Palladium-Catalyzed Preparation of Vinylallenes from 2-Bromo-1,3,5-trienes via an Alkylidene-π-allylpalladium-Mediated Formal $S_N2"$ Pathway

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Supporting Information

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Experimental Section.

General. All anaerobic and/or moisture sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen or with glovebox techniques under prepurified argon. ^{1}H NMR (at 400 MHz) and ^{13}C NMR (at 100 MHz) chemical shifts are reported in ppm downfield of internal tetramethylsilane. Tetrahydrofuran was distilled from benzophenone-ketyl under nitrogen prior to use. Dichloromethane was distilled from CaH_2 under nitrogen prior to use. $Pd(dba)_2$,[1] $[PdCl(\pi\text{-allyl})]_2$,[2] and dpbp[3] were prepared according to the reported methods. All the other chemicals were obtained from commercial sources.

2-Bromo-1,3,5-trienes (1). A typical procedure is given for the synthesis of **1a**. A solution of ^{t}BuCH=CBr$_2$[4] (16.0 g, 66.5 mmol) and $Pd(PPh_3)_4$ (3.84 g, 3.32 mmol) in THF (50 mL) was added at 0 °C by cannula transfer to a suspension of $(CH_2$=CHCH=CH)ZnCl in THF (150 mL), which was prepared from dry $ZnCl_2$ (8.70 g, 63.8 mmol) and $(CH_2$=CHCH=CH)MgCl[5] (57.8 mmol in THF (100 mL)). After the addition, the reaction mixture was stirred at room temperature for 1 h, then at 50 °C for 2 h. The reaction mixture was cooled to room temperature and filtered through a pad of Cellite. The solvent was removed under reduced pressure, and then the residue was purified by column chromatography on silical gel with hexane as an eluent to afford **1a** (2.3 g, 18%) as a colorless oil. The reaction coditions were not optimized. The characterization data of the products are given below.

5-Bromo-7,7-dimethylocta-1,3,5-triene (1a). ^{1}H NMR ($CDCl_3$): δ 1.25 (s, 9H), 5.16 (d, *J* = 9.9 Hz, 1H), 5.33 (d, *J* = 16.5 Hz, 1H), 6.13 (s, 1H), 6.16 (d, *J* = 14.4 Hz, 1H), 6.41 (ddd, *J* = 16.5, 10.8, and 9.9 Hz, 1H), 6.58 (dd, *J* = 14.4 and 10.8 Hz, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 29.7, 33.8, 118.7, 121.3, 132.4, 133.7, 135.9, 144.2. HRMS Calcd for $C_{10}H_{15}Br$: 214.0356. Found: 214.0353.

2-Bromo-1-cyclohexylhexa-1,3,5-triene (1b). ^{1}H NMR ($CDCl_3$): δ 1.07-1.36 (m, 5H), 1.70-1.77 (m, 5H), 2.53-2.61 (m, 1H), 5.17(d, *J* = 9.8 Hz, 1H), 5.33 (d, *J* = 16.5 Hz, 1H), 5.81 (d, *J* = 8.8 Hz, 1H), 6.18 (d, *J* = 14.4 Hz, 1H), 6.42 (ddd, *J* = 16.5, 10.8, and 9.8 Hz, 1H), 6.57 (dd, *J* = 14.4 and 10.8 Hz, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 26.0, 26.3, 32.2, 41.1, 119.2, 123.0, 132.4, 133.6, 136.3, 140.7. HRMS Calcd for $C_{12}H_{17}Br$: 240.0513. Found: 240.0519.

5-Bromotetradeca-1,3,5-triene (1c). ^{1}H NMR ($CDCl_3$): δ 0.88 (t, *J* = 6.6 Hz, 3H), 1.27-1.46 (m, 12H), 2.28-2.35 (m, 2H), 5.17 (d, *J* = 9.6 Hz 1H), 5.33 (d, *J* = 16.2 Hz, 1H), 5.97 (t, *J* = 7.2 Hz, 1H), 6.21 (d, *J* = 14.4Hz, 1H), 6.41(ddd, *J* = 16.2, 10.8, and 9.6 Hz,1H), 6.56 (dd, *J* = 14.4 and 10.8 Hz, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 14.12, 22.67, 28.44, 29.24, 29.28, 29.41, 31.75, 31.86, 118.84, 124.69, 131.91, 132.98, 135.35, 135.92. HRMS Calcd for $C_{14}H_{23}Br$: 270.0982. Found: 270.0974.

Palladium-Catalyzed Reaction of 1 with 2. A typical procedure is given for the reaction of **1a** with **2m** giving **3am**. A mixture of $[PdCl(\eta^3\text{-}C_3H_5)]_2$ (1.5 mg, 8.2 μmol/Pd), dpbp (4.7 mg, 9.0 μmol), NaH (10.0 mg, 416 μmol), and **2m** (60.9 mg, 416 μmol) was dissolved in THF (5 mL) at room temperature. To this mixture was added **1a** (74.0 mg, 344 μmol) by means of syringe. The reaction mixture was stirred at this temperature for 24 h, then filtered through a short pad of SiO_2 to remove precipitated inorganic salts. The silica gel pad was washed with a small amount of Et_2O three times and the combined solution was evaporated to dryness under reduced pressure. The residue was purified by chromatography on silical gel (hexane/Et_2O = 4/1) to afford the vinylallene **3am** (92.7 mg, 96%) as a colorless oil.

The reaction products from **1b** and **1c** contained allylallenes **4** as minor compornents. Because of overlapping of the ^{1}H NMR signals, the allylallenes (**4bm**, **4bn**, and **4cm**) were characterized by ^{13}C{^{1}H} NMR, ^{13}C{^{1}H} DEPT, and low resolution GC-MS analysis. Although each allylallene consisted of two diastereomers, only the major diastereomer was detected by the ^{13}C NMR measurements and the minor diastereomer was observed by GC-MS analysis. The characterization data of the products are given below.

Dimethyl 2-(7,7-dimethyl-2,4,5-octatrienyl)-2-methylpropane-1,3-dioate (3am). ^{1}H NMR ($CDCl_3$): δ 1.03 (s, 9H), 1.40 (s, 3H), 2.63 (d, *J* = 7.5 Hz, 2H), 3.72 (s, 6H), 5.28-5.29 (m, 1H), 5.41-5.51 (m, 1H), 5.77-5.93 (m, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 19.9, 30.0, 32.1, 39.0, 52.3, 52.4, 53.9, 95.3, 104.1, 124.7, 130.8, 172.2, 172.3, 204.3. HRMS Calcd for $C_{16}H_{24}O_4$: 280.1673. Found: 280.1681.

Diethyl 2-(7,7-dimethyl-2,4,5-octatrienyl)-2-acetylaminopropane-1,3-dioate (3an). ^{1}H NMR ($CDCl_3$): δ 1.02 (s, 9H), 1.20 (d, *J* = 7.2 Hz, 6H), 2.04 (s, 3H), 3.09 (d, *J* = 7.5 Hz, 2H), 4.20-4.30 (m, 4H), 5.27-5.36 (m, 2H), 5.74-5.89 (m, 2H), 6.81 (br s, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 13.85, 13.89, 22.8, 29.9, 32.1, 35.8, 62.4 (2C), 66.3, 95.0, 104.1, 123.1, 131.3, 167.4, 167.5, 168.8, 204.3. HRMS Calcd for $C_{19}H_{29}NO_5$: 351.2044. Found: 351.2043.

