Diastereoselective Three-component Reactions of Aryldiazoacetates with Alcohols/Water and Alkynals: Application to Substituted Enelactones

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Supporting Information

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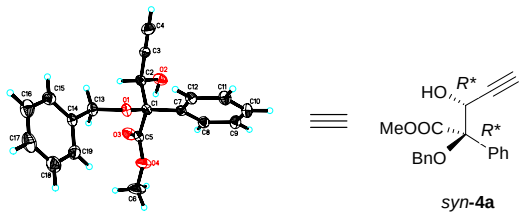
1. Crystal data

Crystal data of *syn*-4a

Single-crystal X-ray structure determinations were performed on a Bruker SMART APEX diffractometer

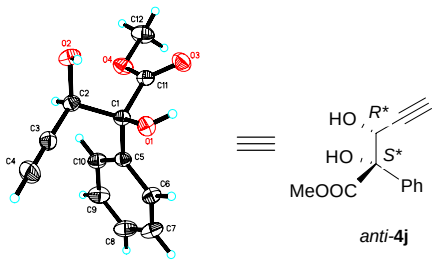


Empirical formula	C19 H18 O4
Formula weight	310.33
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 12.9167(17) A alpha = 90 deg.
	b = 9.8437(13) A beta = 107.208(2) deg.
	c = 13.3798(17) A gamma = 90 deg.
Volume	1625.1(4) A^3
Z, Calculated density	4, 1.268 Mg/m^3
Absorption coefficient	0.089 mm^-1
F(000)	656
Crystal size	0.390 x 0.301 x 0.127 mm
Theta range for data collection	1.93 to 25.99 deg.
Limiting indices	-15<=h<=15, -12<=k<=12, -16<=l<=6
Reflections collected / unique	8645 / 3180 [R(int) = 0.0348]
Completeness to theta = 25.99	99.7 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.7405
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3180 / 1 / 213
Goodness-of-fit on F^2	0.930
Final R indices [I>2sigma(I)]	R1 = 0.0443, wR2 = 0.0955
R indices (all data)	R1 = 0.0668, wR2 = 0.1038
Largest diff. peak and hole	0.167 and -0.150 e.A^-3

CCDC-806582 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Crystal data of *anti*-4j

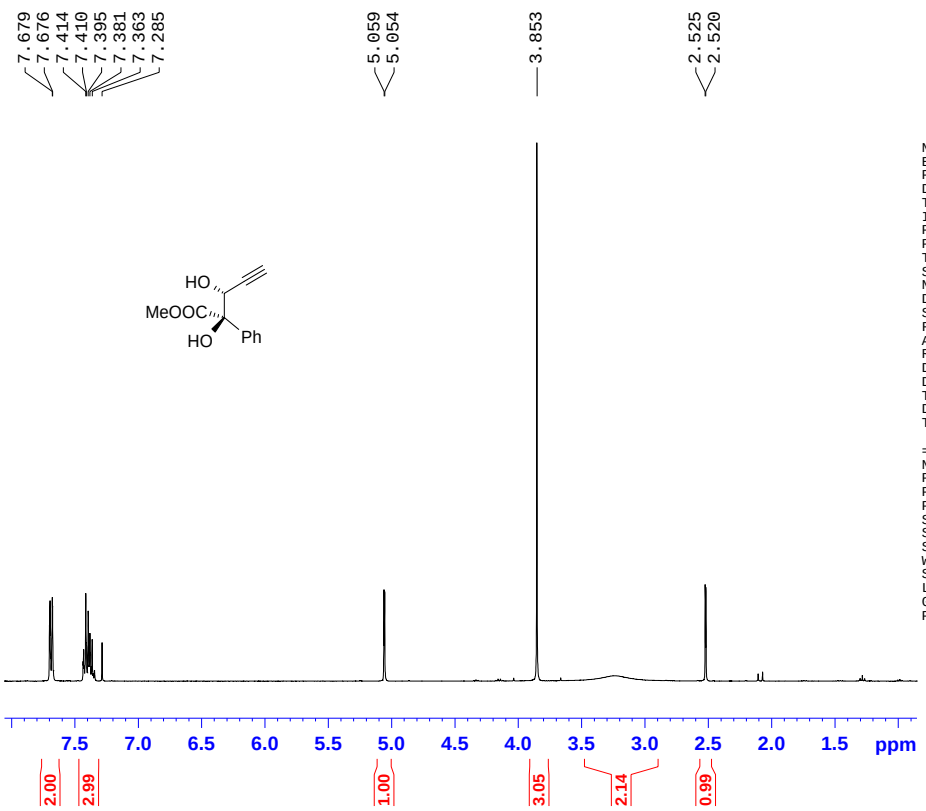
Single-crystal X-ray structure determinations were performed on a Bruker SMART APEX diffractometer



