

Indium(I)-catalyzed alkyl–allyl coupling between ethers and an allylborane

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Electronic Supplementary Information (ESI)

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1 General

Nuclear Magnetic Resonance (NMR) spectra were recorded on a JEOL ECX-400, a JEOL ECA-500, or a JEOL ECX-600 spectrometer, operating at 400 MHz, 500 MHz, or 600 MHz for ^1H NMR, and 100 MHz, 125 MHz, or 150 MHz for ^{13}C NMR (128 MHz for ^{11}B NMR). Chemical shifts were reported downfield from tetramethylsilane (TMS). Infra Red (IR) spectra were measured using a JASSO FT/IR-610 spectrometer. High Resolution Mass Spectra (HRMS) were recorded using a JEOL JMS T100TD (DART) spectrometer. Preparative thin-layer chromatography (PTLC) was carried out using Wakogel B-5F from WAKO. All solvents used were commercially available dry solvents that were further dried and degassed appropriately under an argon atmosphere, and stored over activated molecular sieves in an argon box prior to use.

InOTf was prepared according to a reported procedure, and stored in an argon box at $-30\text{ }^\circ\text{C}$.¹ All other metal salts were purchased from ALDRICH, and stored in an argon box at room temperature. All reactions were carried out under an argon atmosphere in flame-dried glassware. 9-BBN-derived allylborane **2b** was synthesized according to a reported method.²

2 Starting materials

2.1 Procedure for synthesis of starting materials

To a stirred solution of an alcohol (20 mmol) in dry THF (20 mL) at $0\text{ }^\circ\text{C}$ was slowly added NaH (2 equiv). The reaction mixture was further stirred at $0\text{ }^\circ\text{C}$ for 1 h before slow addition of MeI (1.5 equiv). The mixture was then allowed to reach to room temperature overnight. Quenching with water followed by extraction (Et_2O , $3 \times 10\text{ mL}$) and evaporation afforded the corresponding crude ether product, which was then purified by column chromatography to provide substrates **1a–u**.

2.2 Analytical data for unknown ethers

1-Bromo-3-(1-methoxyethyl)-benzene (**1f**):

^1H NMR (CDCl_3 , 600 Hz): $\delta = 1.42$ (s, 3H), 3.23 (s, 3H), 4.23–4.27 (q, $J = 7.2$, 1H), 7.20–7.26 (m, 2H), 7.40–7.46 (m, 2H) ppm.

^{13}C NMR (CDCl_3 , 150 Hz): $\delta = 23.8$, 56.6, 79.0, 122.6, 124.8, 129.2, 130.1, 130.5, 146.0 ppm.

IR (neat): $\nu = 2977$, 2930, 2320, 1117, 1068, 998, 871, 668 cm^{-1} .

HRMS (DART): calculated for $\text{C}_8\text{H}_8^{79}\text{Br}^+ = [\text{M}-\text{OMe}]^+$: $m/z = 182.98094$, found: $m/z = 182.98041$.

1-Methoxy-1-(2-thienyl)-ethane (**1k**):

^1H NMR (CDCl_3 , 600 Hz): $\delta = 1.56$ (s, 3H), 3.27 (s, 3H), 4.56–4.58 (q, $J = 6.9$, 1H), 6.95–6.97 (m, 2H), 7.25–7.26 (m, 1H), ppm.

^{13}C NMR (CDCl_3 , 150 Hz): $\delta = 23.8, 56.2, 74.9, 124.5, 124.7, 126.3, 147.2$ ppm.

IR (neat): $\nu = 2978, 2930, 2820, 1113, 1086, 996, 858, 830, 701$ cm^{-1} .

HRMS (DART): calculated for $\text{C}_6\text{H}_7^{32}\text{S}^+ = [\text{M}-\text{OMe}]^+$: $m/z = 111.02685$, found: $m/z = 182.02728$.

3 In(I)-catalyzed coupling reactions

3.1 Procedure for In(I)-catalyzed C–C bond formation

To a flame-dried 5 ml-screw-vial with magnetic stirring bar in an argon box were added successively InOTf (1–5 mol%), dry toluene or DCM (0.5 M), the corresponding ether **1a–u** (0.4 mmol), and allylborane **2b** (1.2–2.0 equiv). The reaction mixture was stirred at the indicated temperature for the indicated time until complete consumption of the corresponding electrophile **1** (detected by TLC or ^1H NMR analyses of an aliquot of the reaction mixture). The crude reaction mixtures were purified -without further treatment- by preparative thin layer chromatography (PTLC; eluant: hexane \rightarrow hexane/ $\text{Et}_2\text{O} = 95:5$) or column chromatography (eluant: hexane) to afford the corresponding products **3a–u**.

3.2 Analytical data for products

2-(1-Methyl-3-buten-1-yl)-naphthalene (**3a**):

Prepared from ether **1a** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (1 mol%) in DCM (0.5 M) at 25 °C for 90 min. *The obtained analytical data of 3a fit accurately with the reported data.*³

Colorless liquid.

Yield: 86%.

4-Benzyl-1-butene (**3b**):

Prepared from ether **1b** and allylborane **2b** (1.5 equiv) according to the general procedure with InOTf (5 mol%) in DCM (0.5 M) at 25 °C for 14 h. *The obtained analytical data of 3b fit accurately with the reported data.*⁴

Colorless liquid.

Yield 25%.

4-(4-Methoxyphenyl)-1-butene (**3c**):

Prepared from ether **1c** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in toluene (0.5 M) at a temperature range from –20 °C to room temperature for 14 h. *The obtained analytical data of 3c fit accurately with the reported data.*⁵

Colorless liquid.

Yield: 80%.

2-(3-Buten-1-yl)-naphthalene (**3d**):

Prepared from ether **1d** and allylborane **2b** (1.5 equiv) according to the general procedure with InOTf (5

mol%) in DCM (0.5 M) at 25 °C for 14 h. *The obtained analytical data of 3d fit accurately with the reported data.*⁵

Colorless liquid.

Yield 25%.

(1-Methyl-3-buten-1-yl)-benzene (3e):

Prepared from ether **1e** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3e fit accurately with the reported data.*⁴

Colorless liquid.

Yield: 73%.

1-Bromo-3-(1-methyl-3-buten-1-yl)-benzene (3f):

Prepared from ether **1f** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 5 h.

Colorless liquid.

Yield 55%.

¹H NMR (CDCl₃, 600 Hz): δ = 1.22–1.24 (d, *J* = 6.9, 3H), 2.24–2.29 (m, 1H), 2.33–2.37 (m, 1H), 2.74–2.77 (m, 1H), 4.96–5.00 (m, 2H), 5.64–5.71 (m, 1H), 7.11–7.17 (m, 2H), 7.31–7.34 (m, 2H) ppm.

¹³C NMR (CDCl₃, 150 Hz): δ = 21.3, 39.6, 42.4, 116.3, 122.4, 125.7, 129.0, 129.9, 130.1, 136.5, 149.4 ppm

IR (neat): ν = 2961, 1558, 1540, 1260, 1072, 668 cm⁻¹.

HRMS (DART): calculated for C₁₁H₁₄⁸¹Br⁺ = [M+H]⁺: *m/z* = 227.02584, found: *m/z* = 227.02524.

(1-(2-Chloroethyl)-3-buten-1-yl)-benzene (3g):

Prepared from ether **1g** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (5 mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3g fit accurately with the reported data.*⁶

Colorless liquid.

Yield: 62%.

1-Allyl-1,2,3,4-tetrahydronaphthalene (3h):

Prepared from ether **1h** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3h fit accurately with the reported data.*⁷

Colorless liquid.

Yield: 83%.

4,4-Diphenyl-1-butene (3i):

Prepared from ether **1i** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2

mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3i fit accurately with the reported data.*⁷

Colorless liquid.

Yield: 98%.

4- (*p*-Methoxyphenyl)-4-phenyl-1-butene (3j):

Prepared from ether **1j** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3j fit accurately with the reported data.*⁶

Colorless liquid.

Yield: 94%.

4-(2-Thienyl)-1-pentene (3k):

Prepared from ether **1k** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (5 mol%) in DCM (0.5 M) at 25 °C for 30 min.

Colorless liquid.

Yield 45%.

¹H NMR (CDCl₃, 600 Hz): δ = 1.32–1.33 (d, *J* = 7.6, 3H), 2.30–2.35 (m, 1H), 2.43–2.47 (m, 1H), 3.10–3.13 (m, 1H), 5.00–5.06 (m, 2H), 5.73–5.79 (m, 1H), 6.80–6.81 (d, *J* = 3.48, 1H), 6.91–6.93 (m, 1H), 7.12–7.13 (d, *J* = 4.8, 1H) ppm.

¹³C NMR (CDCl₃, 150 Hz): δ = 22.3, 35.1, 43.5, 116.5, 122.5, 122.6, 126.4, 136.4, 151.2 ppm.

IR (neat): ν = 3075, 2964, 2925, 1235, 993, 915, 847, 822, 692 cm⁻¹.

HRMS (DART): calculated for C₉H₁₃³²S⁺ = [M+H]⁺: *m/z* = 153.07380, found: *m/z* = 153.07387.

(1,1-Dimethyl-3-buten-1-yl)-benzene (3l):

Prepared from ether **1l** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at –20 °C for 14 h. *The obtained analytical data of 3l fit accurately with the reported data.*⁶

Colorless liquid.

Yield: 40%.

1-(1,1-Dimethyl-3-buten-1-yl)-4-methoxy-benzene (3m):

Prepared from ether **1m** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at –20 °C for 5 h.

Colorless liquid.

Yield: 78%.

¹H NMR (CDCl₃, 600 Hz): δ = 1.28 (s, 6H), 2.33–2.34 (d, *J* = 6.9, 2H), 3.79 (s, 3H), 4.93–4.97 (m, 2H), 5.52–5.59 (m, 1H), 6.84–6.65 (d, *J* = 8.9, 2H), 7.25–7.47 (d, *J* = 8.9, 2H) ppm.

¹³C NMR (CDCl₃, 150 Hz): δ = 28.7, 36.9, 48.9, 55.2, 113.3, 116.8, 126.8, 135.7, 141.3, 157.3 ppm.

IR (neat): ν = 3077, 2962, 1515, 1249, 1184, 1038, 911, 808 cm⁻¹.

HRMS (DART): calculated for $C_{13}H_{19}O^+ = [M+H]^+$: $m/z = 191.14359$, found: $m/z = 191.14370$.

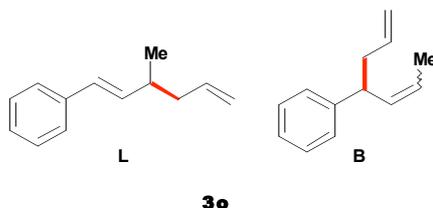
4-(4,4,4-Triphenyl)-1-butane (3n):

Prepared from ether **1n** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3n fit accurately with the reported data.*⁷

White solid.

Yield 95%.

(3-Methyl-1,5-hexadien-1-yl)-benzene (3o-L):

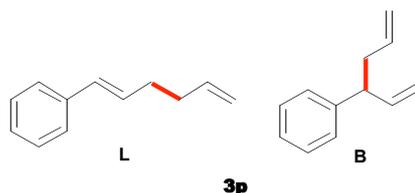


Prepared from ether **1o** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 30 min. *The obtained analytical data of 3o fit accurately with the reported data.*⁸

Colorless liquid.

Yield 93%, L:B = 1:1.

1,5-Hexadien-1-yl-benzene (3p-L):

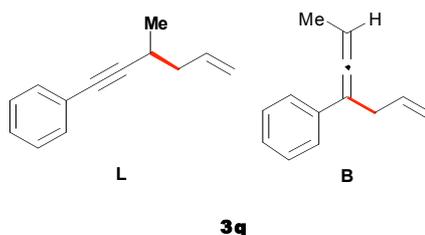


Prepared from ether **1p** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (5 mol%) in DCM (0.5 M) at 25 °C for 14 h. *The obtained analytical data of 3p fit accurately with the reported data.*⁹

Colorless liquid.

Yield: 30%, L:B = 6:1.

(3-Methyl-5-hexen-1-ynyl)benzene (3q-L):



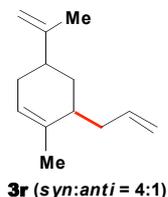
Prepared from ether **1q** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (5 mol%) in DCM (0.5 M) at 25 °C for 5h. *The obtained analytical data of 3q fit accurately with the*

reported data.¹⁰

Colorless liquid.

Yield: 60%, L:B=12:1.

1-Methyl-4-(1-methylethenyl)-6-(2-propenyl)-cyclohexene (**3r**):



Prepared from ether **1r** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (2 mol%) in DCM (0.5 M) at 25 °C for 14 h. *The obtained analytical data of 3r fit accurately with the reported data.*¹¹

Colorless liquid.

Yield: 85%, *syn:anti* = 4:1.

p-4-Pentenylanisole (**3t**):

Prepared from ether **1t** and allylborane **2b** (2.0 equiv) according to the general procedure with InOTf (5 mol%) under neat conditions at 60 °C for 48 h.

Colorless liquid.

Yield 58%.

¹H NMR (CDCl₃, 400 Hz): δ = 1.57–1.65 (m, 2H), 1.98–2.03 (m, 2H), 2.47–2.51 (t, *J* = 7.7, 2H), 3.70 (s, 3H), 4.88–4.97 (m, 2H), 5.70–5.80 (m, 1H), 6.73–6.76 (d, *J* = 8.7, 2H), 7.00–7.03 (d, *J* = 8.7, 2H) ppm.

¹³C NMR (CDCl₃, 100 Hz): δ = 30.8, 33.2, 34.4, 55.2, 113.7, 114.6, 129.3, 134.5, 138.7, 157.7 ppm.

IR (neat): ν = 3073, 2962, 2834, 1244, 1038, 911, 828 cm⁻¹.

HRMS (DART): calculated for C₁₂H₁₇O⁺ = [M+H]⁺: *m/z* = 177.12794, found: *m/z* = 177.12837.

1-Allyladamantane (**3u**):

Prepared from ether **1u** and allylborane **2b** (1.2 equiv) according to the general procedure with InOTf (5 mol%) in DCM (0.5 M) at 25 °C for 14 h. *The obtained analytical data of 3u fit accurately with the reported data.*⁴

Colorless liquid.

Yield 73%.