Di-*tert*-butyl *N*-(7,7-dimethyl-2,4,5-octatrienyl)iminodicarboxylate (3ao): ^{1}H NMR ($CDCl_3$): δ 0.96 (s, 9H), 1.42 (s, 18H), 4.09-4.11 (m, 2H), 5.22 (d, *J* = 6.0 Hz, 1H), 5.54 (dt, *J* = 15.0 and 6.0 Hz, 1H), 5.74 (dd, *J* = 10.2 and 6.0 Hz, 1H), 5.92 (dd, *J* = 15.0 and 10.2 Hz, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 27.9, 30.0, 32.7, 47.7, 82.1, 94.9, 104.1, 126.6, 129.3, 152.1, 204.7. HRMS Calcd for $C_{20}H_{33}NO_4$: 351.2408. Found: 351.2401.

Dimethyl 2-(6-cyclohexyl-2,4,5-hexatrienyl)-2-methylpropane-1,3-dioate (3bm). ^{1}H NMR ($CDCl_3$): δ 1.18-1.34 (m, 6H), 1.40 (s, 3H), 1.64-1.72 (m, 4H), 1.93-2.02 (m, 1H), 2.62 (d, *J* = 7.5 Hz, 2H), 3.71 (s, 6H), 5.27 (br t, *J* = 6.0 Hz, 1H), 5.44 (dt, *J* = 14.7 and 7.5 Hz, 1H), 5.76 (ddd, *J* = 10.2, 6.0, and 2.7 Hz, 1H), 5.89 (dd, *J* = 14.7 and 10.2 Hz, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 19.9, 25.9, 26.1, 32.9 (2C), 37.2, 39.0, 49.1, 52.38, 52.41, 53.9, 94.4, 98.3, 124.6, 130.7, 172.2, 172.3, 205.9. HRMS Calcd for $C_{18}H_{26}O_4$: 306.1830. Found: 306.1838.

Diethyl 2-(6-cyclohexyl-2,4,5-hexatrienyl)-2-acetylaminopropane-1,3-dioate (3bn). ^{1}H NMR ($CDCl_3$): δ 1.03-1.36 (m, 12H), 1.60-1.73 (m, 4H), 2.04 (s, 3H), 1.94-2.09 (m, 1H), 3.08 (d, *J* = 7.5 Hz, 2H), 4.16-4.34 (m, 4H), 5.25-5.37 (m, 2H), 5.73-5.77(m, 1H), 5.84-5.92 (m, 1H), 6.87(br s, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 13.89, 13.91, 22.8, 25.8, 26.0, 32.77, 32.83, 35.8, 37.1, 49.1, 62.4 (2C), 66.3, 94.3, 98.2, 123.0, 131.3, 167.53, 167.49, 168.9, 206.0. HRMS Calcd for $C_{21}H_{31}NO_5$: 377.2200. Found: 377.2197.

Dimethyl 2-(2,4,5-tetradecatrienyl)-2-methylpropane-1,3-dioate (3cm). ^{1}H NMR ($CDCl_3$): δ 79-0.83 (m, 3H), 1.20-1.39 (m, 15H), 1.89-1.97 (m, 2H), 2.55 (d, *J* = 7.5 Hz, 2H), 3.65 (s, 6H), 5.17-5.23(m, 1H), 5.32-5.42 (m, 1H), 5.61-5.69 (m, 1H), 5.78-5.87 (m, 1H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 14.0, 19.8, 22.6, 28.6, 28.9, 29.0, 29.2, 29.3, 31.8, 39.0, 52.3 (2C), 53.8, 92.3, 93.5, 124.7, 130.6, 172.2(2C), 206.8. HRMS Calcd for $C_{20}H_{32}O_4$: 336.2299. Found: 336.2295.

Dimethyl 2-(6-cyclohexyl-1,4,5-hexatrien-3-yl)-2-methylpropane-1,3-dioate (4bm). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 14.0. 17.4, 22.6, 25.9, 31.5, 33.0, 37.1, 49.1, 52.3, 52.4, 57.8, 90.3, 98.0, 117.5, 136.1, 171.2, 171.3, 203.9.

Diethyl 2-(6-cyclohexyl-1,4,5-hexatrien-3-yl)-2-acetylaminopropane-1,3-dioate (4bn). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 13.83, 13.89, 23.0, 25.8, 25.9, 32.77, 32.83, 35.8, 36.8, 48.2, 62.19, 62.25, 68.3, 89.9, 98.1, 117.5, 135.2, 167.0, 167.5, 168.9, 203.7

Dimethyl 2-(1,4,5-tetradecatrien-3-yl)-2-methylpropane-1,3-dioate (4cm). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 17.3, 17.5, 28.6, 28.8, 29.01, 29.03, 29.3, 31.5, 48.9, 49.0, 52.27, 52.31, 57.8, 89.3, 92.1, 117.4, 136.1, 171.2(2C), 205.0.

Palladium-Catalyzed Asymmetric Synthesis of Vinylallene 3an. A typical procedure is given for the reaction of entry 6 in Table 2. A mixture of $Pd(dba)_2$ (23.0 mg, 40.0 μmol), (*R*)-segphos (27.4 mg, 44.8 μmol), NaO^{t}Bu (46.2 mg, 480 μmol), and **2n** (104 mg, 480 μmol) was dissolved in THF (3 mL). After stirring the solution at 40 °C for 15 min, to this was added **1a** (86.0 mg, 400 μmol) by means of syringe. The mixture was stirred at this temperature for 24 h, then filtered through a short pad of silica gel to remove precipitated inorganic salts. The silica gel pad was washed with small amount of Et_2O three times and the combined solution was evaporated to dryness under reduced pressure. The residue was purified by chromatography on silical gel (hexane/Et_2O = 1/2) to afford the vinylallene **3an** (78.7 mg, 56%) as a white solid.

The enantiopurity of **3an** was determined to be 81% ee by HPLC analysis with a chiral stationary phase column (Daicel Chiralpak AS-H; hexane/^{i}PrOH = 50/1; 0.3 mL/min). The absolute configuration was deduced to be (*R*) by the Lowe-Brewster rule[6] from the sign of optical rotation. $[\alpha]^{27.5}_{D} = -26$ (*c* 0.99, $CHCl_3$).