Empirical formula	C12 H12 O4
Formula weight	220.22
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 10.4347(8) A alpha = 90 deg.
	b = 15.9131(12) A beta = 109.4870(10) deg.
	c = 14.1048(10) A gamma = 90 deg.
Volume	2207.9(3) A^3
Z, Calculated density	8, 1.325 Mg/m^3
Absorption coefficient	0.100 mm^-1
F(000)	928
Crystal size	0.412 x 0.369 x 0.315 mm
Theta range for data collection	2.43 to 25.98 deg.
Limiting indices	-10<=h<=12, -18<=k<=19, -17<=l<=14
Reflections collected / unique	5925 / 2171 [R(int) = 0.0385]
Completeness to theta = 25.98	99.8 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.5979
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2171 / 2 / 155
Goodness-of-fit on F^2	1.057
Final R indices [I>2sigma(I)]	R1 = 0.0448, wR2 = 0.1267
R indices (all data)	R1 = 0.0483, wR2 = 0.1299
Extinction coefficient	0.0084(11)
Largest diff. peak and hole	0.227 and -0.224 e.A^-3

CCDC-806589 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

2. NMR analysis spectra for 4a-l, 5-8 and UPLC spectra for 4a

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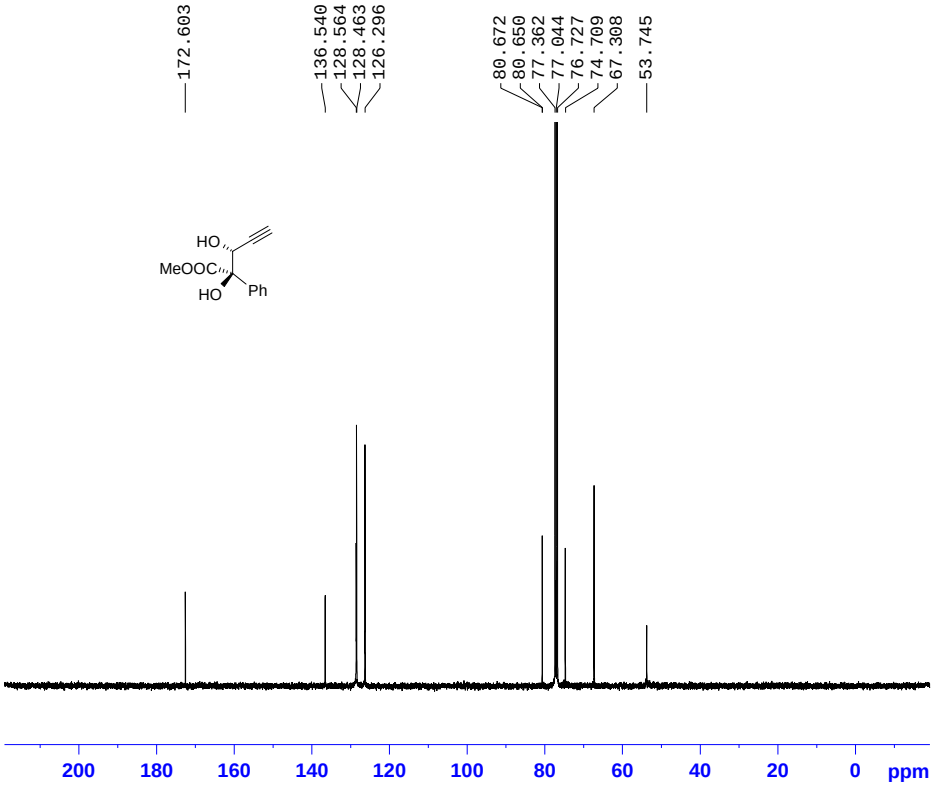
EXPNO                  1
PROCNO                 1
Date_           20101105
Time               19.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 228.1
DW                60.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

