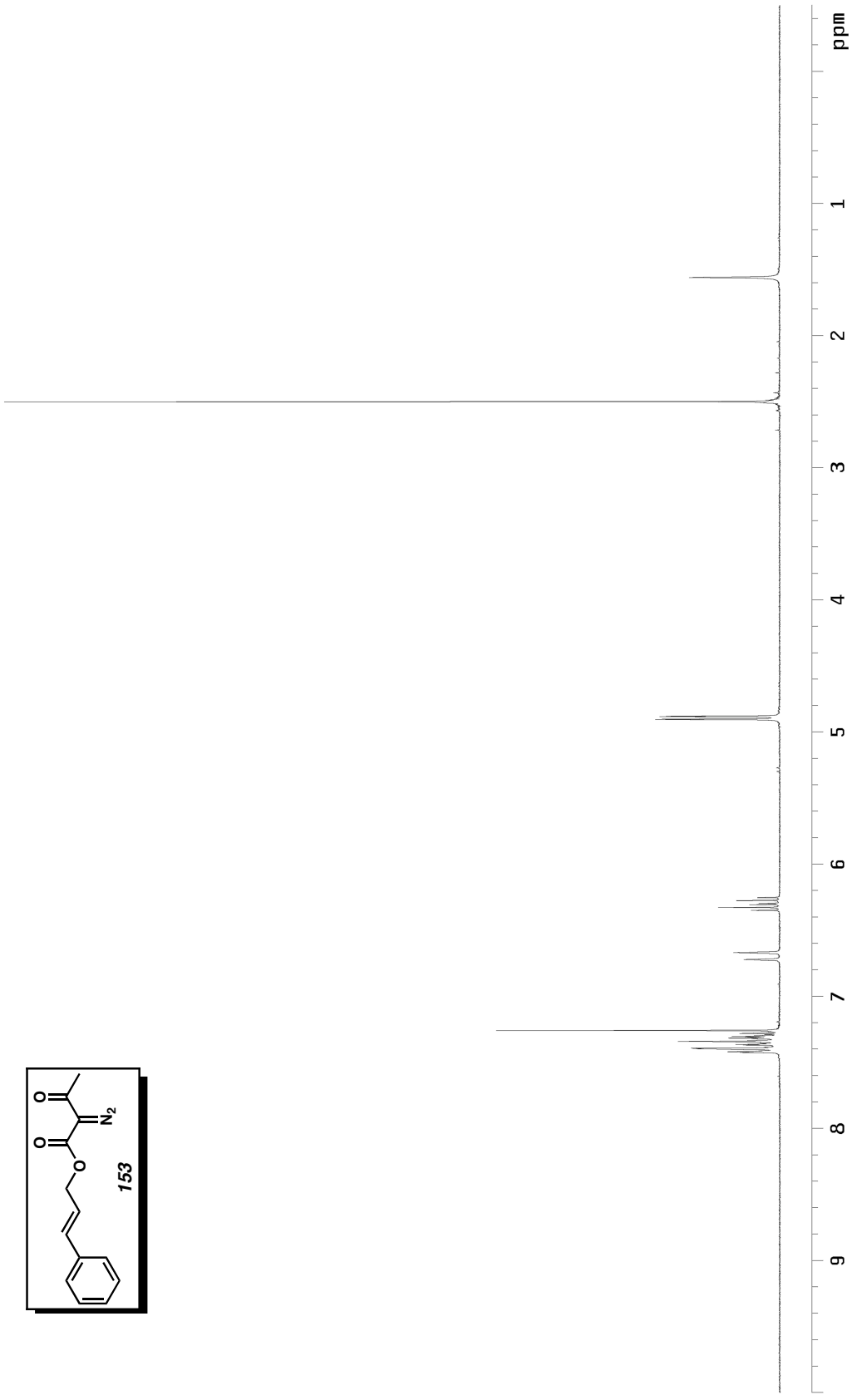


Figure A2.2.28 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **153**

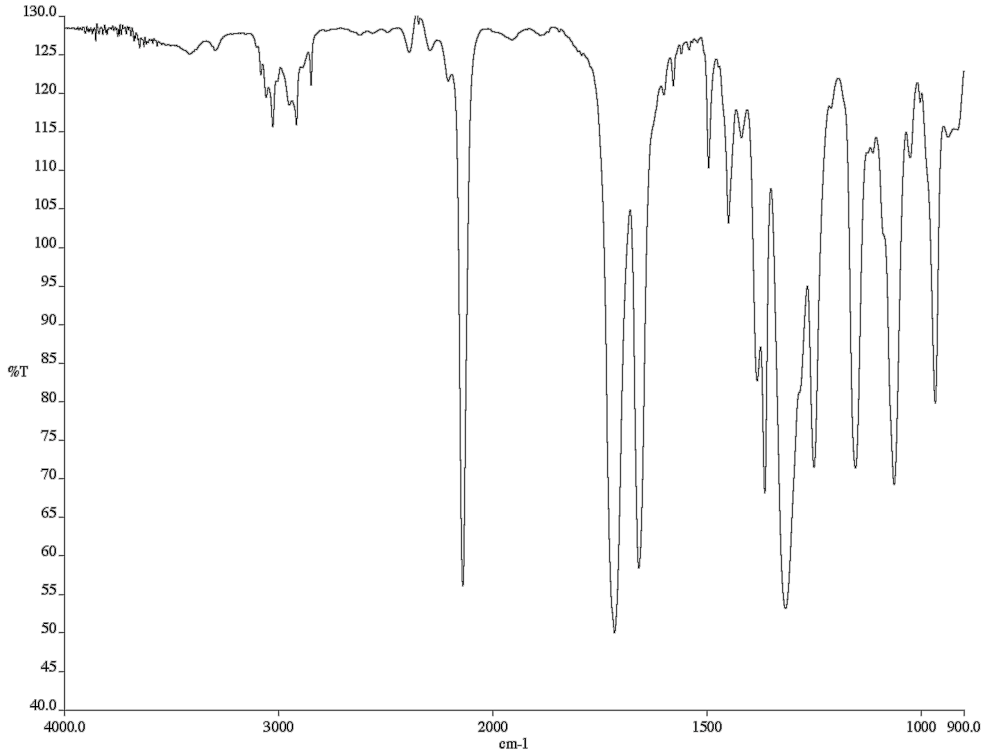


Figure A2.2.29 Infrared spectrum (film/NaCl) of compound **153**

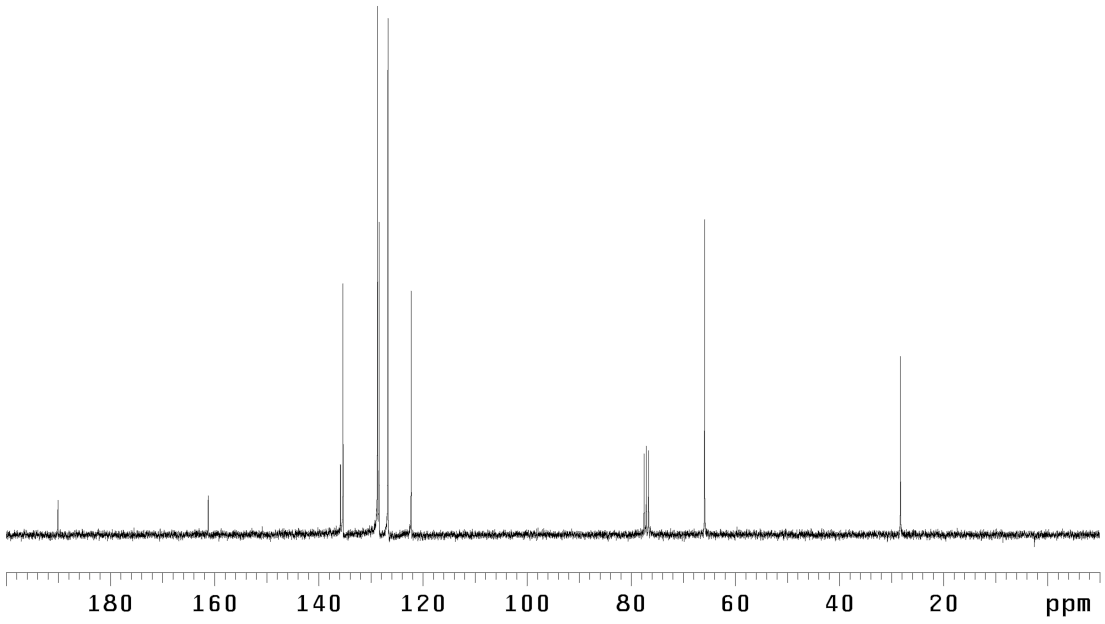


Figure A2.2.30 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **153**

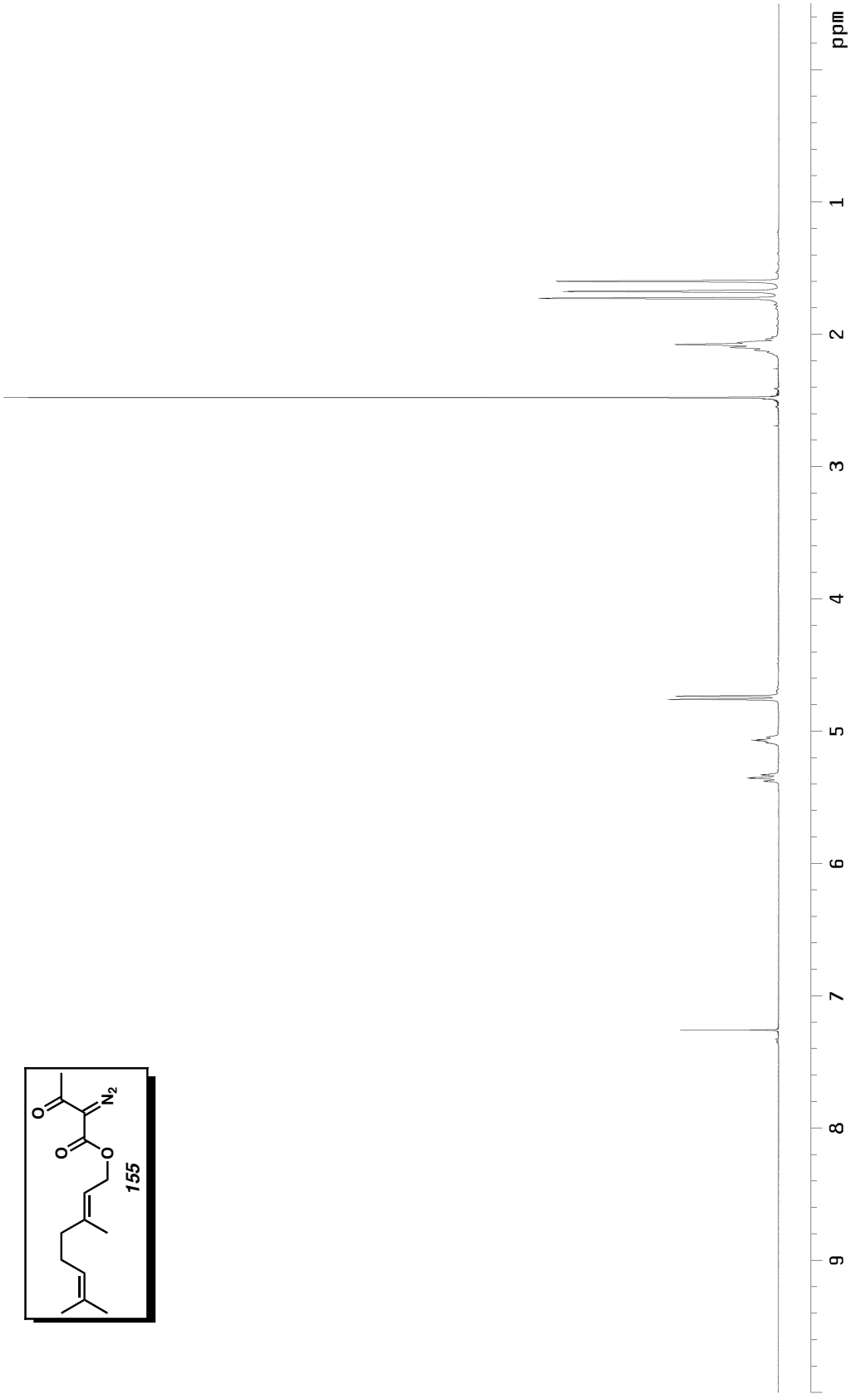


Figure A2.2.31 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **155**

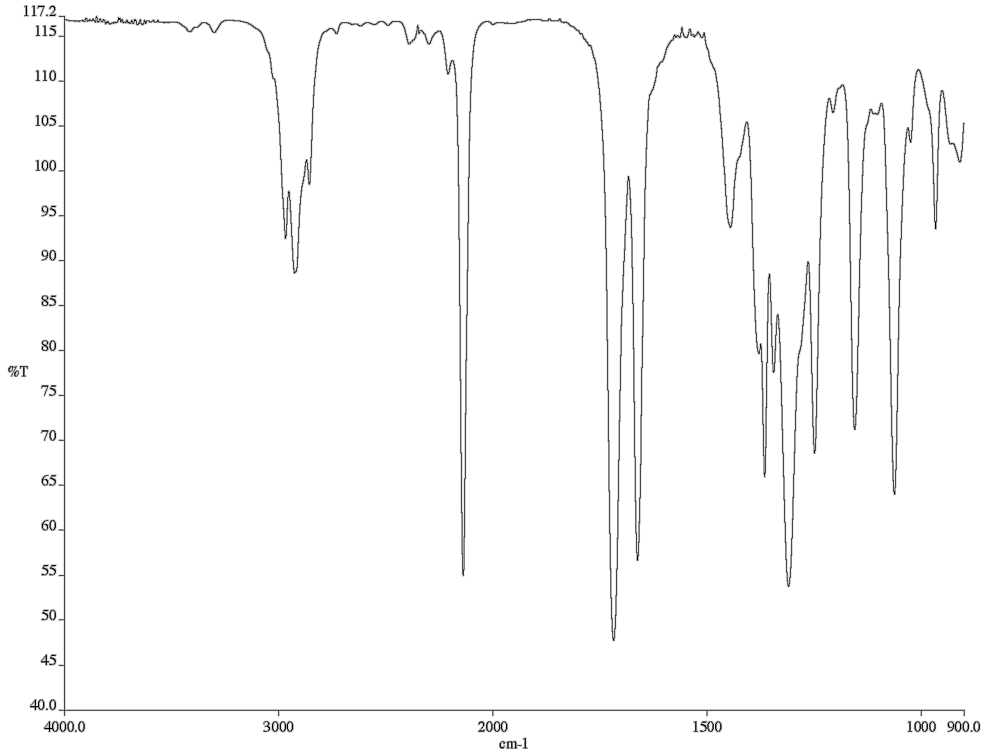


Figure A2.2.32 Infrared spectrum (film/NaCl) of compound **155**

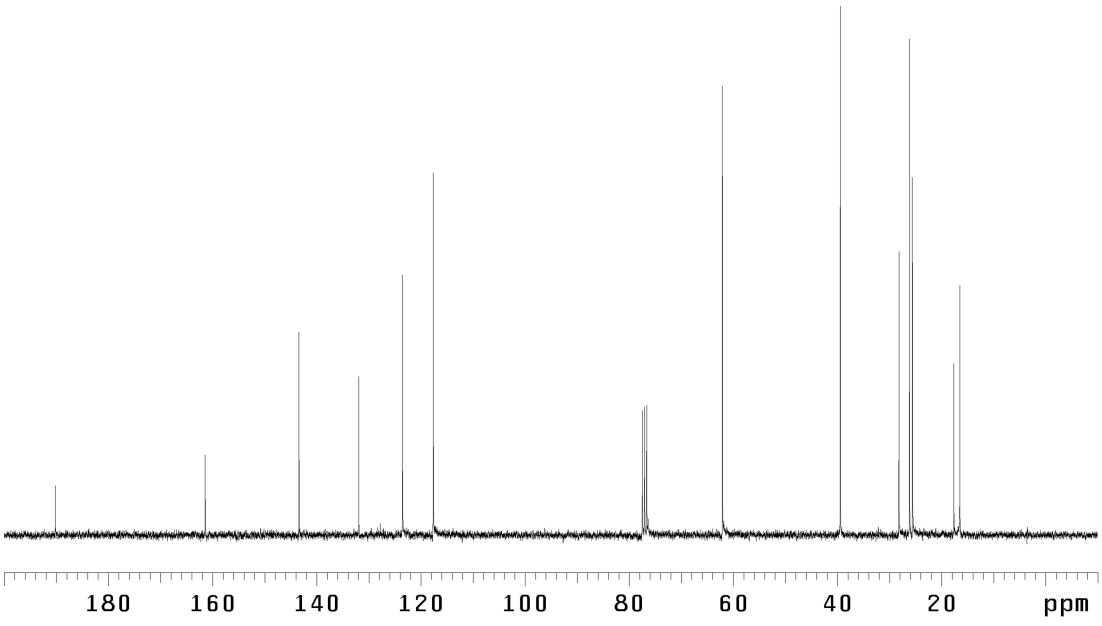


Figure A2.2.33 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **155**

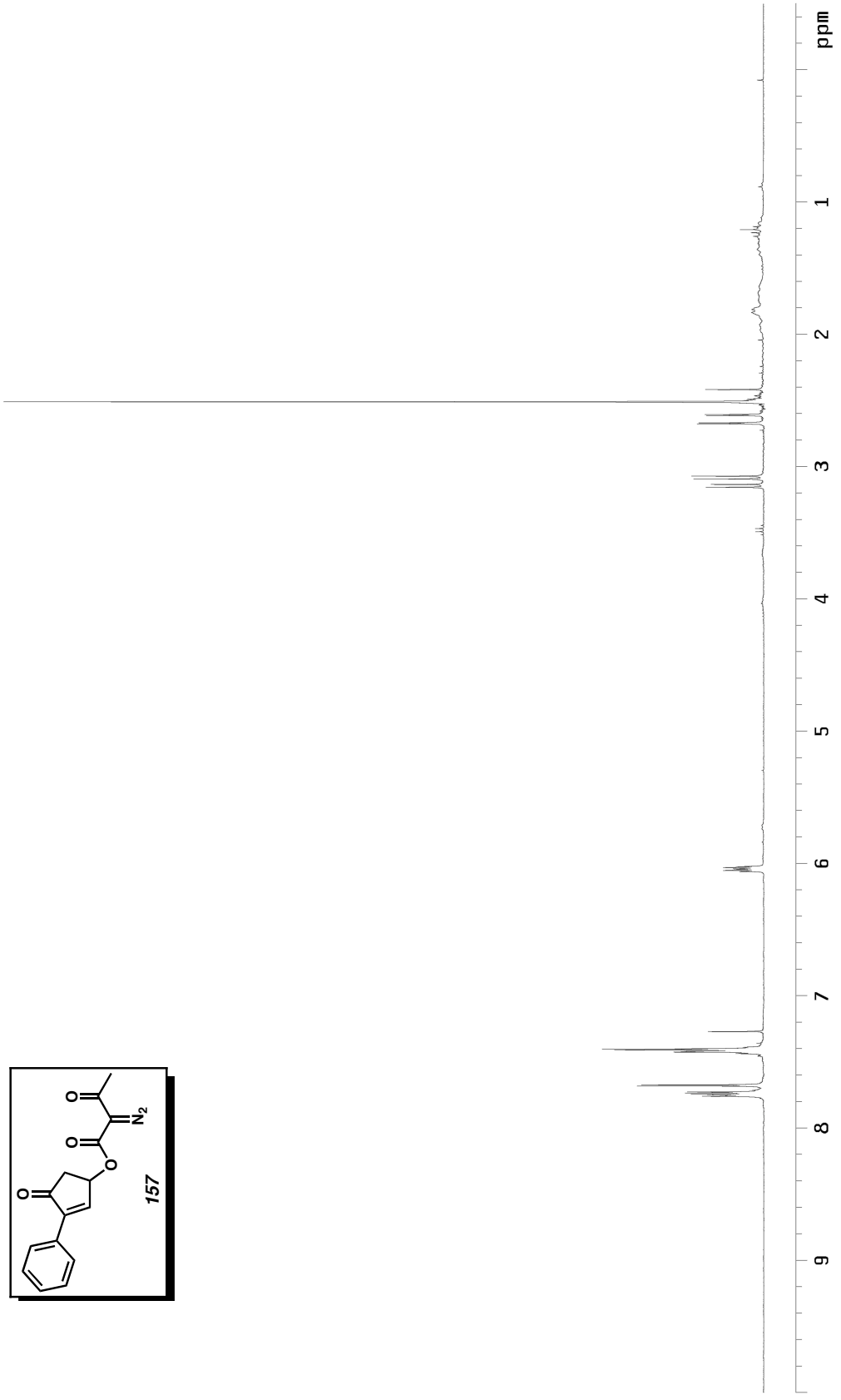


Figure A2.2.34 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **157**

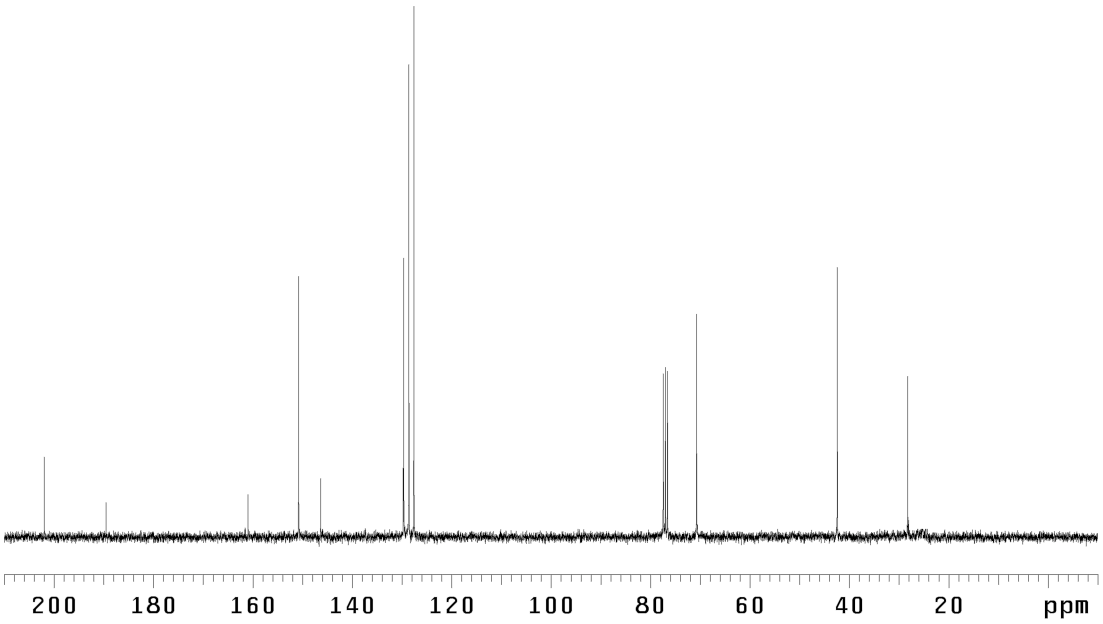


Figure A2.2.35 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **157**

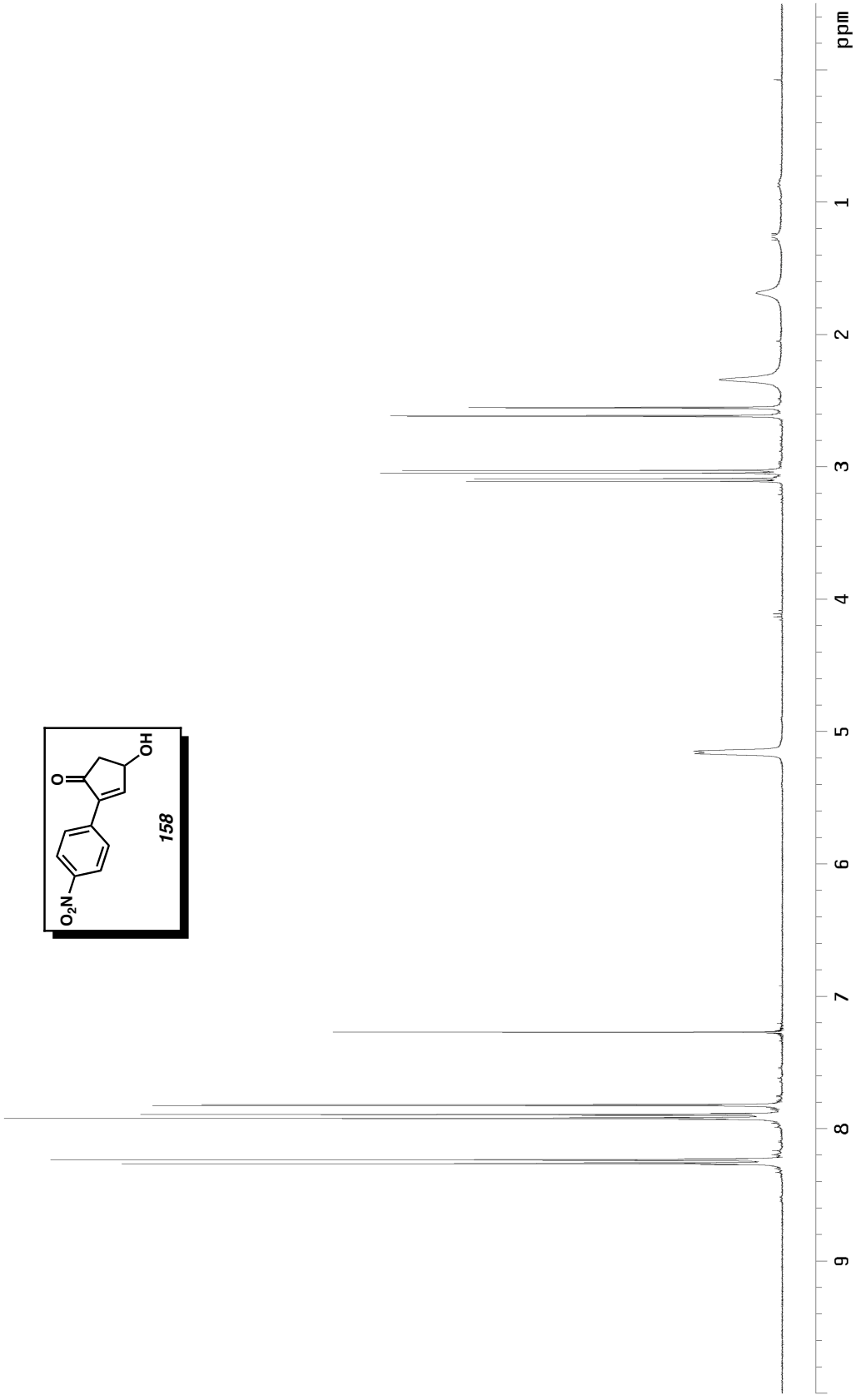


Figure A2.2.36 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **158**

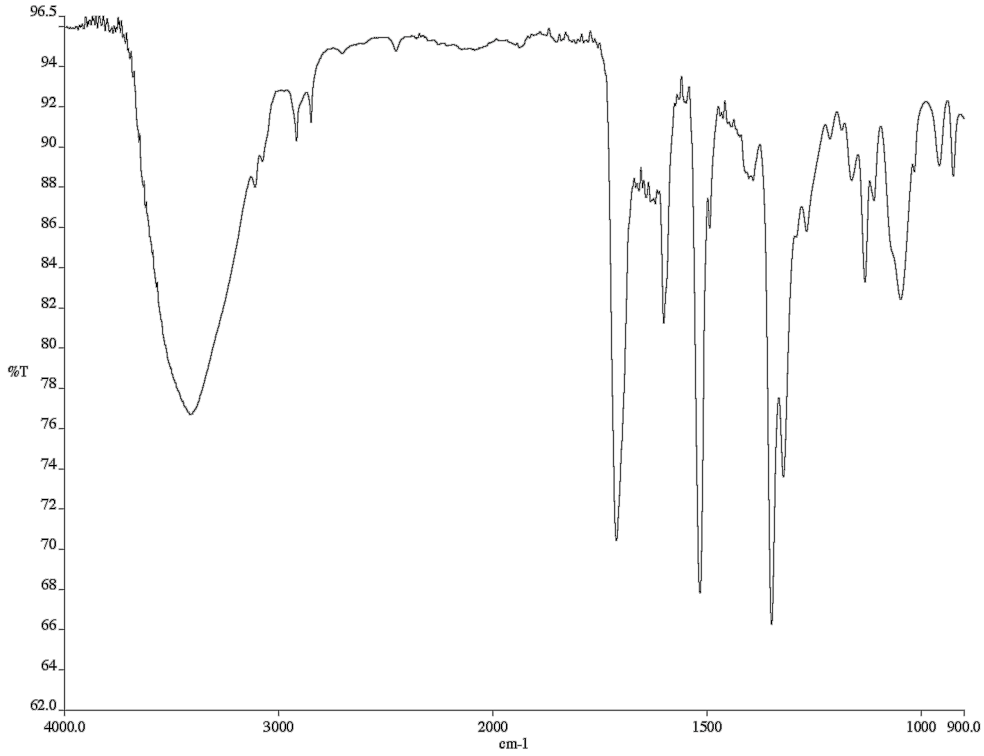


Figure A2.2.37 Infrared spectrum (film/NaCl) of compound **158**

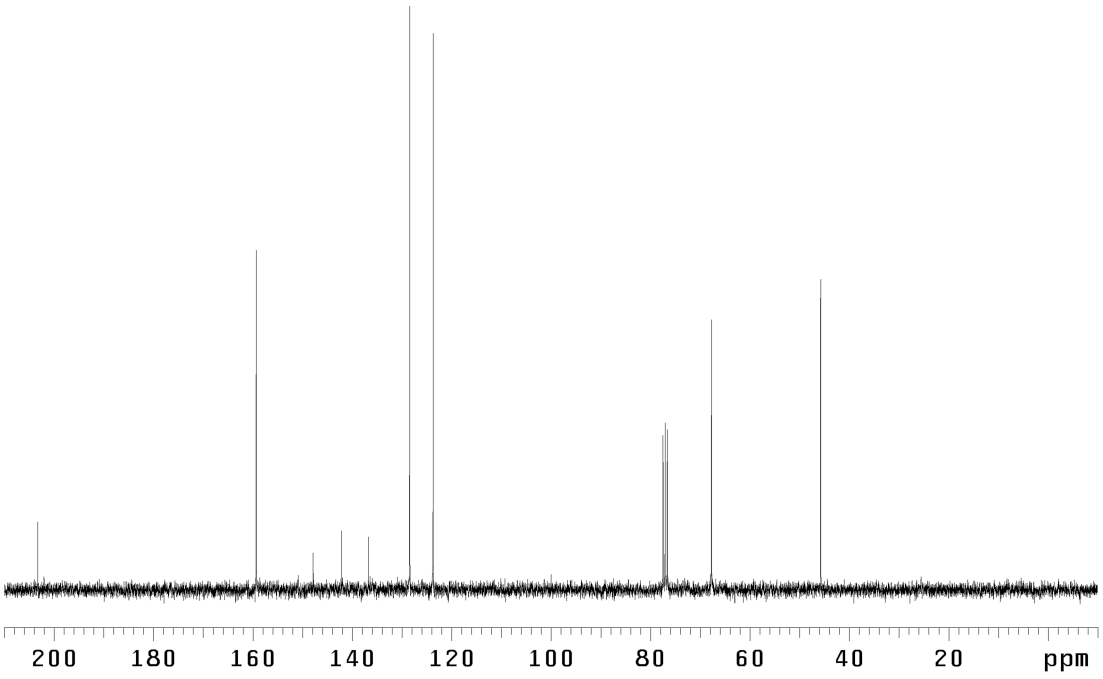


Figure A2.2.38 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **158**

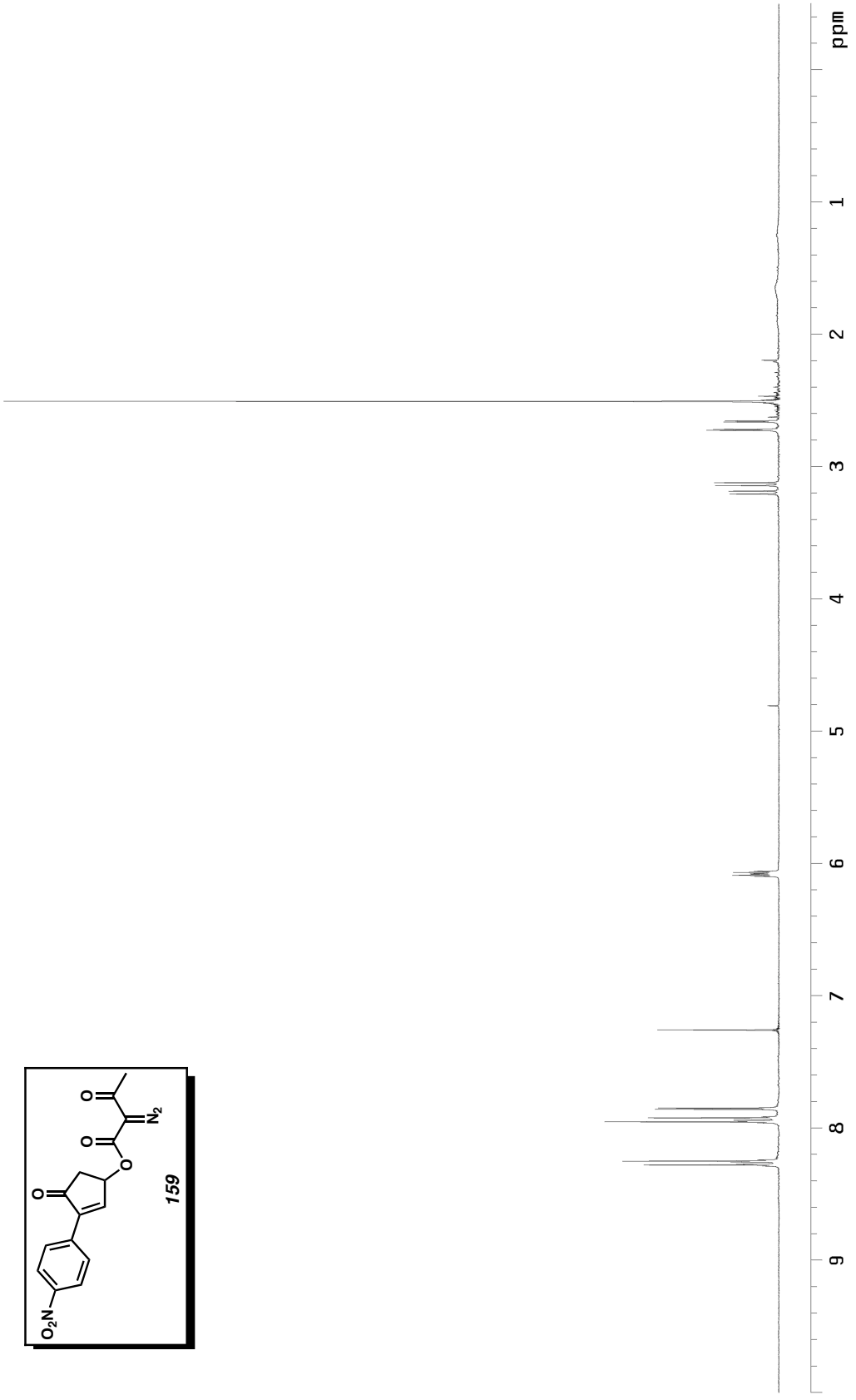


Figure A2.2.39 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **159**

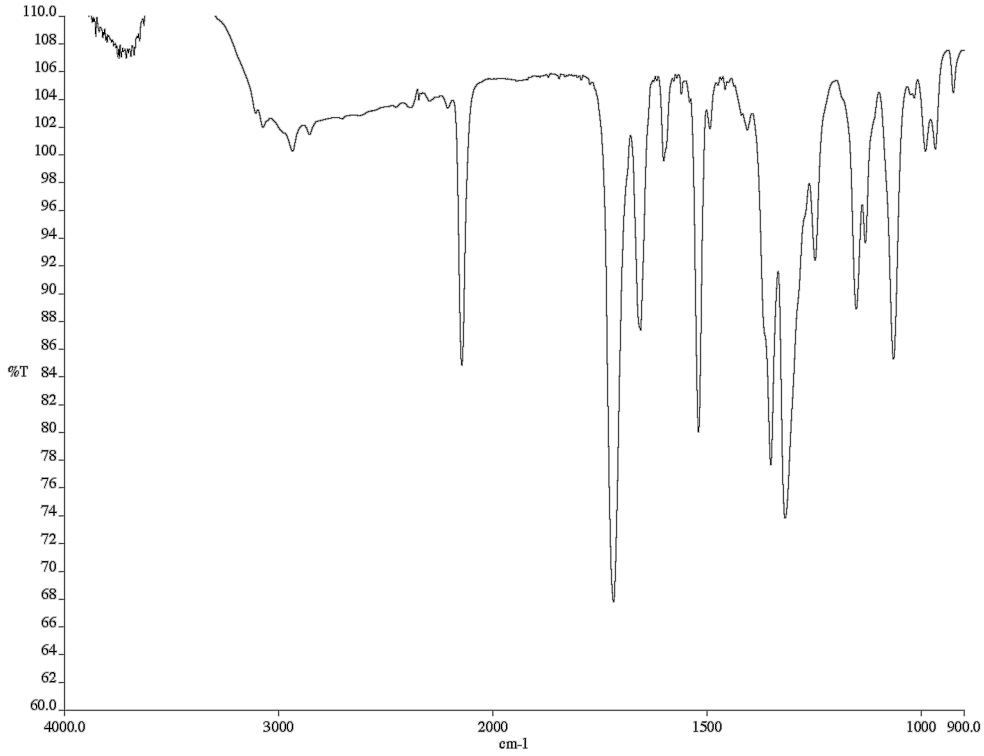


Figure A2.2.40 Infrared spectrum (film/NaCl) of compound **159**

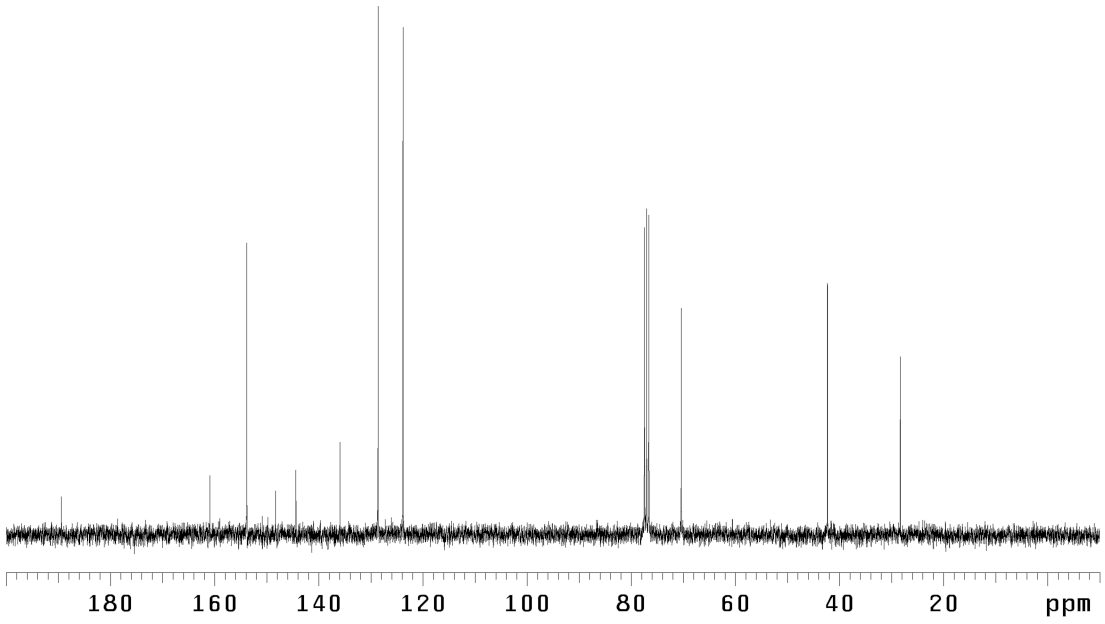


Figure A2.2.41 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **159**

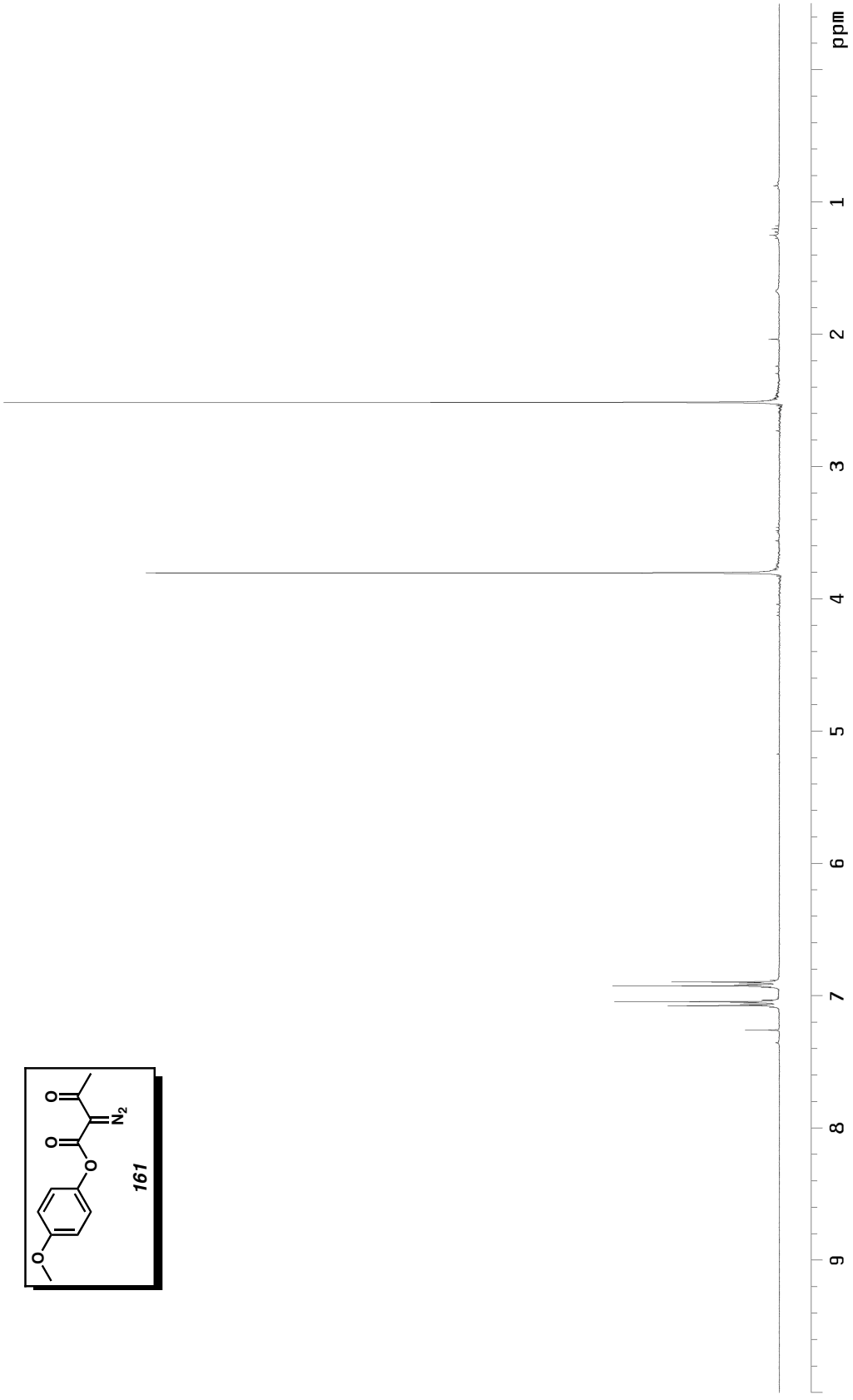


Figure A2.2.42 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **161**

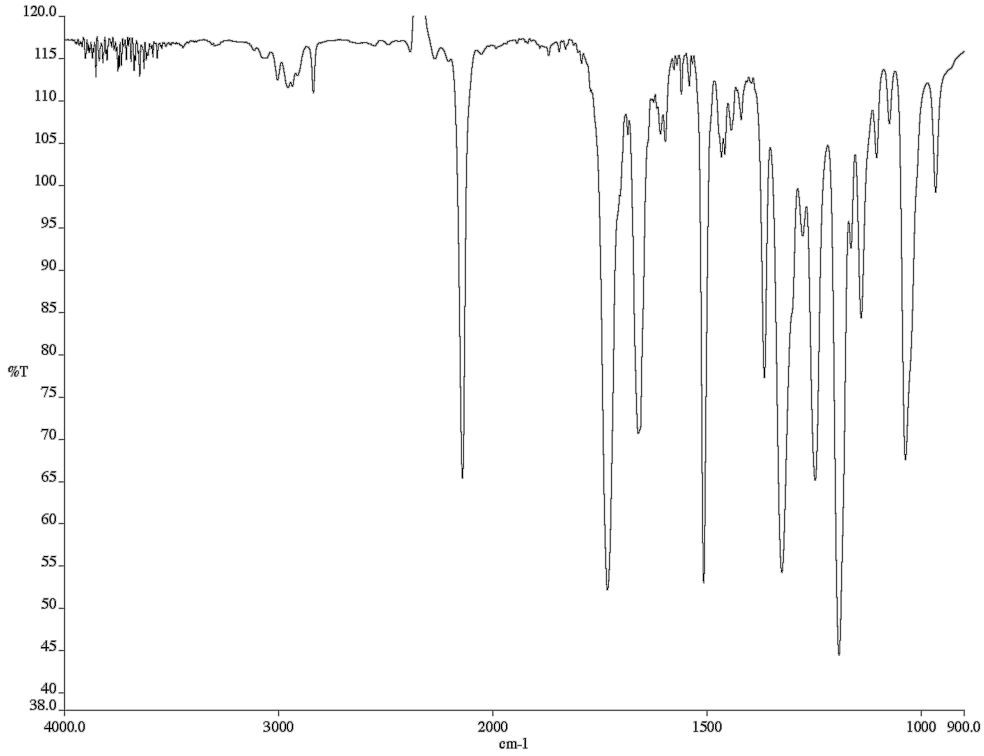


Figure A2.2.43 Infrared spectrum (film/NaCl) of compound **161**

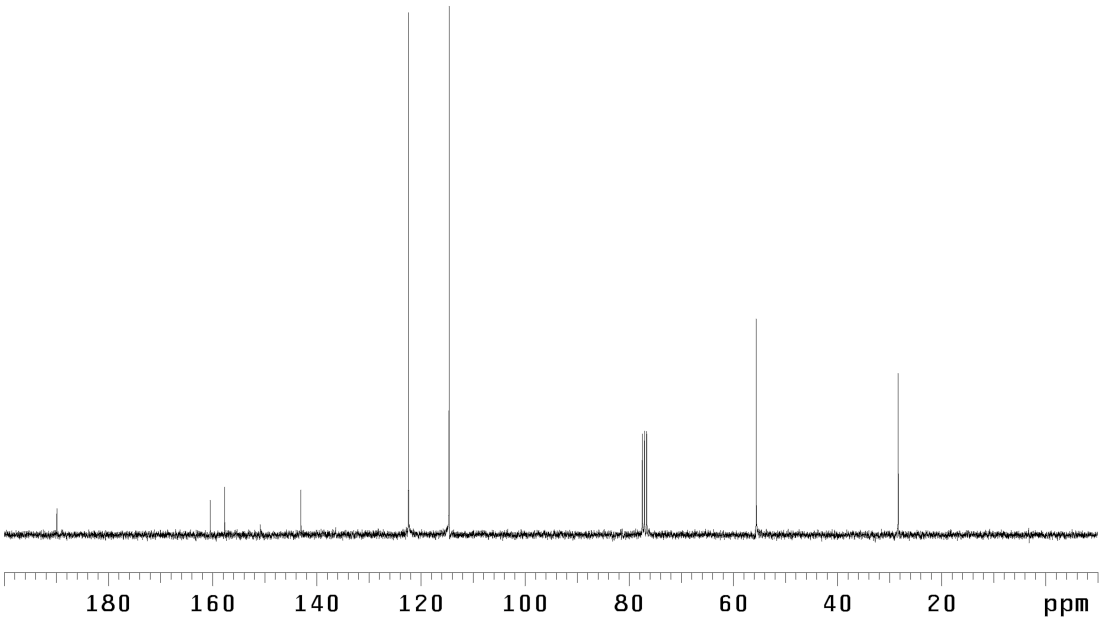


Figure A2.2.44 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **161**

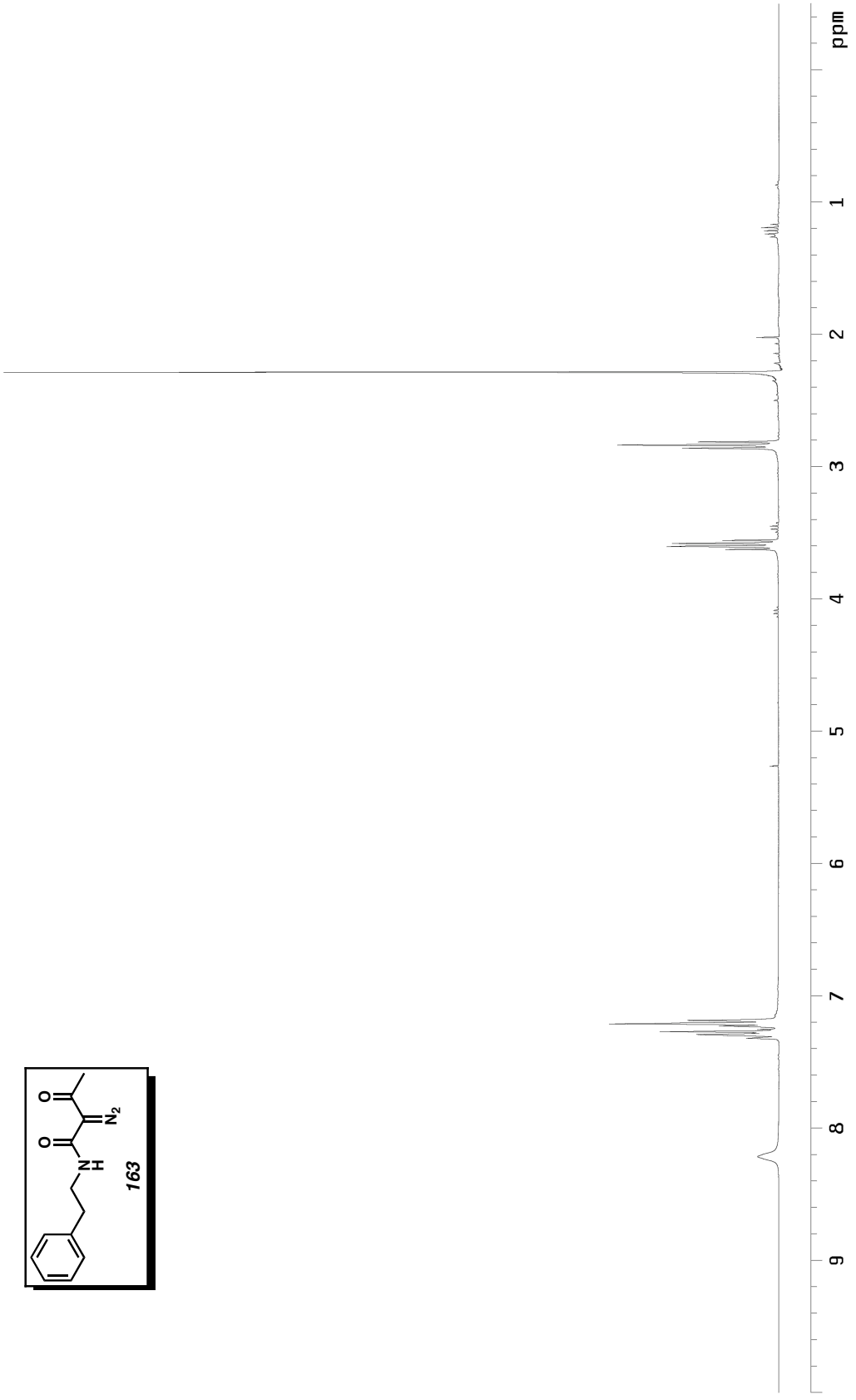


Figure A2.2.45 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **163**

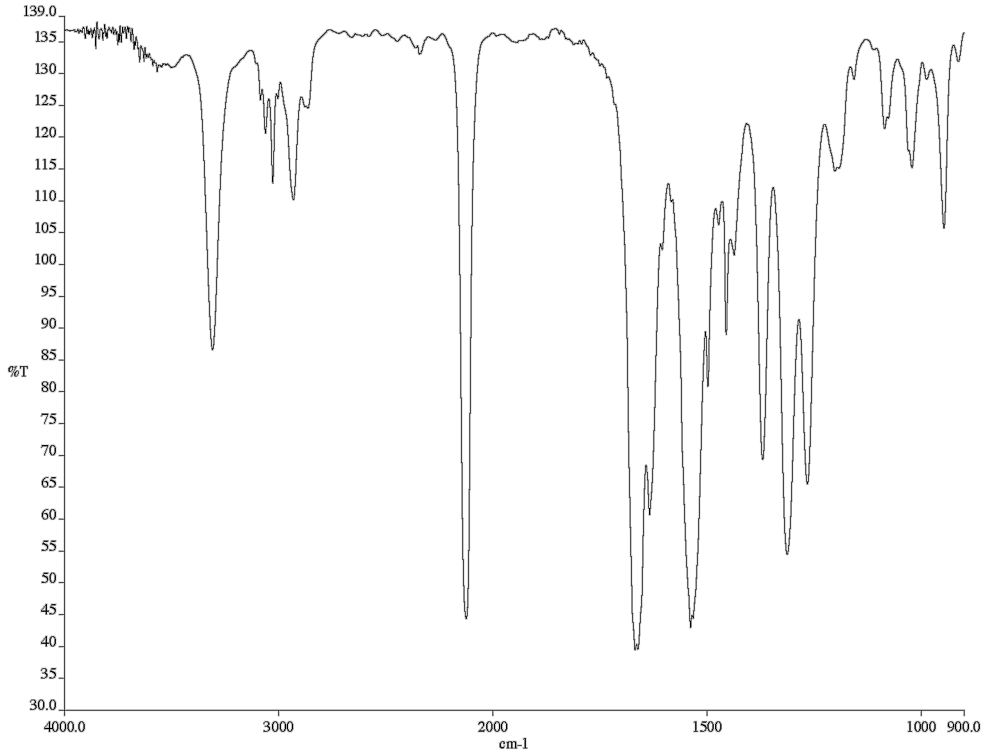


Figure A2.2.46 Infrared spectrum (film/NaCl) of compound **163**

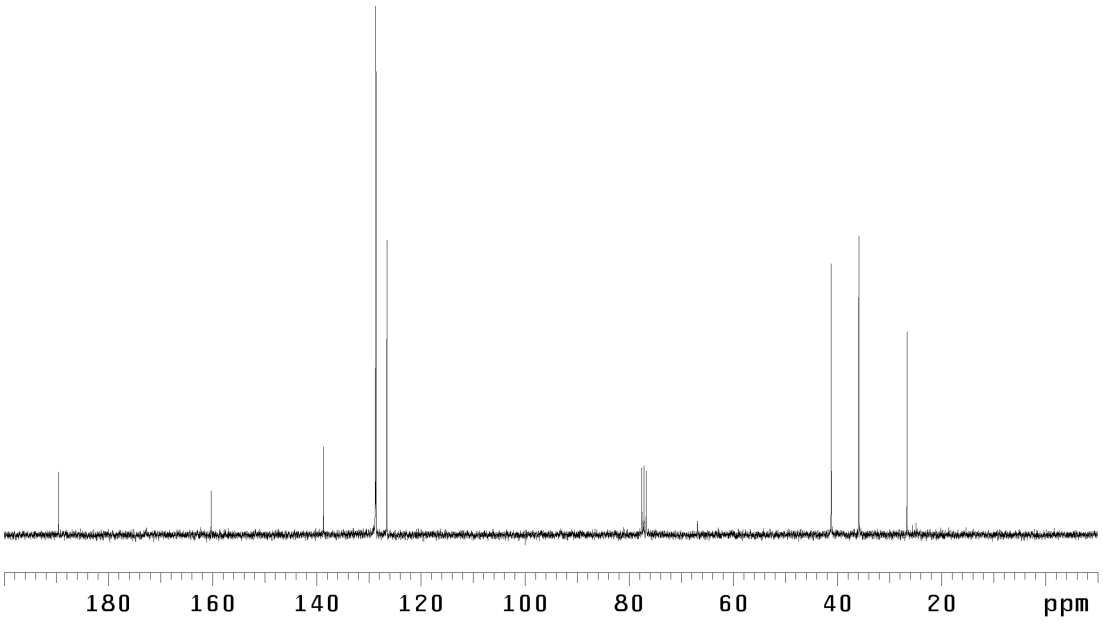


Figure A2.2.47 ^{13}C NMR (75 MHz, $CDCl_3$) of compound **163**

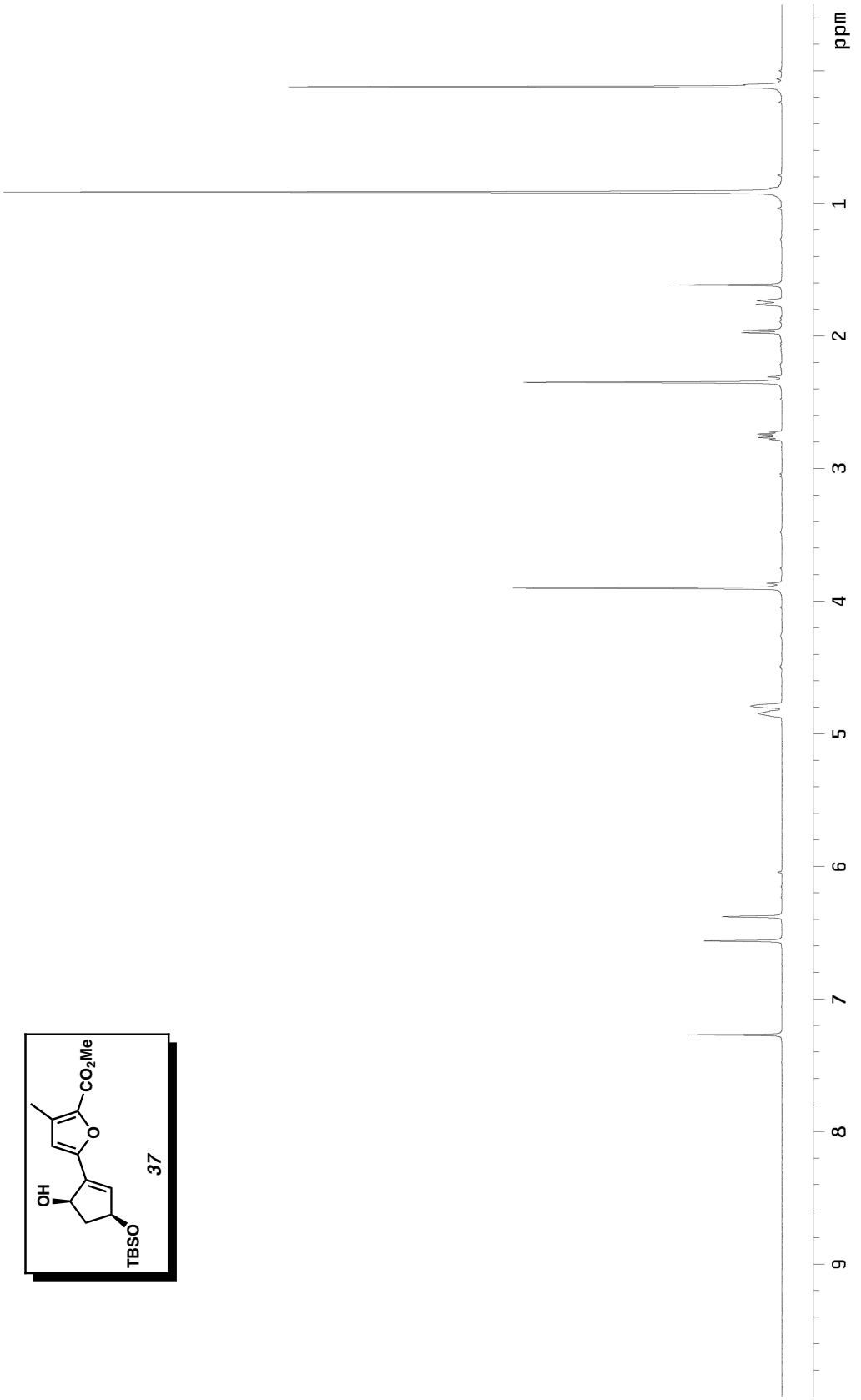


Figure A2.2.48 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **37**

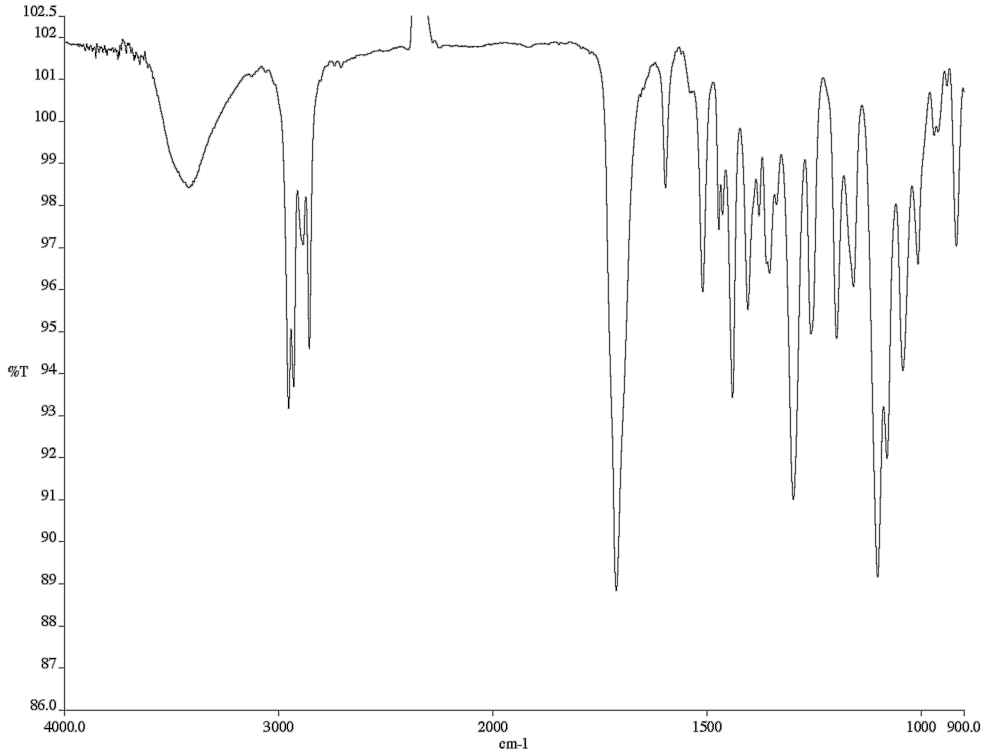


Figure A2.2.49 Infrared spectrum (film/NaCl) of compound **37**

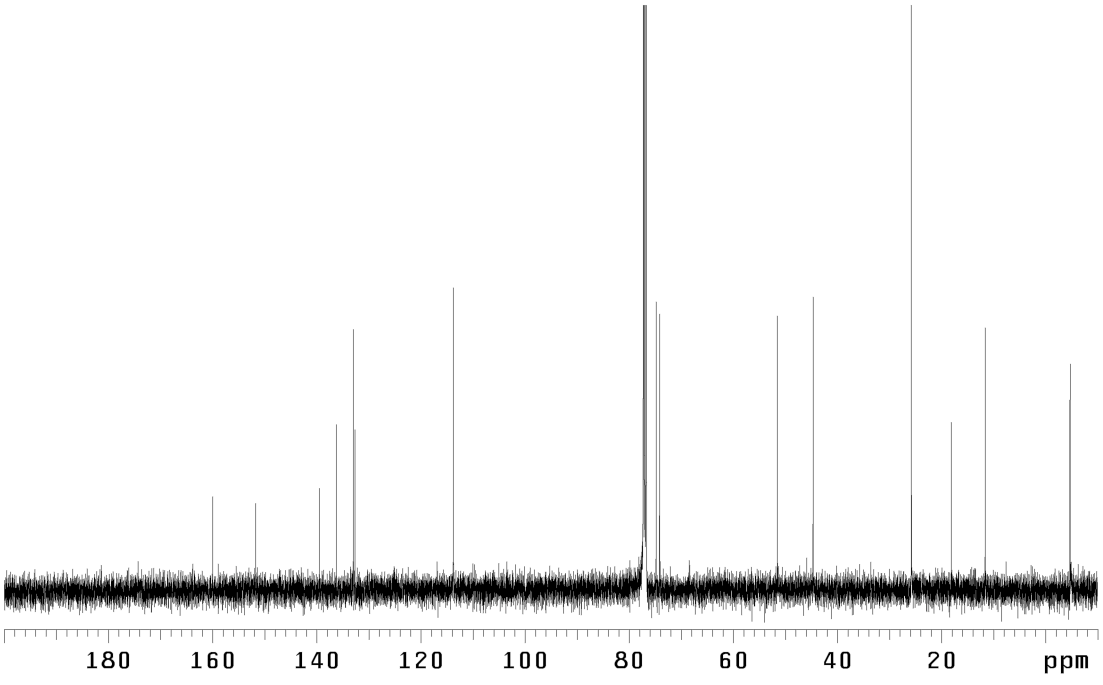


Figure A2.2.50 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **37**

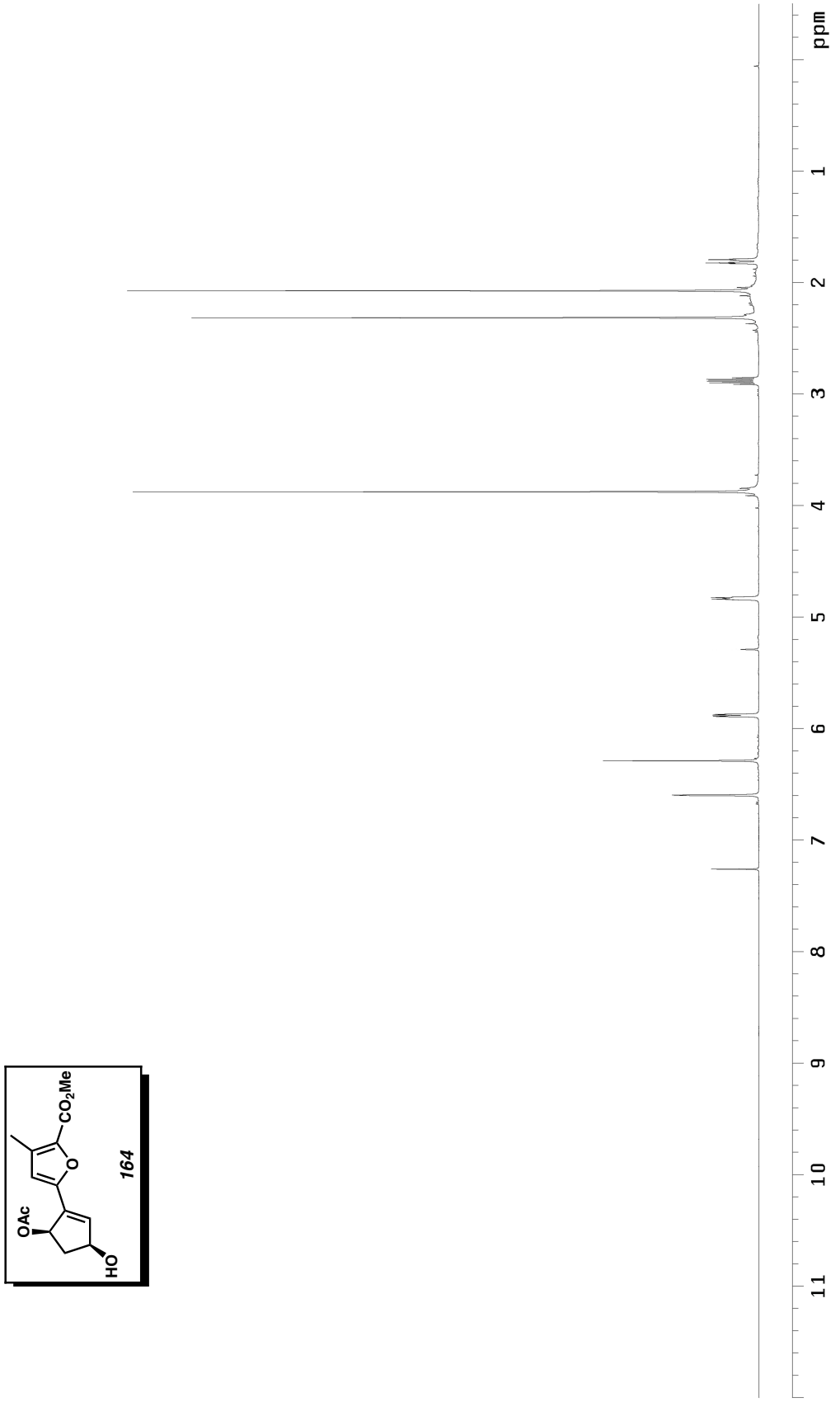


Figure A2.2.51 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **164**

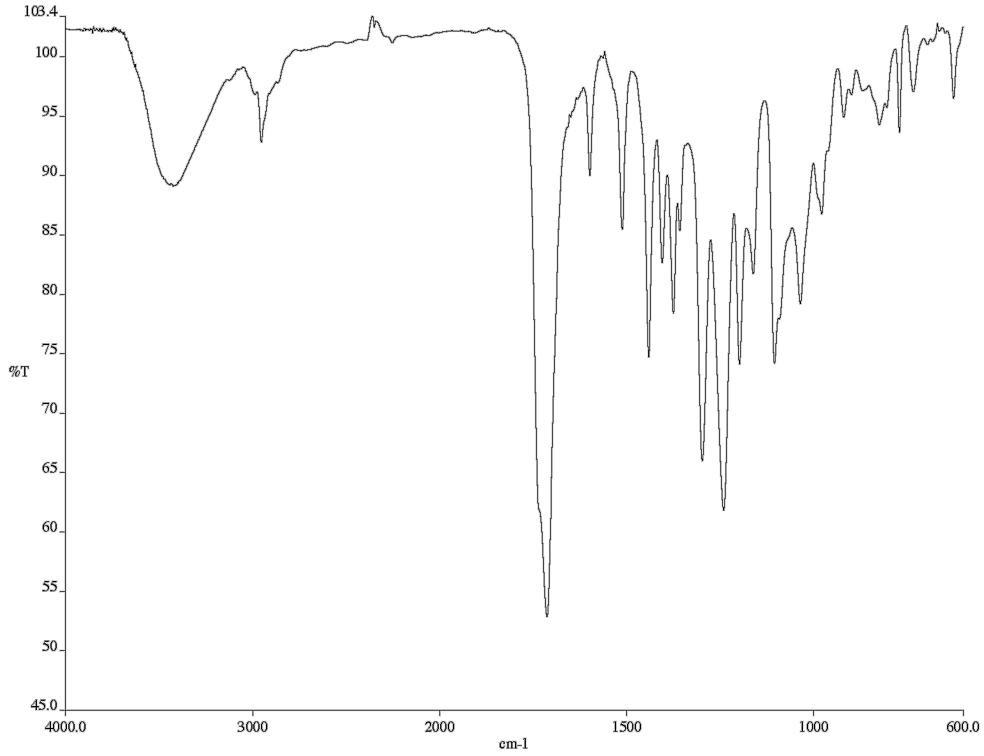


Figure A2.2.52 Infrared spectrum (film/NaCl) of compound **164**

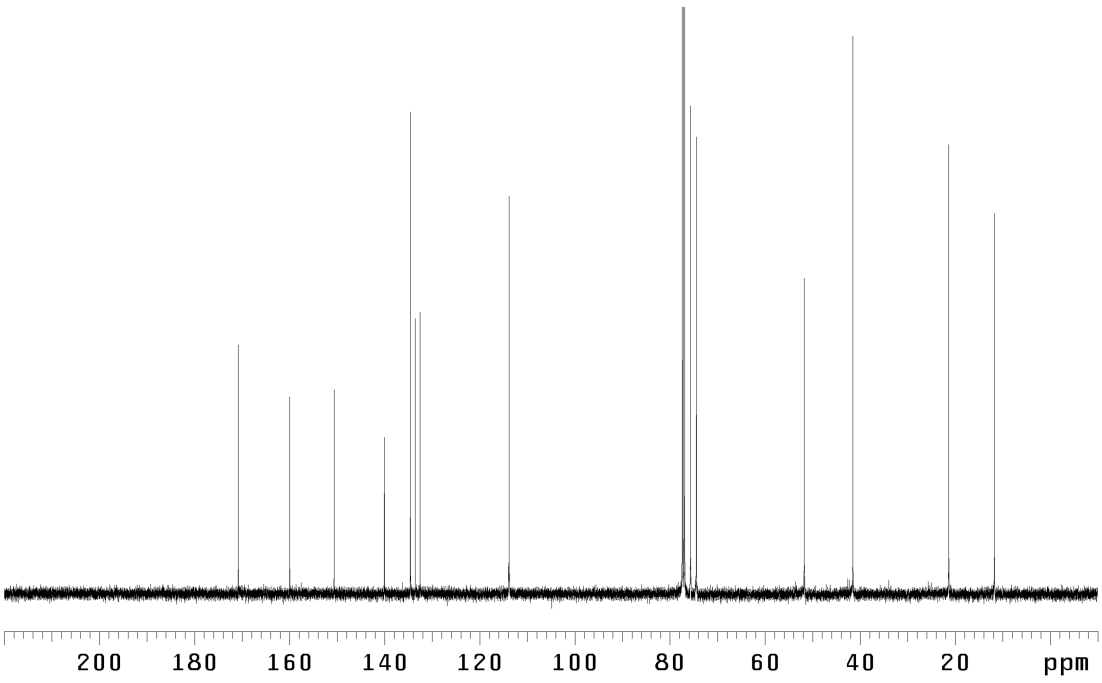