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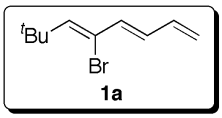

1a

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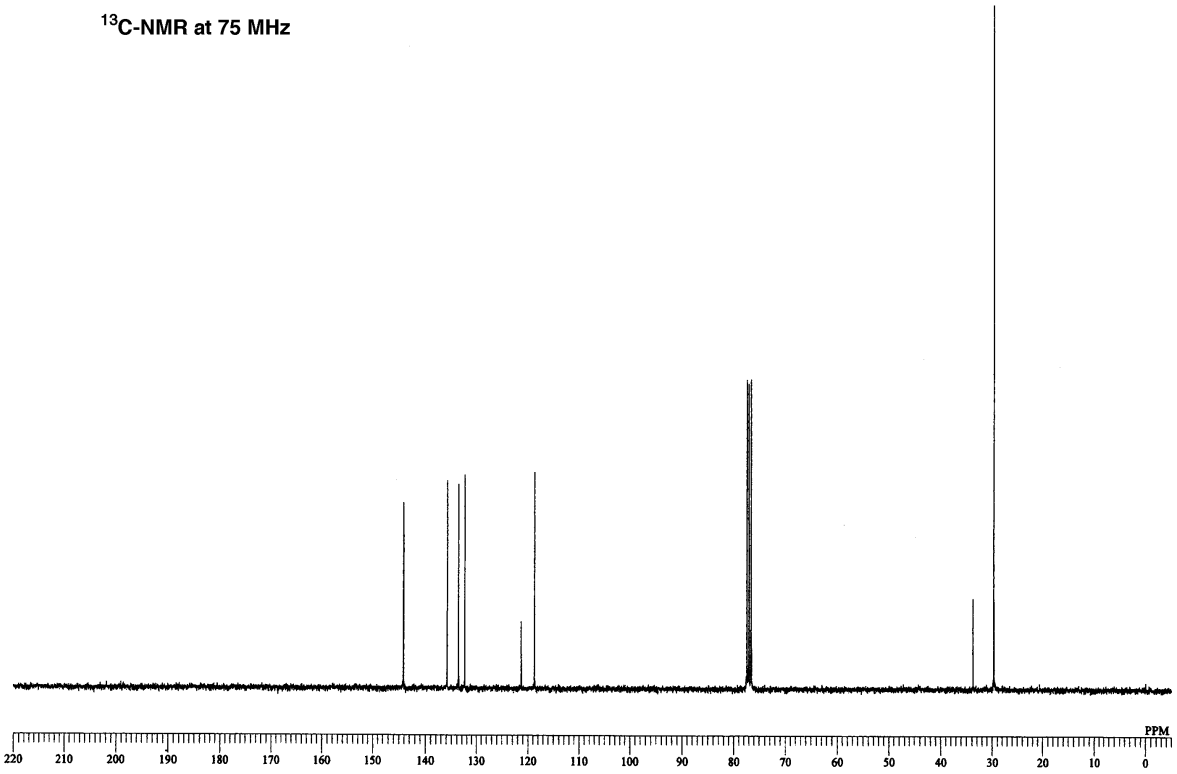
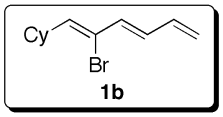
^{13}C-NMR at 75 MHz





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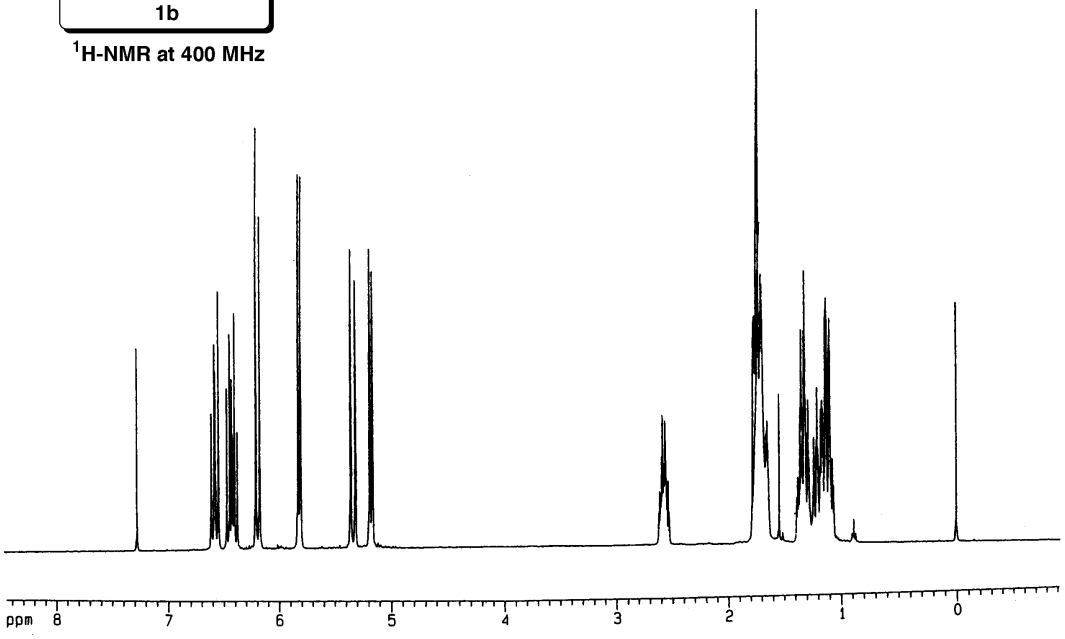

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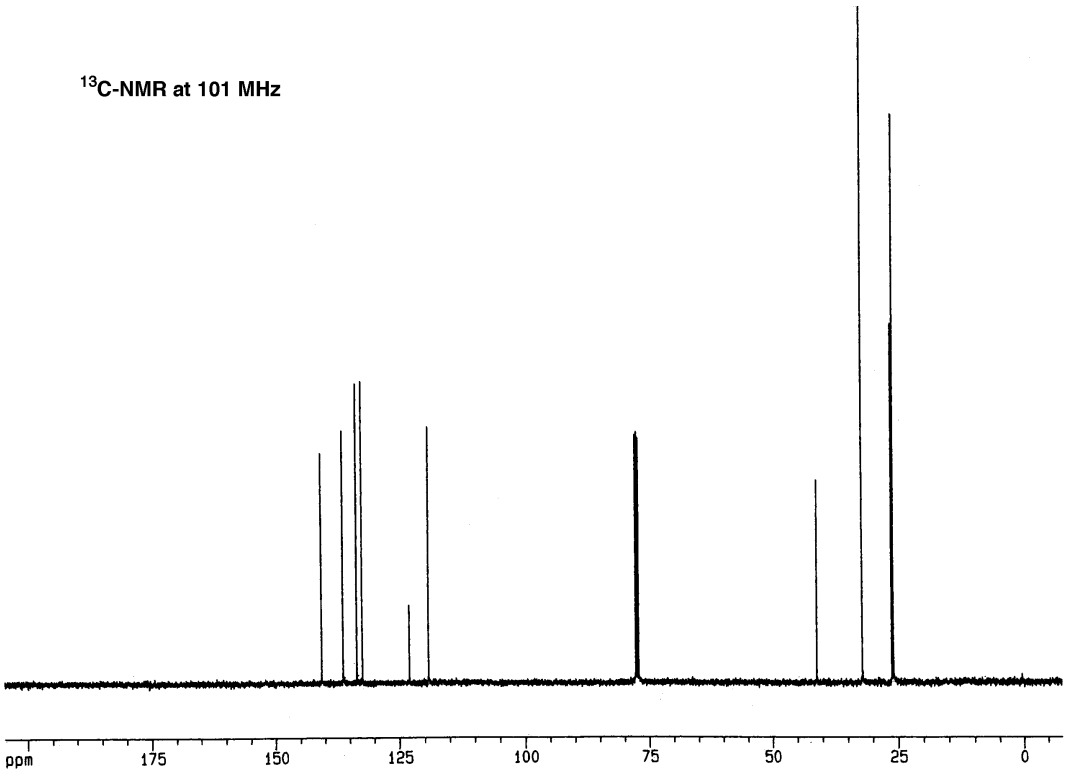

