Supporting Information

Total Synthesis and Structural Revision of (±)-Nidemone

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Comparison of ^{1}H NMR data for natural nidemone, synthetic 1, and our previously synthetic nidemone

Natural nidemone (300 MHz, $CDCl_3$) δ (ppm)	Synthetic **1** (500 MHz, $CDCl_3$) δ (ppm)	Our previously synthetic nidemone (500 MHz, $CDCl_3$) δ (ppm)
12.23 (s, 1H)	12.22 (s, 1H)	12.13 (s, 1H)
7.39 (dd, J = 7.5, 7.5 Hz, 1H)	7.39 (dd, J = 8.5, 7.5 Hz, 1H)	7.42 (dd, J = 8.5, 7.5 Hz, 1H)
6.80 (dd, J = 7.5, 1.0 Hz, 1H)	6.80 (d, J = 8.5 Hz, 1H)	6.84 (d, J = 8.5 Hz, 1H)
6.72 (brdd, J = 7.5, 1.0 Hz, 1H)	6.71 (d, J = 7.5 Hz, 1H)	6.72 (d, J = 7.5 Hz, 1H)
5.27 (t, J = 1.2 Hz, 1H)	5.26 (s, 1H)	5.49 (s, 1H)
3.91 (s, 3H)	3.91 (s, 3H)	3.88 (s, 3H)
3.48 (brddd, J = 16.7, 9.4, 5.0 Hz, 1H)	3.48 (ddd, J = 17.0, 9.5, 5.0 Hz, 1H)	3.06–3.02 (m, 2H)
3.29 (dd, J = 17.5, 1.5 Hz, 1H)	3.29 (d, J = 17.5 Hz, 1H)	
2.87 (ddd, J = 16.8, 5.8, 5.8 Hz, 1H)	2.86 (ddd, J = 17.0, 5.5, 5.5 Hz, 1H)	2.67 (d, J = 17.5 Hz, 1H)
2.48 (dd, J = 17.5, 1.5 Hz, 1H)	2.47 (d, J = 17.5 Hz, 1H)	2.66–2.60 (m, 1H)
2.47 (ddd, J = 14.0, 7.4, 4.0 Hz, 1H)	2.46 (ddd, J = 14.0, 5.5, 5.5 Hz, 1H)	2.57 (d, J = 17.5 Hz, 1H)
2.20 (ddd, J = 13.8, 9.3, 4.8 Hz, 1H)	2.20 (ddd, J = 14.0, 9.5, 5.0 Hz, 1H)	2.04 (ddd, J = 13.5, 5.1, 5.1 Hz, 1H)

Comparison of ^{13}C NMR data for natural nidemone, synthetic 1, and our previously synthetic nidemone

Natural nidemone (75 MHz, $CDCl_3$) δ (ppm)	Synthetic **1** (125 MHz, $CDCl_3$) δ (ppm)	Our previously synthetic nidemone (125 MHz, $CDCl_3$) δ (ppm)
202.7 (C)	202.7 (C)	202.8 (C)
202.3 (C)	202.2 (C)	201.9 (C)
189.6 (C)	189.6 (C)	190.8 (C)
163.4 (C)	163.4 (C)	163.9 (C)
145.4 (C)	145.3 (C)	144.6 (C)
137.0 (CH)	137.0 (CH)	137.5 (CH)
118.9 (CH)	118.9 (CH)	119.2 (CH)
116.4 (C)	116.4 (C)	116.4 (CH)
115.8 (CH)	115.8(CH)	116.4 (C)
101.9 (CH)	101.9 (CH)	105.8 (CH)
59.2 (C)	59.2 (C)	59.6 (CH_3)
59.1 (CH_3)	59.1 (CH_3)	55.7 (C)
39.7 (CH_2)	39.7 (CH_2)	44.9 (CH_2)
32.1 (CH_2)	32.1 (CH_2)	32.1 (CH_2)
25.6 (CH_2)	25.6 (CH_2)	26.5 (CH_2)

Table 1. Crystal data and structure refinement for LCY14.

Identification code	lcy14
Empirical formula	C H O
Formula weight	29.02
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic , Pbca
Unit cell dimensions	a = 10.287(2) A alpha = 90 deg. b = 11.291(2) A beta = 90 deg. c = 20.866(4) A gamma = 90 deg.
Volume	2423.6(9) A3
Z, Calculated density	71, 1.412 Mg/m^3
Absorption coefficient	0.131 mm-1
F(000)	1065
Crystal size	0.38 x 0.16 x 0.15 mm^3
Theta range for data collection	1.95 to 28.33 deg.
Limiting indices	-13<=h<=13, -15<=k<=14, -27<=l<=27
Reflections collected / unique	26046 / 2965 [R(int) = 0.0253]
Completeness to theta = 28.33	98.3 %
Refinement method	Full-matrix least-squares on F2

Data / restraints / parameters	2965 / 0 / 228
Goodness-of-fit on F^2	1.098
Final R indices [I>2sigma(I)]	R1 = 0.0406, wR2 = 0.1169
R indices (all data)	R1 = 0.0506, wR2 = 0.1324
Largest diff. peak and hole	0.340 and -0.300 e.A^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for LCY14.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(10)	1216(1)	1654(1)	4415(1)	22(1)
C(5)	926(1)	1646(1)	3729(1)	24(1)
C(13)	3043(1)	1760(1)	5702(1)	24(1)
C(12)	3781(1)	1104(1)	5300(1)	25(1)
C(9)	2373(1)	2370(1)	4658(1)	23(1)
C(14)	2216(1)	2658(1)	5373(1)	28(1)
C(11)	3530(1)	1474(1)	4650(1)	22(1)
C(6)	1603(1)	2388(1)	3295(1)	30(1)
C(8)	2595(1)	3472(1)	4242(1)	31(1)
C(4)	-58(1)	880(1)	3495(1)	30(1)
C(7)	2656(2)	3210(1)	3527(1)	36(1)
C(3)	-351(2)	861(2)	2843(1)	42(1)
C(1)	1285(2)	2341(2)	2650(1)	42(1)
C(2)	321(2)	1590(2)	2431(1)	46(1)
O(1)	2984(1)	1728(1)	6335(1)	32(1)
C(15)	3704(2)	787(2)	6637(1)	37(1)

O(3)	579(1)	1060(1)	4797(1)	30(1)
O(2)	4080(1)	1141(1)	4162(1)	30(1)
O(4)	-736(1)	142(1)	3877(1)	39(1)

Table 3. Bond lengths [A] and angles [deg] for LCY14.

C(10)-O(3)	1.2307(15)
C(10)-C(5)	1.4637(17)
C(10)-C(9)	1.5243(16)
C(5)-C(6)	1.4159(18)
C(5)-C(4)	1.4180(18)
C(13)-O(1)	1.3227(15)
C(13)-C(12)	1.3516(17)
C(13)-C(14)	1.4915(18)
C(12)-C(11)	1.4439(17)
C(9)-C(8)	1.5331(17)
C(9)-C(14)	1.5354(17)
C(9)-C(11)	1.5623(16)
C(11)-O(2)	1.2232(15)
C(6)-C(1)	1.386(2)
C(6)-C(7)	1.506(2)
C(8)-C(7)	1.522(2)
C(4)-O(4)	1.3476(18)
C(4)-C(3)	1.394(2)
C(3)-C(2)	1.377(3)
C(1)-C(2)	1.383(3)
O(1)-C(15)	1.4403(18)

O(3)-C(10)-C(5)	121.46(11)
O(3)-C(10)-C(9)	119.34(11)
C(5)-C(10)-C(9)	119.14(10)
C(6)-C(5)-C(4)	119.52(12)
C(6)-C(5)-C(10)	121.41(11)
C(4)-C(5)-C(10)	119.08(11)
O(1)-C(13)-C(12)	129.05(12)

O(1)-C(13)-C(14)	116.91(11)
C(12)-C(13)-C(14)	114.02(11)
C(13)-C(12)-C(11)	108.91(11)
C(10)-C(9)-C(8)	111.07(10)
C(10)-C(9)-C(14)	110.64(10)
C(8)-C(9)-C(14)	113.14(10)
C(10)-C(9)-C(11)	104.36(9)
C(8)-C(9)-C(11)	113.92(10)
C(14)-C(9)-C(11)	103.13(9)
C(13)-C(14)-C(9)	104.13(10)
O(2)-C(11)-C(12)	127.63(11)
O(2)-C(11)-C(9)	124.05(11)
C(12)-C(11)-C(9)	108.29(10)
C(1)-C(6)-C(5)	118.75(14)
C(1)-C(6)-C(7)	120.39(13)
C(5)-C(6)-C(7)	120.86(12)
C(7)-C(8)-C(9)	113.72(11)
O(4)-C(4)-C(3)	117.14(13)
O(4)-C(4)-C(5)	122.89(12)
C(3)-C(4)-C(5)	119.96(14)
C(6)-C(7)-C(8)	113.91(11)
C(2)-C(3)-C(4)	119.50(15)
C(2)-C(1)-C(6)	120.99(15)
C(3)-C(2)-C(1)	121.28(14)
C(13)-O(1)-C(15)	115.73(10)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for LCY14.

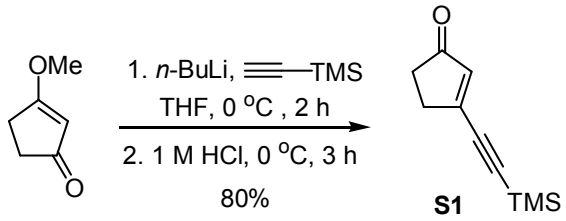
The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

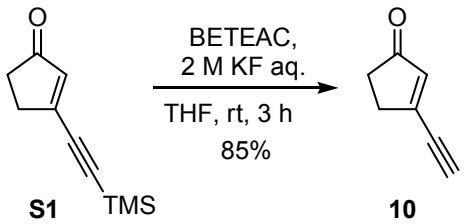
	U11	U22	U33	U23	U13	U12
C(10)	19(1)	19(1)	26(1)	0(1)	1(1)	2(1)
C(5)	23(1)	26(1)	25(1)	-1(1)	1(1)	4(1)
C(13)	20(1)	27(1)	26(1)	-5(1)	-2(1)	-3(1)
C(12)	20(1)	26(1)	29(1)	-2(1)	-1(1)	2(1)
C(9)	21(1)	20(1)	27(1)	-2(1)	-1(1)	1(1)
C(14)	26(1)	26(1)	30(1)	-9(1)	-2(1)	4(1)
C(11)	18(1)	19(1)	29(1)	-2(1)	1(1)	-2(1)
C(6)	33(1)	29(1)	28(1)	4(1)	5(1)	8(1)
C(8)	30(1)	21(1)	43(1)	3(1)	0(1)	-3(1)
C(4)	26(1)	32(1)	33(1)	-6(1)	-3(1)	5(1)
C(7)	36(1)	31(1)	40(1)	12(1)	7(1)	-2(1)
C(3)	42(1)	48(1)	36(1)	-14(1)	-11(1)	8(1)
C(1)	53(1)	44(1)	27(1)	6(1)	7(1)	14(1)
C(2)	58(1)	54(1)	26(1)	-7(1)	-7(1)	21(1)
O(1)	31(1)	38(1)	25(1)	-6(1)	-2(1)	2(1)
C(15)	34(1)	48(1)	28(1)	2(1)	-4(1)	2(1)
O(3)	28(1)	32(1)	29(1)	5(1)	2(1)	-7(1)
O(2)	28(1)	31(1)	30(1)	-2(1)	6(1)	4(1)
O(4)	29(1)	43(1)	43(1)	-4(1)	-5(1)	-11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for LCY14.

	x	y	z	U(eq)
H(1)	3337(17)	39(17)	6507(9)	41(4)
H(2)	3585(19)	898(18)	7093(10)	49(5)
H(3)	4640(20)	808(18)	6538(10)	52(5)
H(4)	4404(15)	496(14)	5404(8)	30(4)
H(6)	1315(18)	2609(16)	5513(8)	41(5)
H(5)	2529(18)	3448(17)	5480(8)	40(5)
H(12)	2572(19)	3952(17)	3304(9)	46(5)
H(11)	3524(17)	2862(16)	3429(9)	40(4)
H(14)	1830(16)	4024(15)	4330(8)	36(4)
H(10)	1749(19)	2859(18)	2359(9)	49(5)
H(13)	3402(17)	3876(16)	4392(8)	38(4)
H(9)	130(20)	1580(20)	1991(11)	64(6)
H(7)	-410(20)	290(20)	4281(12)	65(6)
H(8)	-1030(20)	354(18)	2700(9)	51(5)



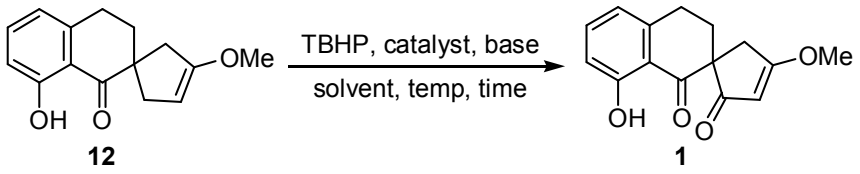
3-(Trimethylsilylethynyl)-2-cyclopenten-1-one (S1). To a stirred solution of trimethylsilylacetylene (336 mg, 3.42 mmol) in THF (3 mL) under Ar atmosphere at 0 °C was added *n*-BuLi (1.6 M in hexanes, 2.0 mL, 3.20 mmol) and stirred at 0 °C for 1 h. 3-Methoxy-2-cyclopenten-1-one (300 mg, 2.68 mmol) was added to the reaction mixture and this mixture was stirred at 0 °C for 2 h. The mixture was quenched with 1 M HCl at 0 °C and stirred at this temperature for 3 h. The aqueous solution was extracted with EtOAc, and the combined extracts were washed with brine, dried over $MgSO_4$, filtered and concentrated. The residue was purified by silica-gel chromatography (hexane/EtOAc = 6:1) to afford **S1** (382 mg, 80%) as a yellowish oil. IR (neat) ν 2961, 2150, 1709, 1582, 1267, 848 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.27 (t, J = 1.8 Hz, 1H), 2.74–2.70 (m, 2H), 2.42–2.39 (m, 2H), 0.23 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.6, 156.7, 136.8, 111.7, 99.7, 34.7, 32.5, –0.5; MS (EI) *m/z* (% base peak) 178 (M+, 22), 163 (100), 133 (1), 119 (1), 107 (8), 97 (2), 83 (4), 77 (3), 75 (3), 53 (2); HRMS (EI) calcd for $C_{10}H_{14}OSi$ 178.0814, Found 178.0815.