Figure A2.2.53 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **164**

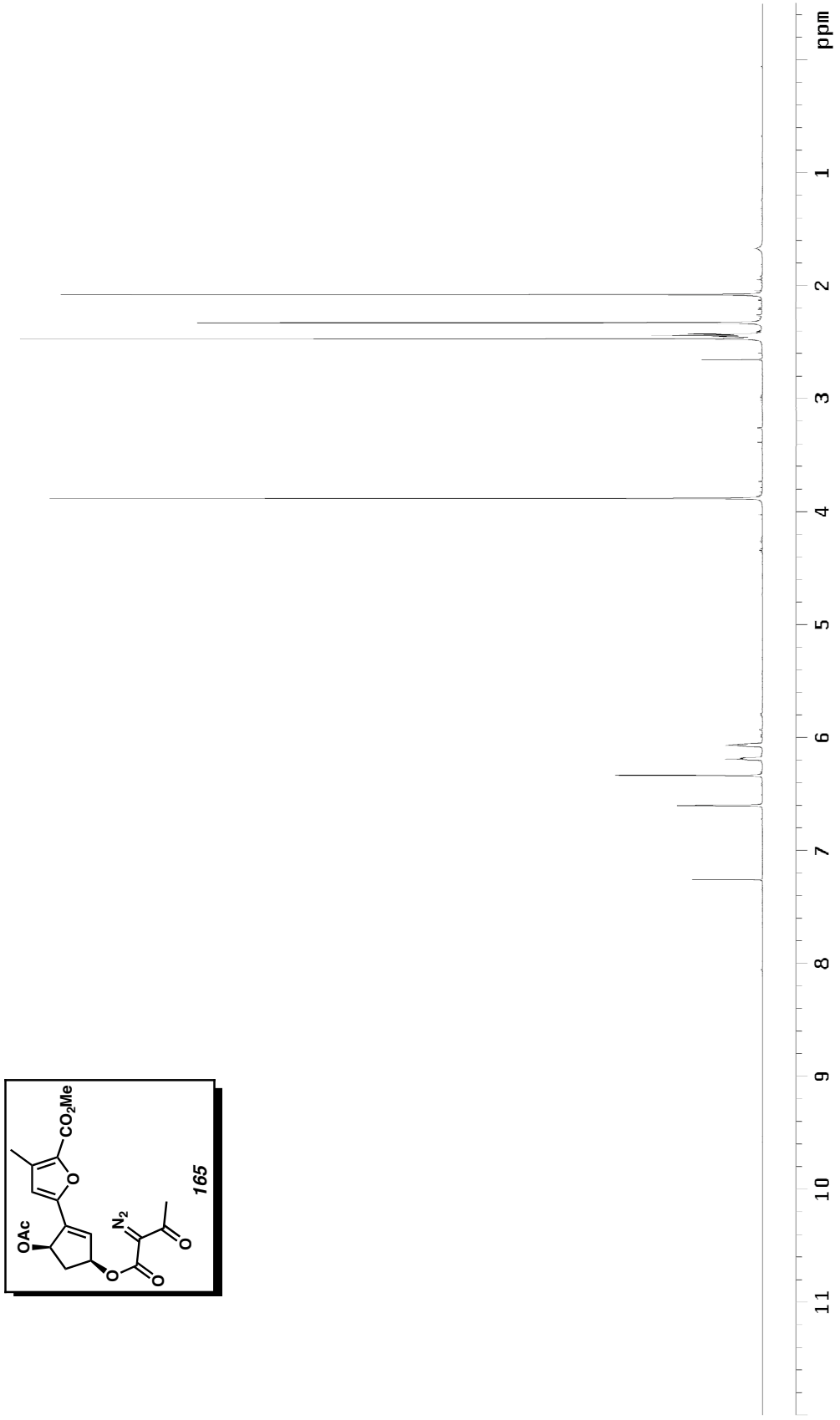


Figure A2.2.54 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **165**

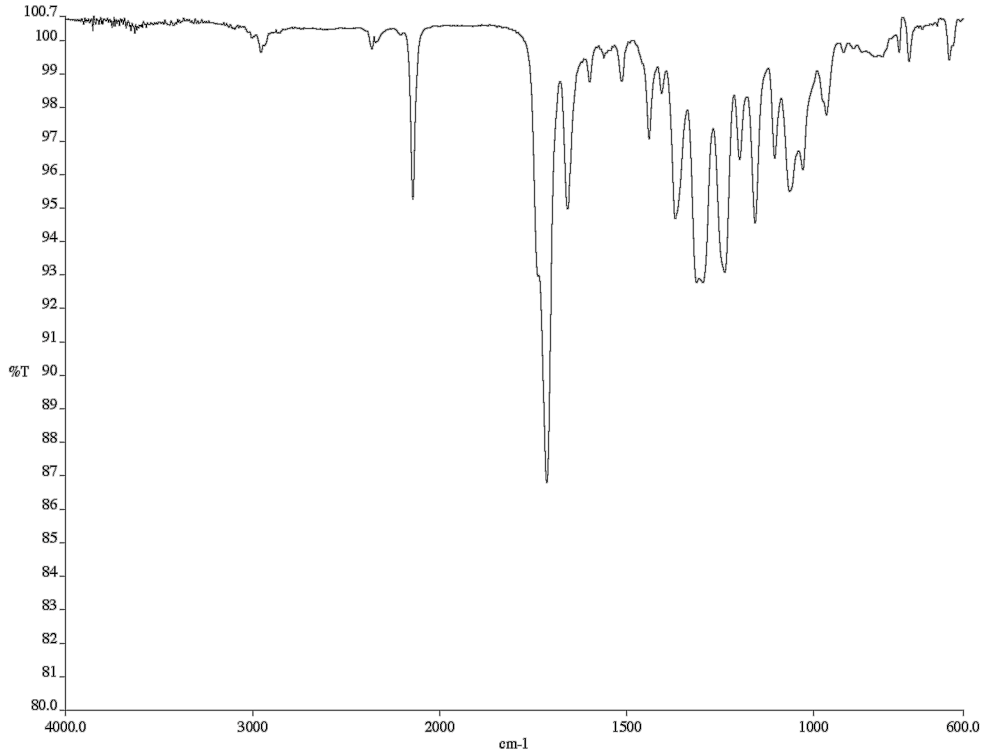


Figure A2.2.55 Infrared spectrum (film/NaCl) of compound **164**

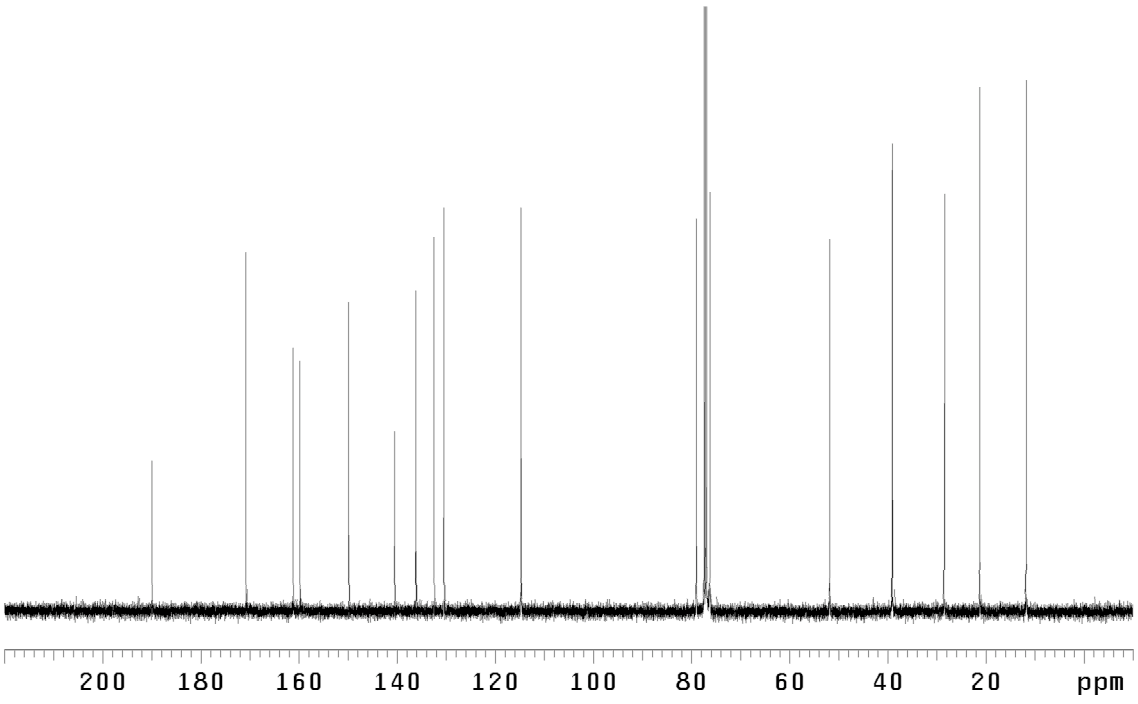


Figure A2.2.56 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **164**

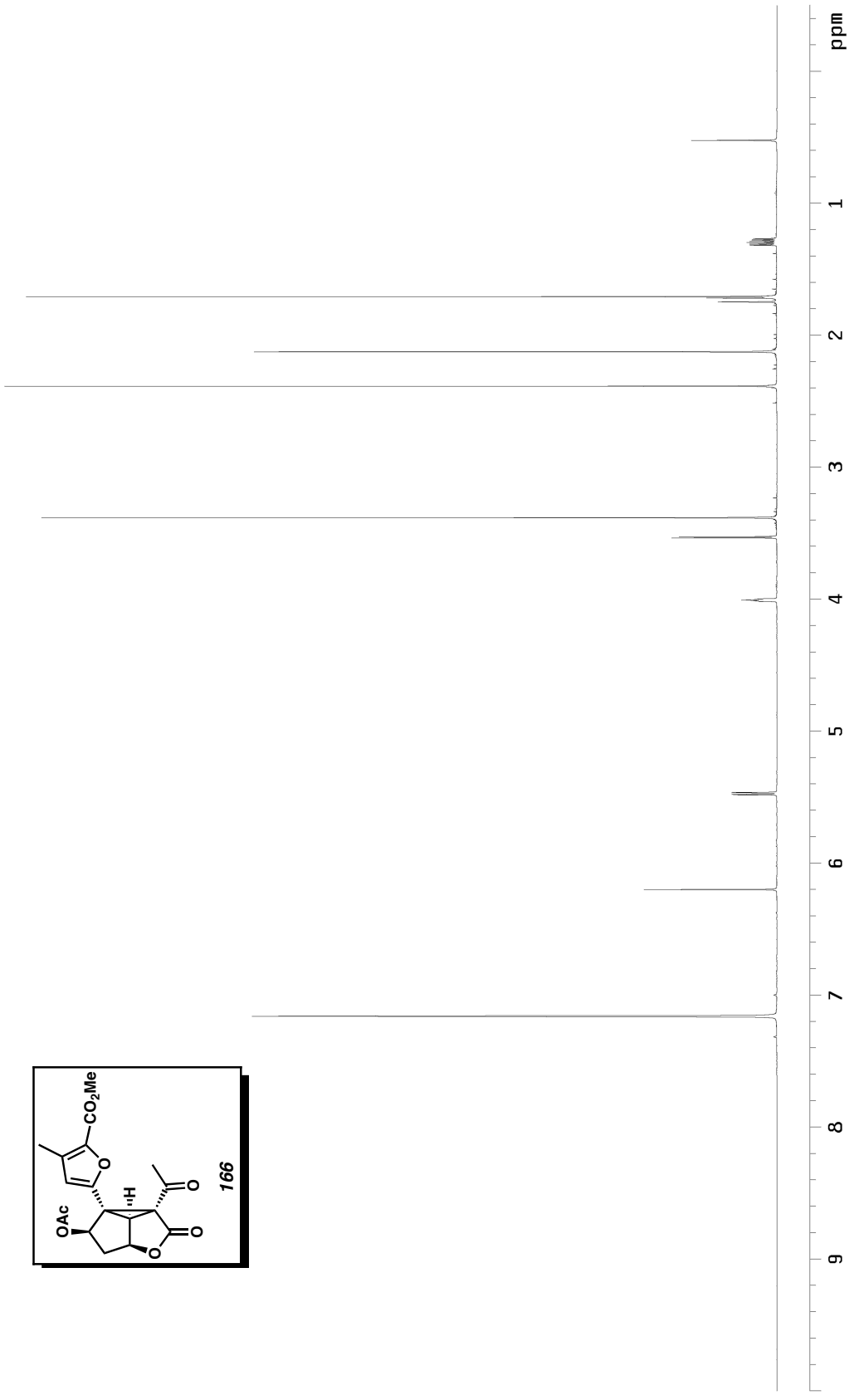


Figure A2.2.57 ^{1}H NMR (500 MHz, C_6D_6) of compound **166**

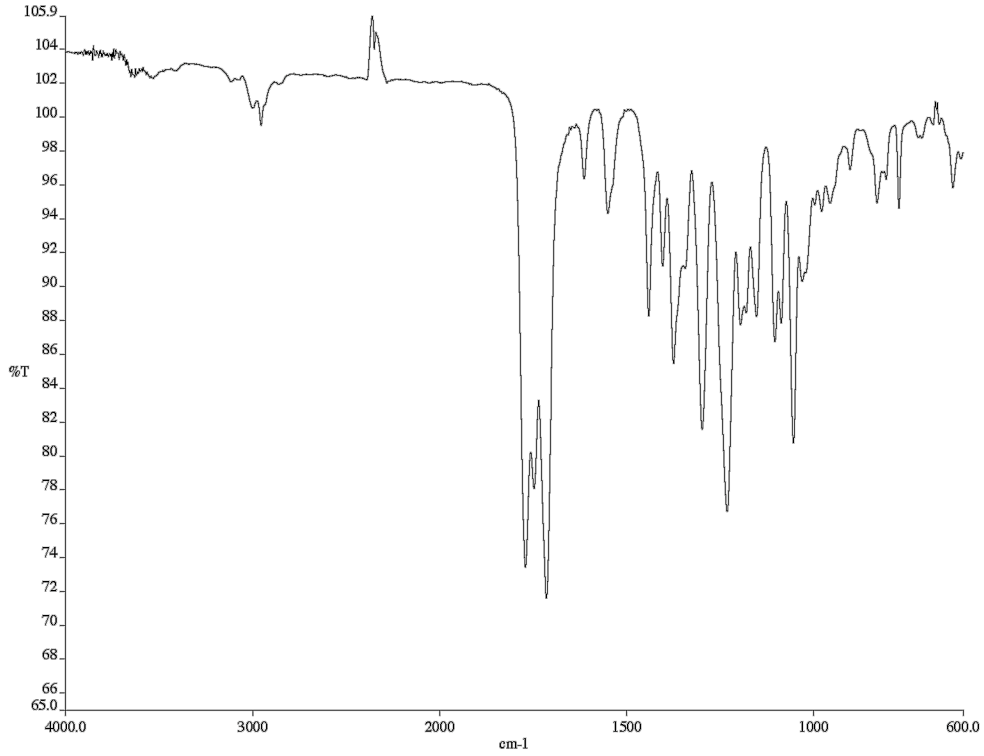


Figure A2.2.58 Infrared spectrum (film/NaCl) of compound **166**

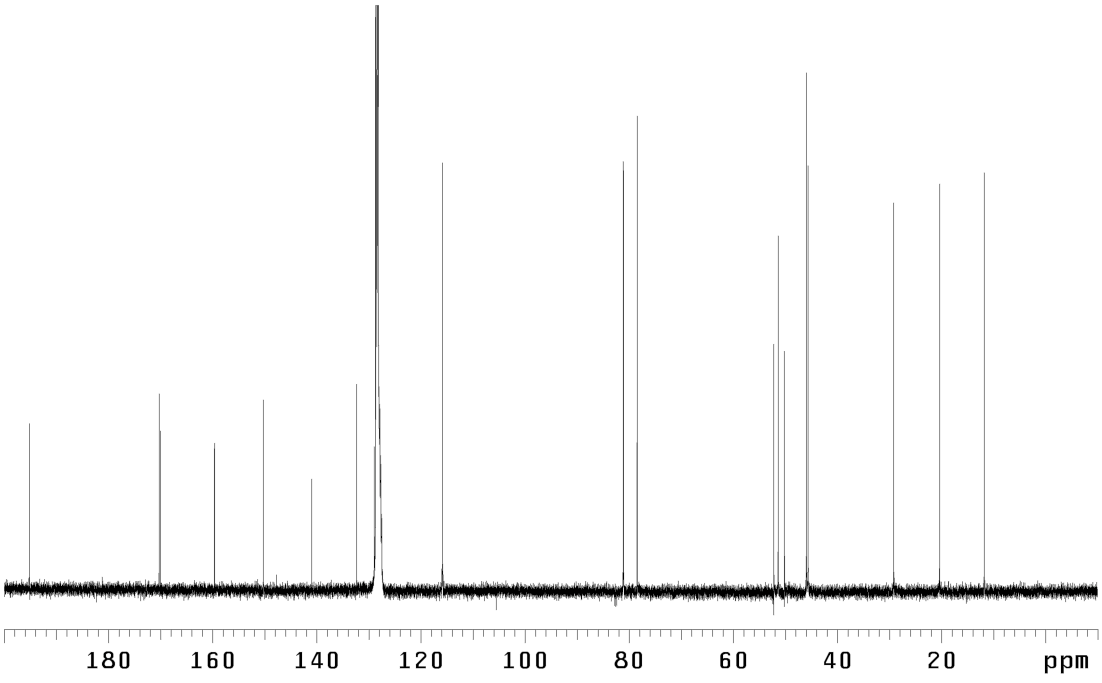


Figure A2.2.59 ^{13}C NMR (125 MHz, C_6D_6) of compound **166**

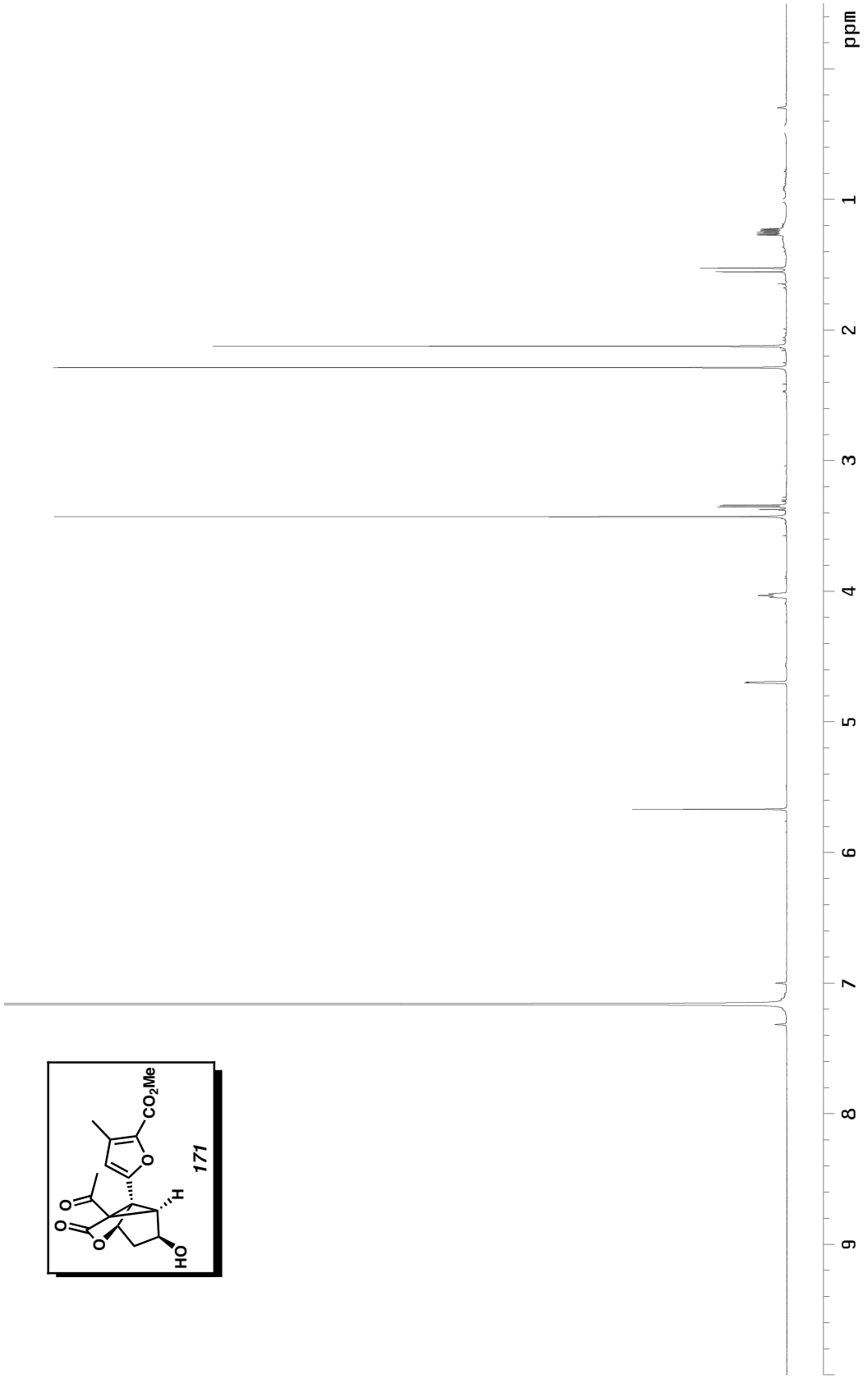


Figure A2.2.60 ^{1}H NMR (500 MHz, C_6D_6) of compound **171**

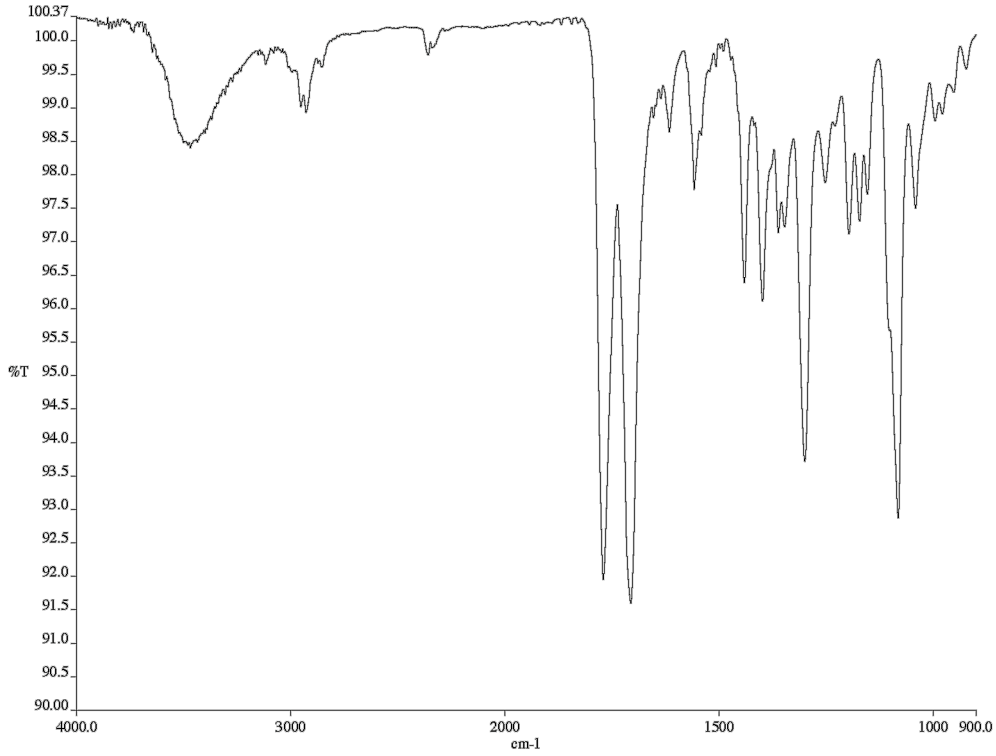


Figure A2.2.61 Infrared spectrum (film/NaCl) of compound **171**

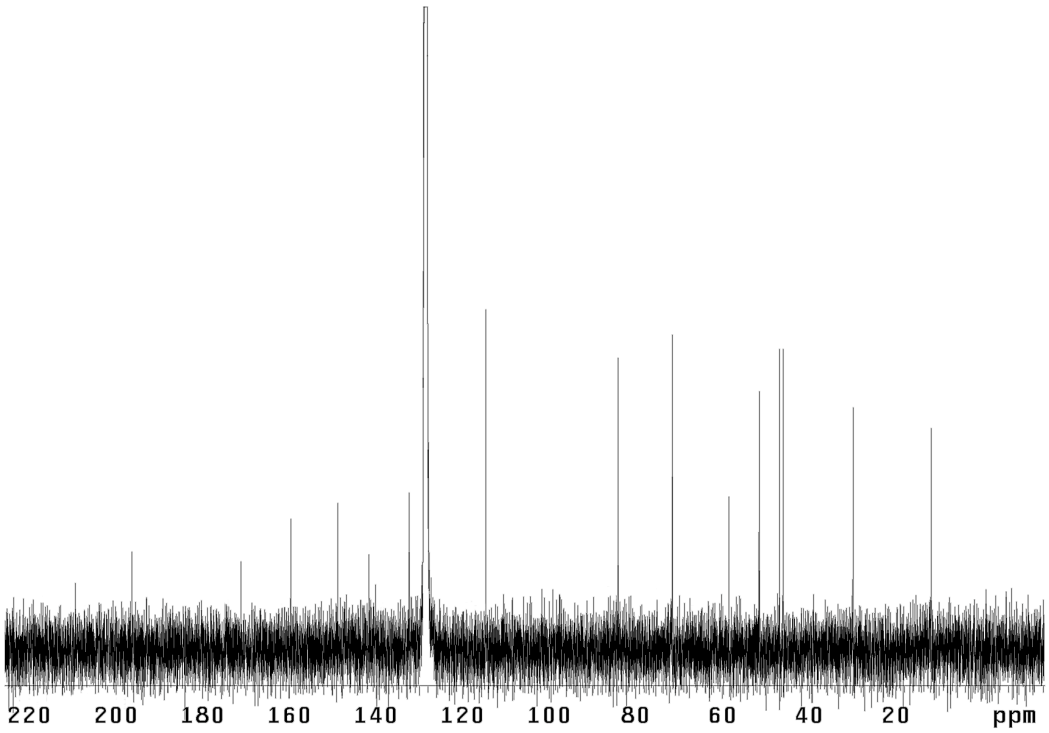


Figure A2.2.62 ^{13}C NMR (125 MHz, C_6D_6) of compound **171**

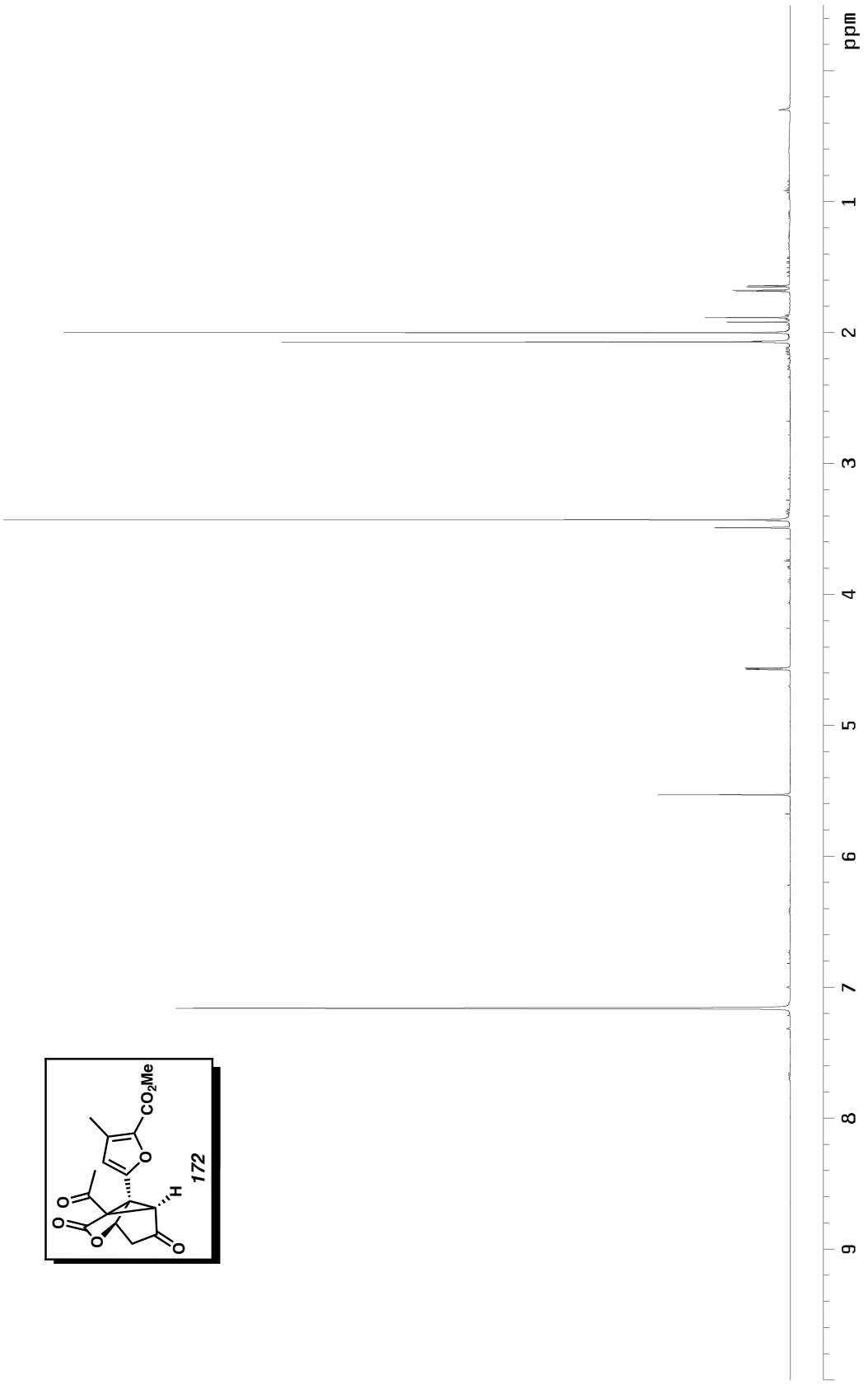


Figure A2.2.63 ^{1}H NMR (500 MHz, C_6D_6) of compound **172**

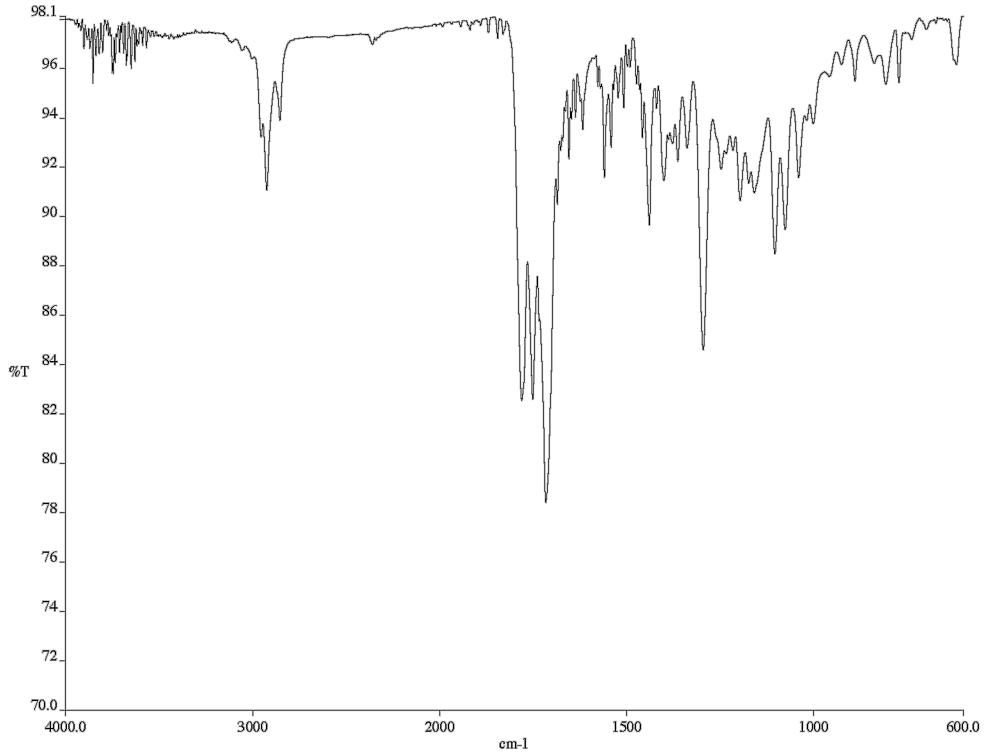


Figure A2.2.64 Infrared spectrum (film/NaCl) of compound **172**

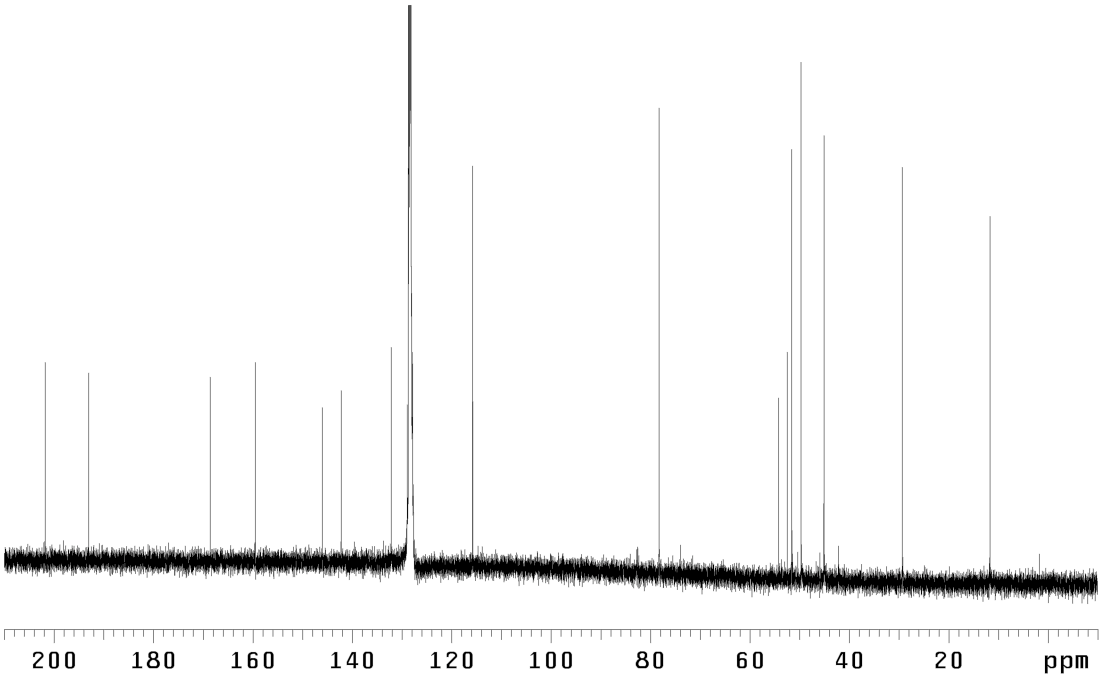


Figure A2.2.65 ^{13}C NMR (125 MHz, C_6D_6) of compound **172**

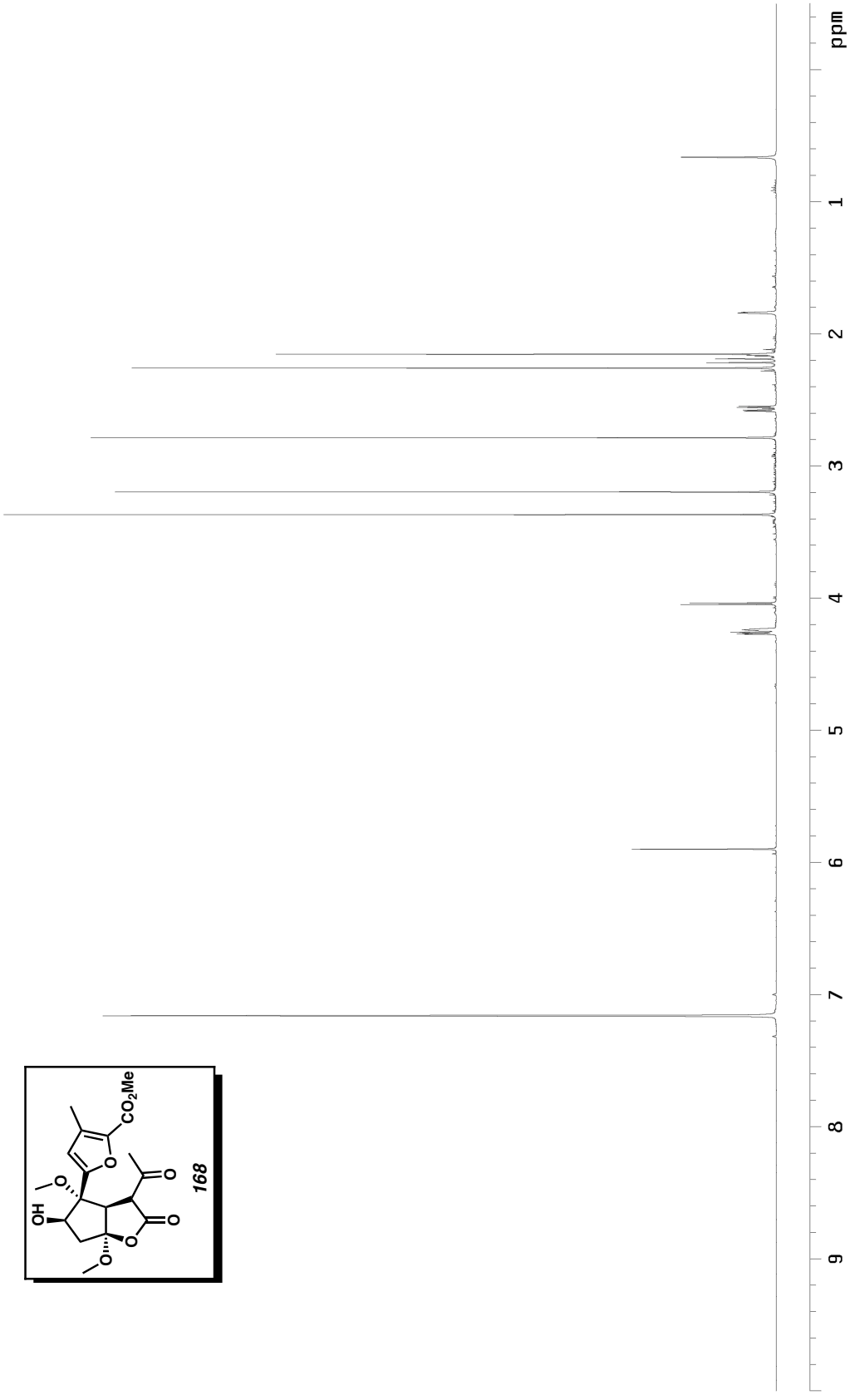


Figure A2.2.66 ^{1}H NMR (500 MHz, C_6D_6) of compound **168**

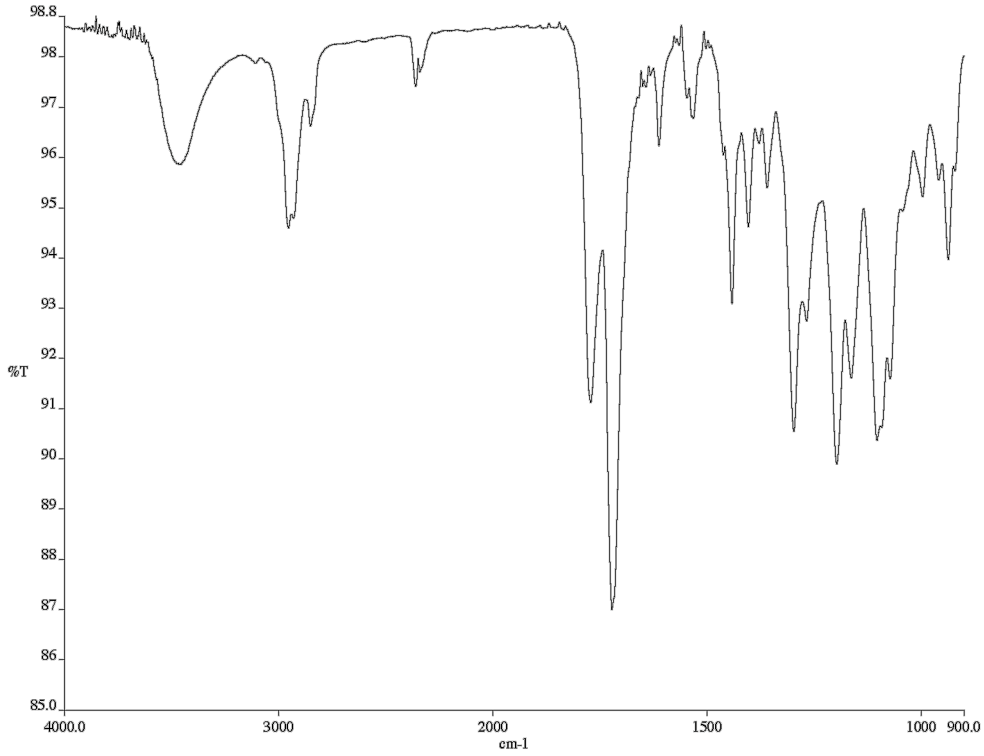


Figure A2.2.67 Infrared spectrum (film/NaCl) of compound **168**

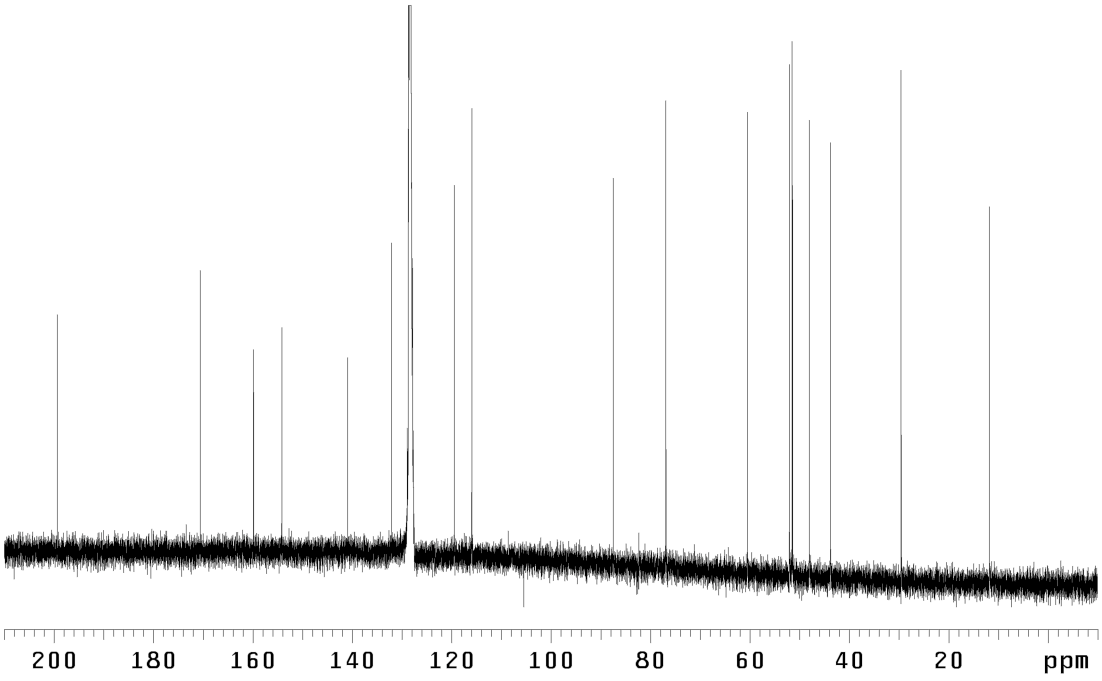


Figure A2.2.68 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **168**

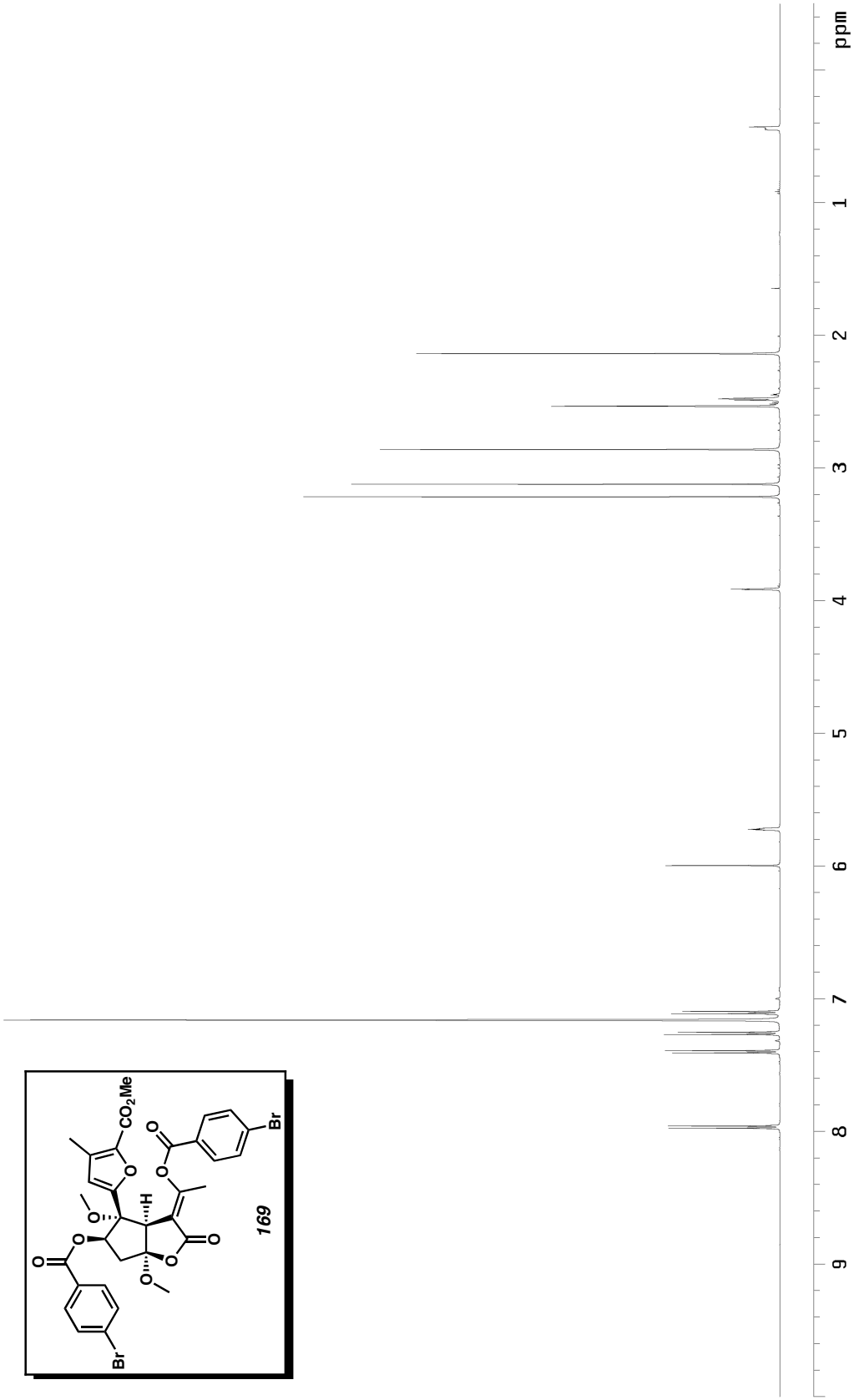


Figure A2.2.69 ^{1}H NMR (500 MHz, C_6D_6) of compound **169**

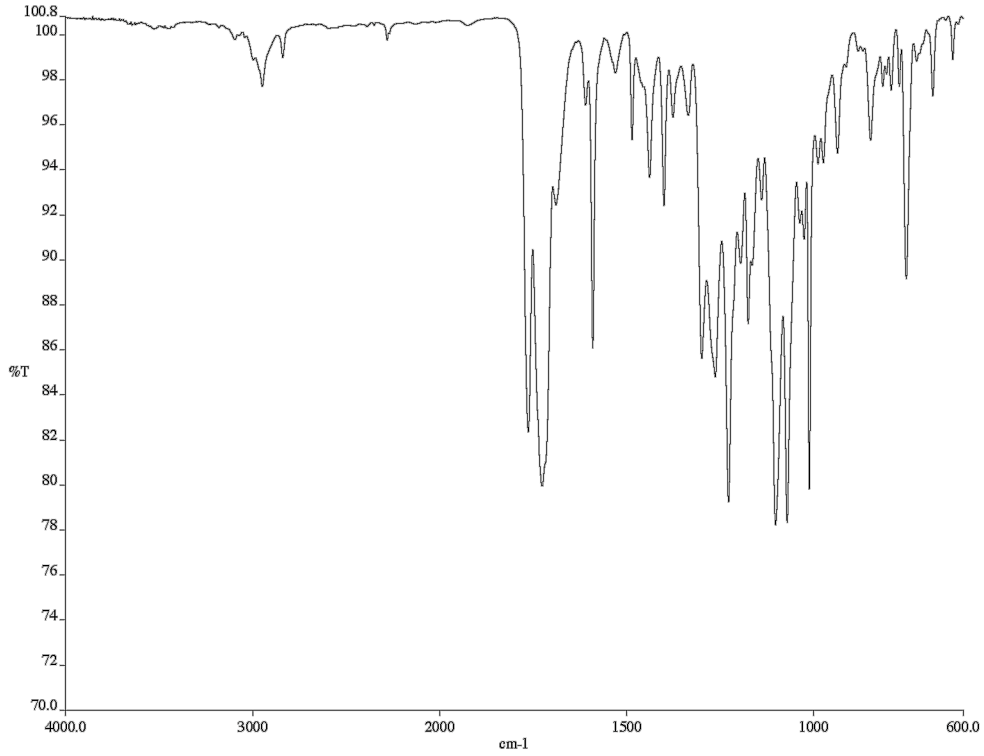


Figure A2.2.70 Infrared spectrum (film/NaCl) of compound **169**

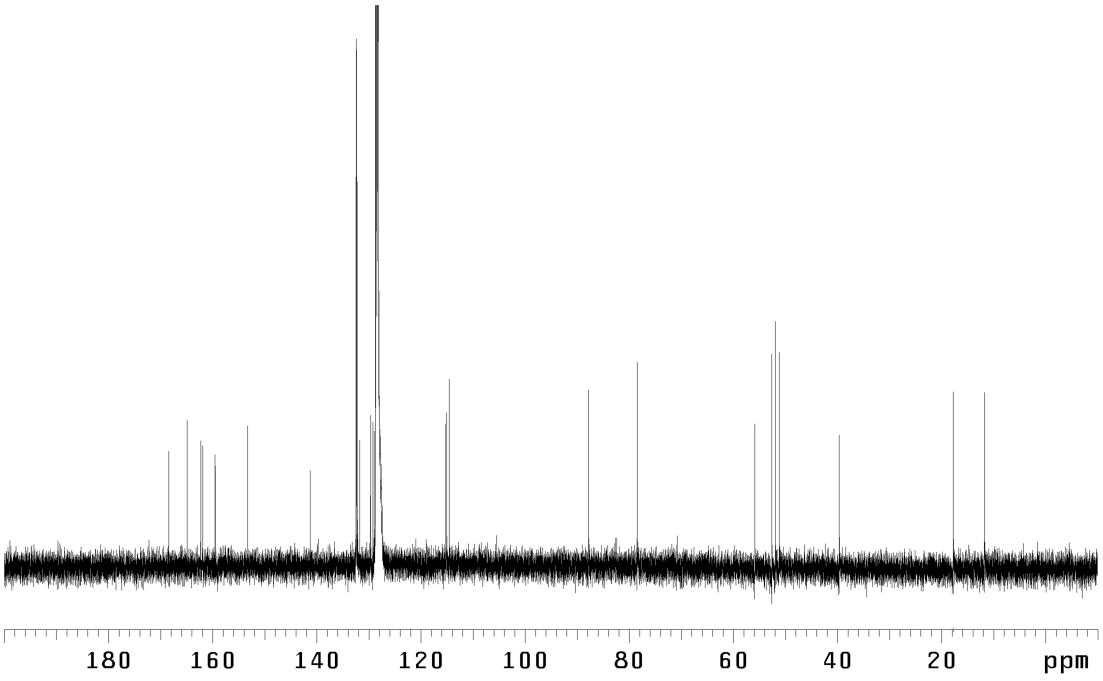


Figure A2.2.71 ^{13}C NMR (125 MHz, C_6D_6) of compound **169**

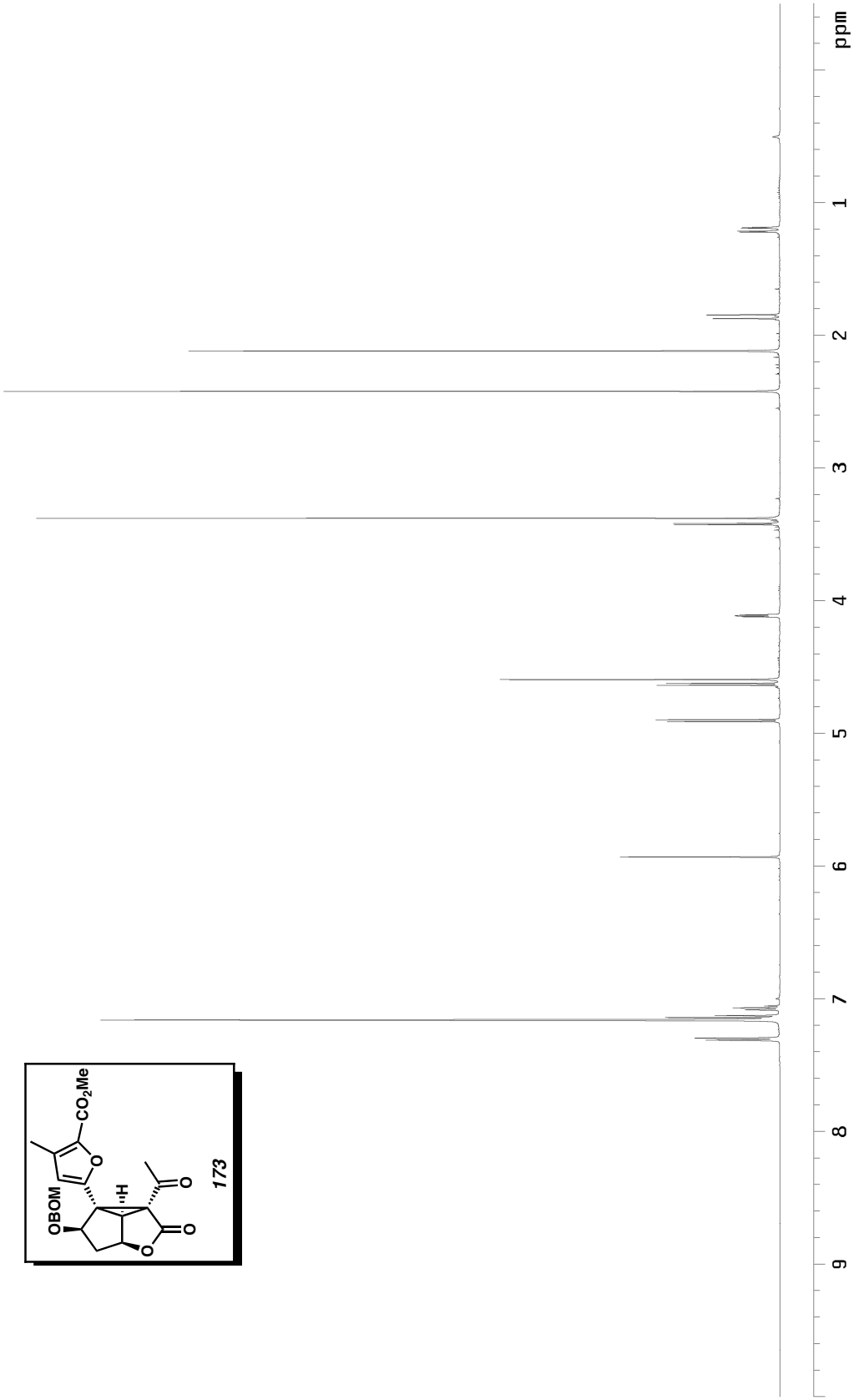


Figure A2.2.72 ^{1}H NMR (500 MHz, C_6D_6) of compound **173**

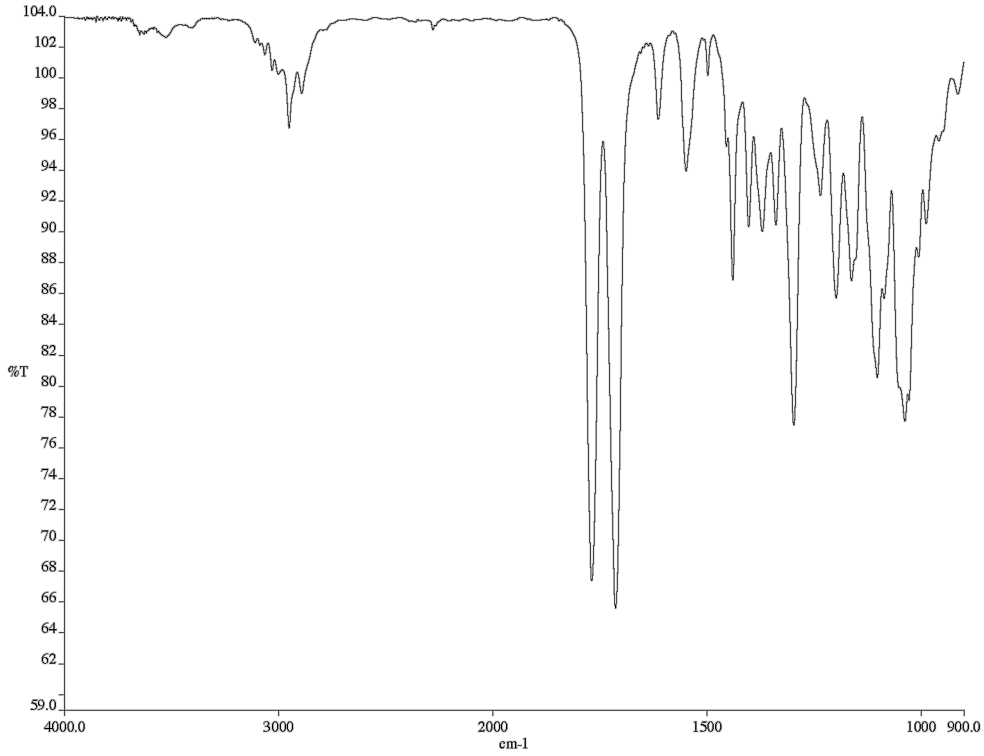


Figure A2.2.73 Infrared spectrum (film/NaCl) of compound **173**

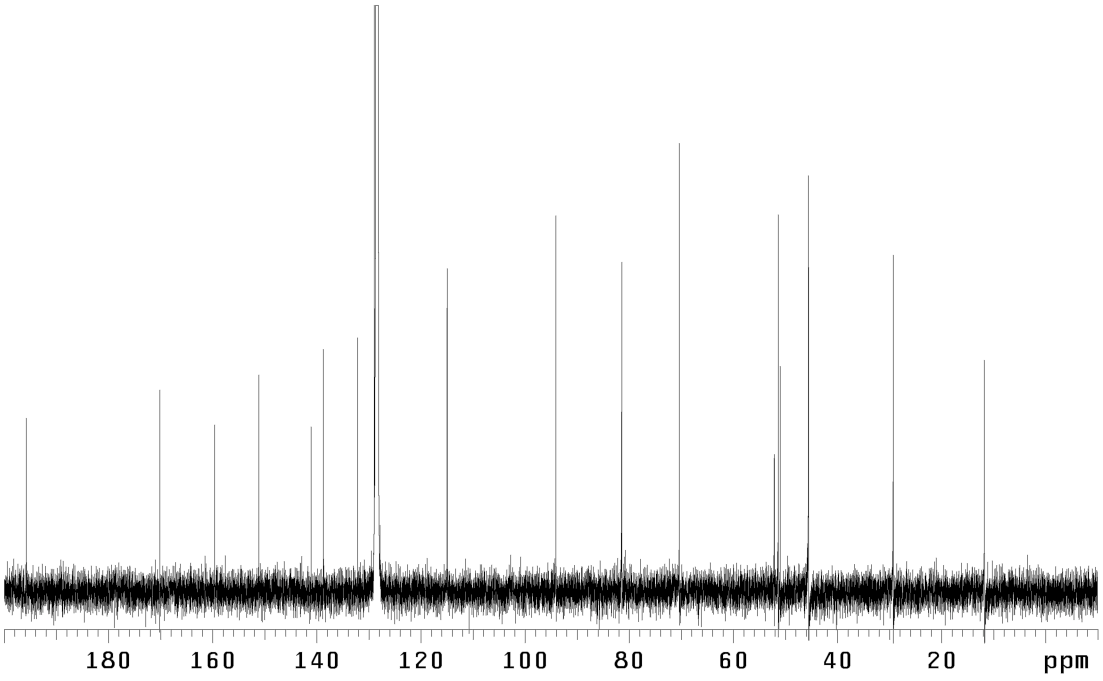


Figure A2.2.74 ^{13}C NMR (125 MHz, C_6D_6) of compound **173**

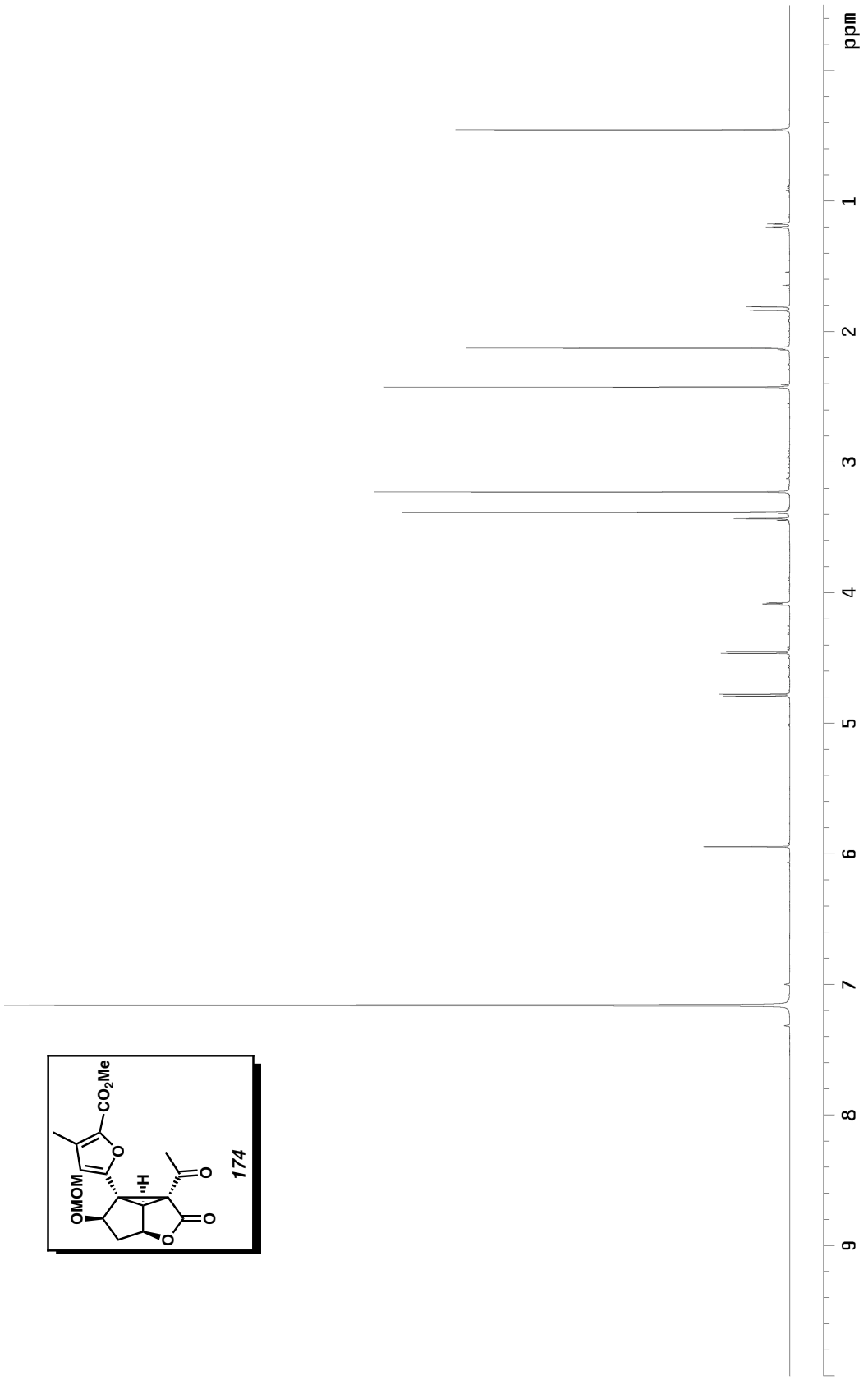


Figure A2.2.75 ^{1}H NMR (500 MHz, C_6D_6) of compound **174**

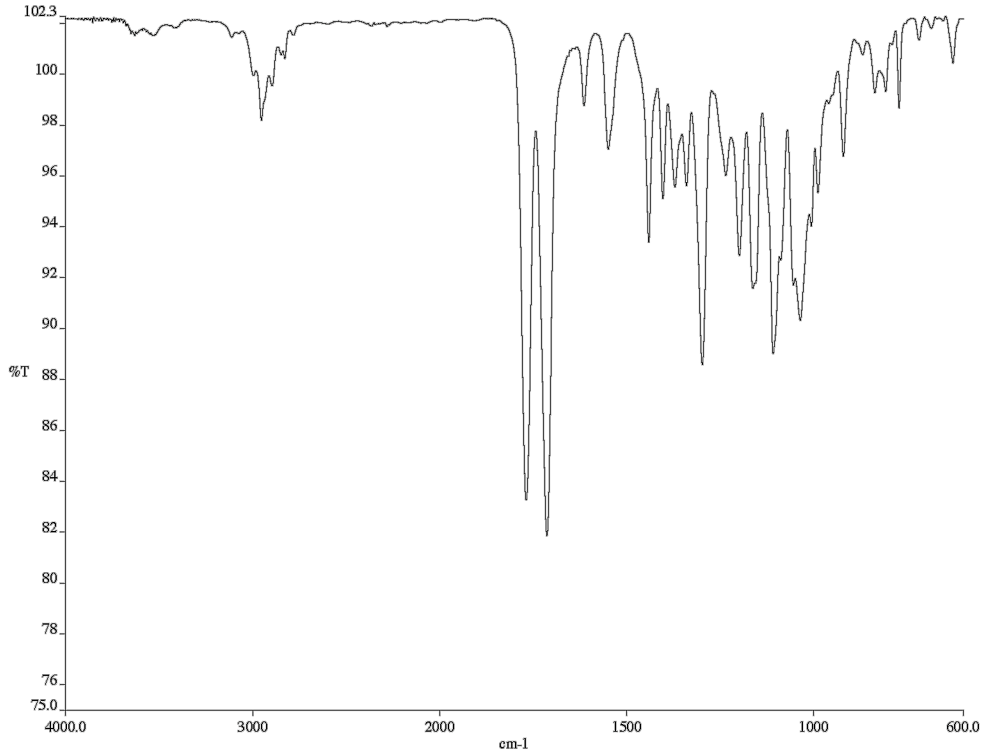


Figure A2.2.76 Infrared spectrum (film/NaCl) of compound **174**

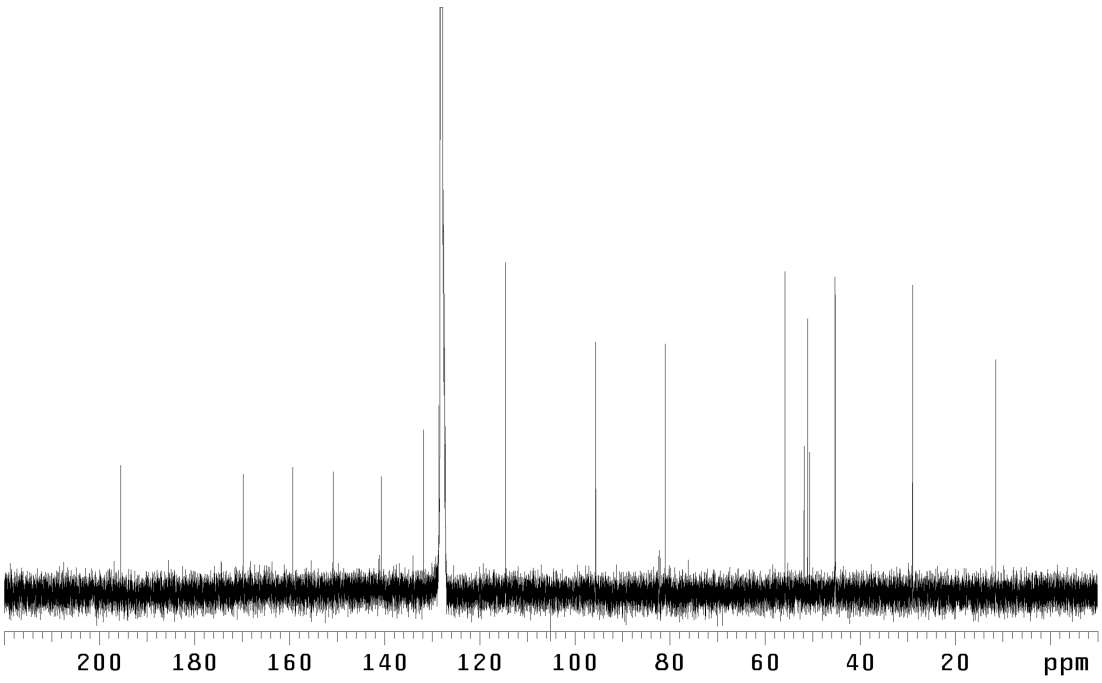


Figure A2.2.77 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **174**

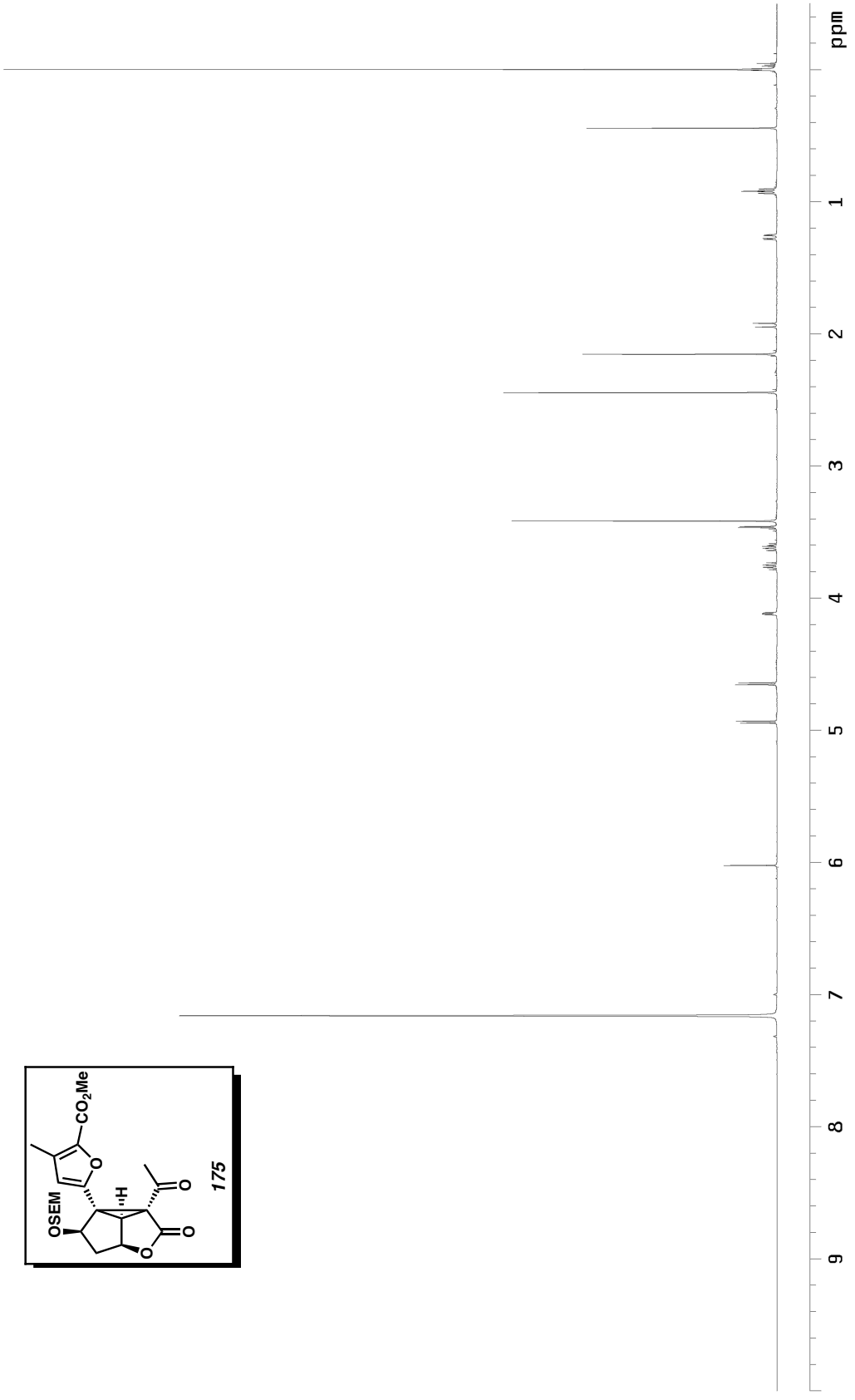


Figure A2.2.78 ^{1}H NMR (500 MHz, C_6D_6) of compound **175**

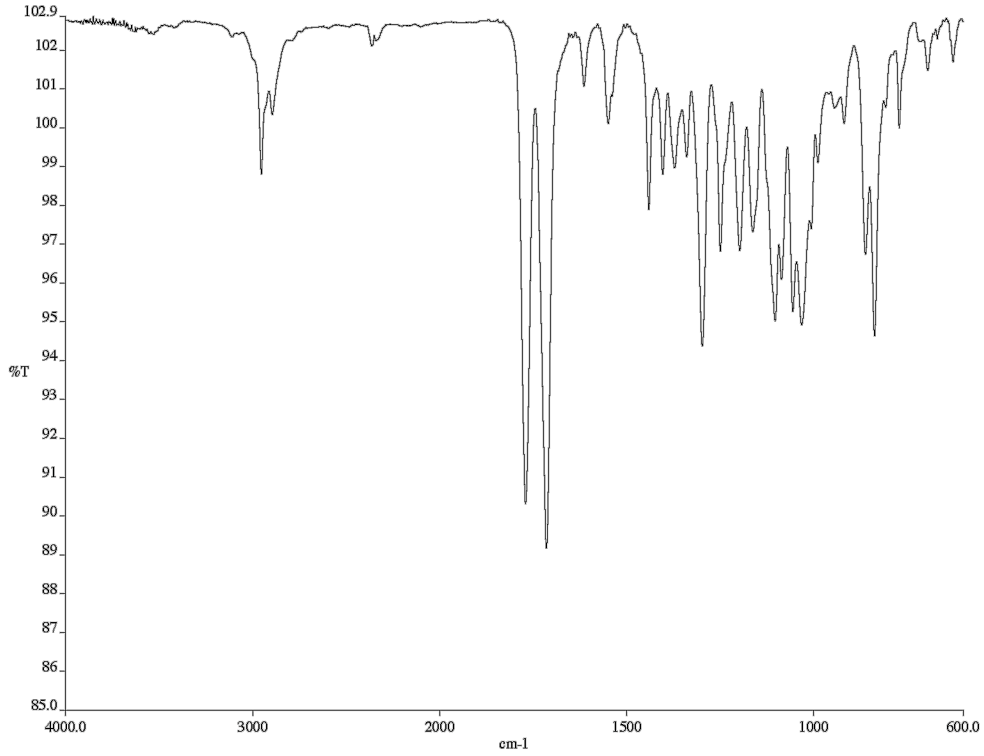


Figure A2.2.79 Infrared spectrum (film/NaCl) of compound **175**