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EXPNO                  2
PROCNO                 1
Date_           20101105
Time               20.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 9195.2
DW                20.850 usec
DE                 6.50 usec
TE                298.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.20 usec
PL1                -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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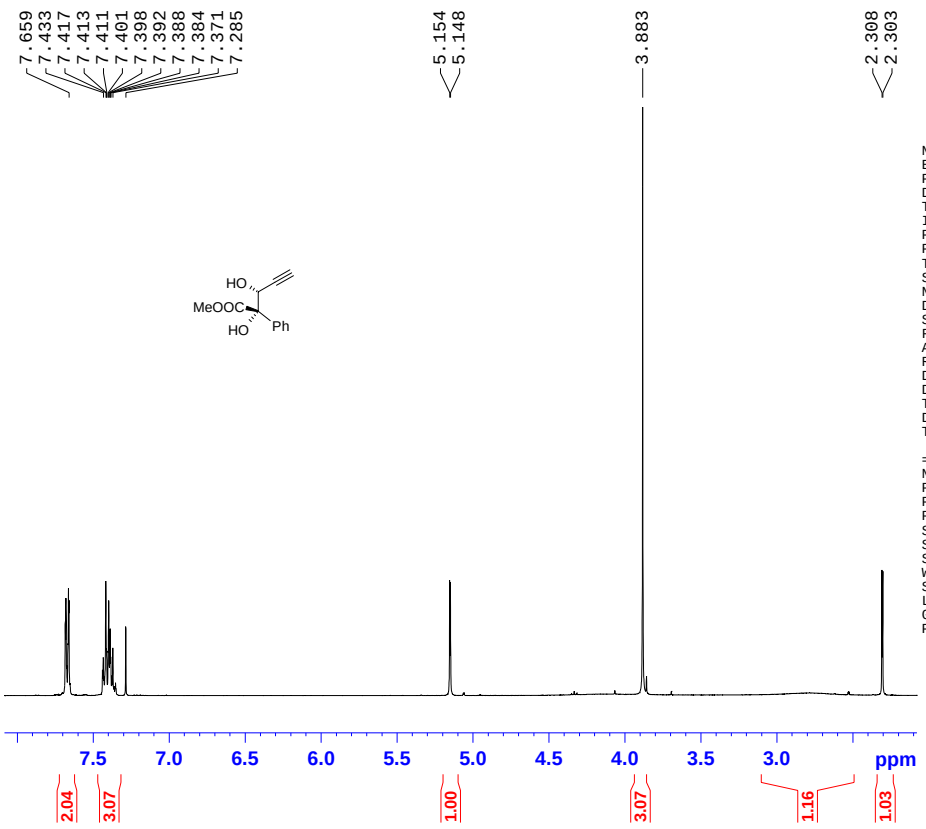