3-Ethynyl-2-cyclopenten-1-one (10). A mixture of **S1** (600 mg, 3.36 mmol) and benzyltriethylammonium chloride (BTEAC, 77 mg, 0.34 mmol) in THF (15 mL) at room temperature was added 2 M KF (3.5 mL). The reaction mixture was stirred at room temperature for 3 h and extracted with Et_2O. The combined extracts were washed with brine, dried over $MgSO_4$, filtered and concentrated.

The crude product was purified by column chromatography on silica gel (Et_2O/hexanes = 1:5) to afford **10** (304 mg, 85%) as a colorless solid. Mp 55–56 ^oC; IR (neat) ν 3194, 2926, 2854, 2095, 1701, 1664, 1578, 1435, 1285, 1177, 872, 729 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.35 (t, J = 1.6 Hz, 1H), 3.84 (s, 1H), 2.77–2.74 (m, 2H), 2.45–2.42 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.2, 155.8, 137.8, 92.2, 79.0, 34.6, 32.3; MS (EI) *m/z* (% base peak) 106 (M^+, 100), 105 (12), 78 (47), 77 (20), 52 (15), 51 (13); HRMS (ESI) calcd for C_7H_6O 106.0419, Found 106.0420.

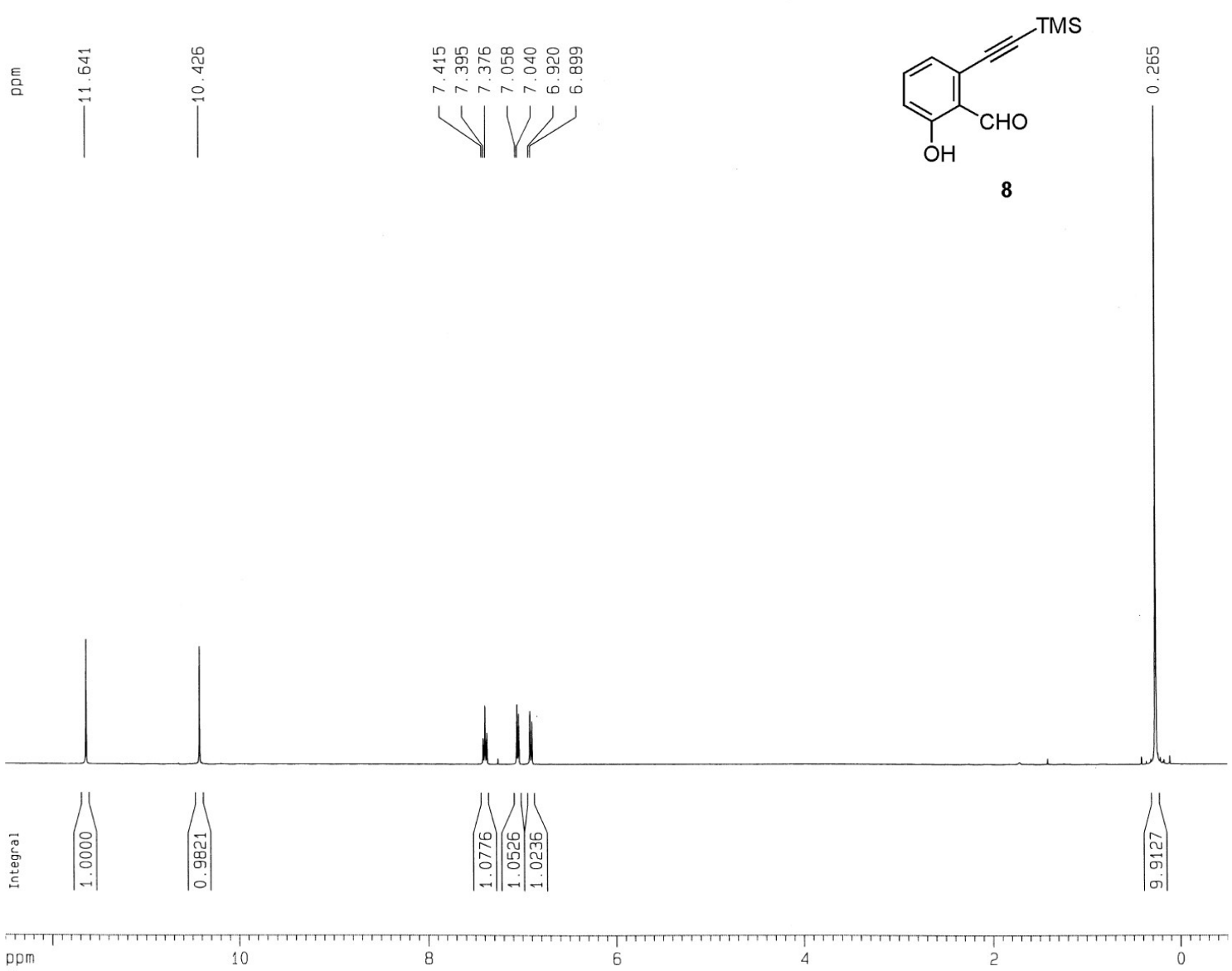
Table S1. Attempts to the regioselective allyic oxidation of 12



12 **1**

entry	catalyst	base	solvent	temp	time	**1** (yield)
1	$Rh(cap)_4$	K_2CO_3	CH_2Cl_2	0 °C to rt	3 h	–[a]
2	$Rh(cap)_4$	K_2CO_3	CH_2Cl_2	0 °C to rt	3 h	10%[b]
3	$Pd(OH)_2$	K_2CO_3	CH_2Cl_2	0 °C	1 d	–[a]
4	$Pd(OH)_2$	Cs_2CO_3	CH_2Cl_2	0 °C	1 d	–[a]
5	$PhI(OAc)_2$	–	EtOAc	0 °C to rt	3 h	7%[b]
6	$PhI(OAc)_2$	K_2CO_3	EtOAc	0 °C to rt	2 h	18%[b]
7	$PhI(OAc)_2$	Cs_2CO_3	EtOAc	0 °C to rt	2 h	14%[b]
8	$PhI(OAc)_2$	K_2CO_3	EtOAc	0 °C	5 h	22%[b]
9	$PhI(OAc)_2$	K_2CO_3	EtOAc	–10 °C	1 d	–[c]
10	$PhI(OAc)_2$	Na_2CO_3	EtOAc	0 °C	6 h	27%[b]
11	$Mn(OAc)_3$	–	EtOAc	rt	1 d	–[a]
12	$Mn(OAc)_3$	–	EtOAc	rt	1 d	–[a,b]

[a]Complicated mixture of products.
[b]Added 4A molecular sieve.
[c]Recovery of starting material.



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DE	6.50 usec
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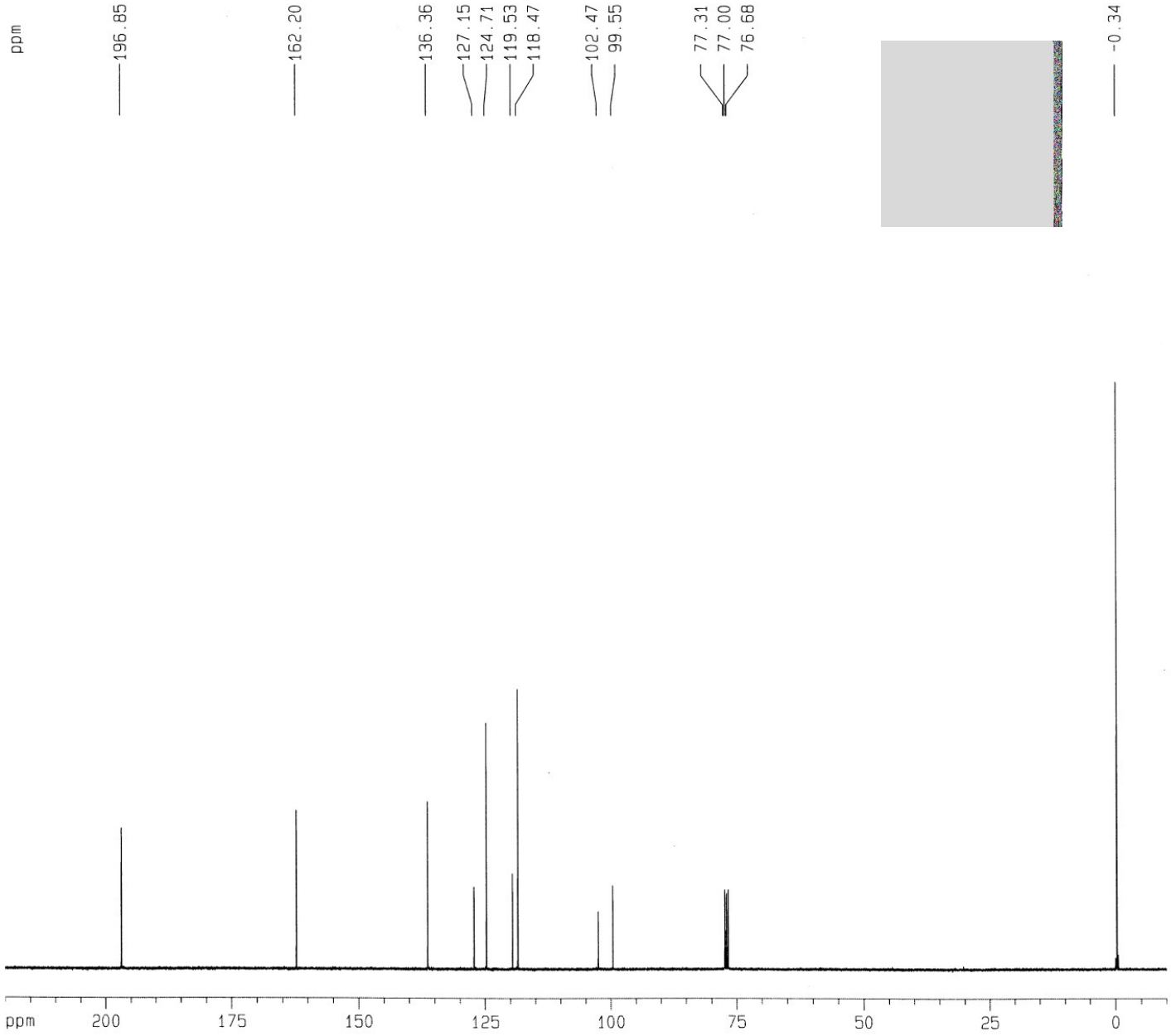
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	8192
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	12.500 ppm
F1	5001.62 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.60465 ppm/cm
HZCM	241.93907 Hz/cm



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d12 0.00002000 sec

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========== CHANNEL f2 ==========
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PL2 -3.00 dB
PL12 14.50 dB
PL13 17.50 dB
SFO2 400.1326008 MHz