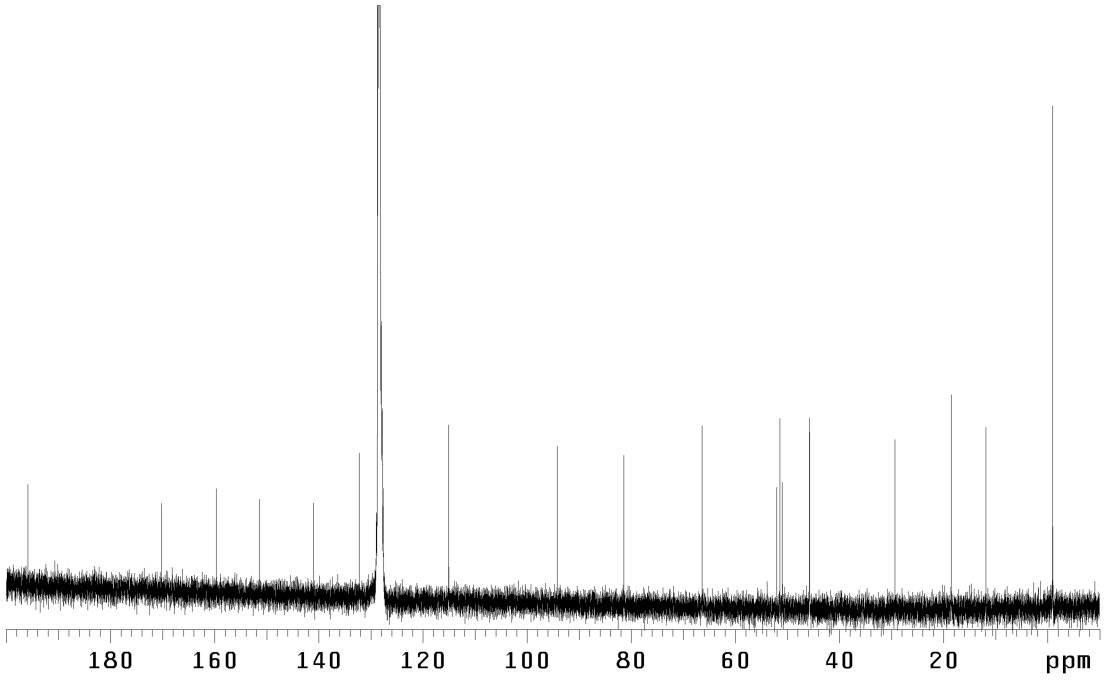


Figure A2.2.80 ^{13}C NMR (125 MHz, C_6D_6) of compound **175**

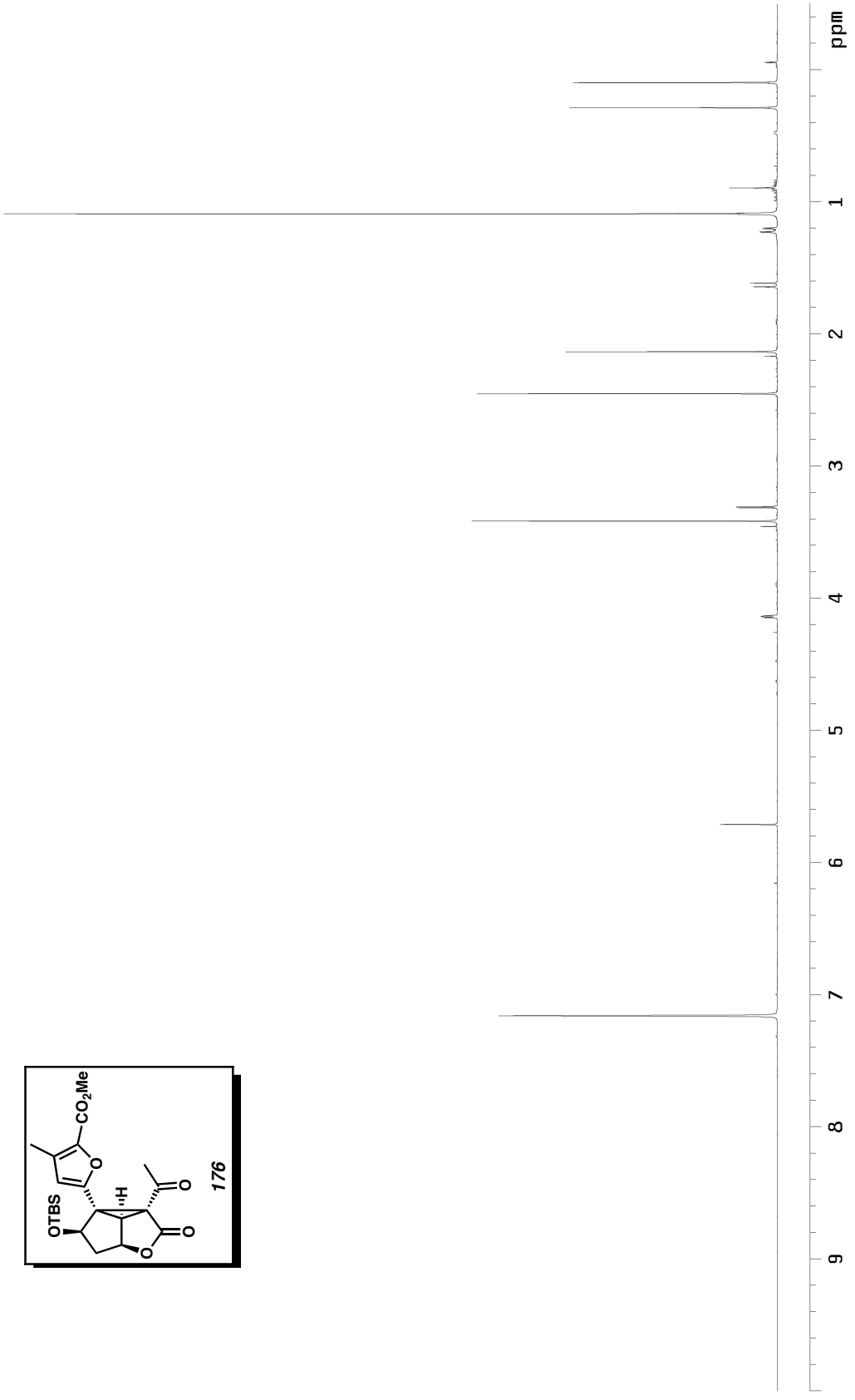


Figure A2.2.81 ^{1}H NMR (500 MHz, C_6D_6) of compound **176**

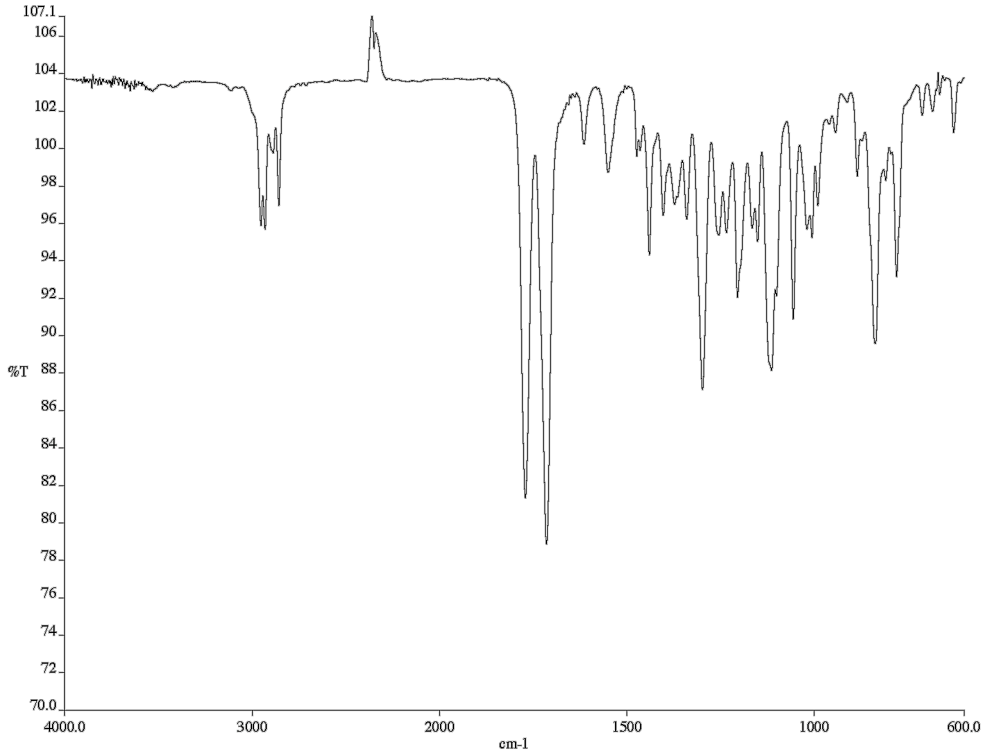


Figure A2.2.82 Infrared spectrum (film/NaCl) of compound **176**

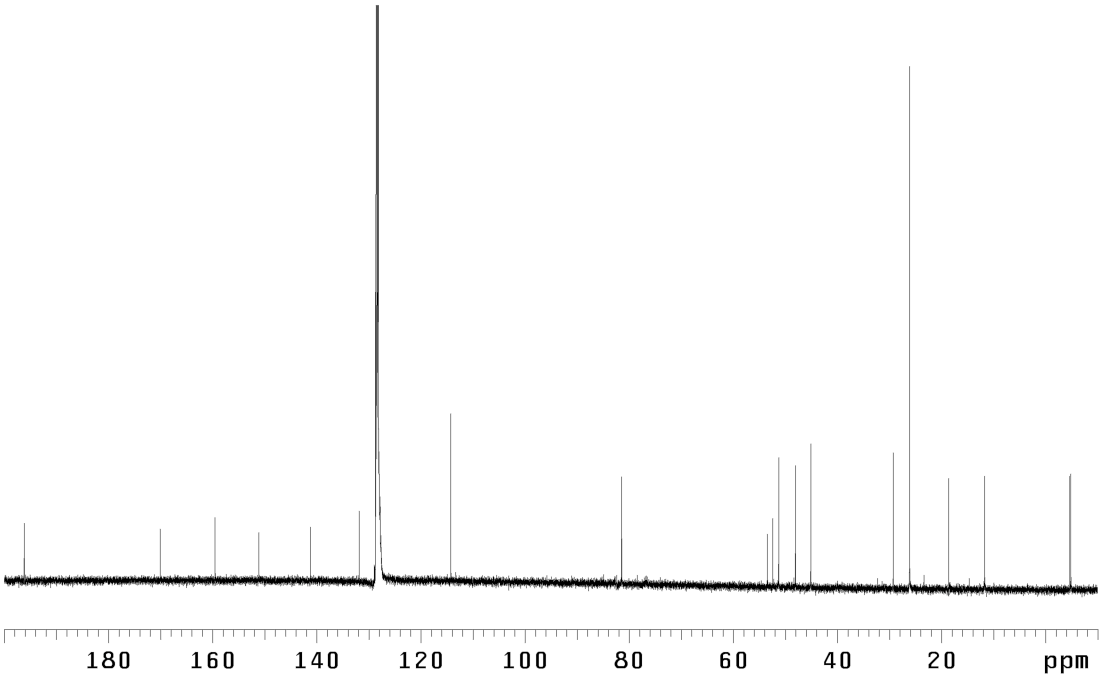


Figure A2.2.83 ^{13}C NMR (125 MHz, C_6D_6) of compound **176**

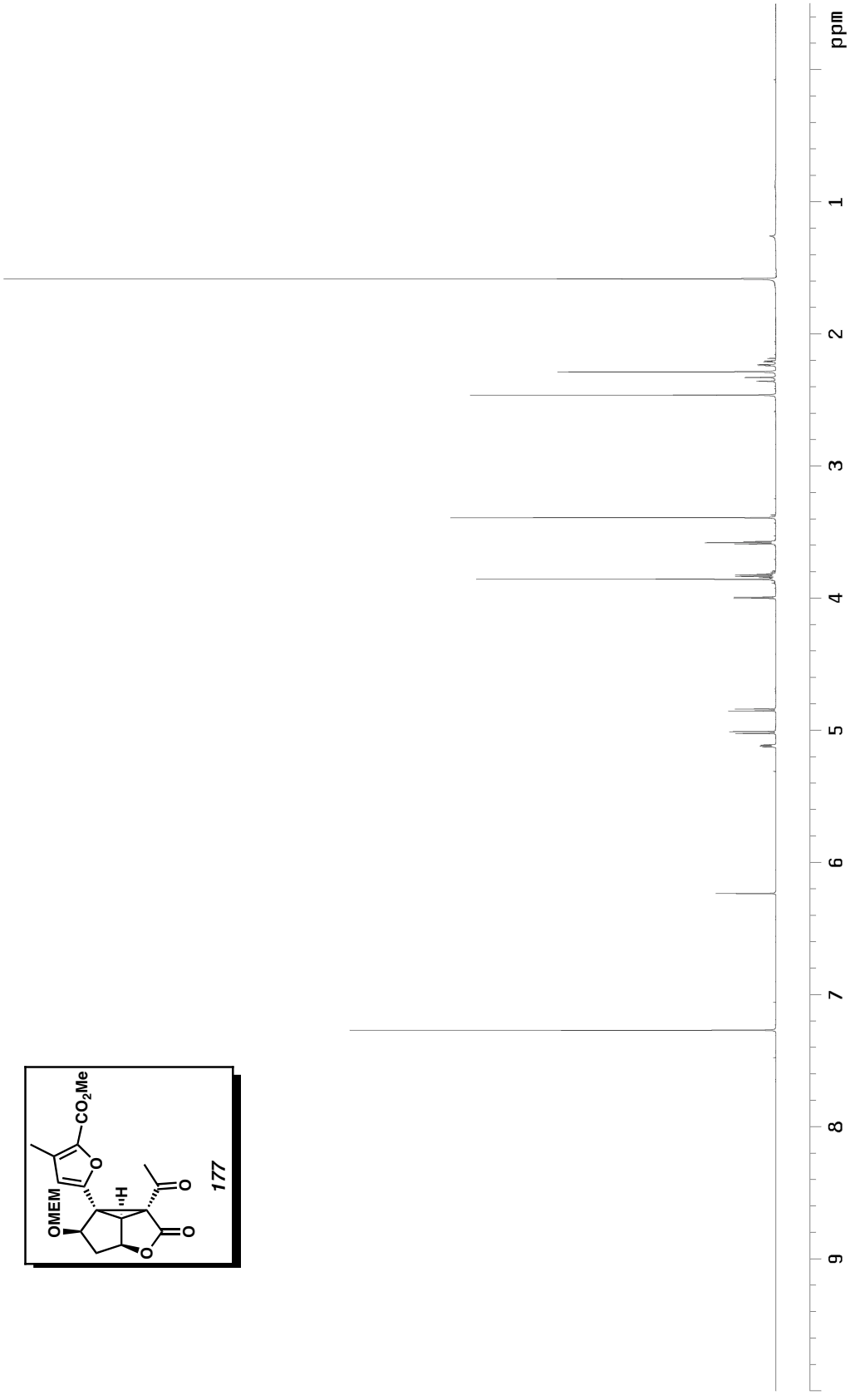


Figure A2.2.84 ^{1}H NMR (500 MHz, C_6D_6) of compound **177**

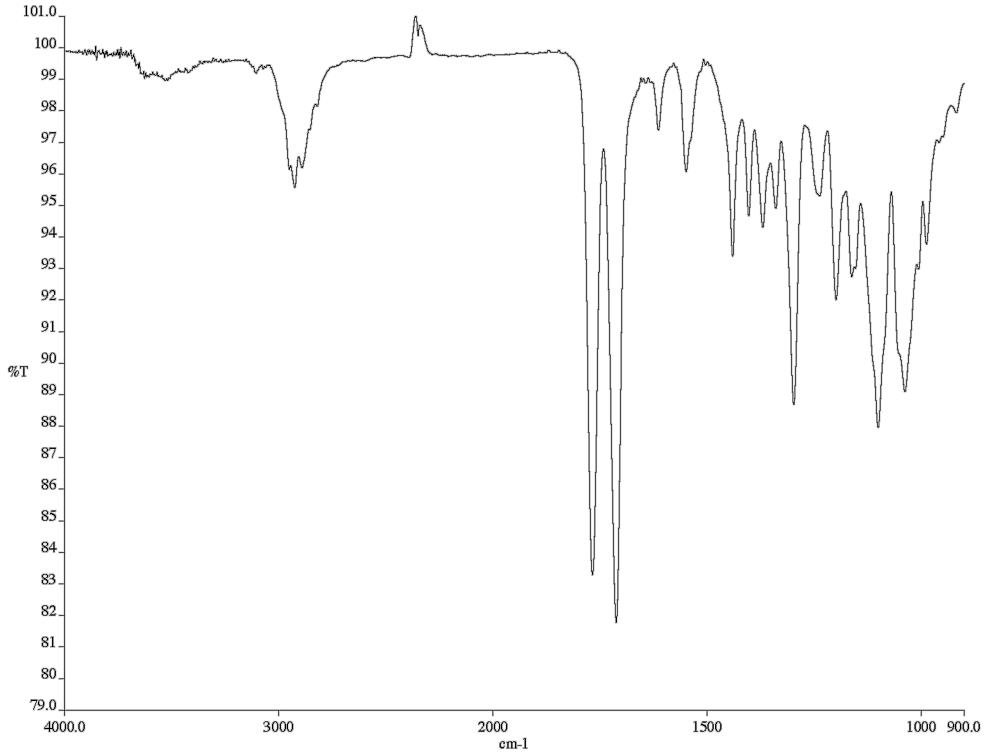


Figure A2.2.85 Infrared spectrum (film/NaCl) of compound **177**

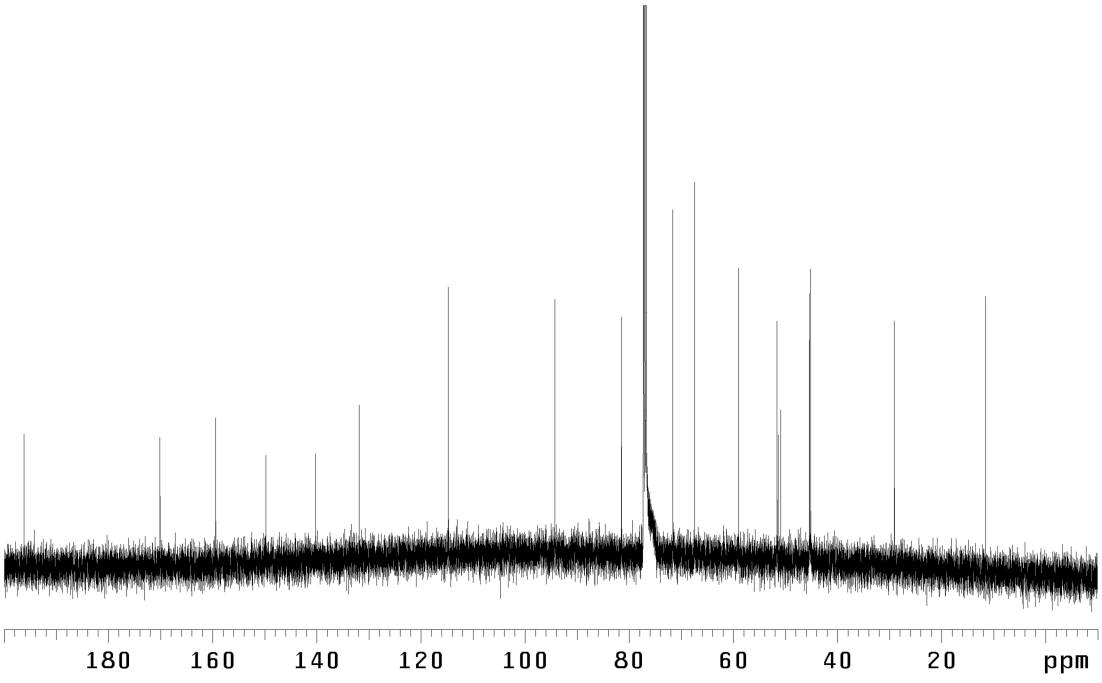


Figure A2.2.86 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **177**

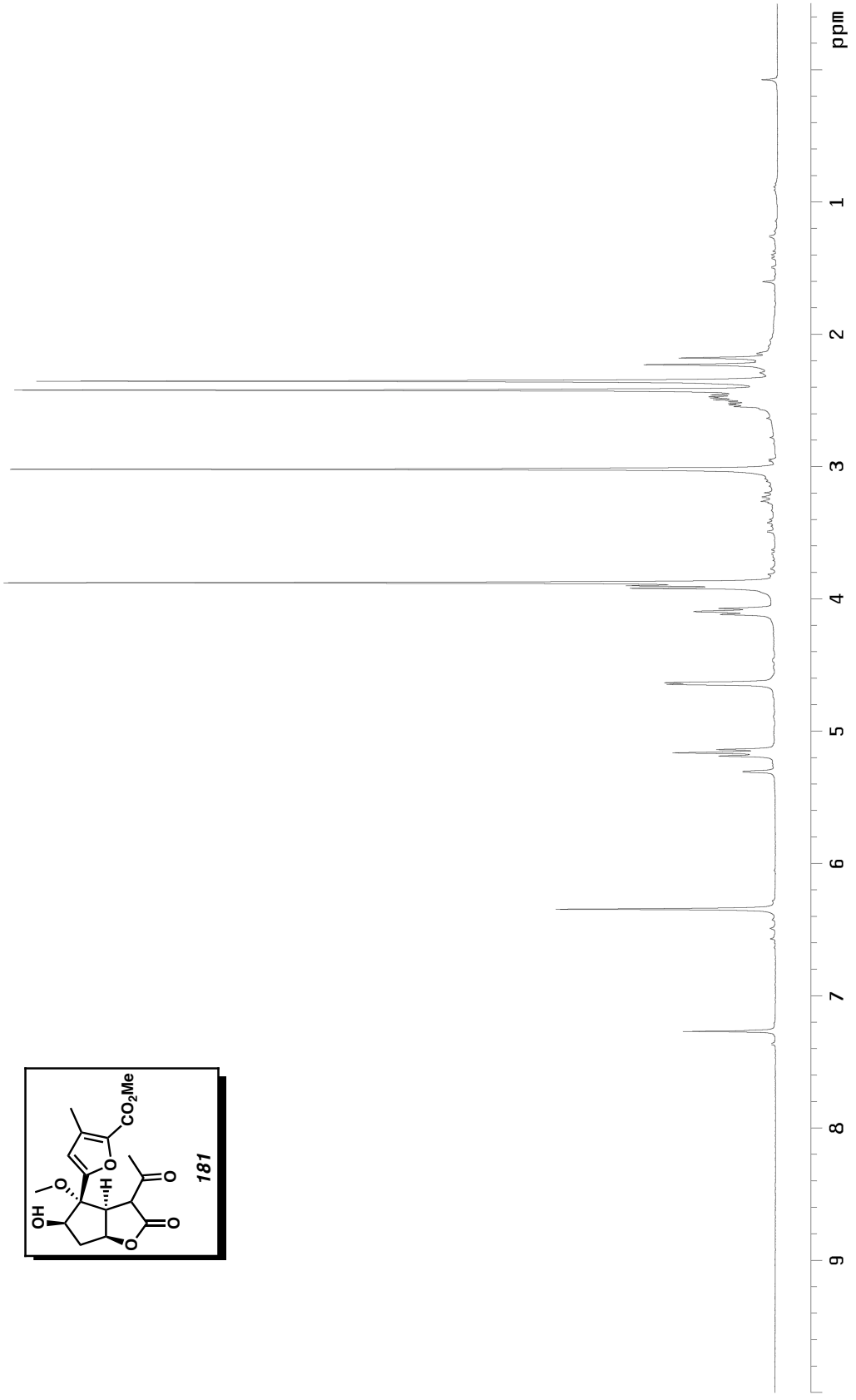


Figure A2.2.87 ^{1}H NMR (300 MHz, $CDCl_3$) of compound **181**

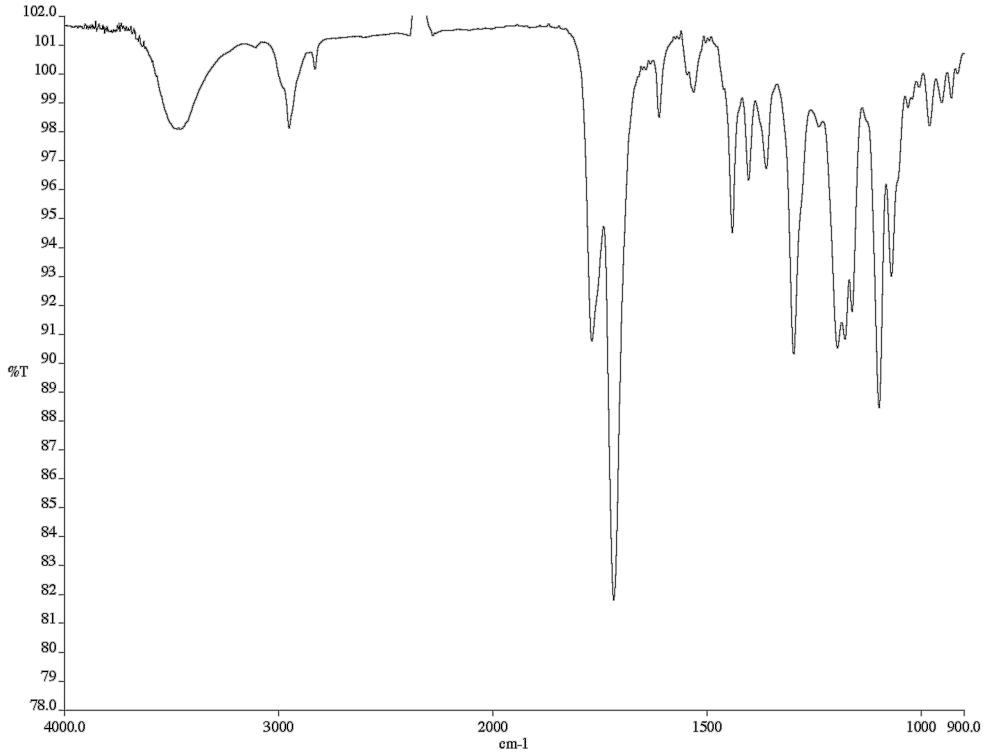


Figure A2.2.88 Infrared spectrum (film/NaCl) of compound **181**

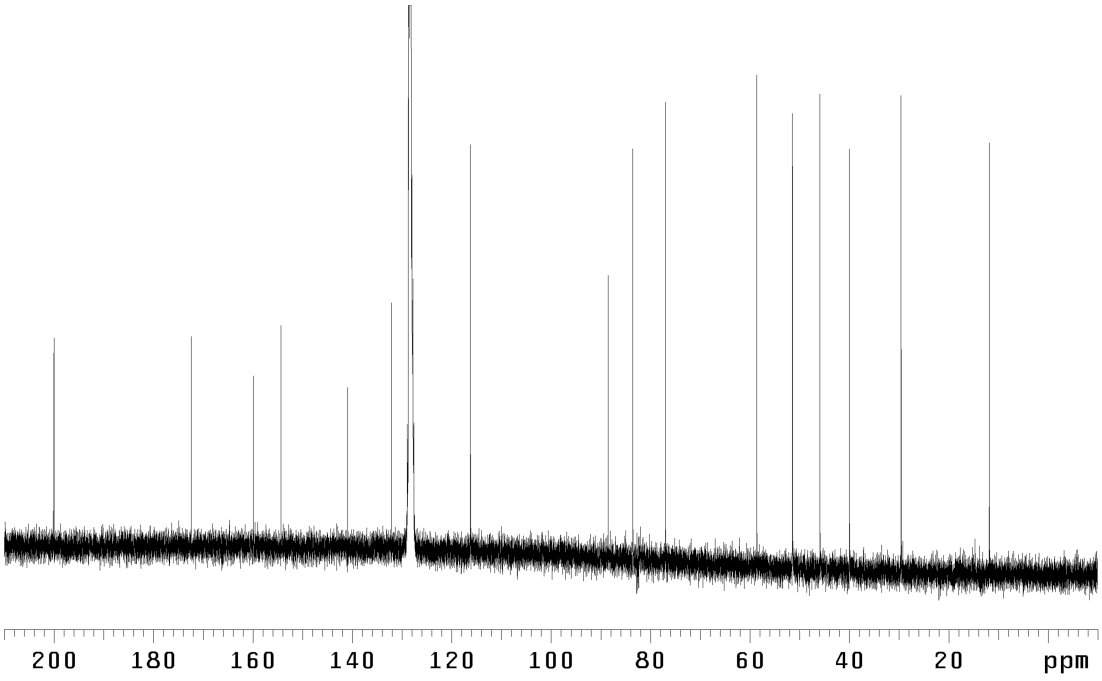


Figure A2.2.89 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **181**

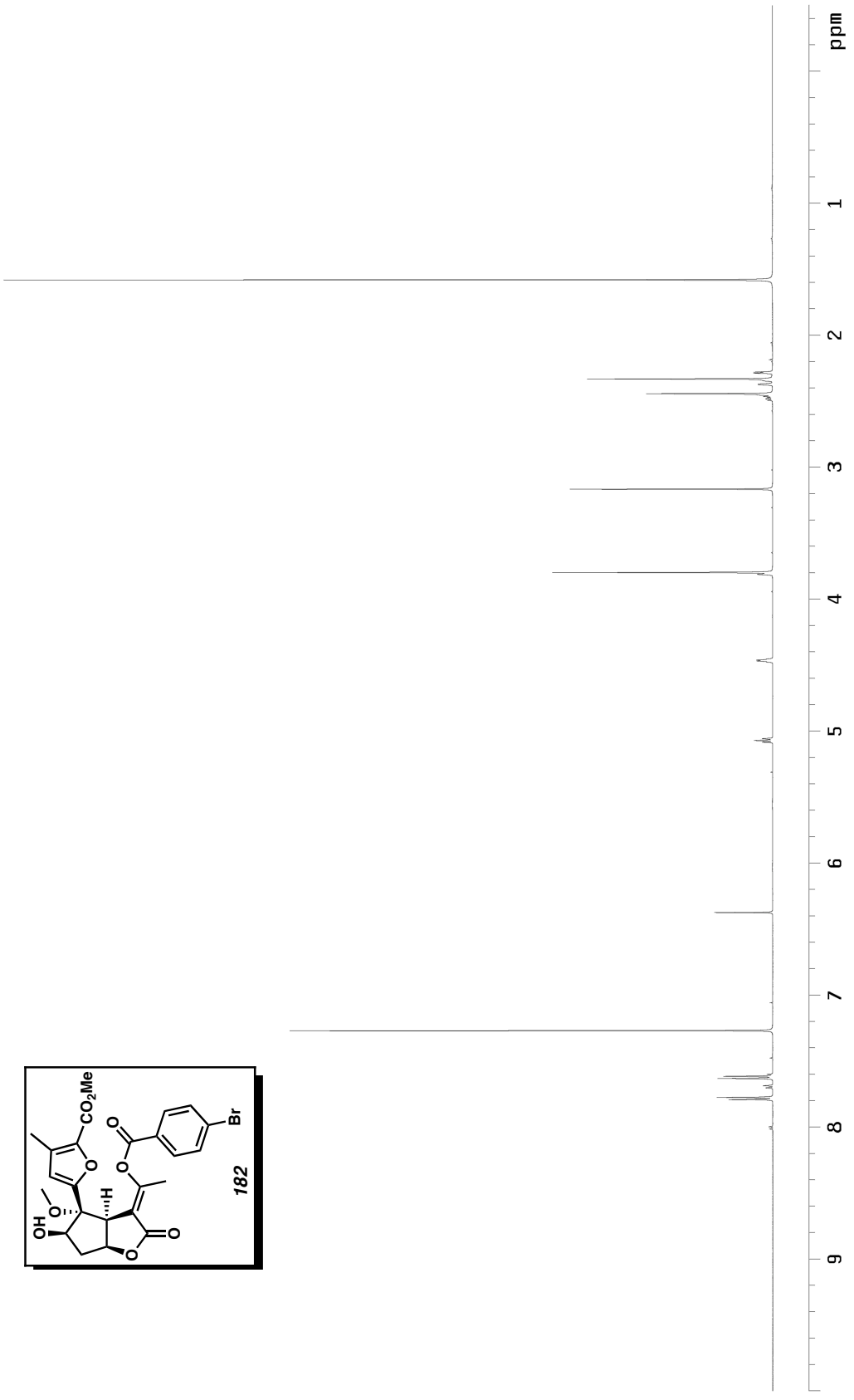


Figure A2.2.90 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **182**

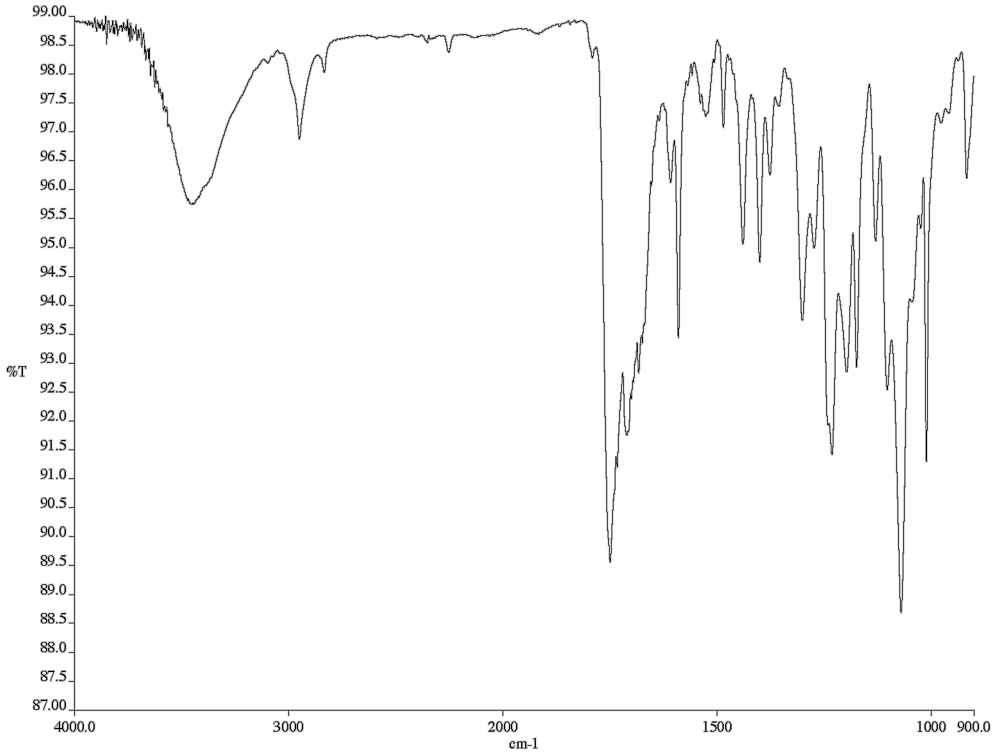


Figure A2.2.91 Infrared spectrum (film/NaCl) of compound **182**

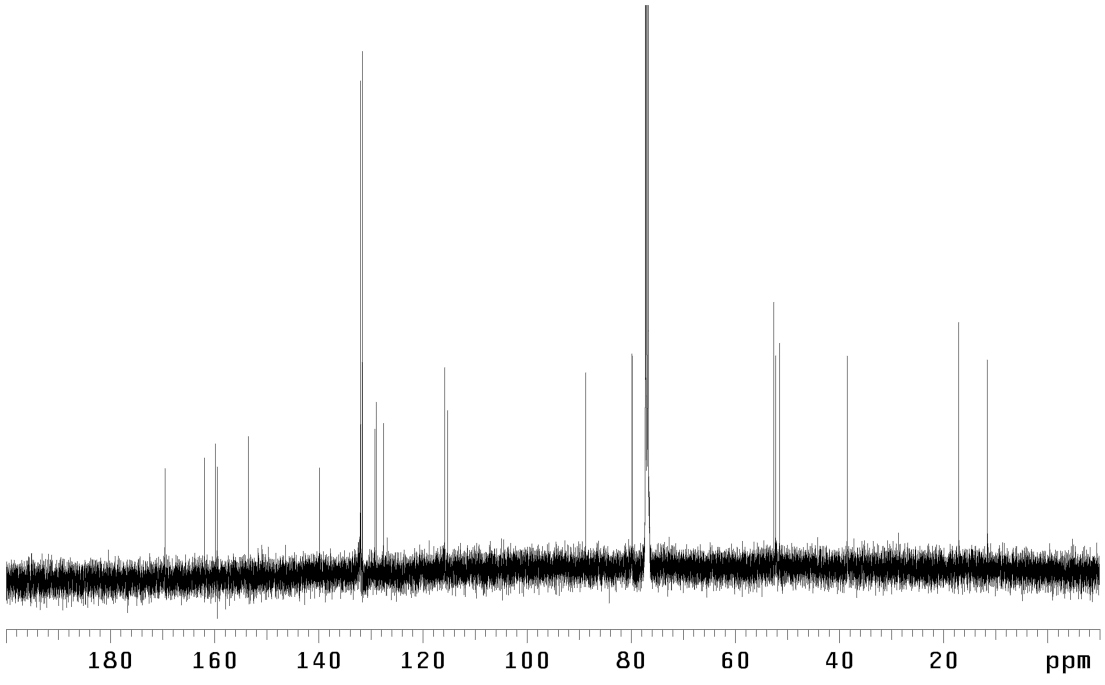


Figure A2.2.92 ^{13}C NMR (125 MHz, $CDCl_3$) of compound **182**

Appendix 2.3

X-Ray Crystallographic Data Relevant to Chapter Two

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Alcohol 182

(CCDC 606989)

Contents

Figure A2.3.1 Representation of alcohol **182**.

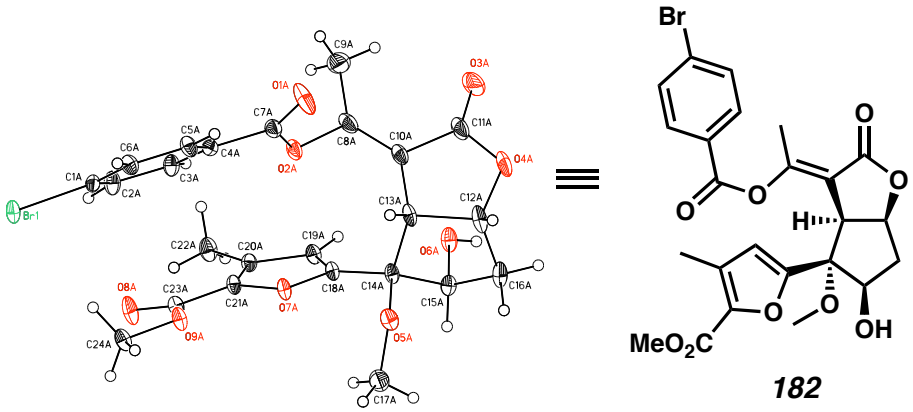


Table 1. Crystal data and structure refinement for alcohol 182 (CCDC 606989).

Empirical formula	$C_{24}H2_3O_9Br$
Formula weight	535.33
Crystallization Solvent	Ethyl acetate/hexanes
Crystal Habit	Fragment
Crystal size	0.34 x 0.26 x 0.18 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 20114 reflections used in lattice determination	2.43 to 41.82°	
Unit cell dimensions	a = 11.1074(2) Å b = 13.5443(3) Å c = 16.7177(4) Å	α= 106.8590(10)° β= 104.9520(10)° γ = 98.6060(10)°
Volume	2255.89(8) $Å^3$	
Z	4	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.576 Mg/m^3	
F(000)	1096	
Data collection program	Bruker SMART v5.630	
θ range for data collection	1.71 to 42.84°	
Completeness to θ = 42.84°	87.4 %	
Index ranges	$-21 \le h \le 21, -24 \le k \le 25, -31 \le l \le 31$	
Data collection scan type	ω scans at 8 ϕ settings	
Data reduction program	Bruker SAINT v6.45A	
Reflections collected	68261	
Independent reflections	28932 [R_{int}= 0.0502]	
Absorption coefficient	1.876 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000000 and 0.820201	

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	28932 / 0 / 797
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.189
