

Supplementary Information

Enantioselective Synthesis of *Anomala Osakana* Pheromone and *Janus integer* Pheromone: A Flexible Approach to Chiral γ -Butyrolactones

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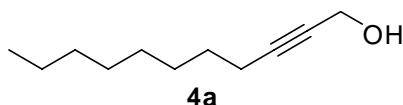
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1. General Methods.

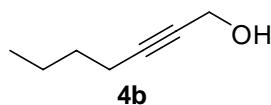
All reactions were carried out under an argon atmosphere and solvents were dried according to established procedures. Reactions were monitored by thin layer chromatography (TLC). Column chromatography purifications were carried out using silica gel. All solvents and reagents were freshly distilled before used. Diethylzinc was prepared from EtI with Zn, and then was diluted with toluene to 1.0 M. ^1H and ^{13}C NMR spectral measurements were measured on a 300-Bruker spectrometer with TMS as an internal standard. Optical rotations were recorded on a Perkin–Elmer 341 polarimeter. The ESI-MS was recorded on a Mariner biomassspectrometer. The ee value determination was carried out using chiral HPLC with a Daicel Chiracel OD-H column on Waters with a 996 UV-detector.

2. General preparation of propargylic alcohol 4:

To a solution of propargyl alcohol (**3**, 1.12 g, 20 mmol) in tetrahydrofuran (THF, 30 ml) and hexamethylphosphoric triamide (HMPA, 8 ml) was added *n*-BuLi (1.5 M in hexane, 26.7 ml, 40 mmol) at -78 °C. After the reaction temperature was warmed to -30 °C, 1-bromoocetane or 1-bromobutane (15 mmol) was added to the mixture and stirred at room temperature overnight. The reaction mixture was treated with aqueous saturated NH₄Cl and extracted with EtOAc. The organic extract was washed with brine and dried over anhy. Na₂SO₄. The solvent was evaporated under vacuum. The residue was purified by silica gel column chromatography (PE : EA = 9:1) to afford **4** as colorless oil.

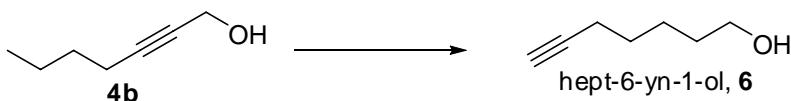


Undec-2-yn-1-ol (4a): 1.76g, 70% yield. ^1H NMR (300 MHz, CDCl₃) δ : 4.25 (2H, t, *J* = 2.1 Hz), 2.21 (2H, tt, *J* = 2.1, 7.2 Hz), 1.67 (1H, bs), 1.51 (2H, m), 1.39-1.27 (10H, m), 0.88 (3H, t, *J* = 6.9 Hz). ^{13}C NMR (75 MHz, CDCl₃) δ : 86.6, 78.2, 51.4, 31.8, 29.2, 29.1, 28.9, 28.6, 22.6, 18.7, 14.1. EI-MS (*m/z*): 168 (M⁺), 111 (M⁺-57), 99 (M⁺-69), 97 (M⁺-71), 85 (M⁺-83), 83 (M⁺-85), 71 (M⁺-97), 69 (M⁺-99).



Hept-2-yn-1-ol, 4b: 1.19g, 71% yield. ^1H NMR (300 MHz, CDCl₃) δ : 4.25 (2H, s), 2.22 (2H, tt, *J* = 2, 7 Hz), 1.69 (1H, br), 1.55-1.35 (4H, m), 0.91 (3H, t, *J* = 7.2 Hz). ^{13}C NMR (75 MHz, CDCl₃) δ : 86.6, 78.2, 51.4, 30.6, 21.9, 18.4, 13.5. EI-MS (*m/z*): 112 (M⁺), 111 (M⁺-1), 95 (M⁺-17), 81 (M⁺-31), 67 (M⁺-45).

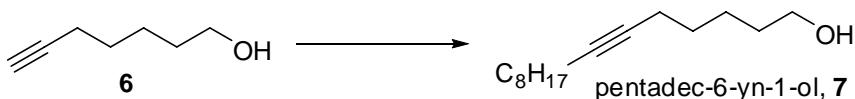
3. Zipper isomerization for the preparation of 6:



The mixture of Li (0.56 g, 80 mmol) in 1,3-diaminopropane (30 ml) was stirred and heated in an

oil bath at 70°C until the blue color discharges (1 h), affording a white suspension of the lithium amide. After cooling to room temperature, potassium tert-butoxide (5.3 g, 48 mmol,) was added to the mixture. The resultant pale yellow solution is stirred for 20 min at room temperature, and then **4b** (0.9 g, 8 mmol) was added. Residual **4b** was washed into the mixture with small portion of 1,3-diaminopropane. After stirring at room temperature for 3h, the reaction mixture was poured into plenty of ice-water and extracted with Et₂O for three times. The organic phase was combined and washed with 5% aqueous HCl and brine, then dried over anhy. Na₂SO₄. The solvent was evaporated under vacuum. The residue was purified by silica gel column chromatography (PE : EA = 7:1) to afford **6** (0.82 g, 90%) as colorless oil. ¹H NMR (300 MHz, CDCl₃) δ: 3.65 (2H, t, *J*= 6 Hz), 2.21 (2H, td, *J*= 3, 6.9 Hz), 1.96 (1H, t, *J*= 3 Hz), 1.64-1.55 (5H, m), 1.53-1.45 (2H, m). ¹³C NMR (75 MHz, CDCl₃) δ: 84.4, 68.3, 62.7, 32.1, 28.1, 24.8, 18.3. EI-MS (*m/z*): 112 (M⁺), 111 (M⁺-1), 97 (M⁺-15), 95 (M⁺-17), 83 (M⁺-29), 81 (M⁺-31), 69 (M⁺-43), 57 (M⁺-55), 55 (M⁺-57).

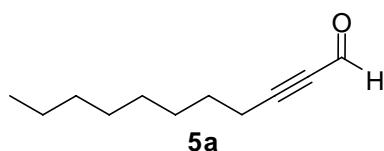
4. Coupling reaction of **6** with 1-bromooctane for the preparation of **7**:



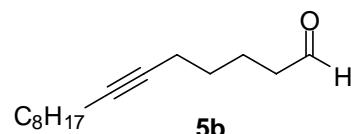
The ratio of **6**, 1-bromooctane and *n*-BuLi was 1:1:1. Using a similar procedure as the preparation of **4** afforded **7** in 90% yield base on 40% of the recovered **6**. ¹H NMR (300 MHz, CDCl₃) δ: 3.65 (2H, t, *J*= 6 Hz), 2.19-2.10 (4H, m), 1.63-1.54 (2H, m), 1.52-1.42 (6H, m), 1.40-1.33 (3H, m), 1.27 (8H, m), 0.88 (3H, m). ¹³C NMR (75 MHz, CDCl₃) δ: 80.5, 79.8, 62.9, 32.3, 31.8, 29.2, 29.1, 28.7, 24.9, 24.1, 22.6, 18.71, 18.70, 14.1. ESI-MS: [M+Na]⁺ 247.2.

5. Swern oxidation for the preparation of **5**:

Oxalyl chloride (0.52 ml, 6 mmol, 1.2 equiv) was added to dry DCM (20 mL) and cooled to -60°C under Ar. Dry DMSO (0.85 ml, 12 mmol, 2.4 equiv) was then added to the oxalyl chloride solution in drop wise. After stirring for 5mins, the solution of **4a** or **7** in 10 ml DCM was added to the mixture in 1min. The resulting cloudy white mixture was stirred for 15 min at -60°C. TEA 3.5 mL, 25 mol, 5.0 equiv) was added and the solution was stirred for 15 min at -60°C and 10 min at 0°C. Ice-water was added to quench the reaction and extracted with DCM for three times. The organic phase was combined and washed with 5% aqueous HCl and brine, then dried over anhy. Na₂SO₄. The solvent was evaporated under vacuum. The residue was purified by silica gel column chromatography (PE : EA = 15:1) to afford **5**.



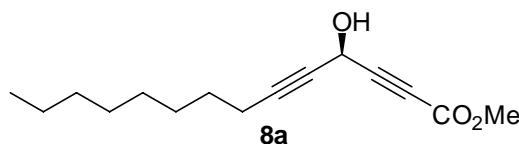
Undec-2-yneal (5a): yellow oil, 0.63g, 75% yield. ¹H NMR (300 MHz, CDCl₃) δ: 9.18 (1H, s), 2.41 (2H, t, *J* = 7.2 Hz), 1.65-1.55 (2H, m), 1.43-1.36 (2H, m), 1.28 (8H, m), 0.89 (3H, t, *J* = 6.9 Hz). ¹³C NMR (75 MHz, CDCl₃) δ: 177.3, 99.4, 81.7, 31.8, 29.1, 29.0, 28.8, 27.5, 22.6, 19.1, 14.1. EI-MS (*m/z*): 166 (M⁺), 165 (M⁺-1), 137 (M⁺-29), 123 (M⁺-43), 109 (M⁺-67), 95 (M⁺-71), 81 (M⁺-85), 71 (M⁺-95), 67 (M⁺-99).



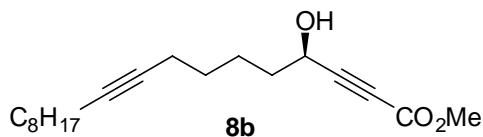
Pentadec-6-yneal (5b): colorless oil, 0.81 g, 73% yield. ^1H NMR (300 MHz, CDCl_3) δ : 9.78 (1H, t, J = 2 Hz), 2.46 (2H, td, J = 2, 7.2 Hz), 2.22-2.10 (4H, m) 1.75 (2H, q, J = 7.5 Hz), 1.57-1.42 (4H, m), 1.38-1.33 (2H, m), 1.27 (8H, m), 0.88 (3H, t, J = 6.9 Hz). ^{13}C NMR (75 MHz, CDCl_3) δ : 202.5, 80.9, 79.2, 43.4, 31.8, 29.2, 29.1, 28.9, 28.4, 22.6, 21.2, 18.7, 18.5, 14.1. ESI-MS: $[\text{M}+\text{Na}]^+$ 245.2.

6. General procedure for the asymmetric addition of methyl propiolate to 5:

To a stirred solution of **L*** (21.6m g, 0.06 mmol, 30 mol %), DME (20.7 μL , 0.2 mmol) and Et_2Zn (0.4 mL, 0.4 mmol) in toluene (2 mL) under Ar, methyl propiolate (35.7 μL , 0.2 mmol) was added in one portion. After the solution has been stirred at 25°C overnight, $\text{Ti(O}^{\text{i}}\text{Pr})_4$ (17.5 μL , 0.06 mmol, 30mol%) was added and stirred for 1h. Then, the aldehyde **5** (0.2 mmol) was added in one portion at 0°C. Ammonium chloride (saturated aq.) was added to quench the reaction until the reaction was complete (monitoring with TLC), and the mixture was extracted with methylene chloride and dried with sodium sulfate. After column chromatography on silica gel eluted with 10% ethyl acetate in hexanes, the optically active γ -hydroxy- α,β -acetylenic ester **8** was isolated. The enantiomeric purity of the product was determined by using HPLC.



(R)-Methyl 4-hydroxytetradeca-2, 5-dynoate (8a): colorless oil, 80% yield. 84% ee was determined by HPLC analysis (OD column, 1.0mL/min, 3% i-PrOH in hexane); retention times: 17.5 min (major) and 19.4 min (minor). $[\alpha]^{20}_D = -4$ ($c = 1.31, \text{CHCl}_3$). ^1H NMR (300 MHz, CDCl_3) δ : 5.21 (1H, bs), 3.80 (3H, s), 2.64 (1H, bs), 2.23 (2H, td, J = 2.1, 7.2 Hz), 1.57-1.47 (2H, m), 1.41-1.32 (2H, m), 1.27 (8H, m), 0.88 (3H, t, J = 6.9 Hz). ^{13}C NMR (75 MHz, CDCl_3) δ : 153.6, 87.5, 84.1, 75.2, 74.6, 52.9, 52.1, 31.8, 29.1, 29.0, 28.8, 28.1, 22.6, 18.6, 14.0. ESI-MS: $[\text{M}+\text{Na}]^+$ 273.1.