F2 - Processing parameters
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
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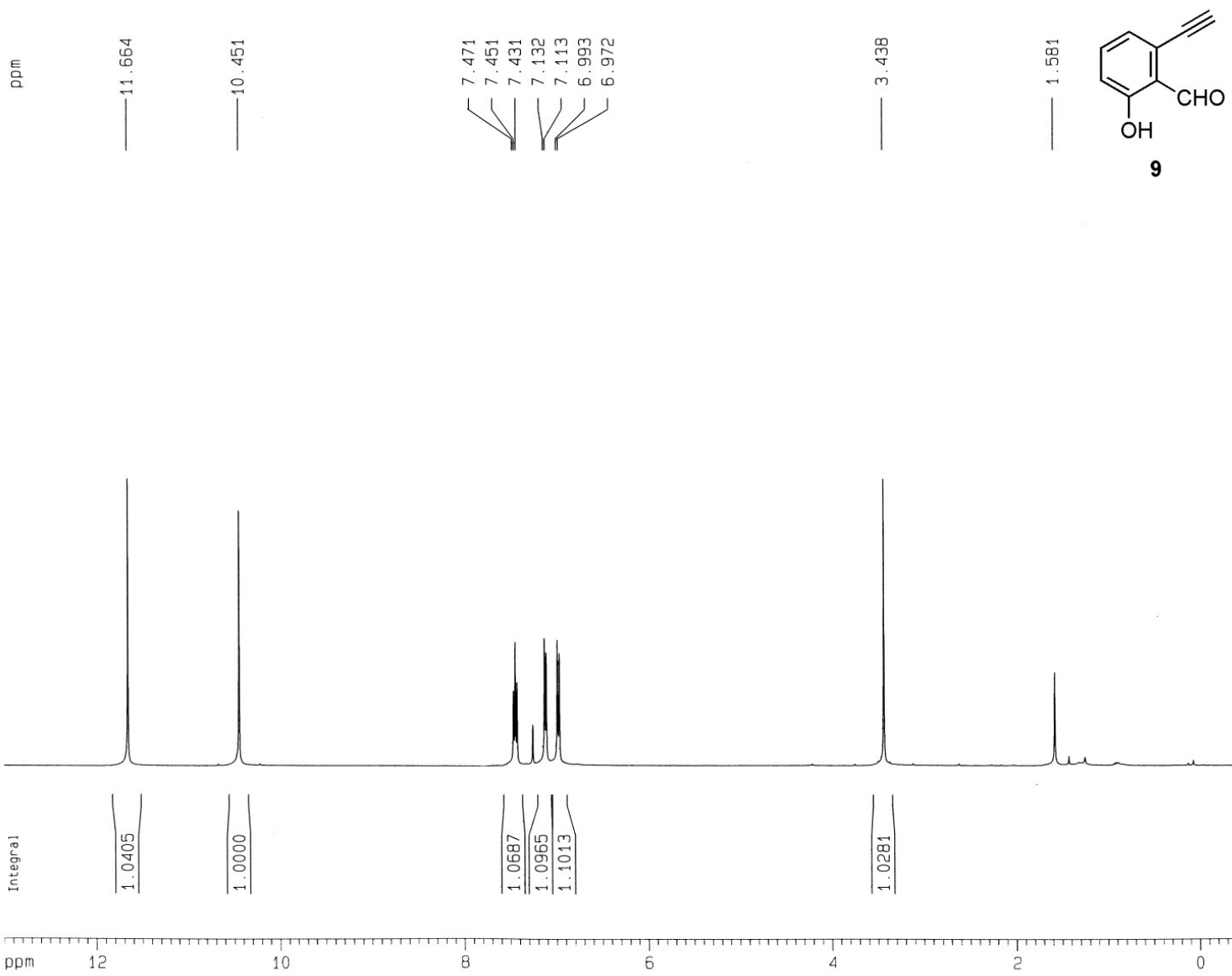

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AQ	1.3664756 sec
RG	4597.6
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	8192
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	13.000 ppm
F1	5201.69 Hz
F2P	-0.500 ppm
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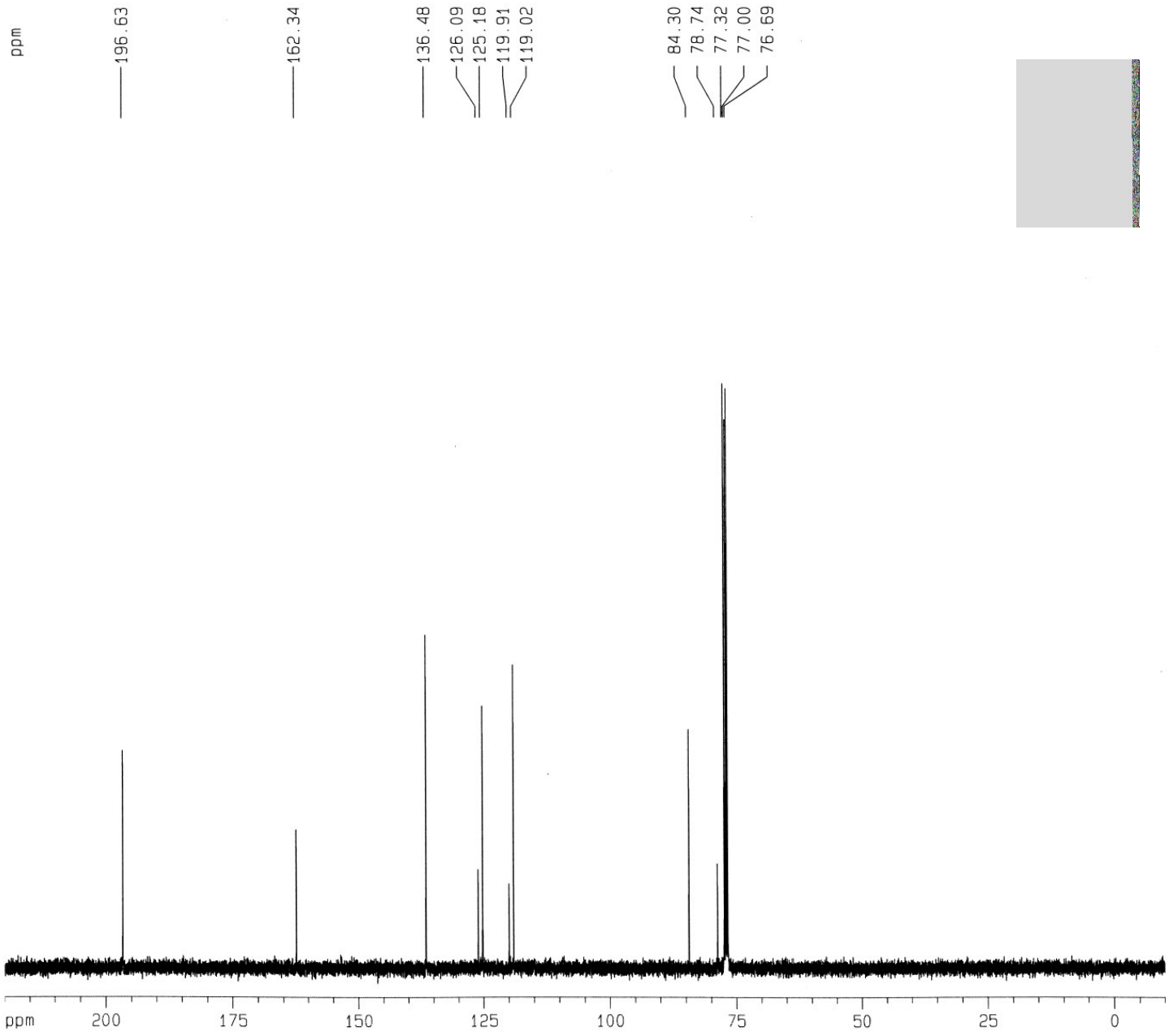
PPMCM	0.62791 ppm/cm
HZCM	251.24442 Hz/cm

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PROCNO                     1

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NS                      768
DS                        4
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FIDRES            0.383387 Hz
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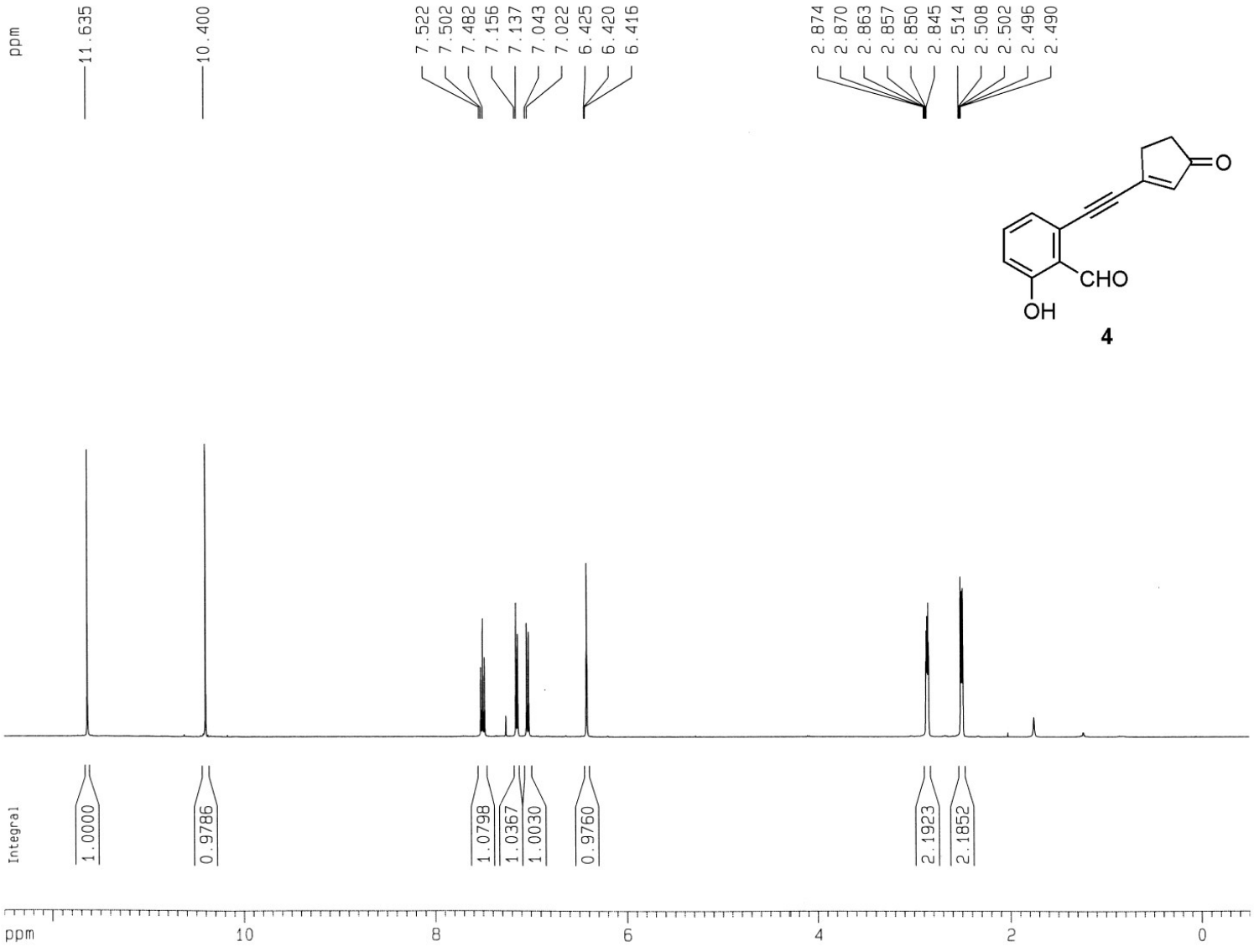
DW                 19.900 usec
DE                  6.50 usec
TE                  300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                    13C
P1                   10.20 usec
PL1                   0.00 dB
SFO1            100.6237959 MHz

============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -3.00 dB
PL12                 14.50 dB
PL13                 17.50 dB
SFO2            400.1326008 MHz

F2 - Processing parameters
SI                      32768
SF              100.6127700 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 220.000 ppm
F1                22134.81 Hz
F2P                 -10.000 ppm
F2                -1006.13 Hz
PPMCM             11.50000 ppm/cm
HZCM           1157.04688 Hz/cm
```



4

```
Current Data Parameters
NAME                  lcy-9
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20160305
Time                  19.30
INSTRUM               spect
PROBHD     5 mm QNP  1H
PULPROG                zg30
TD                    16384
SOLVENT               CDCl3
NS                       16
DS                        0
SWH               5995.204 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                   2298.8
DW                  83.400 usec
DE                   6.50 usec
TE                  300.0 K
D1               1.50000000 sec