Final R indices [I>2σ(I), 18290 reflections]	R1 = 0.0407, *w*R2 = 0.0734
R indices (all data)	R1 = 0.0831, *w*R2 = 0.0812
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.007
Average shift/error	0.000
Largest diff. peak and hole	0.844 and -0.543 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 alcohol 182 (CCDC 606989). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Br(1)	11019(1)	1482(1)	10581(1)	19(1)
O(1A)	4709(1)	873(1)	8210(1)	34(1)
O(2A)	5774(1)	1287(1)	7327(1)	20(1)
O(3A)	2091(1)	777(1)	5288(1)	36(1)
O(4A)	2261(1)	2445(1)	6087(1)	25(1)
O(5A)	5897(1)	4595(1)	7735(1)	19(1)
O(6A)	4432(1)	3010(1)	5395(1)	20(1)
O(7A)	7570(1)	3438(1)	7662(1)	16(1)
O(8A)	10665(1)	2911(1)	8096(1)	26(1)
O(9A)	9571(1)	3647(1)	8987(1)	18(1)
C(1A)	9361(1)	1314(1)	9804(1)	16(1)
C(2A)	9229(1)	1148(1)	8922(1)	20(1)
C(3A)	8037(1)	1072(1)	8348(1)	18(1)
C(4A)	6990(1)	1154(1)	8661(1)	15(1)
C(5A)	7140(1)	1289(1)	9543(1)	18(1)
C(6A)	8330(1)	1375(1)	10125(1)	18(1)
C(7A)	5707(1)	1080(1)	8070(1)	18(1)
C(8A)	4599(1)	1014(1)	6651(1)	22(1)
C(9A)	4200(2)	-138(1)	6108(1)	34(1)
C(10A)	4018(1)	1783(1)	6549(1)	19(1)
C(11A)	2731(1)	1566(1)	5895(1)	24(1)
C(12A)	3206(1)	3353(1)	6785(1)	22(1)
C(13A)	4414(1)	2941(1)	7101(1)	17(1)
C(14A)	5506(1)	3668(1)	6955(1)	15(1)
C(15A)	4787(1)	3946(1)	6159(1)	17(1)
C(16A)	3600(1)	4196(1)	6402(1)	21(1)
C(17A)	6832(1)	5468(1)	7761(1)	25(1)
C(18A)	6643(1)	3217(1)	6874(1)	14(1)
C(19A)	6986(1)	2623(1)	6200(1)	16(1)
C(20A)	8221(1)	2463(1)	6582(1)	16(1)
C(21A)	8535(1)	2986(1)	7470(1)	16(1)
C(22A)	8979(1)	1840(1)	6099(1)	22(1)
C(23A)	9698(1)	3171(1)	8201(1)	17(1)
C(24A)	10696(1)	3849(1)	9740(1)	20(1)
Br(2)	3493(1)	3385(1)	-883(1)	18(1)
O(1B)	9806(1)	4309(1)	1640(1)	28(1)
O(2B)	8714(1)	3943(1)	2530(1)	19(1)
O(3B)	12418(1)	4788(1)	4624(1)	30(1)
O(4B)	12534(1)	3196(1)	3826(1)	23(1)
O(5B)	9146(1)	747(1)	2103(1)	20(1)
O(6B)	10420(1)	2346(1)	4444(1)	19(1)
O(7B)	7219(1)	1646(1)	2160(1)	16(1)
O(8B)	4007(1)	1961(1)	1733(1)	21(1)
O(9B)	5099(1)	1226(1)	841(1)	18(1)
C(1B)	5143(1)	3643(1)	-75(1)	15(1)
C(2B)	5228(1)	3750(1)	794(1)	17(1)

C(3B)	6417(1)	3885(1)	1395(1)	17(1)
C(4B)	7512(1)	3913(1)	1131(1)	14(1)
C(5B)	7415(1)	3819(1)	261(1)	17(1)
C(6B)	6229(1)	3685(1)	-347(1)	17(1)
C(7B)	8798(1)	4077(1)	1764(1)	17(1)
C(8B)	9866(1)	4308(1)	3237(1)	19(1)
C(9B)	10039(1)	5424(1)	3807(1)	28(1)
C(10B)	10611(1)	3636(1)	3334(1)	18(1)
C(11B)	11900(1)	3974(1)	4011(1)	21(1)
C(12B)	11713(1)	2235(1)	3104(1)	22(1)
C(13B)	10398(1)	2503(1)	2764(1)	17(1)
C(14B)	9408(1)	1648(1)	2882(1)	16(1)
C(15B)	10213(1)	1430(1)	3687(1)	18(1)
C(16B)	11459(1)	1343(1)	3466(1)	22(1)
C(17B)	8407(1)	-225(1)	2091(1)	25(1)
C(18B)	8174(1)	1940(1)	2947(1)	14(1)
C(19B)	7771(1)	2456(1)	3621(1)	15(1)
C(20B)	6467(1)	2480(1)	3241(1)	14(1)
C(21B)	6179(1)	1972(1)	2353(1)	15(1)
C(22B)	5613(1)	2941(1)	3728(1)	20(1)
C(23B)	4983(1)	1725(1)	1626(1)	15(1)
C(24B)	3965(1)	998(1)	90(1)	20(1)

Table 3. Bond lengths [Å] and angles [°] for alcohol 182 (CCDC 606989).