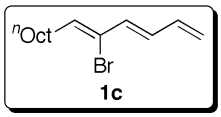

1c

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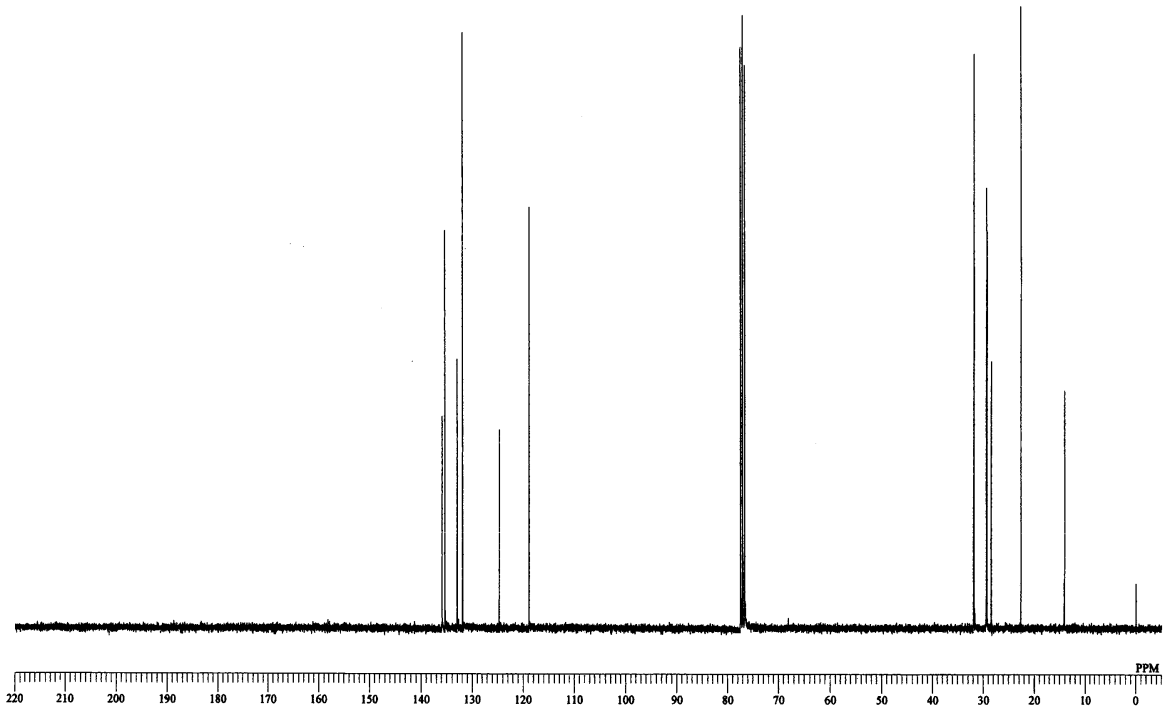
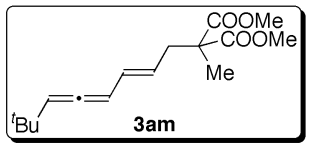
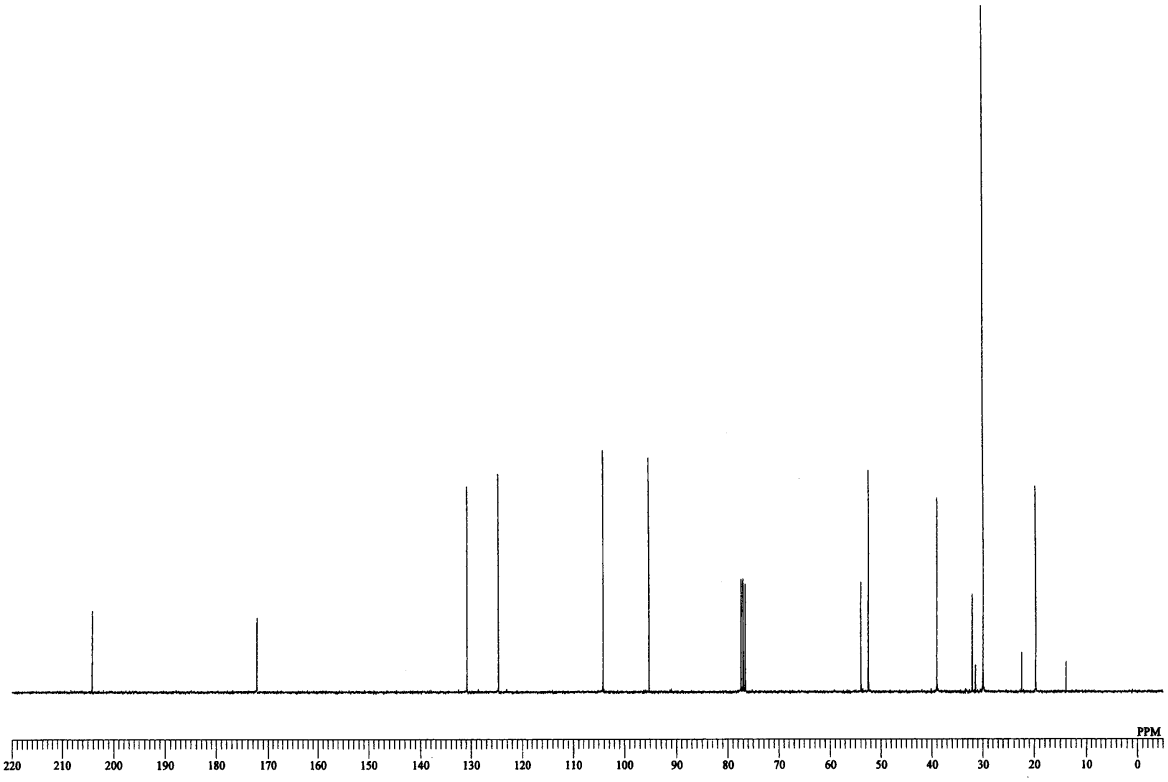