============ CHANNEL f1 =============
NUC1                      1H
P1                     11.90 usec
PL1                    -3.00 dB
SFO1            400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF              400.1300091 MHz
WDW                       EM
SSB                       0
LB                     0.10 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    21.50 cm
F1P                  12.500 ppm
F1                  5001.62 Hz
F2P                  -0.500 ppm
F2                  -200.07 Hz
PPMCM              0.60465 ppm/cm
HZCM              241.93907 Hz/cm
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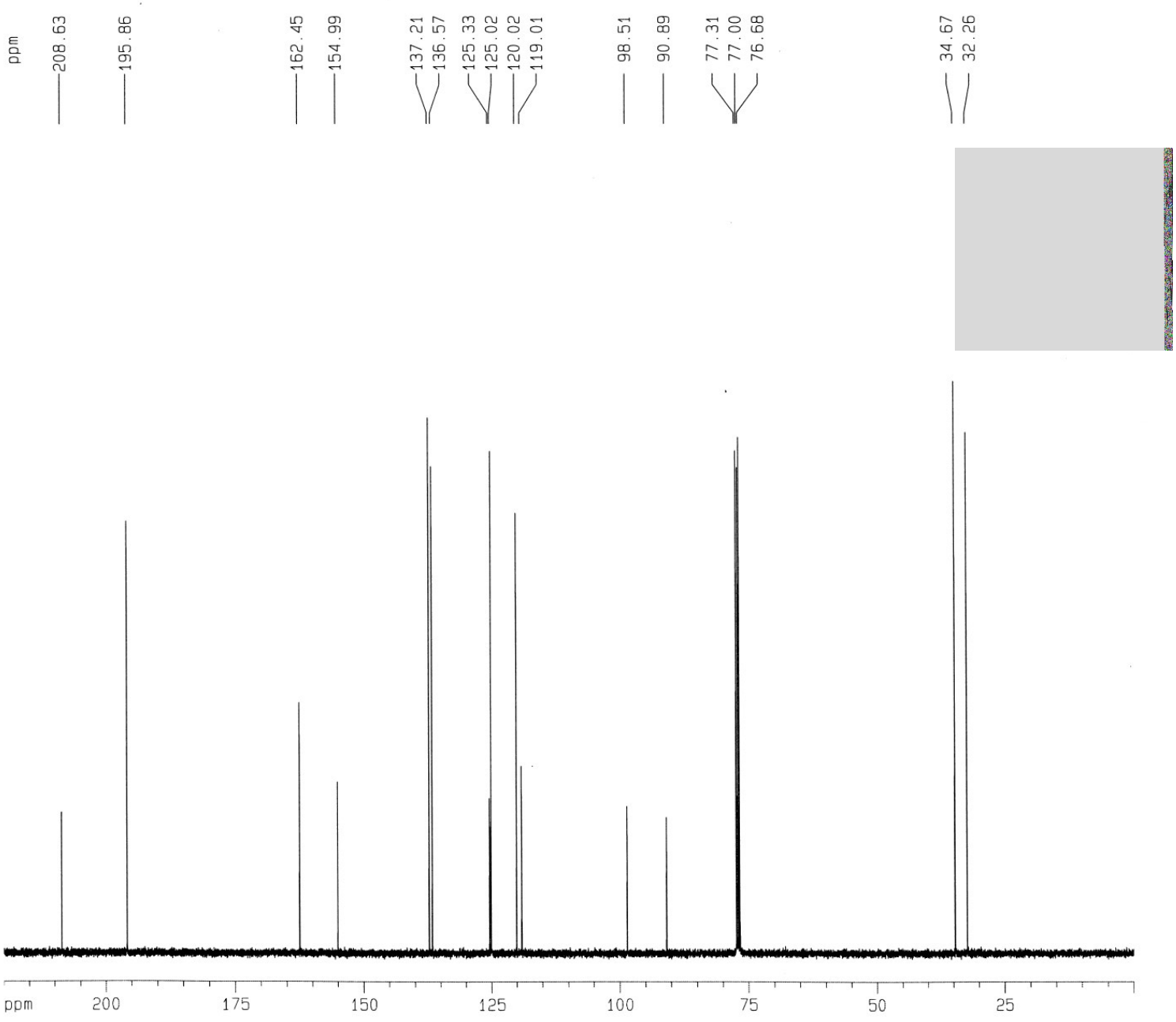


Current Data Parameters

NAME	lcy-9
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20160305
Time	20.27
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	256
DW	19.900 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	10.20 usec
PL1	0.00 dB
SF01	100.6237959 MHz

========== CHANNEL f2 ==========

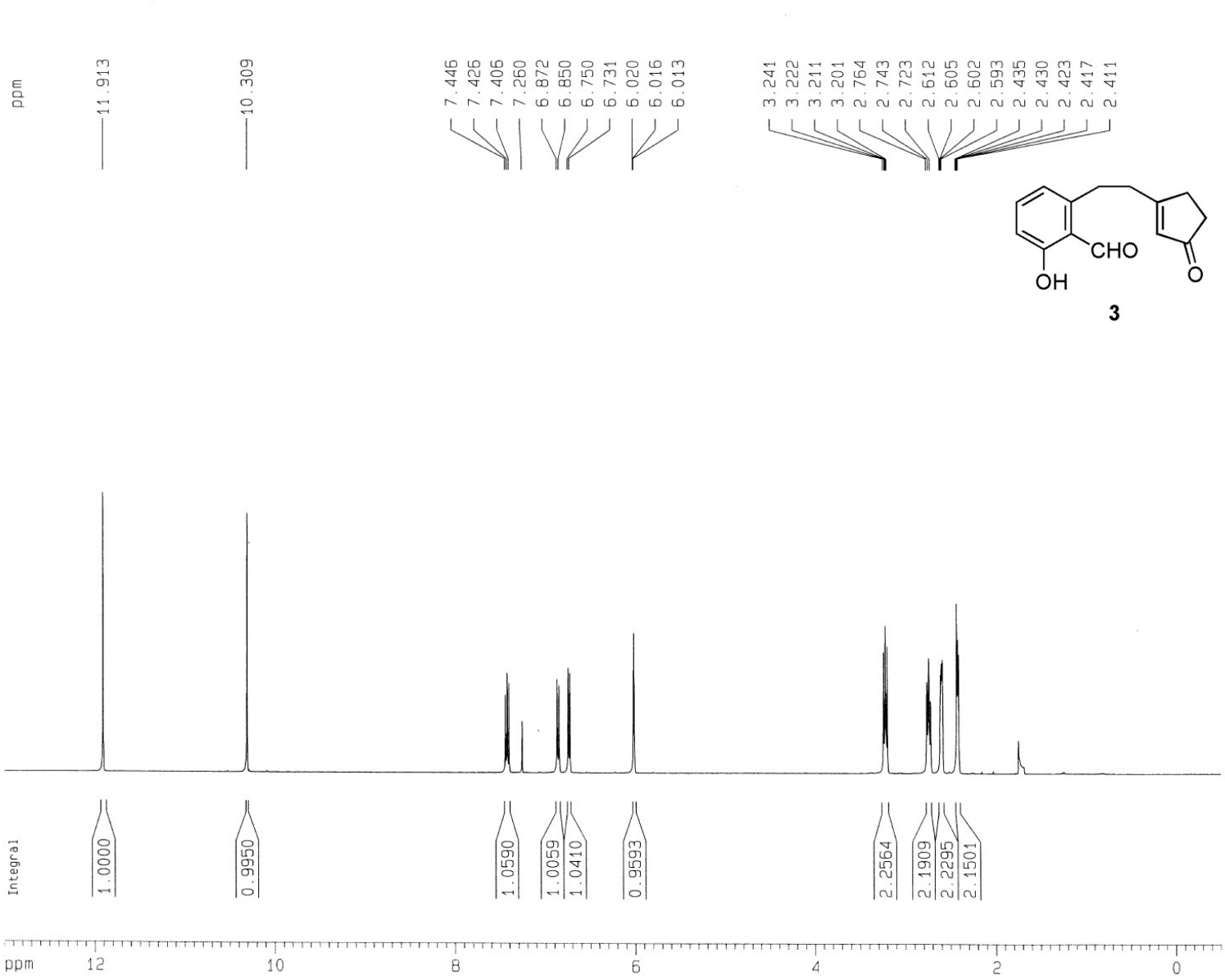
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-3.00 dB
PL12	14.50 dB
PL13	17.50 dB
SF02	400.1326008 MHz

F2 - Processing parameters

SI	32768
SF	100.6127754 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	11.00000 ppm/cm
HZCM	1106.74048 Hz/cm



Current Data Parameters

NAME	LYC-10
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160705
Time	21.32
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	228.1
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

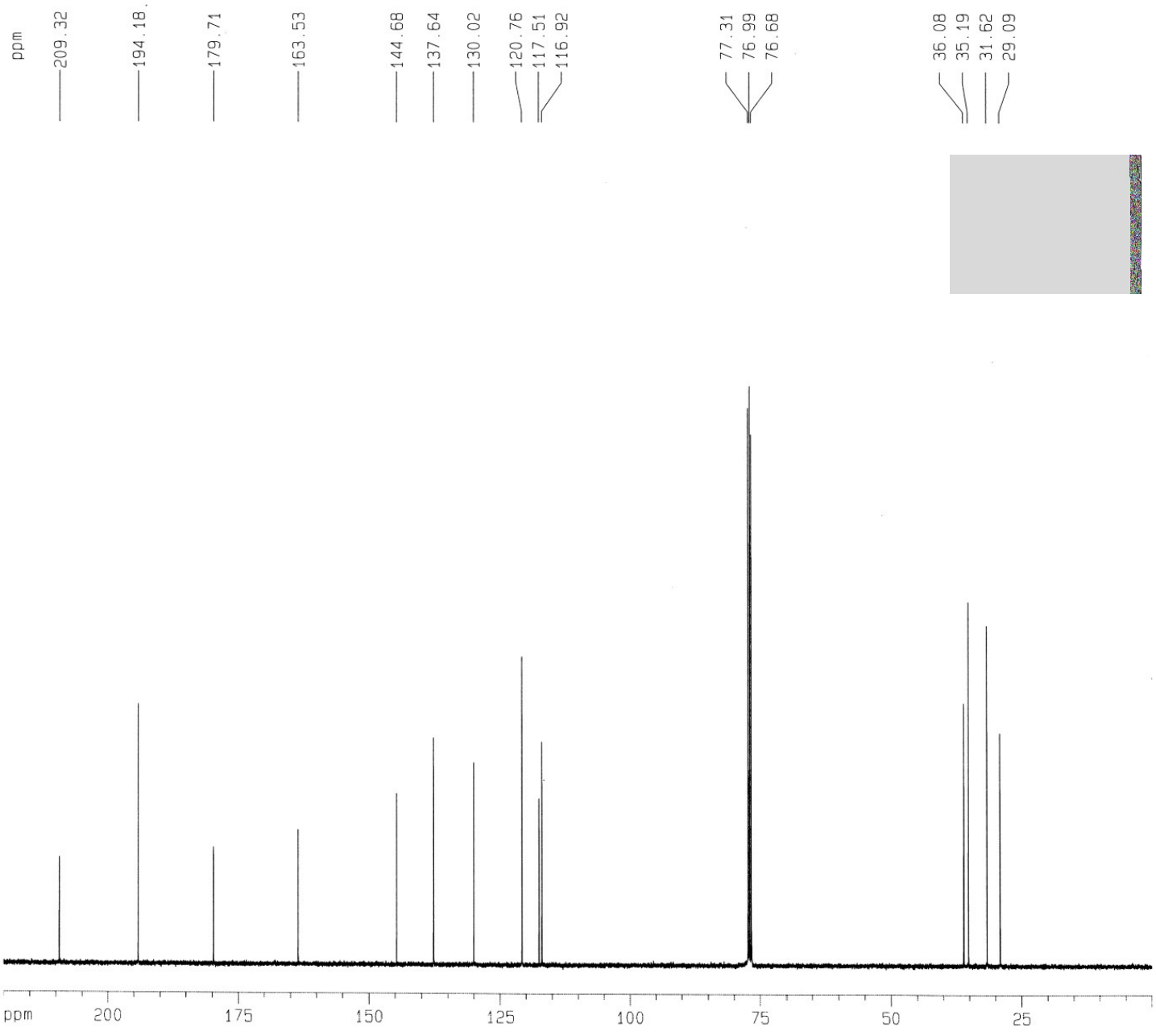
NUC1	1H
P1	14.30 usec
PL1	-0.30 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	8192
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	13.000 ppm
F1	5201.69 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.62791 ppm/cm
HZCM	251.24442 Hz/cm



Current Data Parameters

NAME	LYC-10
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20160706
Time	1.43
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4505
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	256
DW	19.900 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	10.20 usec
PL1	0.00 dB
SFO1	100.6237959 MHz