Br(1)-C(1A)	1.8967(11)	C(20A)-C(21A)	1.3699(16)
O(1A)-C(7A)	1.2006(15)	C(20A)-C(22A)	1.4966(15)
O(2A)-C(7A)	1.3673(15)	C(21A)-C(23A)	1.4612(15)
O(2A)-C(8A)	1.4031(14)	C(22A)-H(22A)	0.86(2)
O(3A)-C(11A)	1.2030(18)	C(22A)-H(22B)	0.89(2)
O(4A)-C(11A)	1.3573(18)	C(22A)-H(22C)	0.91(2)
O(4A)-C(12A)	1.4575(16)	C(24A)-H(24A)	0.945(19)
O(5A)-C(17A)	1.4344(17)	C(24A)-H(24B)	0.950(18)
O(5A)-C(14A)	1.4363(14)	C(24A)-H(24C)	0.921(17)
O(6A)-C(15A)	1.4308(15)	Br(2)-C(1B)	1.8886(10)
O(6A)-H(6A)	0.76(2)	O(1B)-C(7B)	1.2031(14)
O(7A)-C(18A)	1.3641(13)	O(2B)-C(7B)	1.3679(14)
O(7A)-C(21A)	1.3745(12)	O(2B)-C(8B)	1.4040(13)
O(8A)-C(23A)	1.2169(13)	O(3B)-C(11B)	1.2033(17)
O(9A)-C(23A)	1.3406(14)	O(4B)-C(11B)	1.3588(17)
O(9A)-C(24A)	1.4513(14)	O(4B)-C(12B)	1.4660(16)
C(1A)-C(6A)	1.3878(15)	O(5B)-C(17B)	1.4358(18)
C(1A)-C(2A)	1.3900(17)	O(5B)-C(14B)	1.4305(13)
C(2A)-C(3A)	1.3897(16)	O(6B)-C(15B)	1.4301(15)
C(2A)-H(2A)	0.969(18)	O(6B)-H(6B)	0.79(2)
C(3A)-C(4A)	1.3984(15)	O(7B)-C(18B)	1.3659(13)
C(3A)-H(3A)	0.929(18)	O(7B)-C(21B)	1.3763(12)
C(4A)-C(5A)	1.3950(16)	O(8B)-C(23B)	1.2158(13)
C(4A)-C(7A)	1.4795(15)	O(9B)-C(23B)	1.3397(14)
C(5A)-C(6A)	1.3897(16)	O(9B)-C(24B)	1.4497(14)
C(5A)-H(5A)	0.891(17)	C(1B)-C(6B)	1.3940(14)
C(6A)-H(6A1)	0.951(16)	C(1B)-C(2B)	1.3935(16)
C(8A)-C(10A)	1.3341(19)	C(2B)-C(3B)	1.3865(16)
C(8A)-C(9A)	1.489(2)	C(2B)-H(2B)	0.901(16)
C(9A)-H(9A1)	0.88(2)	C(3B)-C(4B)	1.3958(15)
C(9A)-H(9A2)	0.97(2)	C(3B)-H(3B)	0.927(18)
C(9A)-H(9A3)	0.96(2)	C(4B)-C(5B)	1.3976(16)
C(10A)-C(11A)	1.4857(15)	C(4B)-C(7B)	1.4823(15)
C(10A)-C(13A)	1.5001(18)	C(5B)-C(6B)	1.3904(15)
C(12A)-C(16A)	1.525(2)	C(5B)-H(5B)	0.910(18)
C(12A)-C(13A)	1.5563(15)	C(6B)-H(6B1)	0.944(15)
C(12A)-H(12A)	0.902(19)	C(8B)-C(10B)	1.3346(18)
C(13A)-C(14A)	1.5609(16)	C(8B)-C(9B)	1.4872(19)
C(13A)-H(13A)	0.941(16)	C(9B)-H(9B1)	0.91(2)
C(14A)-C(18A)	1.5024(14)	C(9B)-H(9B2)	0.99(2)
C(14A)-C(15A)	1.5430(16)	C(9B)-H(9B3)	0.93(2)
C(15A)-C(16A)	1.5315(15)	C(10B)-C(11B)	1.4851(15)
C(15A)-H(15A)	0.966(16)	C(10B)-C(13B)	1.4994(18)
C(16A)-H(16A)	0.928(17)	C(12B)-C(16B)	1.522(2)
C(16A)-H(16B)	0.958(17)	C(12B)-C(13B)	1.5604(15)
C(17A)-H(17A)	0.95(2)	C(12B)-H(12B)	0.921(16)
C(17A)-H(17B)	0.95(2)	C(13B)-C(14B)	1.5654(17)
C(17A)-H(17C)	0.88(2)	C(13B)-H(13B)	0.937(16)
C(18A)-C(19A)	1.3602(15)	C(14B)-C(18B)	1.5037(14)
C(19A)-C(20A)	1.4326(15)	C(14B)-C(15B)	1.5461(16)
C(19A)-H(19A)	0.968(16)	C(15B)-C(16B)	1.5338(15)

C(15B)-H(15B)	0.959(16)
C(16B)-H(16C)	0.941(18)
C(16B)-H(16D)	0.940(19)
C(17B)-H(17D)	0.965(17)
C(17B)-H(17E)	0.967(18)
C(17B)-H(17F)	0.940(19)
C(18B)-C(19B)	1.3621(15)
C(19B)-C(20B)	1.4325(14)
C(19B)-H(19B)	1.020(17)
C(20B)-C(21B)	1.3693(15)
C(20B)-C(22B)	1.4940(14)
C(21B)-C(23B)	1.4673(15)
C(22B)-H(22D)	0.88(2)
C(22B)-H(22E)	0.92(2)
C(22B)-H(22F)	0.95(2)
C(24B)-H(24D)	0.925(17)
C(24B)-H(24E)	0.949(19)
C(24B)-H(24F)	0.96(2)

C(7A)-O(2A)-C(8A)	115.48(9)
C(11A)-O(4A)-C(12A)	112.39(9)
C(17A)-O(5A)-C(14A)	115.97(9)