4 References

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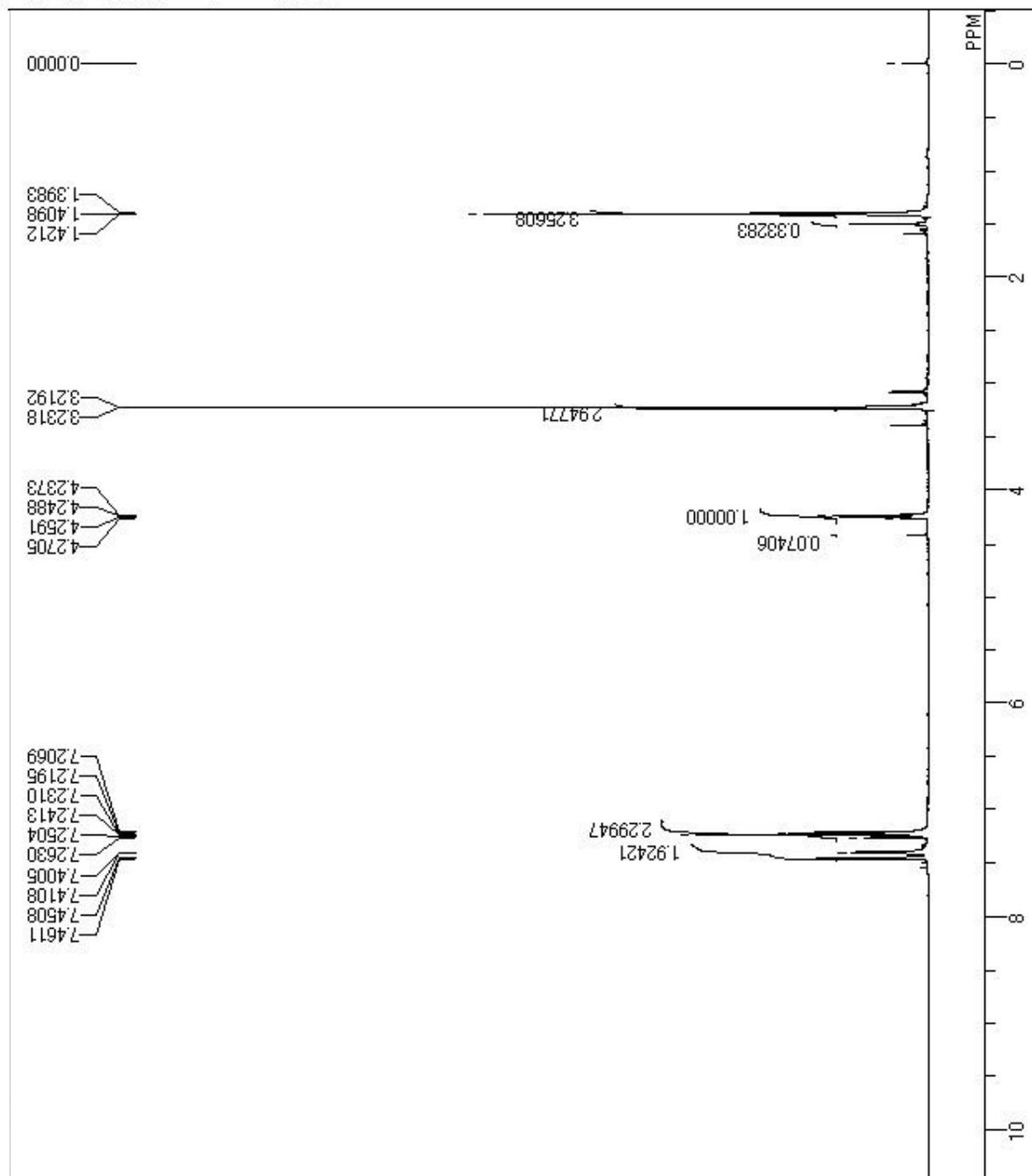
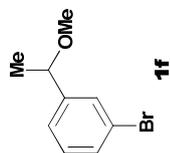
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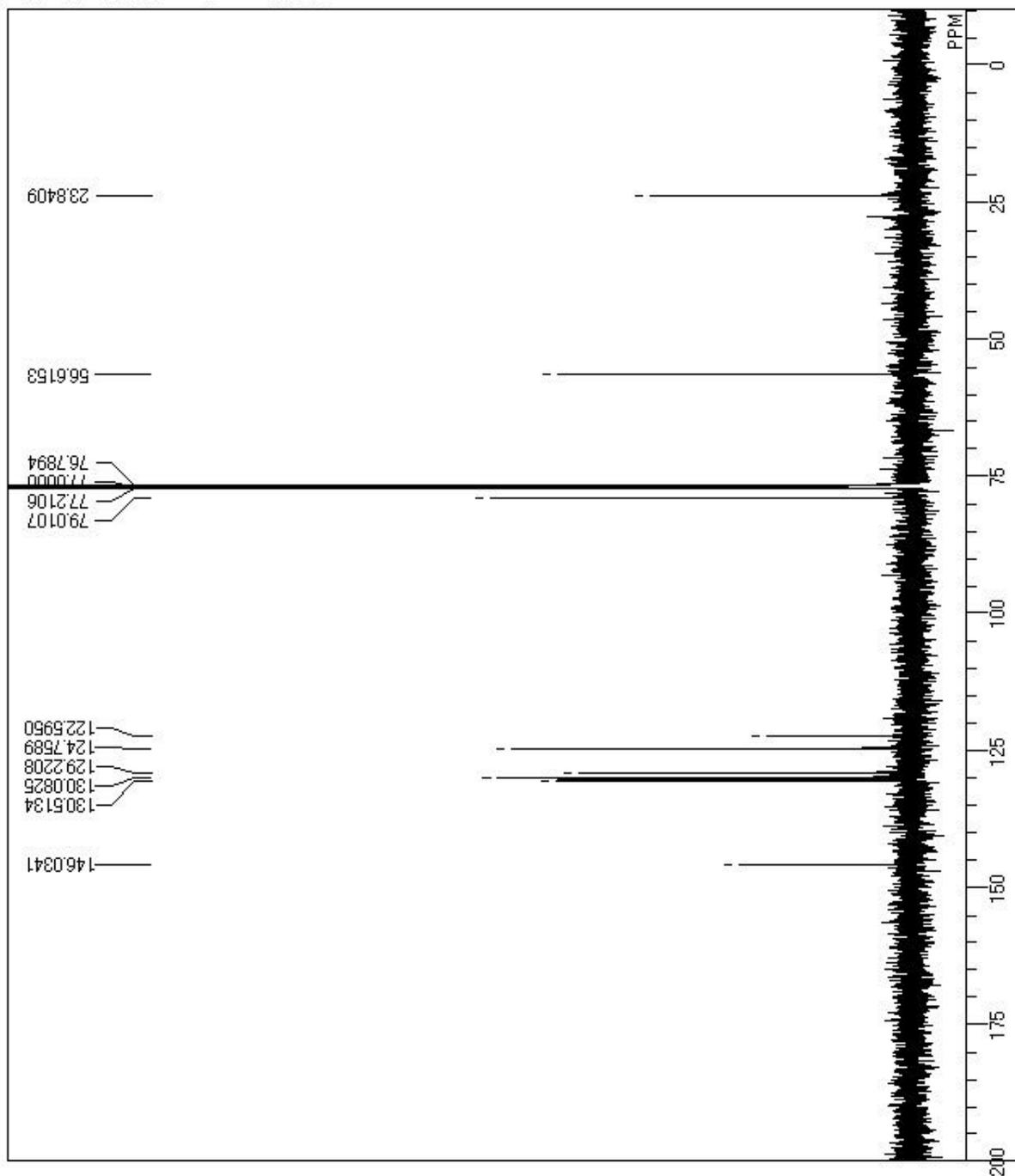
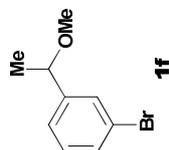
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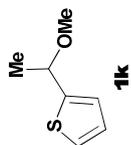
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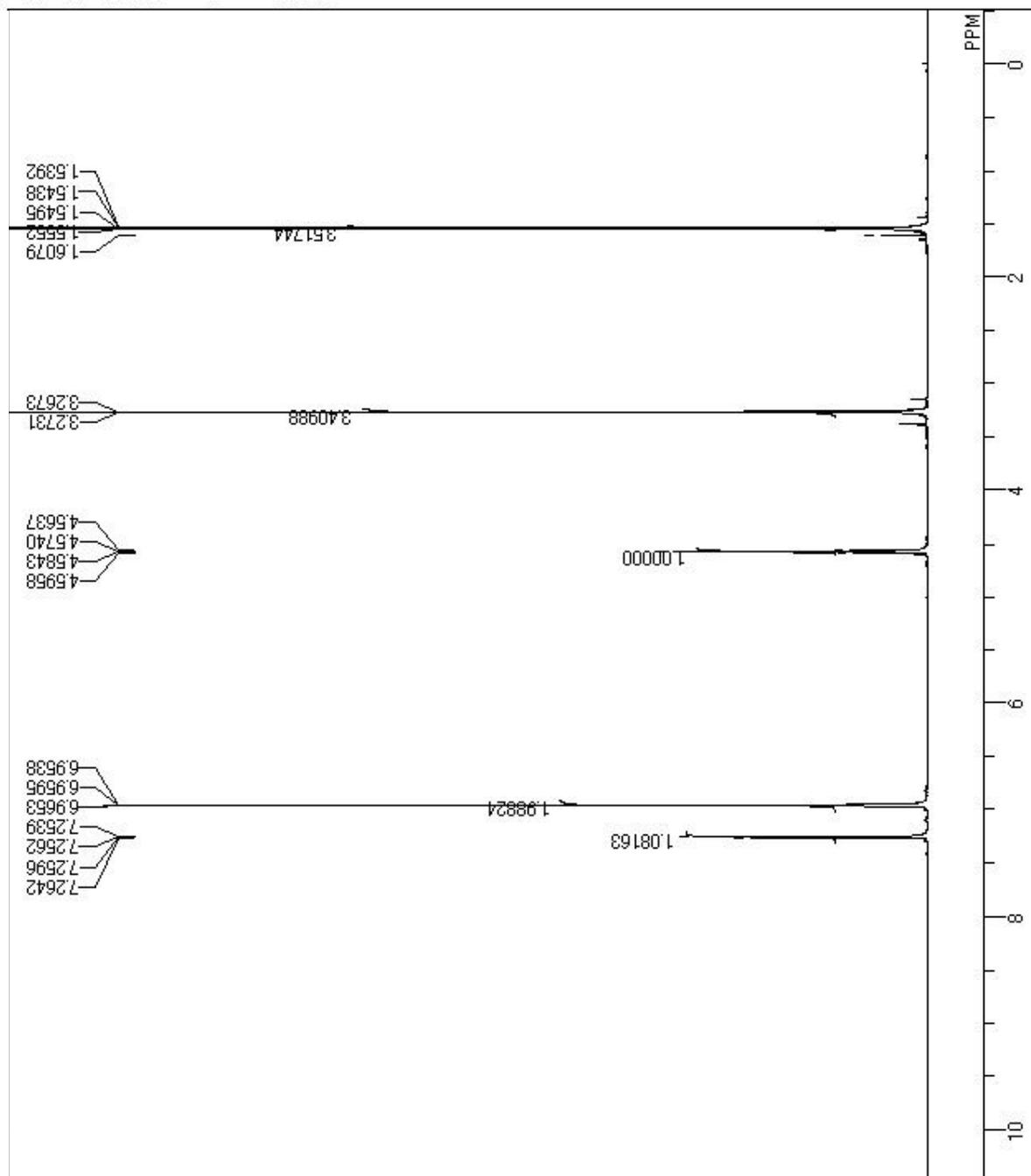
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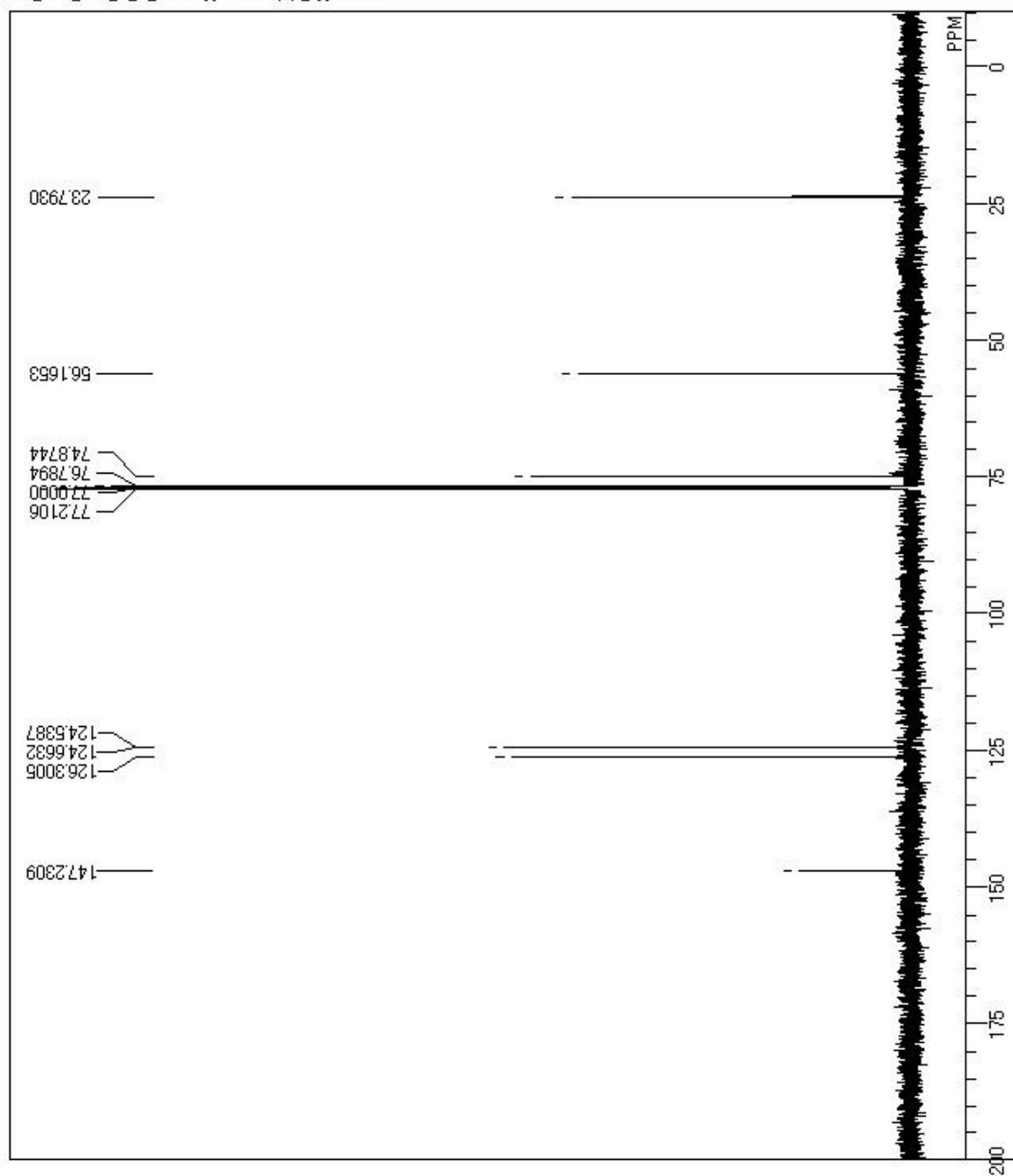
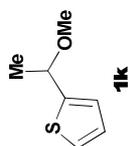
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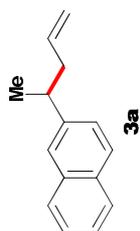
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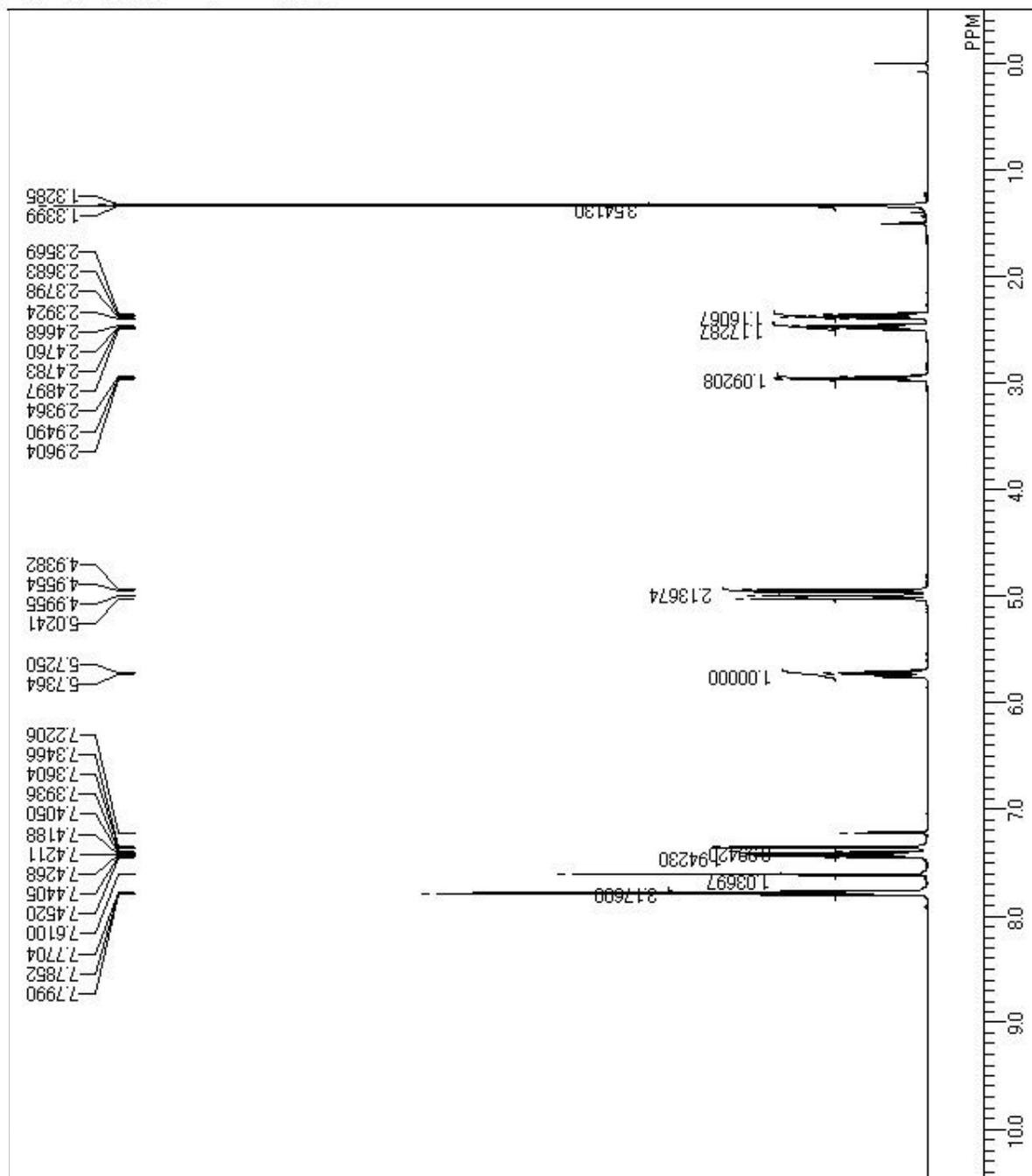
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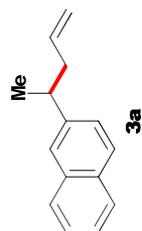
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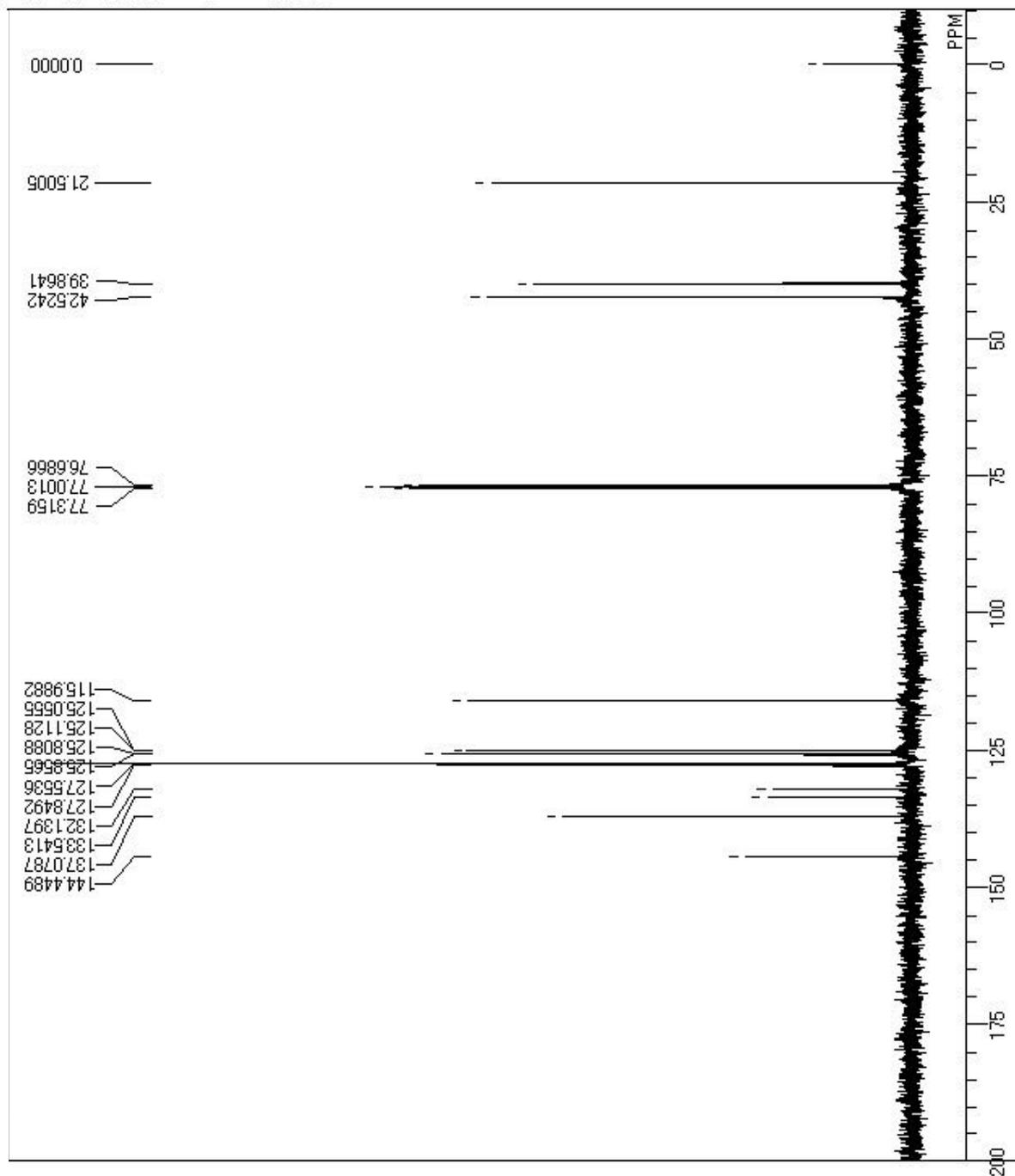
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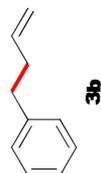
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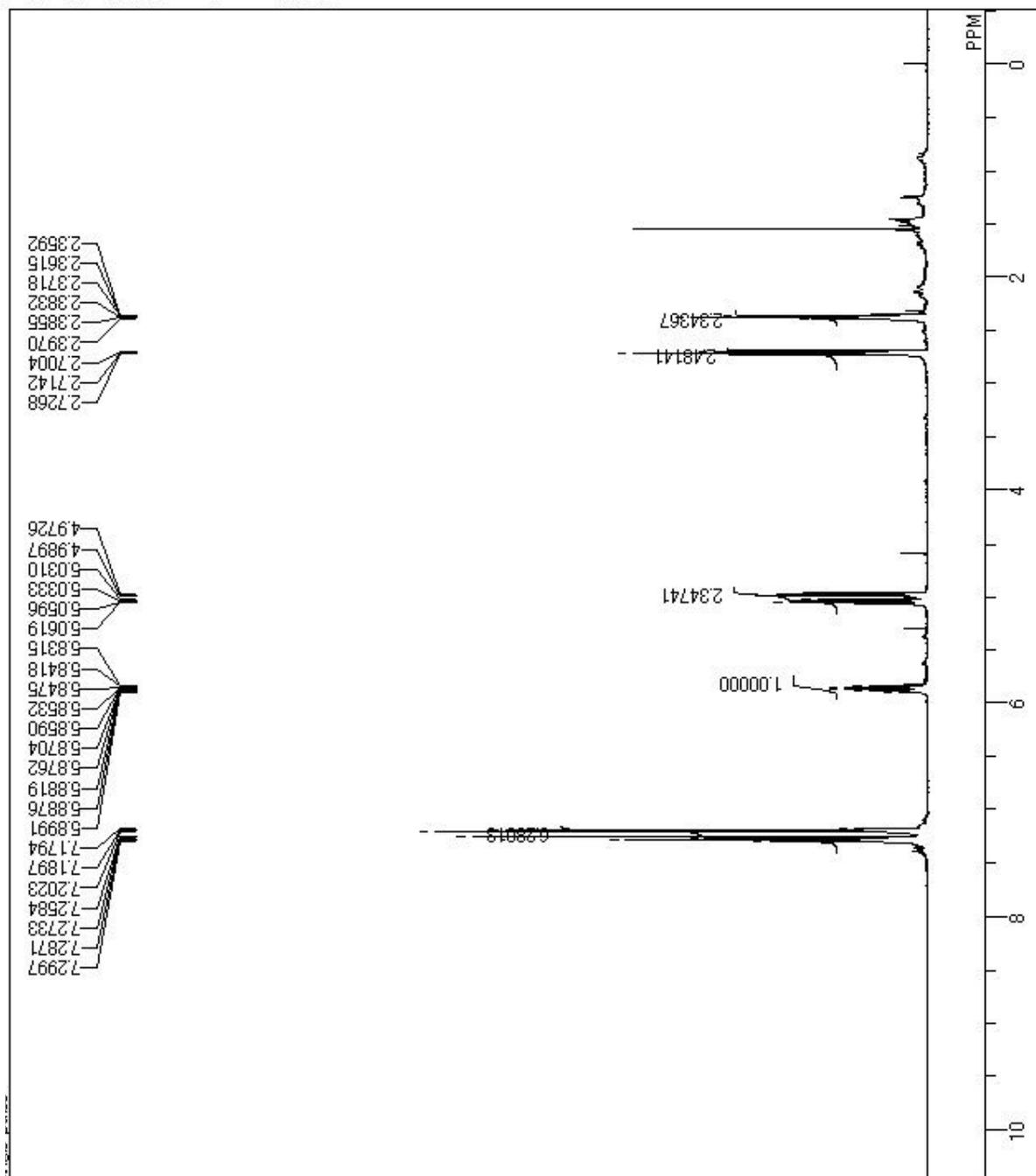
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18.1 c
CDCL3
0.00 ppm
1.20 Hz
42



DFILE
COMNT
DATIM
DANUC
EXMOD
OBFREQ
OBSET
OBFREQ
POINT
FREQ
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

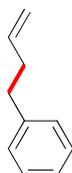
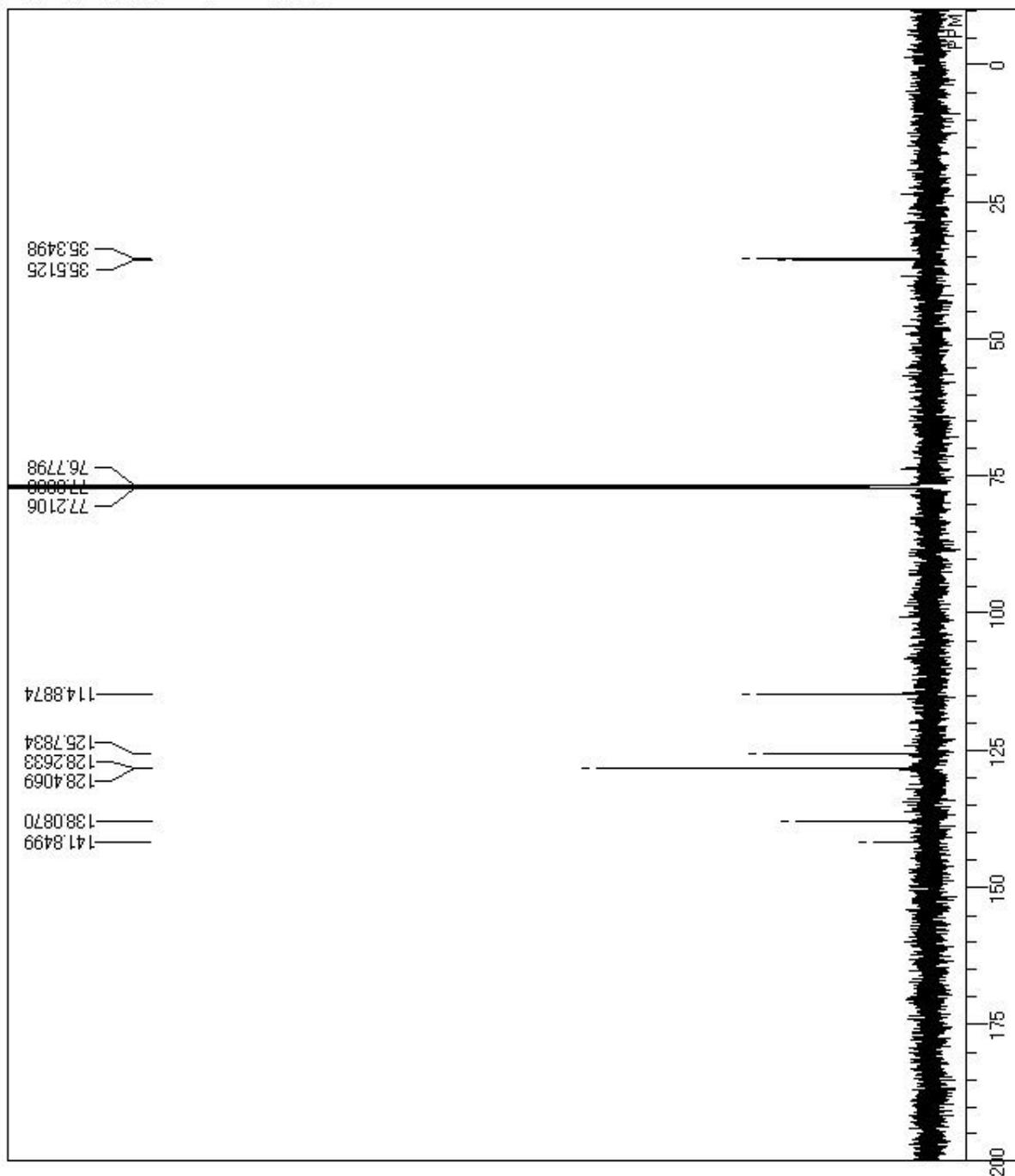


C:\Documents and Settings\delta\My Dr

22-02-2010 03:24:47

13C
shik pulse dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
168
0.6921 sec
2.0000 sec
2.87 usec
1H
192 c
CDCL3
77.00 ppm
1.20 Hz
60

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

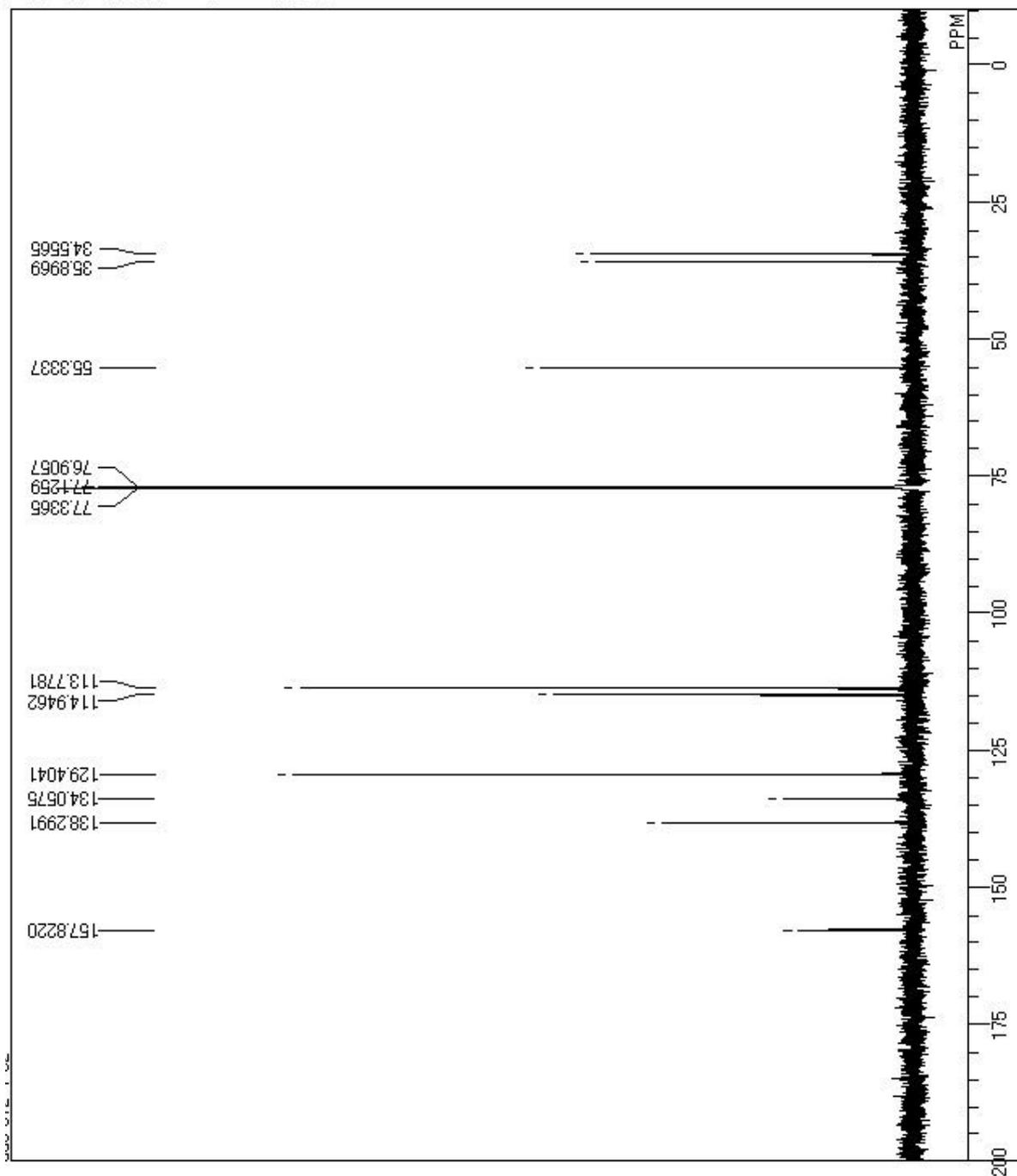
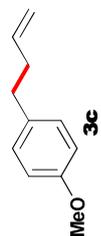


3b

C:\Documents and Settings\delta\My Dc

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

dao 512-1 c2
17-12-2009 02:18:34
13C
single pulse dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
79
0.6921 sec
2.0000 sec
2.87 usec
1H
20.0 c
CDCL3
225.50 ppm
0.12 Hz
60



C:\Documents and Settings\delta\My D

27-07-2010 19:57:04

1H

single pulse ex2

399.78 MHz

4.19 KHz

7.29 Hz

16384

POINT

7503.00 Hz

8

2.1837 sec

2.0000 sec

6.50 usec

1H

407.6 c

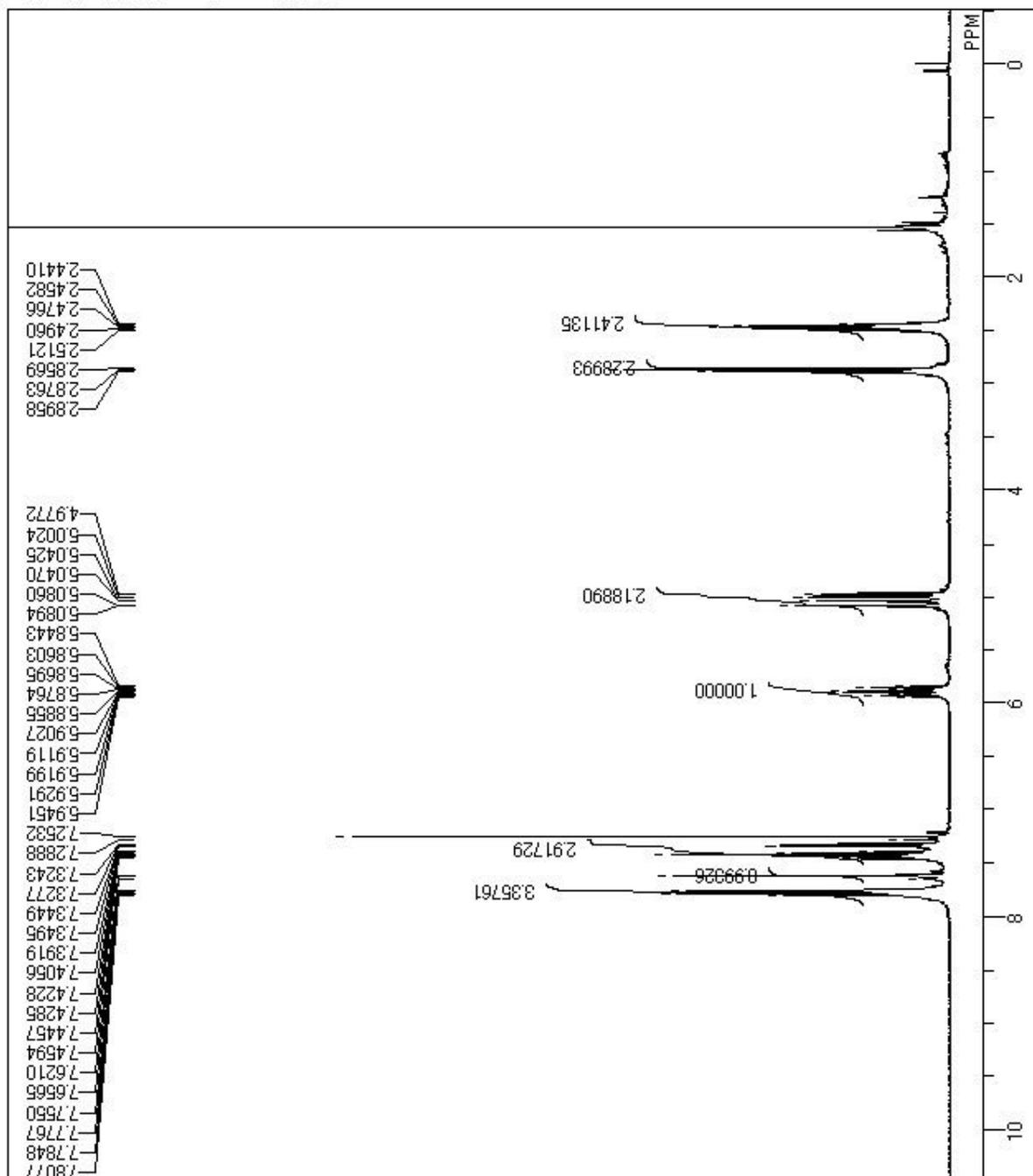
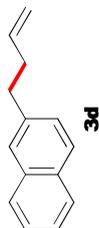
CDCL3

0.00 ppm

0.71 Hz

38

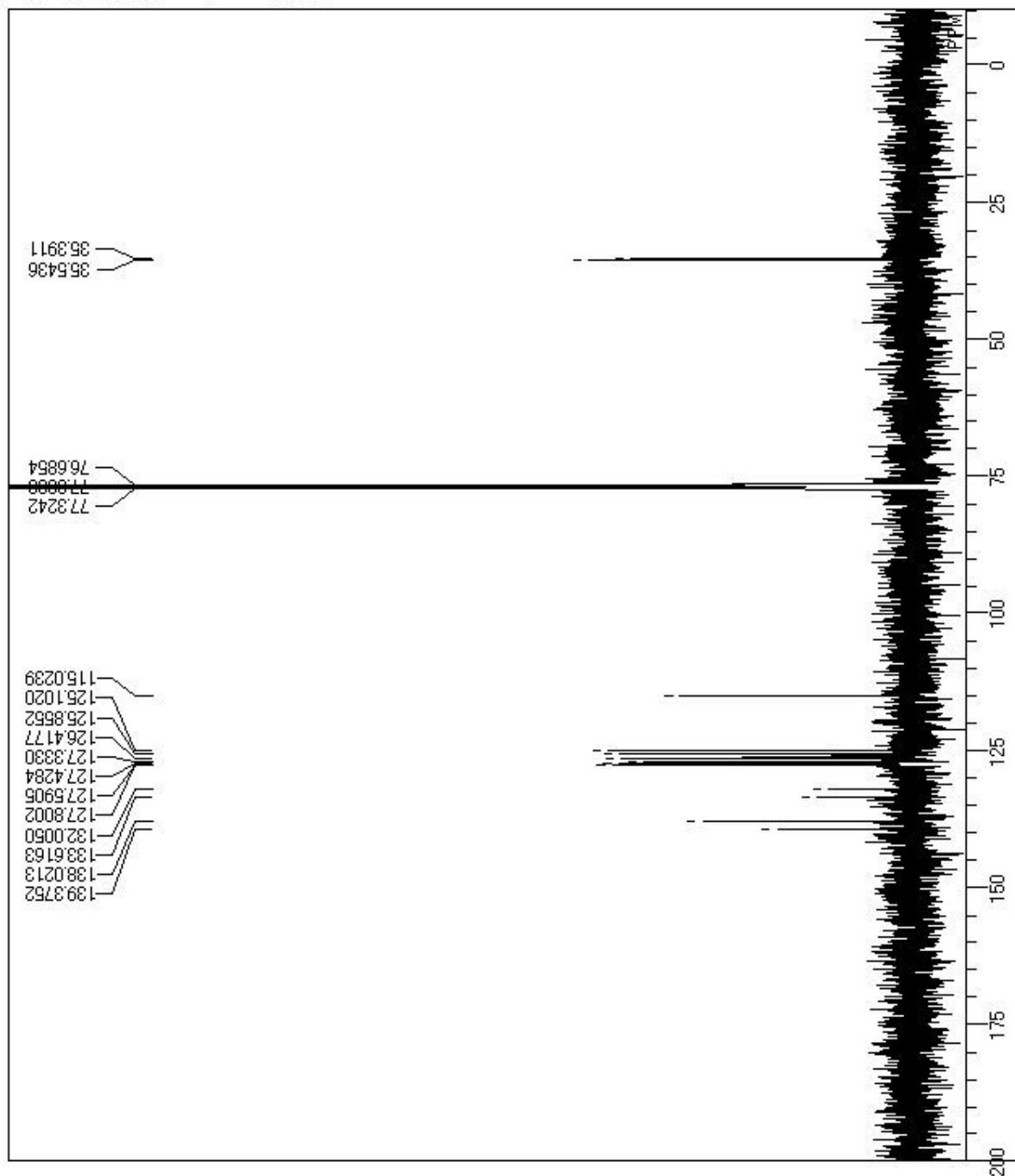
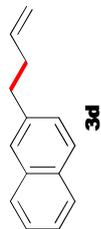
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



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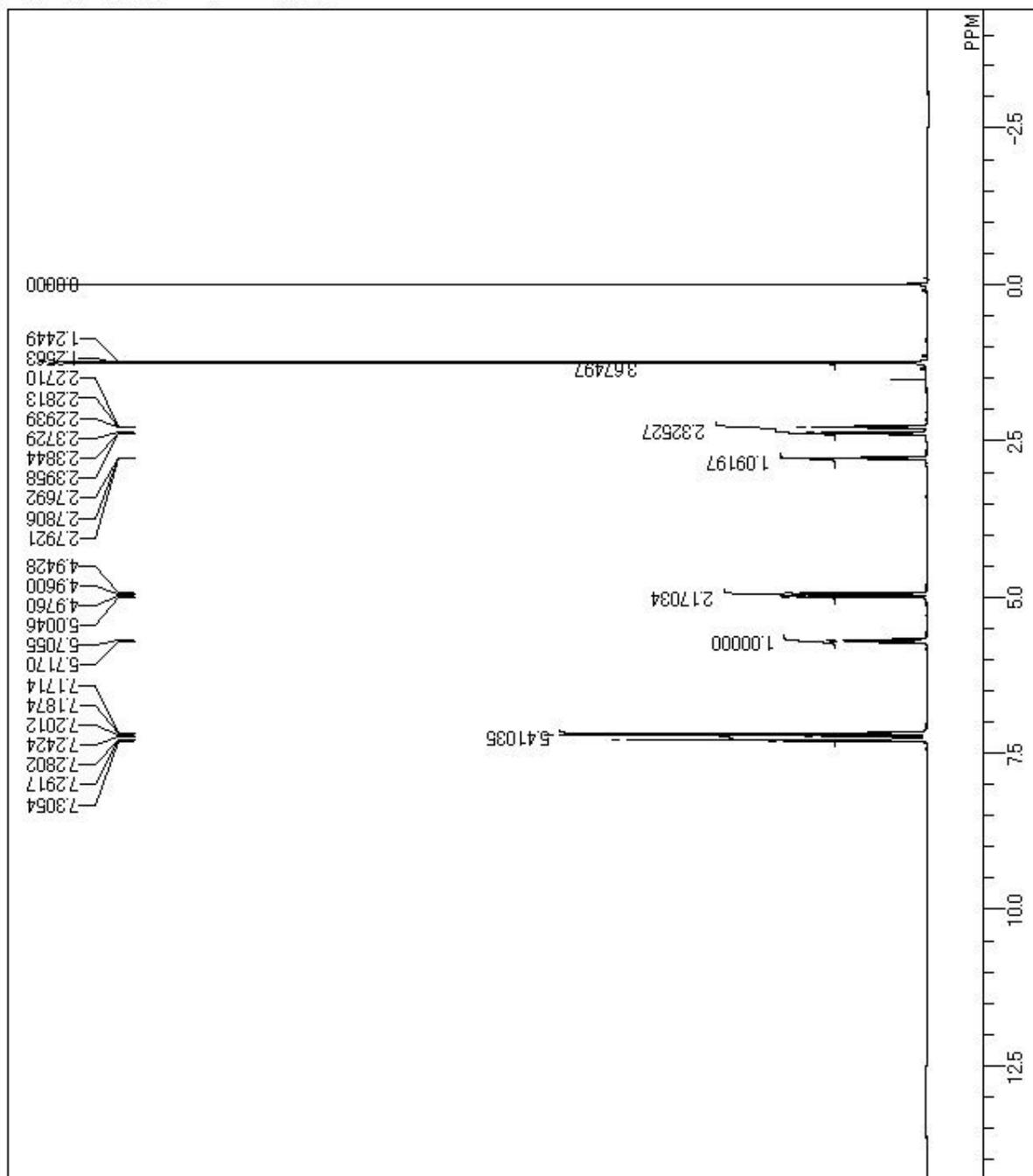
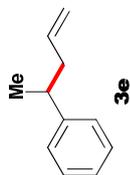
C:\Documents and Settings\delta\My Dr

DFILE
COMNT
DATIM 28-07-2010 21:52:14
OBNUC 13C
strs pulse_dec
EXMOD 100.53 MHz
OBFRQ 5.35 KHz
OBSET 5.86 Hz
OBFIN 32768
POINT 31407.04 Hz
FREQU 326
SCANS 1.0433 sec
ACQTM 2.0000 sec
PD 3.50 usec
PW1 1H
IRNUC 406.8 c
CTEMP CDCL3
SLVNT 77.00 ppm
EXREF 0.71 Hz
BF 54
RGAIN



C:\Documents and Settings\delta\My Dr

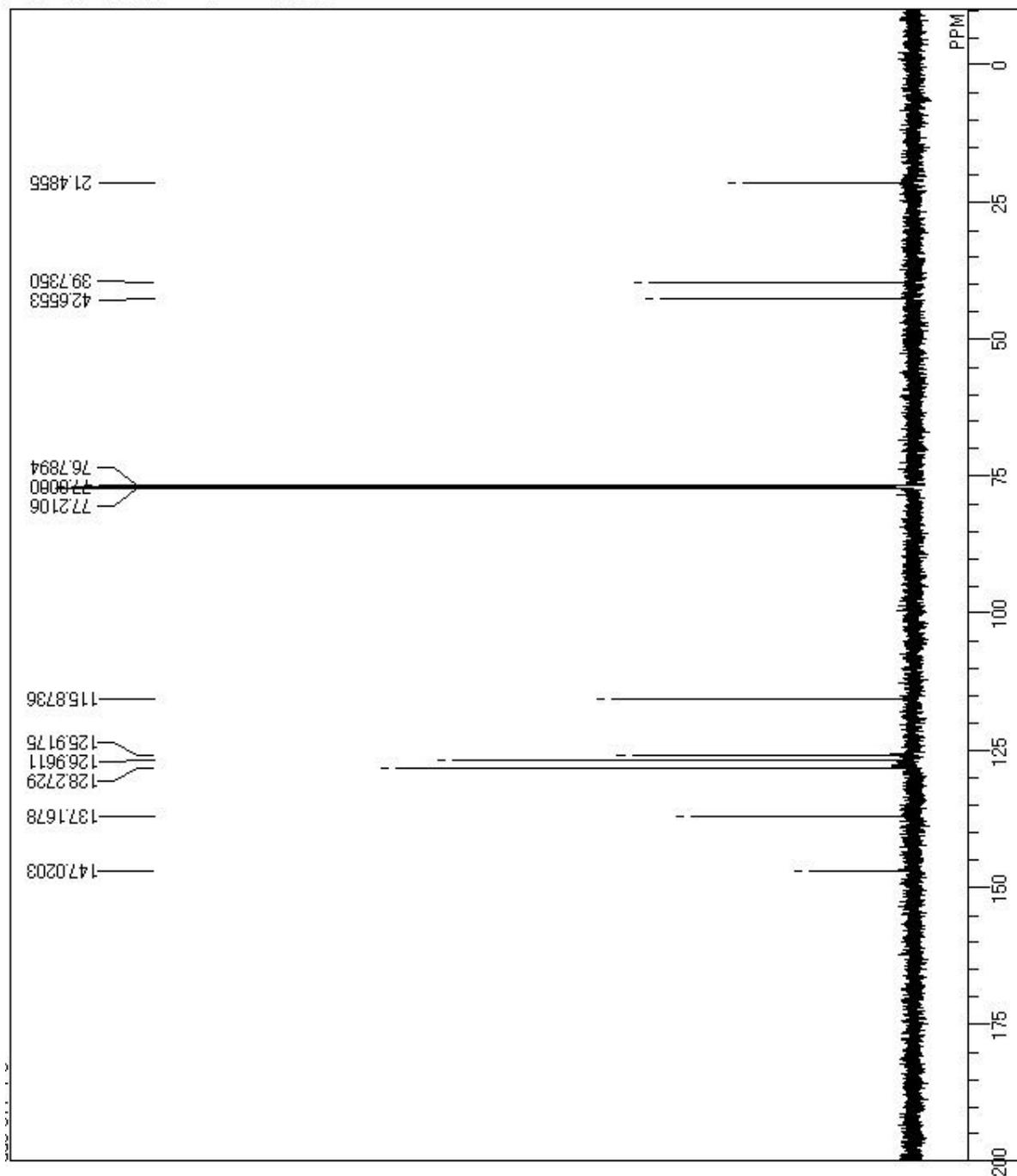
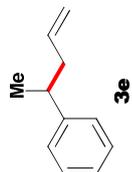
DFILE
COMNT
DATIM 15-01-2010 06:10:03
OBNUC 1H
EXMOD shake_pulsex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 8
ACQTM 1.4549 sec
PD 2.0000 sec
PWI 6.50 usec
IRNUC 1H
CTEMP 183 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 38



C:\Documents and Settings\delta\My D

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

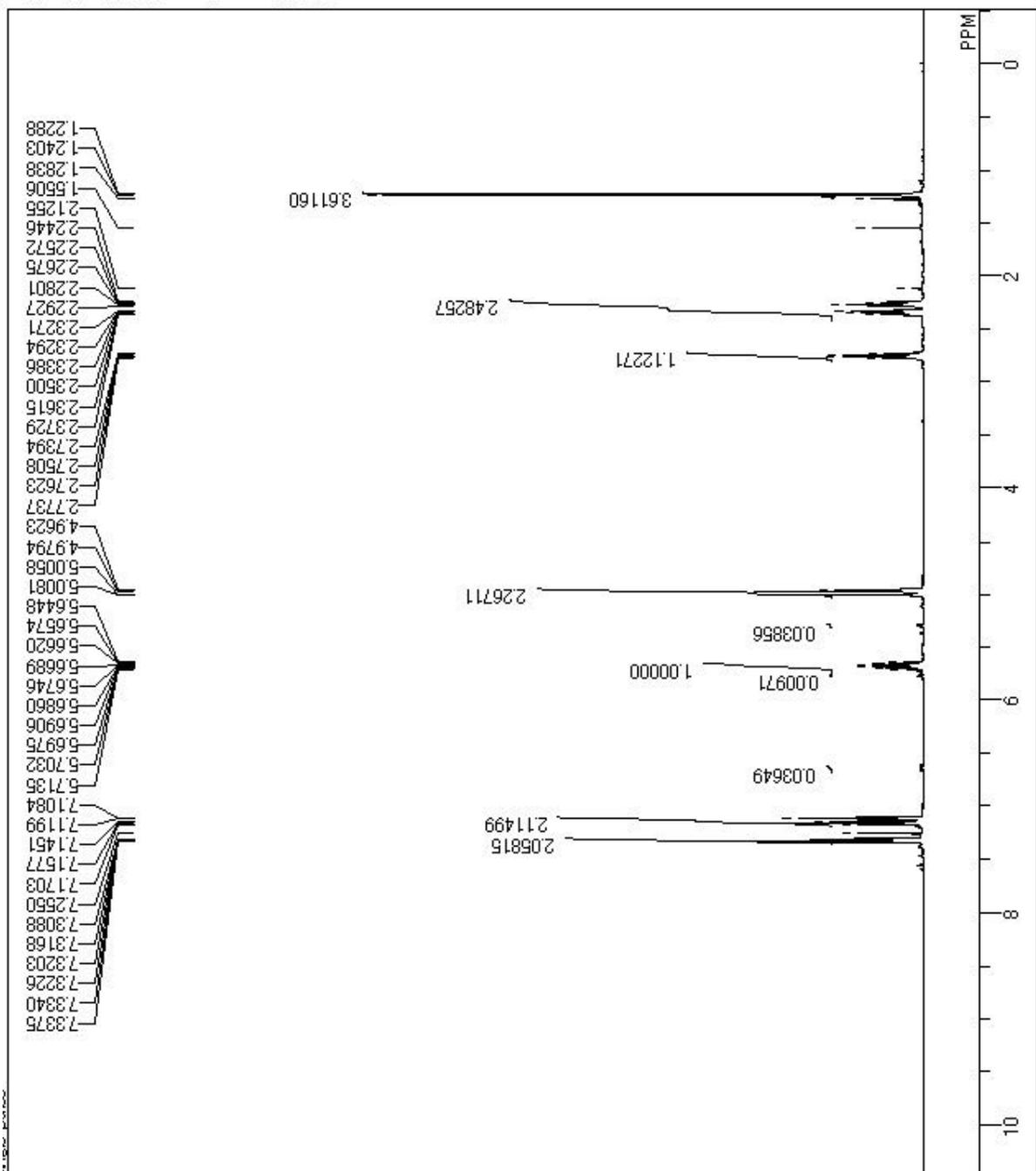
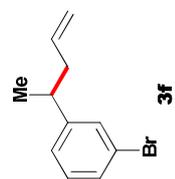
dao 511-1 c
17-12-2009 02:04:06
13C
sine, pulse dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
79
0.6921 sec
2.0000 sec
2.87 usec
1H
19.8 c
CDCL3
77.00 ppm
0.12 Hz
60