========== CHANNEL f2 ==========

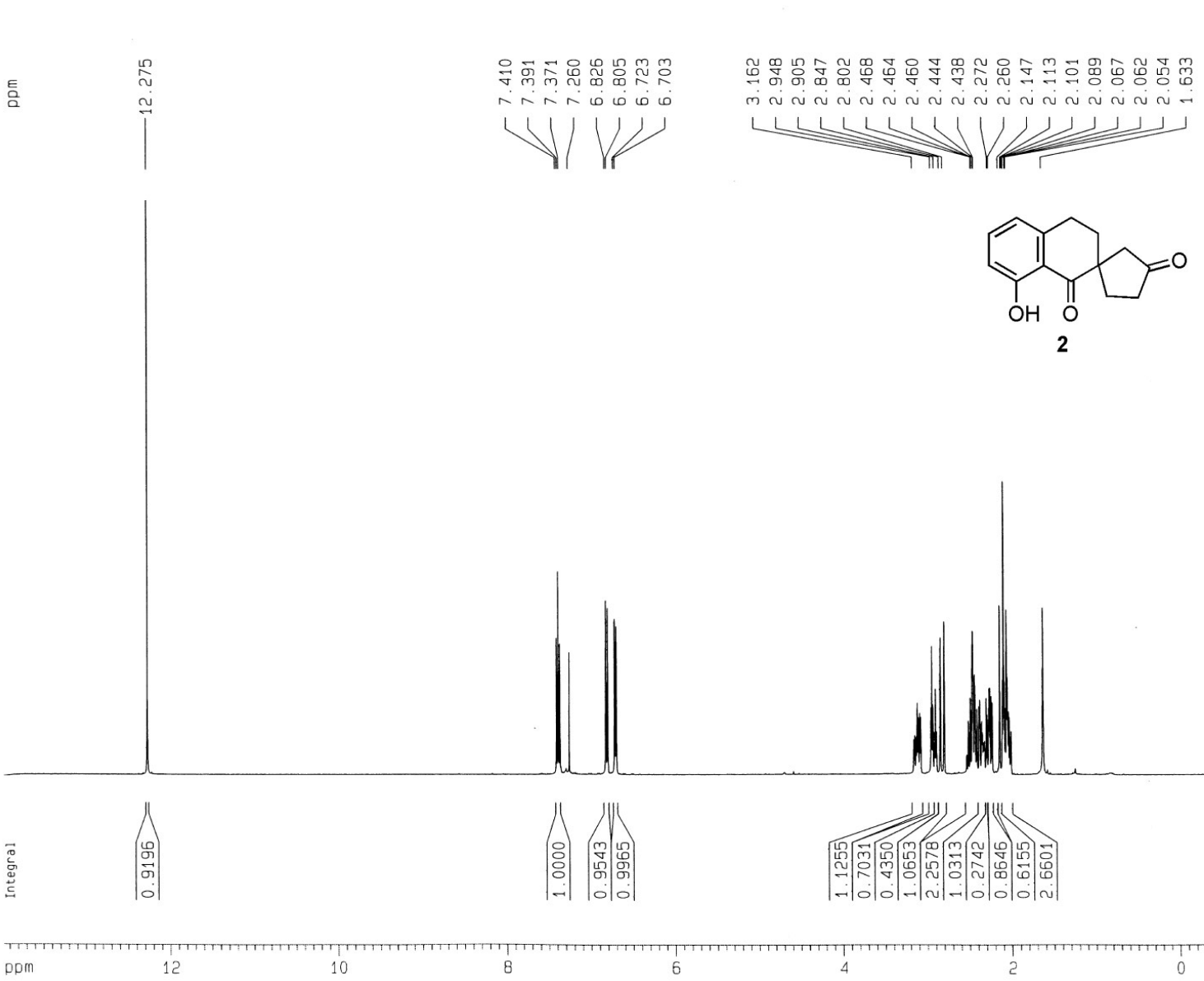
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-3.00 dB
PL12	14.50 dB
PL13	17.50 dB
SFO2	400.1326008 MHz

F2 - Processing parameters

SI	32768
SF	100.6127746 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	11.00000 ppm/cm
HZCM	1106.74048 Hz/cm



Current Data Parameters

NAME	LCY
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160308
Time	23.51
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	3649.1
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

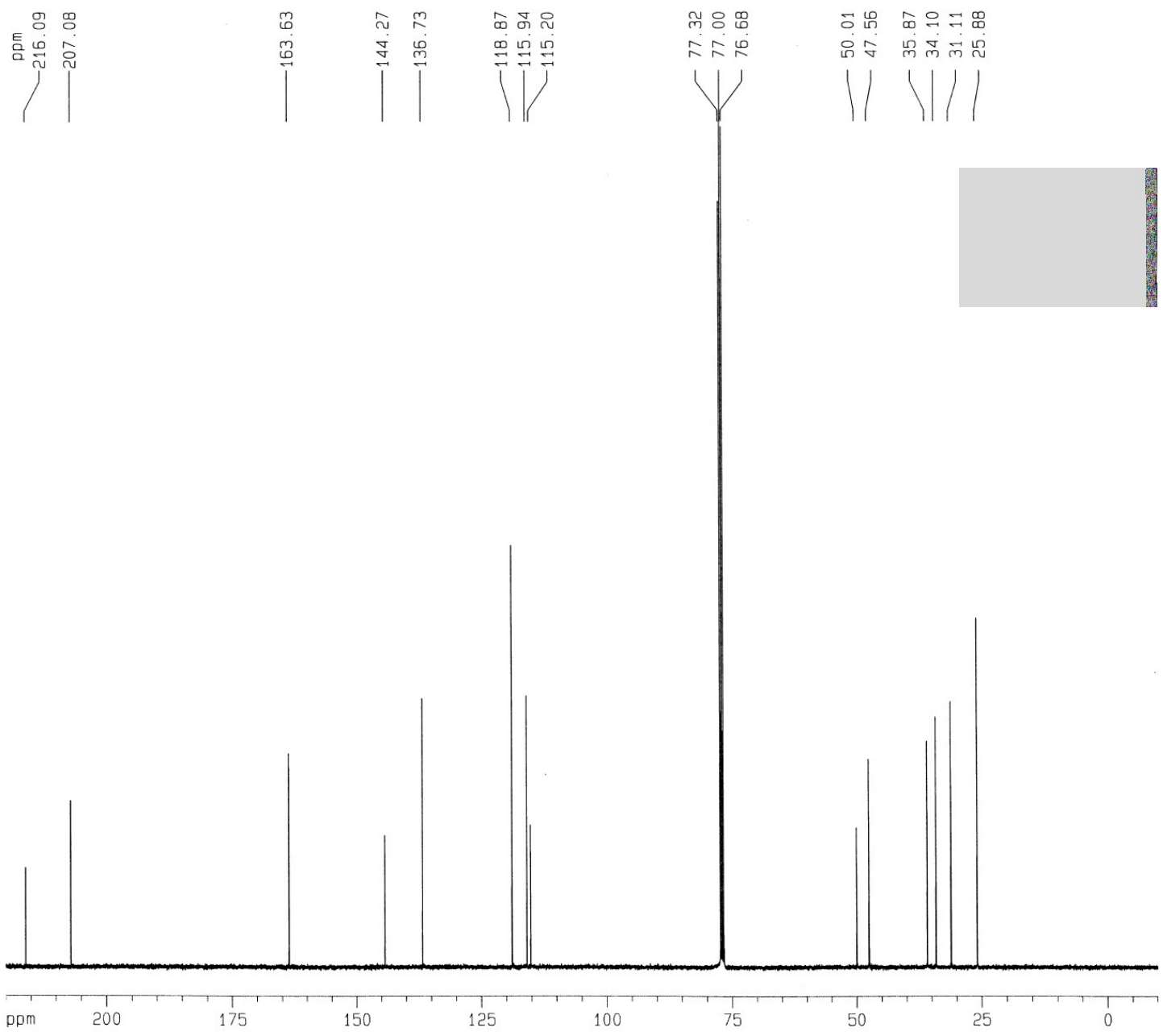
NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	8192
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	14.000 ppm
F1	5601.82 Hz
F2P	-0.500 ppm
F2	-200.06 Hz
PPMCM	0.67442 ppm/cm
HZCM	269.85510 Hz/cm



Current Data Parameters
NAME LCY
EXPNO 2
PROCNO 1

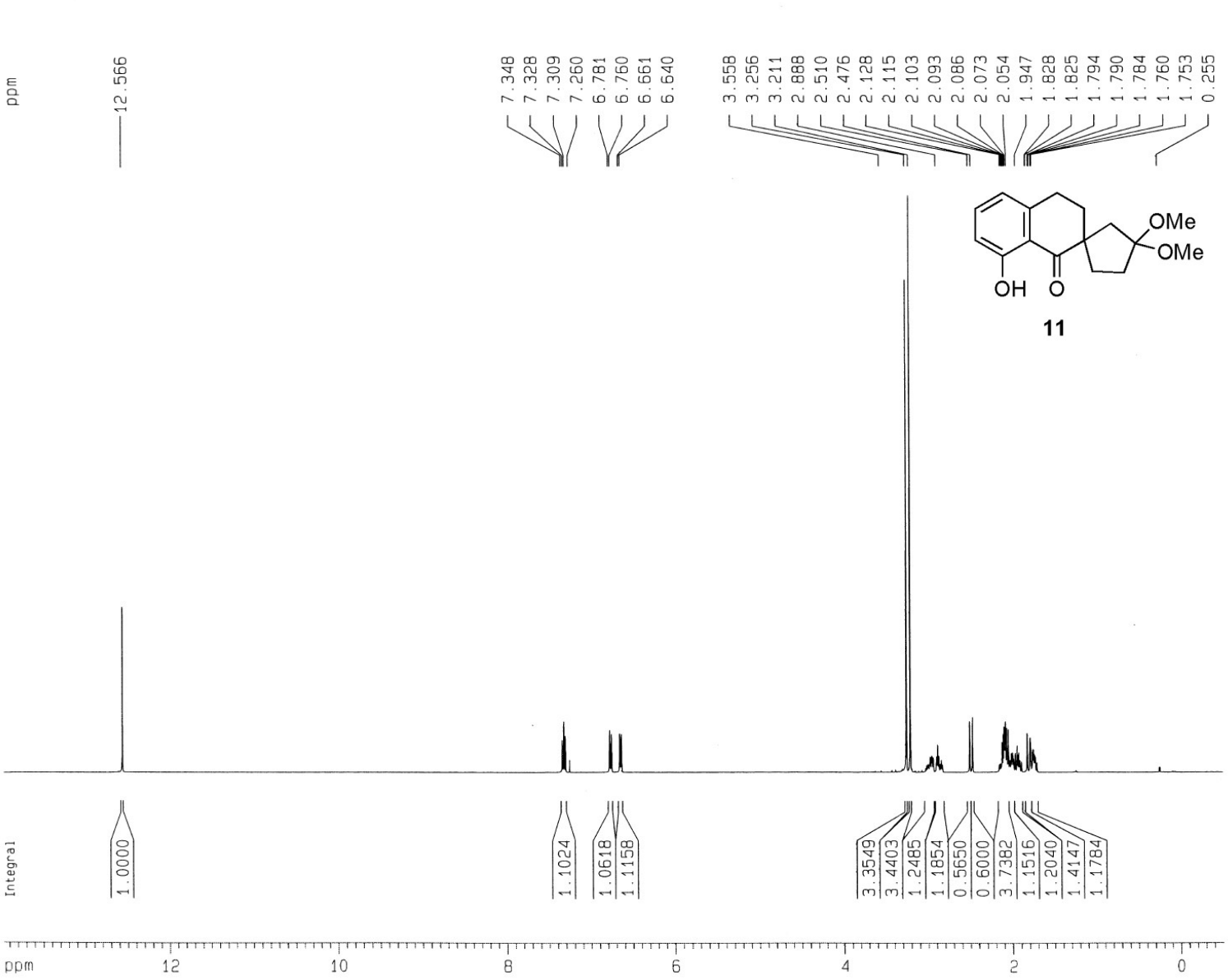
F2 - Acquisition Parameters
Date_ 20160309
Time 6.34
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7168
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 256
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 10.20 usec
PL1 0.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.50 dB
PL13 17.50 dB
SFO2 400.1326008 MHz

F2 - Processing parameters
SI 32768
SF 100.6127723 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm



Current Data Parameters

NAME	WYC-test
EXPNO	70
PROCNO	1

F2 - Acquisition Parameters

Date_	20160301
Time	22.34
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	912.3
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