C(15A)-O(6A)-H(6A)	108.2(15)
C(18A)-O(7A)-C(21A)	106.33(8)
C(23A)-O(9A)-C(24A)	114.99(9)
C(6A)-C(1A)-C(2A)	121.98(10)
C(6A)-C(1A)-Br(1)	120.04(9)
C(2A)-C(1A)-Br(1)	117.97(8)
C(3A)-C(2A)-C(1A)	119.10(10)
C(3A)-C(2A)-H(2A)	117.5(11)
C(1A)-C(2A)-H(2A)	123.2(11)
C(2A)-C(3A)-C(4A)	119.89(11)
C(2A)-C(3A)-H(3A)	120.5(11)
C(4A)-C(3A)-H(3A)	119.6(11)
C(3A)-C(4A)-C(5A)	119.86(10)
C(3A)-C(4A)-C(7A)	121.46(10)
C(5A)-C(4A)-C(7A)	118.68(10)
C(6A)-C(5A)-C(4A)	120.73(10)
C(6A)-C(5A)-H(5A)	121.7(11)
C(4A)-C(5A)-H(5A)	117.5(11)
C(1A)-C(6A)-C(5A)	118.40(11)
C(1A)-C(6A)-H(6A1)	122.4(10)
C(5A)-C(6A)-H(6A1)	119.2(10)
O(1A)-C(7A)-O(2A)	122.51(11)
O(1A)-C(7A)-C(4A)	125.43(12)
O(2A)-C(7A)-C(4A)	112.05(10)
C(10A)-C(8A)-O(2A)	118.54(12)
C(10A)-C(8A)-C(9A)	128.62(12)
O(2A)-C(8A)-C(9A)	112.79(12)
C(8A)-C(9A)-H(9A1)	110.0(14)
C(8A)-C(9A)-H(9A2)	108.7(12)
H(9A1)-C(9A)-H(9A2)	112.6(18)
C(8A)-C(9A)-H(9A3)	107.5(13)
H(9A1)-C(9A)-H(9A3)	111.6(18)
H(9A2)-C(9A)-H(9A3)	106.3(17)
C(8A)-C(10A)-C(11A)	122.62(12)
C(8A)-C(10A)-C(13A)	128.54(10)
C(11A)-C(10A)-C(13A)	108.45(11)
O(3A)-C(11A)-O(4A)	119.81(12)
O(3A)-C(11A)-C(10A)	131.28(14)
O(4A)-C(11A)-C(10A)	108.86(11)
O(4A)-C(12A)-C(16A)	109.45(10)
O(4A)-C(12A)-C(13A)	106.21(11)
C(16A)-C(12A)-C(13A)	107.12(9)
O(4A)-C(12A)-H(12A)	105.6(12)
C(16A)-C(12A)-H(12A)	114.7(12)
C(13A)-C(12A)-H(12A)	113.4(12)
C(10A)-C(13A)-C(12A)	103.48(9)
C(10A)-C(13A)-C(14A)	117.70(9)
C(12A)-C(13A)-C(14A)	103.81(10)
C(10A)-C(13A)-H(13A)	109.9(10)
C(12A)-C(13A)-H(13A)	113.2(10)
C(14A)-C(13A)-H(13A)	108.6(10)
O(5A)-C(14A)-C(18A)	109.54(9)
O(5A)-C(14A)-C(15A)	109.05(10)
C(18A)-C(14A)-C(15A)	116.10(9)
O(5A)-C(14A)-C(13A)	102.55(8)
C(18A)-C(14A)-C(13A)	115.37(10)
C(15A)-C(14A)-C(13A)	103.20(9)
O(6A)-C(15A)-C(16A)	110.76(10)
O(6A)-C(15A)-C(14A)	107.37(10)
C(16A)-C(15A)-C(14A)	101.58(9)
O(6A)-C(15A)-H(15A)	111.0(9)
C(16A)-C(15A)-H(15A)	115.1(9)
C(14A)-C(15A)-H(15A)	110.4(9)
C(12A)-C(16A)-C(15A)	104.43(10)
C(12A)-C(16A)-H(16A)	110.8(11)
C(15A)-C(16A)-H(16A)	108.7(11)
C(12A)-C(16A)-H(16B)	110.1(10)
C(15A)-C(16A)-H(16B)	113.1(10)
H(16A)-C(16A)-H(16B)	109.6(14)
O(5A)-C(17A)-H(17A)	112.3(12)
O(5A)-C(17A)-H(17B)	106.5(12)
H(17A)-C(17A)-H(17B)	109.6(16)
O(5A)-C(17A)-H(17C)	109.7(15)
H(17A)-C(17A)-H(17C)	110.7(18)
H(17B)-C(17A)-H(17C)	108.0(18)
C(19A)-C(18A)-O(7A)	110.52(9)
C(19A)-C(18A)-C(14A)	135.62(10)
O(7A)-C(18A)-C(14A)	113.85(9)
C(18A)-C(19A)-C(20A)	107.01(10)
C(18A)-C(19A)-H(19A)	126.8(10)
C(20A)-C(19A)-H(19A)	126.2(10)
C(21A)-C(20A)-C(19A)	105.30(9)
C(21A)-C(20A)-C(22A)	128.17(10)
C(19A)-C(20A)-C(22A)	126.52(10)
C(20A)-C(21A)-O(7A)	110.82(9)
C(20A)-C(21A)-C(23A)	131.59(10)

O(7A)-C(21A)-C(23A)	117.52(9)	C(11B)-C(10B)-C(13B)	108.95(10)
C(20A)-C(22A)-H(22A)	109.5(13)	O(3B)-C(11B)-O(4B)	120.34(11)
C(20A)-C(22A)-H(22B)	109.0(13)	O(3B)-C(11B)-C(10B)	130.84(13)
H(22A)-C(22A)-H(22B)	113.7(18)	O(4B)-C(11B)-C(10B)	108.76(11)
C(20A)-C(22A)-H(22C)	113.4(14)	O(4B)-C(12B)-C(16B)	109.34(10)