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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   362
DW                60.400 usec
DE                 6.50 usec
TE                297.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
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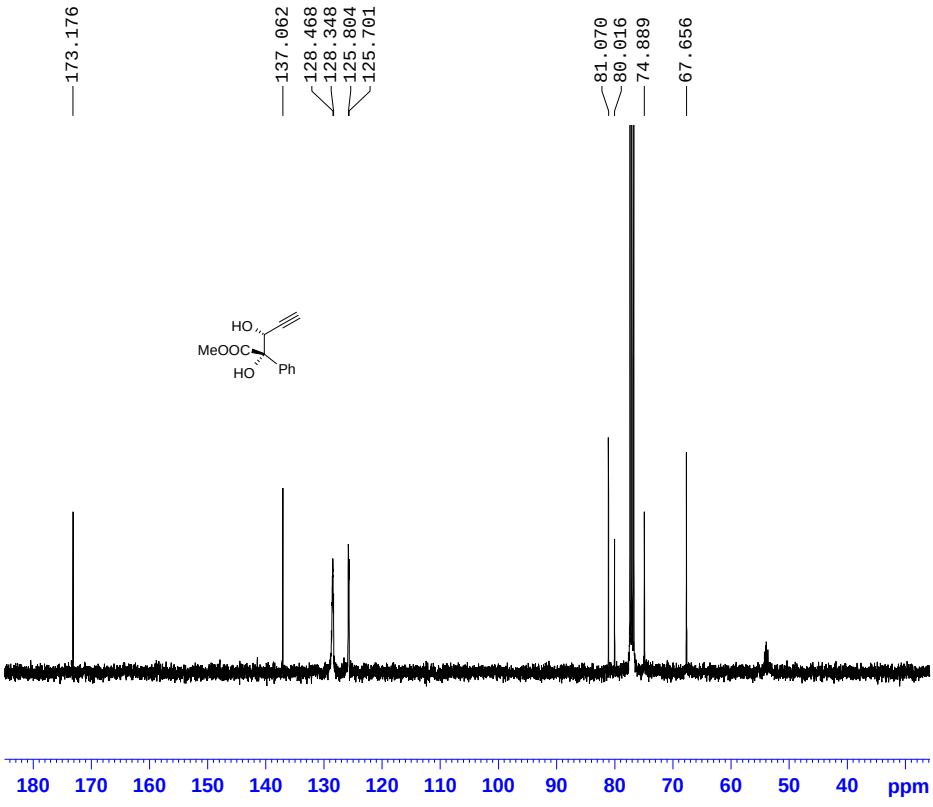


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PROCNO                 1
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Time                21.43
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 32768
DW                20.850 usec
DE                 6.50 usec
TE                297.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.20 usec
PL1               -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              10.83 dB
PL13              10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
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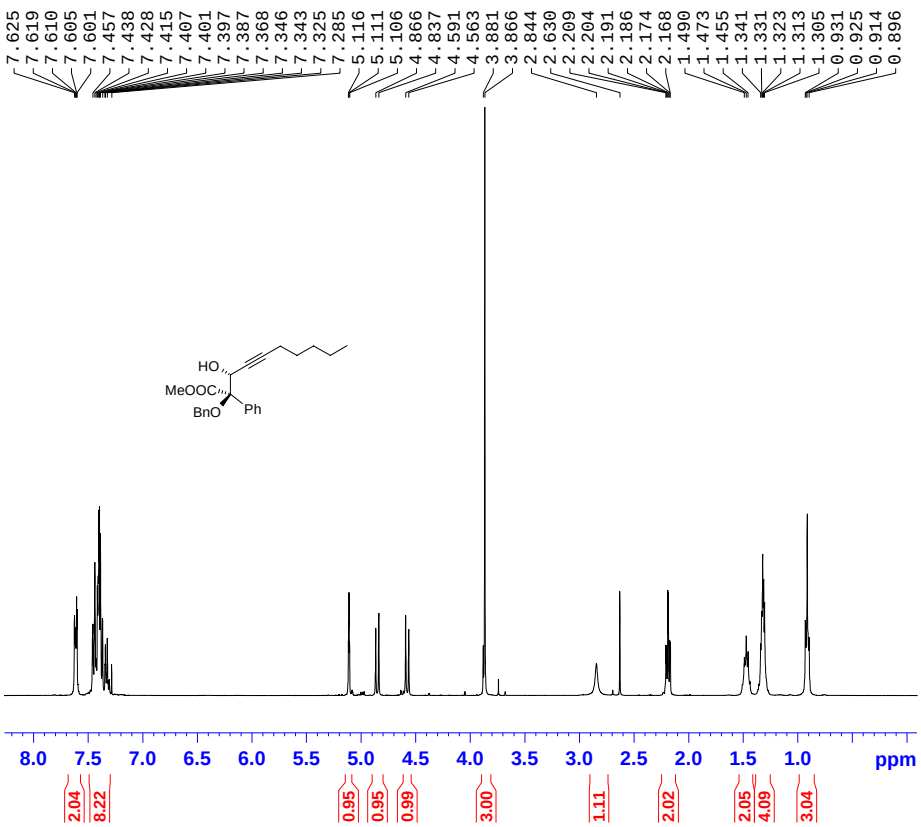