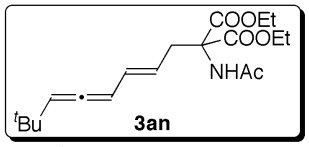


3am

^{1}H-NMR at 300 MHz

^{13}C-NMR at 75 MHz





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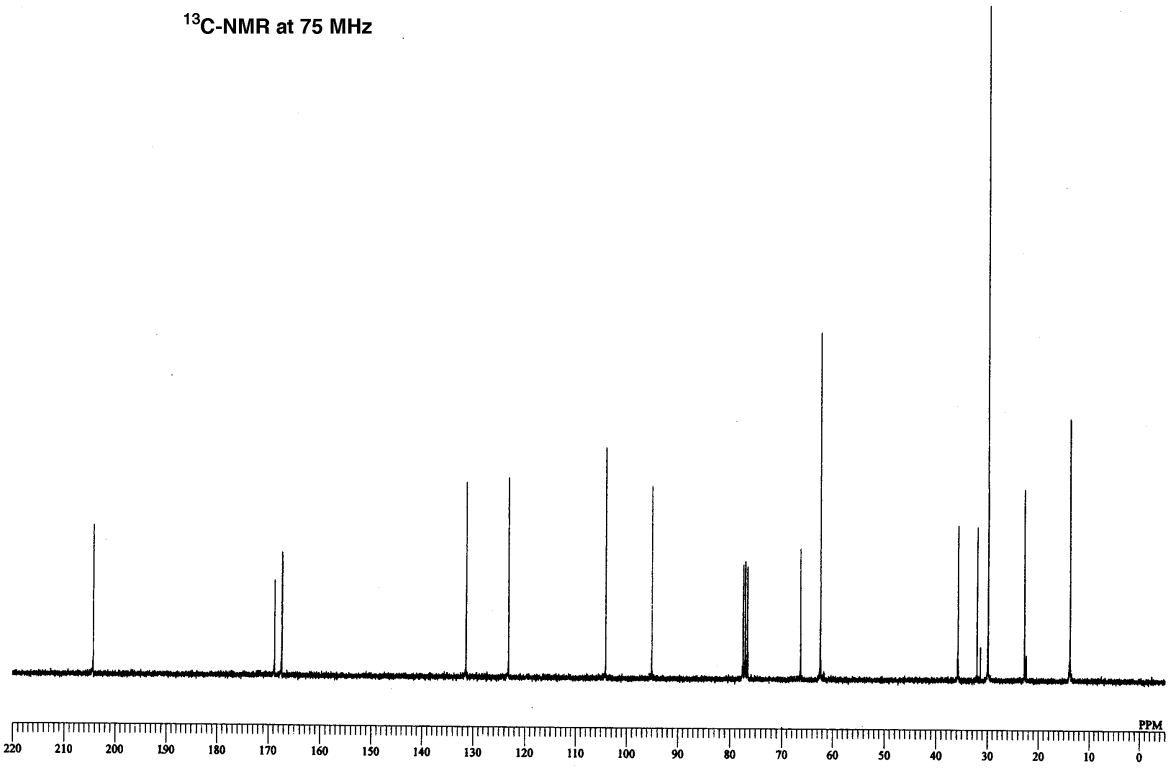
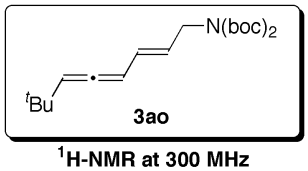
^{13}C-NMR at 75 MHz





3ao

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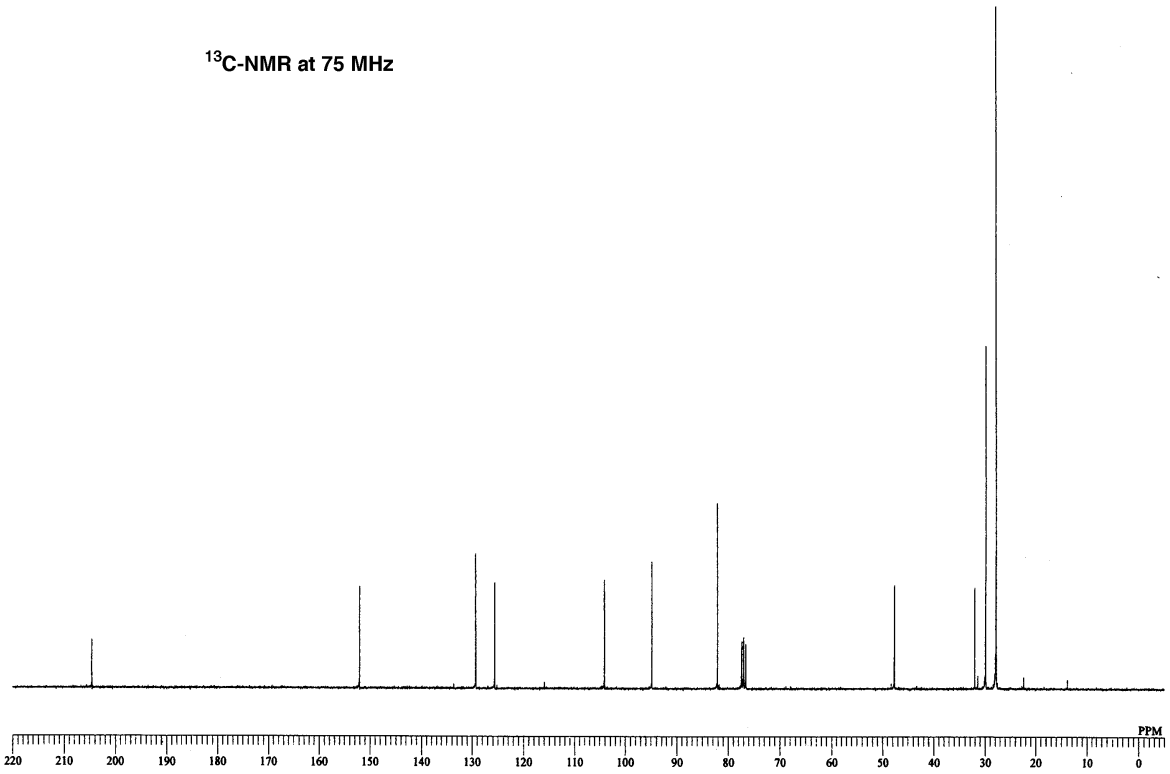
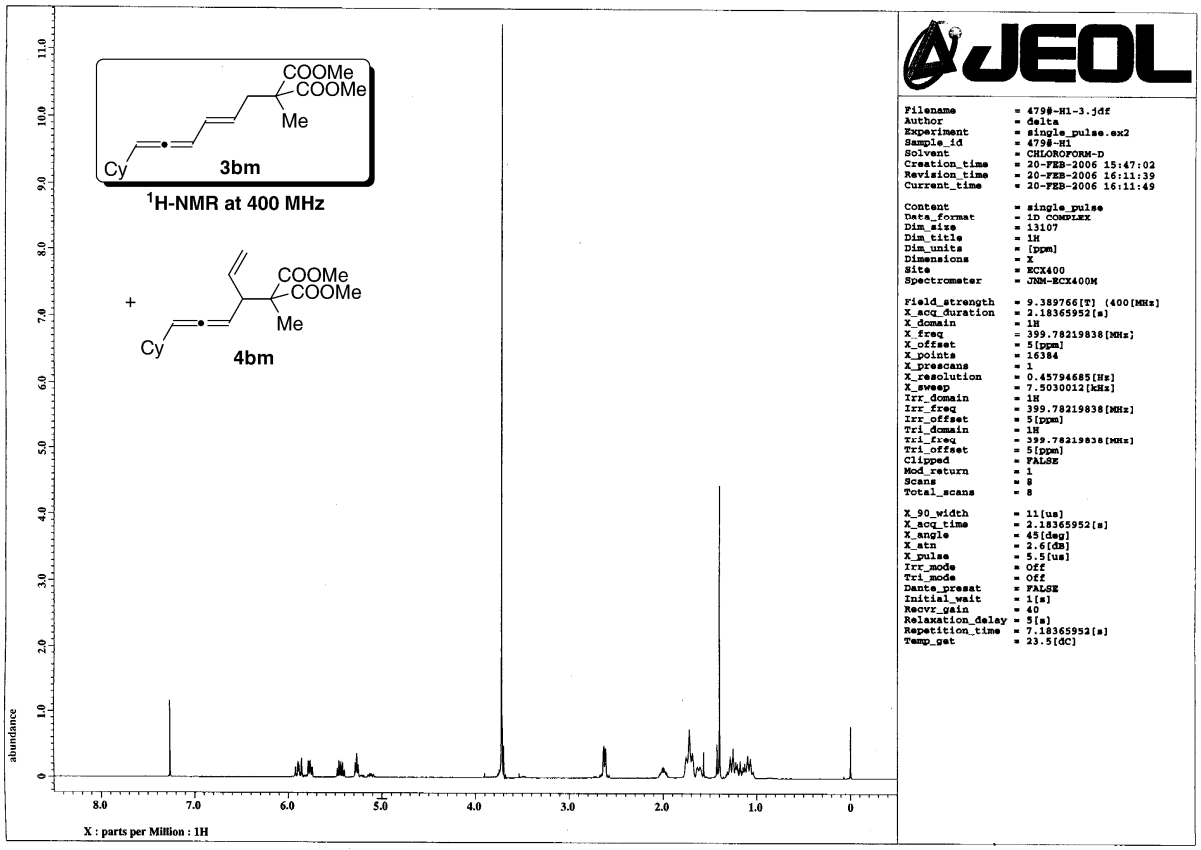
^{13}C-NMR at 75 MHz