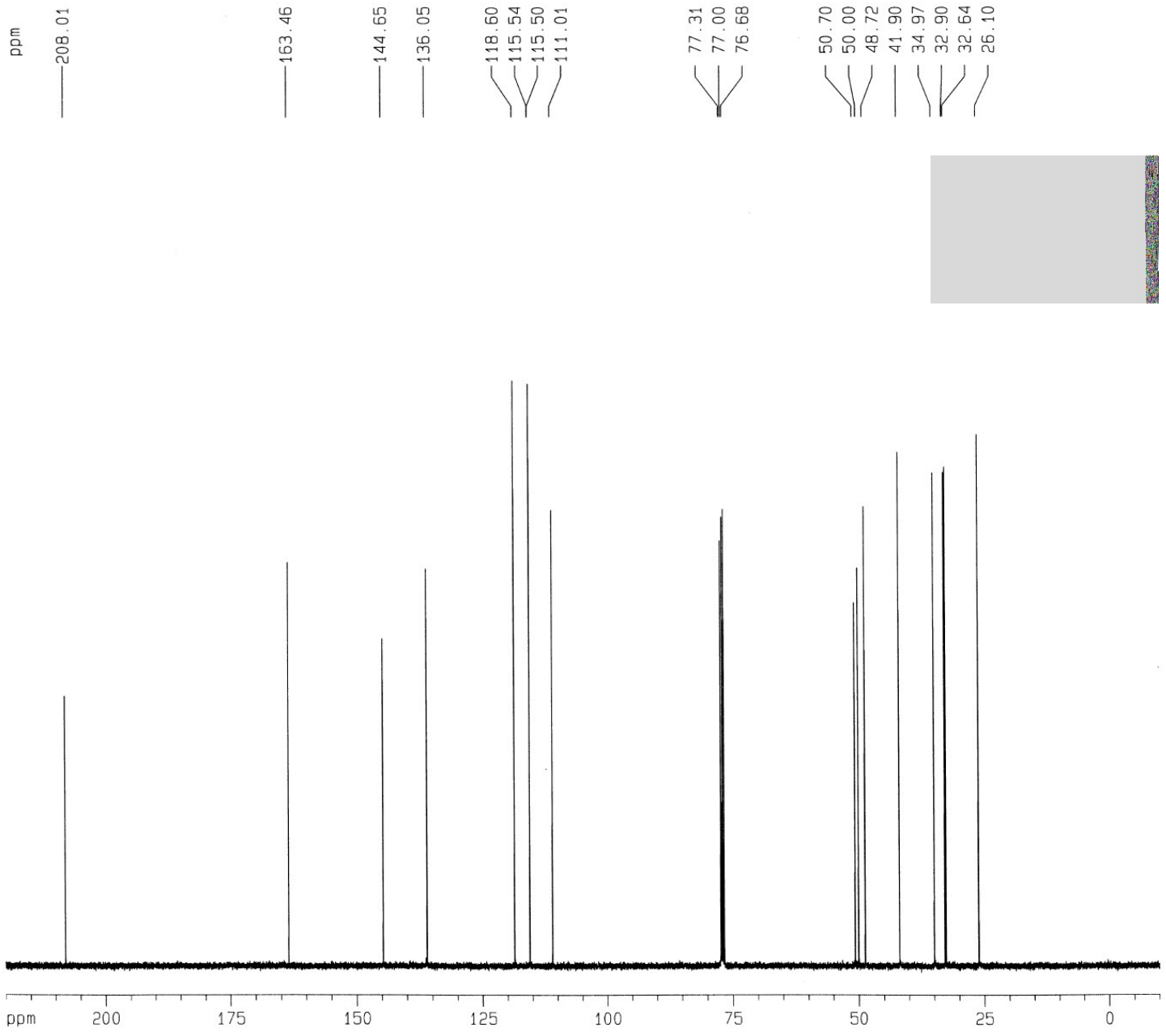
NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	8192
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	14.000 ppm
F1	5601.82 Hz
F2P	-0.500 ppm
F2	-200.06 Hz
PPMCM	0.67442 ppm/cm
HZCM	269.85510 Hz/cm



Current Data Parameters
NAME lcy-10
EXPNO 1
PROCNO 1

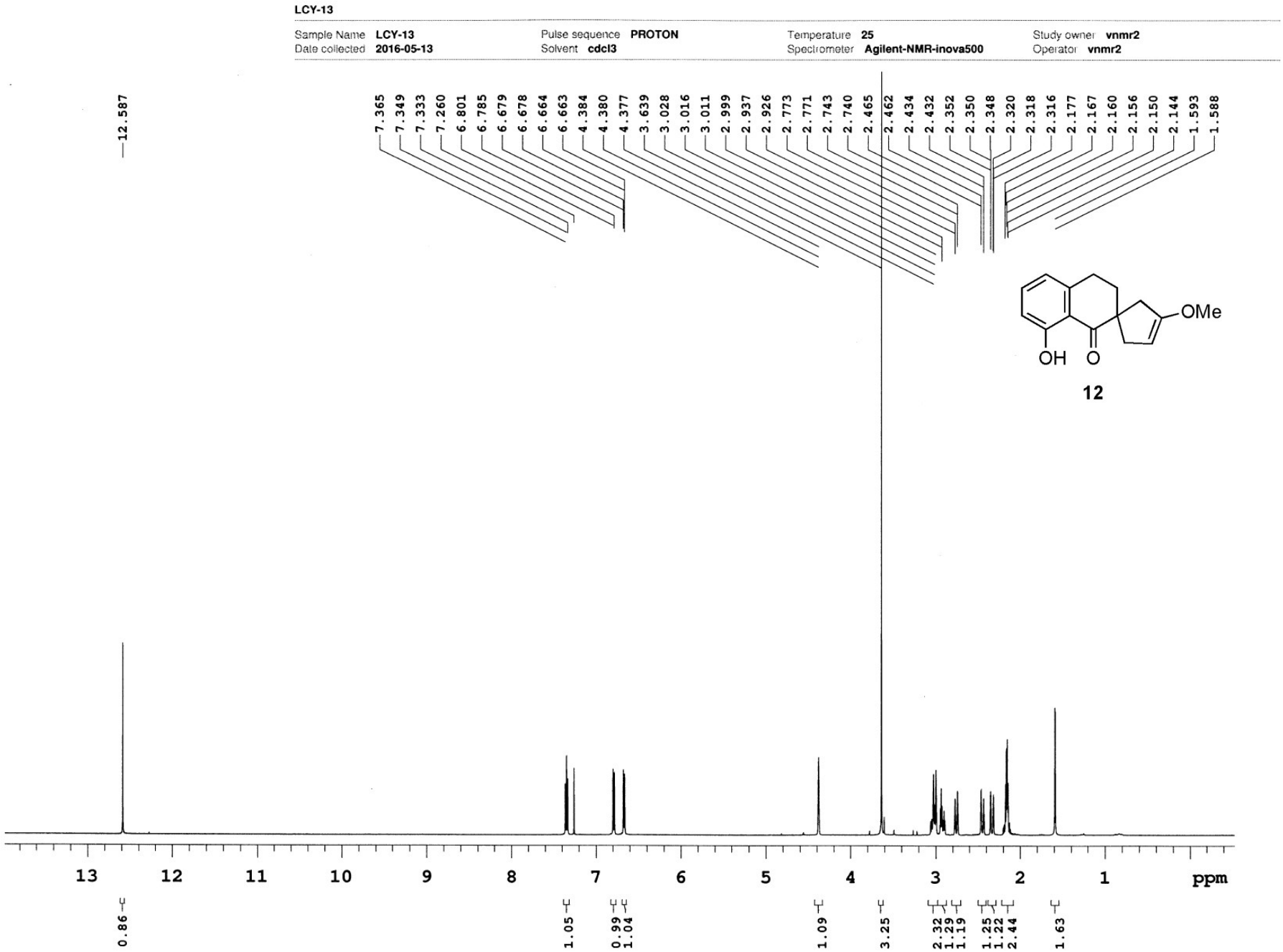
F2 - Acquisition Parameters
Date_ 20160302
Time 11.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 256
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 10.20 usec
PL1 0.00 dB
SFO1 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.50 dB
PL13 17.50 dB
SFO2 400.1326008 MHz

F2 - Processing parameters
SI 32768
SF 100.6127754 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm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Sample Name LCY-13
Date collected 2016-05-13
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Spectrometer Agilent-NMR-inova500
Study owner vnmr2
Operator vnmr2
12.587
7.365
7.349
7.333
7.260
6.801
6.785
6.679
6.678
6.664
6.663
4.384
4.380
4.377
3.639
3.028
3.016
3.011
2.999
2.937
2.926
2.773
2.771
2.743
2.740
2.465
2.462
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2.320
2.318
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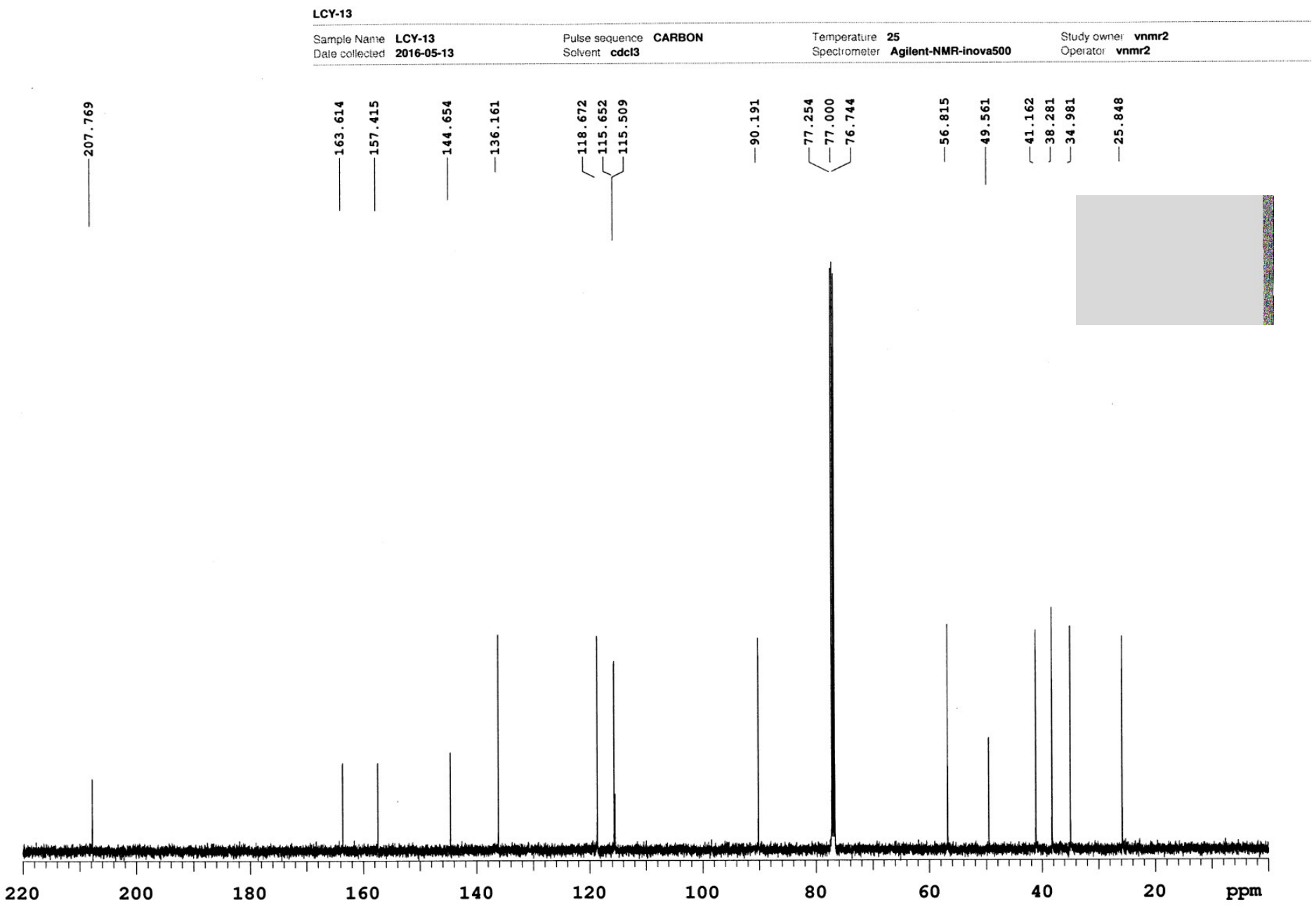
2.177
2.167
2.160
2.156
2.150
2.144
1.593
1.588
OMe
OH
O
12
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.86
1.05
0.99
1.04
1.09
3.25
2.32
1.29
1.19
1.25
1.22
2.44
1.63


LCY-13
Sample Name LCY-13
Date collected 2016-05-13
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Spectrometer Agilent-NMR-inova500
Study owner vnmr2
Operator vnmr2
207.769
163.614
157.415
144.654
136.161
118.672
115.652
115.509
90.191
77.254
77.000
76.744
56.815
49.561
41.162
38.281
34.981
25.848
220
200
180
160
140
120
100
80
60
40
20
ppm