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PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    35.9
DW                  60.400 usec
DE                   6.50 usec
TE                  298.1 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -1.00 dB
PL1W          10.40389347 W
SFO1          400.1124708 MHz
SI                   32768
SF            400.1100000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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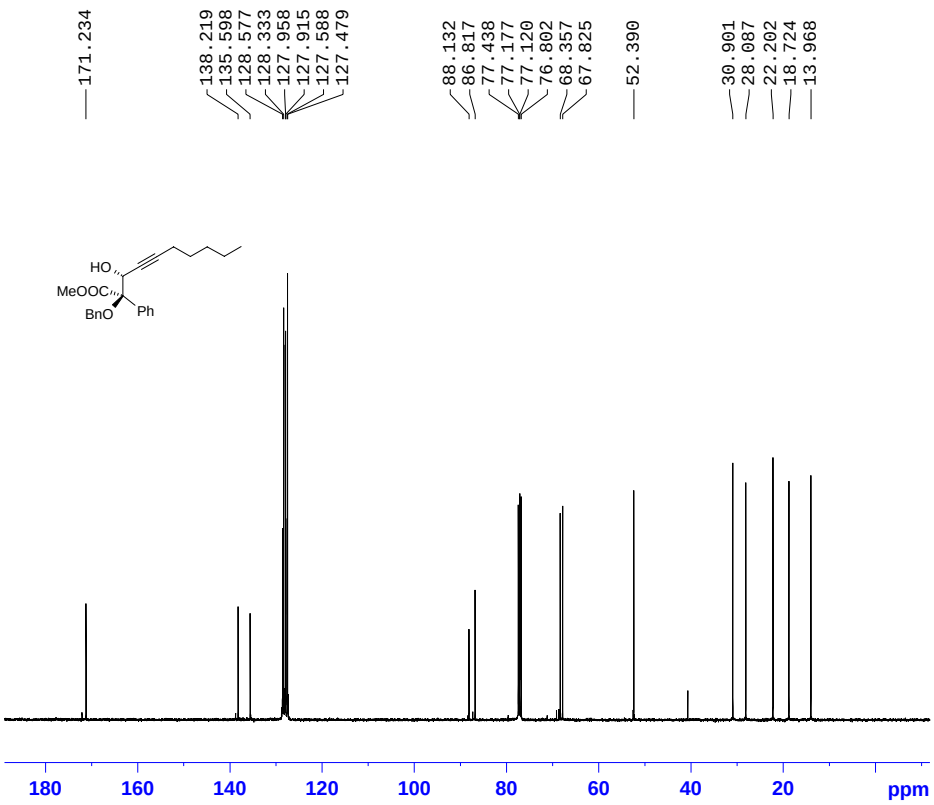