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COOMe
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3bm
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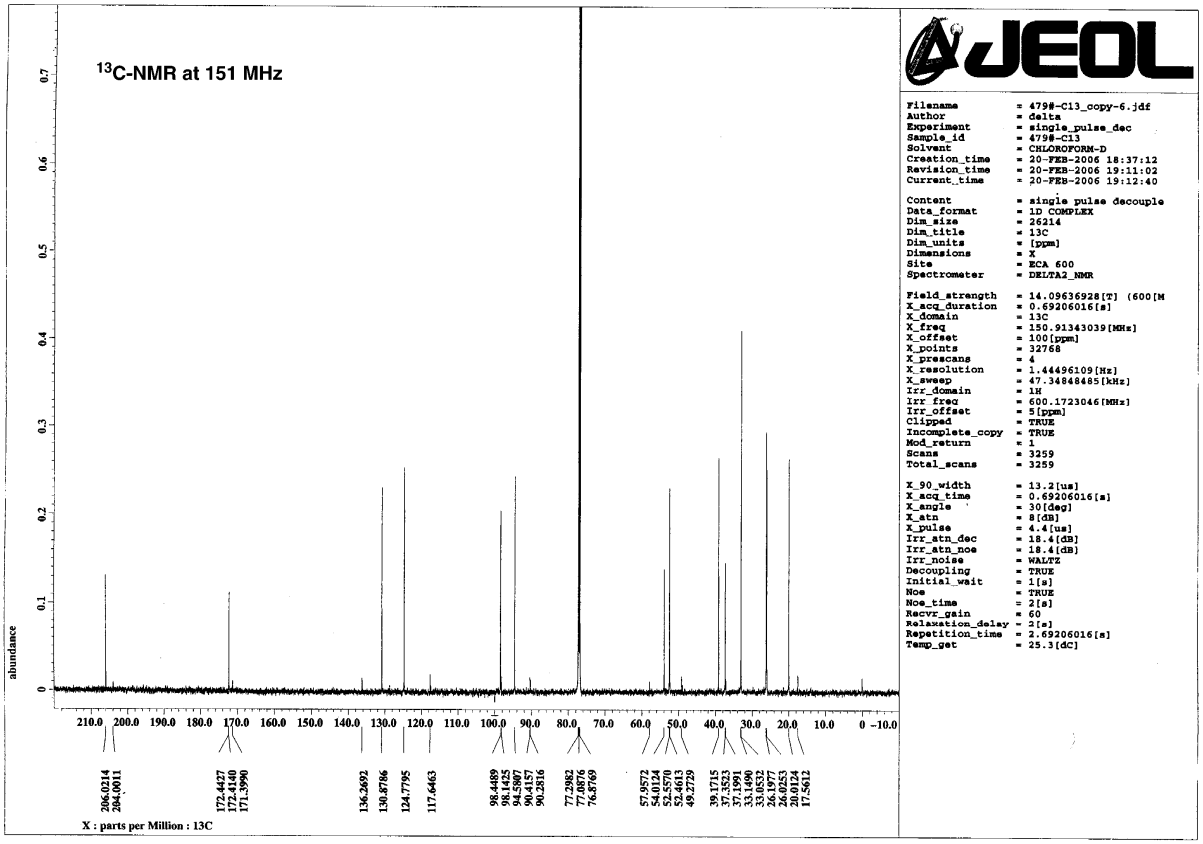