LCY-14
Sample Name LCY-14
Date collected 2016-12-12
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Spectrometer Agilent-NMR-inova500
Study owner vnmr2
Operator vnmr2
1.579
2.168
2.177
2.187
2.196
2.205
2.214
2.224
2.435
2.447
2.457
2.474
2.485
2.491
2.834
2.845
2.856
2.867
2.879
2.890
3.271
3.306
3.447
3.457
3.466
3.478
3.491
3.500
3.510
3.908
5.264
6.701
6.716
6.787
6.804
7.260
7.369
7.385
7.401
12.218
OMe
OH
O
O
1
ppm
1
2
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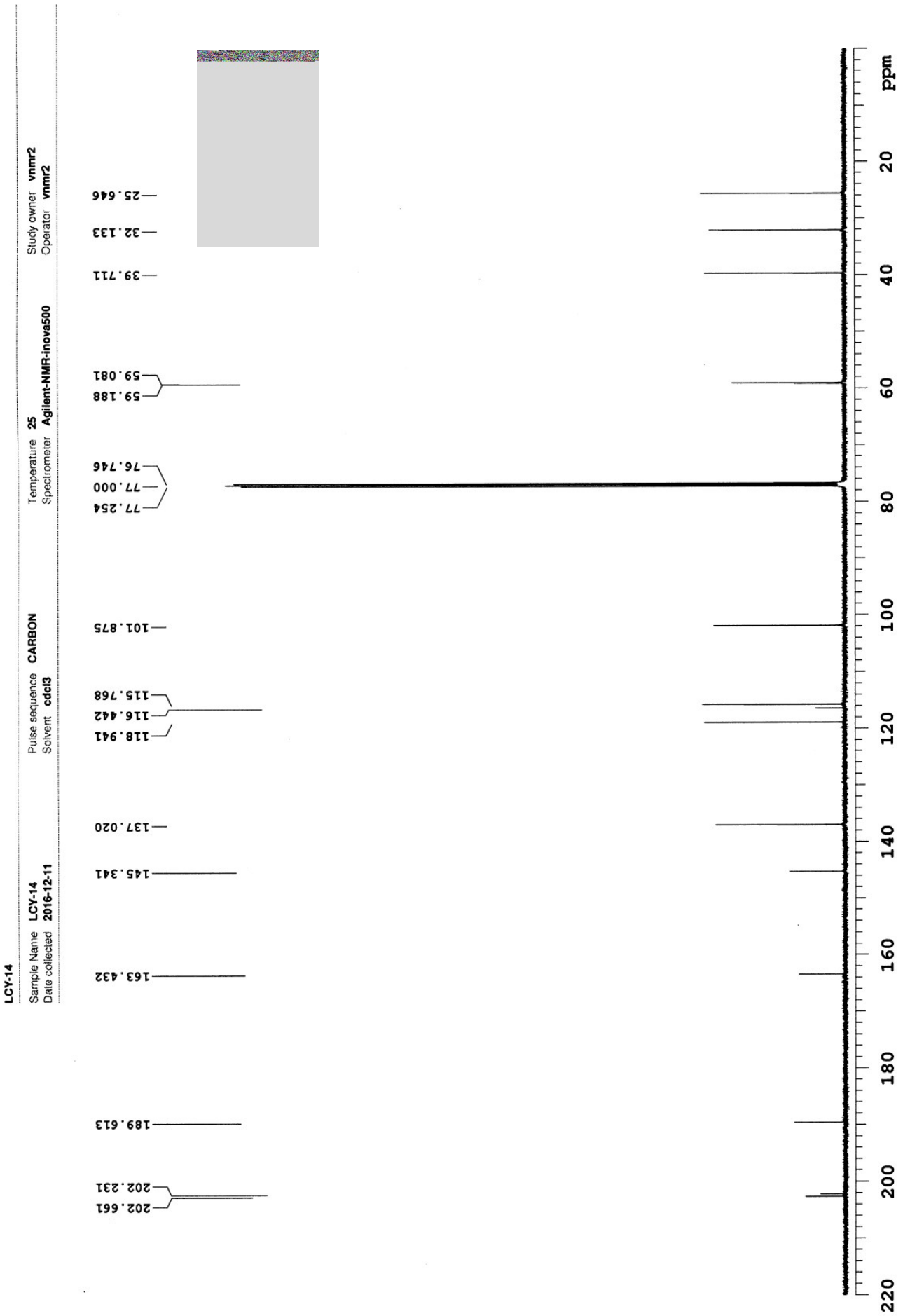
7.81
15.16
7.56
7.32
7.61
22.17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Sample Name LCY-14
Date collected 2016-12-11
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Spectrometer Agilent-NMR-inova500
Study owner vnmr2
Operator vnmr2
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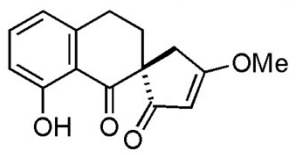
39.711
59.081
59.188
76.746
77.000
77.254
101.875
115.768
116.442
118.941
137.020
145.341
163.432
189.613
202.231
202.661
ppm

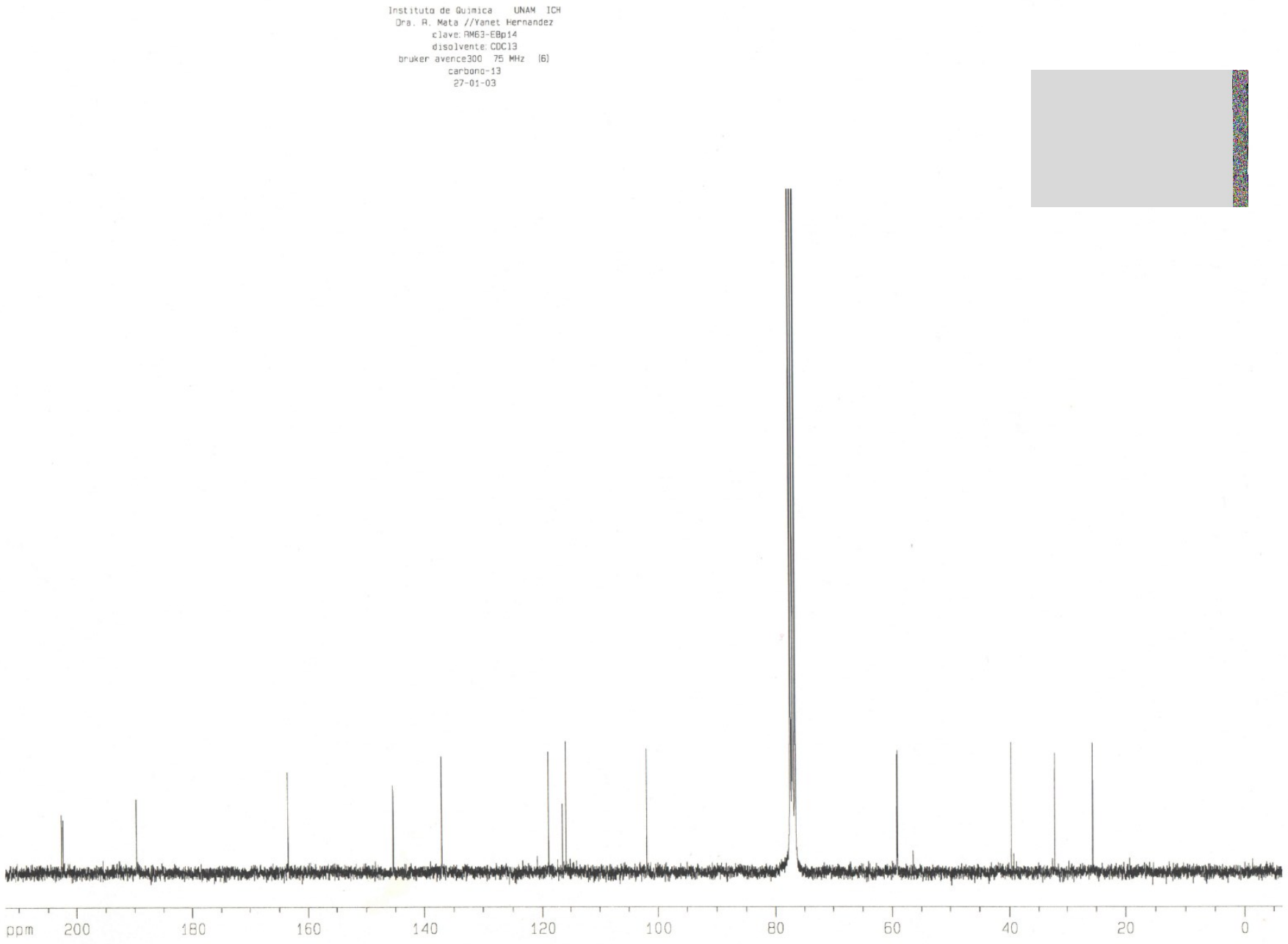
Instituto de Quimica UNAM ICH
Dra. R. Mata
Yanet Hernandez
clave:RM63_EB-P14
unity unity plus500 (7)
disolvente:CDCl3
hidrogeno-1+D2O
28-01-03

Pulse Sequence: s2pul

$C_{15}H_{19}O_4$



natural (–)-nidemone


Instituto de Quimica UNAM ICH
Dra. R. Mata //Yanet Hernandez
clave: RM63-EBp14
disolvente: CDCl3
bruker avence300 75 MHz (6)
carbono-13
27-01-03
ppm
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180
160
140
120
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80
60
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20
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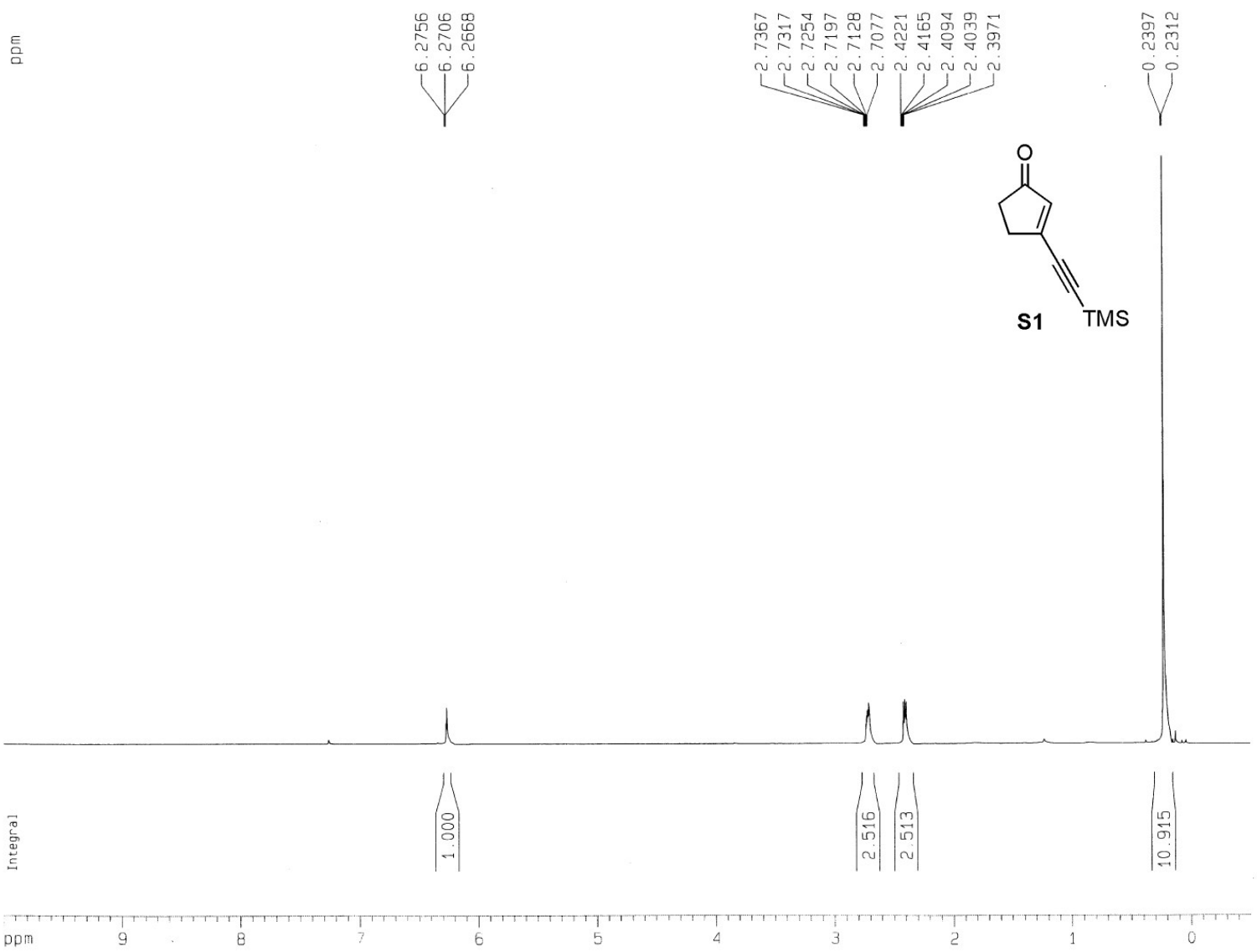


Current Data Parameters

NAME	LSP-test
EXPNO	13
PROCNO	1

F2 - Acquisition Parameters

Date_	20150322
Time	17.29
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	1024
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec

========== CHANNEL f1 ==========

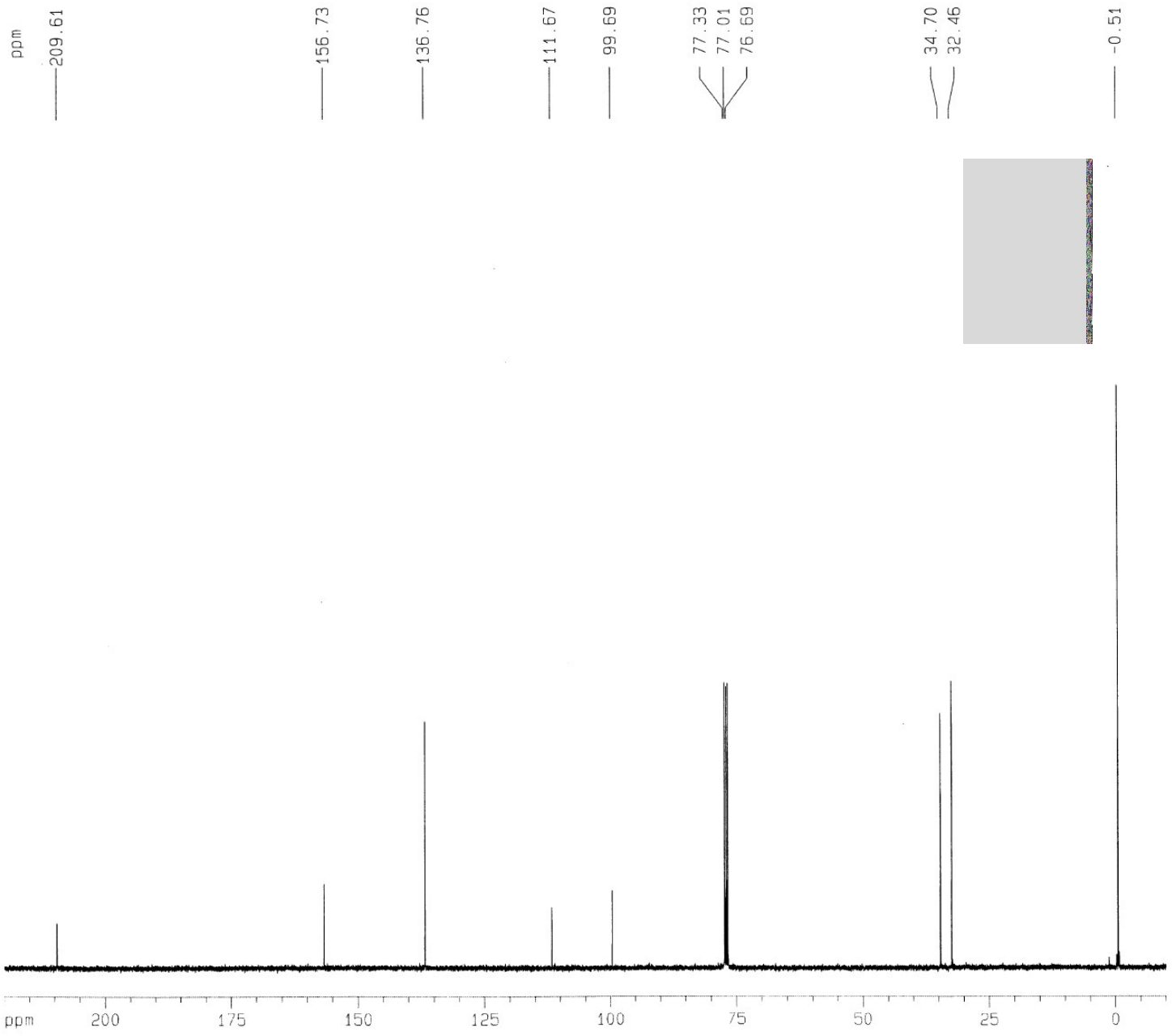
NUC1	1H
P1	11.90 usec
PL1	-3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	8192
SF	400.1300099 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.50 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.06 Hz
PPMCM	0.48837 ppm/cm
HZCM	195.41232 Hz/cm



Current Data Parameters

NAME	LSP-test
EXPNO	14
PROCNO	1

F2 - Acquisition Parameters

Date_	20150322
Time	18.27
INSTRUM	spect
PROBHD	5 mm QNP 1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	256
DW	19.900 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

============ CHANNEL f1 ============

NUC1	13C
P1	10.20 usec
PL1	0.00 dB
SFO1	100.6237959 MHz

============ CHANNEL f2 ============

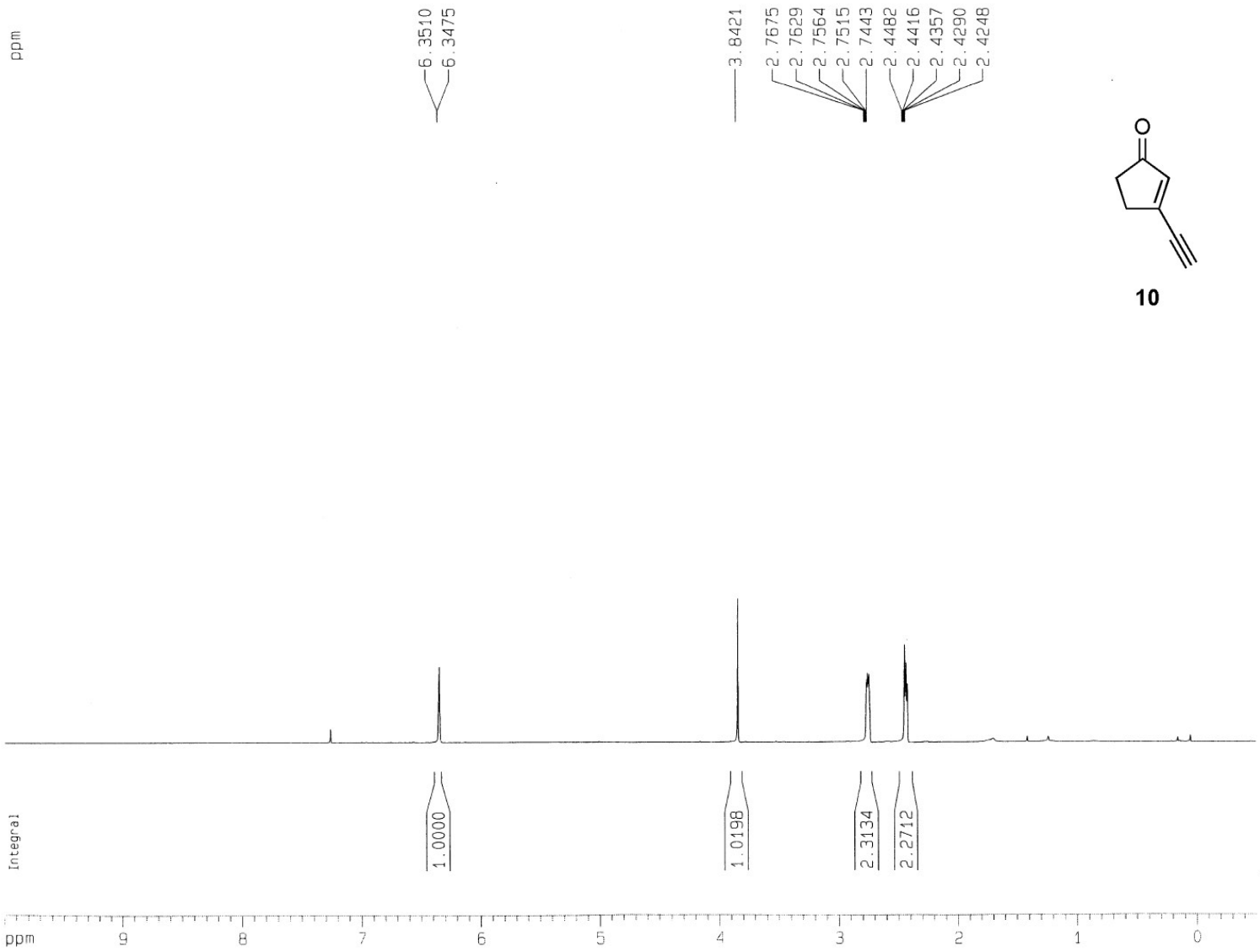
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-3.00 dB
PL12	14.50 dB
PL13	17.50 dB
SFO2	400.1326008 MHz

F2 - Processing parameters

SI	32768
SF	100.6127731 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	11.50000 ppm/cm
HZCM	1157.04688 Hz/cm



10

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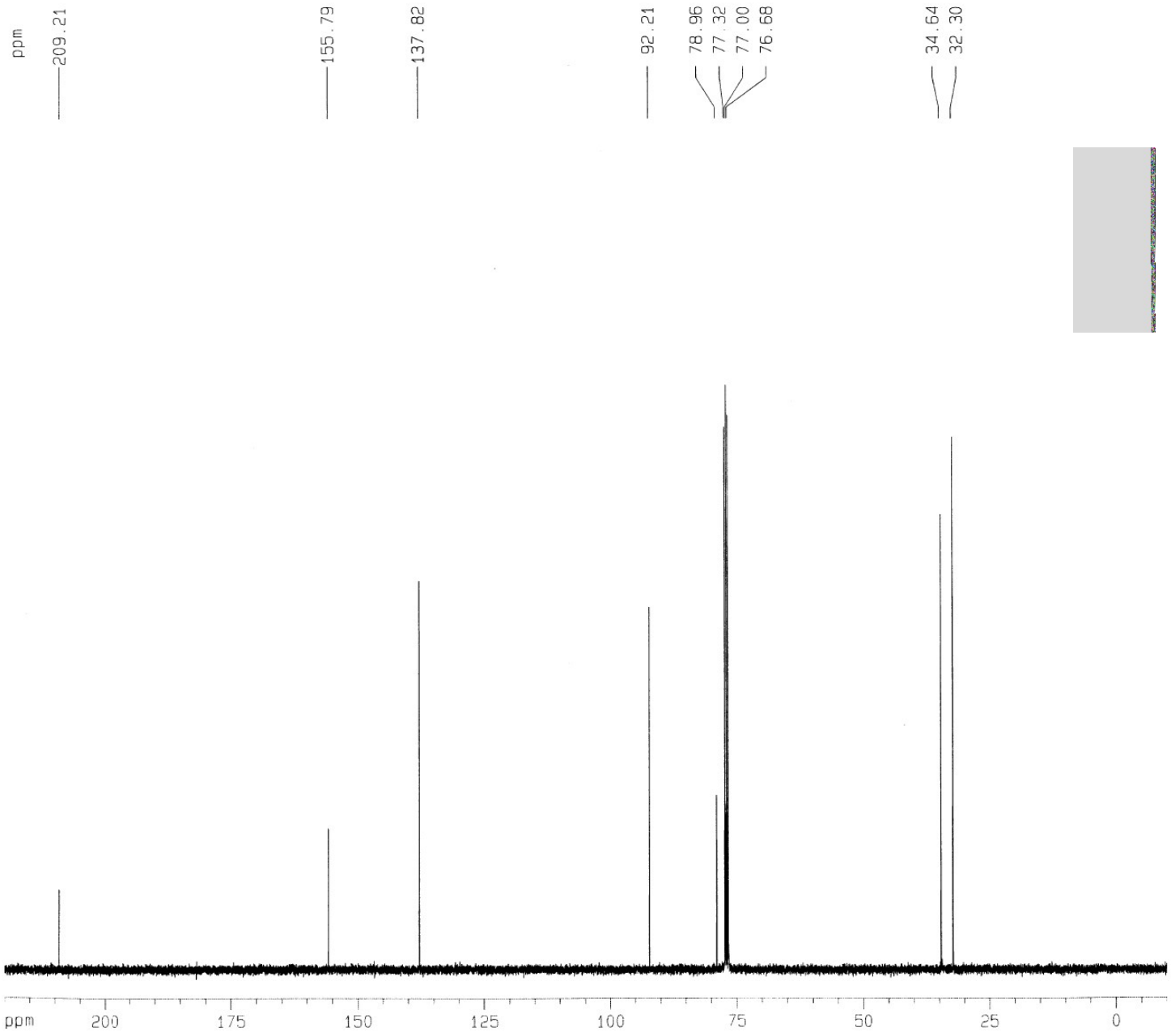
Current Data Parameters
NAME                  LSP-test
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20150322
Time                     16.41
INSTRUM                  spect
PROBHD     5 mm QNP  1H
PULPROG                  zg30
TD                      16384
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                5995.204 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    2896.3
DW                   83.400 usec
DE                    6.50 usec
TE                   300.0 K
D1                 1.50000000 sec

============ CHANNEL f1 =============
NUC1                        1H
P1                       11.90 usec
PL1                      -3.00 dB
SF01              400.1326008 MHz

F2 - Processing parameters
SI                        8192
SF                400.1300099 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       21.50 cm
F1P                     10.000 ppm
F1                     4001.30 Hz
F2P                     -0.500 ppm
F2                     -200.06 Hz
PPMCM                 0.48837 ppm/cm
HZCM                195.41232 Hz/cm
```



Current Data Parameters
NAME LSP-test
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150322
Time 17.24
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 757
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 256
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 10.20 usec
PL1 0.00 dB
SF01 100.6237959 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.50 dB
PL13 17.50 dB
SF02 400.1326008 MHz

F2 - Processing parameters
SI 32768
SF 100.6127738 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm