Supporting Information

Synthesis of crispine-A analogues *via* intramolecular Schmidt reaction

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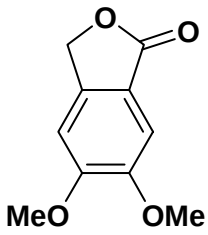
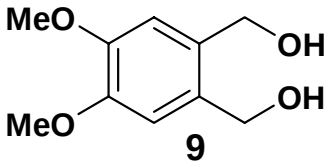


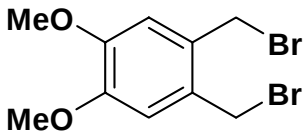
5,6-dimethoxyisobenzofuran-1(3H)-one ^{1}H NMR [400 MHz, $CDCl_3$] δ 7.27 (s, 1H), 6.87 (s, 1H), 5.19 (s, 2H), 3.96 (s, 3H), 3.93 (s, 3H), ^{13}C NMR [100 MHz, $CDCl_3$] δ 171.49, 154.88 150.46, 141.05, 117.63, 103.42, 69.16, 56.39, 56.28; MS (ESI) $C_{10}H_{11}O_4$ $(M+H)^+$ 195.



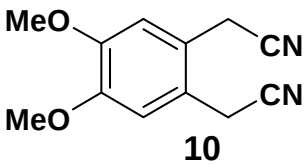
9

1,2-Bis(hydroxymethyl)-4,5-dimethoxybenzene (9): To a stirred solution of LAH (430 mg, 11.34 mmol) in dry THF (20 ml) at 0 °C was slowly added a solution of 5,6-dimethoxyisobenzofuran-1(3H)-one (2 g, 10.306 mmol) and stirred at 0 °C for 15 mins. The reaction mixture was then heated to reflux for 4h, cooled to 0 °C, quenched with moist Na_2SO_4 and filtered. The filtrated was then concentrated under reduced pressure to give the corresponding diol (**9**) (1.92 g, 94% yield). The crude product was taken for further reaction without purification. (IR (Neat): 3460, 3337, 2922, 1607, 1511, 1460, 1103, 754 cm^{-1}; ^{1}H NMR [400 MHz, $CDCl_3$] δ 6.89 (s, 2H), 4.66 (s, 4H), 3.89 (s,

6H), 3.08 (bs, 1H), 1.76 (bs, 1H), ^{13}C NMR [100 MHz, $CDCl_3$] δ 148.50, 132.02, 113.26, 63.84, 56.03; MS (ESI) $C_{10}H_{14}O_4$ $(M+Na)^+$ 221.



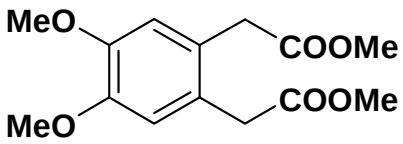
1,2-Bis(bromomethyl)-4,5-dimethoxybenzene: To a suspension of diol (**9**) (2 g, 10.10 mmol) in benzene (20 ml) was slowly added PBr_3 (2.06 ml, 22 mmol) and stirred at 50 °C for 1h then cooled to rt and stirred for 24h. The reaction mixture was then basified with saturated Na_2CO_3 solution (PH = 9). Organic layer was washed with water and dried over anhydrous Na_2SO_4, concentrated under reduced pressure and purified by recrystallization to afford the corresponding **1,2-bis(bromomethyl)-4,5-dimethoxybenzene** (3.01 g, 92% yield). IR (neat): 2955, 1605, 1522, 1460, 1358, 1277, 1204, 1128, 1000, 871, 603 cm^{-1}; ^1H NMR [400 MHz, $CDCl_3$] δ 6.84 (s, 2H), 4.63 (s, 4H), 3.89 (s, 6H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 149.54, 129.07, 113.74, 56.06, 30.54.



10

2,2'-(4,5-dimethoxy-1,2-phenylene)diacetonitrile (10): To a stirred solution of NaCN (3.16 g, 64.51 mmol) in dry DMSO (50 ml) was slowly added a solution of 1,2-bis(bromomethyl)-4,5-dimethoxybenzene (9.5 g, 29.321

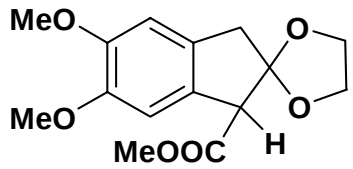
mmol) at 90 °C. The reaction mixture was stirred at 90 °C for additional 3h. Then quenched with water and extracted with chloroform. Organic layer was dried over anhydrous Na_2SO_4, concentrated under reduced pressure and purified by column chromatography over silica gel using gradient elution with 10 – 20 % EtOAc in hexane to yield the corresponding dicyano compound (**10**) (5.71 g, 90% yield). IR (neat): 2959, 2941, 2837, 2250, 1609, 1525, 1417, 1272, 1180, 1089, 873, 756 cm^{-1}; 1H NMR [400 MHz, $CDCl_3$] δ 6.90 (s, 2H), 3.90 (s, 6H), 3.70 (s, 4H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 149.32, 120.25, 116.99, 112.82, 56.17, 21.07; MS (ESI) $C_{12}H_{12}N_2O_2$ $(M+Na)^+$ 239.



11

Dimethyl 2,2'-(4,5-dimethoxy-1,2-phenylene)diacetate (11a): To a solution of dicyano compound (**10**) (2.12 g, 8.62 mmol) in methanol (15 ml), thionyl chloride was slowly added (4.13 ml, 56.96 mmol) and heated to reflux for 2 days. Reaction mixture was diluted with EtOAc and washed with water. Organic layer was dried over anhydrous Na_2SO_4, concentrated and purified by column chromatography over silica gel using gradient elution with 10 – 20 % EtOAc in hexane to yield the corresponding diester (**11**) (2.86 g, 94% yield). IR (neat): 2952, 2851, 1729, 1520, 1273, 1192, 1009, 754 cm^{-1}; ^1H NMR

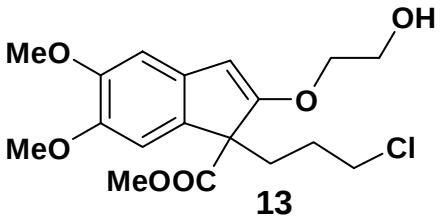
[400 MHz, $CDCl_3$] δ 6.76 (s, 2H), 3.85 (s, 6H), 3.67 (s, 6H), 3.62 (s, 4H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 171.75, 148.33, 125.28, 114.10, 55.97, 51.88, 38.27, 29.60; MS (ESI) $C_{14}H_{18}O_6$ $(M+Na)^+$ 305.



12

Methyl 5',6'-dimethoxy-1',3'-dihydrospiro[[1,3]dioxolane-2,2'-indene]-1'-carboxylate (12): To a stirred solution of β-ketoester (**7)** (100 mg, 0.378 mmol), triethyl orthoformate (251 µl, 1.51 mmol) and ethylene glycol (212 µl, 3.77 mmol) in dry DCM (10 ml) added catalytic amount of PTSA (7 mg, 0.03 mmol) and the reaction mixture was stirred for about 3 days. Reaction mixture was then diluted with DCM, washed with saturated $NaHCO_3$ followed by brine and extracted with DCM. Organic layer was dried over anhydrous Na_2SO_4, conc. and purified by column chromatography over silica gel using gradient elution with 10 – 20 % EtOAc in hexane to yield the corresponding ethylene ketal (**12**) (53%).IR (neat): 2957, 2836, 1728, 1508, 1466, 1257, 1180, 1031, 857, 572 cm^{-1}; ^1H NMR [400 MHz, $CDCl_3$] δ 6.77 (s, 1H), 6.74 (s, 1H), 4.01-4.11 (m, 4H), 3.86 (s, 3H), 3.84 (s, 3H), 3.73 (s, 3H), 3.73 (s, 3H), 3.43 (d, *J* = 16 Hz, 1H), 3.06 (d, *J* = 16 Hz, 1H); ^{13}C NMR [100 MHz,

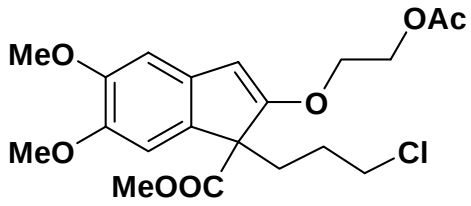
$CDCl_3$] δ 171.02, 149.44, 148.54, 132.34, 129.44, 117.911, 108.39, 107.79, 65.45, 64.55, 59.39, 56.05, 55.98, 52.06, 42.99; MS (ESI) $C_{15}H_{18}O_6$ $(M+Na)^+$ 317.



13

Methyl 1-(3-chloropropyl)-2-(2-hydroxyethoxy)-5,6-dimethoxy-1H-indene-1-carboxylate (13): To a suspension of NaH (14 mg, 0.35 mmol) in dry DMF (3 ml) at 0 °C was slowly added a solution of ethylene ketal (**12**) (100 mg, 0.325 mmol) in dry DMF (3 ml) and stirred at 0 °C for 5 min. Then into this added 1-chloro-3-iodopropane (52 µl, 0.487 mmol) and stirred at 0 °C for 30 min. The reaction mixture was then quenched, washed with water and extracted with EtOAc. Organic layer was dried over anhydrous Na_2SO_4, concentrated under reduced pressure and purified by column chromatography over silica gel using gradient elution with 10 – 20 % EtOAc in hexane to yield the alkylated product (**13**) (87.3 mg, 70% yield). IR (neat): 3513, 2951, 2835, 1727, 1607, 1491, 1454, 1312, 1213, 1075, 1032, 761 cm^{-1}; ^1H NMR [400 MHz, $CDCl_3$] δ 6.86 (s, 1H), 6.72 (s, 1H), 5.66 (s, 1H), 4.15-4.10 (m, 4H), 3.87 (s, 3H), 3.86 (s, 3H), 3.67 (s, 3H), 3.39 (m, 2H), 2.32 (t, J = 8 Hz, 1H),

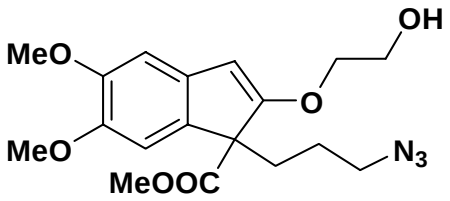
1.46-1.31 (m, 2H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 172.69, 164.39, 149,48, 146.11, 136.22, 129.91, 107.20, 104.09, 101.23, 71.58, 60.7, 60.41, 56.60,



14

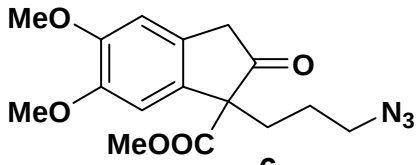
Methyl 1-(3-chloropropyl)-2-(2-hydroxyethoxy)-5,6-dimethoxy-1H-indene-1-carboxylate (14): To a stirred solution of alkylated compound (13) (9 mg, 0.024 mmol) in dry DCM (1 ml), acetic anhydride (3.47 µl, 0.0364 mmol) and triethyl amine (3.23 µl, 0.0243 mmol) was added followed by catalytic amount DMAP and stirred at rt for 2h. The reaction mixture was then diluted with DCM and washed with water. Organic layer was dried over anhydrous Na_2SO_4, conc. under reduced pressuren and purified by column chromatography over silica gel using gradient elution with 10-15 % EtOAc in hexane to yield the acetylated compound (**14**) (7 mg, 70% yield). IR (neat): 2952, 1731, 1608, 1491, 1455, 1316, 1232, 1058, 761 cm^{-1}; ^{1}H NMR [400 MHz, $CDCl_3$] δ 6.83 (s, 1H), 6.73 (s, 1H), 5.64 (s, 1H), 4.40 (t, J = 4 HZ, 4.17 (m, 2H), 3.87 (s, 3H), 3.86 (s, 3H), 3.63 (s, 3H), 3.39 (m, 2H), 2.27 (m, 2H), 2.09 (s, 3H), 1.29.1.25 (m, 2H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 172.45,

170.85, 164.88, 149.54, 146.28, 136.19, 130.45, 107.13, 104.28, 100.97, 68.09, 62.17, 61.12, 56.63, 56.13, 52.60, 44.99, 30.58, 26.62, 20.79; MS (ESI) $C_{20}H_{25}ClO_7$ $(M+H)^+$ 413.



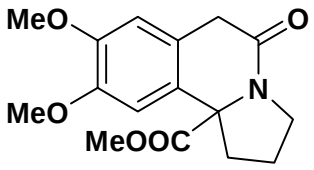
15

Methyl 1-(3-azidopropyl)-2-(2-hydroxyethoxy)-5,6-dimethoxy-1H-indene-1-carboxylate (15): To a solution of alkylated compound (**13**) (70 mg, 0.0189 mmol) in DMF (2 ml) was added NaN_3 (13.5 mg, 0.020 mmol) and stirred at 60 °C for 24h. Reaction mixture was then diluted with EtOAc, washed with water. Organic layer was dried, conc. and purified by column chromatography over silica gel using gradient elution with 10 – 20 % EtOAc in hexane to yield the corresponding azido compound (**15**) (59 mg, 83% yield). IR (neat): 3498, 2935, 2094, 1727, 1607, 1491, 1455, 1309, 1214, 1966, 762 cm^{-1}; ^1H NMR [400 MHz, $CDCl_3$] δ 6.85 (s, 1H), 6.73 (s, 1H), 5.67 (s, 1H), 4.1-3.9 (m, 4H), 3.87 (s, 3H), 3.86 (s, 3H), 3.67 (s, 3H), 3.19 (m, 2H), 2.25 (m, 2H), 1.29-1.25 (m, 2H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 172.55, 164.51, 149.73, 146.34, 136.36, 130.11, 107.62, 104.45, 100.26, 71.63, 62.35, 60.69, 56.71, 56.12, 52.63, 51.26, 30.35, 22.76; MS (ESI) $C_{18}H_{23}N_3O_6$ $(M+Na)^+$ 400.



6

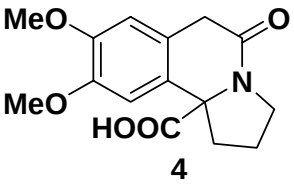
Methyl 1-(3-azidopropyl)-5,6-dimethoxy-2-oxo-2,3-dihydro-1H-indene-1-carboxylate (6): To a stirred solution of azide (**15**) (488 mg, 1.294 mmol) in methanol (10 ml) was added Dowex activated using 1N HCl and heated to reflux for 20h. The reaction mixture was then filtered, concentrated under reduced pressure and the residue was purified by column chromatography over silica gel using gradient elution with 10 – 20 % EtOAc in hexane to yield the corresponding keto azide (**6**) (349 mg, 81% yield). IR (neat): 2913, 2095, 1613, 1494, 1454, 1286, 1164, 1032, 763 cm^{-1}; ^{1}H NMR [400 MHz, $CDCl_3$] δ 6.87 (s, 1H), 6.76 (s, 1H), 3.90 (s, 3H), 3.89 (s, 3H), 3.76 (d, *J* = 22.4 Hz, 1H), 3.65 (s, 3H), 3.44 (d, *J* = 22.4 Hz), 3.16 (m, 2H), 2.25 (m, 2H), 1.29 (m, 2H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 212.20, 170.87, 149,95, 145.59, 131.60, 129.15, 107.77, 106.47, 64.60, 56.19, 56.05, 52.84, 51.15, 43.42, 31.03, 23.70.



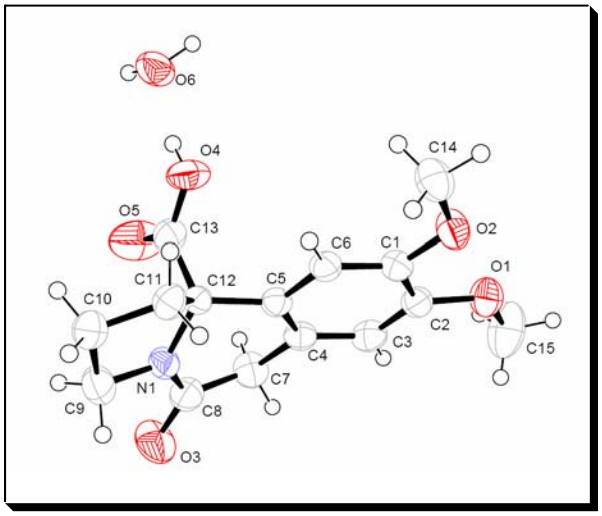
3

Methyl 8,9-dimethoxy-5-oxo-1,2,3,5,6,10b-hexahydropyrrolo[2,1-a]isoquinoline-10b-carboxylate (3): In a clean flame dried RB flask, ketoazide (**15**) (364 mg, 1.04 mmol) was taken and dried azeotropically with dry benzene. Into this added DCM (5 ml) and cooled to -5 °C. Triflic acid (139 µl, 1.57 mmol) was slowly added and stirred at -5 °C for 15min. Reaction mixture was then diluted with DCM and washed with water. Organic layer was dried over anhydrous Na_2SO_4 concentrated under reduced pressure and purified by column chromatography over silica gel using gradient elution with 30 – 40 % EtOAc in hexane to yield the corresponding cyclized product (**3**) (180.69 mg, 54% yield). IR (neat): 2953, 1731, 1650, 1518, 1433, 1411, 1254, 1217, 1133, 805, 628 cm^{-1}; ^1H NMR [400 MHz, $CDCl_3$] δ 6.77 (s, 1H), 6.55 (s, 1H), 3.83 (s, 3H), 3.80 (s, 3H), 3.70 (d, *J* = 19.2 Hz, 1H), 3.59 (s, 3H), 3.59 (m, 1H) 3.41 (d, *J* = 18.8 Hz), 3.05 (m, 1H), 2.01 (m, 2H), 1.85 (m, 2H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 172.15, 168.19, 149.41, 148.13, 126.05, 125.10, 110.0, 108.66 71.60, 56.23, 56.02, 53.14, 44.77, 37.63, 36.23, 21.50; HRMS (ESI) calcd for $C_{16}H_{19}NO_5$ $(M+H)^+$: 306.134; found: 306.1350.

Single Crystal X-Ray Analysis of Hydroxy analogue of Crispine A (4):



4



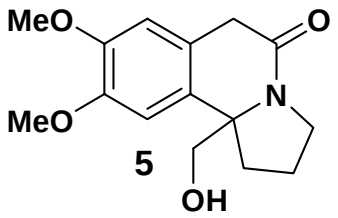
To a solution of ester (**3**) (40 mg, 0.125 mmol) in dioxane **:** water (3:1) (3 ml), KOH was added and stirred at rt for 5h. Then diluted with water and neutralized (PH = 6) with 1N HCl and extracted with EtOAc. Organic layers were dried over anhydrous Na_2SO_4, concentrated under reduced pressure and purifired by recrystallisation from EtOAc:Hexane (1:5) (5 mg/mL) afforded suitable size and quality crystals for x-ray diffraction in the form of pale yellow needles. CCDC Number: CCDC 663178

Table 1. Crystal data and structure refinement for ajoy.

Identification code	ajoy
Empirical formula	C15 H19 N O6
Formula weight	309.31
Temperature	298(2) K
Wavelength	0.71073 A

Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 10.3243(9) A alpha = 90 deg.
	b = 10.6834(10) A beta = 90 deg.
	c = 13.6598(12) A gamma = 90 deg.
Volume	1506.7(2) A^3
Z, Calculated density	4, 1.364 Mg/m^3
Absorption coefficient	0.106 mm^-1
F(000)	656
Crystal size	0.25 x 0.22 x 0.22 mm
Theta range for data collection	2.42 to 28.46 deg.
Limiting indices	-13<=h<=9, -12<=k<=14, -17<=l<=14
Reflections collected / unique	19002 / 3594 [R(int) = 0.0738]
Completeness to theta = 25.00	100.0 %
Absorption correction	None
Max. and min. transmission	0.9771 and 0.9740
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3594 / 3 / 214
Goodness-of-fit on F^2	0.740
Final R indices [I>2sigma(I)]	R1 = 0.0481, wR2 = 0.1125
R indices (all data)	R1 = 0.1352, wR2 = 0.1597

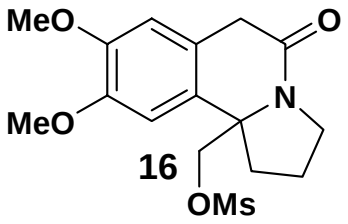
Absolute structure parameter	-0.3(19)
Extinction coefficient	0.009(3)
Largest diff. peak and hole	0.151 and -0.167 e.A^-3



5

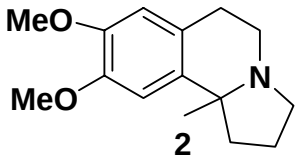
10b-(hydroxymethyl)-8,9-dimethoxy-1,2,3,10b-tetrahydropyrrolo[2,1-a]isoquinolin-5(6H)-one (5): To a suspension of LAH (7.5 mg, 0.19 mmol) in dry THF (2 ml) at 0 °C, a solution of ester (**3**) (55 mg, 0.18 mmol) in dry THF (2 ml) was slowly added and stirred at 0 °C for 8h. Reaction mixture was then quenched with moist Na_2SO_4. The solid was filtered, washed with EtOAc. Filtrate was concentrated under reduced pressure and purified by column chromatography over silica gel using gradient elution with 30–50 % EtOAc in hexane to yield the corresponding alcohol (5) (4.8 mg, 70 % yield). IR (neat): 3385, 2940, 1618, 1514, 1452, 1298, 1214, 1065, 765 cm^{-1}; ^{1}H NMR [400 MHz, $CDCl_3$] δ 6.67 (s, 1H), 6.64 (s, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.78 (d, *J* = 19.2 Hz, 1H), 3.62 (m, 4H), 3.43 (d, *J* = 19.2 Hz), 2.60 (bs, 1H), 2.55 (m, 1H), 2.10 (m, 4H); ^{13}C NMR [100 MHz, $CDCl_3$] δ 168.52, 148.74, 147.94, 129.45, 124.90, 110.26, 108.46, 69.33, 67.95, 60.41, 56.25,

56.04, 44.97, 37.51, 33.88, 29.65, 21.20, 21.05, 14.19; MS (ESI) $C_{15}H_{19}NO_4$ $(M+H)^+$ 278.



(8,9-dimethoxy-5-oxo-1,2,3,5,6,10b-hexahydropyrrolo[2,1-a]isoquinolin-10b-yl)methyl methanesulfonate (16): To a solution of alcohol (**5**) (5 mg, 0.0180 mmol) in dry DCM at 0 °C was added triethyl amine (5.5 µl, 0.03969 mmol) and stirred for 10 mins. Into this slowly added methane sulfonylchloride (1.54 µl, 0.0198 mmol) and stirred at 0 °C for 20 min. The temperature was then slowly raised to rt and stirred for 6h. Reaction mixture was then diluted with DCM and washed with water. Organic layer was dried over anhydrous Na_2SO_4, conc. and purified by column chromatography over silica gel using gradient elution with 30–50 % EtOAc in hexane to yield the corresponding mesylate (**16**) (6 mg, 93 % yield). IR (neat): 2940, 1618, 1514, 1452, 1366, 1298, 1214, 1065, 765 cm^{-1}; ^1H NMR [400 MHz, $CDCl_3$] δ 6.68 (s, 1H), 6.65 (s, 1H), 4.15 (dd, *J* = 23.2, 10 Hz, 2H), 3.89 (s, 3H), 3.88 (s, 3H), 3.77 (d, *J* = 19.6 Hz, 1H), 3.69 (m, 1H), 3.49 (d, *J* = 19.6 Hz, 1H), 2.82 (s, 3H), 2.63 (m, 1H), 2.24 (m, 2H), 1.41 (m, 2H); ^{13}C NMR [100 MHz, $CDCl_3$]

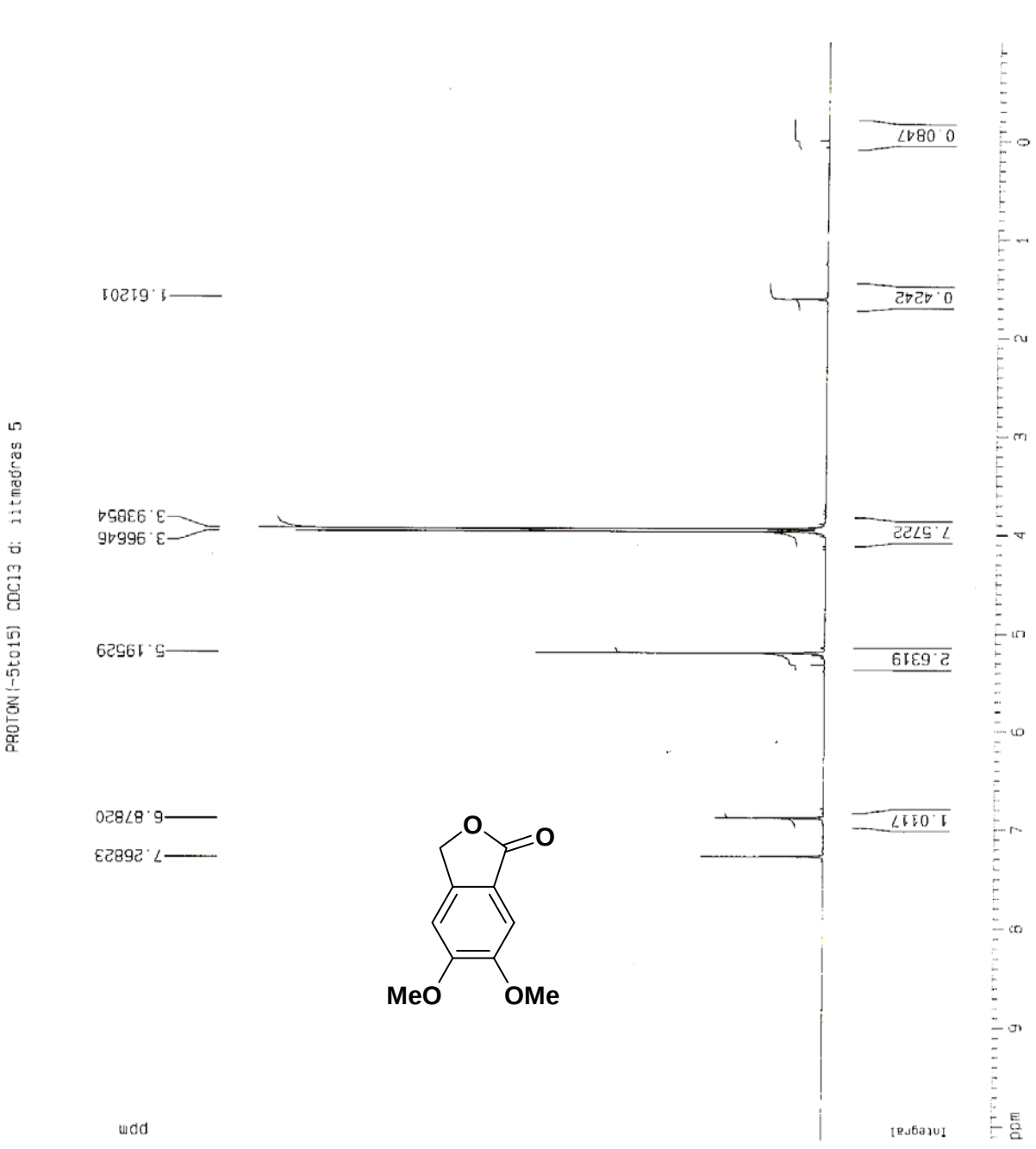
δ 168.12, 149.49, 148.33, 128.36, 127.85, 124.98, 110.49, 108.93, 72.43, 67.033, 56.43, 56.11, 46.27, 45.21, 37.55, 34.63, 31.56, 21.12; HRMS (ESI) calcd for $C_{16}H_{21}NO_6S$ $(M+Na)^+$: 378.0987; found: 378.0988.

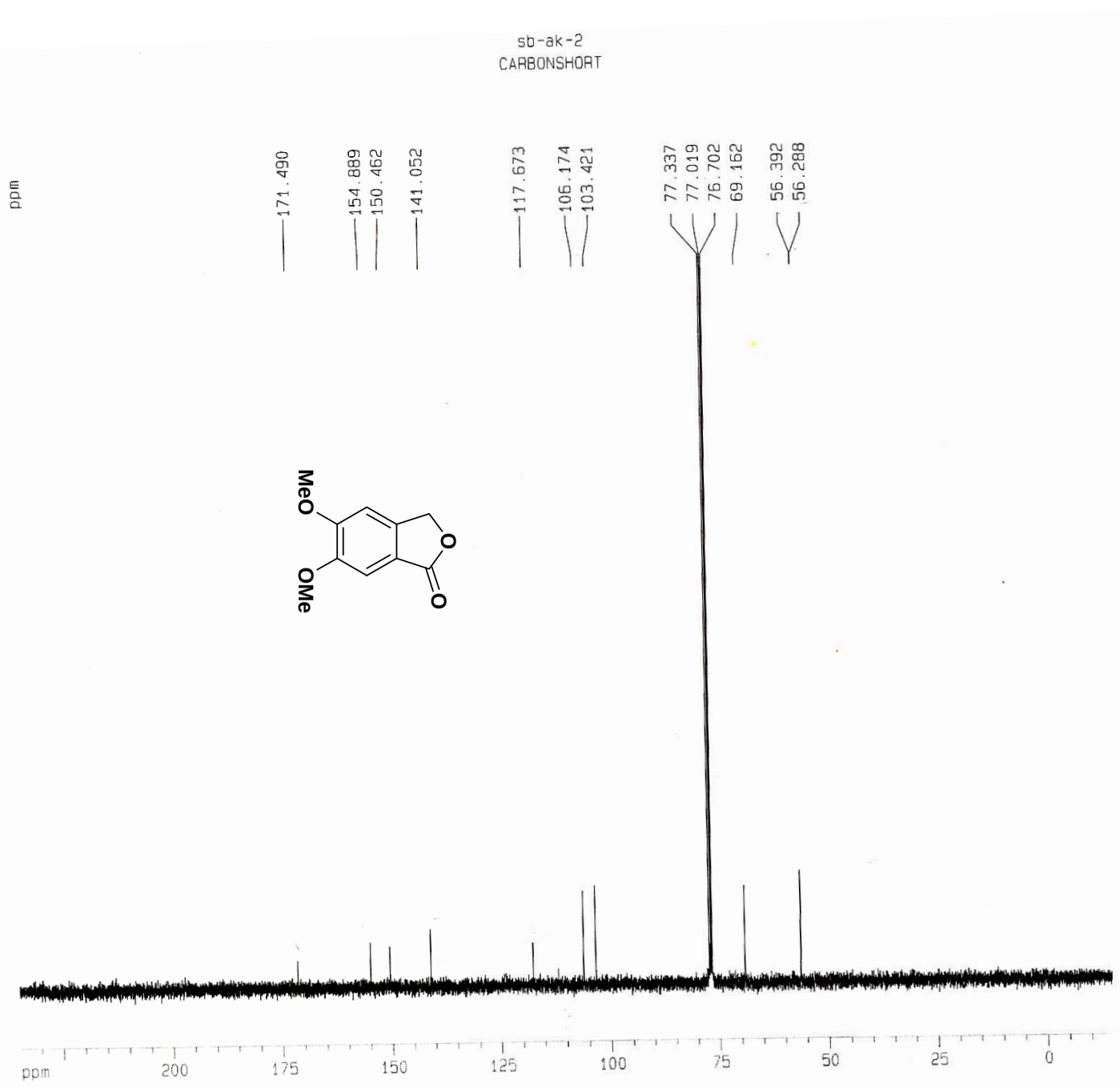


2

Pyrroridino[a]-1-methyl-1,2,3,4-terahydroisoquinoline (2): To a stirred solution of LAH (1 mg, 0.0264 mmol) in dry THF (25 µl)at 0 °C under argon atmosphere was slowly added a solution of Conc.H_2SO_4 (0.5 µl) in 50 µl of dry THF. After stirring at 0 °C for 15 min, compound (**16**) (2 mg, 0.0072 mmol) in dry THF (20 µl) was added in one portion and the reaction was stirred at room temperature for 24 h. The reaction mixture was then cooled to 0 °C and 2 N NaOH (6 µl) was then carefully added and the reaction mixture stirred for 15 min. The solid was collected and washed with ether. Organic layer was dried over anhydrous Na_2SO_4, conc. under reduced pressure and purified by column chromatography over deactivated silica gel using gradient elution with 30–50% methanol in DCM to yield the corresponding methyl analogue ofCrispine A (**2**) in 80% yield. IR (neat) 2962, 2931, 2862, 1609, 1510, 1464, 1357, 1254, 1211, 1167, 1078, 996, 858, 770 cm-1; ^{1}H-NMR

values were consistent with those previously reported;[26] HRMS (ESI) calcd for $C_{15}H_{21}NO_2$ $(M+Na)^+$: 248.1651; found: 248.1657.


sb-ak-34
PROTON(-5to15) CDCl3 d: litmadras 5
Current Data Parameters
NAME sbak
EXPNO 50
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060110
Time 20.54
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.35 usec
PL1 0.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
7.26823
6.87820
5.19529
3.96646
3.93854
1.61201
1.0117
2.6319
7.5722
0.4242
0.0847
MeO
OMe
O
O
ppm
Integral
ppm



Current Data Parameters

NAME	sb
EXPNO	452
PROCNO	1

F2 - Acquisition Parameters

Date_	20050527
Time	18.58
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	303
DS	2
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	90.5
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec

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

NUC1	13C
P1	5.50 usec
PL1	0.00 dB
SFO1	100.6238364 MHz

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

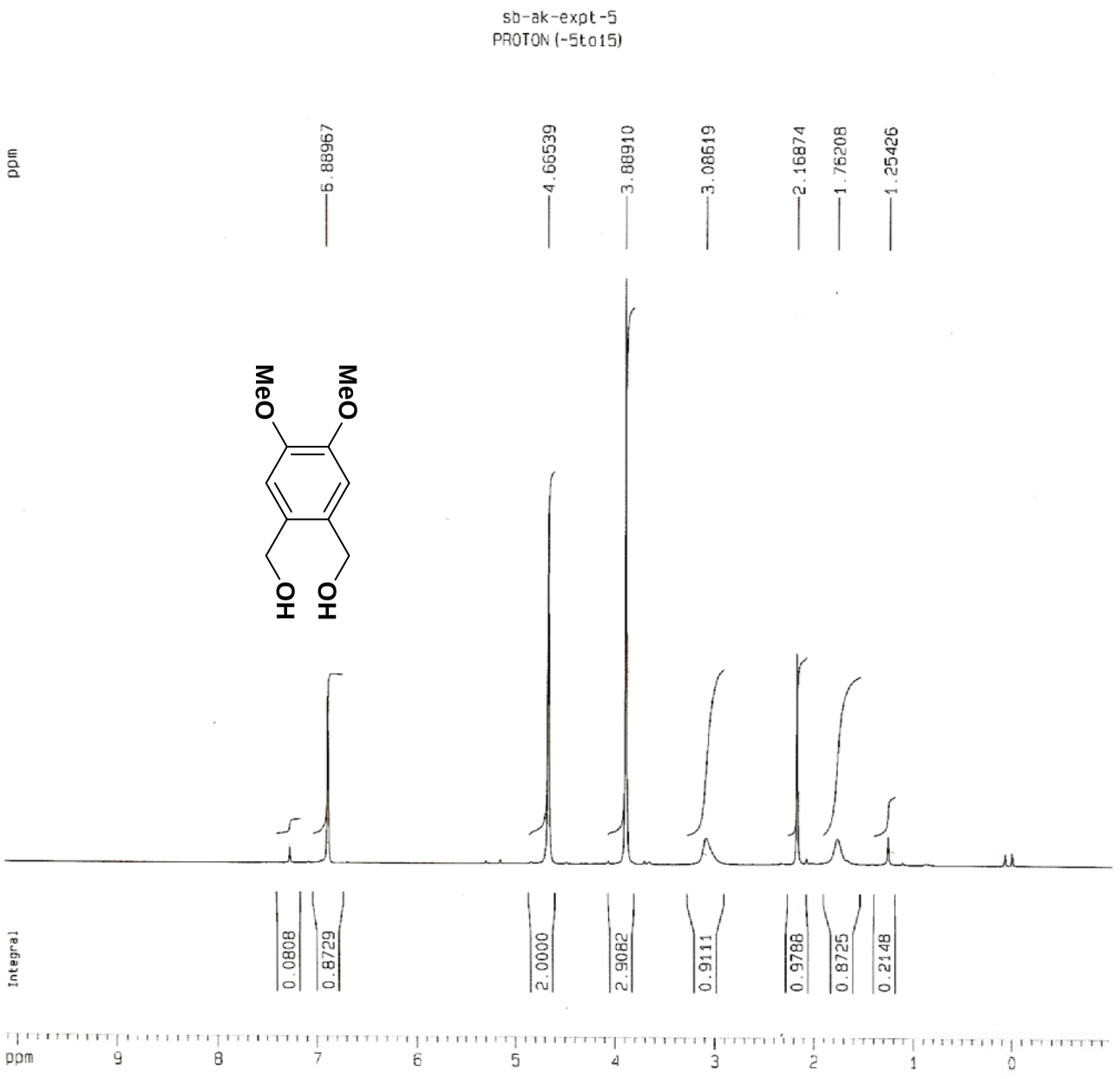
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	120.00 dB
PL12	18.00 dB
SFO2	400.1320007 MHz

F2 - Processing parameters

SI	131072
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	14.00 cm
F1P	234.849 ppm
F1	23628.84 Hz
F2P	-14.877 ppm
F2	-1496.79 Hz
PPMCM	12.48630 ppm/cm
HZCM	1256.28137 Hz/cm



Current Data Parameters
NAME sbak
EXPNO 4
PROCNO 1

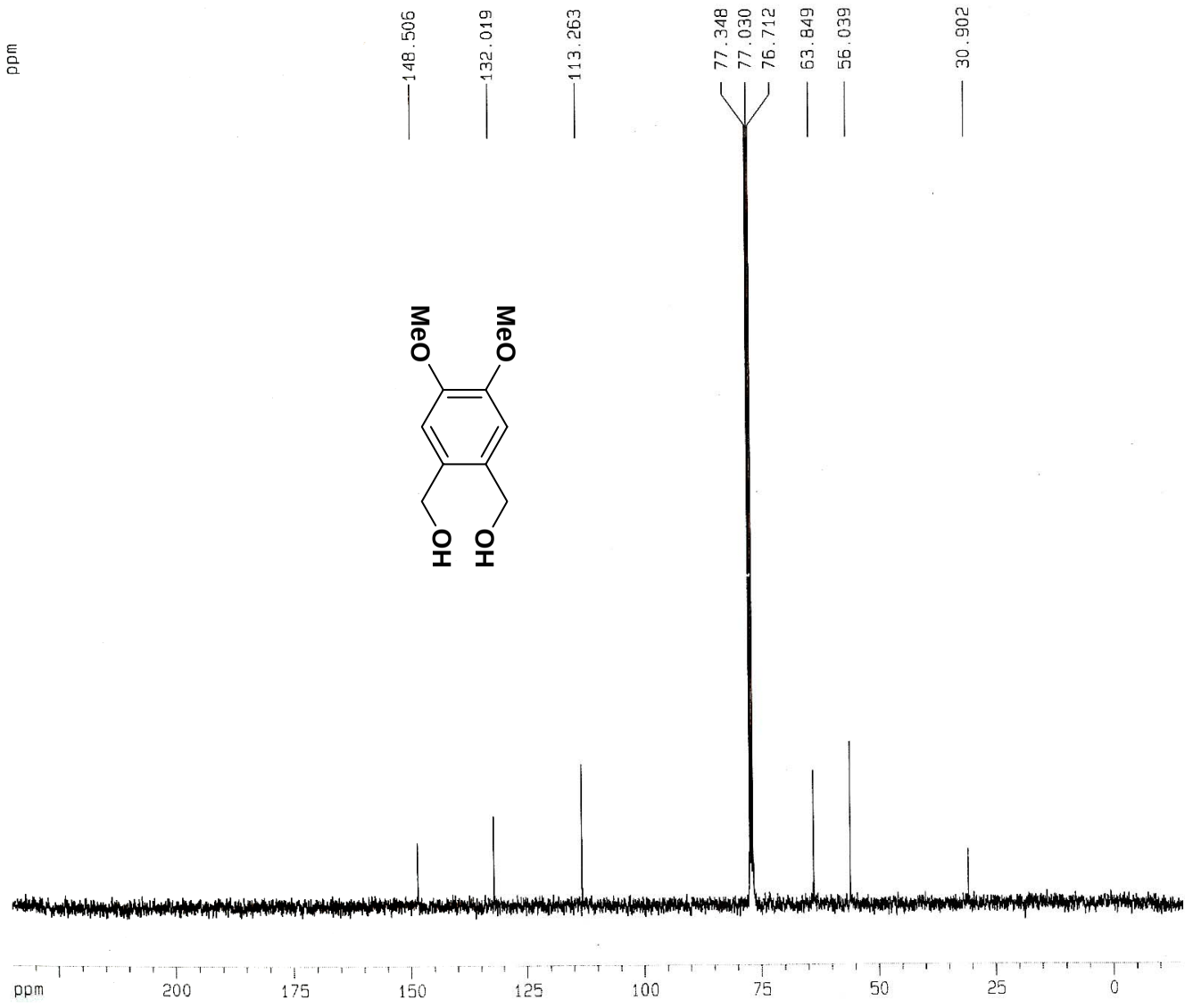
F2 - Acquisition Parameters
Date_ 20050608
Time 13.16
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.35 usec
PL1 0.00 dB
SFO1 400.1319460 MHz

F2 - Processing parameters
SI 16384
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm

sb-ak-expt-5
CARBONSHORT



Current Data Parameters

NAME	sbak
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20050608
Time	13.20
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	215
DS	2
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	1448.2
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec

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

NUC1	13C
P1	5.50 usec
PL1	0.00 dB
SF01	100.6238364 MHz

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

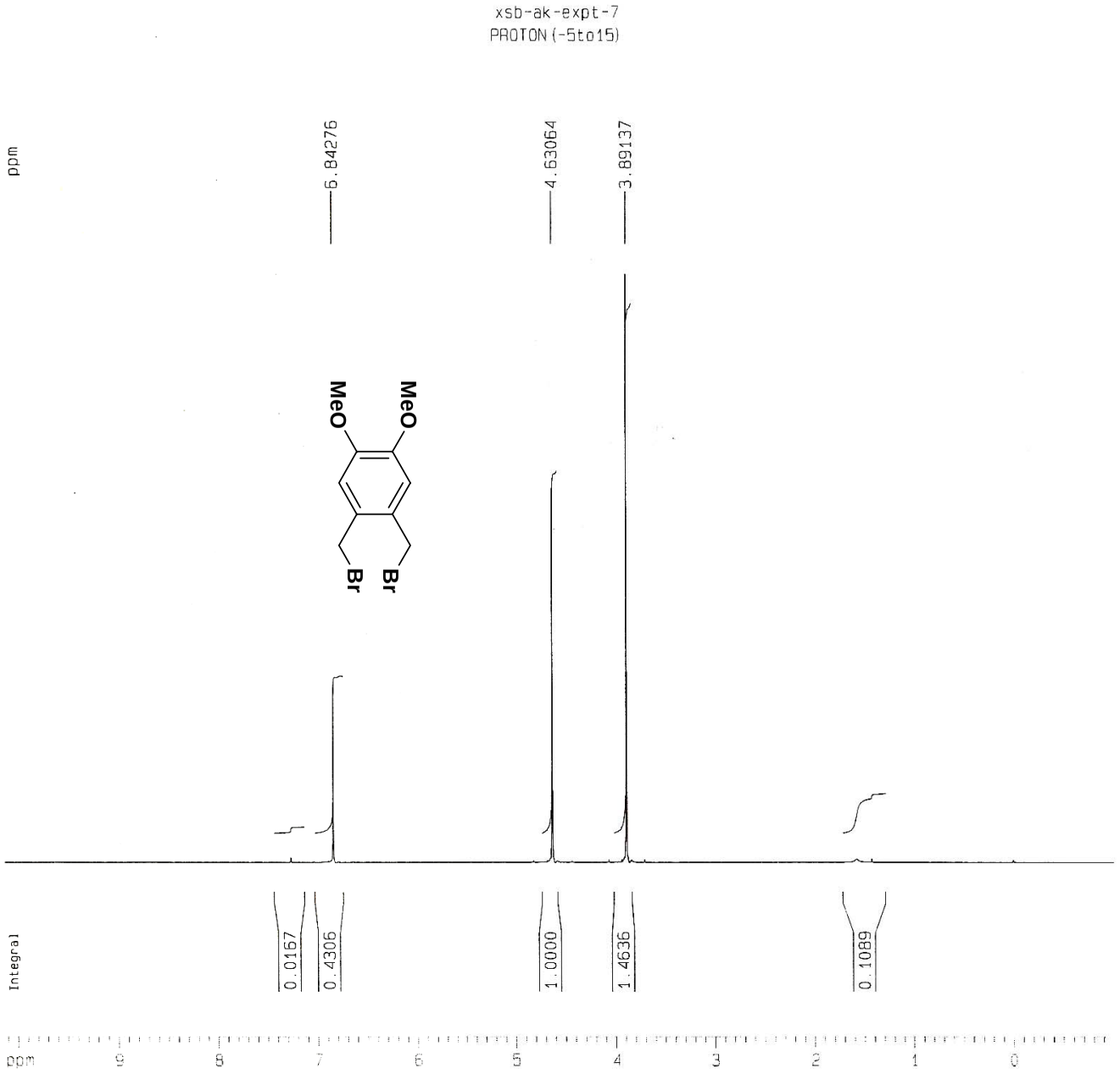
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	120.00 dB
PL12	18.00 dB
SF02	400.1320007 MHz

F2 - Processing parameters

SI	131072
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	14.00 cm
F1P	234.849 ppm
F1	23628.84 Hz
F2P	-14.877 ppm
F2	-1496.79 Hz
PPMCM	12.48630 ppm/cm
HZCM	1256.28137 Hz/cm



Current Data Parameters

NAME	sbak
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20050708
Time	11.01
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8389.262 Hz
FIDRES	0.256020 Hz
AQ	1.9530228 sec
RG	64
DW	59.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec

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

NUC1	1H
P1	8.35 usec
PL1	0.00 dB
SFO1	400.1319460 MHz

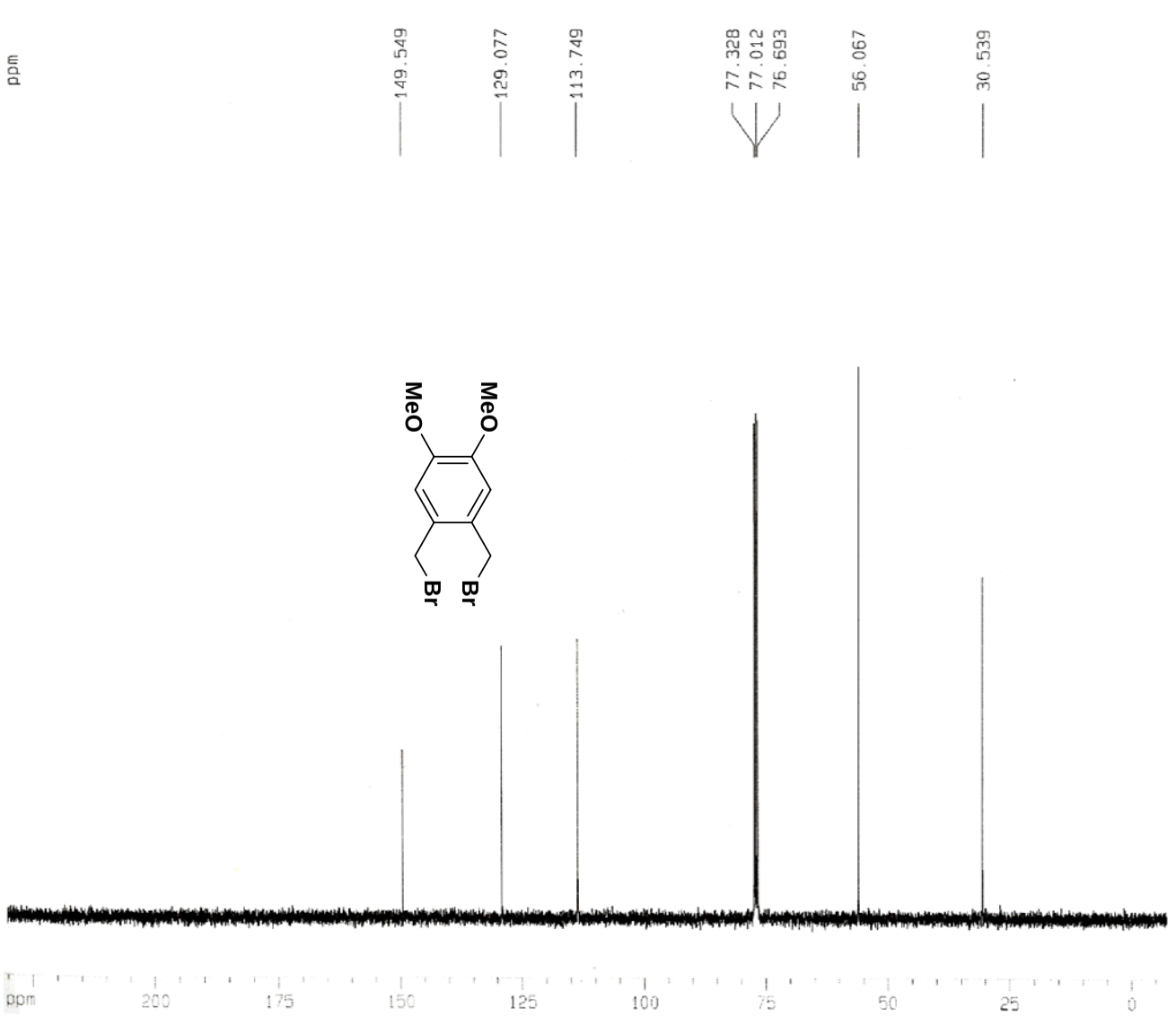
F2 - Processing parameters

SI	16384
SF	400.1300070 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	3.00

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	10.129 ppm
F1	4052.73 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.55643 ppm/cm
HZCM	222.64290 Hz/cm

sb-ak-expt-7
CARBONSHORT



Current Data Parameters
NAME sbak
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050708
Time 10.42
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 363
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 181
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 9.10 cm
F1P 230.605 ppm
F1 23201.76 Hz
F2P -7.259 ppm
F2 -730.32 Hz
PPMCM 11.89316 ppm/cm
HZCM 1196.60425 Hz/cm

SB/AK/10
PROTON (-5to15)

Current Data Parameters

NAME	sbak
EXPNO	18
PROCNO	1

F2 - Acquisition Parameters

Date_	20050722
Time	17.25
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8389.262 Hz
FIDRES	0.256020 Hz
AQ	1.9530228 sec
RG	22.6
DW	59.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec

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

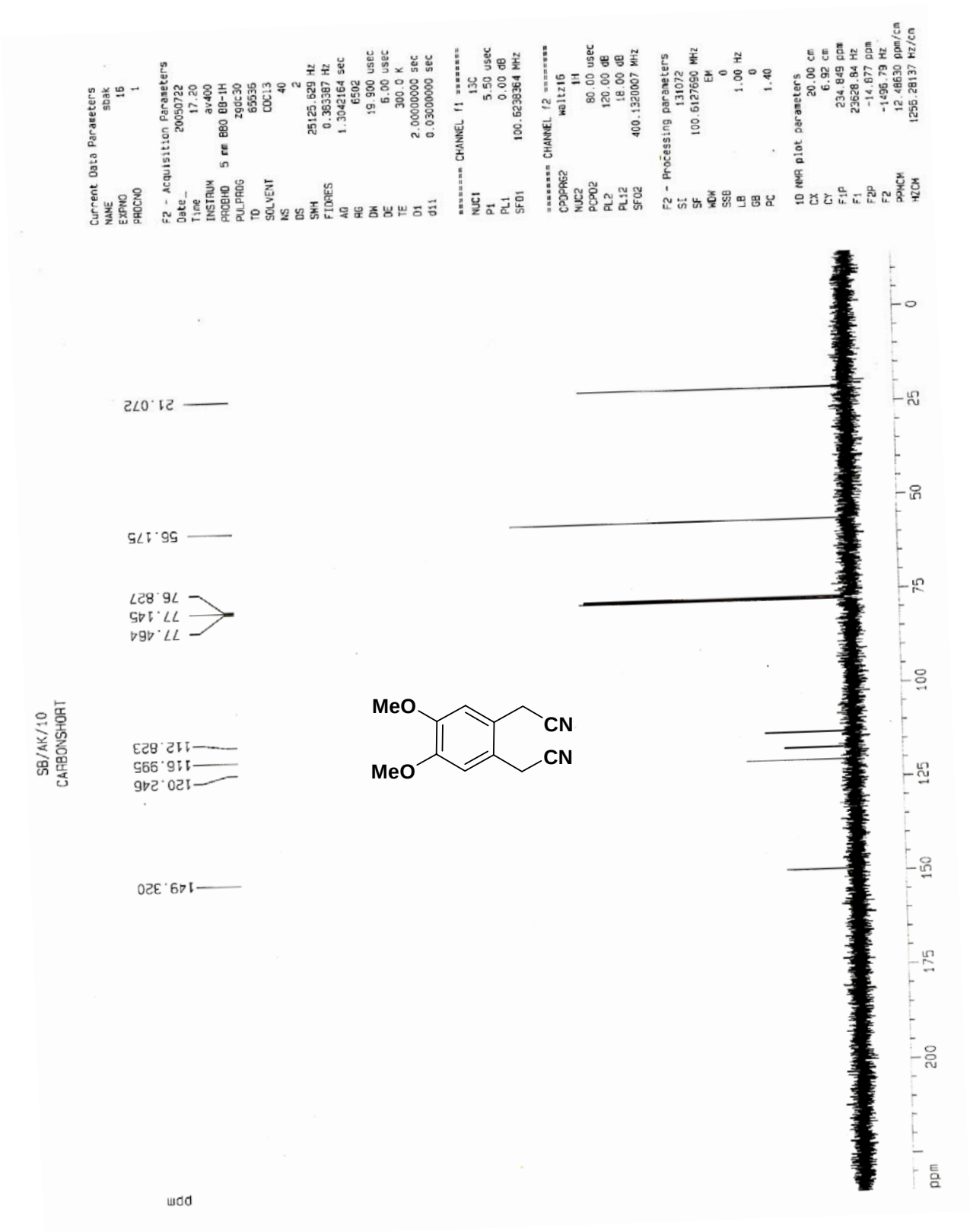
NUC1	1H
P1	8.35 usec
PL1	0.00 dB
SFO1	400.1319460 MHz

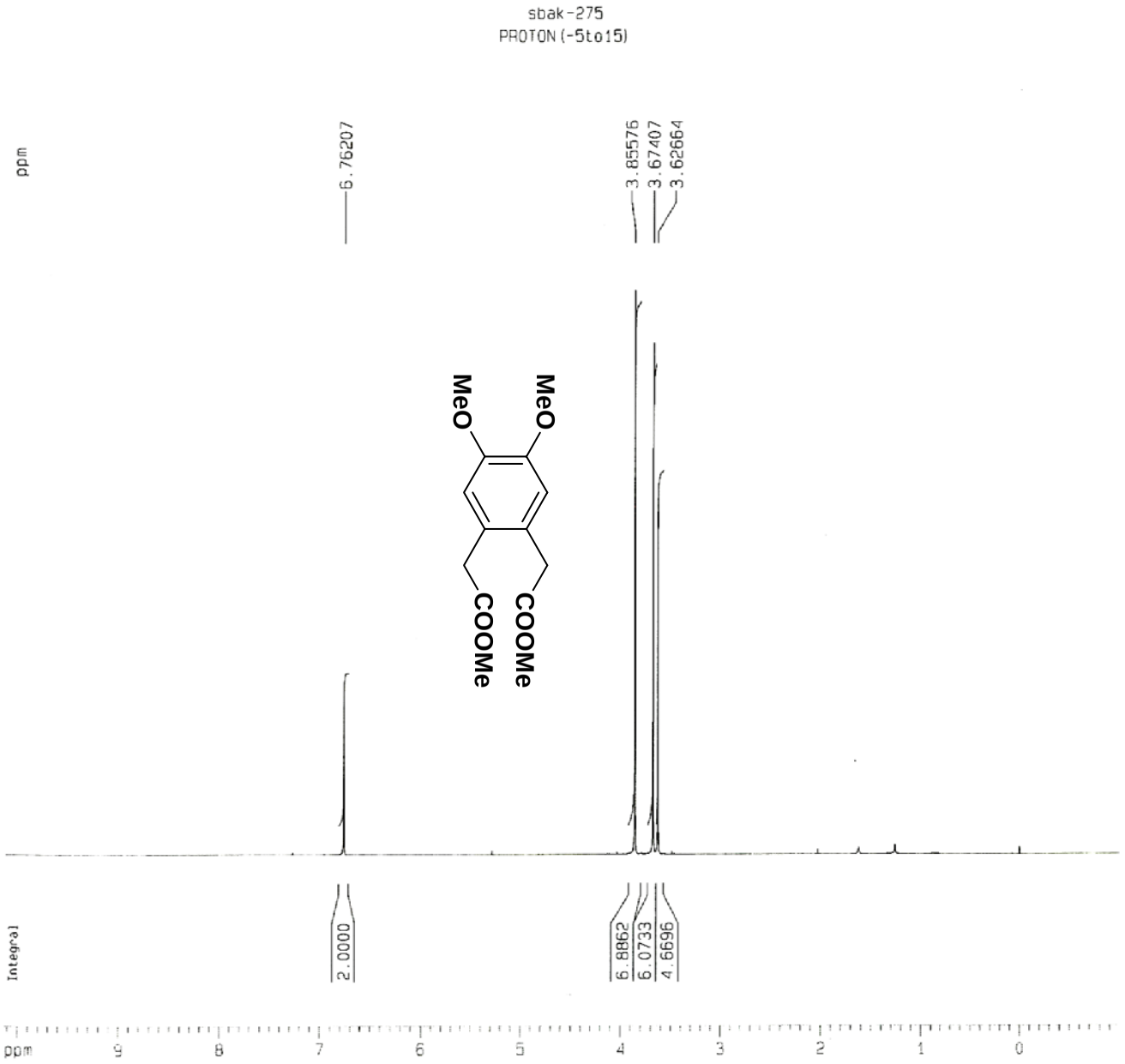
F2 - Processing parameters

SI	16384
SF	400.1299951 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	3.00

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	10.129 ppm
F1	4052.73 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.55643 ppm/cm
HZCM	222.64290 Hz/cm


SB/AK/10
CARBONSHORT
MeO
MeO
CN
CN
21.072
56.175
76.827
77.145
77.464
112.823
116.995
120.246
149.320
Current Data Parameters
NAME soak
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050722
Time 17.20
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 40
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 6502
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.92 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.677 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28137 Hz/cm
ppm
0
25
50
75
100
125
150
175
200



Current Data Parameters

NAME	sb2
EXPNO	628
PROCNO	1

F2 - Acquisition Parameters

Date_	20070609
Time	11.25
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8389.262 Hz
FIDRES	0.256020 Hz
AQ	1.9530228 sec
RG	57
DW	59.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec

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

NUC1	1H
P1	11.00 usec
PL1	3.00 dB
SFO1	400.1319460 MHz

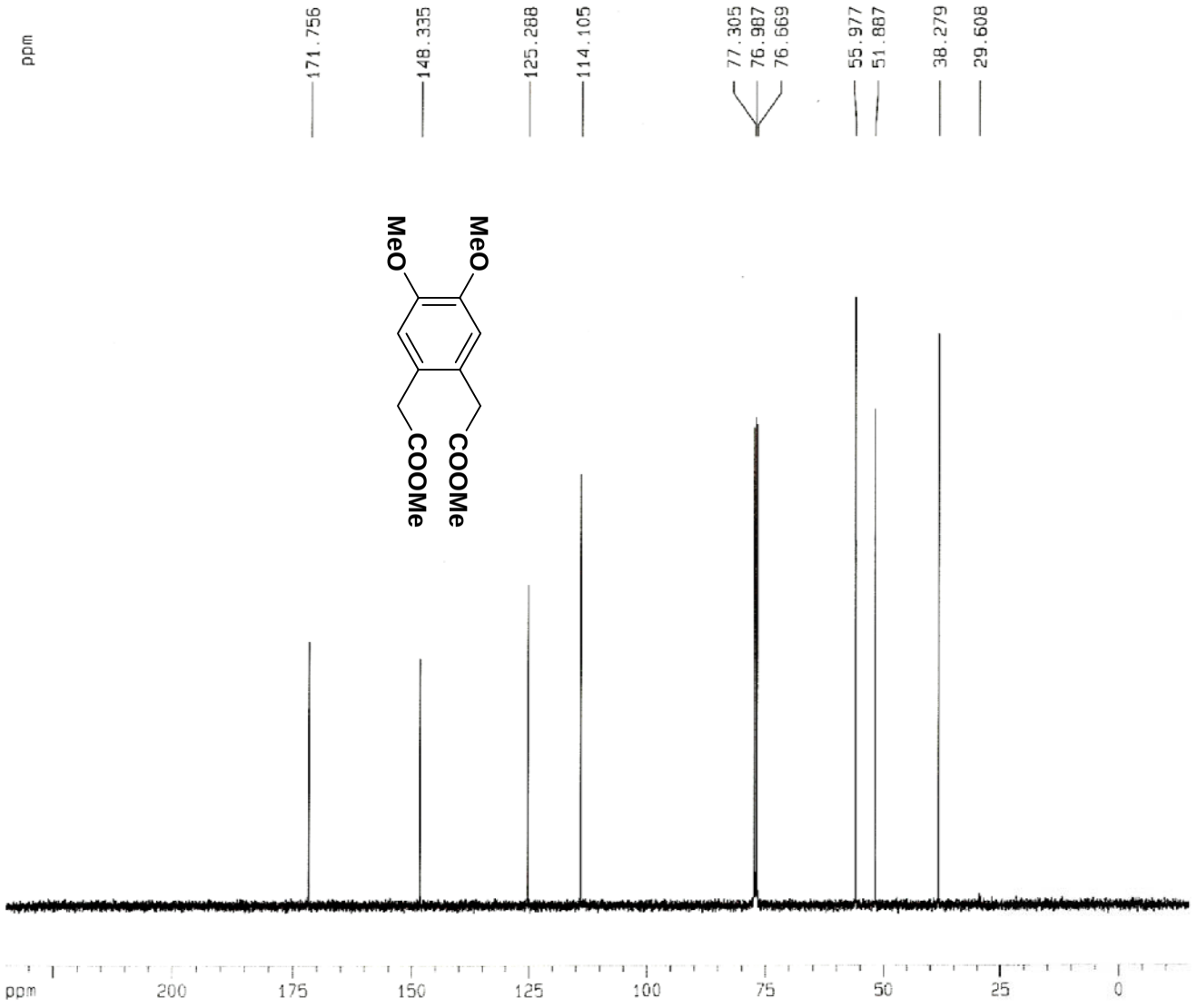
F2 - Processing parameters

SI	16384
SF	400.1300055 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	3.00

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	10.129 ppm
F1	4052.73 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.55643 ppm/cm
HZCM	222.64290 Hz/cm

sbak-275
CARBONSHORT

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Current Data Parameters
NAME                  sb2
EXPNO                 629
PROCNO                  1

F2 - Acquisition Parameters
Date_              20070609
Time                 11.55
INSTRUM              av400
PROBHD   5 mm BBO BB-1H
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      2
SWH             25125.629 Hz
FIDRES              0.383387 Hz
AQ                1.3042164 sec
RG                   1149.4
DW                  19.900 usec
DE                   6.00 usec
TE                  300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.00 usec
PL1                  0.00 dB
SFO1          100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                  3.00 dB
PL12                20.23 dB
PL13                16.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                  131072
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   9.94 cm
F1P                234.849 ppm
F1                23628.84 Hz
F2P                -14.877 ppm
F2                -1498.79 Hz
PPMCM             12.48630 ppm/cm
HZCM          1256.28137 Hz/cm
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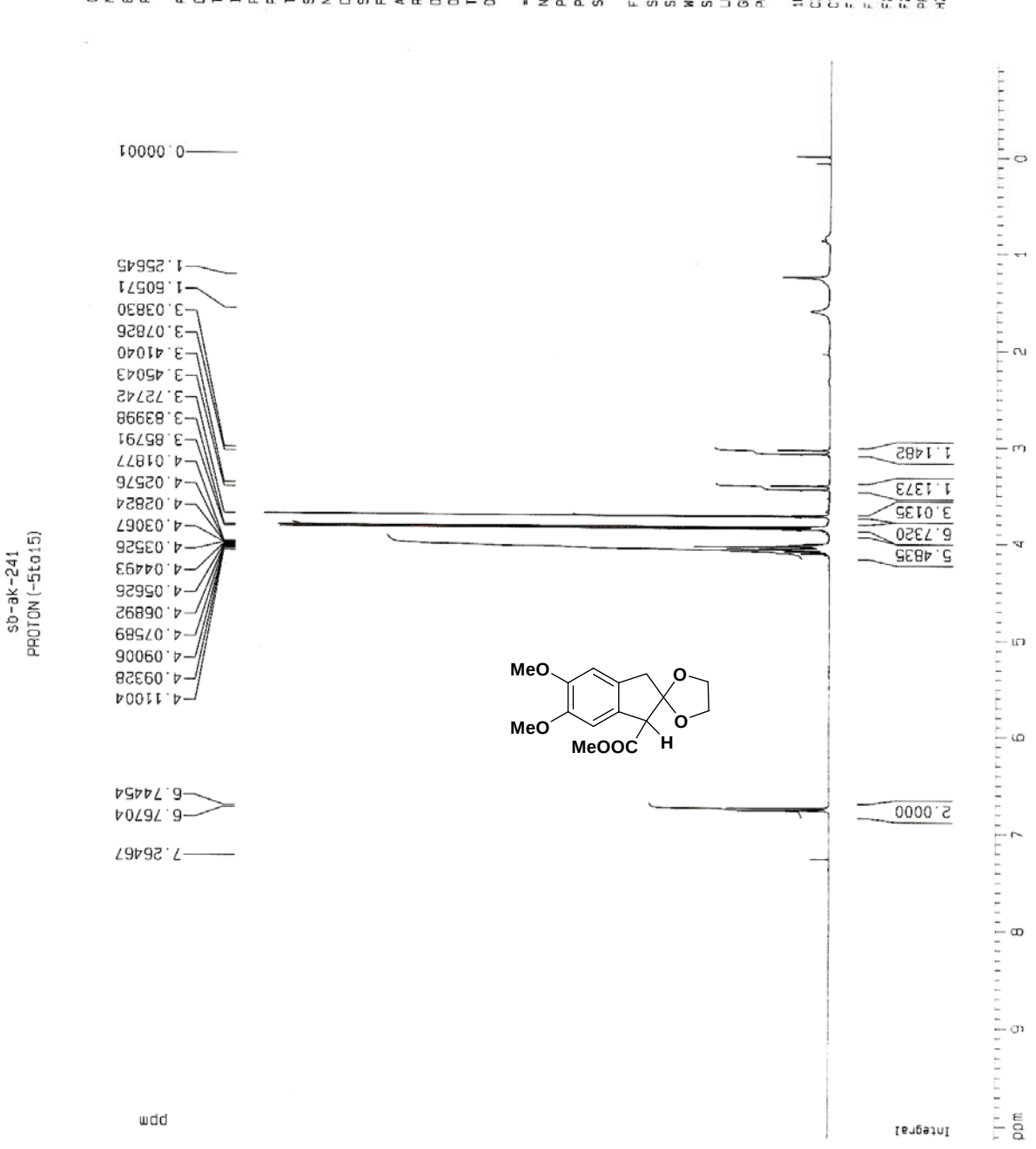
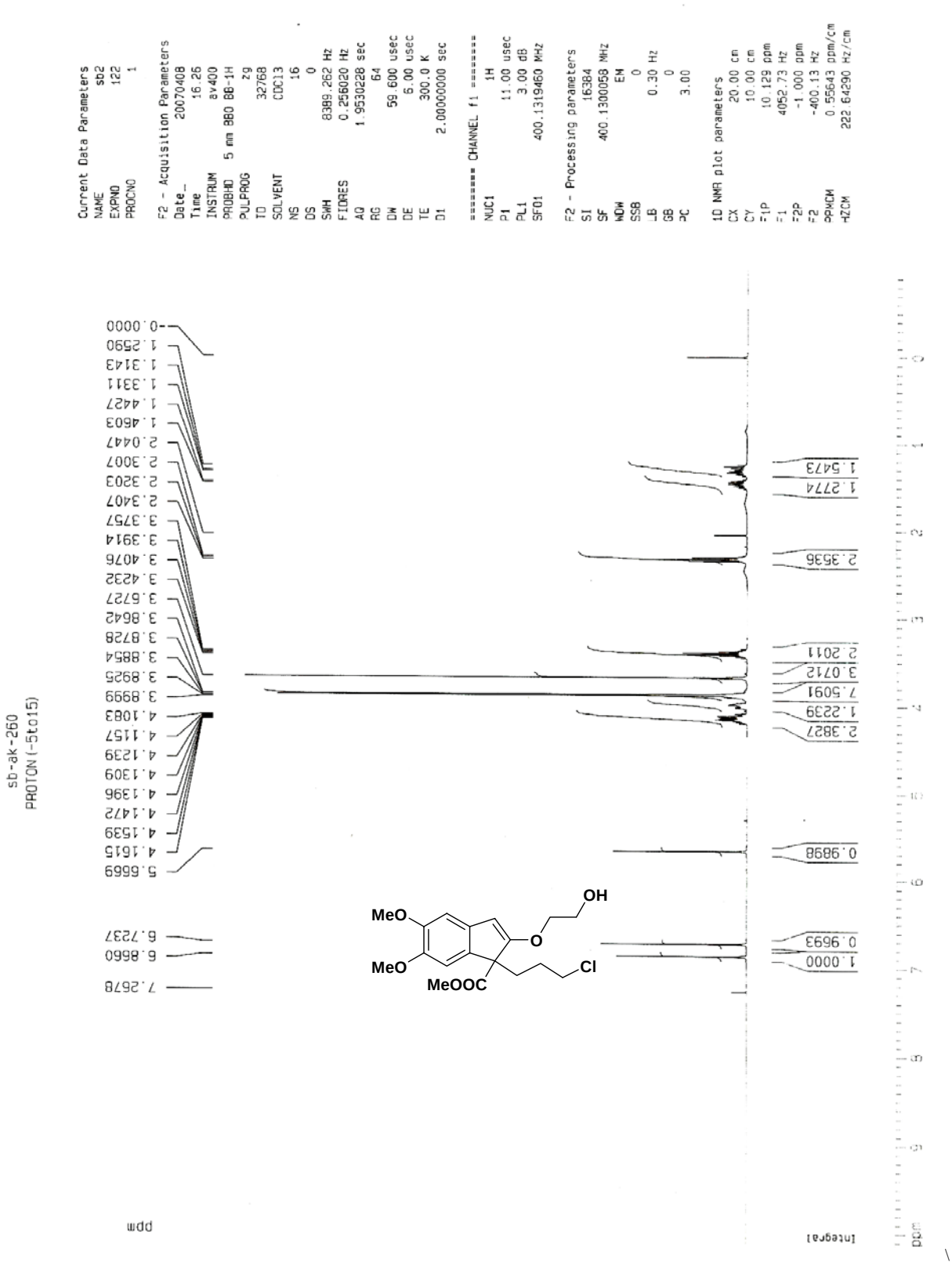
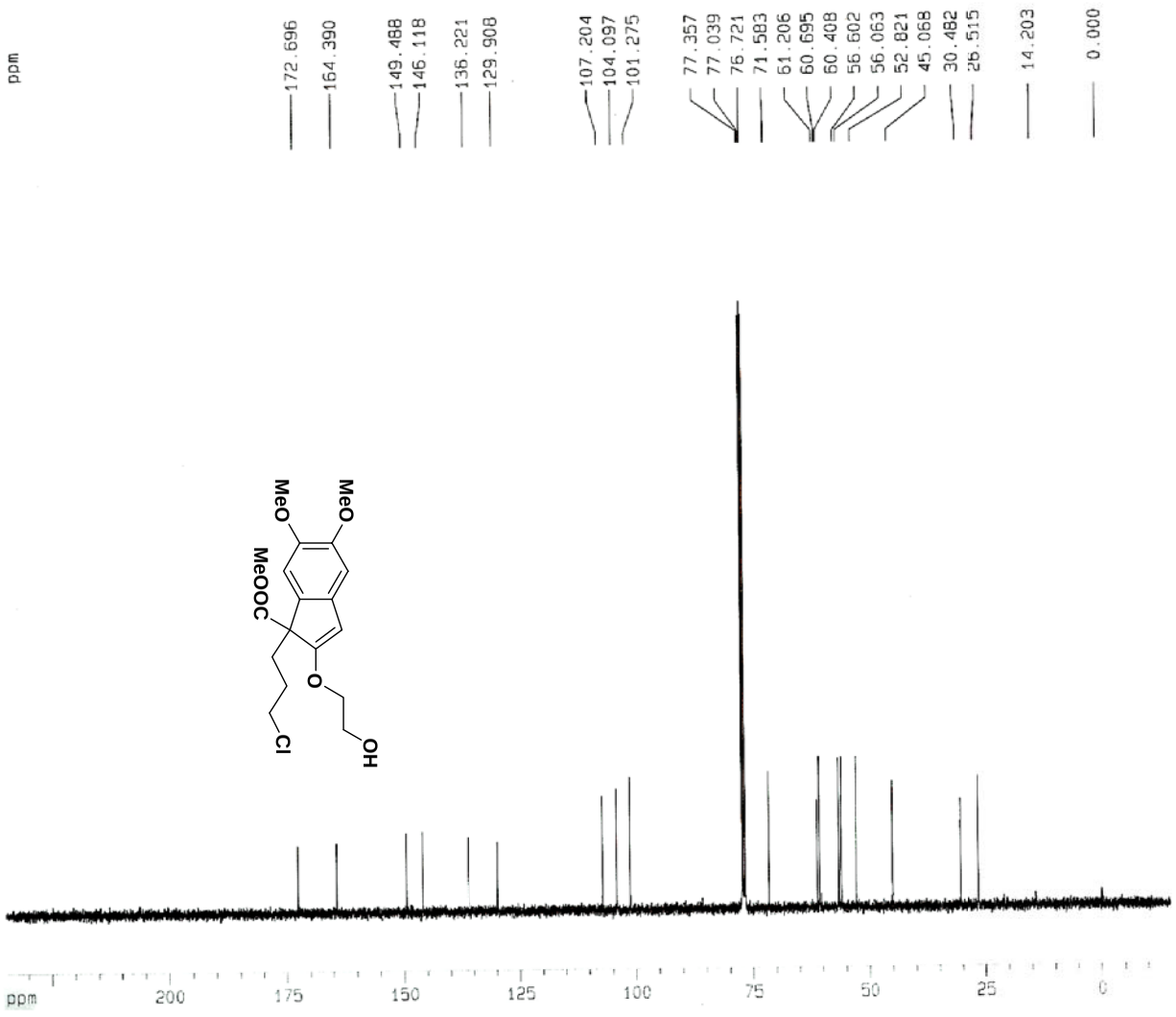

sb-ak-241
PROTON (-5to15)
Current Data Parameters
NAME sb1
EXPNO 1088
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070304
Time 17.20
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 90.5
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 -3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
0.00001
1.25645
1.60571
3.03830
3.07826
3.41040
3.45043
3.72742
3.83998
3.85791
4.01877
4.02576
4.02824
4.03067
4.03526
4.04493
4.05626
4.06892
4.07589
4.09006
4.09328
4.11004
6.74654
6.76704
7.26467
1.1482
1.1373
3.0135
5.7320
5.4835
2.0000
MeO
MeO
MeOOC
O
O
H
ppm
Integral
ppm

sb-ak-241
CARBONSHORT
ppm
42.998
52.067
55.979
56.054
59.392
64.546
65.475
76.690
77.008
77.326
107.789
108.391
117.911
129.444
132.343
148.547
149.436
171.015
MeO
MeO
MeOOC
H
O
O
Current Data Parameters
NAME sb1
EXPNO 1089
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070304
Time 17.22
INSTRUM av400
PROBHD 5 mm BBO BB-IH
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 -4.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 15.98 dB
PL13 16.00 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28137 Hz/cm
ppm
200
175
150
125
100
75
50
25
0


sb-ak-260
PROTON(-5to15)
Current Data Parameters
NAME sb2
EXPNO 122
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070408
Time 16.26
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 64
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SF01 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
MeO
MeO
MeOOC
O
OH
Cl
ppm
Integral
ppm

sb-ak-260
CARBONSHORT



Current Data Parameters

NAME	sb2
EXPNO	123
PROCNO	1

F2 - Acquisition Parameters

Date_	20070408
Time	23.15
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	2
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	14596.5
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	11.00 usec
PL1	0.00 dB
SF01	100.6238364 MHz

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

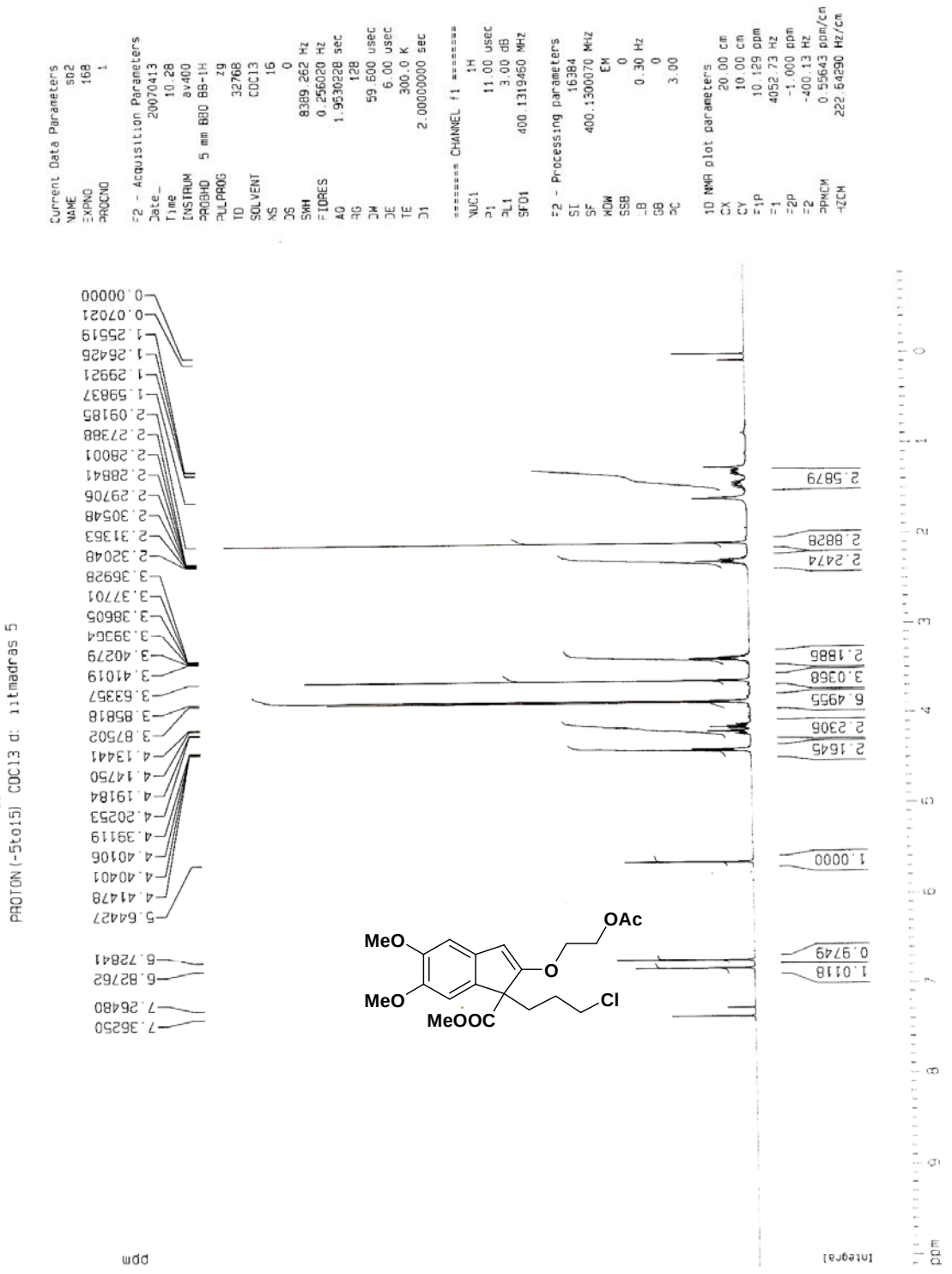
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.00 dB
PL12	20.23 dB
PL13	16.00 dB
SF02	400.1320007 MHz

F2 - Processing parameters

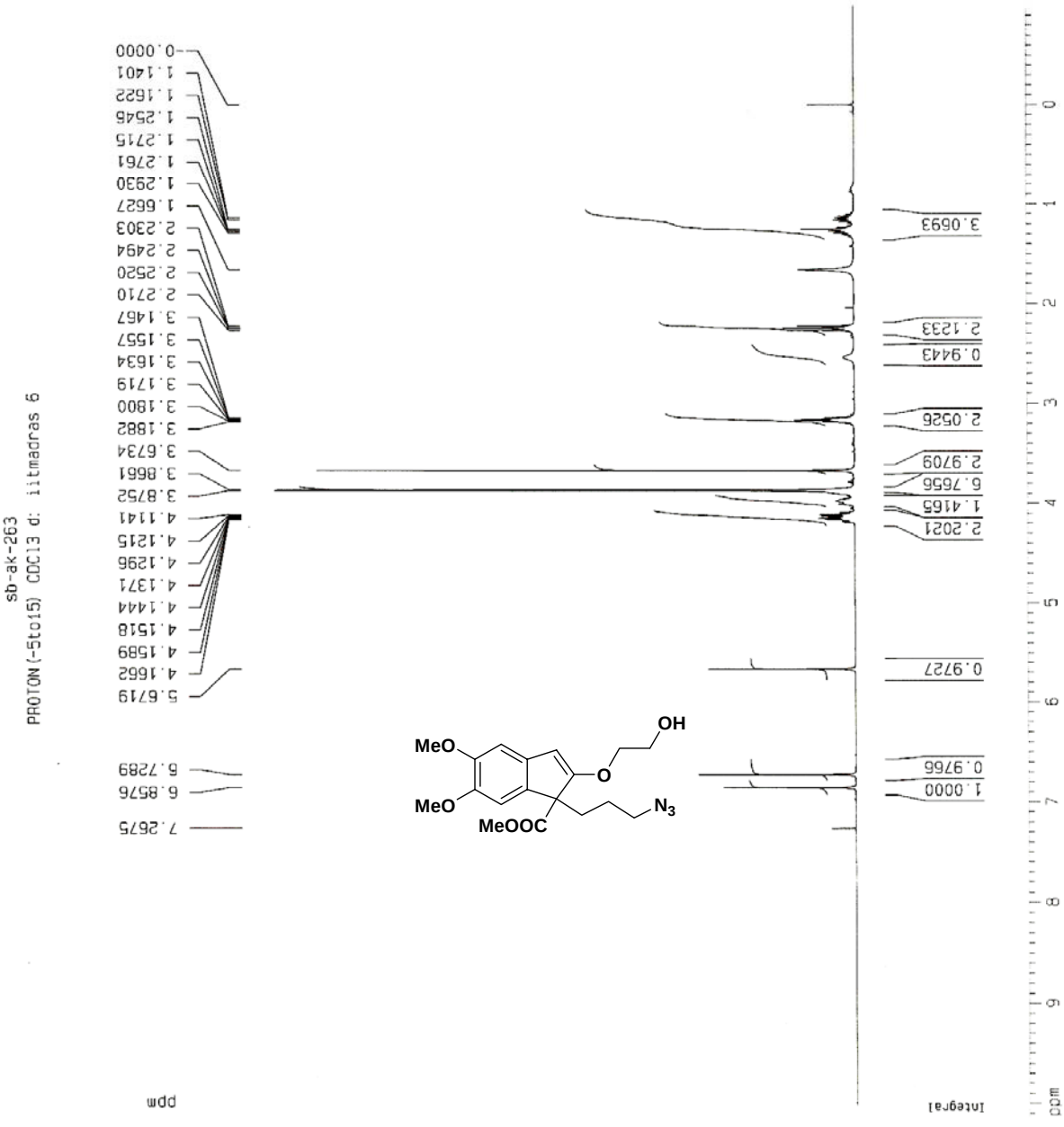
SI	131072
SF	100.6127670 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

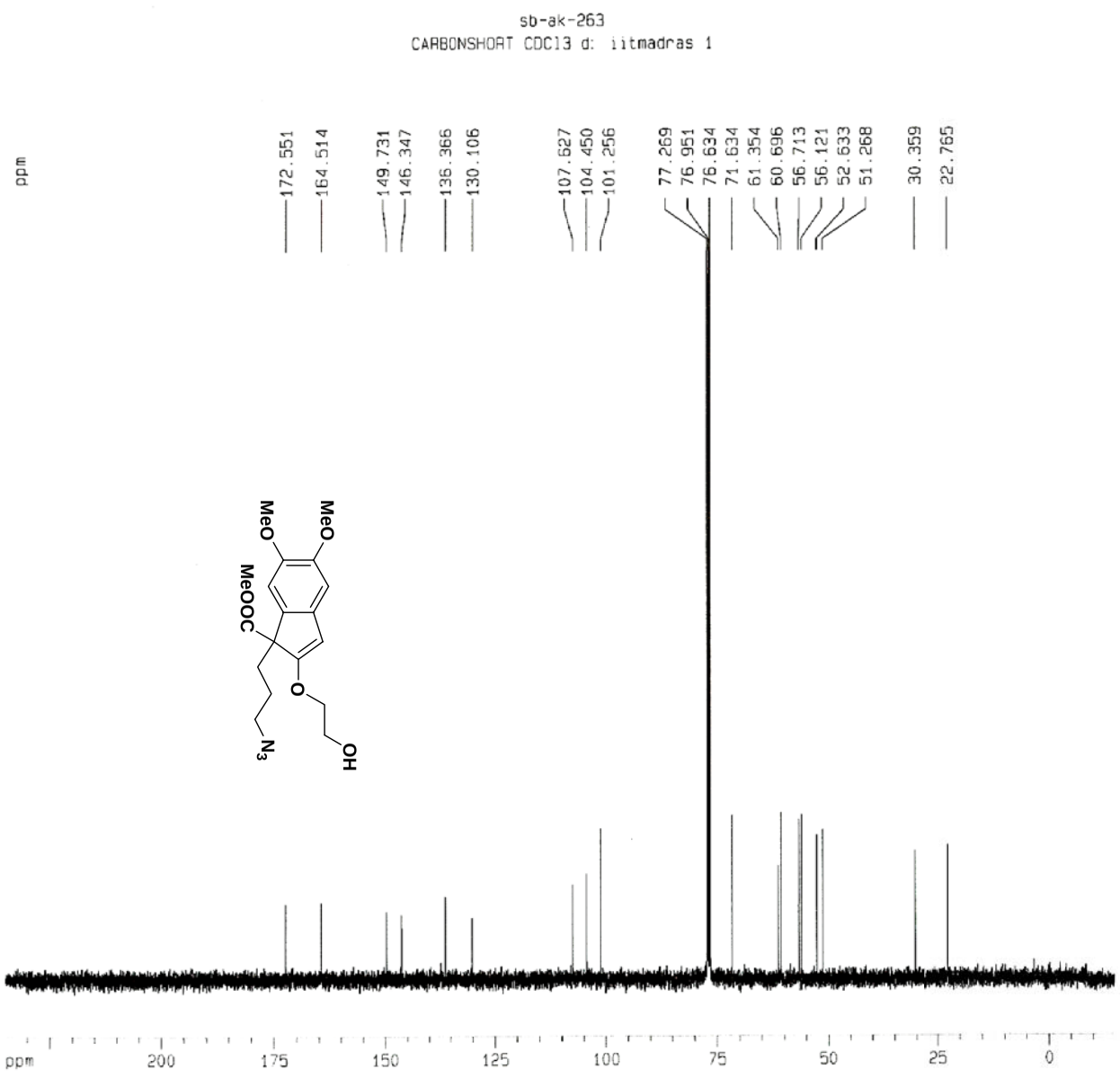
1D NMR plot parameters

CX	20.00 cm
CY	9.94 cm
F1P	234.849 ppm
F1	23628.84 Hz
F2P	-14.877 ppm
F2	-1496.79 Hz
PPMCM	12.48630 ppm/cm
HZCM	1256.28137 Hz/cm


sb-ak-261
PROTON(-5to15) CDCl3 d: iitmadras 5
Current Data Parameters
NAME sb2
EXPNO 168
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070413
Time 10.28
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 128
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
0.00000
0.07021
1.25519
1.26426
1.29921
1.59837
2.09185
2.27388
2.28001
2.28841
2.29706
2.30548
2.31353
2.32048
3.36928
3.37701
3.38605
3.39364
3.40279
3.41019
3.83357
3.85818
3.87502
4.13441
4.14750
4.19184
4.20253
4.39119
4.40106
4.40401
4.41478
5.64427
6.72841
6.82762
7.26480
7.36250
2.5879
2.8828
2.2474
2.1885
3.0368
6.4955
2.2305
2.1645
1.0000
0.9749
1.0118
MeO
MeO
MeOOC
O
OAc
Cl
ppm
Integral

sbak-261
CARBONSHOT
Current Data Parameters
NAME sb2
EXPNO 167
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070413
Time 7.08
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 16.00 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127637 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23620.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28125 Hz/cm
-0.000
20.793
26.627
29.710
30.582
44.988
52.602
56.130
56.631
61.121
62.173
68.098
76.717
77.034
77.352
100.966
104.276
107.130
128.354
130.453
136.194
146.276
149.544
164.882
170.853
172.449
ppm
MeO
MeO
MeOOC
O
OAc
Cl


sb-ak-263
PROTON(-5to15) CDCl3 d: litmadras 6
Current Data Parameters
NAME sb2
EXPNO 583
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070605
Time 16.34
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 101.6
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
0.0000
1.1401
1.1622
1.2545
1.2715
1.2761
1.2930
1.6627
2.2303
2.2494
2.2520
2.2710
3.1467
3.1557
3.1634
3.1719
3.1800
3.1882
3.6734
3.8661
3.8752
4.1141
4.1215
4.1296
4.1371
4.1444
4.1518
4.1589
4.1662
5.6719
6.7289
6.8576
7.2675
3.0693
2.1233
0.9443
2.0526
2.9709
6.7656
1.4165
2.2021
0.9727
0.9766
1.0000
MeO
MeO
MeOOC
O
OH
N3
ppm
Integral



Current Data Parameters

NAME	sb2
EXPNO	622
PROCNO	1

F2 - Acquisition Parameters

Date_	20070608
Time	23.11
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1000
DS	2
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	1149.4
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	11.00 usec
PL1	0.00 dB
SF01	100.6238364 MHz

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

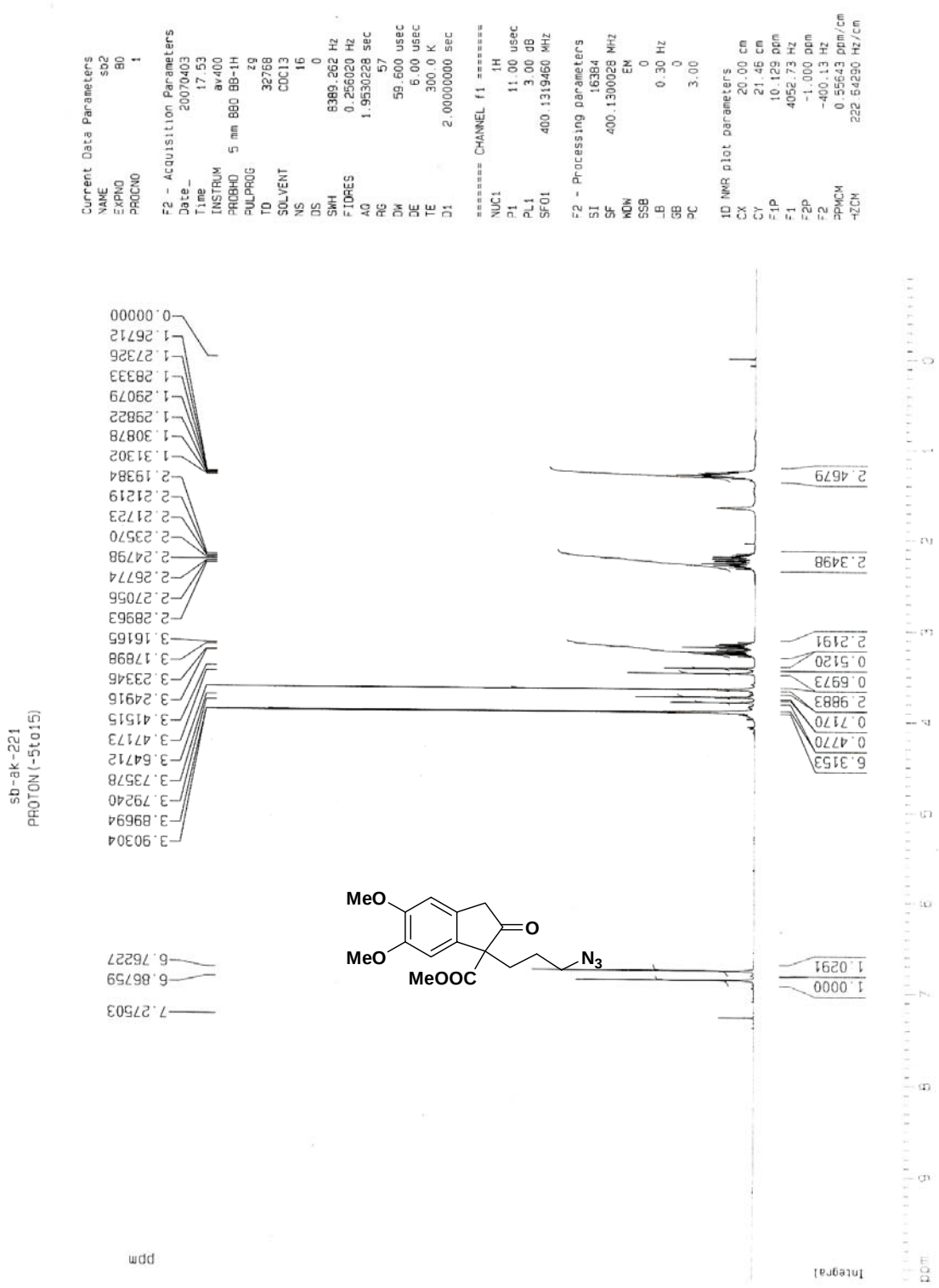
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.00 dB
PL12	20.23 dB
PL13	16.00 dB
SF02	400.1320007 MHz

F2 - Processing parameters

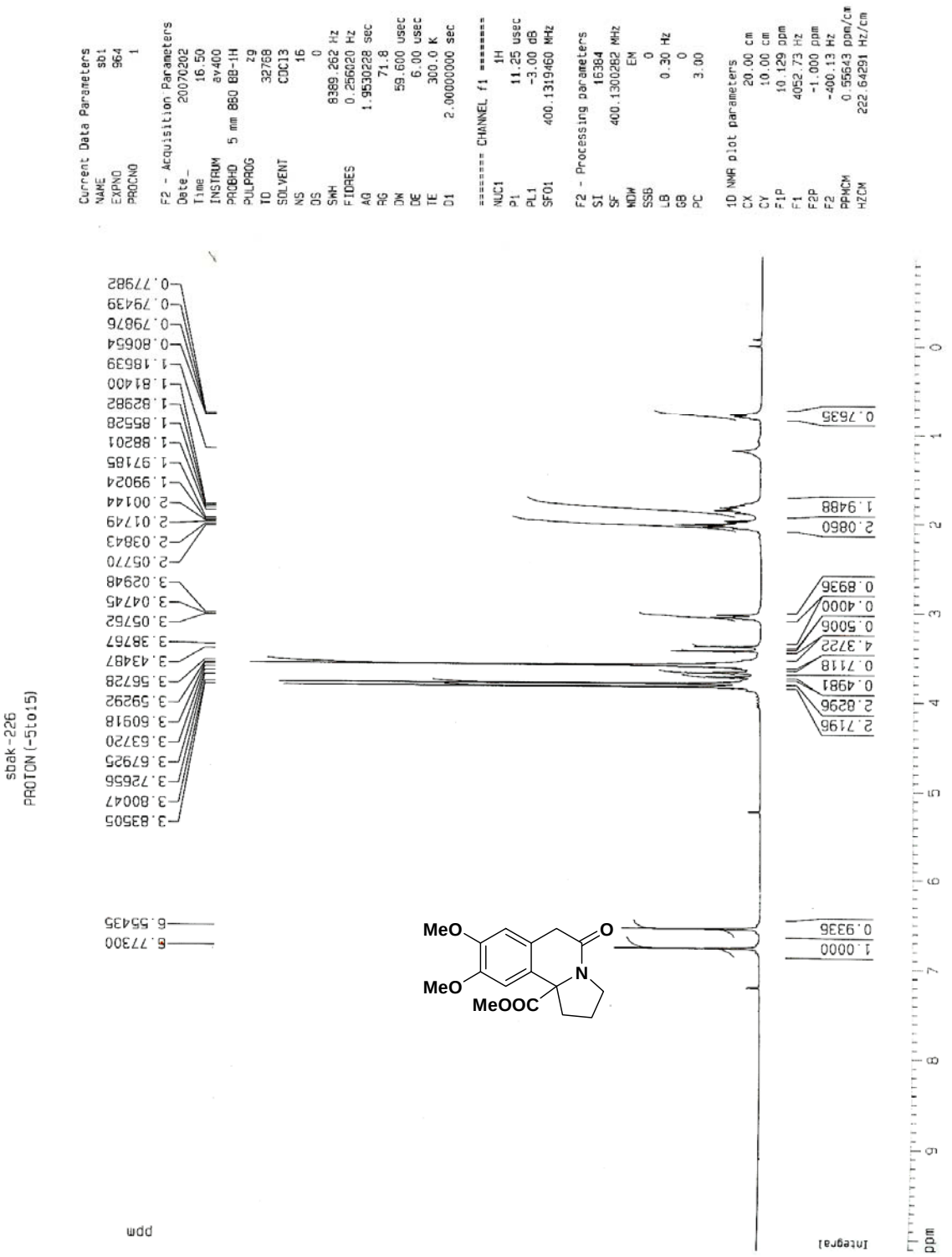
SI	131072
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

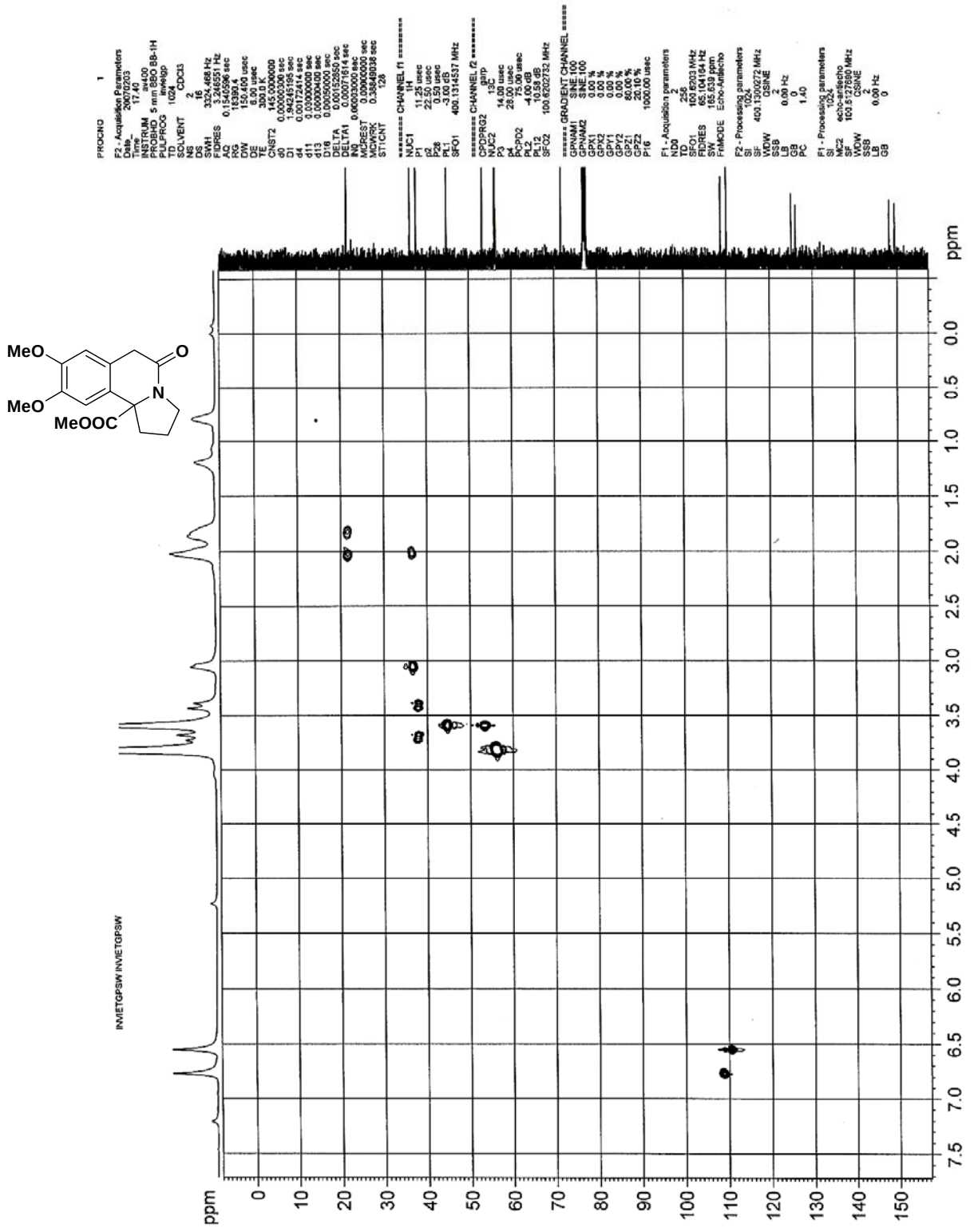
CX	20.00 cm
CY	20.47 cm
F1P	234.849 ppm
F1	23628.84 Hz
F2P	-14.877 ppm
F2	-1495.79 Hz
PPMCM	12.48630 ppm/cm
HZCM	1256.28137 Hz/cm

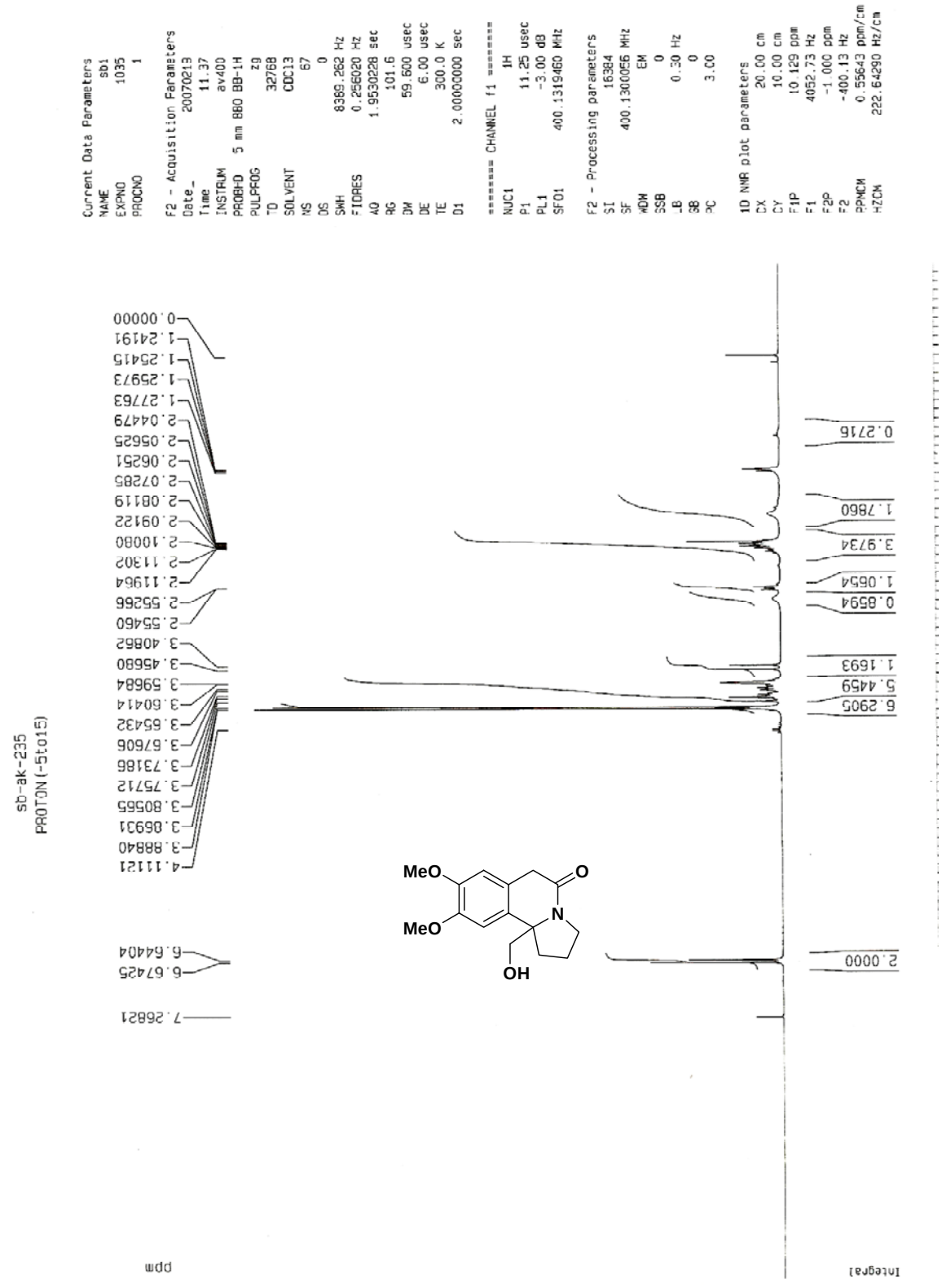

sb-ak-221
PROTON(-5to15)
Current Data Parameters
NAME sb2
EXPNO 80
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070403
Time 17.53
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 57
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 21.46 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
0.00000
1.26712
1.27326
1.28333
1.29079
1.29822
1.30878
1.31302
2.19384
2.21219
2.21723
2.23570
2.24798
2.26774
2.27056
2.28963
3.16165
3.17898
3.23346
3.24916
3.41515
3.47173
3.64712
3.73578
3.79240
3.86964
3.90304
6.76227
6.86759
7.27503
2.4679
2.3498
2.2191
0.5120
0.6973
2.9883
0.7170
0.4770
6.3153
1.0291
1.0000
MeO
MeO
MeOOC
O
N3
ppm
Integral

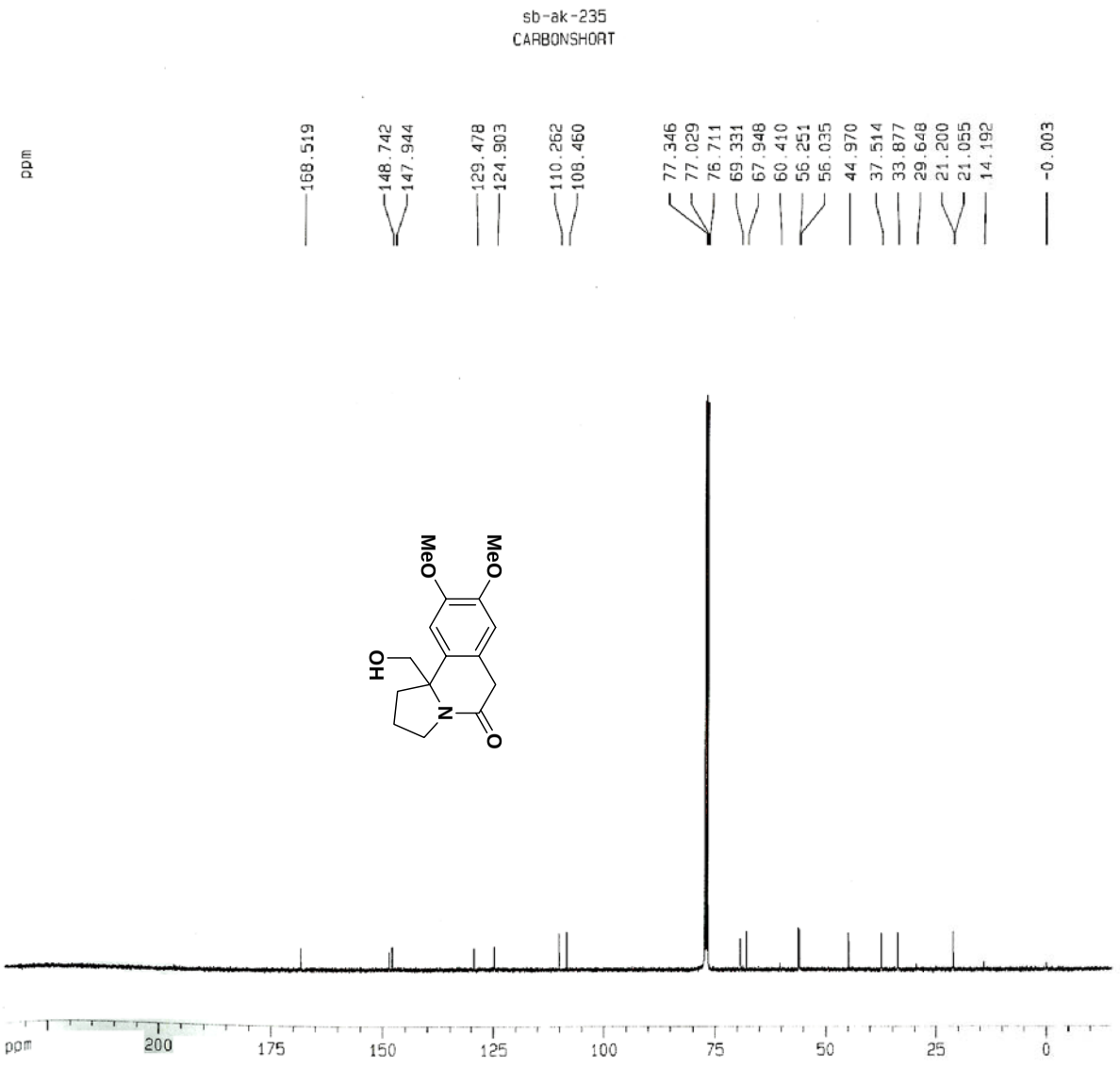
sb-ak-221
CARBONSHORT
Current Data Parameters
NAME sb2
EXPNO 81
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070403
Time 18.00
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 97
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 2896.3
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SF01 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 16.00 dB
SF02 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.49630 ppm/cm
HZCM 1256.28137 Hz/cm
MeO
MeO
MeOOC
O
N3
23.707
31.034
43.419
51.153
52.841
56.050
56.195
64.607
76.722
77.040
77.358
106.469
107.796
129.158
131.600
149.590
149.950
170.871
212.201
ppm
200
150
100
50
0


sbak-226
PROTON (-5to15)
Current Data Parameters
NAME sb1
EXPNO 964
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070202
Time 16.50
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 71.8
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 -3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300282 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64291 Hz/cm
0.77982
0.79439
0.79876
0.80654
1.18539
1.81400
1.82982
1.85528
1.88201
1.97185
1.99024
2.00144
2.01749
2.03843
2.05770
3.02948
3.04745
3.05762
3.38767
3.43487
3.56728
3.59292
3.60918
3.63720
3.67925
3.72656
3.80047
3.83505
6.55435
6.77300
0.7635
1.9488
2.0860
0.8936
0.4000
0.5006
4.3722
0.7118
0.4981
2.8296
2.7196
0.9336
1.0000
MeO
MeO
MeOOC
O
N
ppm
Integral

sb-ak-226
CARBONSHORT
Current Data Parameters
NAME sb1
EXPNO 966
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070202
Time 19.21
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 116
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 -4.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 15.98 dB
PL13 16.00 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 9.94 cm
F1P 234.849 ppm
F1 23628.64 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28137 Hz/cm
21.505
36.230
37.632
44.768
53.139
56.019
56.234
71.603
76.748
77.066
77.384
108.658
110.010
125.103
126.053
148.127
149.413
168.199
172.154
MeO
MeO
MeOOC
N
O
ppm
0
25
50
75
100
125
150
175
200


MeO
MeO
MeOOC
O
N
INVIETGPSW INVIETGPSW
ppm


sb-ak-235
PROTON(-5to15)
Current Data Parameters
NAME sb1
EXPNO 1035
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070219
Time 11.37
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 67
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 101.6
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 -3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
MeO
MeO
O
N
OH
ppm
Integral



Current Data Parameters

NAME	sb1
EXPNO	1031
PROCNO	1

F2 - Acquisition Parameters

Date_	20070217
Time	9.04
INSTRUM	av400
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8003
DS	2
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042164 sec
RG	1149.4
DW	19.900 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	14.00 usec
PL1	-4.00 dB
SFO1	100.6238364 MHz

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

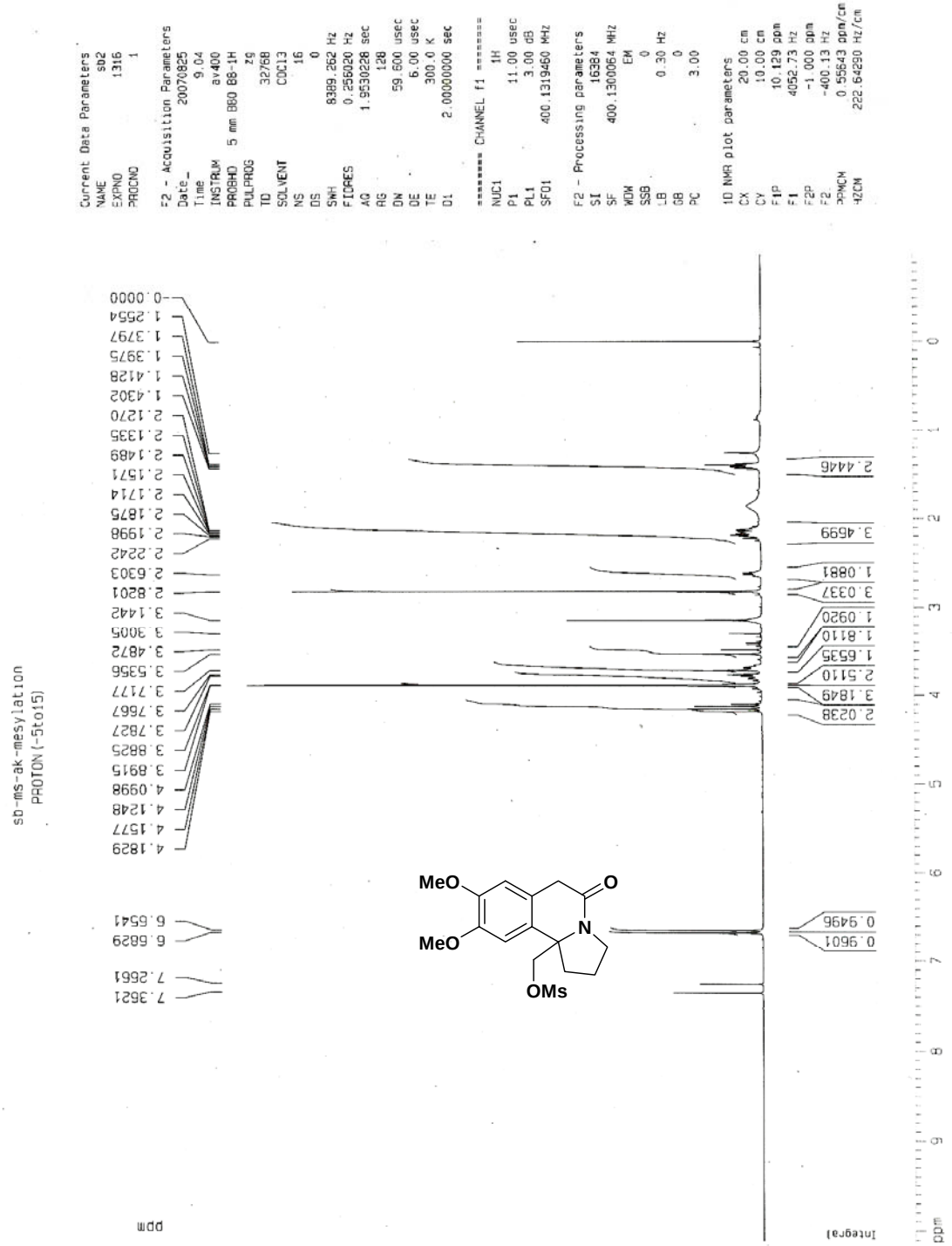
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-3.00 dB
PL12	15.98 dB
PL13	16.00 dB
SFO2	400.1320007 MHz

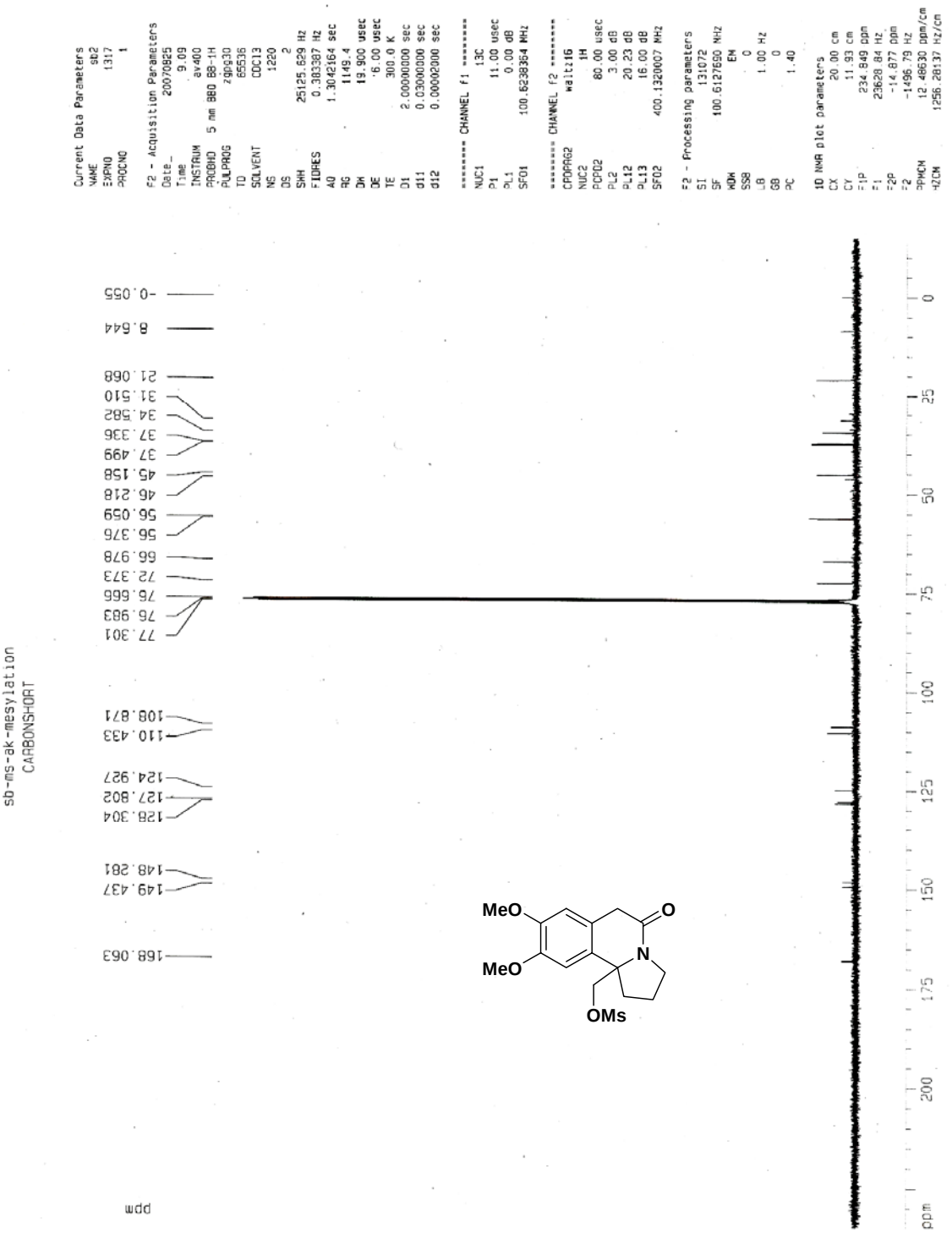
F2 - Processing parameters

SI	131072
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	9.94 cm
F1P	234.849 ppm
F1	23628.84 Hz
F2P	-14.877 ppm
F2	-1496.79 Hz
PPMCM	12.48630 ppm/cm
HZCM	1256.28137 Hz/cm


sb-ms-ak-mesylation
PROTON (-5to15)
Current Data Parameters
NAME sb2
EXPNO 1316
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070825
Time 9.04
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 128
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 3.00 dB
SFO1 400.1319460 MHz
F2 - Processing parameters
SI 16384
SF 400.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 3.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.129 ppm
F1 4052.73 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55643 ppm/cm
HZCM 222.64290 Hz/cm
0.0000
1.2554
1.3797
1.3975
1.4128
1.4302
2.1270
2.1335
2.1489
2.1571
2.1714
2.1875
2.1998
2.2242
2.6303
2.8201
3.1442
3.3005
3.4872
3.5356
3.7177
3.7667
3.7827
3.8825
3.8915
4.0998
4.1248
4.1577
4.1829
6.6541
6.6829
7.2661
7.3621
2.4446
3.4699
1.0881
3.0337
1.0920
1.8110
1.6535
2.5110
3.1849
2.0238
0.9496
0.9601
MeO
MeO
O
N
OMs
ppm
Integral


sb-ms-ak-mesylation
CARBONSHORT
Current Data Parameters
NAME sb2
EXPNO 1317
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070825
Time 9.09
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1220
DS 2
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1149.4
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.23 dB
PL13 16.00 dB
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 131072
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 11.93 cm
F1P 234.849 ppm
F1 23620.84 Hz
F2P -14.877 ppm
F2 -1496.79 Hz
PPMCM 12.49630 ppm/cm
HZCM 1256.28137 Hz/cm
-0.055
8.644
21.068
31.510
34.582
37.336
37.499
45.158
46.218
56.059
56.376
66.978
72.373
76.666
76.983
77.301
108.871
110.433
124.927
127.802
128.304
148.281
149.437
168.063
MeO
MeO
O
N
OMs
ppm
0
25
50
75
100
125
150
175
200