H(22A)-C(22A)-H(22C)	107.2(18)	O(4B)-C(12B)-C(13B)	106.11(10)
H(22B)-C(22A)-H(22C)	104.0(18)	C(16B)-C(12B)-C(13B)	107.02(9)
O(8A)-C(23A)-O(9A)	124.47(11)	O(4B)-C(12B)-H(12B)	103.6(10)
O(8A)-C(23A)-C(21A)	123.07(11)	C(16B)-C(12B)-H(12B)	115.6(10)
O(9A)-C(23A)-C(21A)	112.46(9)	C(13B)-C(12B)-H(12B)	114.6(10)
O(9A)-C(24A)-H(24A)	109.4(11)	C(10B)-C(13B)-C(12B)	103.30(9)
O(9A)-C(24A)-H(24B)	104.7(11)	C(10B)-C(13B)-C(14B)	117.00(9)
H(24A)-C(24A)-H(24B)	114.6(15)	C(12B)-C(13B)-C(14B)	103.80(10)
O(9A)-C(24A)-H(24C)	110.5(10)	C(10B)-C(13B)-H(13B)	112.2(10)
H(24A)-C(24A)-H(24C)	110.9(15)	C(12B)-C(13B)-H(13B)	111.4(10)
H(24B)-C(24A)-H(24C)	106.6(14)	C(14B)-C(13B)-H(13B)	108.7(10)
C(7B)-O(2B)-C(8B)	115.66(9)	O(5B)-C(14B)-C(18B)	109.68(8)
C(11B)-O(4B)-C(12B)	112.32(9)	O(5B)-C(14B)-C(15B)	109.46(10)
C(17B)-O(5B)-C(14B)	115.45(10)	C(18B)-C(14B)-C(15B)	115.52(9)
C(15B)-O(6B)-H(6B)	106.6(16)	O(5B)-C(14B)-C(13B)	102.99(9)
C(18B)-O(7B)-C(21B)	106.33(8)	C(18B)-C(14B)-C(13B)	115.51(10)
C(23B)-O(9B)-C(24B)	115.07(8)	C(15B)-C(14B)-C(13B)	102.78(9)
C(6B)-C(1B)-C(2B)	121.43(10)	O(6B)-C(15B)-C(16B)	111.39(10)
C(6B)-C(1B)-Br(2)	120.97(8)	O(6B)-C(15B)-C(14B)	106.82(10)
C(2B)-C(1B)-Br(2)	117.56(8)	C(16B)-C(15B)-C(14B)	101.42(9)
C(3B)-C(2B)-C(1B)	119.09(10)	O(6B)-C(15B)-H(15B)	111.5(9)
C(3B)-C(2B)-H(2B)	118.7(10)	C(16B)-C(15B)-H(15B)	114.3(9)
C(1B)-C(2B)-H(2B)	122.2(10)	C(14B)-C(15B)-H(15B)	110.7(9)
C(2B)-C(3B)-C(4B)	120.33(10)	C(12B)-C(16B)-C(15B)	104.35(10)
C(2B)-C(3B)-H(3B)	118.2(11)	C(12B)-C(16B)-H(16C)	112.9(11)
C(4B)-C(3B)-H(3B)	121.5(11)	C(15B)-C(16B)-H(16C)	111.9(11)
C(3B)-C(4B)-C(5B)	119.95(10)	C(12B)-C(16B)-H(16D)	111.3(12)
C(3B)-C(4B)-C(7B)	121.41(10)	C(15B)-C(16B)-H(16D)	108.7(12)
C(5B)-C(4B)-C(7B)	118.61(9)	H(16C)-C(16B)-H(16D)	107.6(15)
C(6B)-C(5B)-C(4B)	120.25(10)	O(5B)-C(17B)-H(17D)	111.8(10)
C(6B)-C(5B)-H(5B)	119.5(11)	O(5B)-C(17B)-H(17E)	105.7(11)
C(4B)-C(5B)-H(5B)	120.2(11)	H(17D)-C(17B)-H(17E)	107.5(14)
C(1B)-C(6B)-C(5B)	118.94(10)	O(5B)-C(17B)-H(17F)	111.6(11)
C(1B)-C(6B)-H(6B1)	119.9(9)	H(17D)-C(17B)-H(17F)	109.7(15)
C(5B)-C(6B)-H(6B1)	121.2(9)	H(17E)-C(17B)-H(17F)	110.4(15)
O(1B)-C(7B)-O(2B)	122.88(10)	C(19B)-C(18B)-O(7B)	110.36(9)
O(1B)-C(7B)-C(4B)	125.44(11)	C(19B)-C(18B)-C(14B)	134.82(10)
O(2B)-C(7B)-C(4B)	111.68(9)	O(7B)-C(18B)-C(14B)	114.81(9)
C(10B)-C(8B)-O(2B)	118.62(11)	C(18B)-C(19B)-C(20B)	107.13(9)
C(10B)-C(8B)-C(9B)	129.16(11)	C(18B)-C(19B)-H(19B)	129.1(10)
O(2B)-C(8B)-C(9B)	112.17(11)	C(20B)-C(19B)-H(19B)	123.7(10)
C(8B)-C(9B)-H(9B1)	108.9(13)	C(21B)-C(20B)-C(19B)	105.29(9)
C(8B)-C(9B)-H(9B2)	108.8(11)	C(21B)-C(20B)-C(22B)	128.21(10)
H(9B1)-C(9B)-H(9B2)	111.2(17)	C(19B)-C(20B)-C(22B)	126.48(10)
C(8B)-C(9B)-H(9B3)	110.2(13)	C(20B)-C(21B)-O(7B)	110.88(9)
H(9B1)-C(9B)-H(9B3)	109.1(17)	C(20B)-C(21B)-C(23B)	130.75(9)
H(9B2)-C(9B)-H(9B3)	108.6(16)	O(7B)-C(21B)-C(23B)	118.37(9)
C(8B)-C(10B)-C(11B)	122.54(12)	C(20B)-C(22B)-H(22D)	112.6(13)
C(8B)-C(10B)-C(13B)	128.24(10)	C(20B)-C(22B)-H(22E)	111.6(13)