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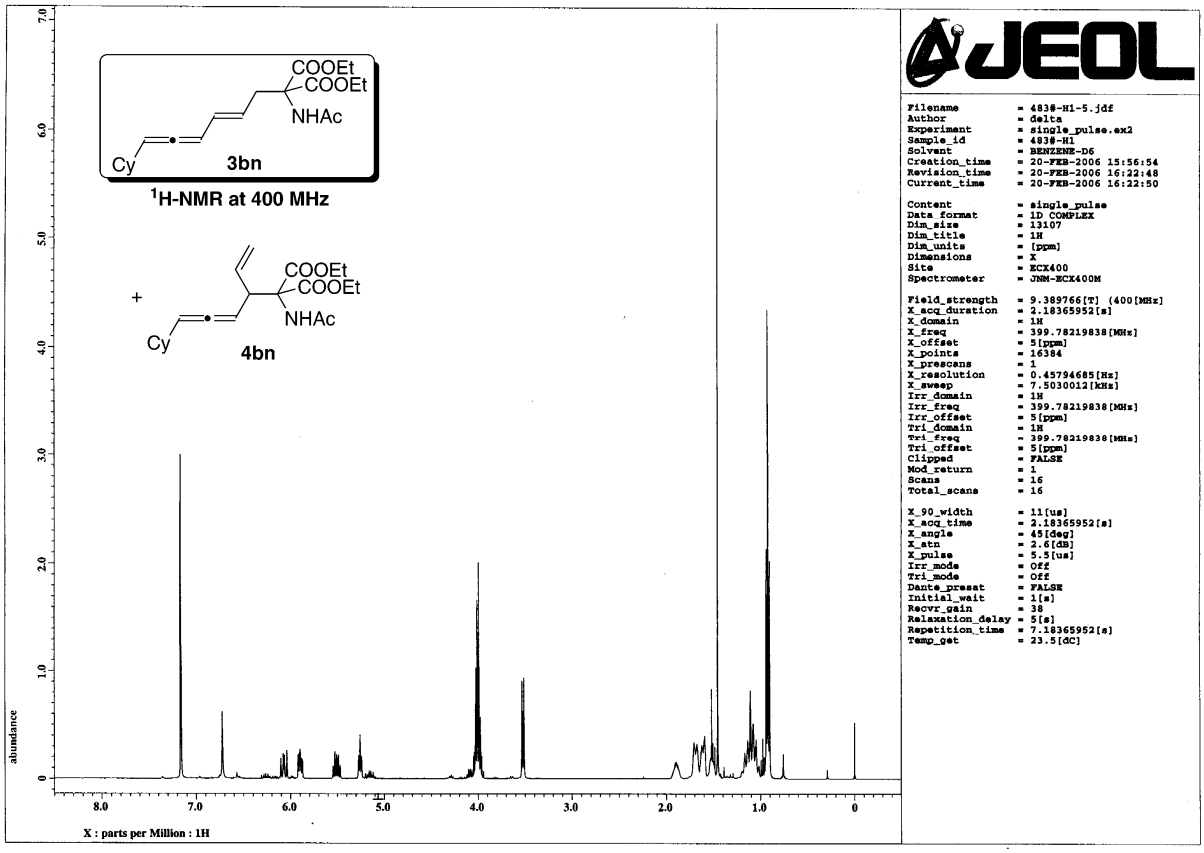
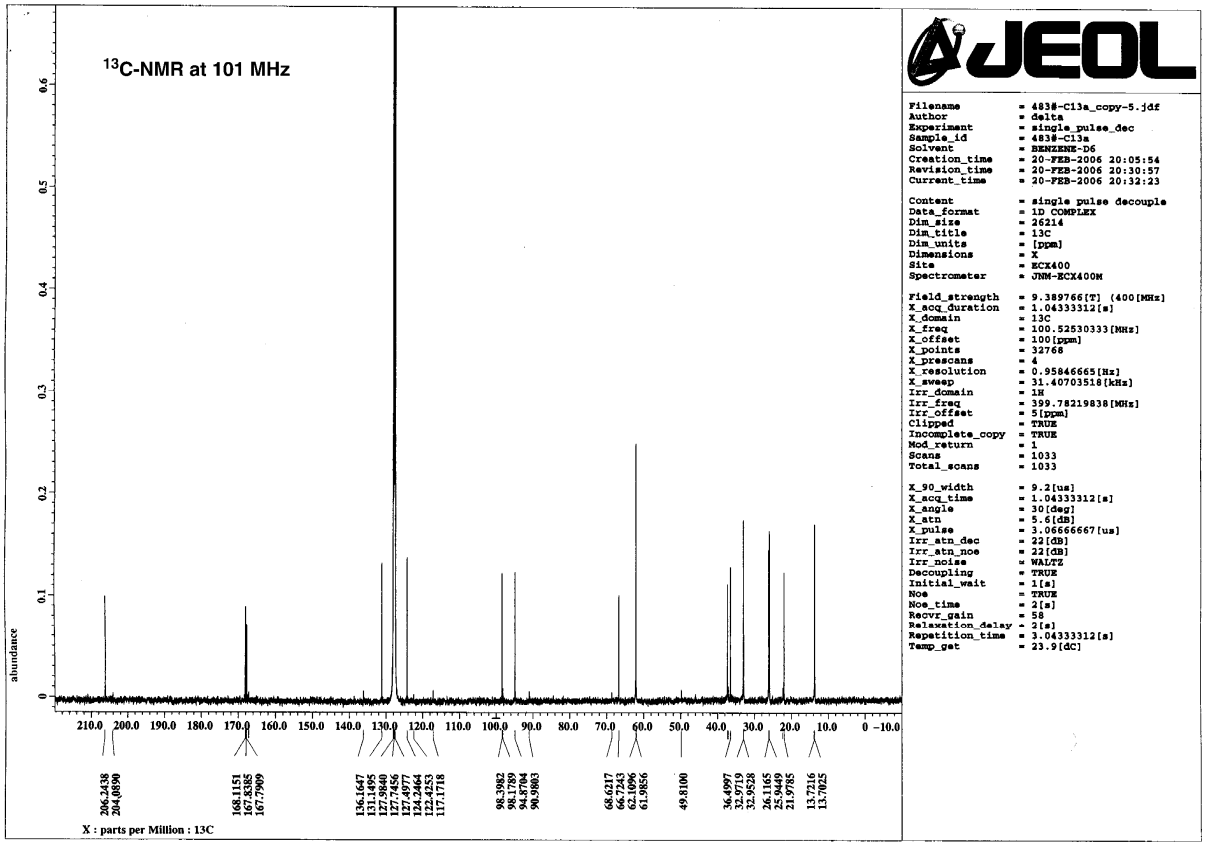
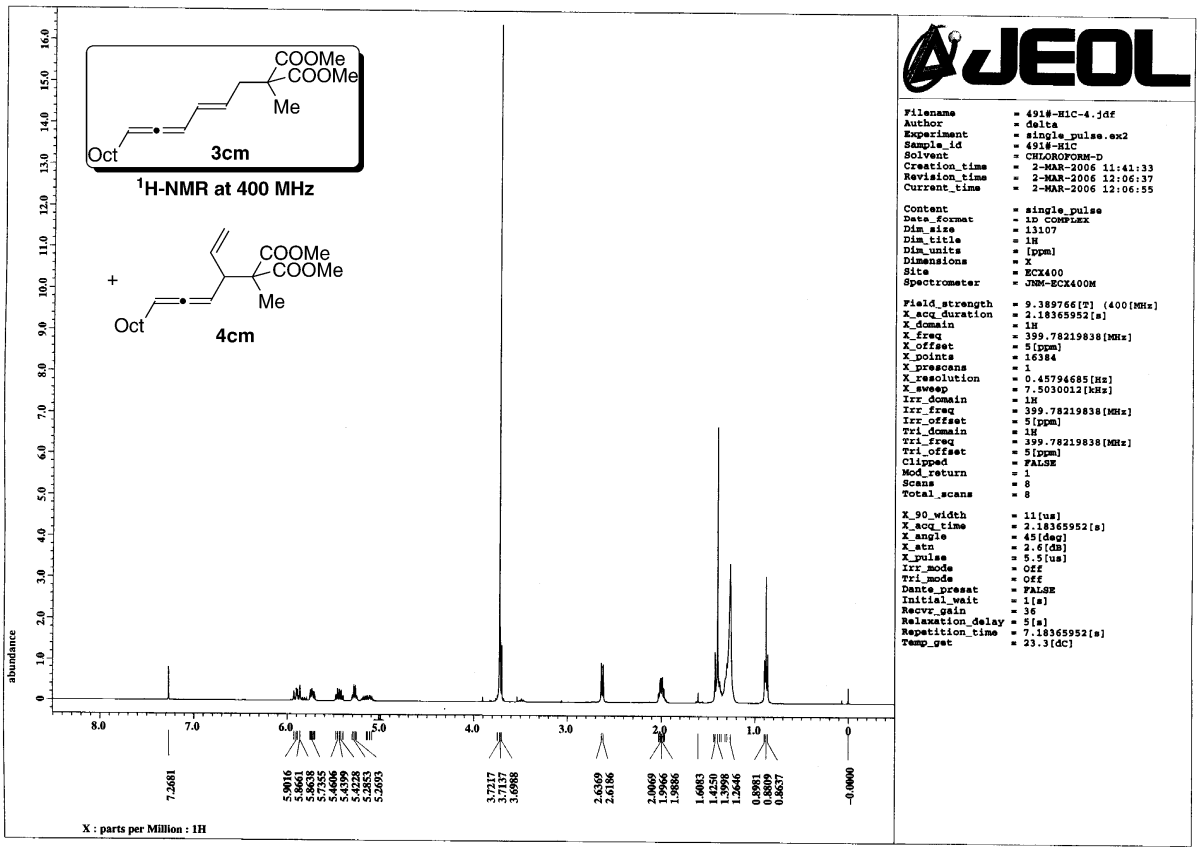
Repetition_time = 2.69206016[s]
Temp_get = 25.3[dC]


COOEt
COOEt
NHAc
Cy
3bn
1H-NMR at 400 MHz
+
COOEt
COOEt
NHAc
Cy
4bn
abundance
X : parts per Million : 1H
Filename = 483#-H1-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 483#-H1
Solvent = BENZENE-D6
Creation_time = 20-FEB-2006 15:56:54
Revision_time = 20-FEB-2006 16:22:48
Current_time = 20-FEB-2006 16:22:50
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX400
Spectrometer = JNM-ECX400M
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 2.18365952[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.45794685[Hz]
X_sweep = 7.5030012[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 11[us]
X_acq_time = 2.18365952[s]
X_angle = 45[deg]
X_atn = 2.6[dB]
X_pulse = 5.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 7.18365952[s]
Temp_get = 23.5[dC]


13C-NMR at 101 MHz
abundance
X : parts per Million : 13C
206.2438
204.0890
168.1151
167.8385
167.7909
136.1647
131.1495
127.9840
127.7456
127.4977
124.3464
122.4253
117.1718
98.3982
98.1789
94.8704
90.9803
68.6217
66.7243
62.1096
61.9856
49.8100
36.4997
33.9719
32.9528
26.1165
25.9449
21.9785
13.7216
13.7025
Filename = 483#-C13a_copy-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 483#-C13a
Solvent = BENZENE-D6
Creation_time = 20-FEB-2006 20:05:54
Revision_time = 20-FEB-2006 20:30:57
Current_time = 20-FEB-2006 20:32:23
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX400
Spectrometer = JNM-ECX400M
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Incomplete_copy = TRUE
Mod_return = 1
Scans = 1033
Total_scans = 1033
X_90_width = 9.2[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 5.6[dB]
X_pulse = 3.06666667[us]
Irr_atn_dec = 22[dB]
Irr_atn_noe = 22[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 58
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 23.9[dC]

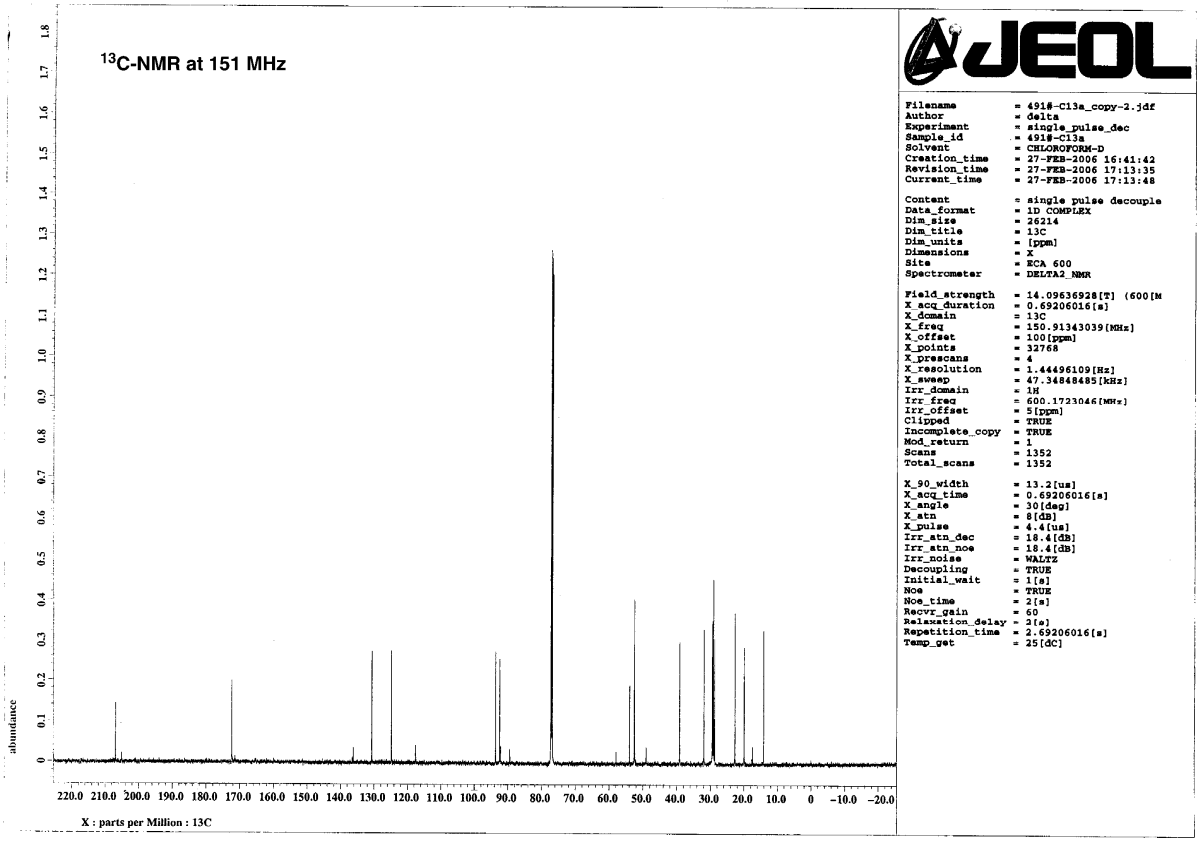


Filename = 491#-H1C-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 491#-H1C
Solvent = CHLOROFORM-D
Creation_time = 2-MAR-2006 11:41:33
Revision_time = 2-MAR-2006 12:06:37
Current_time = 2-MAR-2006 12:06:55

Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX400
Spectrometer = JNM-ECX400M

Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 2.18365952[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.45794685[Hz]
X_sweep = 7.5030012[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11[us]
X_acq_time = 2.18365952[s]
X_angle = 45[deg]
X_atn = 2.6[dB]
X_pulse = 5.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 7.18365952[s]
Temp_get = 23.3[dC]



Filename = 491#-C13a_copy-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 491#-C13a
Solvent = CHLOROFORM-D
Creation_time = 27-FEB-2006 16:41:42
Revision_time = 27-FEB-2006 17:13:35
Current_time = 27-FEB-2006 17:13:48

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCA 600
Spectrometer = DELTA2_NMR

Field_strength = 14.09636928[T] (600[M
X_acq_duration = 0.69206016[s]
X_domain = 13C
X_freq = 150.91343039[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.44496109[Hz]
X_sweep = 47.34848485[kHz]
Irr_domain = 1H
Irr_freq = 600.1723046[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Incomplete_copy = TRUE
Mod_return = 1
Scans = 1352
Total_scans = 1352

X_90_width = 13.2[us]
X_acq_time = 0.69206016[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 4.4[us]
Irr_atn_dec = 18.4[dB]
Irr_atn_noe = 18.4[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.69206016[s]
Temp_get = 25[dC]