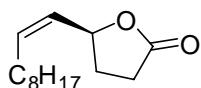
(R)-Methyl 4-hydroxyoctadeca-2, 9-dynoate (8b) colorless oil, 78% yield, 84% ee was determined by HPLC analysis (OD column, 1.0mL/min, 3% i-PrOH in hexane); retention times: 14.8 min (major) and 19.1 min (minor). $[\alpha]^{20}_D = -9$ ($c = 1.27, \text{CHCl}_3$). ^1H NMR (300 MHz, CDCl_3) δ : 4.50 (1H, t, J = 6 Hz), 3.79 (3H, s), 2.20-2.10 (4H, m), 2.01 (1H, bs), 1.83-1.75 (2H, m), 1.63-1.53 (4H, m), 1.51-1.43 (2H, m), 1.38-1.33 (2H, m), 1.27 (8H, m), 0.88 (3H, t, J = 6.9 Hz). ^{13}C NMR (75 MHz, CDCl_3) δ : 153.7, 88.0, 79.5, 76.3, 62.0, 52.8, 36.4, 31.8, 29.2, 29.1, 28.9, 28.6, 24.1, 22.6, 18.7, 18.6, 14.1. ESI-MS: $[\text{M}+\text{Na}]^+$ 329.2.

7. General procedure for the conversion of **8** to (4S, 5Z)-**1** or (4R, 9Z)-**2**:

Lindlar's Pd catalyst (5 wt.-% on BaSO₄, 20 mg) was added to a solution of **8** (0.2 mmol) in hexane (10 mL). The suspension was vigorously stirred under H₂ (1 atm) for 2h until the reaction completed (monitored by ¹H NMR). The catalyst was then removed by filtration through a small portion of Celite®, and washed with hexane. The hexane solution was concentrated in vacuo and used for the next step without purification.

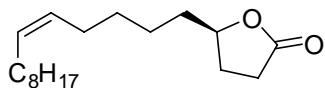
CuCl (21.9 mg, 1.1 equiv relative to the amount of **8** in the first) was added to the stirred solution of the above residue in 5 ml of MeOH. After cooling to 0°C. NaBH₄ (38 mg, 5 equiv) was added to the mixture in small portions until the reduction completed (monitored by TLC, 0.5 h). 1 ml 2M HCl was then added to quench the reaction. The mixture was extracted with EtOAc (5 ml × 3) and washed with brine, then dried over anhy. Na₂SO₄. The solvent was evaporated under vacuum to afford **10**.

The residue obtained from the above step was added 10 ml benzene and 3mg TsOH • H₂O. The mixture was heated at reflux for 2h. After the lactonization completed, benzene was evaporated under vacuum and diluted with Et₂O. The organic phase was washed with 5% aqueous NaHCO₃, 5% aqueous HCl and brine, then dried over anhy. Na₂SO₄. The solvent was evaporated under vacuum. The residue was purified by silica gel column chromatography (PE : EA = 9:1), and afforded (4S, 5Z)-**1** or (4R, 9Z)-**2** as both colorless oil.



(4S, 5Z)-**1**, *Anomala Osakana* Pheromone

(4S, 5Z)-**1**: 87 % overall yield. $[\alpha]^{20}_D = +49$ ($c = 1.14$, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ: 5.67 (1H, dtd, $J = 0.9, 7.5, 10.8$ Hz), 5.46 (1H, tt, $J = 1.5, 10.8$ Hz), 5.25 (1H, dtd, $J = 0.9, 8.4, 6.6$ Hz), 2.60-2.54 (2H, m), 2.43-2.33 (1H, m), 2.19-2.04 (2H, m), 2.01-1.88 (1H, m), 1.39-1.35 (1H, m), 1.39-1.26 (1H, m), 0.88 (3H, t, $J = 6.9$ Hz). ¹³C NMR (75 MHz, CDCl₃) δ: 177.2, 135.9, 127.2, 76.4, 31.8, 29.4, 29.3, 29.2, 29.0, 27.8, 22.6, 14.1. HRMS (ESI): calcd. for C₁₄H₂₄O₂ [M+Na]⁺ 247.1699, found 247.1700, error = -0.4 ppm. Lit.¹: $[\alpha]^{26}_D = +70.5$ ($c = 5.5$, CHCl₃). ¹H NMR (CDCl₃): δ: 0.85 (t, $J = 6.8$ Hz, 3H), 1.20-1.30 (12H, m), 2.02-2.14 (2H, m), 2.31-2.39 (1H, m), 2.49-2.56 (2H, m), 5.22 (1H, tdd, $J = 8.3, 6.6, 1.0$ Hz), 5.42 (1H, ddt, $J = 10.7, 8.6, 1.5$ Hz), 5.64 (1H, dtd, $J = 10.9, 7.7, 1.0$ Hz). ¹³C NMR (CDCl₃) δ: 14.48, 23.05, 28.23, 29.41, 29.60, 29.63, 29.71, 29.80, 29.82, 32.26, 76.83, 127.64, 136.25, 177.52.

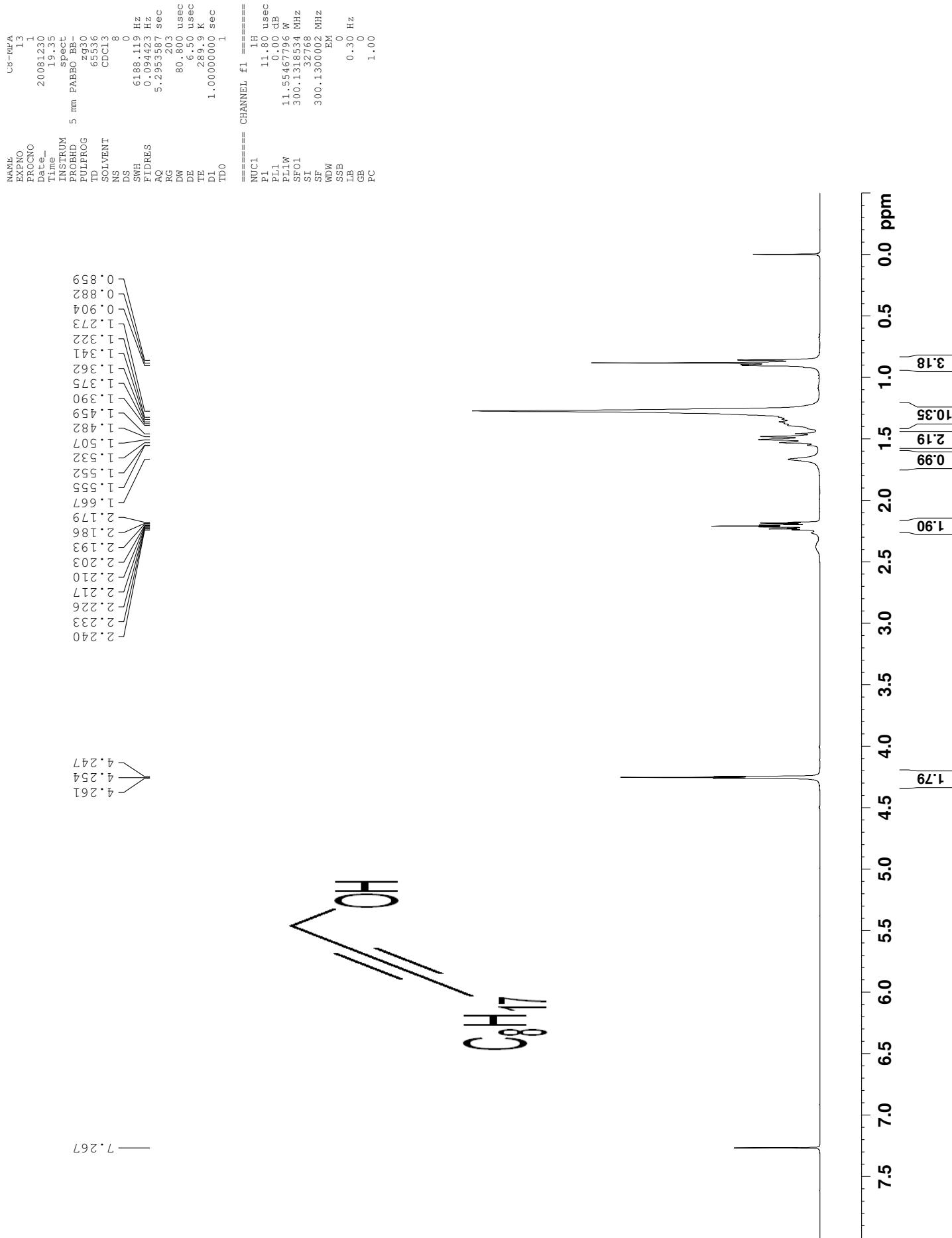


(4R, 9Z)-**2**, *Janus integer* Pheromone

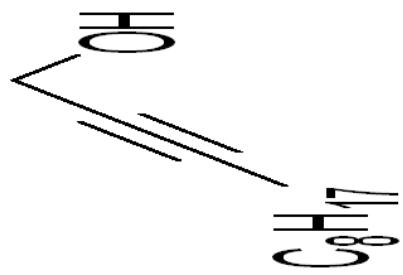
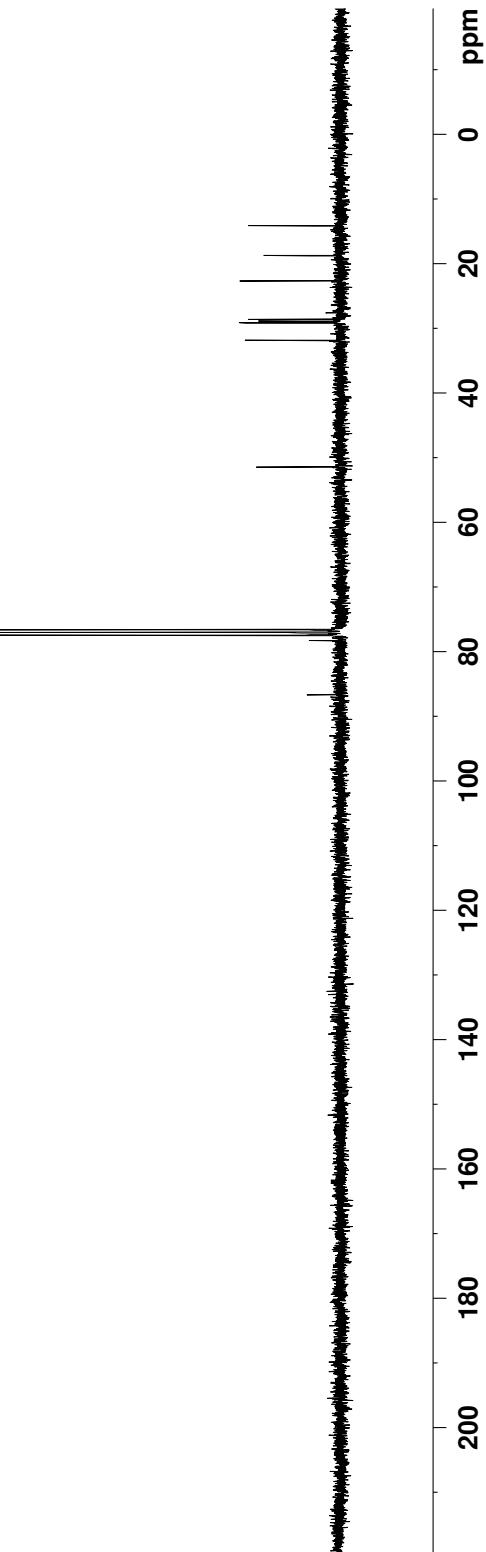
(4R, 9Z)-**2**: 89% overall yield. $[\alpha]^{20}_D = +13$ ($c = 1.19$, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ: 5.42-5.28 (2H, m), 4.53-4.44 (1H, m), 2.56-2.51 (2H, dd, $J = 6.9, 9.6$ Hz), 2.38-2.27 (1H, m), 2.08-1.98 (4H, m), 1.92-1.81 (1H, m), 1.80-1.69 (1H, m), 1.67-1.55 (1H, m), 1.53-1.37 (4H, m), 1.36-1.27 (13H, m), 0.88 (3H, t, $J = 6.9$ Hz). ¹³C NMR (75 MHz, CDCl₃) δ: 177.2, 130.4, 129.1,

(1) (a) J. H. Tumlinson, M. G. Klein, R. E. Doolittle, T. L. Ladd, A. T. Proveaux, *Science* **1977**, *197*, 789; (b) A. A. Dos Santos, W. Francke, *Tetrahedron: Asymmetry* **2006**, *17*, 2487

80.9, 35.5, 31.9, 29.7, 29.5, 29.4, 29.3, 28.8, 28.0, 27.2, 26.9, 24.8, 22.6, 14.1. HRMS (ESI): calcd. for C₁₈H₃₀O₂ [M+Na]⁺ 303.2295, found 303.2291, error = 1.3 ppm. Lit.:² [α]²⁶_D = +24 (c = 0.50, CDCl₃). ¹H NMR (500 MHz, CDCl₃): δ : 0.88 (t, J = 7.0 Hz, 3H), 1.27-1.76 (m, 19 H), 1.81-1.89 (m, 1 H), 1.99-2.06 (m, 4 H), 2.32 (ddt, J = 6.5, 7.5, 13 Hz, 1 H), 2.53 (ddd, J = 1.3, 7.5, 9.2 Hz, 2 H), 4.48 (quint, J = 6.5 Hz, 1 H), 5.35 (dtt, J = 7.1, 12, 18 Hz, 2 H) ppm. ¹³C NMR (100 MHz, CDCl₃): δ : 14.10, 14.11, 22.7, 24.9, 27.0, 27.2, 28.0, 28.9, 29.3, 29.4, 29.5, 29.7, 31.9, 35.5, 81.0, 129.1, 130.5, 177.3.



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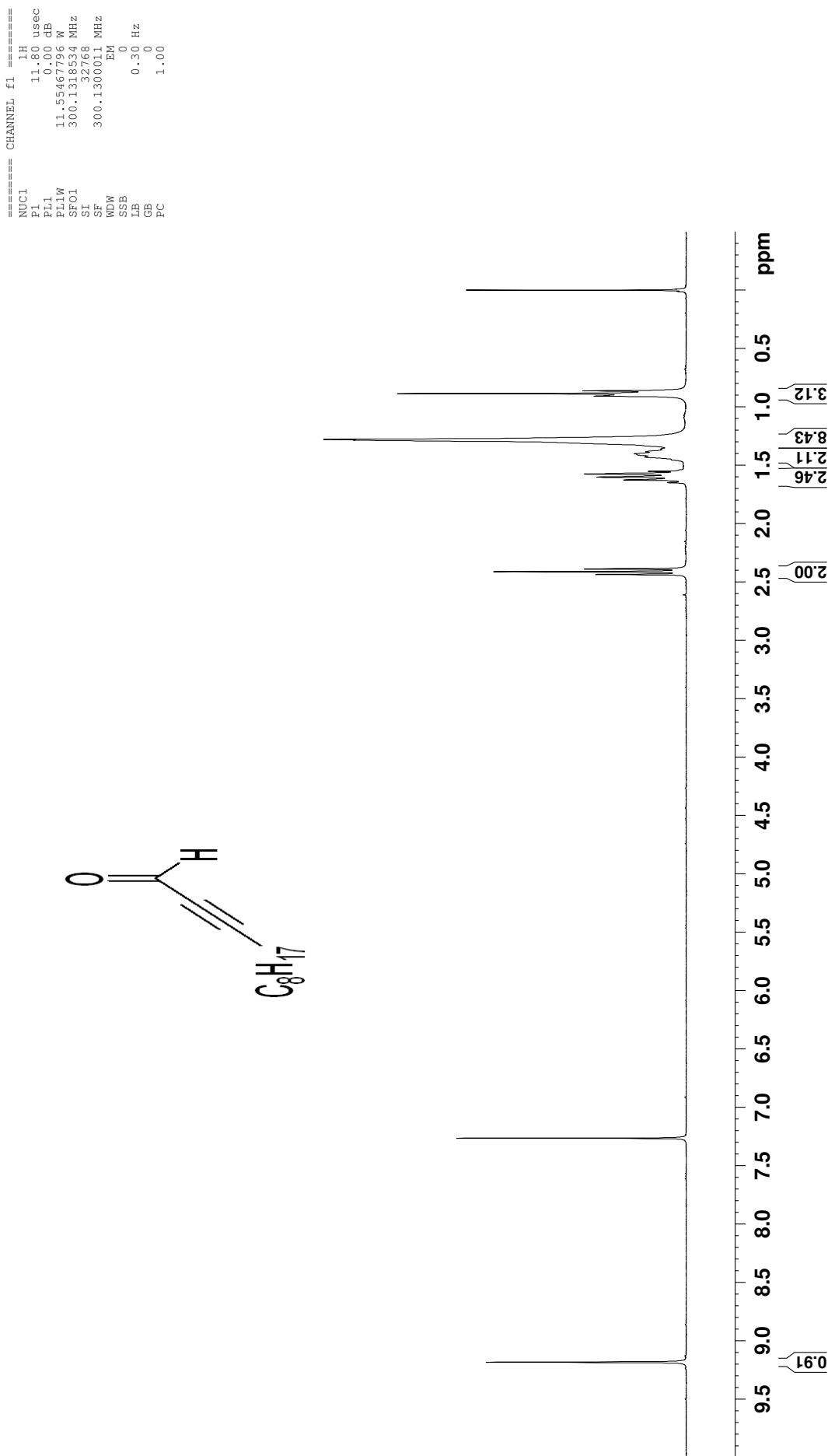
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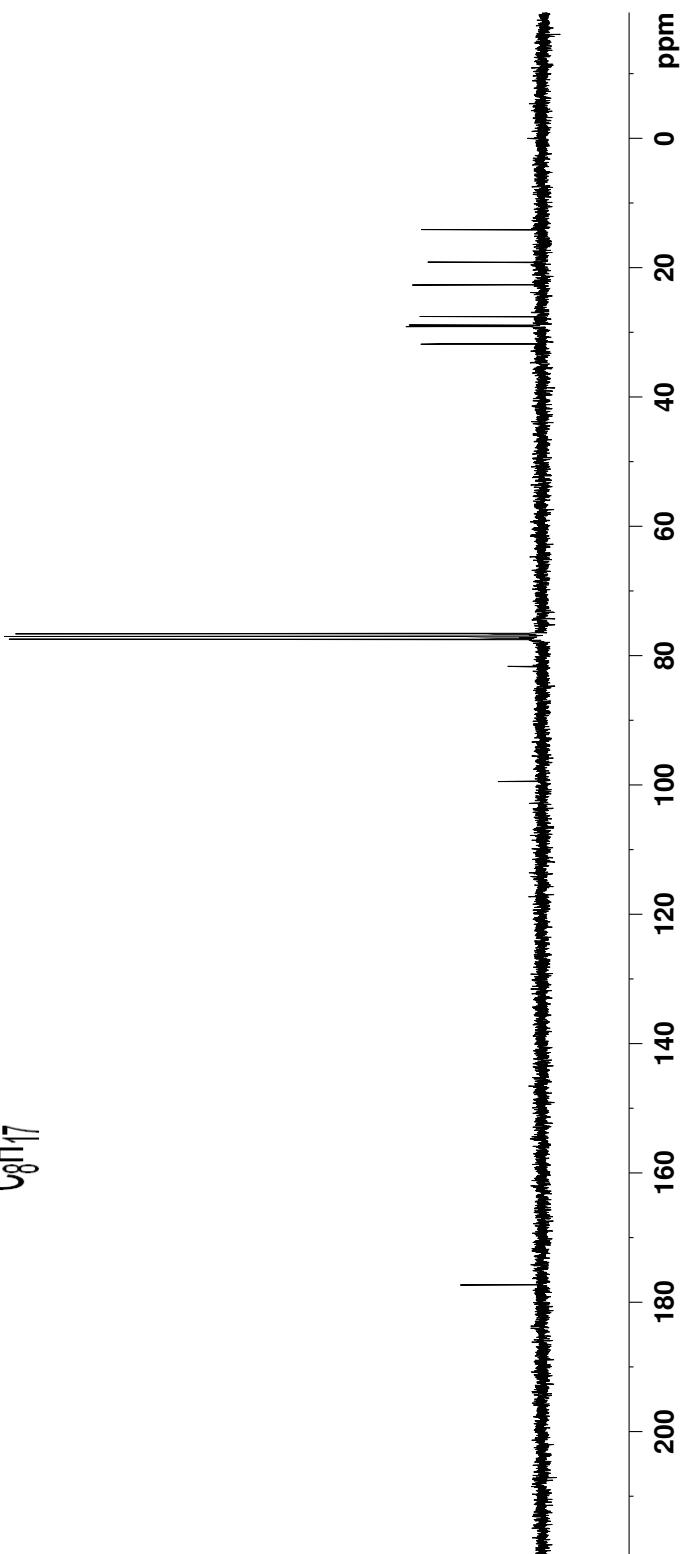
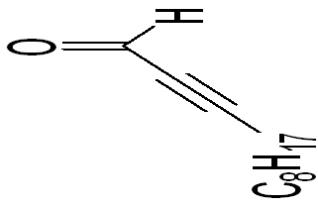
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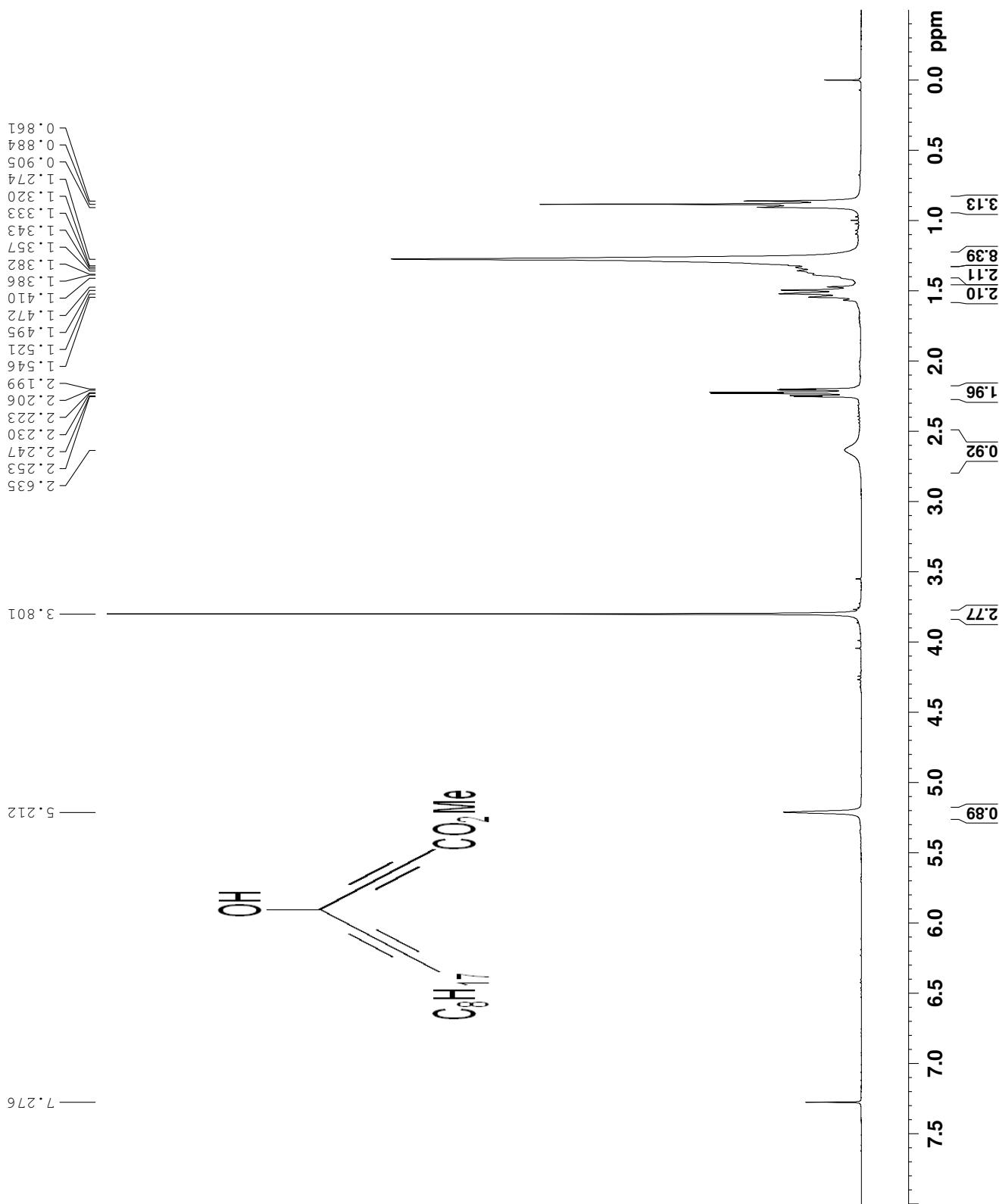


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Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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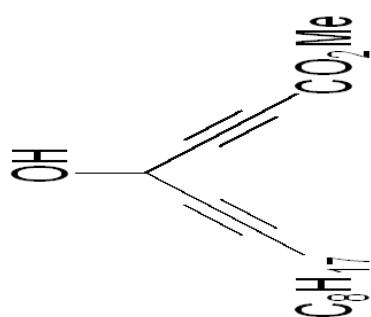
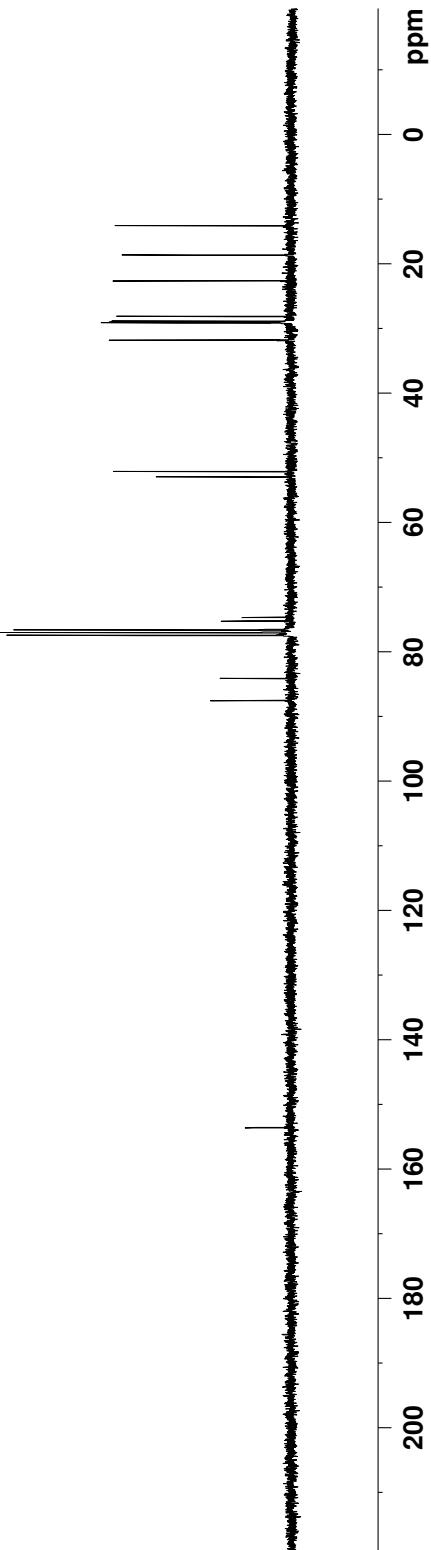
```

NAME          0.81230
EXENO         4
PROCNO       1
Date_        20081230
Time_        17.31
INSTRUM      spect
PROBHD      5 mm PABBO BB-
PULPROG     zgpp36
TD           65536
SOLVENT      CDCl3
NS            105
DS            4
SWH          18028.816 Hz
FIDRES      0.250988 Hz
AQ           1.8175818 sec
RG           27.733
DW           6.50 usec
DE           290.7 K
TE           2.0000000 sec
D1           0.03000000 sec
D11          1
TD0          1

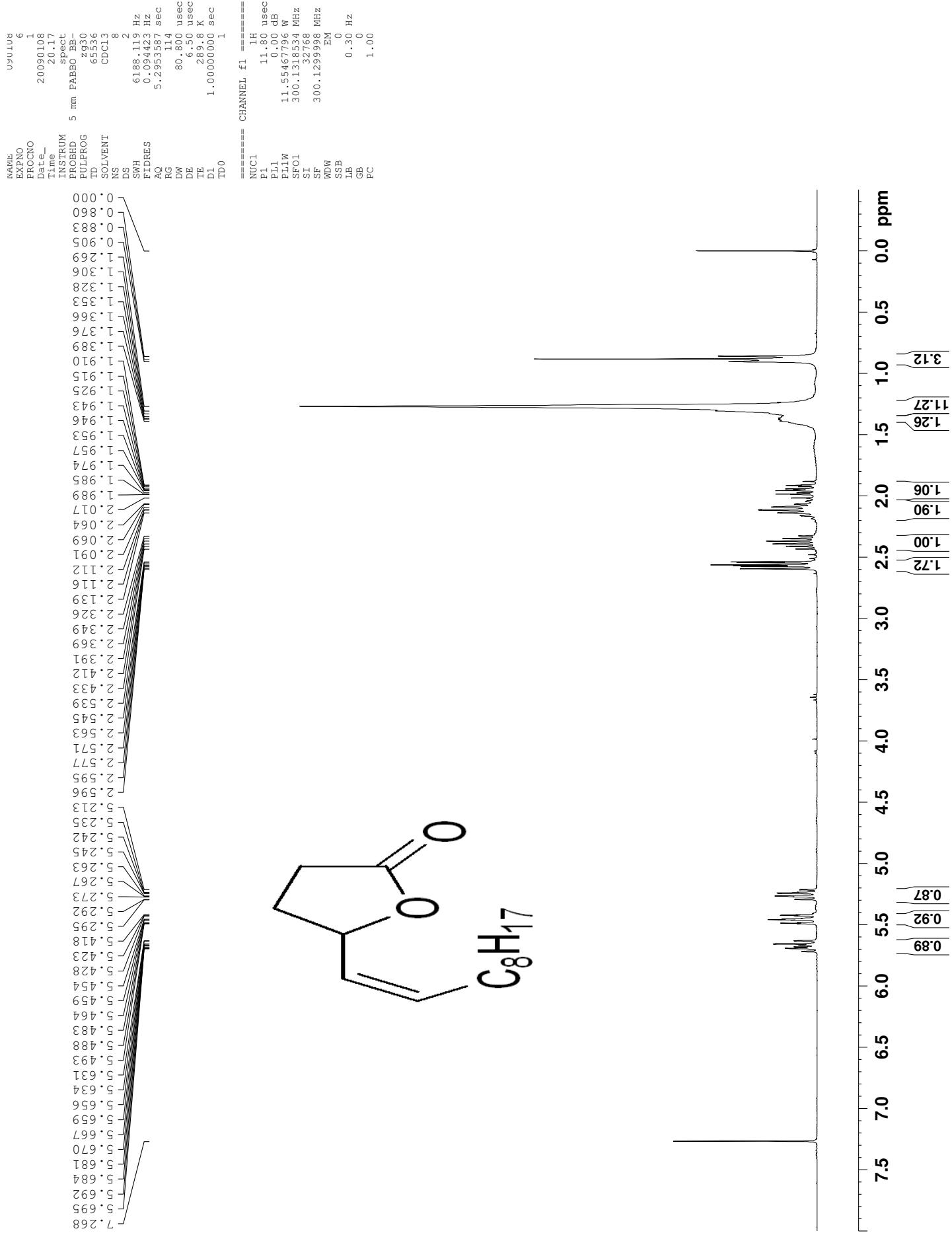
===== CHANNEL f1 =====
NUC1        13C
P1           9.70 usec
PL1          0.00 dB
PL1W         29.38907051 W
SF01         75.4732933 MHz

===== CHANNEL f2 =====
CPDPGR2    wait z16
NUC2        1H
PCPD2       80.00 usec
PL2          1.00 dB
PL12         17.00 dB
PL13         17.00 dB
PL2W         9.17820644 W
PL12W        0.23034613 W
PL13W        0.23034613 W
SF02         300.1312005 MHz
SI           75.467754 MHz
SF           EM
WDW          0
SSB          1.00 Hz
LB           0
GB           1.40
PC           1.40

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153.58
 87.55
 84.09
 77.42
 77.00
 76.58
 75.21
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 52.92
 52.07
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 28.91
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 22.60
 22.05
 18.62
 14.05



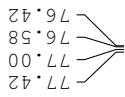
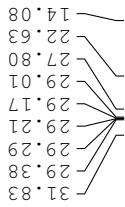
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NAME          0.90108
EXENO         7
PROCNO       1
Date_        20090108
Time_        20.38
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG     zgpg36
TD           65536
SOLVENT      CDCl3
NS            144
DS            4
SWH          18028.816 Hz
FIDRES       0.225098 Hz
AQ           1.8175818 sec
RG           27.203
DW           27.733 usec
DE           6.50 usec
TE           290.5 K
D1           2.0000000 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1             9.70 usec
PL1           0.00 dB
PL1W          29.38907051 W
SF01          75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           1.00 dB
PL12          17.00 dB
PL13          17.00 dB
PL2W          9.17820644 W
PL12W         0.23034613 W
PL13W         0.23034613 W
SF02          300.1312005 MHz
SI             75.467753 MHz
SF             EM
WDW           0
SSB           1.00 Hz
LB             0
GB             1.40
PC             1.40

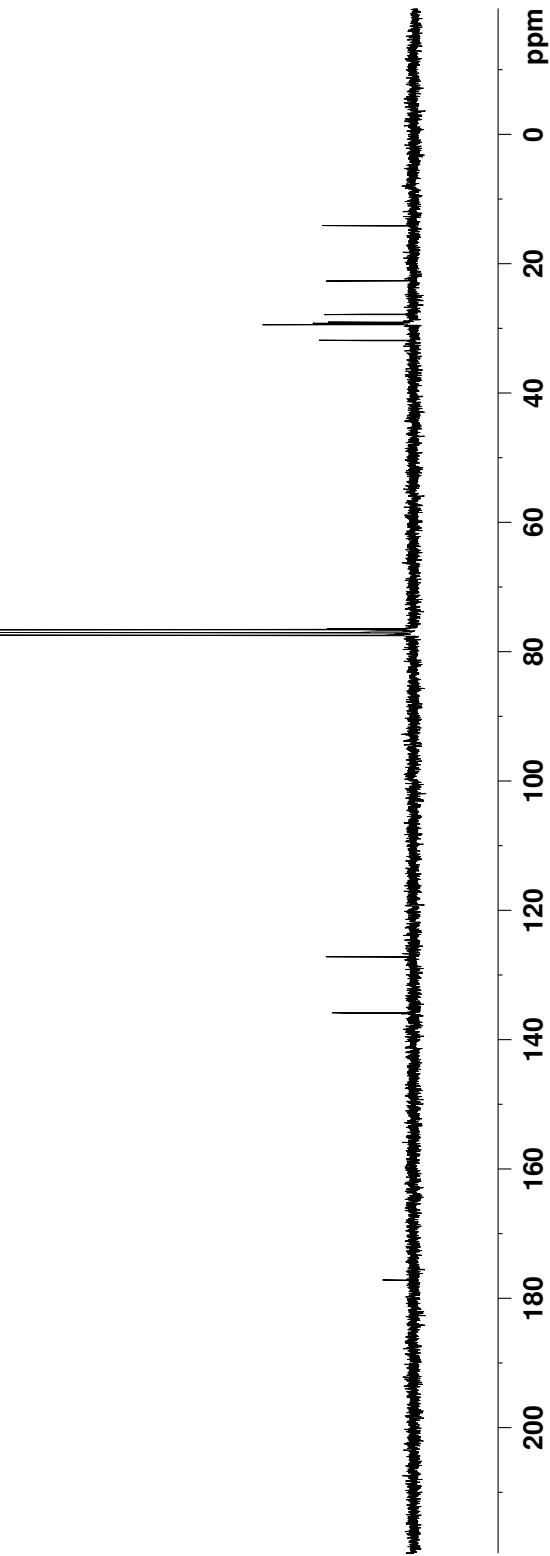
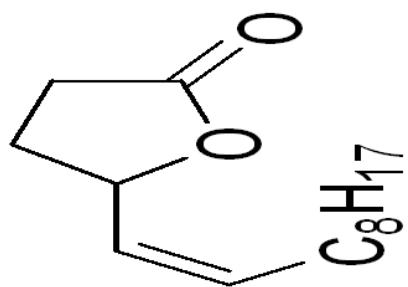
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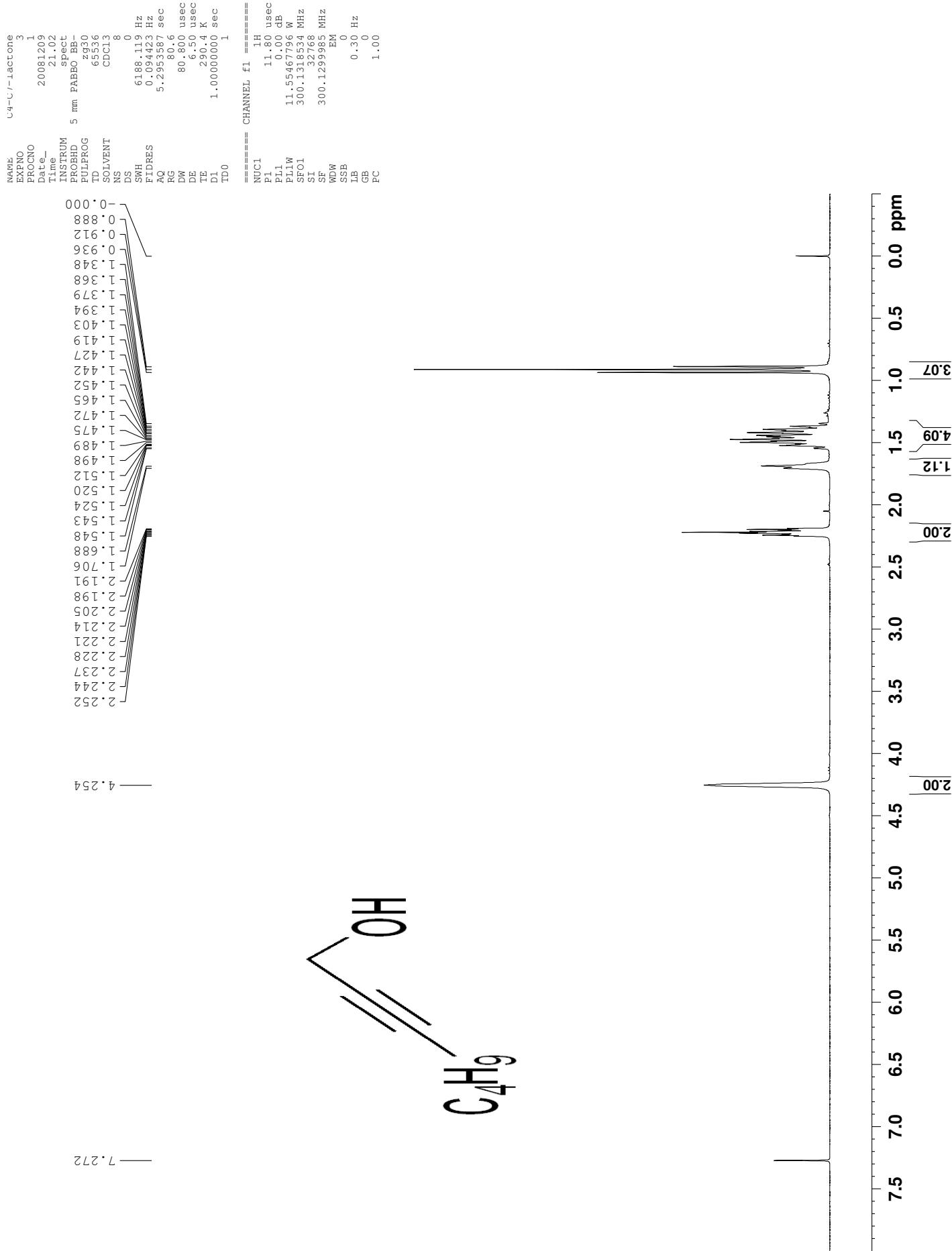


127.16

135.86

177.17





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```

NAME          C4-C7-lactone
EXENO         4
PROCNO       1
Date_        20081209
Time_        21.09
INSTRUM      spect
PROBHD      5 mm PABBO BB-
PULPROG     zgpg36
TD           65536
SOLVENT      CDC13
NS            38
DS            0
SWH          18028.816 Hz
FIDRES      0.25098 Hz
AQ           1.8175818 sec
RG           27.733 usec
DW           6.500 usec
DE           290.7 K
TE           2.0000000 sec
D1           0.03000000 sec
D11          1
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1             9.70 usec
PL1          0.00 dB
PL1W         29.38907051 W
SF01         75.4732933 MHz

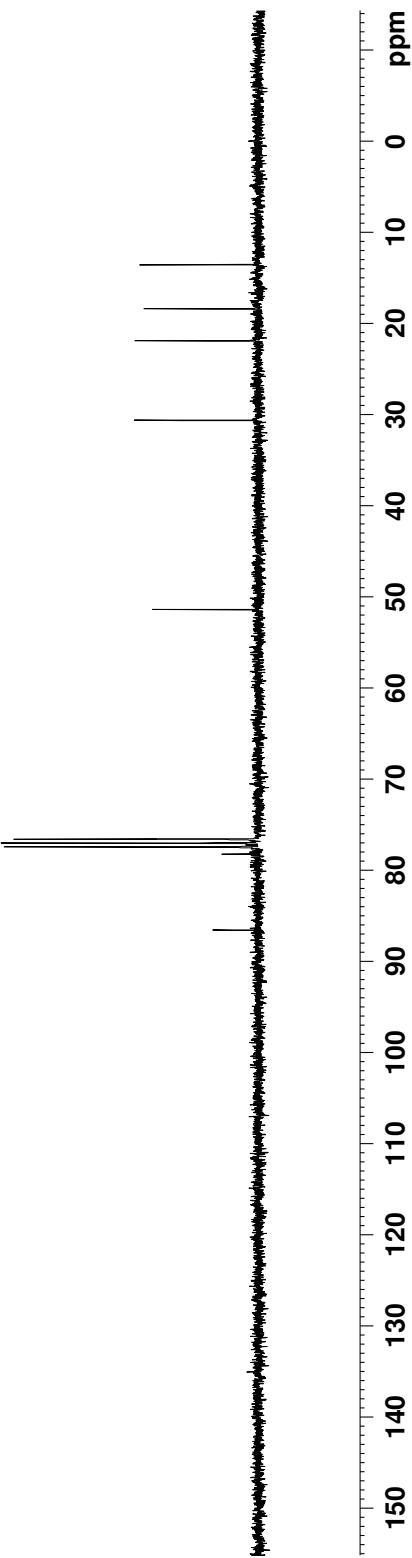
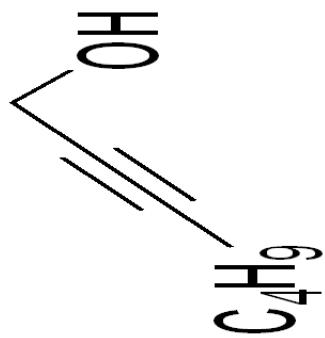
===== CHANNEL f2 =====
CPDPGR22    waltz16
NUC2          1H
PCPD2        80.00 usec
PL2          1.00 dB
PL12         17.00 dB
PL13         17.00 dB
PL2W         9.17820644 W
PL12W        0.23034613 W
PL13W        0.23034613 W
SF02         300.1312005 MHz
SI             32.768
SF             EM
WDW           75.4677539 MHz
SSB           0
LB             1.00 Hz
GB             0
PC             1.40

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— 13.54
 — 18.37
 — 21.88
 — 30.60

— 51.37

— 76.57
 — 77.00
 — 77.42
 — 78.21
 — 86.56



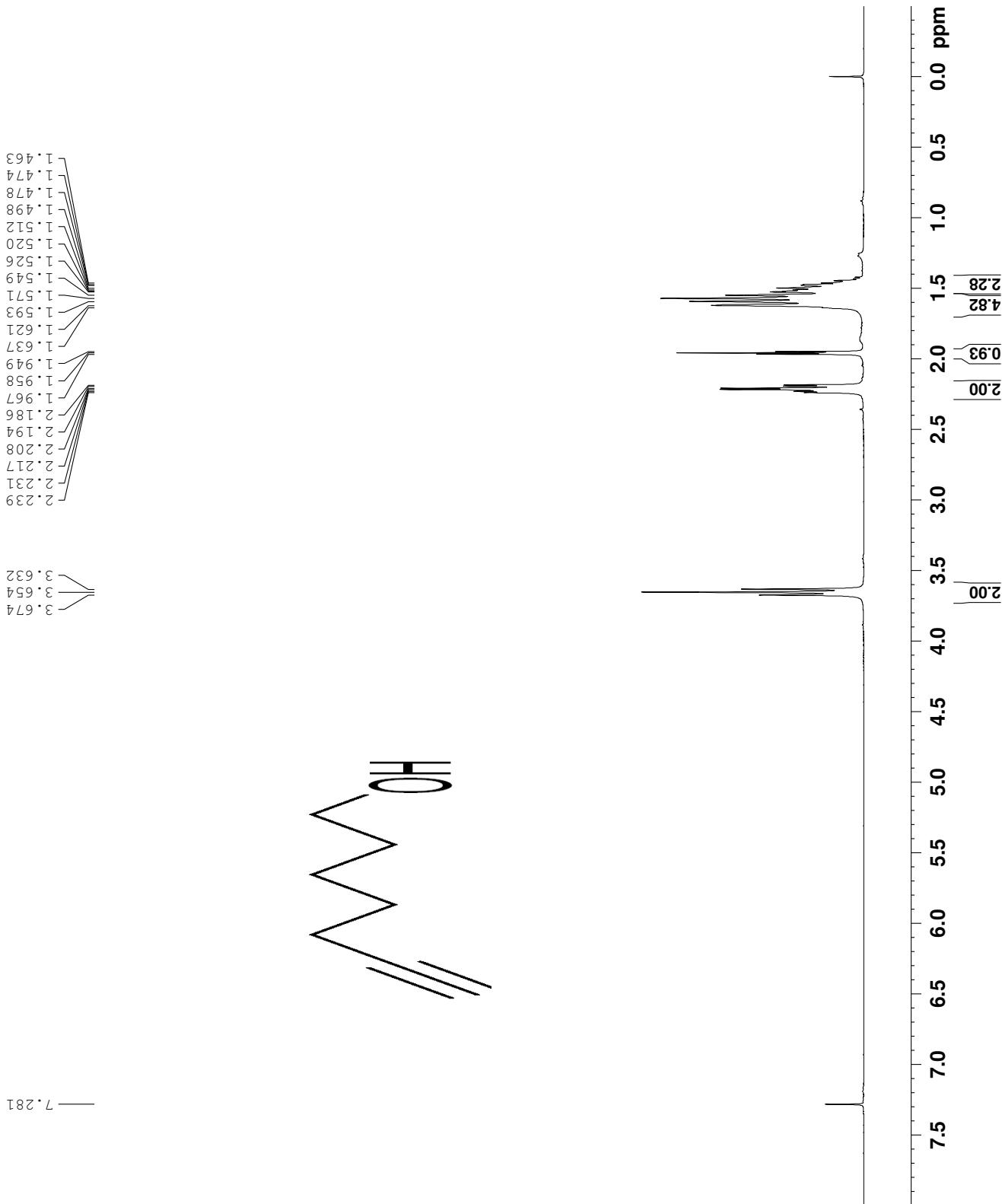
Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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```

NAME: EXPNO: 10
PROCNO: 1
Date: 20081219
Time: 10.47
INSTRUM: spect
PROBOD: 5 mm PABBO BB
PULPROG: zg30
TD: 65536
SOLVENT: NS
NS: 8
DS: 0
SWH: 61.88,11.9 Hz
FIDRES: 0.034423 Hz
AQ: 5.293587 sec
RG: 57
DW: 80.800 usec
DE: 6.50 usec
TE: 28.97 K
D1: 1.0000000 sec
TDO: 1

=====
CHANNEL f1 =====
NUC1: 1H
P1: 11.80 usec
PL1: 0.00 dB
PL1W: 11.5546796 W
SFO1: 300.1318534 MHz
SI: 300.1327662 MHz
SF: 300.12939962 MHz
WDW: EM
SSB: 0
LB: 0.34 Hz
GB: 0
PC: 1.00

```



Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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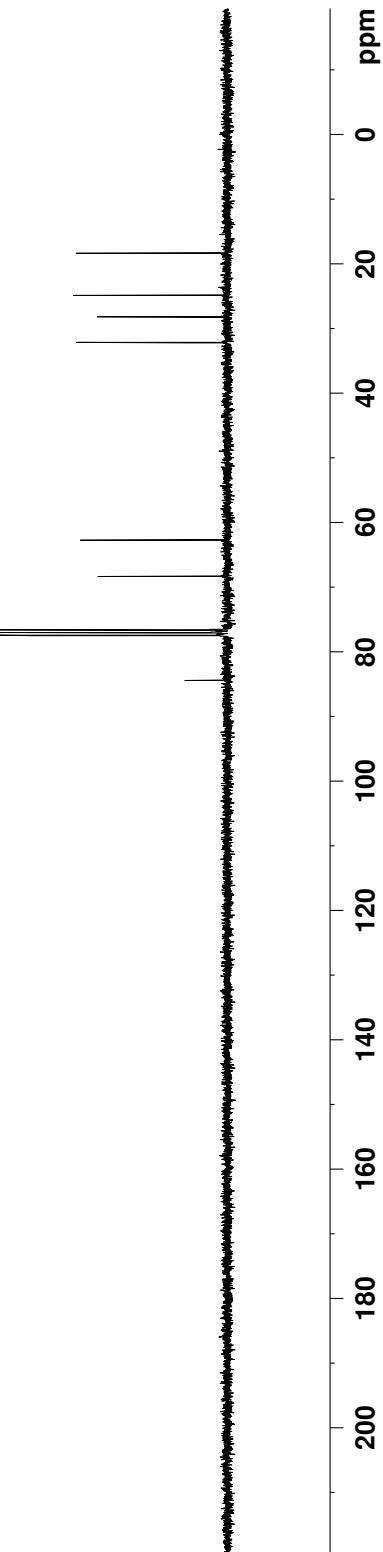
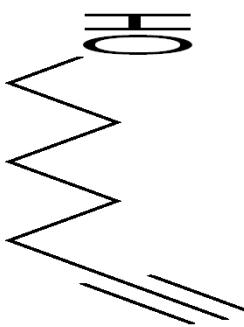
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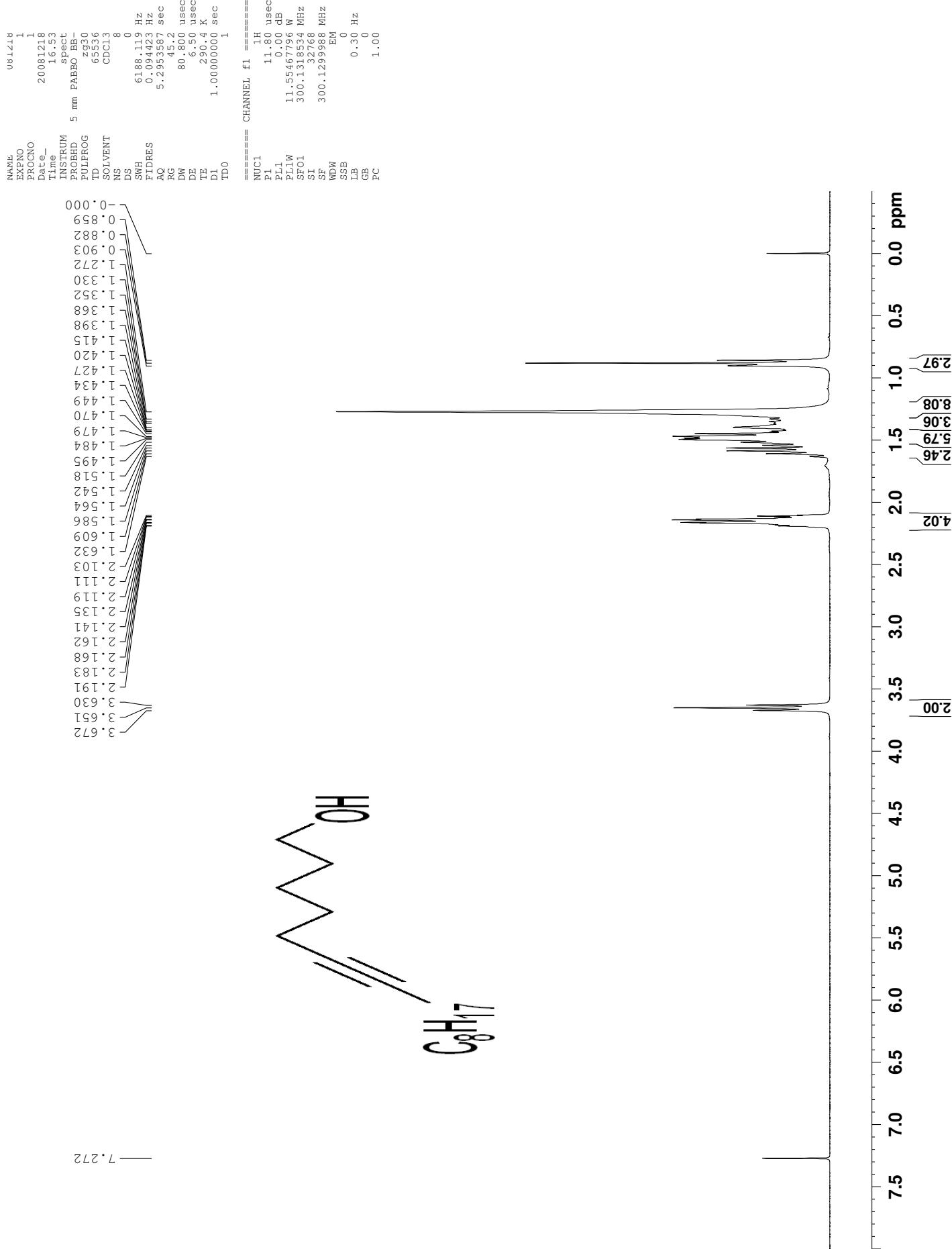
NAME          C4-C7-lactone
EXPTNO.      1
PROCNO.      1
Date_        20081219
Time_        10.47
INSTRUM.     spect
PROBHD.      5 mm PABBO BB-
PULPROG.    zgpg36
TD.          65536
SOLVENT.    CDCl3
NS.          40
DS.          0
SWH.         18028.816 Hz
FIDRES.     0.225098 Hz
AQ.          1.8175818 sec
RG.          27.733 usec
DW.          6.500 usec
DE.          289.6 K
TE.          2.0000000 sec
D1.          0.03000000 sec
D11.         1
TD0.         1

===== CHANNEL f1 =====
NUC1          13C
P1.          9.70 usec
PL1.         0.00 dB
PL1W.        29.38907051 W
SF01.        75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2.    waltz16
NUC2          1H
PCPD2.       80.00 usec
PL2.          1.00 dB
PL12.         17.00 dB
PL13.         17.00 dB
PL2W.        9.17820644 W
PL12W.       0.23034613 W
PL13W.       0.23034613 W
SF02.        300.1312005 MHz
SI.           75.4677520 MHz
SF.            EM
WDW.          0
SSB.          1.00 Hz
LB.            0
GB.          1.40
PC.          1.40

```

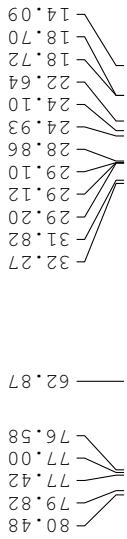




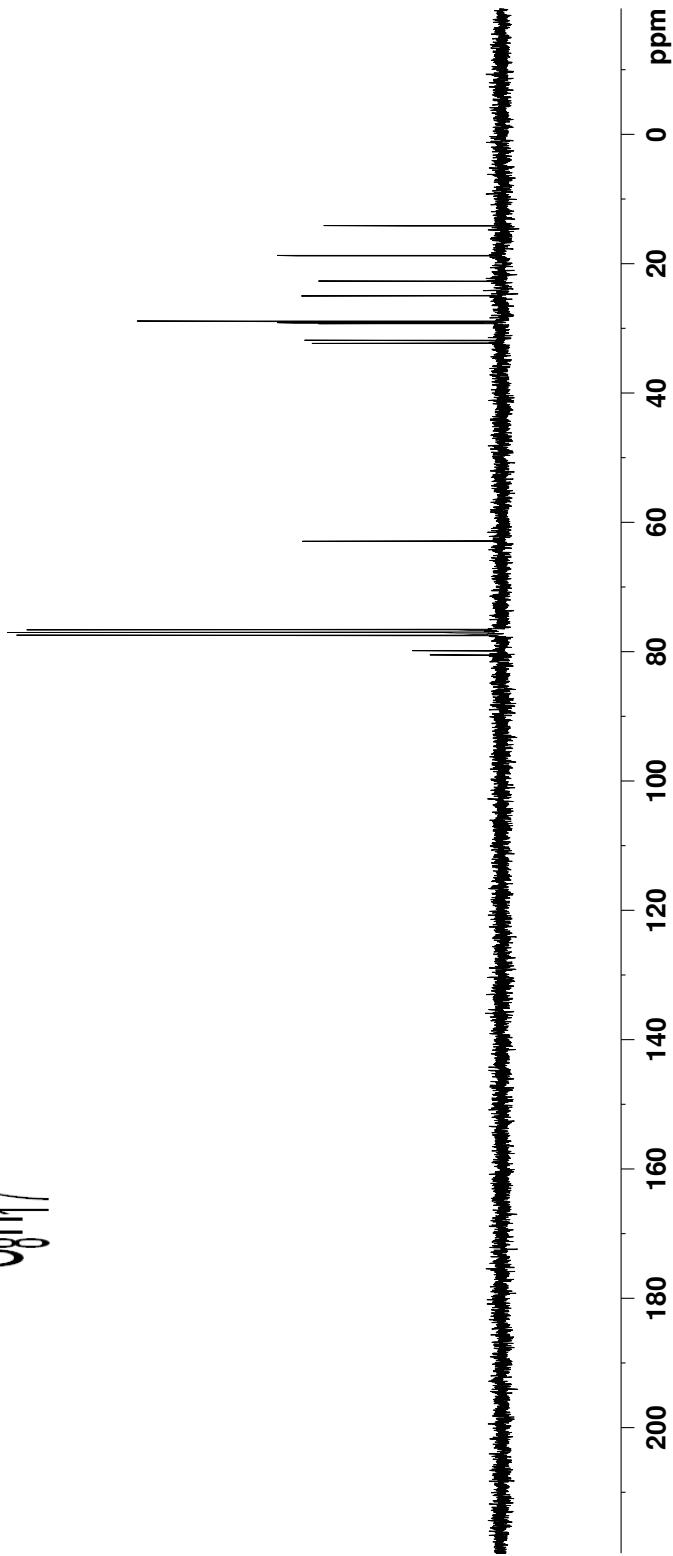
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NAME          081218
EXENO         2
PROCNO        1
Date_         20081218
Time          16.56
INSTRUM       spect
PROBHD       5 mm PABBO BB-
PULPROG      ZGP930
TD           65536
SOLVENT       CDCl3
NS            65
DS            0
SWH          18028.816 Hz
FIDRES       0.25098 Hz
AQ            1.8175818 sec
RG            27.203
DW           27.733 usec
DE            6.50 usec
TE            290.4 K
D1           2.0000000 sec
D11          0.03000000 sec
TD0          1

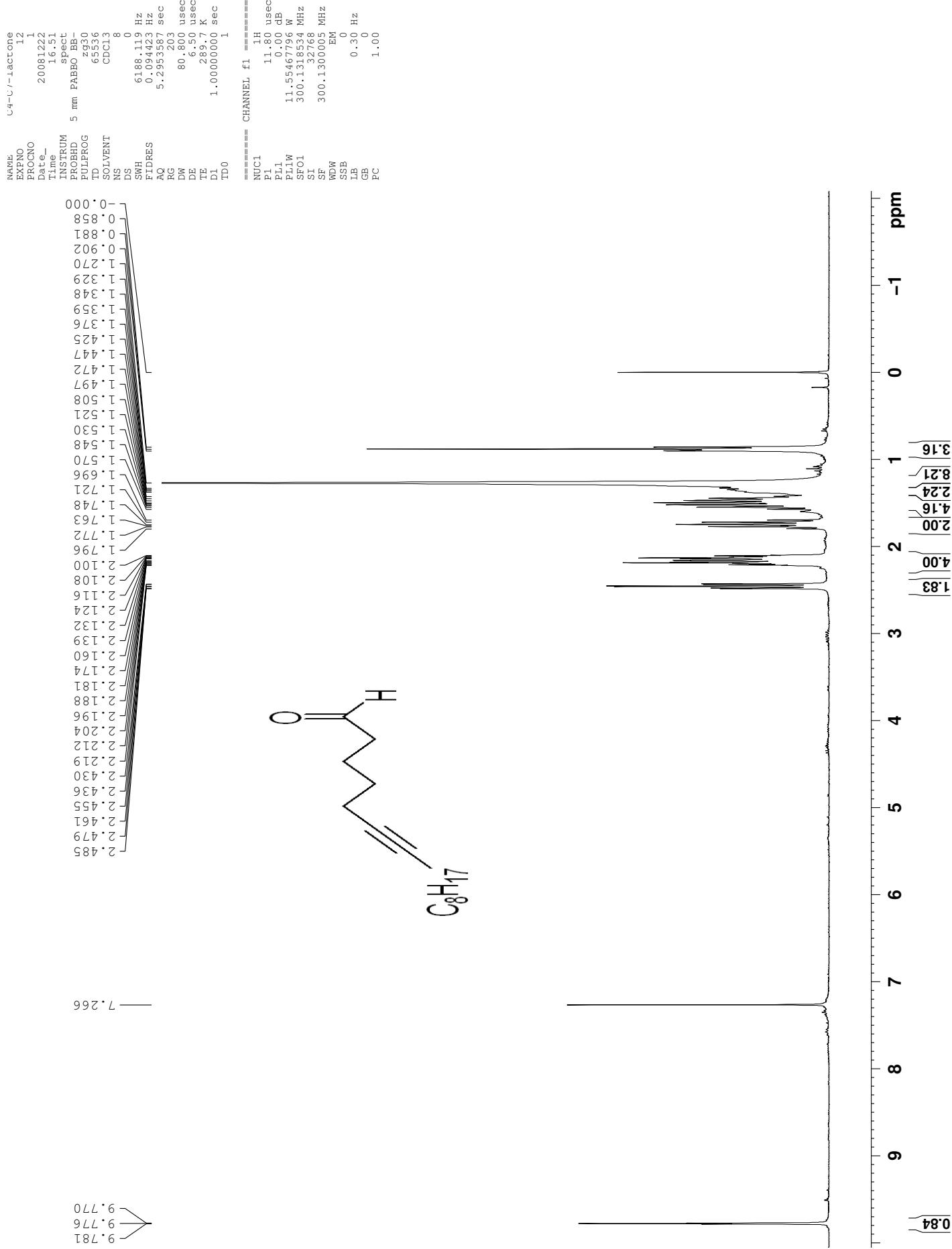
===== CHANNEL f1 =====
NUC1          13C
P1            9.70 usec
PL1          0.00 dB
PL1W         29.38907051 W
SF01         75.4732933 MHz

===== CHANNEL f2 =====
CPDPGR2      waitz16
NUC2          1H
PCPD2        80.00 usec
PL2          1.00 dB
PL12         17.00 dB
PL13         17.00 dB
PL2W         9.17820644 W
PL12W        0.23034613 W
PL13W        0.23034613 W
SF02         300.1312005 MHz
SI            75.467753 MHz
SF            EM
WDW           0
SSB           1.00 Hz
LB            0
GB            1.10
PC            1.10
```



62.87





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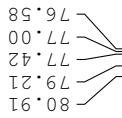
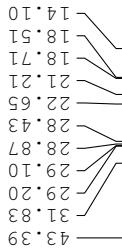
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NAME      C4-C7-lactone
EXNNO     13
PROCNO    1
Date_     20081222
Time      16.55
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpp36
TD       65536
SOLVENT   CDCl3
NS        187
DS         0
SWH      18028.816 Hz
FIDRES   0.225098 Hz
AQ        1.8175818 sec
RG        27.733 usec
DW       6.500 usec
DE        290.4 K
TE       2.0000000 sec
D1      0.03000000 sec
D11     1
TD0      1

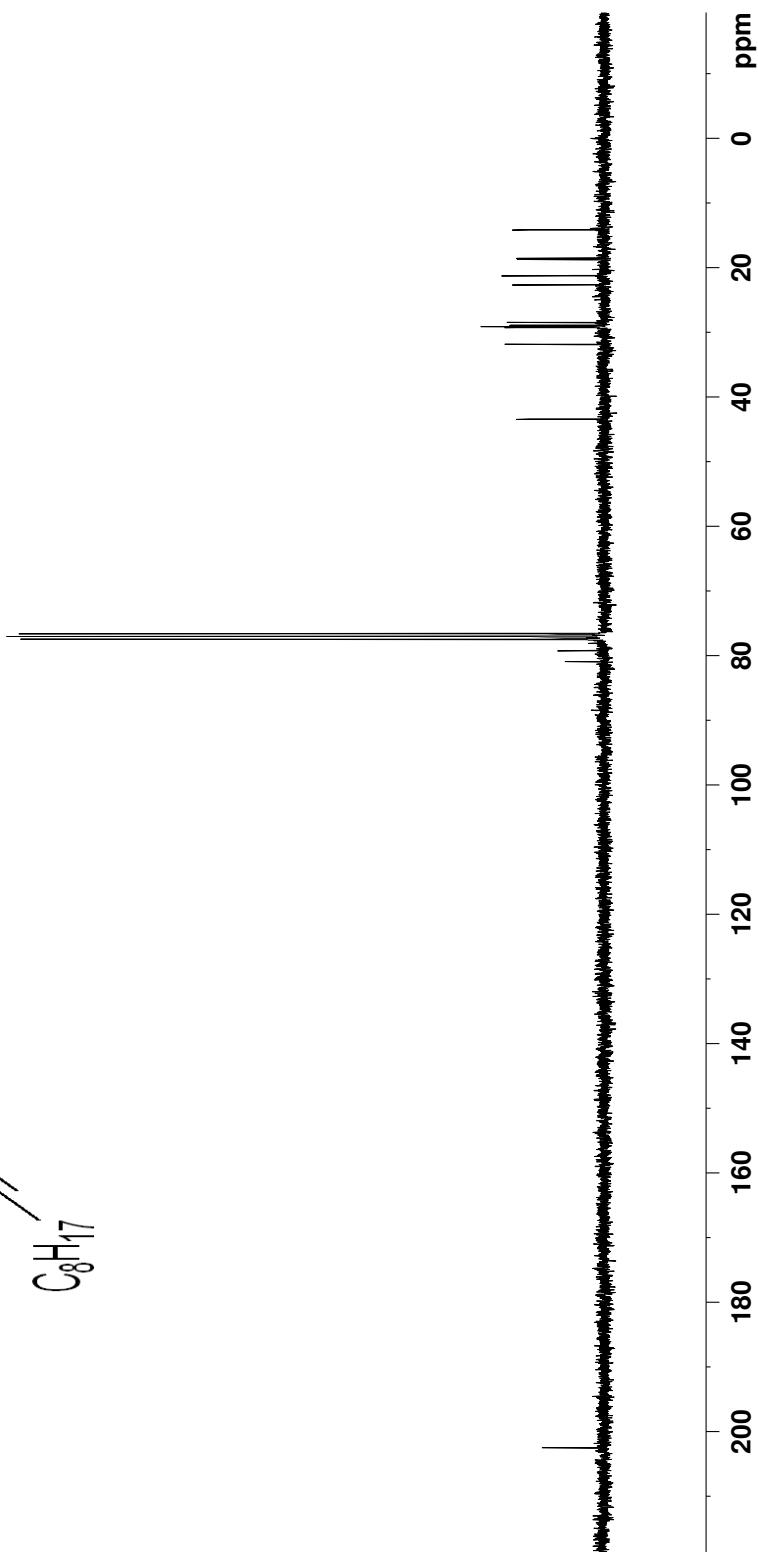
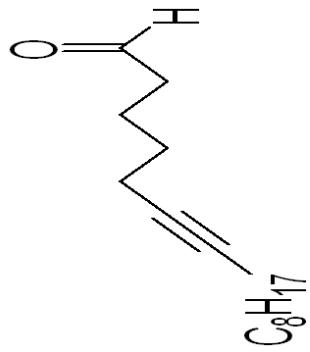
===== CHANNEL f1 =====
NUC1      13C
P1        9.70 usec
PL1      0.00 dB
PL1W    29.38907051 W
SF01    75.4732933 MHz

===== CHANNEL f2 =====
CPDPGR2  waitz16
NUC2      1H
PCPD2    80.00 usec
PL2      1.00 dB
PL12    17.00 dB
PL13    9.17820644 W
PL2W    0.23034613 W
PL12W   0.23034613 W
PL13W   300.1312005 MHz
SF02    75.4677438 MHz
SI        EM
SF        0
WDW      1.00 Hz
SSB      0
LB        1.40
GB      0
PC      1.40

```



202.49



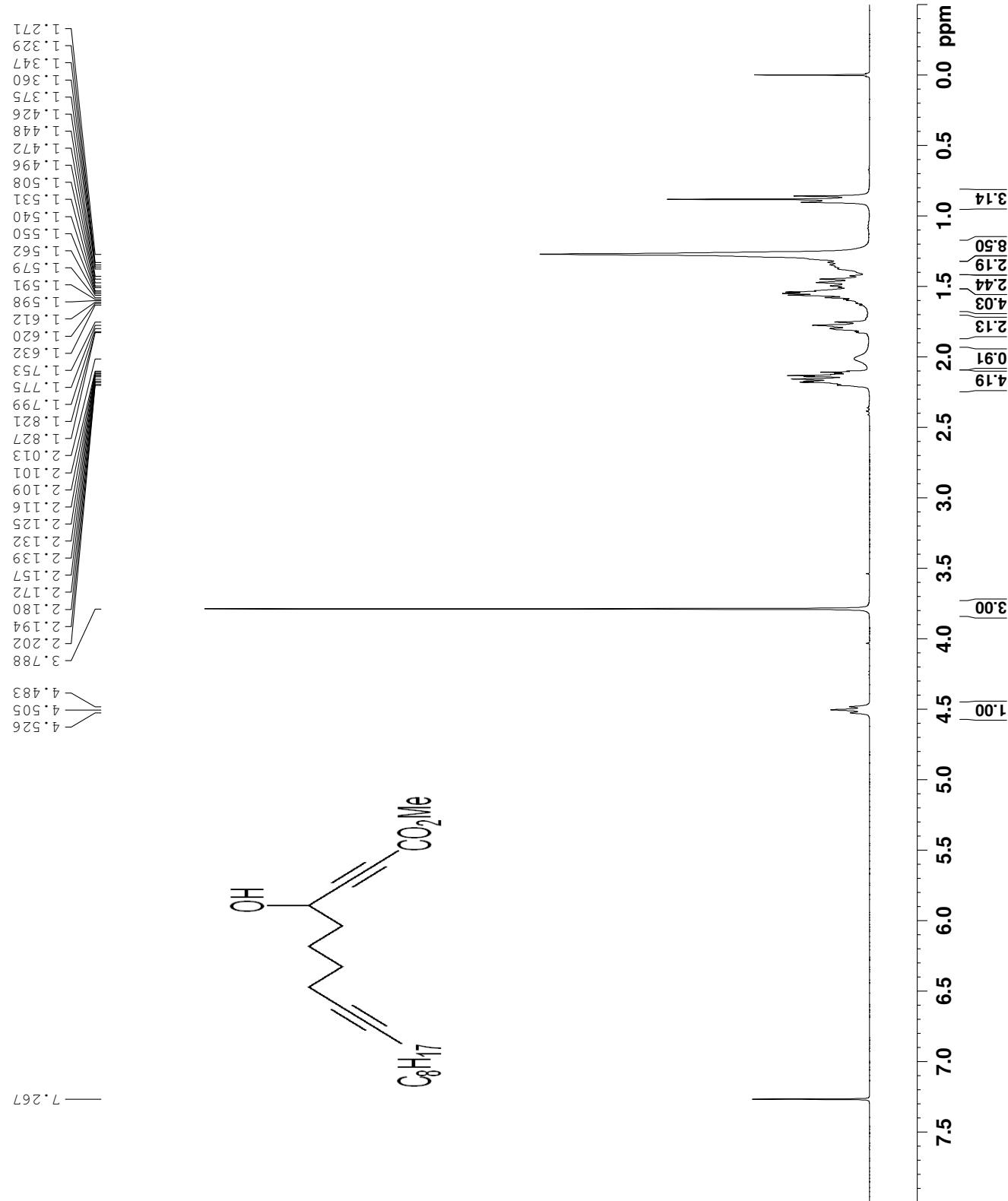
Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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```

INSTRUMENTAL
NAME: EXPNO
EXPNO: 1
IPROCN: 1
Date: 20/08/224
Time: 15.09
PROBID: 5
INSTRUM: PABBO BB-
PULPROG: z930
TD: 51356
TDTime: CDC13
SOLVENT: INS
DS: 8
FWHM: 61.88
FDURES: 0.09423
PAQ: 5.2945587
TRG: sec
RGW: 80.6
DE: 80.800
DE: 6.50
TE: 29.01
TE: 1.0000000
DD1: sec
DD2D0: 1

=====
CHANNEL f1 =====
NUNC1: 1H
P1: 11.80 usec
PPL1: 0.00 dB
PPL1W: 11.55467796 W
SF01: 300.318534 MHz
SI: 32768
SF: EM
WDW: 0
SSB: 0.30 Hz
ILB: 0
RGB: 0
PC: 1.00

```



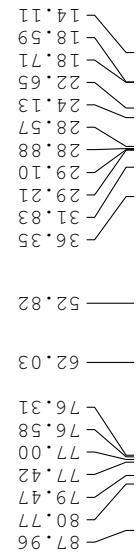
```

NAME      C4-C7-lactone
EXNNO          1
PROCNO         1
Date_        20081224
Time       15.13
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG ZPP930
TD        65536
SOLVENT    CDCl3
NS           240
DS            0
SWH       18028.816 Hz
FIDRES   0.225098 Hz
AQ        1.8175818 sec
RG        27.733 usec
DW        6.500 usec
DE        290.9 K
TE        2.0000000 sec
D1        0.03000000 sec
D11       1
TD0          1

===== CHANNEL f1 =====
NUC1      13C
P1        9.70 usec
PL1      0.00 dB
PL1W     29.38907051 W
SF01     75.4732933 MHz

===== CHANNEL f2 =====
CPDPGR2  waitz16
NUC2      1H
PCPD2     80.00 usec
PL2      1.00 dB
PL12     17.00 dB
PL13     17.00 dB
PL2W     9.17820644 W
PL12W    0.23034613 W
PL13W    0.23034613 W
SF02     300.1312005 MHz
SI        75.4677439 MHz
SF          EM
WDW        0
SSB        1.00 Hz
LB          0
GB          1.40
PC          1.40

```

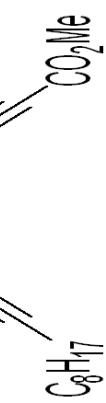
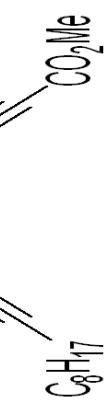
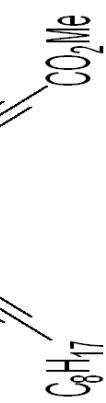
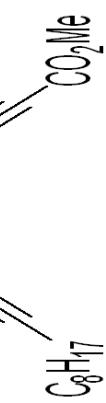
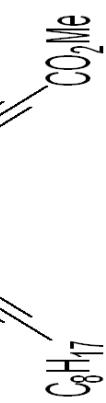
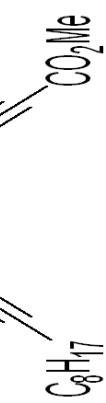
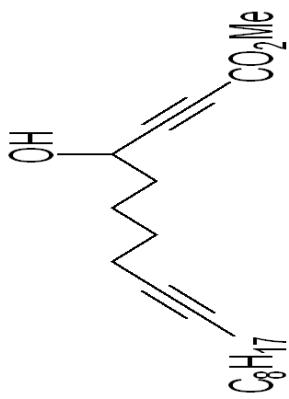


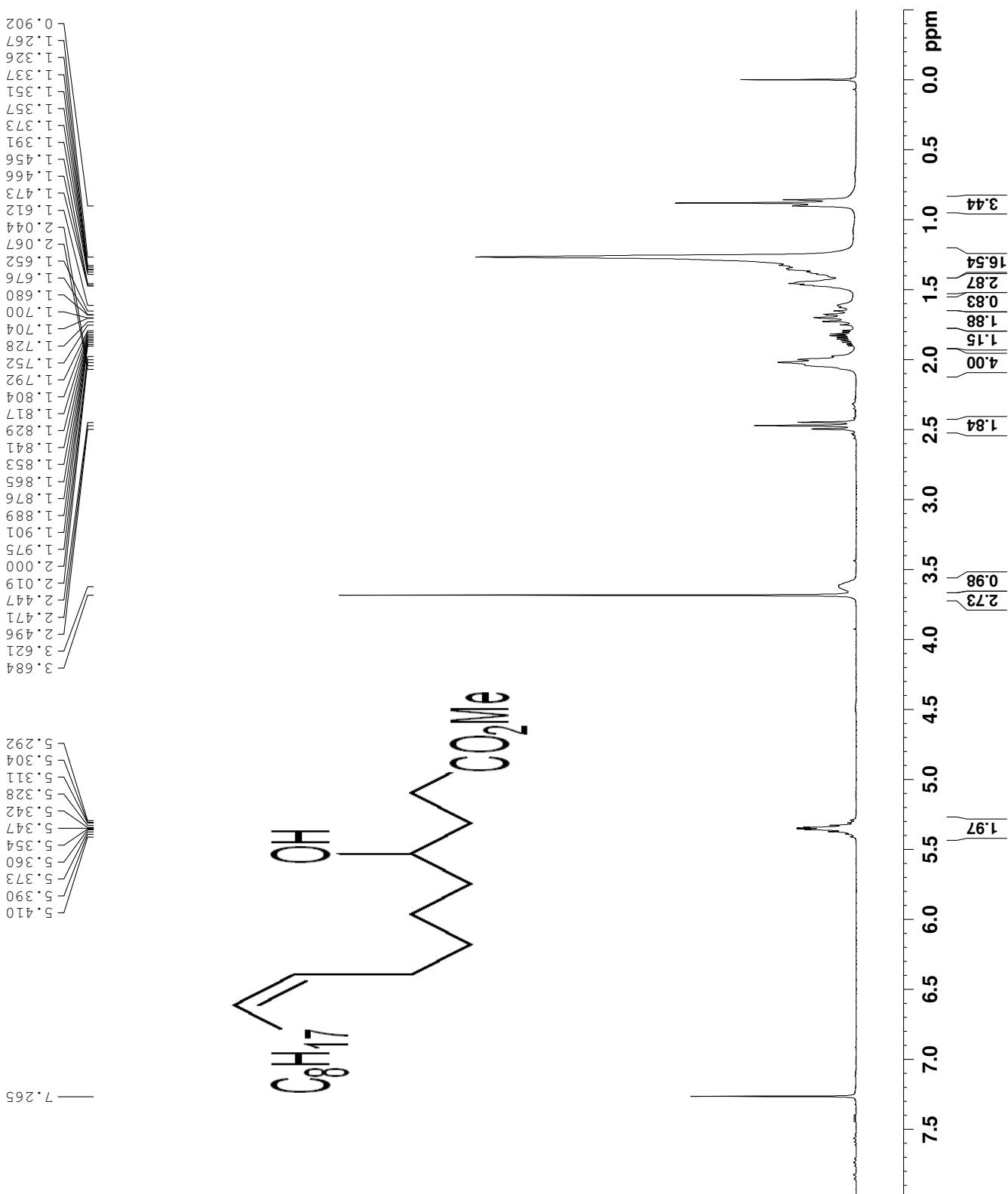
52.82

62.03

76.31
76.58
77.00
77.42
79.47
80.77
87.96

153.74



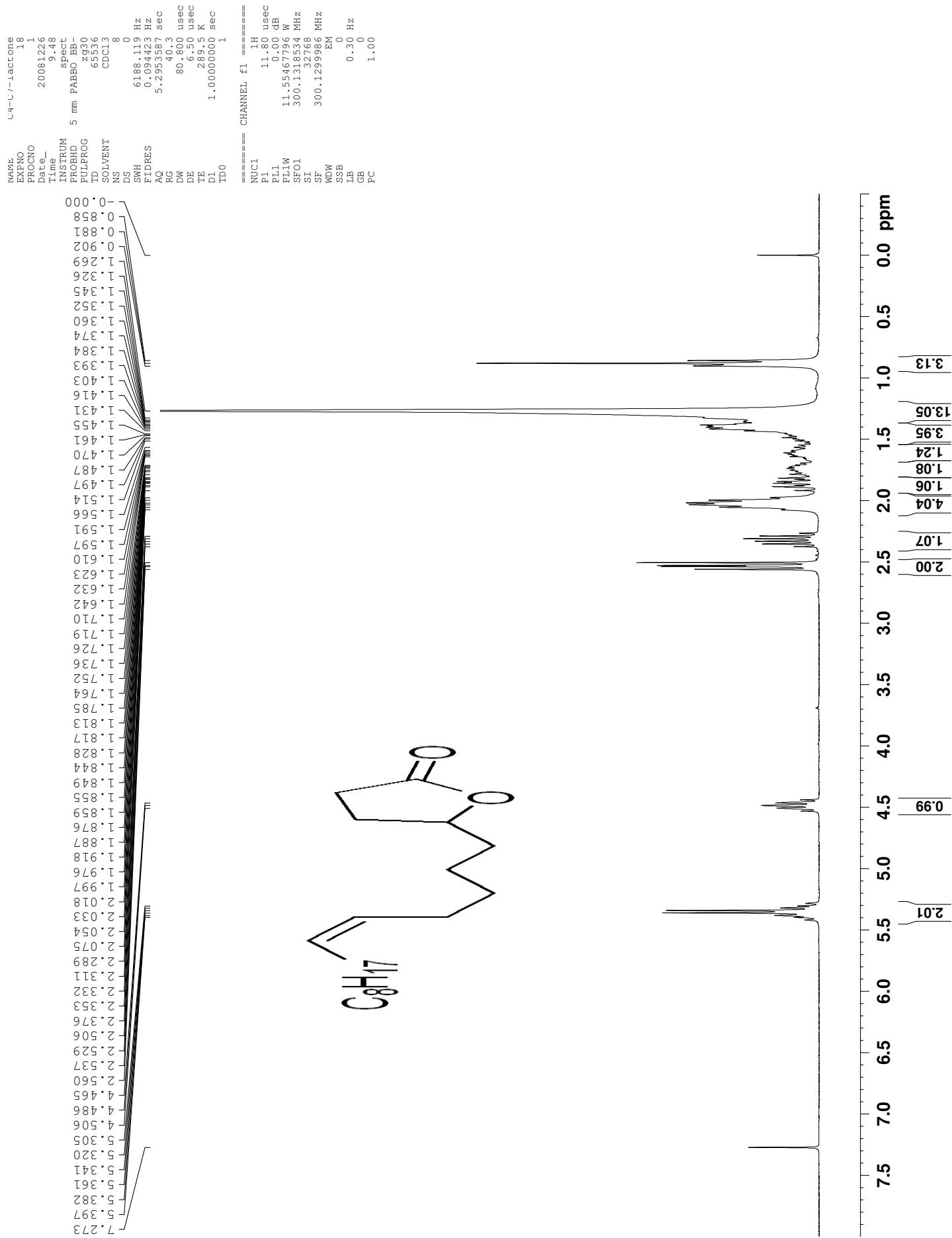


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NAME      LIN11      1
EXENO     1
PROCNO   1
Date_     20081226
Time_    15.47
INSTRUM  spect
PROBHD  5 mm PABBO BB-
PULPROG ZPP936
TD       65536
SOLVENT  CDC13
NS      412
DS        4
SWH     18028.816 Hz
FIDRES  0.225098 Hz
AQ      1.8175818 sec
RG      27.203
DW      27.733 usec
DE      6.50 usec
TE      291.0 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1
===== CHANNEL f1 =====
NUC1      13C
P1        9.70 usec
PL1      0.00 dB
PL1W     29.38907051 W
SF01     75.4732933 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2      1H
PCPD2     80.00 usec
PL2      1.00 dB
PL12     17.00 dB
PL13     17.00 dB
PL2W     9.17820644 W
PL12W    0.23034613 W
PL13W    0.23034613 W
SF02     300.1312005 MHz
SI        75.4677494 MHz
SF        EM
WDW      0
SSB      1.00 Hz
LB        0
GB        1.40
PC

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14.11
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