H(22D)-C(22B)-H(22E)	110.7(18)
C(20B)-C(22B)-H(22F)	107.9(13)
H(22D)-C(22B)-H(22F)	108.0(18)
H(22E)-C(22B)-H(22F)	105.6(18)
O(8B)-C(23B)-O(9B)	124.50(10)
O(8B)-C(23B)-C(21B)	123.17(10)
O(9B)-C(23B)-C(21B)	112.32(9)
O(9B)-C(24B)-H(24D)	111.4(10)
O(9B)-C(24B)-H(24E)	107.6(11)
H(24D)-C(24B)-H(24E)	107.5(14)
O(9B)-C(24B)-H(24F)	109.6(11)
H(24D)-C(24B)-H(24F)	109.1(15)
H(24E)-C(24B)-H(24F)	111.6(15)

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for alcohol 182 (CCDC 606989). 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}
Br(1)	146(1)	261(1)	176(1)	80(1)	24(1)	90(1)
O(1A)	132(4)	651(8)	255(5)	227(5)	30(3)	27(4)
O(2A)	141(3)	272(5)	191(4)	141(3)	1(3)	29(3)
O(3A)	266(5)	363(6)	294(6)	129(5)	-109(4)	-32(4)
O(4A)	120(3)	405(6)	231(4)	158(4)	-4(3)	56(3)
O(5A)	164(3)	229(4)	146(4)	15(3)	41(3)	60(3)
O(6A)	186(3)	296(5)	108(4)	66(3)	31(3)	101(3)
O(7A)	113(3)	235(4)	120(3)	52(3)	32(3)	71(3)
O(8A)	138(3)	413(6)	195(4)	45(4)	41(3)	119(4)
O(9A)	129(3)	280(4)	136(4)	62(3)	27(3)	68(3)
C(1A)	139(4)	167(5)	153(5)	58(4)	20(3)	59(4)
C(2A)	169(4)	287(6)	186(5)	88(5)	69(4)	112(4)
C(3A)	170(4)	245(6)	148(5)	81(4)	47(4)	82(4)
C(4A)	140(4)	165(5)	161(5)	75(4)	32(3)	32(4)
C(5A)	129(4)	256(6)	171(5)	88(4)	50(4)	40(4)
C(6A)	152(4)	226(6)	152(5)	73(4)	36(4)	42(4)
C(7A)	151(4)	212(5)	186(5)	97(4)	26(4)	31(4)
C(8A)	157(4)	274(6)	187(5)	120(5)	-6(4)	-10(4)
C(9A)	345(7)	232(7)	320(8)	102(6)	-71(6)	0(6)
C(10A)	132(4)	275(6)	146(5)	108(4)	7(3)	21(4)
C(11A)	154(4)	342(7)	211(6)	158(5)	-20(4)	11(4)
C(12A)	135(4)	384(7)	144(5)	92(5)	46(4)	110(4)
C(13A)	115(4)	285(6)	117(4)	83(4)	30(3)	65(4)
C(14A)	124(4)	208(5)	123(4)	46(4)	37(3)	64(4)
C(15A)	169(4)	226(5)	144(5)	77(4)	52(4)	87(4)
C(16A)	190(5)	311(7)	157(5)	77(5)	47(4)	143(4)
C(17A)	214(5)	221(6)	275(7)	41(5)	46(5)	56(5)
C(18A)	112(4)	194(5)	124(4)	61(4)	31(3)	50(3)
C(19A)	132(4)	214(5)	130(5)	53(4)	38(3)	54(4)
C(20A)	132(4)	188(5)	163(5)	67(4)	59(3)	57(4)
C(21A)	113(4)	210(5)	161(5)	74(4)	49(3)	61(3)
C(22A)	174(5)	281(6)	196(6)	42(5)	74(4)	101(4)
C(23A)	131(4)	214(5)	163(5)	66(4)	37(4)	49(4)
C(24A)	139(4)	276(6)	168(5)	68(4)	18(4)	43(4)
Br(2)	116(1)	277(1)	151(1)	84(1)	26(1)	65(1)
O(1B)	129(3)	509(6)	203(4)	165(4)	39(3)	30(4)
O(2B)	126(3)	287(5)	155(4)	117(3)	11(3)	22(3)
O(3B)	251(4)	294(5)	232(5)	88(4)	-79(4)	-27(4)
O(4B)	104(3)	370(5)	184(4)	100(4)	-9(3)	25(3)
O(5B)	137(3)	262(4)	155(4)	1(3)	29(3)	61(3)
O(6B)	161(3)	297(5)	114(4)	66(3)	14(3)	82(3)
O(7B)	97(3)	248(4)	115(3)	45(3)	21(2)	59(3)
O(8B)	131(3)	308(5)	183(4)	55(3)	41(3)	89(3)
O(9B)	120(3)	273(4)	128(4)	50(3)	18(3)	60(3)

C(1B)	116(4)	195(5)	132(4)	60(4)	22(3)	39(3)
C(2B)	138(4)	241(6)	150(5)	71(4)	58(4)	58(4)
C(3B)	149(4)	226(6)	130(5)	71(4)	42(3)	49(4)
C(4B)	120(4)	170(5)	140(4)	62(4)	30(3)	30(3)
C(5B)	128(4)	237(6)	164(5)	81(4)	48(4)	39(4)
C(6B)	139(4)	250(6)	130(5)	79(4)	42(3)	46(4)
C(7B)	137(4)	217(5)	141(5)	78(4)	31(3)	37(4)
C(8B)	143(4)	259(6)	144(5)	88(4)	11(4)	2(4)
C(9B)	261(6)	229(6)	276(7)	70(5)	-1(5)	26(5)
C(10B)	121(4)	283(6)	110(4)	75(4)	8(3)	2(4)
C(11B)	146(4)	313(7)	150(5)	110(5)	-6(4)	-2(4)
C(12B)	107(4)	382(7)	132(5)	51(5)	20(3)	67(4)
C(13B)	100(4)	294(6)	110(4)	62(4)	14(3)	50(4)
C(14B)	107(4)	233(5)	109(4)	31(4)	15(3)	56(4)
C(15B)	134(4)	240(6)	153(5)	64(4)	19(3)	69(4)
C(16B)	136(4)	309(7)	189(5)	51(5)	19(4)	103(4)
C(17B)	158(5)	254(6)	273(7)	15(5)	36(4)	52(4)
C(18B)	99(4)	213(5)	109(4)	50(4)	18(3)	42(3)
C(19B)	127(4)	204(5)	117(4)	61(4)	33(3)	50(3)
C(20B)	127(4)	174(5)	141(4)	66(4)	53(3)	51(3)
C(21B)	105(4)	209(5)	135(4)	65(4)	41(3)	51(3)
C(22B)	167(4)	261(6)	174(5)	54(4)	78(4)	90(4)
C(23B)	118(4)	194(5)	148(5)	70(4)	37(3)	44(3)
C(24B)	137(4)	256(6)	154(5)	51(4)	7(4)	40(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for alcohol 182 (CCDC 606989).

	x	y	z	U_{iso}
H(6A)	4022(19)	3125(16)	5005(13)	34(5)
H(2A)	9944(17)	1133(14)	8691(12)	29(5)
H(3A)	7926(17)	956(14)	7756(12)	29(5)
H(5A)	6455(17)	1346(14)	9721(11)	25(4)
H(6A1)	8415(15)	1494(12)	10728(10)	16(4)
H(9A1)	3480(20)	-272(16)	5676(14)	44(6)
H(9A2)	4130(20)	-548(16)	6491(14)	43(6)
H(9A3)	4890(20)	-310(17)	5886(14)	45(6)
H(12A)	2831(18)	3565(15)	7202(12)	33(5)
H(13A)	4643(15)	3019(12)	7703(10)	16(4)
H(15A)	5328(15)	4527(12)	6094(10)	15(4)
H(16A)	3834(16)	4872(13)	6823(11)	22(4)
H(16B)	2907(16)	4157(13)	5905(11)	20(4)
H(17A)	7453(19)	5236(15)	7498(13)	36(5)
H(17B)	7248(19)	5884(16)	8368(14)	39(5)
H(17C)	6440(20)	5868(18)	7502(15)	53(7)
H(19A)	6485(16)	2343(13)	5581(11)	20(4)
H(22A)	9760(20)	1990(16)	6439(13)	39(5)
H(22B)	8910(20)	1969(17)	5596(14)	42(6)
H(22C)	8670(20)	1125(18)	5922(14)	50(6)
H(24A)	10882(17)	3196(15)	9758(12)	29(5)
H(24B)	10482(17)	4239(14)	10234(12)	28(5)
H(24C)	11386(16)	4291(13)	9709(10)	19(4)
H(6B)	10850(20)	2237(17)	4856(14)	45(6)
H(2B)	4532(16)	3722(13)	977(11)	20(4)
H(3B)	6458(17)	3951(14)	1970(12)	27(4)
H(5B)	8127(17)	3839(14)	87(11)	26(4)
H(6B1)	6152(15)	3634(12)	-935(10)	15(4)
H(9B1)	9470(20)	5439(16)	4110(14)	43(6)
H(9B2)	9907(19)	5870(16)	3429(13)	38(5)
H(9B3)	10870(20)	5685(16)	4206(13)	42(6)
H(12B)	12161(15)	2122(12)	2706(10)	17(4)
H(13B)	10187(16)	2419(13)	2165(11)	21(4)
H(15B)	9775(15)	799(13)	3743(10)	18(4)
H(16C)	12138(17)	1399(14)	3962(12)	27(4)
H(16D)	11321(18)	673(15)	3039(12)	33(5)
H(17D)	8936(16)	-580(13)	2410(11)	19(4)
H(17E)	8073(17)	-680(14)	1479(12)	29(5)
H(17F)	7731(18)	-109(14)	2320(12)	30(5)
H(19B)	8272(17)	2803(14)	4277(12)	28(5)
H(22D)	4980(20)	3105(15)	3392(13)	37(5)
H(22E)	6070(20)	3520(17)	4226(14)	45(6)
H(22F)	5250(20)	2420(18)	3935(14)	50(6)
H(24D)	3248(16)	632(13)	160(11)	20(4)
H(24E)	4109(18)	552(15)	-413(12)	31(5)
H(24F)	3812(18)	1649(15)	20(12)	32(5)

Table 6. Hydrogen bonds for alcohol 182 (CCDC 606989) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(6A)-H(6A)...O(4B)#1	0.76(2)	2.26(2)	3.0125(12)	169(2)
O(6B)-H(6B)...O(4A)#2	0.79(2)	2.16(2)	2.9238(12)	163(2)

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

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Furan 169

(CCDC 602164)

Contents

Figure A2.3.2 Representation of Furan **169**.

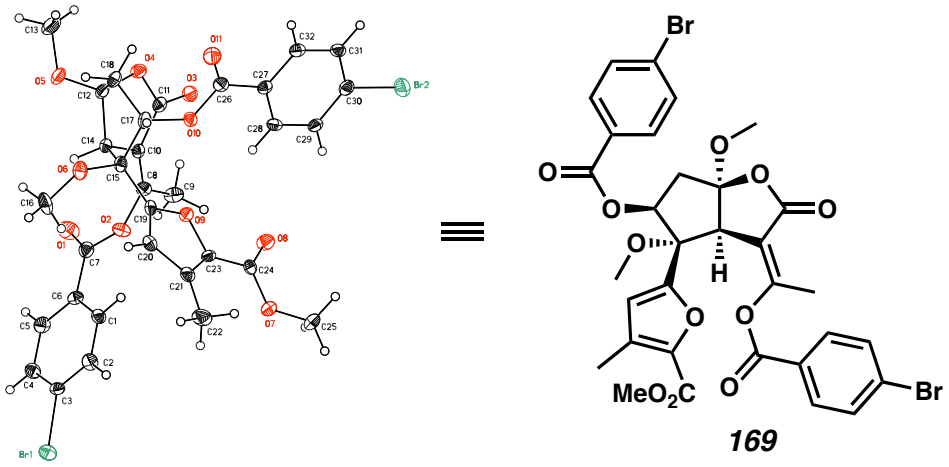


Table 1. Crystal data and structure refinement for furan 169 (CCDC 602164).

Empirical formula	$C_{32}H_{28}O_{11}Br_2$
Formula weight	748.36
Crystallization Solvent	Ethylacetate/n-heptane
Crystal Habit	Blade
Crystal size	0.31 x 0.24 x 0.11 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 10496 reflections used in lattice determination	2.30 to 28.23°
Unit cell dimensions	a = 17.2333(11) Å b = 10.2395(7) Å β= 104.5150(10)° c = 18.3237(12) Å
Volume	3130.2(4) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.588 Mg/m^3
F(000)	1512
Data collection program	Bruker SMART v5.630
θ range for data collection	1.22 to 28.58°
Completeness to θ = 28.58°	93.7 %
Index ranges	$-22 \le h \le 22$, $-13 \le k \le 13$, $-23 \le l \le 23$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	49507
Independent reflections	49509 [R_{int}= 0.0820]
Absorption coefficient	2.651 mm^{-1}
Absorption correction	TWINABS
Max. and min. transmission	1.0000 and 0.6884

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	Geometric positions
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	49509 / 0 / 412
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.273
Final R indices [I>2σ(I), 35818 reflections]	R1 = 0.0465, *w*R2 = 0.0991
R indices (all data)	R1 = 0.0708, *w*R2 = 0.1032
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Largest diff. peak and hole	1.202 and -0.725 e.Å^{-3}

Special Refinement Details

The crystal is twinned. CELL_NOW was used to define the two domains and produce a matrix for the twin law as follows;

751 reflections within 0.100 of an integer index assigned to domain 1,
751 of them exclusively; 248 reflections not yet assigned to a domain

Rotated from first domain by 179.7 degrees about
reciprocal axis 1.000 0.002 0.000 and real axis 1.000 -0.003 0.237

474 reflections within 0.100 of an integer index assigned to domain 2,
233 of them exclusively; 14 reflections not yet assigned to a domain

Twin Law:
Transforms h1 -> h2
1.00010 -0.00089 0.47107
0.00022 -0.99985 0.00031
-0.00044 -0.00057 -1.00002

The data was integrated with SAINT then TWINABS was used to write the file used for refinement. From TWINABS;

13830 data (3969 unique) involve component 1 only, mean I/sigma 7.8
13557 data (3916 unique) involve component 2 only, mean I/sigma 5.4
23827 data (6687 unique) involve 2 components, mean I/sigma 7.3
36 data (36 unique) involve 3 components, mean I/sigma 4.0

Refinement using an HKLF 5 type file produced a final set of atomic parameters and a scale factor between the two domains, BASF=0.33261

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.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for furan 169 (CCDC 602164). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Br(1)	1046(1)	2805(1)	-1563(1)	22(1)
Br(2)	6652(1)	13142(1)	1456(1)	27(1)
O(1)	1187(1)	9543(1)	-1711(1)	35(1)
O(2)	2058(1)	9038(1)	-609(1)	20(1)
O(3)	3071(1)	12918(1)	41(1)	21(1)
O(4)	2149(1)	12868(1)	709(1)	19(1)
O(5)	786(1)	12476(1)	499(1)	23(1)
O(6)	1221(1)	9189(1)	1375(1)	22(1)
O(7)	4613(1)	6506(1)	716(1)	22(1)
O(8)	4502(1)	8654(1)	420(1)	23(1)
O(9)	3103(1)	8783(1)	845(1)	16(1)
O(10)	3048(1)	10949(1)	1772(1)	17(1)
O(11)	3322(1)	12169(1)	2836(1)	25(1)
C(1)	1757(1)	6474(2)	-736(1)	18(1)
C(2)	1655(1)	5142(2)	-808(1)	21(1)
C(3)	1145(1)	4653(2)	-1455(1)	16(1)
C(4)	728(1)	5454(2)	-2020(1)	20(1)
C(5)	839(1)	6796(2)	-1941(1)	21(1)
C(6)	1362(1)	7304(2)	-1303(1)	17(1)
C(7)	1500(1)	8738(2)	-1257(1)	20(1)
C(8)	2396(1)	10292(2)	-517(1)	17(1)
C(9)	2936(1)	10604(2)	-1014(1)	25(1)
C(10)	2250(1)	10981(2)	45(1)	14(1)
C(11)	2562(1)	12316(2)	235(1)	17(1)
C(12)	1542(1)	11995(2)	853(1)	18(1)
C(13)	607(1)	13733(2)	765(1)	32(1)
C(14)	1668(1)	10657(2)	502(1)	14(1)
C(15)	1918(1)	9702(2)	1191(1)	17(1)
C(16)	684(1)	8458(2)	792(1)	30(1)
C(17)	2263(1)	10637(2)	1858(1)	17(1)
C(18)	1709(1)	11803(2)	1706(1)	19(1)
C(19)	2474(1)	8589(2)	1155(1)	15(1)
C(20)	2489(1)	7352(2)	1426(1)	19(1)
C(21)	3171(1)	6719(2)	1281(1)	18(1)
C(22)	3450(1)	5356(2)	1519(1)	28(1)
C(23)	3523(1)	7618(2)	921(1)	15(1)
C(24)	4257(1)	7678(2)	656(1)	17(1)
C(25)	5373(1)	6476(2)	504(1)	26(1)
C(26)	3520(1)	11758(2)	2291(1)	19(1)
C(27)	4283(1)	12027(2)	2093(1)	16(1)
C(28)	4504(1)	11392(2)	1500(1)	17(1)
C(29)	5210(1)	11697(2)	1320(1)	17(1)
C(30)	5693(1)	12672(2)	1724(1)	19(1)
C(31)	5490(1)	13312(2)	2315(1)	19(1)
C(32)	4787(1)	12986(2)	2499(1)	18(1)

Table 3. Bond lengths [Å] and angles [°] for furan 169 (CCDC 602164).

Br(1)-C(3)	1.9056(18)	C(11)-O(4)-C(12)	111.90(14)
Br(2)-C(30)	1.900(2)	C(12)-O(5)-C(13)	114.44(15)
O(1)-C(7)	1.198(2)	C(15)-O(6)-C(16)	116.16(14)
O(2)-C(7)	1.363(2)	C(24)-O(7)-C(25)	114.82(15)
O(2)-C(8)	1.402(2)	C(19)-O(9)-C(23)	106.47(14)
O(3)-C(11)	1.196(2)	C(26)-O(10)-C(17)	118.12(15)
O(4)-C(11)	1.376(2)	C(2)-C(1)-C(6)	120.62(18)
O(4)-C(12)	1.450(2)	C(1)-C(2)-C(3)	118.70(18)
O(5)-C(12)	1.392(2)	C(4)-C(3)-C(2)	122.03(18)
O(5)-C(13)	1.438(2)	C(4)-C(3)-Br(1)	119.91(15)
O(6)-C(15)	1.425(2)	C(2)-C(3)-Br(1)	118.05(14)
O(6)-C(16)	1.437(2)	C(3)-C(4)-C(5)	118.68(18)
O(7)-C(24)	1.340(2)	C(6)-C(5)-C(4)	120.12(18)
O(7)-C(25)	1.458(2)	C(1)-C(6)-C(5)	119.81(18)
O(8)-C(24)	1.206(2)	C(1)-C(6)-C(7)	121.52(18)
O(9)-C(19)	1.357(2)	C(5)-C(6)-C(7)	118.65(17)
O(9)-C(23)	1.385(2)	O(1)-C(7)-O(2)	123.04(19)
O(10)-C(26)	1.366(2)	O(1)-C(7)-C(6)	127.05(19)
O(10)-C(17)	1.436(2)	O(2)-C(7)-C(6)	109.91(17)
O(11)-C(26)	1.209(2)	C(10)-C(8)-O(2)	115.29(18)
C(1)-C(2)	1.377(2)	C(10)-C(8)-C(9)	129.09(19)
C(1)-C(6)	1.383(2)	O(2)-C(8)-C(9)	115.29(16)
C(2)-C(3)	1.381(2)	C(8)-C(10)-C(11)	123.14(18)
C(3)-C(4)	1.374(2)	C(8)-C(10)-C(14)	127.04(18)
C(4)-C(5)	1.390(2)	C(11)-C(10)-C(14)	109.23(16)
C(5)-C(6)	1.386(2)	O(3)-C(11)-O(4)	120.67(18)
C(6)-C(7)	1.486(3)	O(3)-C(11)-C(10)	131.11(19)
C(8)-C(10)	1.324(2)	O(4)-C(11)-C(10)	108.22(17)
C(8)-C(9)	1.489(2)	O(5)-C(12)-O(4)	109.29(15)
C(10)-C(11)	1.478(2)	O(5)-C(12)-C(18)	115.84(17)
C(10)-C(14)	1.495(2)	O(4)-C(12)-C(18)	107.84(16)
C(12)-C(18)	1.529(2)	O(5)-C(12)-C(14)	109.33(16)
C(12)-C(14)	1.552(2)	O(4)-C(12)-C(14)	106.62(15)
C(14)-C(15)	1.570(2)	C(18)-C(12)-C(14)	107.52(15)
C(15)-C(19)	1.502(3)	C(10)-C(14)-C(12)	102.71(15)
C(15)-C(17)	1.548(2)	C(10)-C(14)-C(15)	120.16(16)
C(17)-C(18)	1.511(2)	C(12)-C(14)-C(15)	104.72(14)
C(19)-C(20)	1.359(2)	O(6)-C(15)-C(19)	108.41(15)
C(20)-C(21)	1.423(3)	O(6)-C(15)-C(17)	102.80(15)
C(21)-C(23)	1.361(2)	C(19)-C(15)-C(17)	112.34(16)
C(21)-C(22)	1.504(2)	O(6)-C(15)-C(14)	110.07(16)
C(23)-C(24)	1.467(3)	C(19)-C(15)-C(14)	119.06(16)
C(26)-C(27)	1.475(3)	C(17)-C(15)-C(14)	102.90(15)
C(27)-C(32)	1.397(3)	O(10)-C(17)-C(18)	111.90(16)
C(27)-C(28)	1.398(2)	O(10)-C(17)-C(15)	103.75(15)
C(28)-C(29)	1.374(3)	C(18)-C(17)-C(15)	104.04(15)
C(29)-C(30)	1.388(2)	C(17)-C(18)-C(12)	103.76(15)
C(30)-C(31)	1.384(2)	O(9)-C(19)-C(20)	110.22(17)
C(31)-C(32)	1.377(3)	O(9)-C(19)-C(15)	119.53(17)
		C(20)-C(19)-C(15)	130.16(19)
C(7)-O(2)-C(8)	119.53(15)	C(19)-C(20)-C(21)	107.29(18)

C(23)-C(21)-C(20)	105.73(17)
C(23)-C(21)-C(22)	128.2(2)
C(20)-C(21)-C(22)	125.96(18)
C(21)-C(23)-O(9)	110.29(17)
C(21)-C(23)-C(24)	135.72(19)
O(9)-C(23)-C(24)	113.77(16)
O(8)-C(24)-O(7)	125.31(19)
O(8)-C(24)-C(23)	124.23(19)
O(7)-C(24)-C(23)	110.46(17)
O(11)-C(26)-O(10)	123.0(2)
O(11)-C(26)-C(27)	126.35(19)
O(10)-C(26)-C(27)	110.68(17)
C(32)-C(27)-C(28)	119.08(19)
C(32)-C(27)-C(26)	118.29(17)
C(28)-C(27)-C(26)	122.59(19)
C(29)-C(28)-C(27)	120.74(19)
C(28)-C(29)-C(30)	118.97(18)
C(31)-C(30)-C(29)	121.5(2)
C(31)-C(30)-Br(2)	119.30(16)
C(29)-C(30)-Br(2)	119.23(15)
C(32)-C(31)-C(30)	119.16(19)
C(31)-C(32)-C(27)	120.55(18)

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for furan 169 (CCDC 602164). 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}
Br(1)	259(1)	175(1)	232(1)	-2(1)	60(1)	-29(1)
Br(2)	217(1)	295(1)	321(1)	-33(1)	84(1)	-52(1)
O(1)	409(12)	190(8)	336(9)	75(7)	-96(8)	28(8)
O(2)	278(10)	146(8)	156(8)	-5(6)	22(7)	-13(7)
O(3)	187(9)	183(8)	266(8)	10(7)	98(7)	-23(7)
O(4)	202(9)	162(8)	232(8)	-2(6)	101(7)	6(7)
O(5)	163(9)	238(9)	276(8)	6(6)	50(7)	74(7)
O(6)	161(9)	270(9)	240(8)	29(6)	98(7)	-26(7)
O(7)	190(9)	174(8)	288(8)	-13(6)	64(7)	32(7)
O(8)	256(10)	190(8)	273(8)	27(7)	119(7)	14(7)
O(9)	163(9)	151(7)	180(7)	25(6)	57(7)	5(6)
O(10)	149(9)	199(8)	161(7)	-36(6)	39(7)	-12(7)
O(11)	250(9)	324(9)	186(8)	-78(7)	87(7)	4(8)
C(1)	176(13)	181(12)	158(11)	-17(9)	2(10)	-4(10)
C(2)	213(14)	201(12)	191(12)	42(9)	30(11)	-11(10)
C(3)	184(13)	135(11)	176(11)	-18(9)	72(10)	6(10)
C(4)	208(13)	229(12)	150(11)	-37(9)	22(10)	-9(10)
C(5)	231(14)	200(12)	178(11)	35(9)	14(10)	33(10)
C(6)	201(13)	150(11)	168(11)	1(9)	72(9)	21(10)
C(7)	183(14)	225(12)	189(12)	-11(10)	50(10)	12(11)
C(8)	207(14)	113(11)	174(11)	51(9)	5(10)	19(10)
C(9)	322(15)	188(12)	250(12)	-10(10)	115(11)	16(11)
C(10)	132(12)	131(11)	153(11)	47(9)	15(10)	18(9)
C(11)	165(13)	184(12)	134(11)	34(9)	7(10)	42(10)
C(12)	144(13)	179(12)	208(11)	3(9)	56(10)	35(10)
C(13)	287(15)	274(13)	411(15)	14(11)	101(12)	117(12)
C(14)	125(12)	159(11)	144(11)	9(9)	21(9)	12(9)
C(15)	135(13)	194(12)	188(12)	26(9)	72(10)	-18(10)
C(16)	183(14)	335(14)	345(14)	41(11)	18(11)	-110(11)
C(17)	138(13)	236(12)	145(11)	18(9)	59(10)	11(10)
C(18)	153(13)	230(12)	198(11)	4(9)	87(10)	35(10)
C(19)	110(12)	199(12)	125(11)	2(9)	17(9)	-8(10)
C(20)	174(13)	191(12)	190(11)	-3(10)	44(10)	-45(10)
C(21)	182(14)	187(11)	147(11)	-20(9)	-24(10)	-6(10)
C(22)	338(16)	166(12)	330(14)	39(10)	67(12)	-22(11)
C(23)	172(13)	119(11)	142(11)	-10(9)	9(9)	36(9)
C(24)	147(13)	197(12)	136(11)	-28(9)	-20(9)	-4(10)
C(25)	201(14)	243(13)	332(13)	-28(10)	62(11)	77(11)
C(26)	203(14)	134(11)	193(11)	43(9)	-5(10)	24(10)
C(27)	180(13)	143(11)	128(10)	24(9)	14(9)	44(10)
C(28)	213(14)	120(11)	152(11)	-15(9)	11(10)	7(10)
C(29)	181(13)	163(11)	171(11)	-23(9)	48(10)	41(10)
C(30)	184(13)	177(11)	214(11)	34(10)	44(10)	39(10)
C(31)	208(14)	135(11)	199(12)	-13(9)	-12(10)	24(10)
C(32)	218(14)	159(12)	137(11)	-11(9)	-3(10)	48(10)