C:\Documents and Settings\deltaelta\デスク

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

1H
1H
single pulse
single pulse
600.17 MHz
5.30 KHz
5.47 Hz
16384
11261.26 Hz
8
1.4549 sec
2.0000 sec
6.50 usec
1H
17.8 c
CDCL3
0.00 ppm
1.20 Hz
40

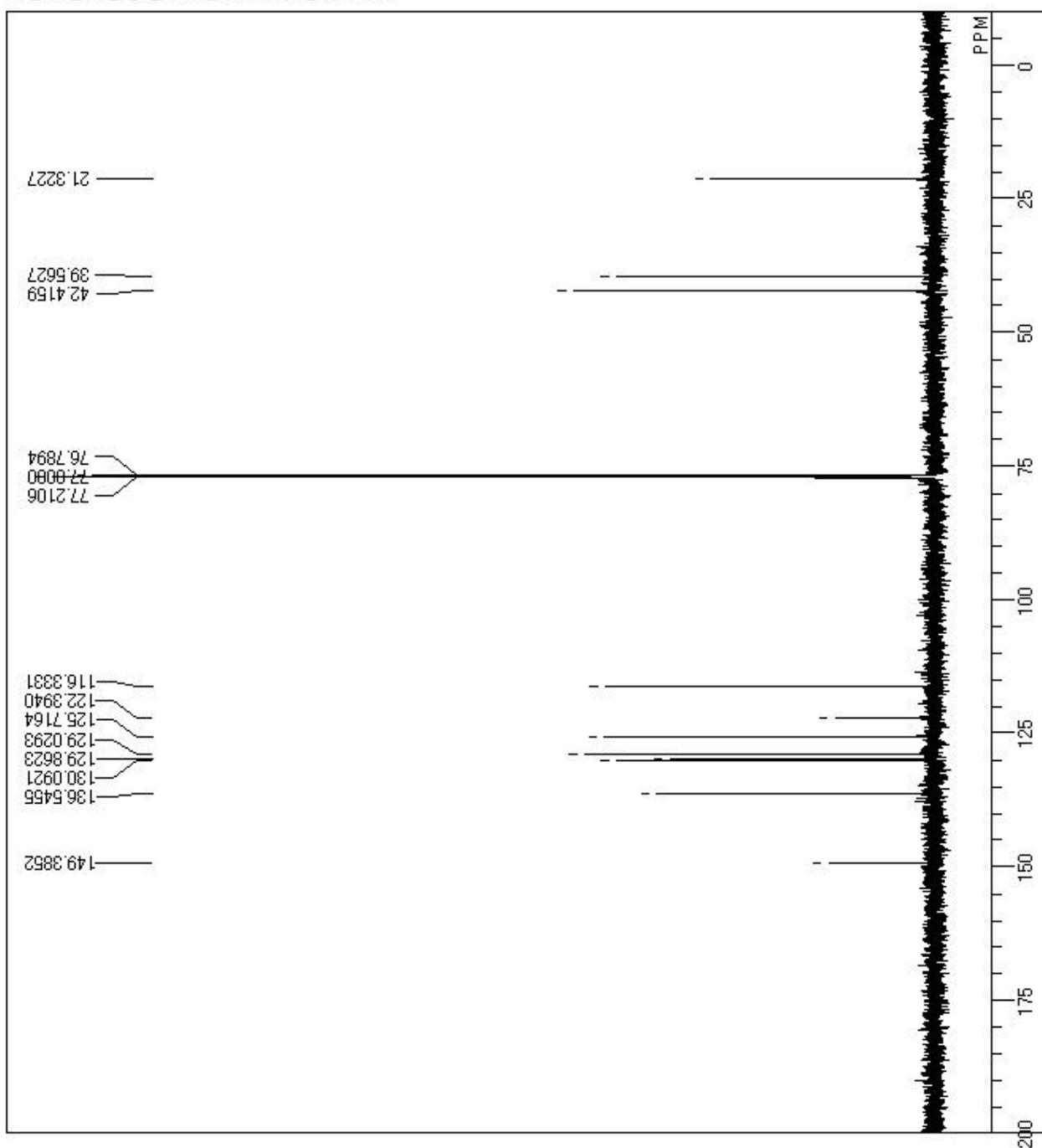
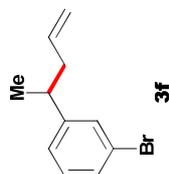


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D.
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

22-02-2010 03:00:45
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
69
0.6921 sec
2.0000 sec
2.87 usec
1H
18.9 c
CDCL3
77.00 ppm
1.20 Hz
60

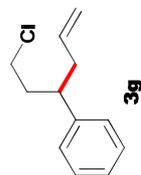
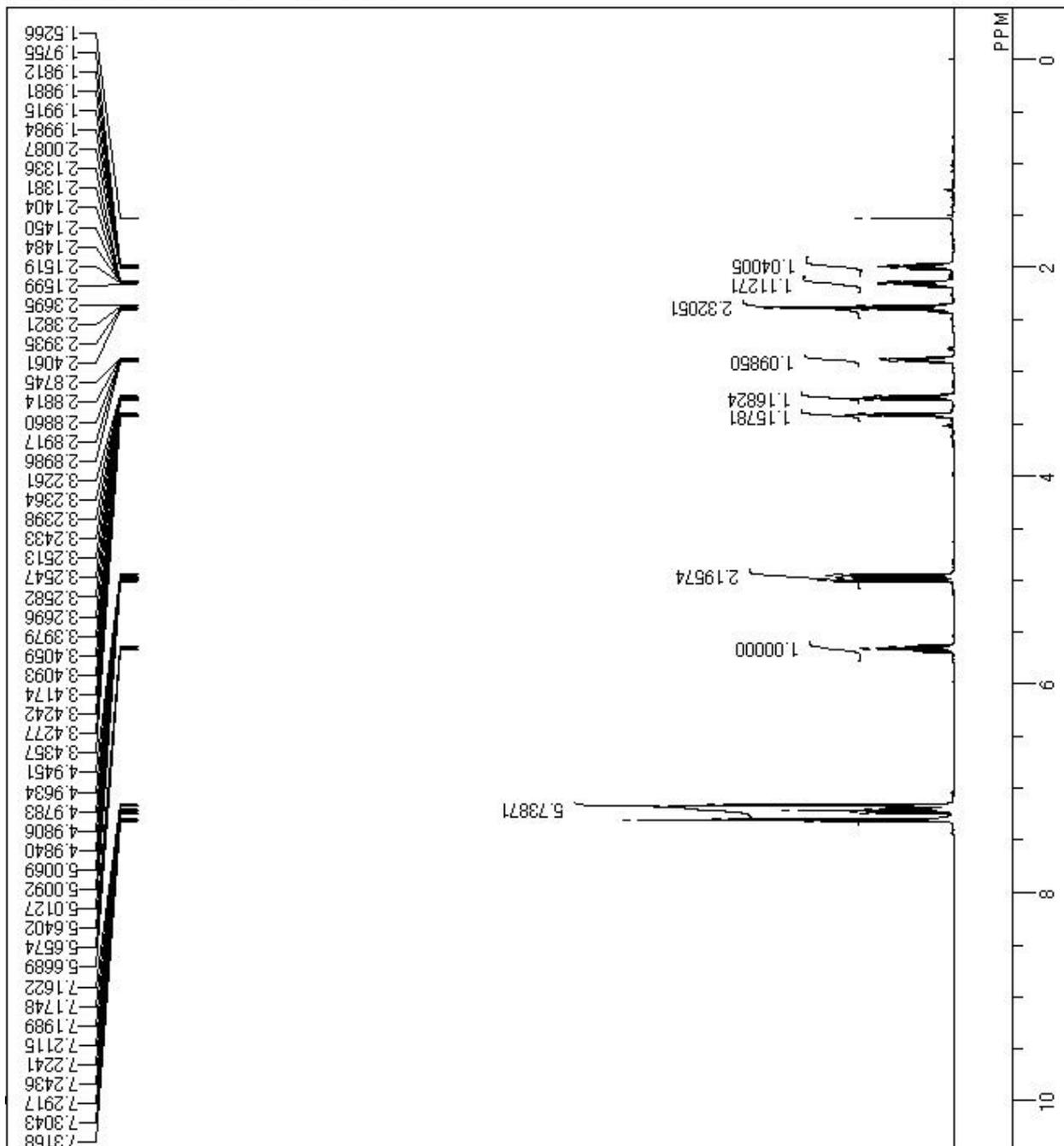


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D.
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

single_pulse
14-06-2010 20:30:46
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
16384
11261.26 Hz
8
1.4549 sec
2.0000 sec
6.50 usec
1H
21.9 c
CDCL3
0.00 ppm
0.12 Hz
38

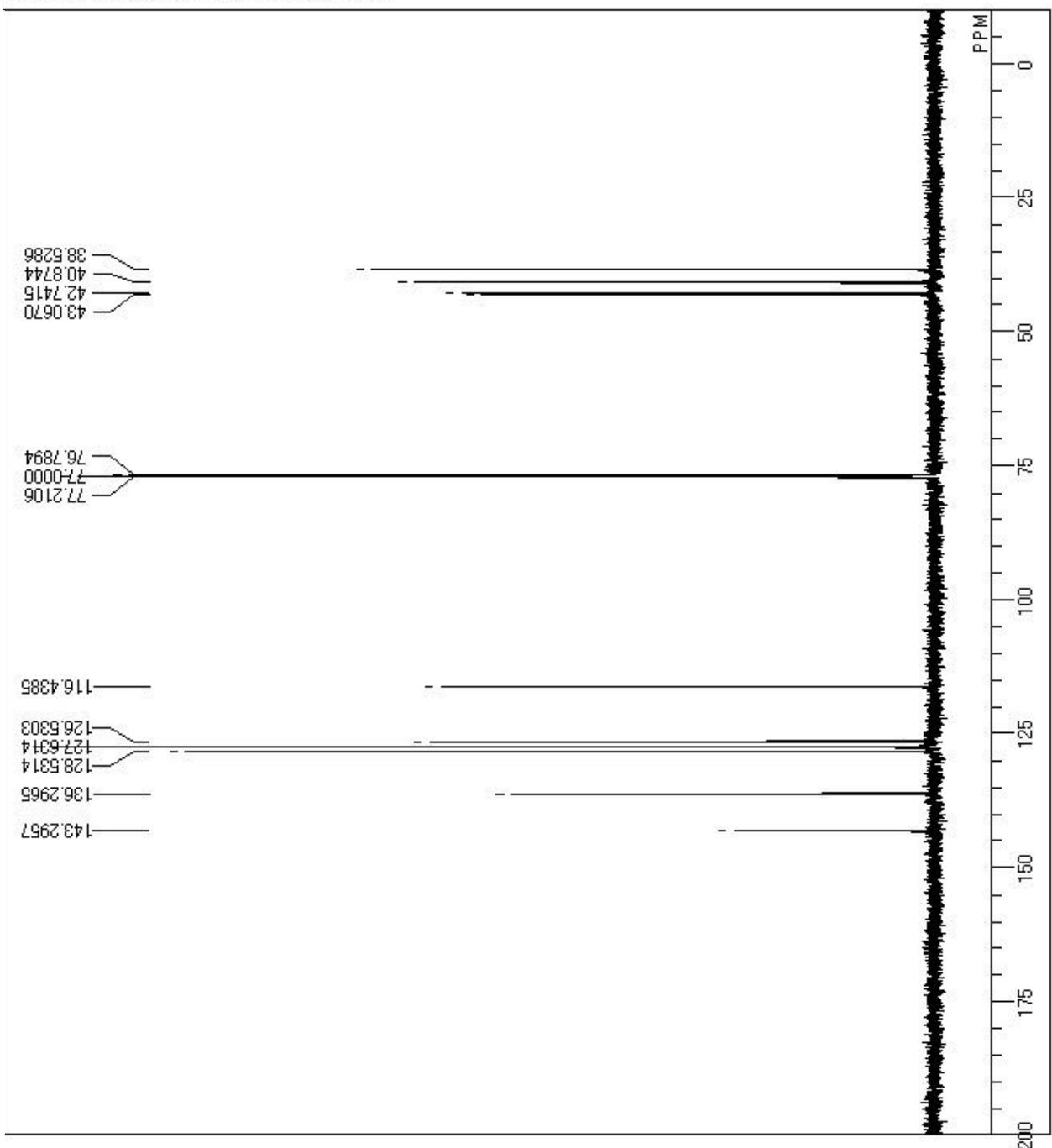
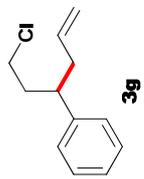


C:\Documents and Settings\delta\My [

14-06-2010 20:42:08

13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
152
0.6921 sec
2.0000 sec
2.87 usec
1H
22.9 c
CDCL3
77.00 ppm
0.12 Hz
60

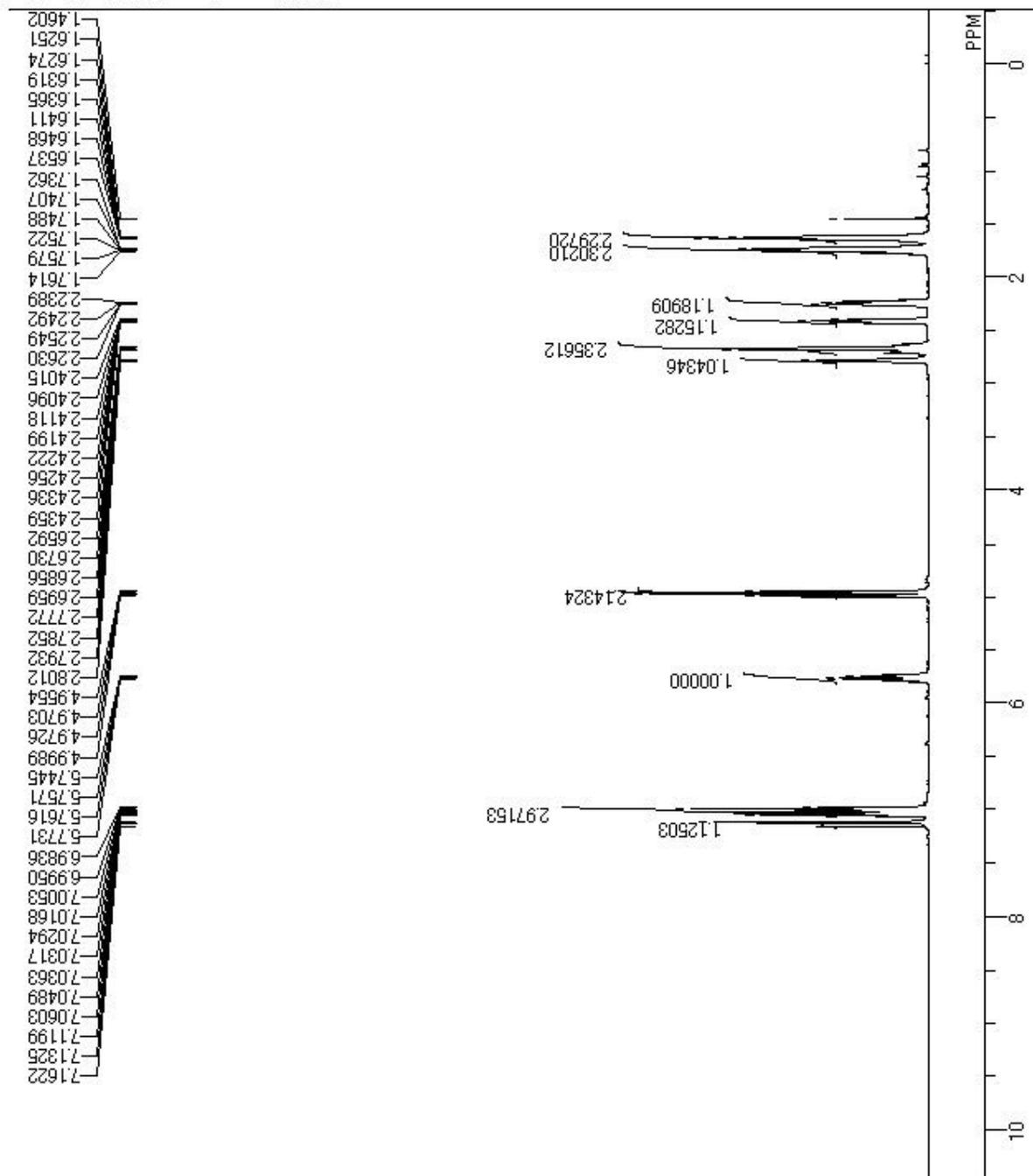
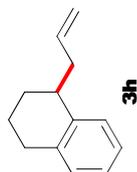
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



C:\Documents and Settings\delta\My Dr

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWT
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

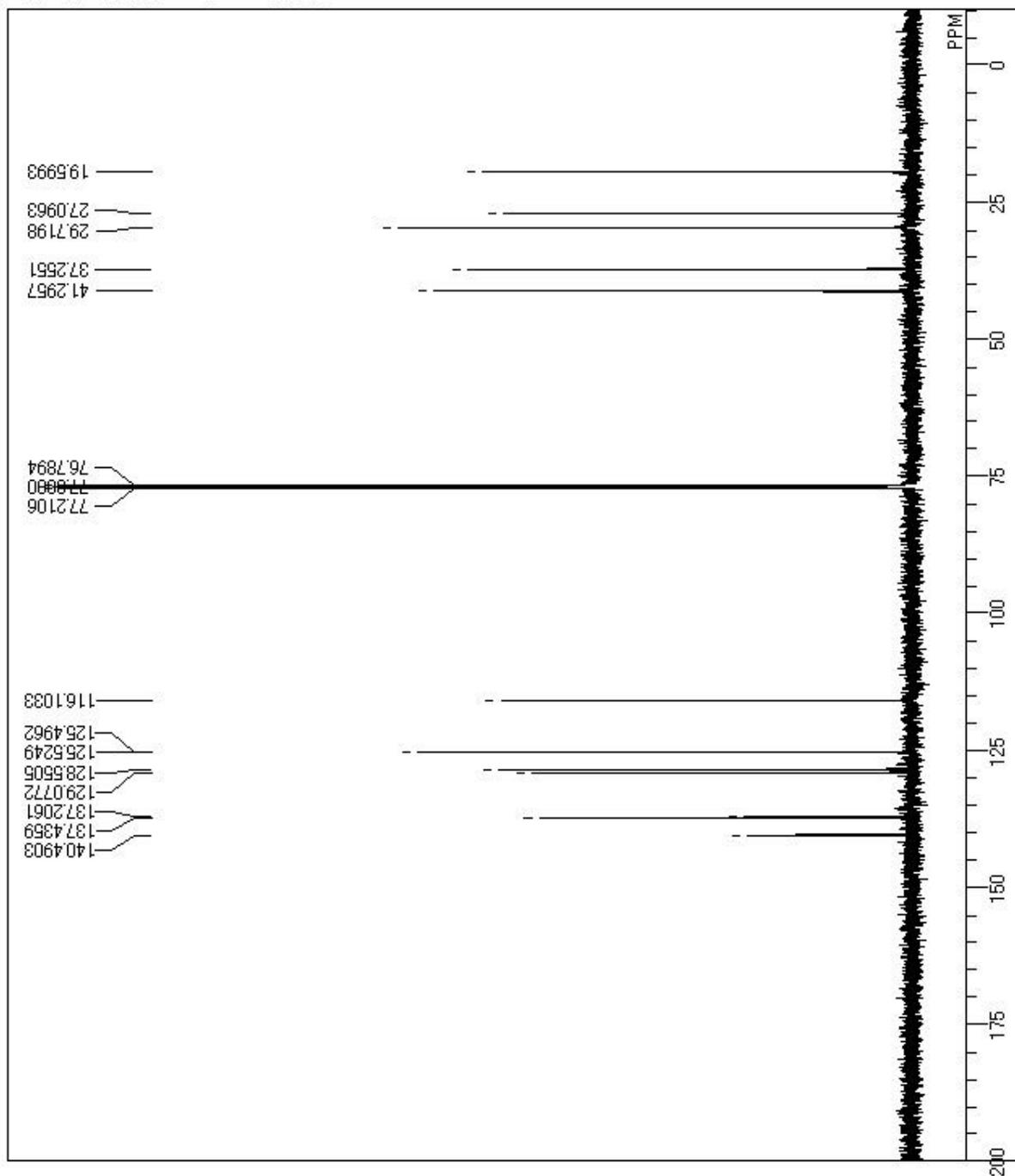
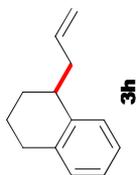
24-02-2010 00:59:37
1H
shale_pulsex2
600.17 MHz
5.30 KHz
5.47 Hz
16384
11261.26 Hz
8
1.4549 sec
2.0000 sec
6.50 usec
1H
18.4 c
CDCL3
0.00 ppm
1.20 Hz
36



L2-S

C:\Documents and Settings\delta\My Dr

DFILE
COMNT
DATIM 24-02-2010 01:06:60
OBNUC 13C
EXMOD sine pulse dec
OBFRQ 160.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.48 Hz
SCANS 117
ACQTM 0.6921 sec
PD 2.0000 sec
PWI 2.87 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



C:\Documents and Settings\delta\My I

single_pulse
07-06-2010 21:31:34

1H
single_pulse.ex2

600.17 MHz
5.30 KHz

5.47 Hz
16384

11261.26 Hz
8

1.4549 sec
2.0000 sec

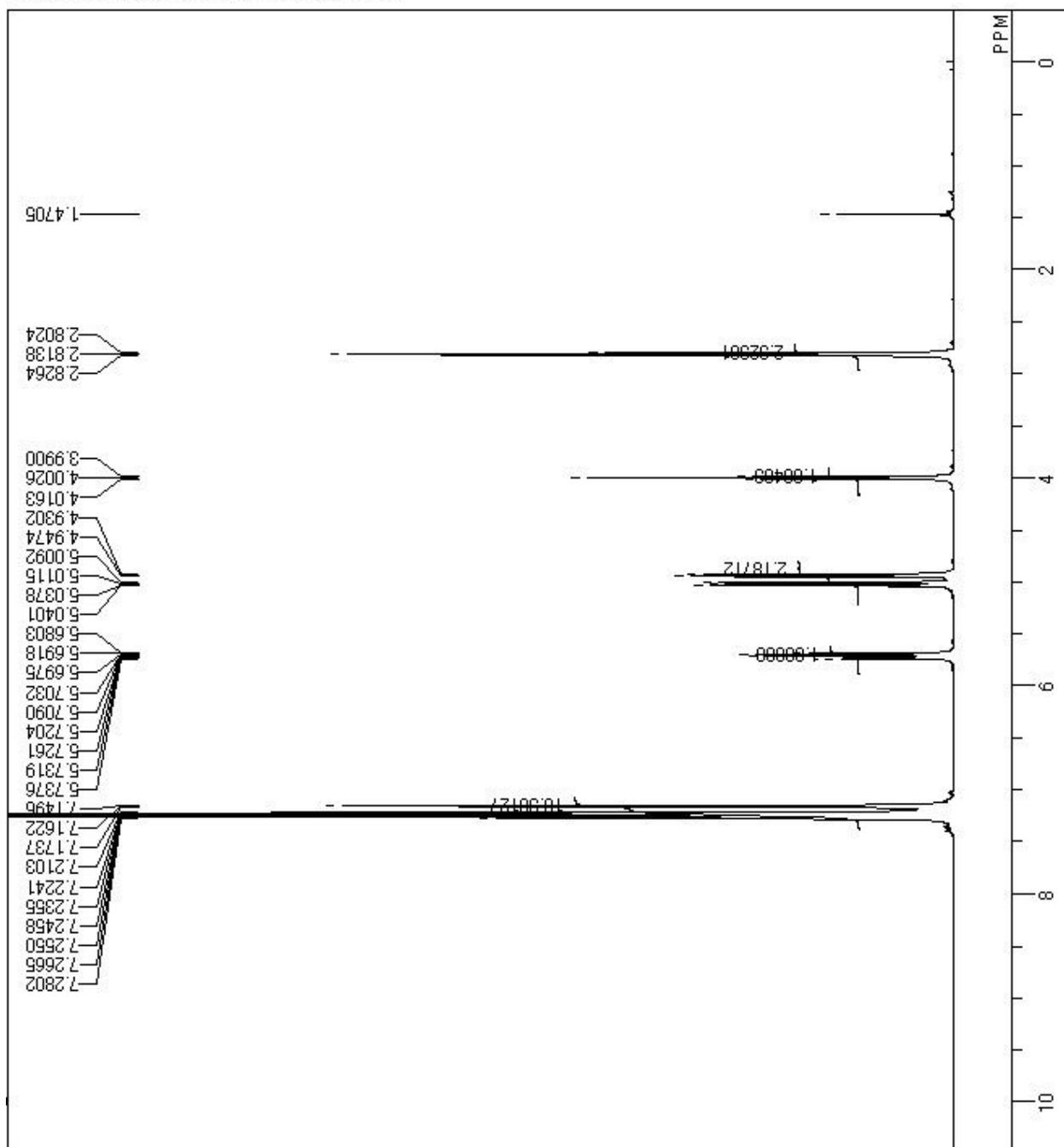
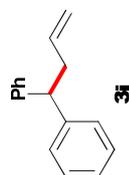
6.50 usec
1H

22.4 c
CDCL3

0.00 ppm
0.12 Hz

36
RGAIN

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D.
P.WI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

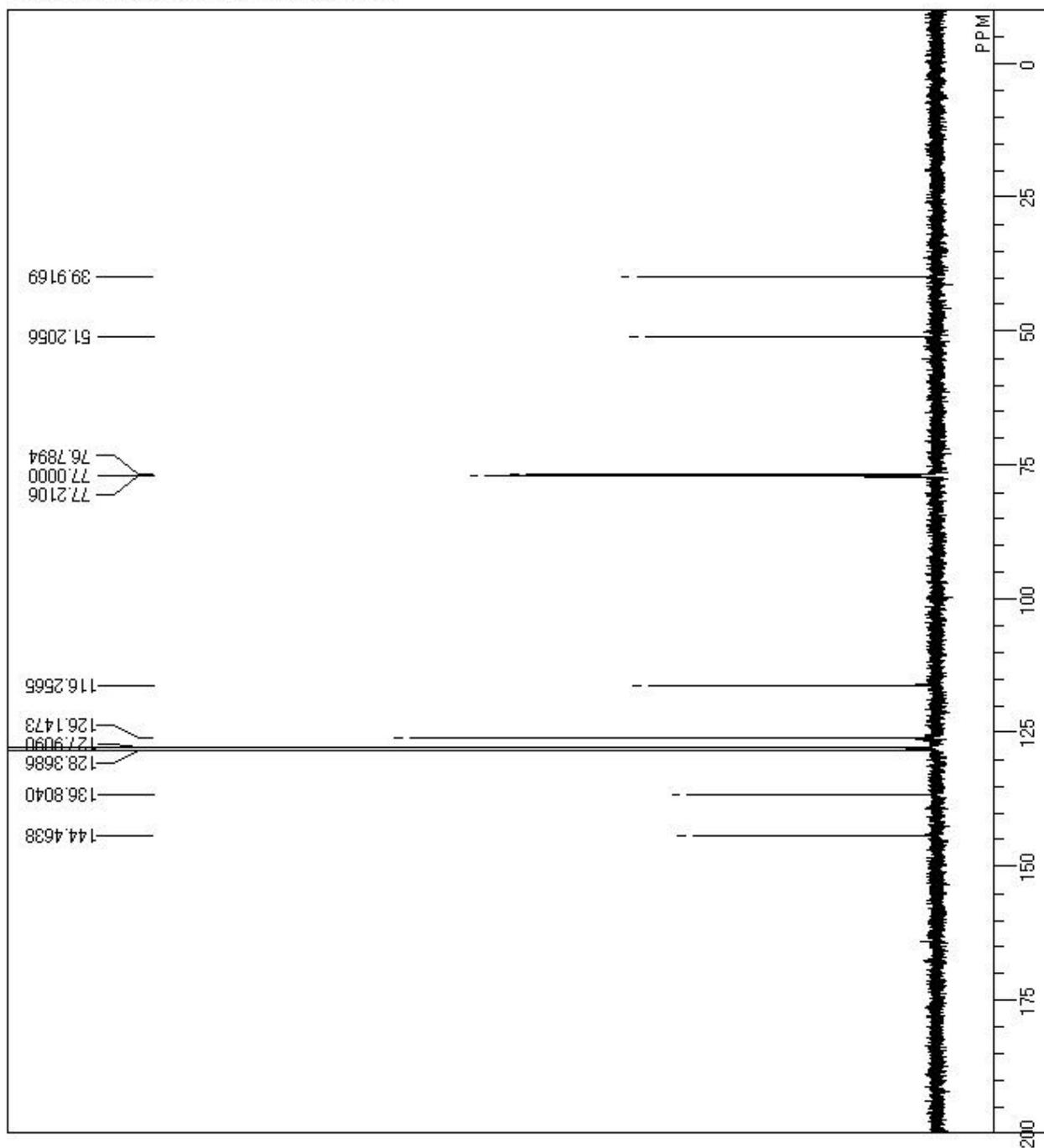
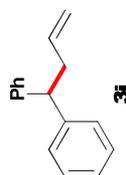


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

07-06-2010 21:36:19
13C
single pulse dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
50
0.6921 sec
2.0000 sec
2.87 usec
1H
23.1 c
CDCL3
77.00 ppm
0.12 Hz
60

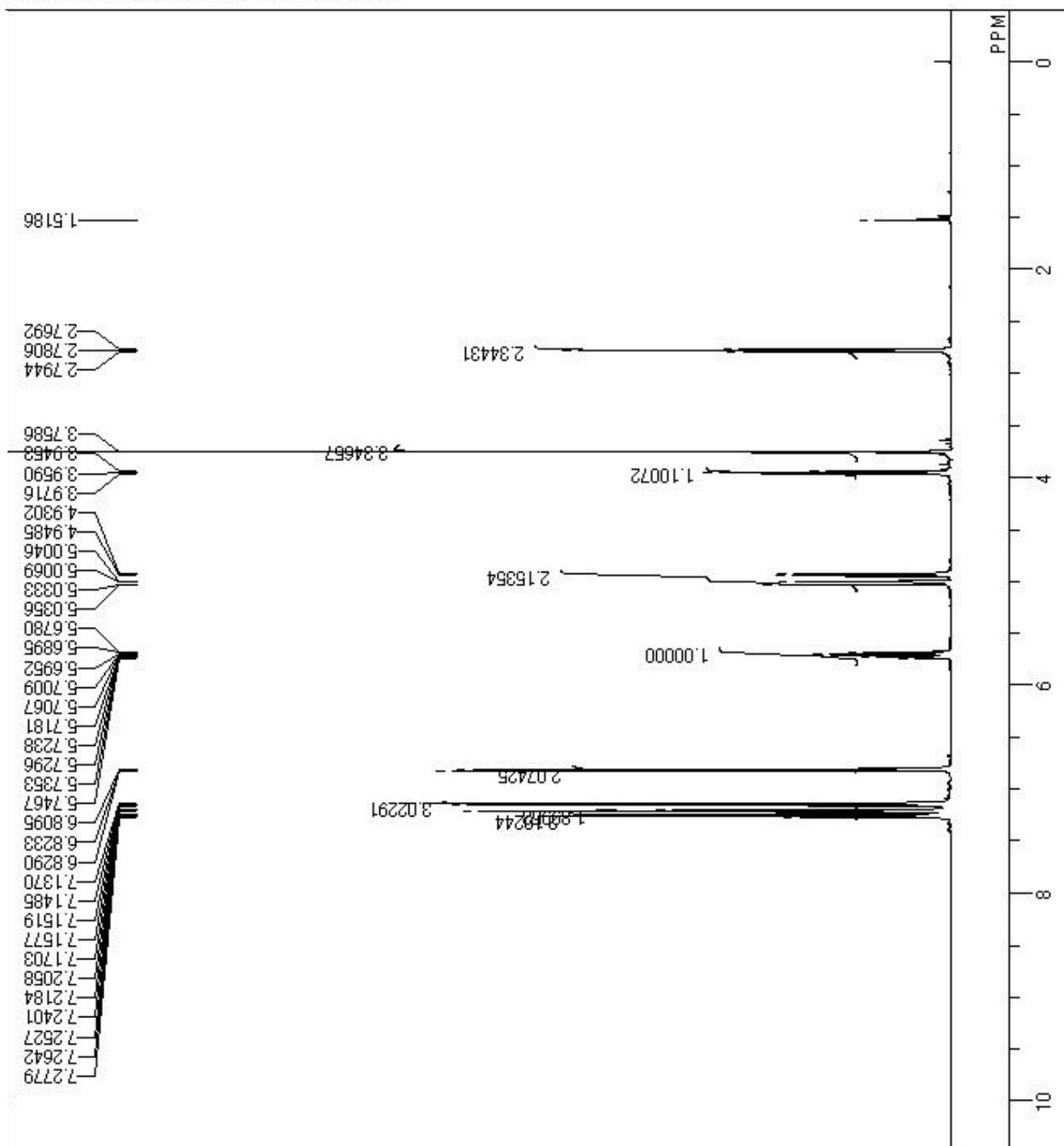
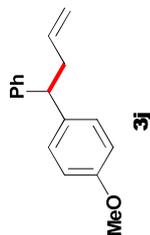


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
P.W1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

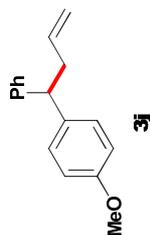
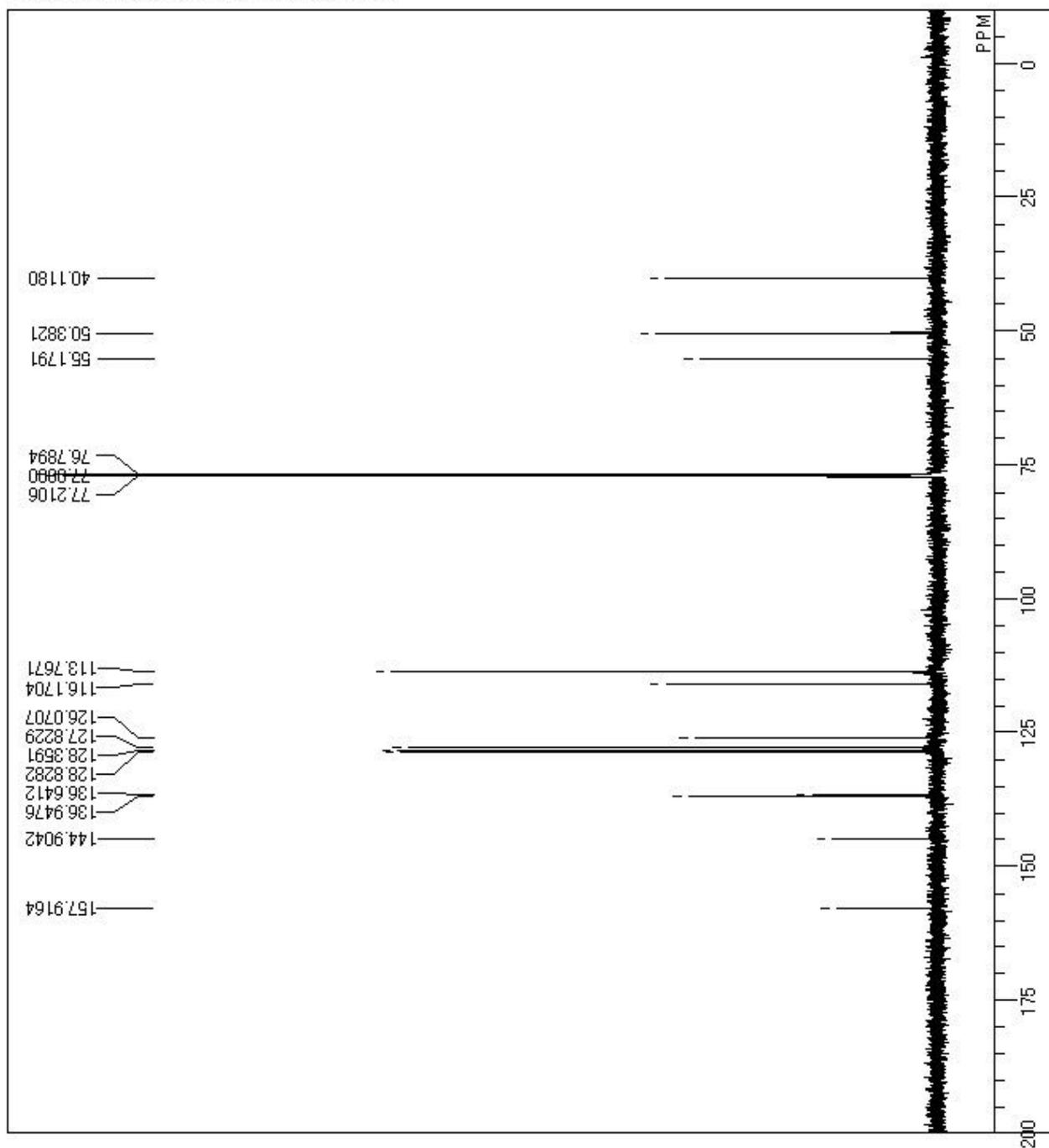
single_pulse
18-06-2010 03:52:01
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
16384
11261.26 Hz
8
1.4549 sec
2.0000 sec
6.50 usec
1H
25.4 c
CDCL3
0.00 ppm
0.12 Hz
40



C:\Documents and Settings\delta\My I

18-06-2010 04:00:05
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
129
0.6921 sec
2.0000 sec
2.87 usec
1H
26.2 c
CDCl3
77.00 ppm
0.12 Hz
60

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



C:\Documents and Settings\delta\My I

single_pulse
07-06-2010 22:04:29

1H
single_pulse.ex2

600.17 MHz

5.30 KHz

5.47 Hz

16384

11261.26 Hz

8

1.4549 sec

2.0000 sec

6.50 usec

1H

22.7 c

CDCL3

0.00 ppm

0.12 Hz

42

DFILE

COMNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

PD

PW1

IRNUC

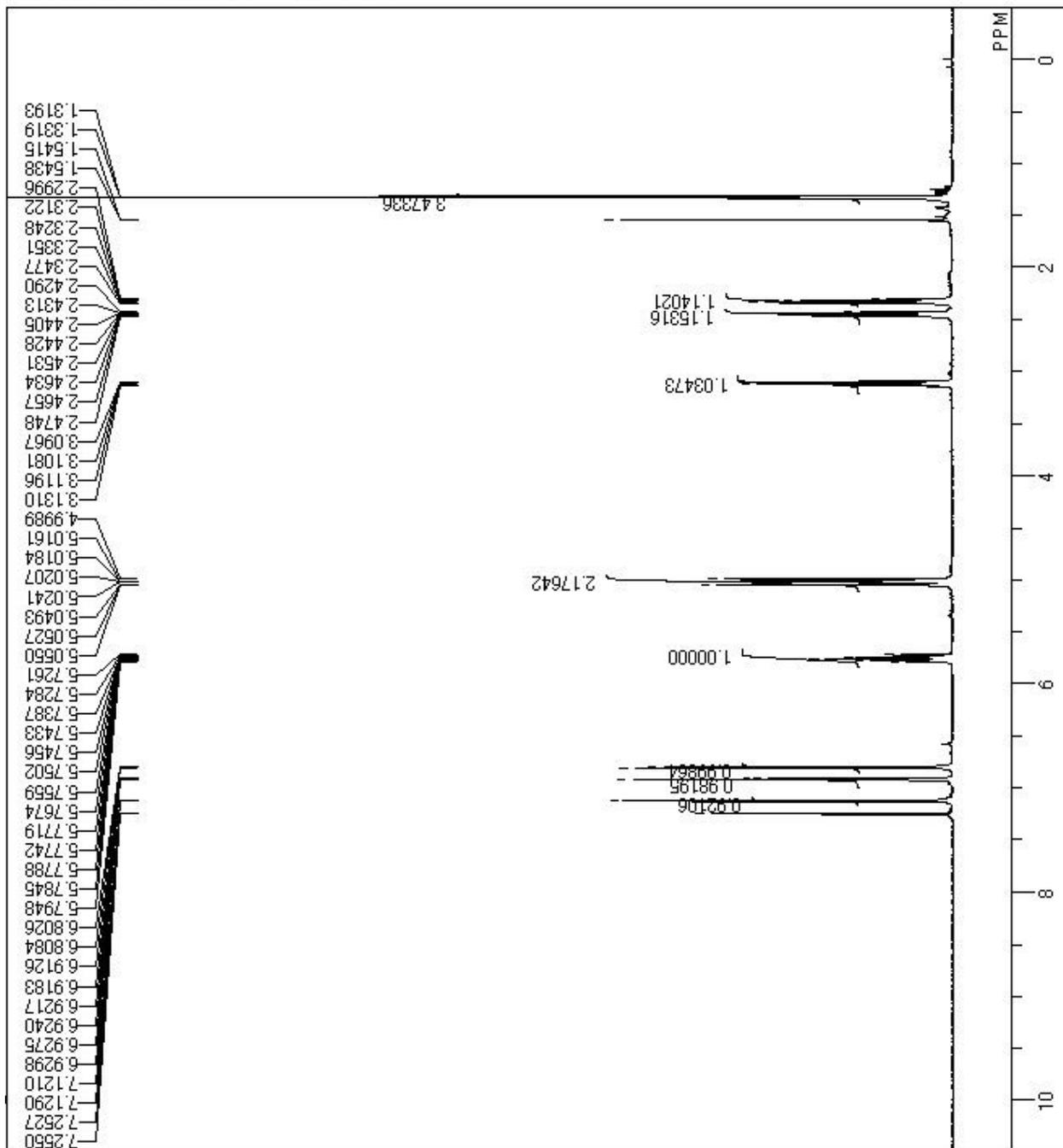
CTEMP

SLVNT

EXREF

BF

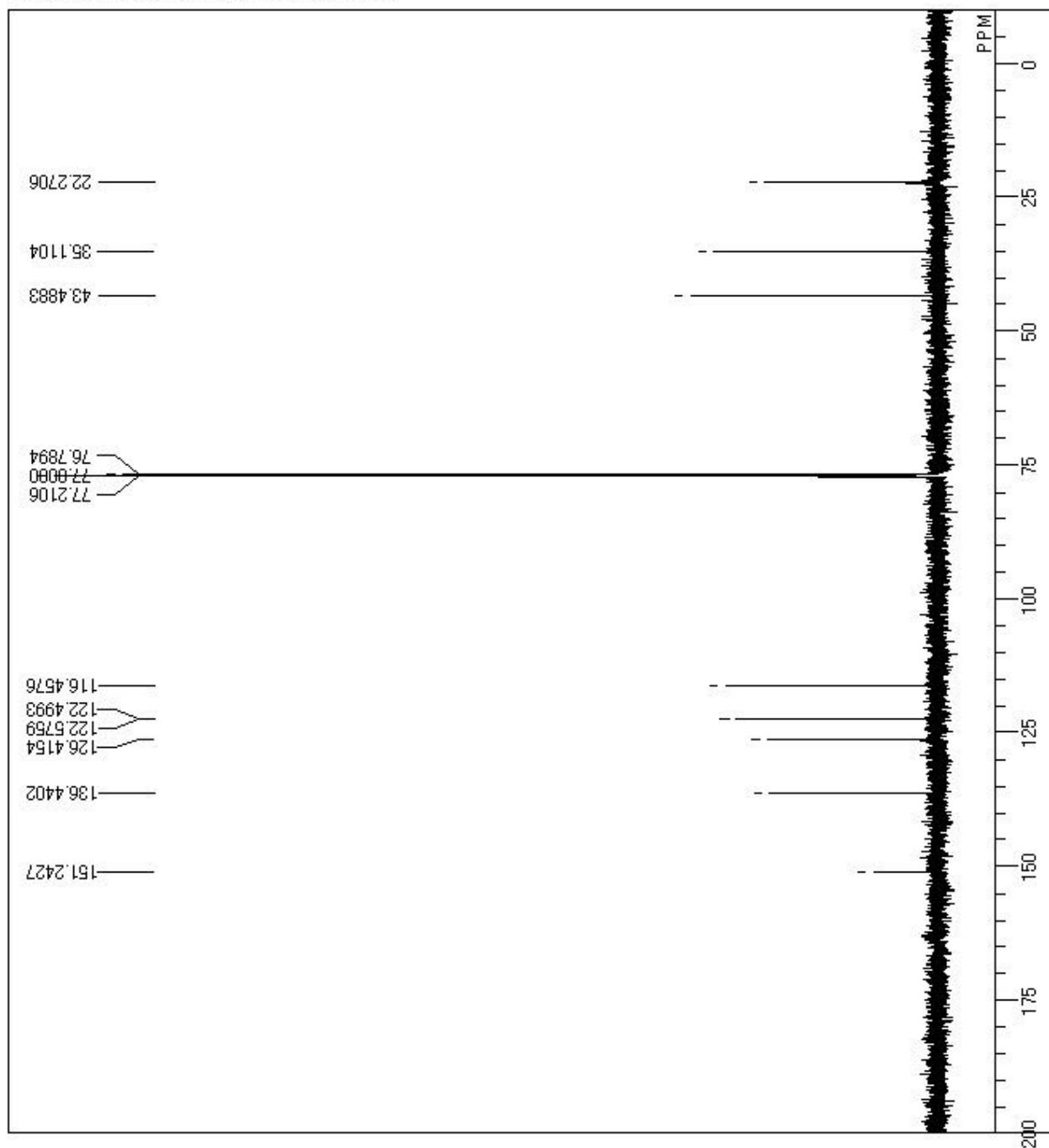
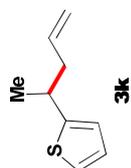
RGAIN



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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM 07-06-2010 22:10:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 32768
FREQU 47348.48 Hz
SCANS 86
ACQTM 0.6921 sec
P.D. 2.0000 sec
PWI 2.87 usec
IRNUC 1H
CTEMP 23.5 c
SOLVT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

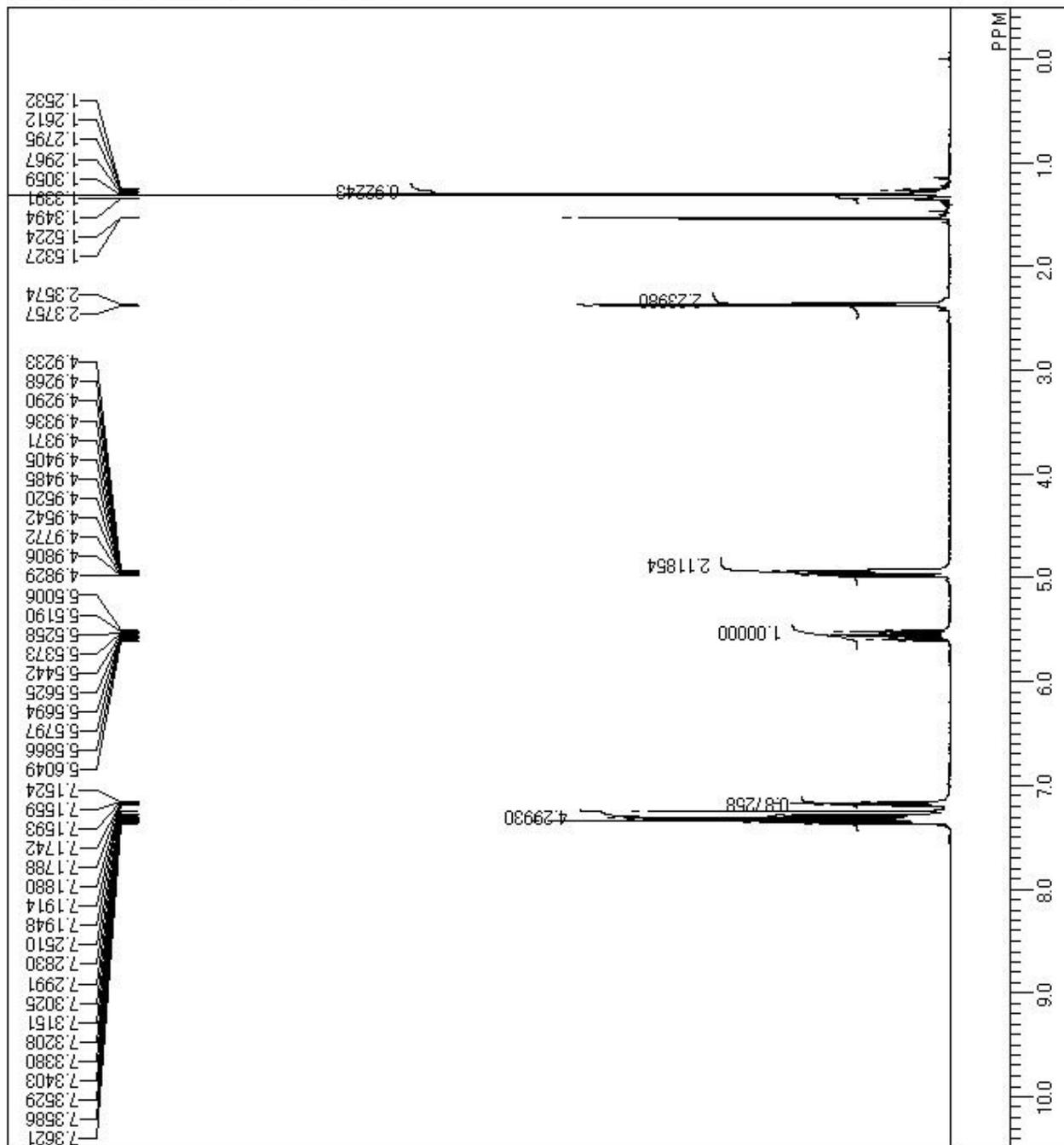
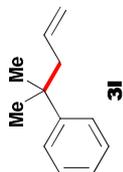


S-34

C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

22-06-2010 16:23:39
1H
single_pulse_ex2
399.78 MHz
4.19 KHz
7.29 Hz
16384
7503.00 Hz
8
2.1837 sec
2.0000 sec
6.50 usec
1H
406.5 c
CDCl3
0.00 ppm
1.20 Hz
34

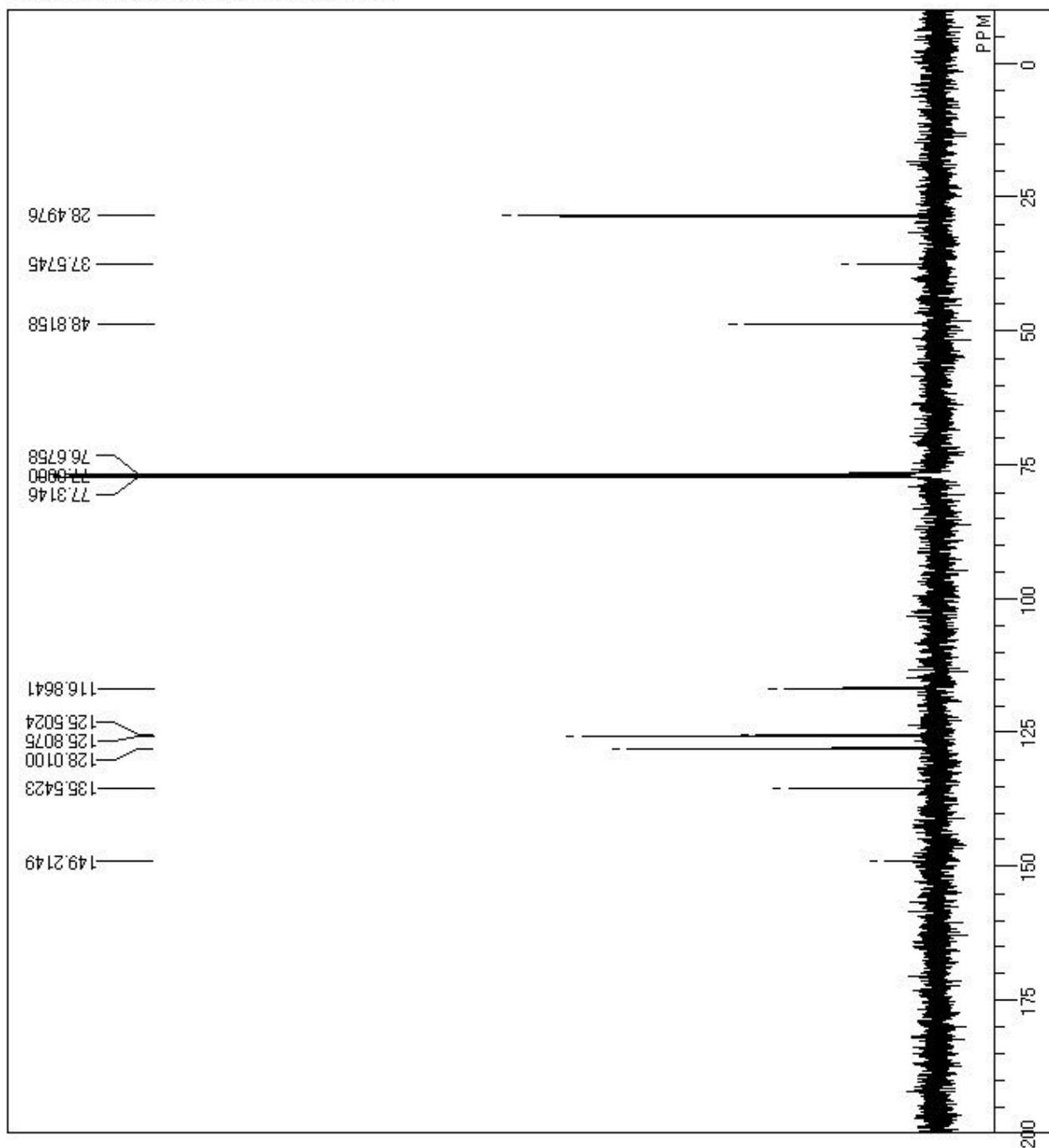
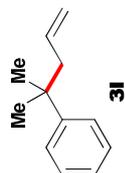


C:\Documents and Settings\delta\My I

22-06-2010 16:30:10

13C
single_pulse_dec
100.53 MHz
5.35 KHz
5.86 Hz
32768
31407.04 Hz
62
1.0433 sec
2.0000 sec
3.50 usec
1H
406.5 c
CDCl3
77.00 ppm
1.20 Hz
56

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



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C:\Documents and Settings\delta\My I

single_pulse
25-05-2010 01:19:15

1H
single_pulse.ex2

600.17 MHz
5.30 KHz

5.47 Hz
16384

11261.26 Hz
8

1.4549 sec
2.0000 sec

6.50 usec
1H

21.3 c
CDCL3

0.00 ppm
0.20 Hz

38
RGAIN

DFILE

COMNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

P.D.

PW1

IRNUC

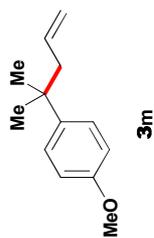
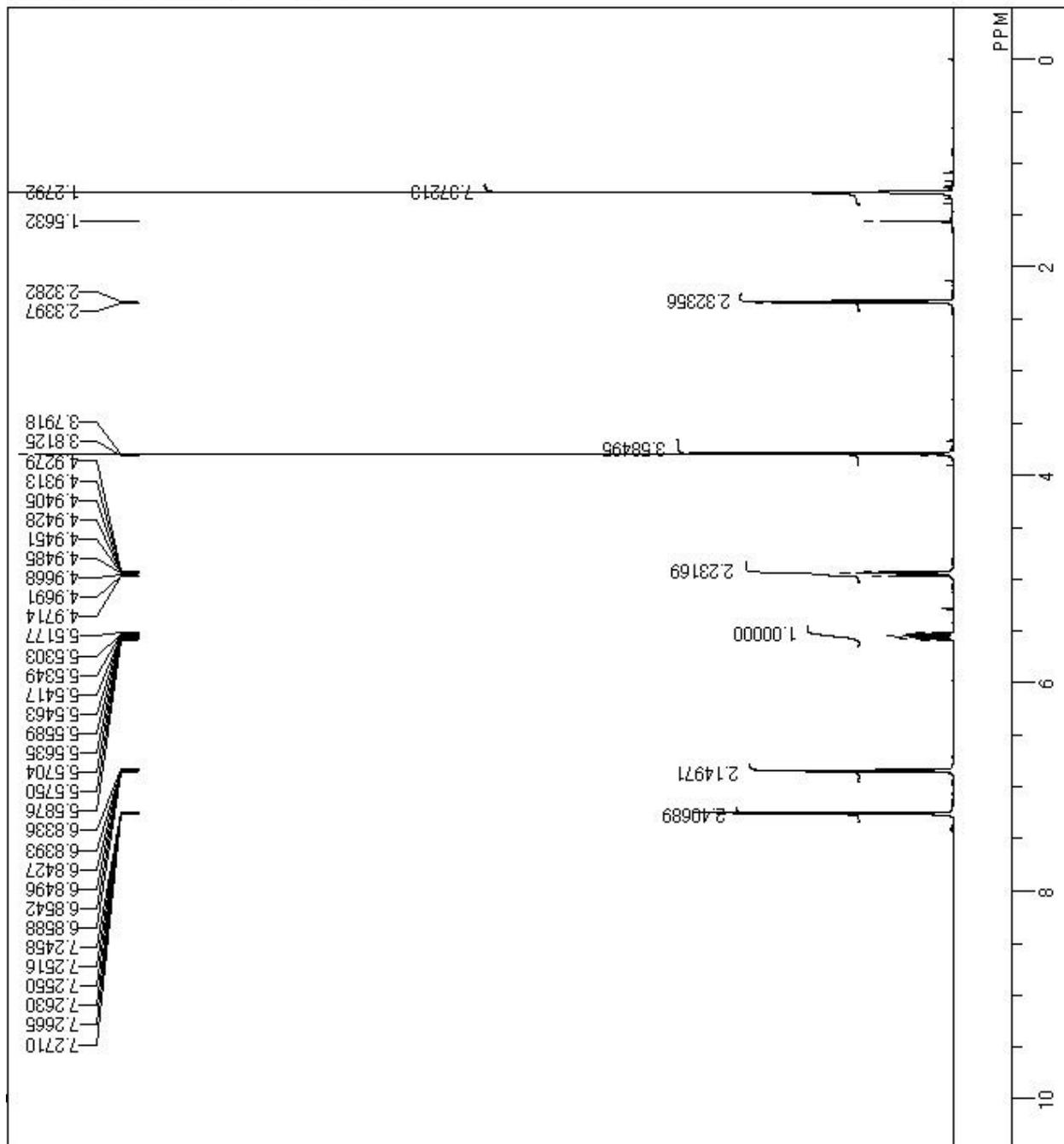
CTEMP

SLVNT

EXREF

BF

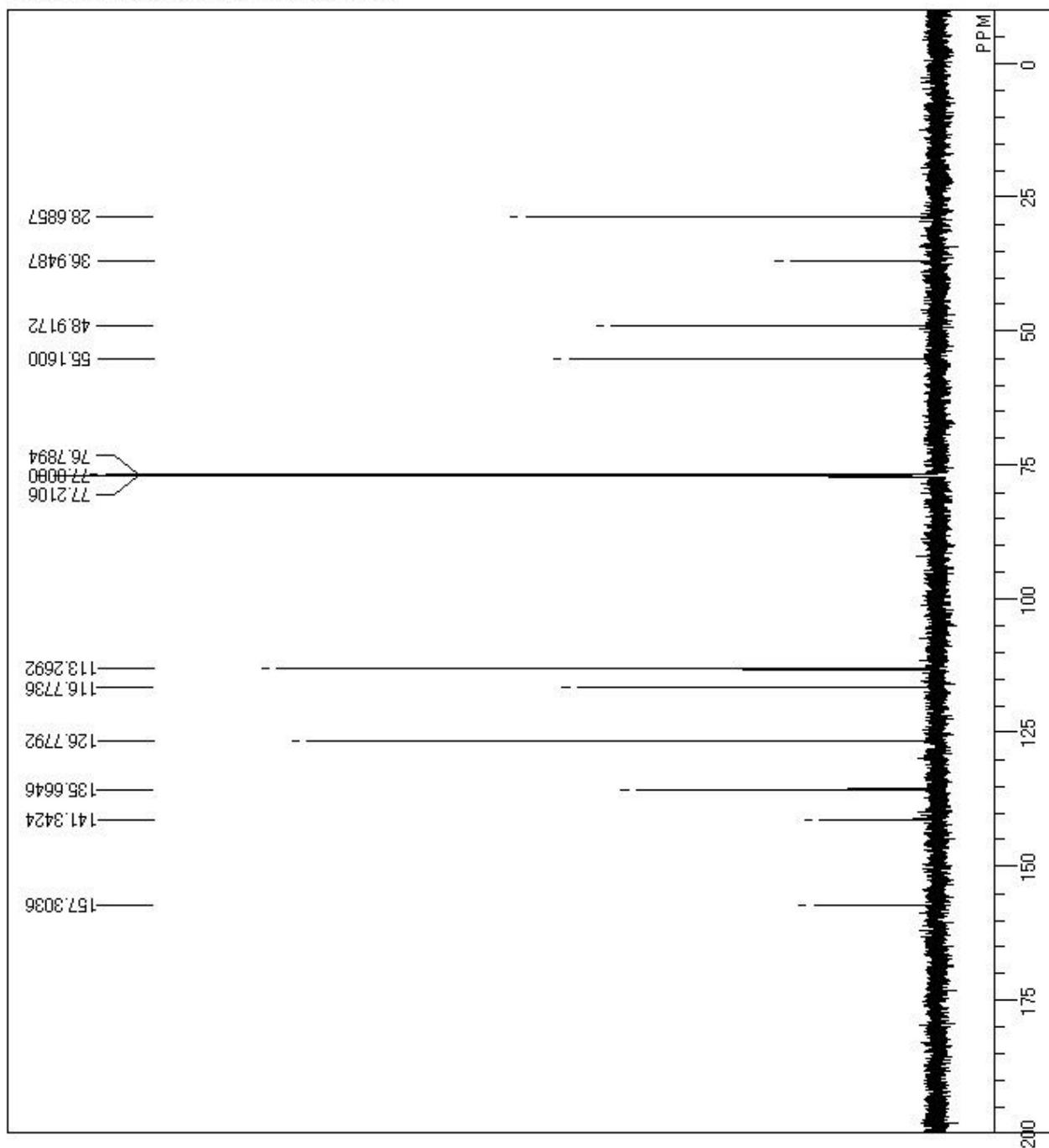
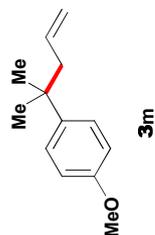
RGAIN



C:\Documents and Settings\delta\My [

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

25-05-2010 01:25:02
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
60
0.6921 sec
2.0000 sec
2.87 usec
1H
22.0 c
CDCl3
77.00 ppm
0.20 Hz
60

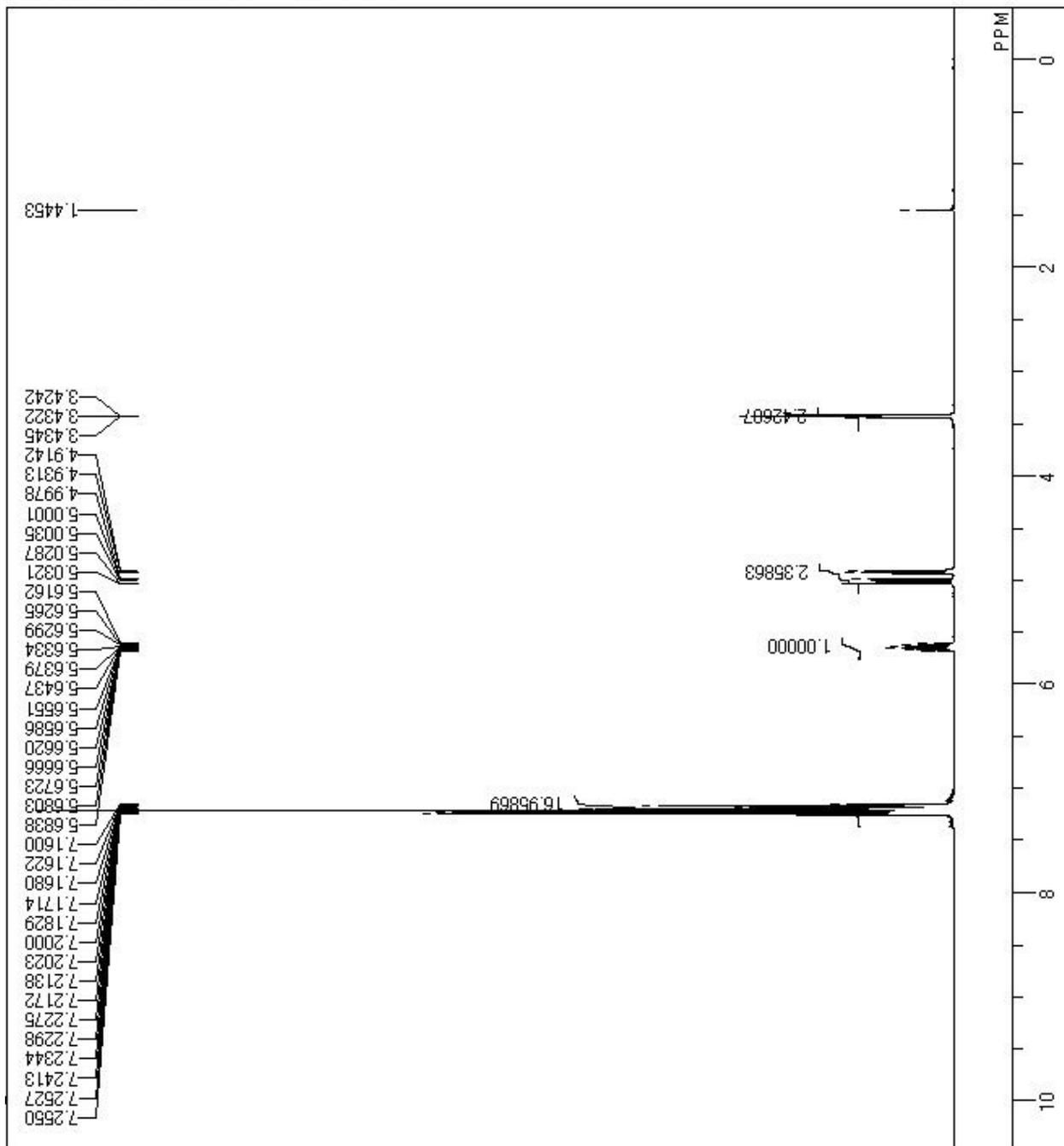
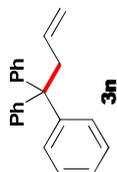


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
DNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D.
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

single_pulse
07-06-2010 21:40:47
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
16384
11261.26 Hz
8
1.4549 sec
2.0000 sec
6.50 usec
1H
22.6 c
CDCL3
0.00 ppm
0.12 Hz
32

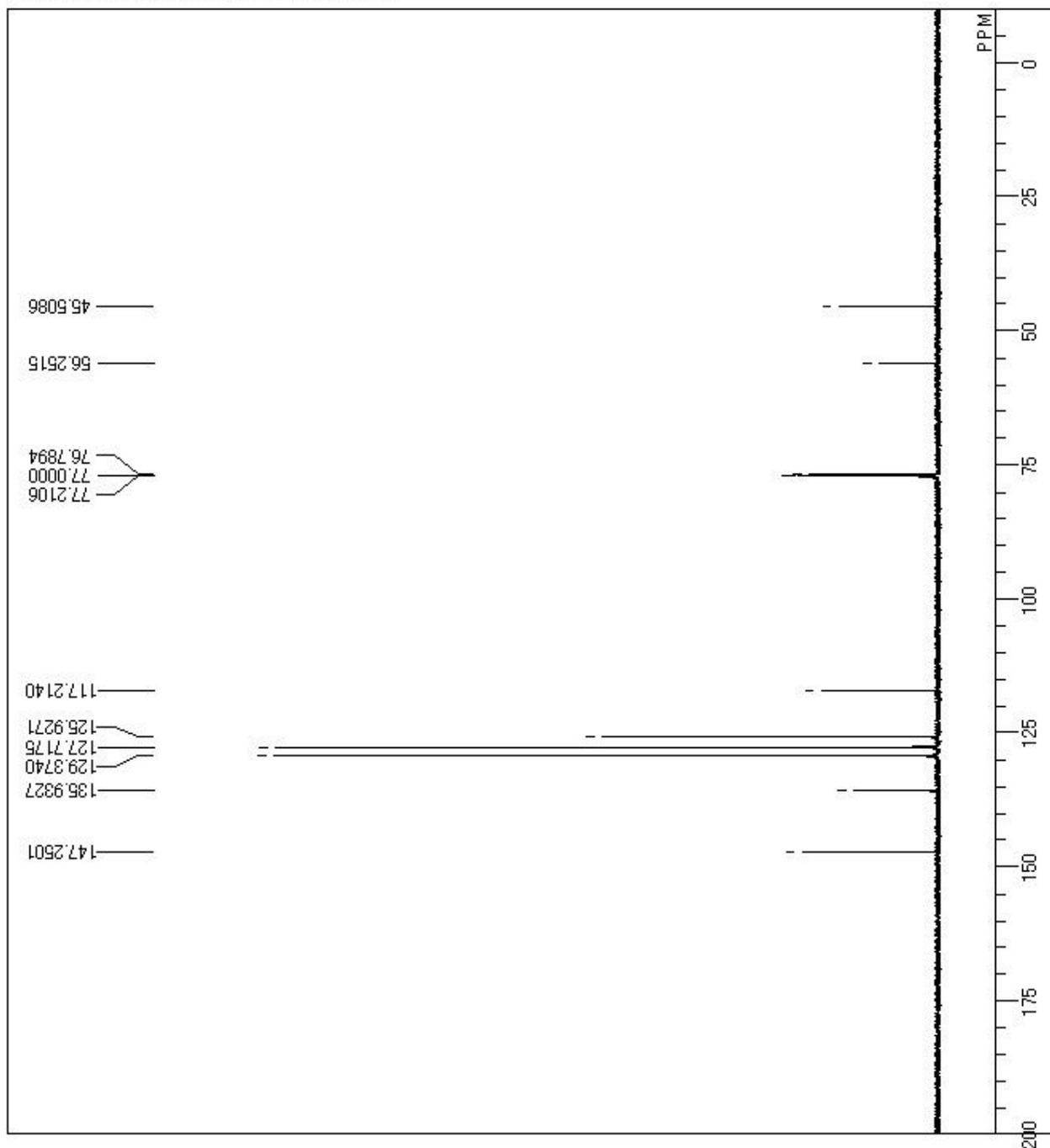
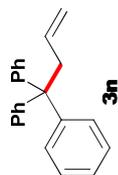


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

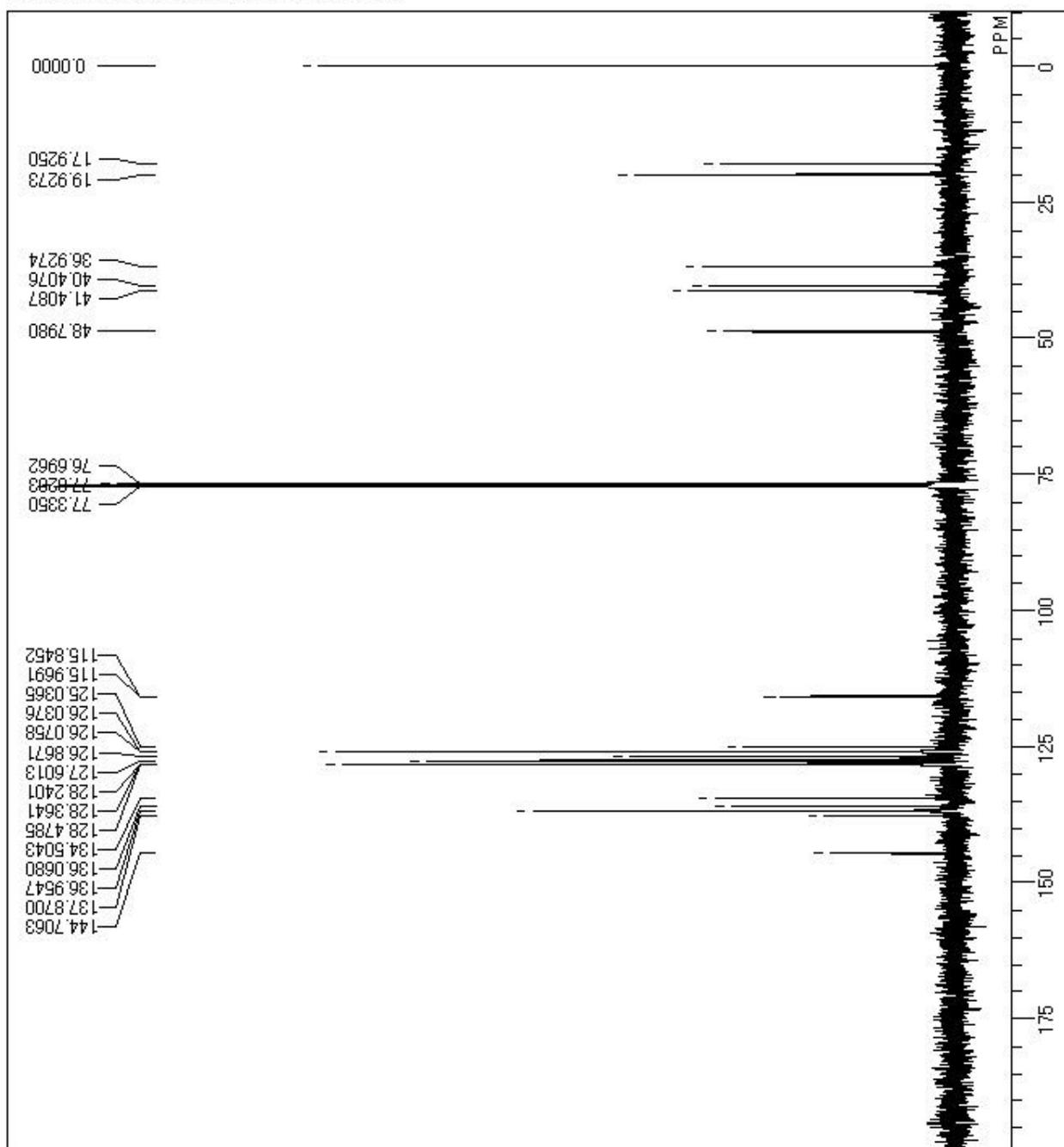
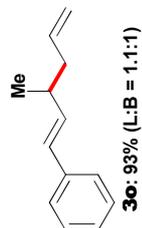
07-06-2010 21:45:28
13C
single_pulse_dec
150.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
E4
0.6921 sec
2.0000 sec
2.87 usec
1H
23.4 c
CDCL3
77.00 ppm
0.12 Hz
60



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C:\Documents and Settings\delta\タテス

DFILE
COMNT
DATIM 20-08-2010 19:41:60
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.63 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40961
FREQU 39259.39 Hz
SCANS 98
ACQTM 1.0433 sec
PD 2.0000 sec
PWI 3.50 usec
IRNUC 1H
CTEMP 409.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.71 Hz
RGAIN 56



C:\Documents and Settings\delta\My I

single_pulse
11-02-2010 05:21:32

1H
single_pulse.ex2

600.17 MHz
5.30 KHz

5.47 Hz
13107

9008.87 Hz
8

1.4549 sec
2.0000 sec

6.50 usec
1H

21.4 c
CDCL3

0.00 ppm
1.20 Hz

42
RGAIN

DFILE

COMNT

DATIM

OBNUC

EXMOD

OBFRQ

OBSET

OBFIN

POINT

FREQU

SCANS

ACQTM

P.D.

PW1

IRNUC

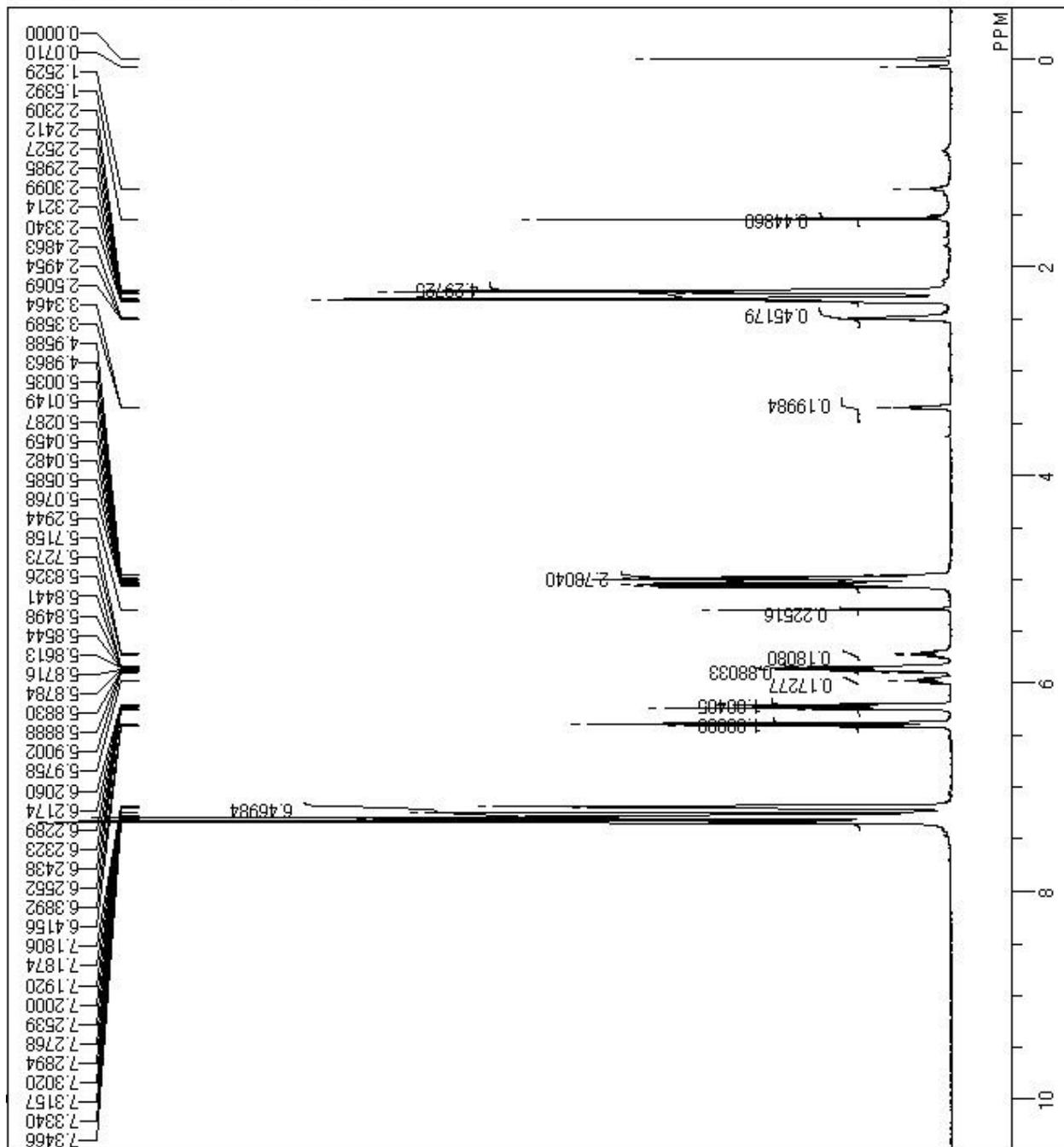
CTEMP

SLVNT

EXREF

BF

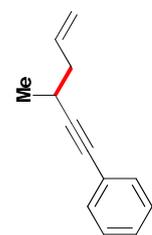
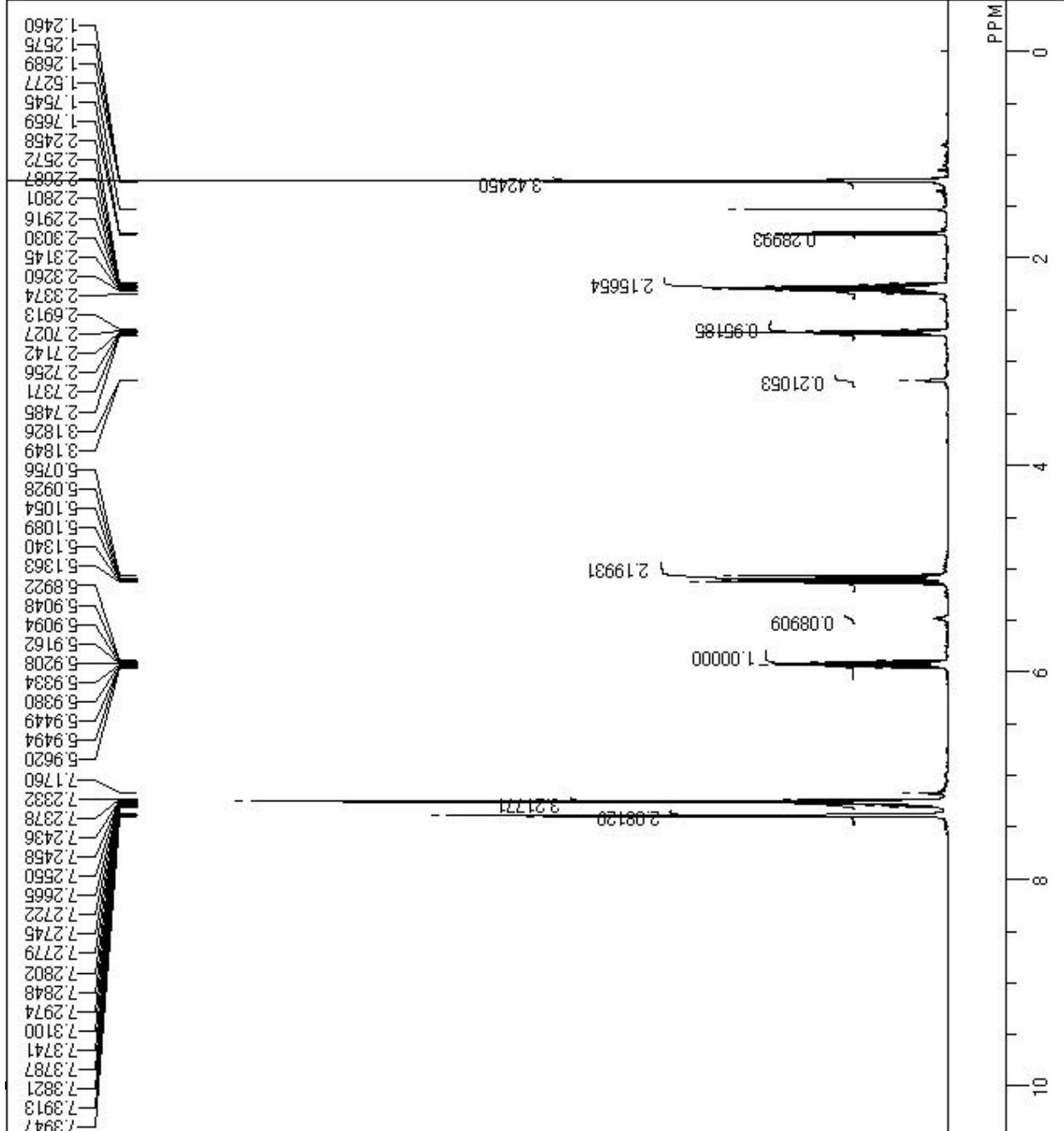
RGAIN



3p, 30% (L:B = 6:1)

C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D.
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



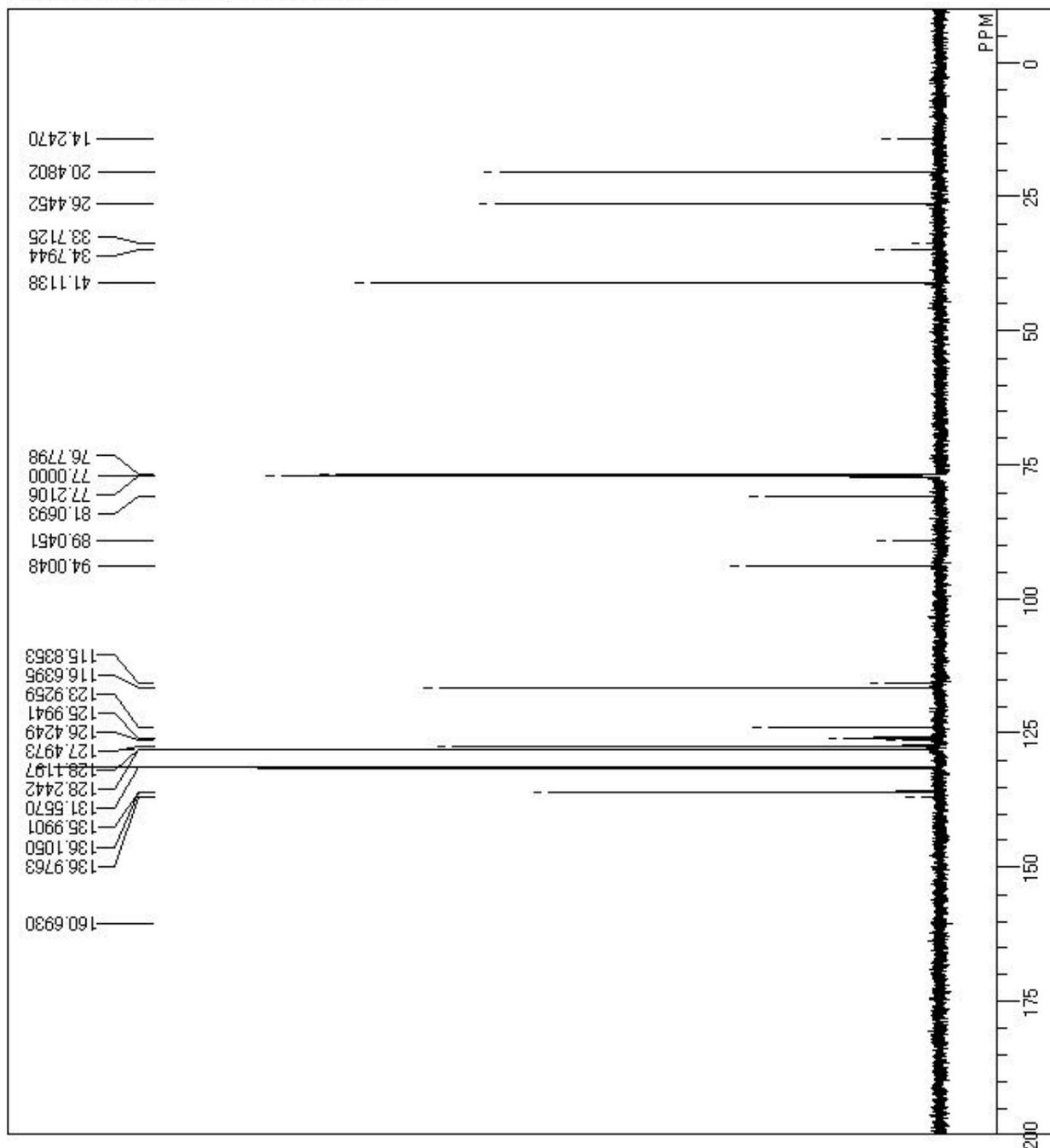
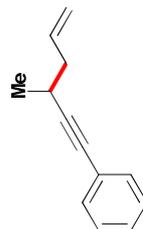
single_pulse
18-06-2010 03:38:51
1H
single_pulse.ex2
600.17 MHz
5.30 KHz
5.47 Hz
16384
11261.26 Hz

1.4549 sec
2.0000 sec
6.50 usec
1H
25.2 c
CDCL3
0.00 ppm
0.12 Hz
36

C:\Documents and Settings\delta\My I

DFILE
COMINT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

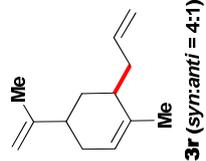
18-06-2010 03:48:03
13C
single_pulse_dec
160.92 MHz
8.52 KHz
1.74 Hz
32768
47348.48 Hz
147
0.6921 sec
2.0000 sec
2.87 usec
1H
26.1 c
CDCL3
77.00 ppm
0.12 Hz
60



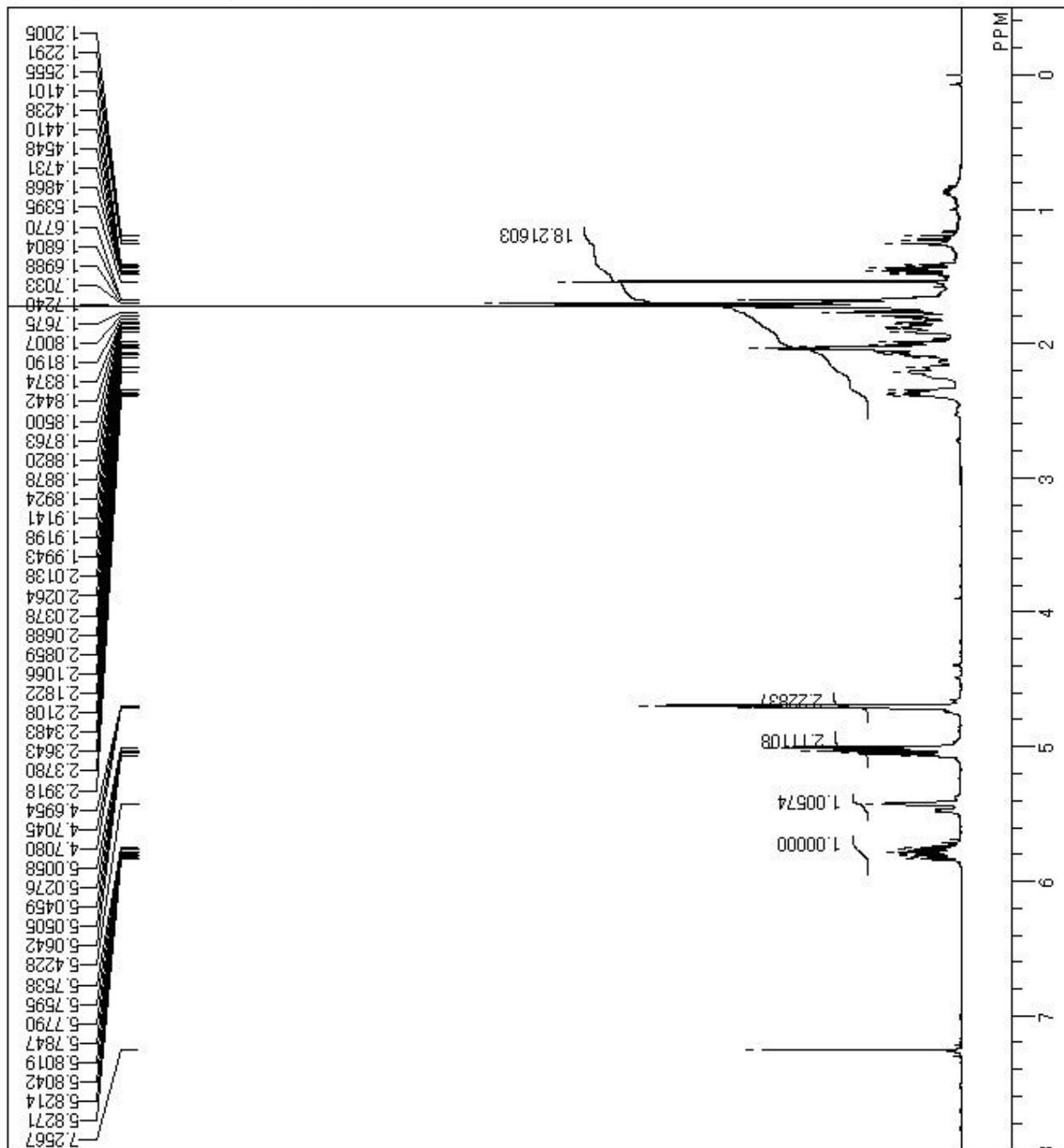
C:\Documents and Settings\delta\My I

27-07-2010 20:31:09

1H
single_pulse.ex2
399.78 MHz
4.19 KHz
7.29 Hz
16384
7503.00 Hz
8
2.1837 sec
2.0000 sec
6.50 usec
1H 407.6 c
CDCL3
0.00 ppm
0.71 Hz
34



DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D.
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



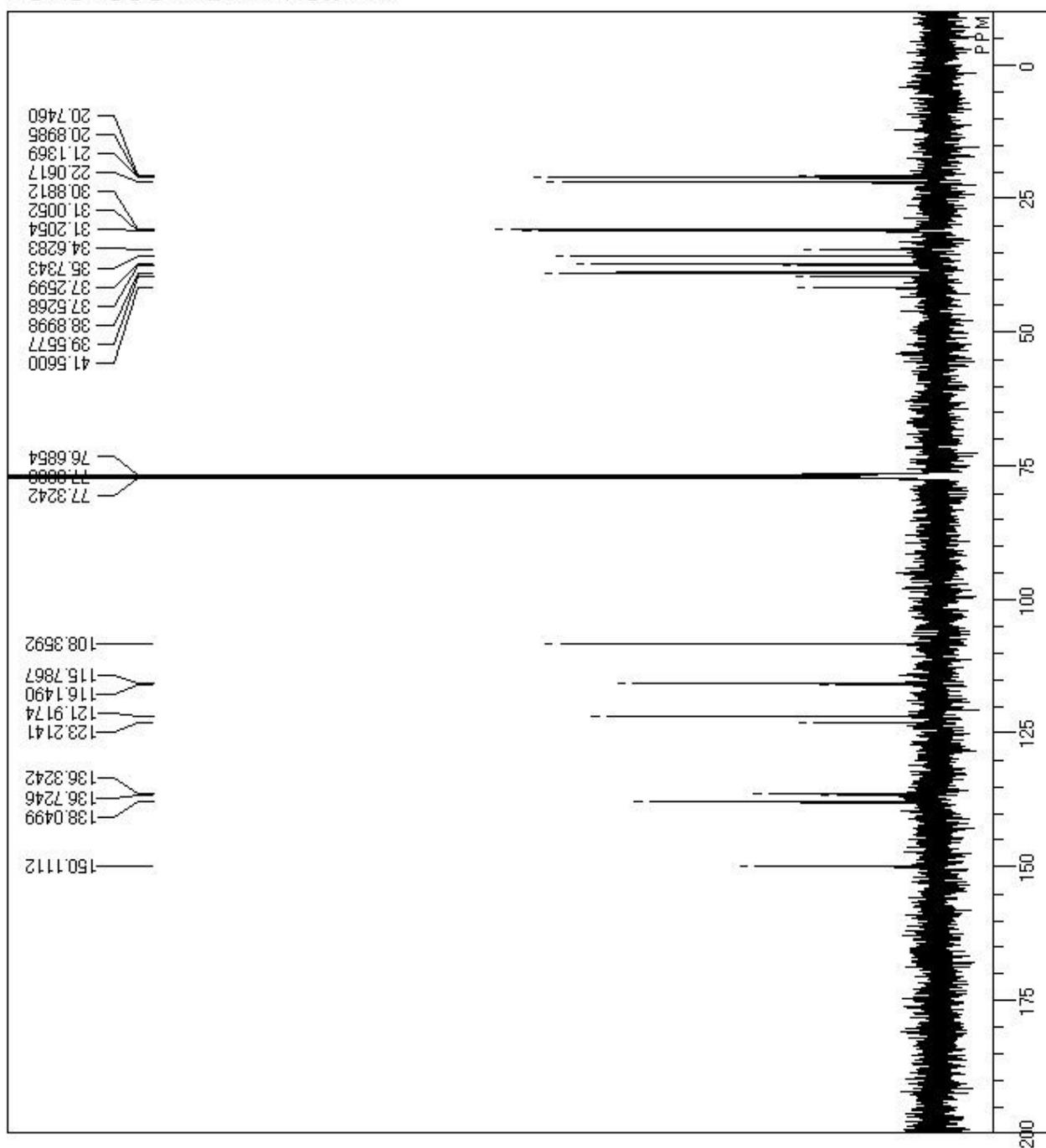
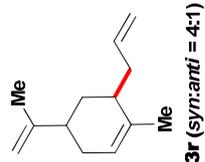
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C:\Documents and Settings\delta\My [

27-07-2010 20:46:16

13C
single_pulse_dec
100.53 MHz
5.35 KHz
5.86 Hz
32768
31407.04 Hz
208
1.0433 sec
2.0000 sec
3.50 usec
1H
407.6 c
CDCl3
77.00 ppm
0.12 Hz
56

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
P.W1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

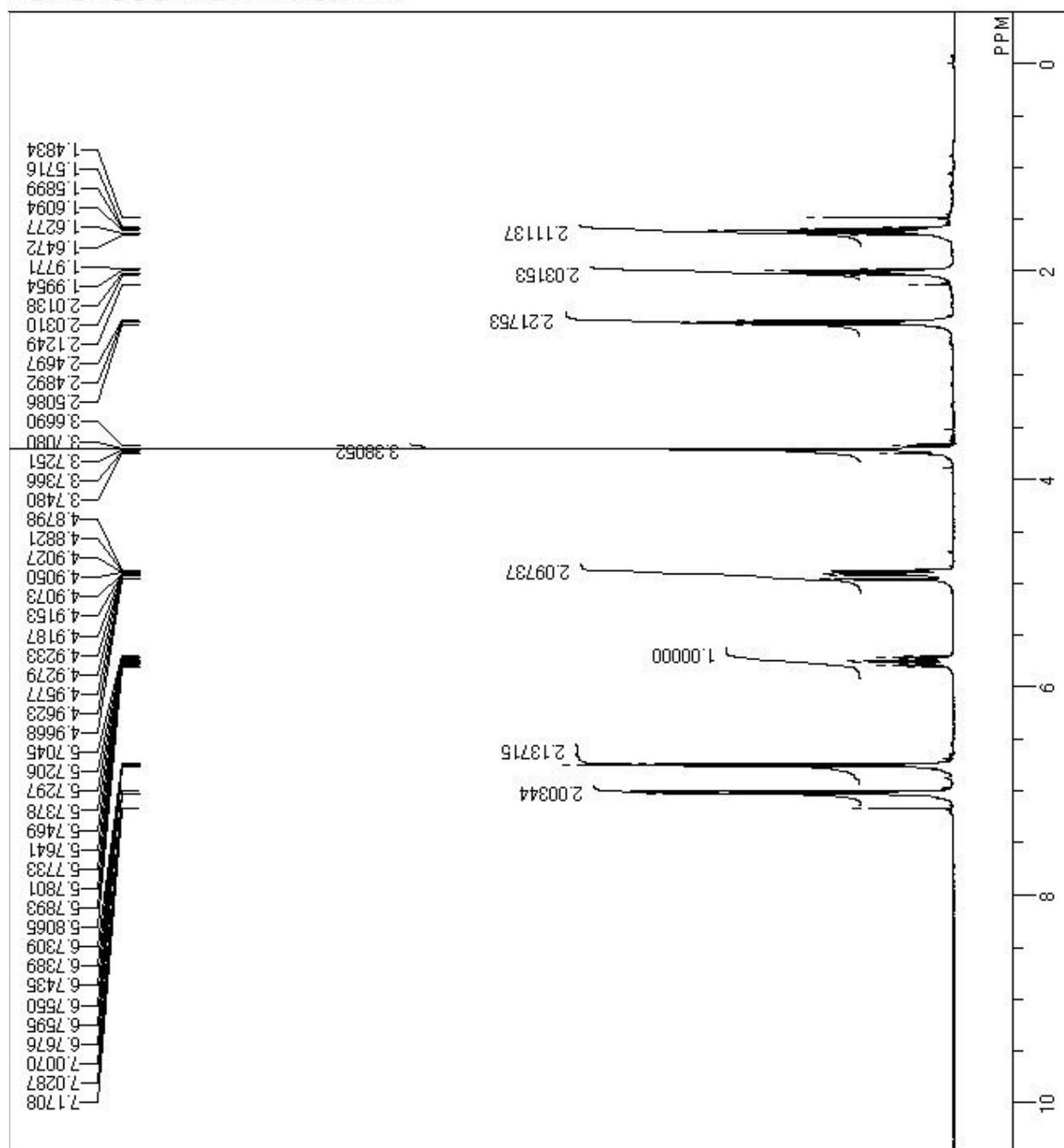
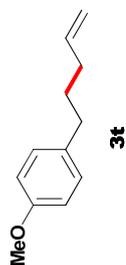


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
P.W1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

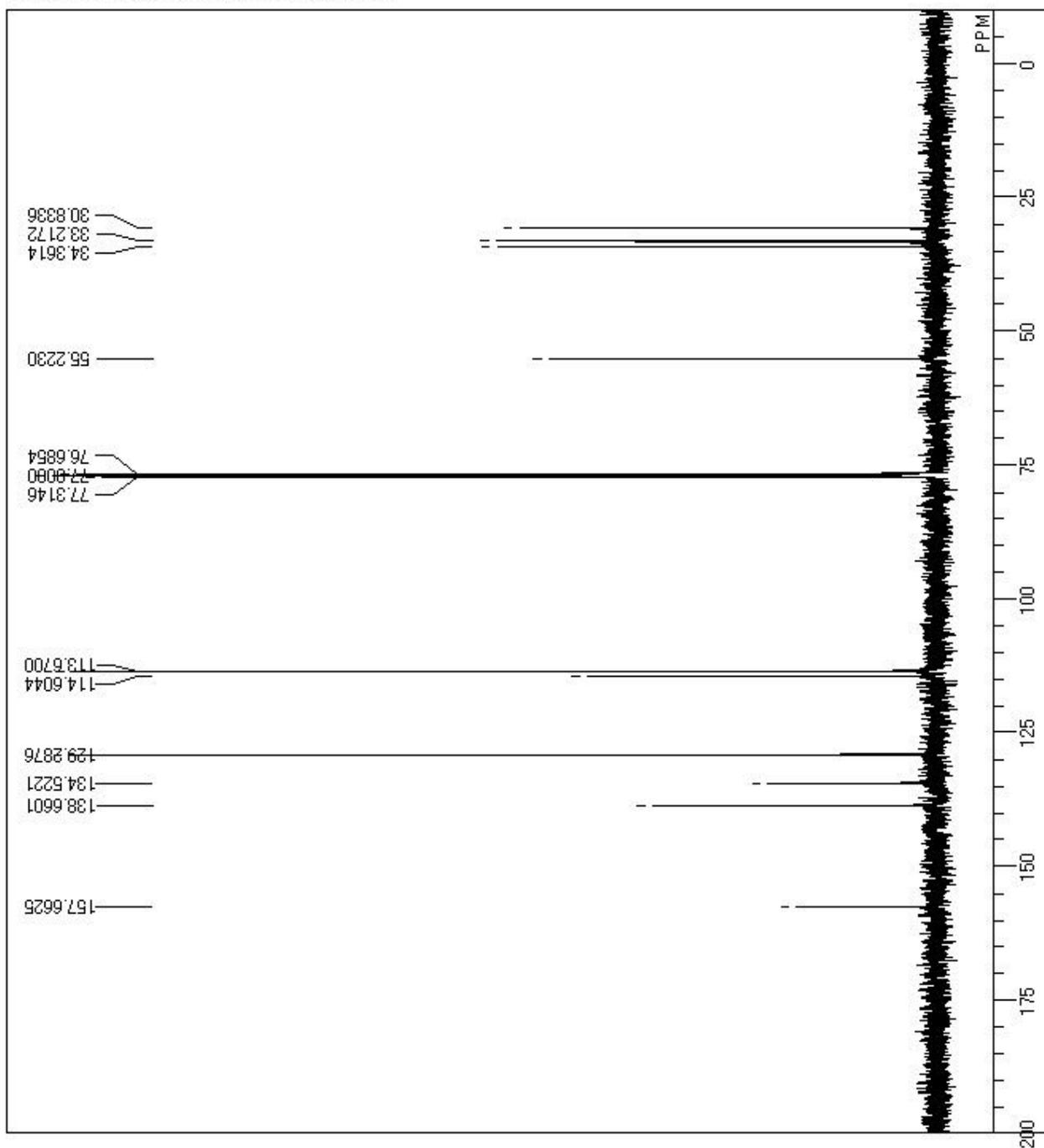
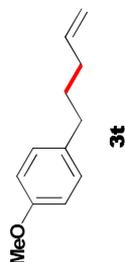
dao 718-1
20-06-2010 18:21:25
1H
single pulse ex2
399.78 MHz
4.19 KHz
7.29 Hz
16384
7503.00 Hz
2.1837 sec
2.0000 sec
6.50 usec
1H
405.1 c
CDCL3
0.00 ppm
0.12 Hz
34



C:\Documents and Settings\delta\My I

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

20-06-2010 18:31:43
13C
single_pulse_dec
100.53 MHz
5.35 KHz
5.86 Hz
32768
31407.04 Hz
134
1.0433 sec
2.0000 sec
3.50 usec
1H
405.1 c
CDCL3
77.00 ppm
0.20 Hz
56

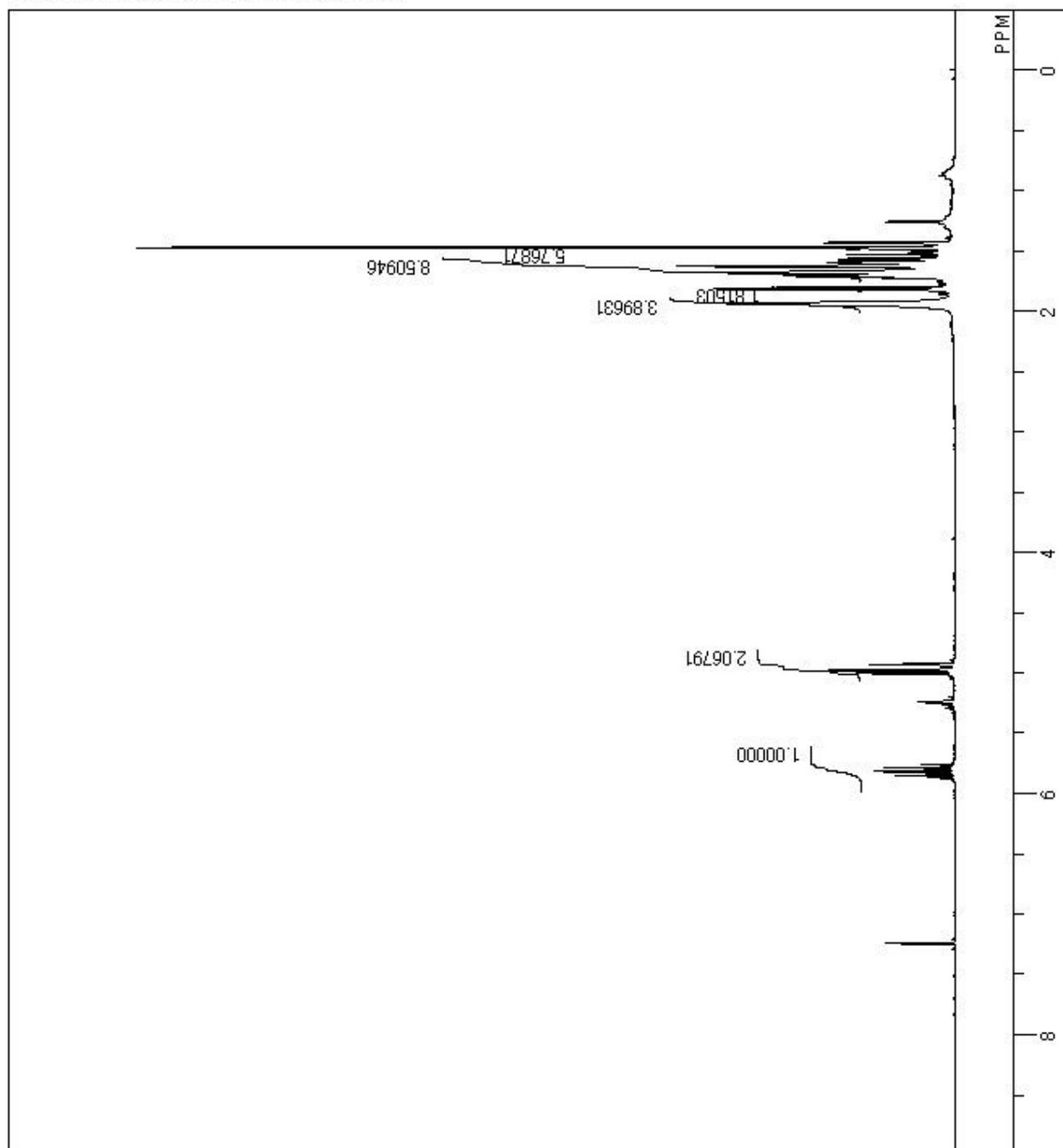


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C:\Documents and Settings\delta\My I

DFILE
COMNT
DATM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

27-07-2010 20:20:11
1H
single_pulse_ex2
399.78 MHz
4.19 KHz
7.29 Hz
13107
6002.31 Hz
8
2.1837 sec
2.0000 sec
6.50 usec
1H
407.6 c
CDCl3
0.00 ppm
0.71 Hz
28



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C:\Documents and Settings\delta\My I

27-07-2010 20:25:04

13C
single_pulse_dec

100.53 MHz

5.35 KHz

5.86 Hz

32768

31407.04 Hz

36

1.0433 sec

2.0000 sec

3.50 usec

1H

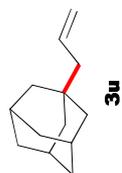
407.6 c

CDCL3

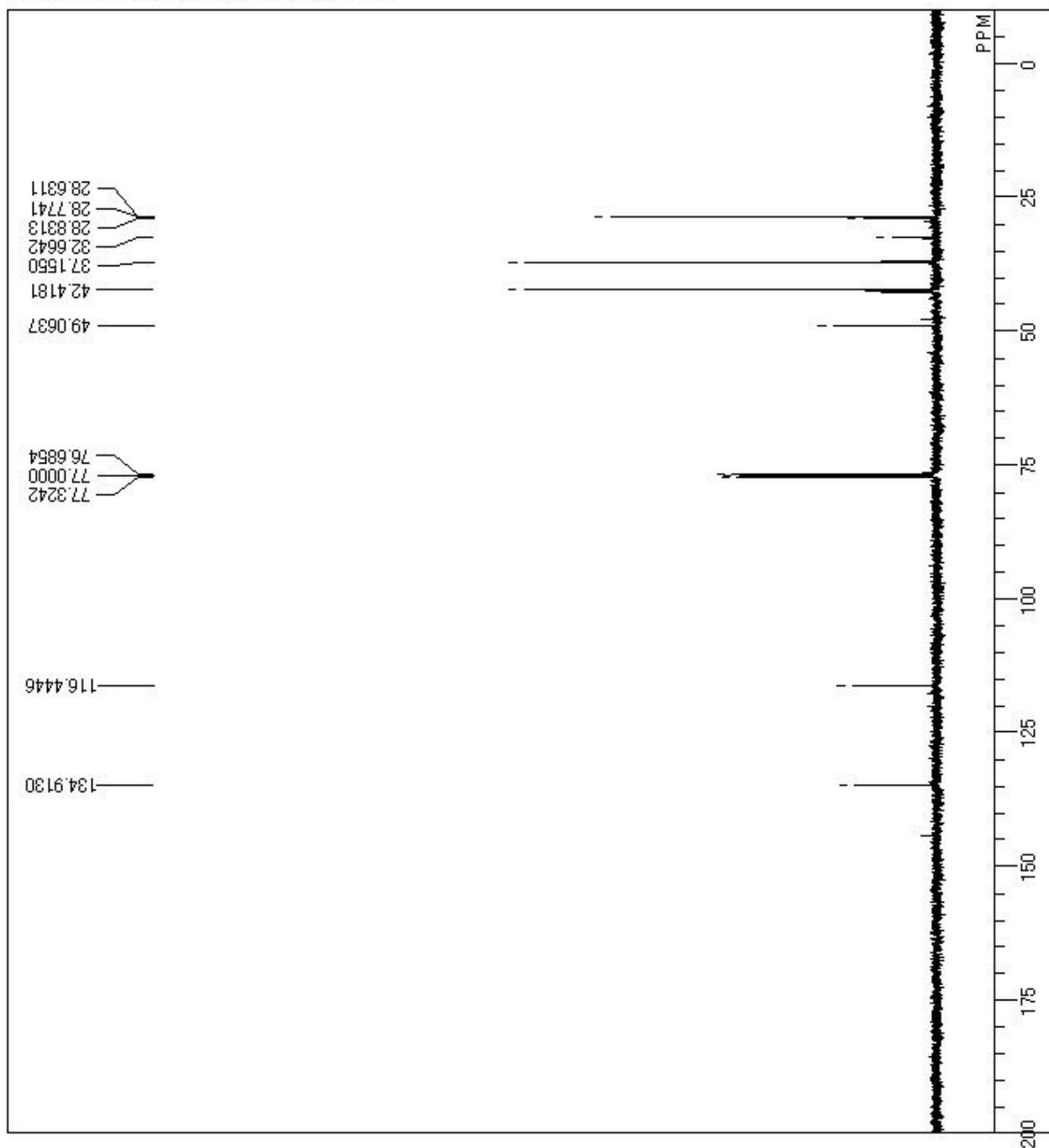
77.00 ppm

0.71 Hz

56



DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
P.D
P.W1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



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