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NAME      NHB1-2005005-110-01
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PROCNO                  1
Date_           20100419
Time                17.36
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                   32768
DW                  20.850 usec
DE                   6.50 usec
TE                  298.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.20 usec
PL1                 -2.00 dB
PL1W          61.30850601 W
SFO1          100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                14.46 dB
PL13                14.00 dB
PL2W          10.40389347 W
PL12W          0.29593471 W
PL13W          0.32900000 W
SFO2          400.1116004 MHz
SI                   32768
SF            100.6077400 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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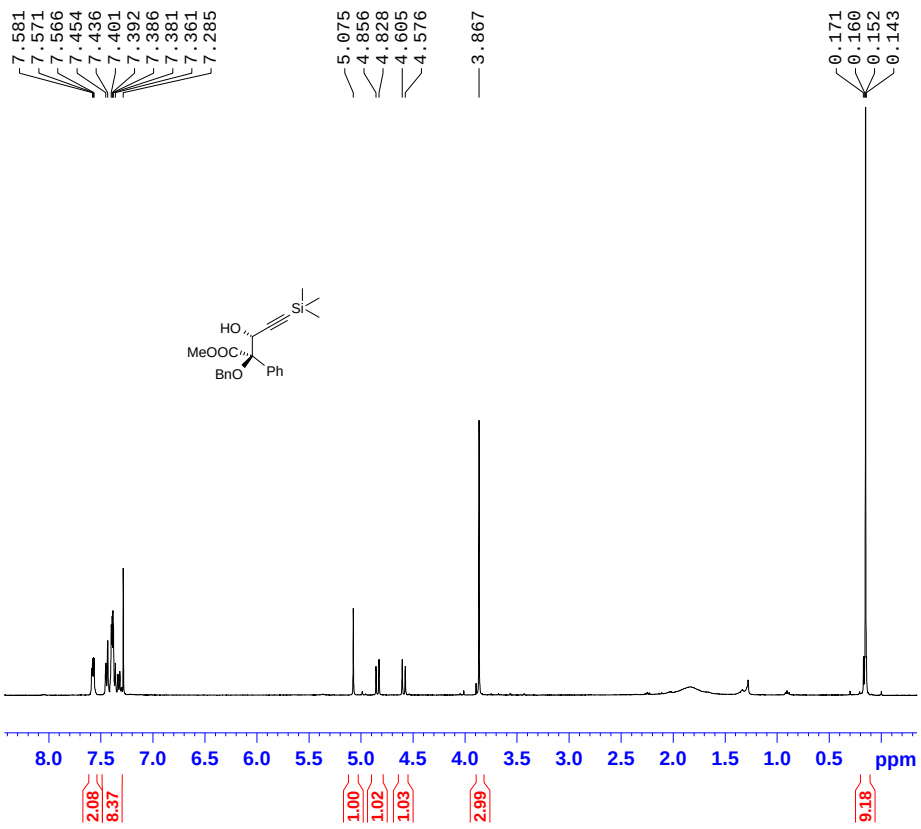


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PROCNO                  1
Date_           20100926
Time                9.42
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    362
DW                 60.400 usec
DE                  6.50 usec
TE                 296.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -1.00 dB
PL1W          10.40389347 W
SFO1          400.1124708 MHz
SI                  32768
SF            400.1100000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

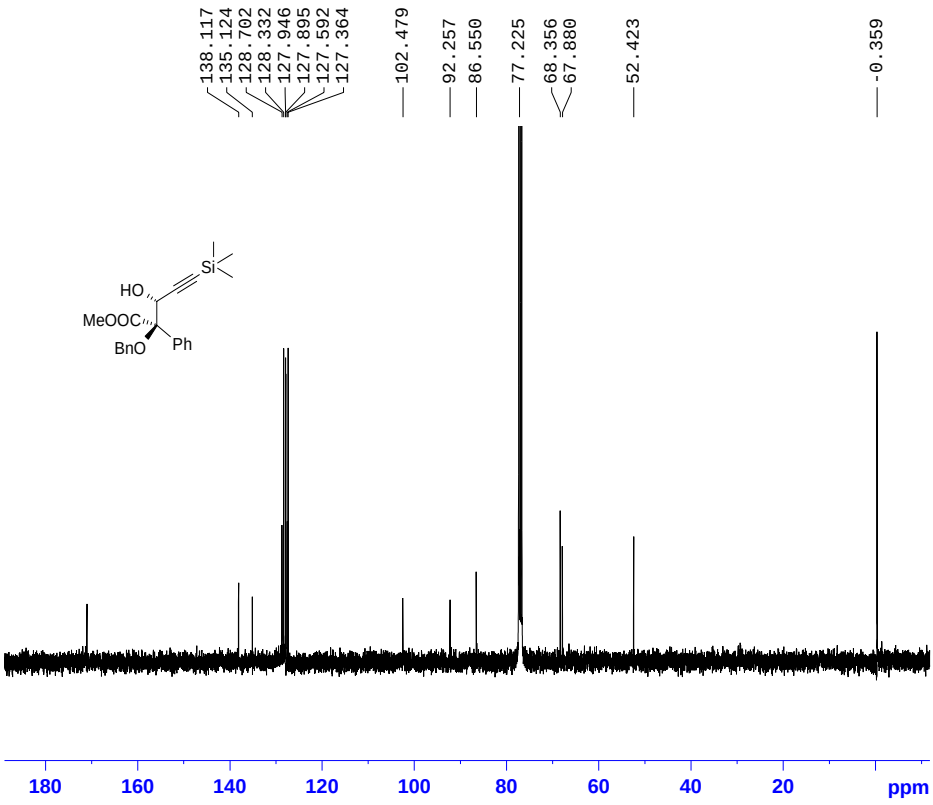


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EXPNO                   2
PROCNO                  1
Date_           20100926
Time               11.43
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                   5160.6
DW                 20.850 usec
DE                  6.50 usec
TE                 297.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.20 usec
PL1                 -2.00 dB
PL1W          61.30850601 W
SFO1          100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                14.46 dB
PL13                14.00 dB
PL2W          10.40389347 W
PL12W          0.29593471 W
PL13W          0.32900000 W
SFO2          400.1116004 MHz
SI                  32768
SF            100.6077400 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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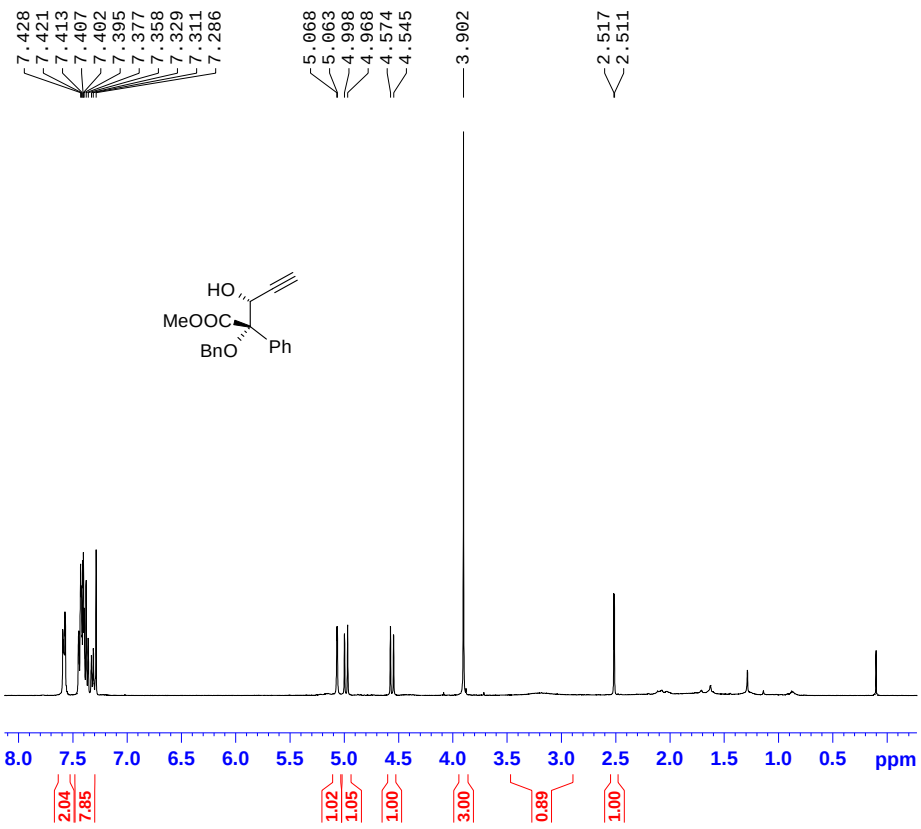


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PROCNO                 1
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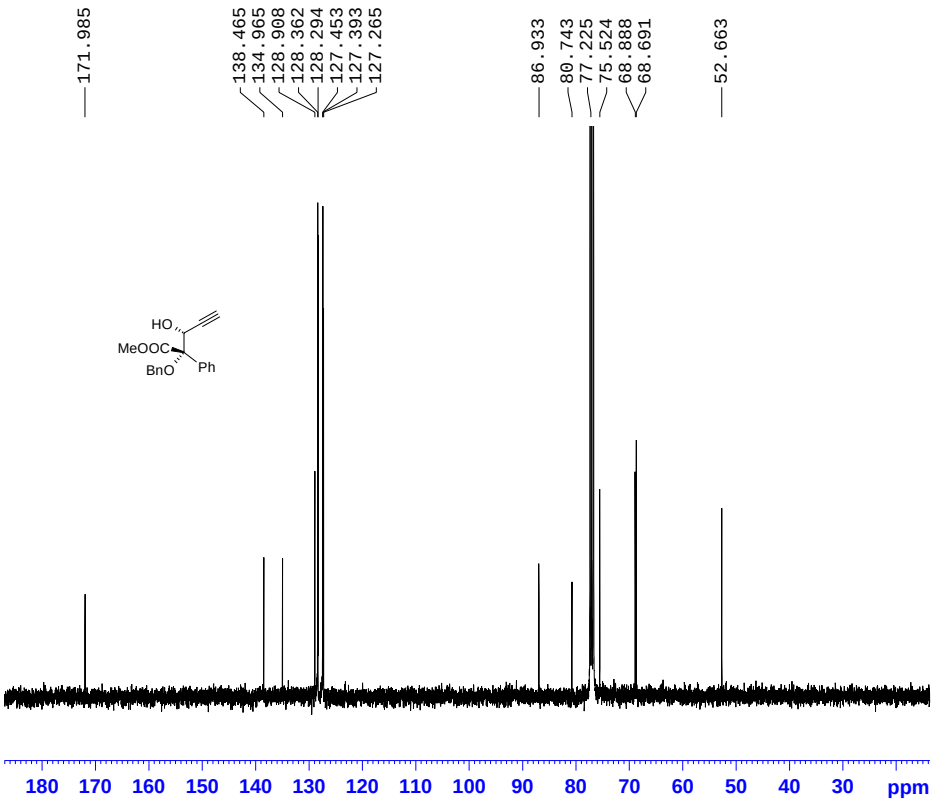
Time                16.32
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   256
DW                60.400 usec
DE                 6.50 usec
TE                299.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

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PROCNO                 1
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Time                20.08
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 32768
DW                20.850 usec
DE                 6.50 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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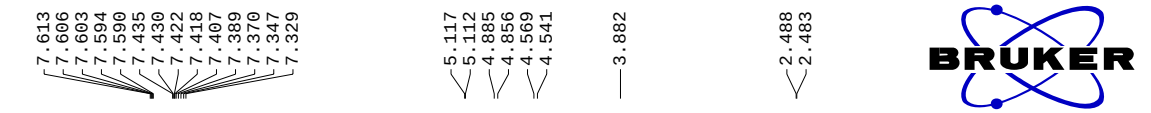

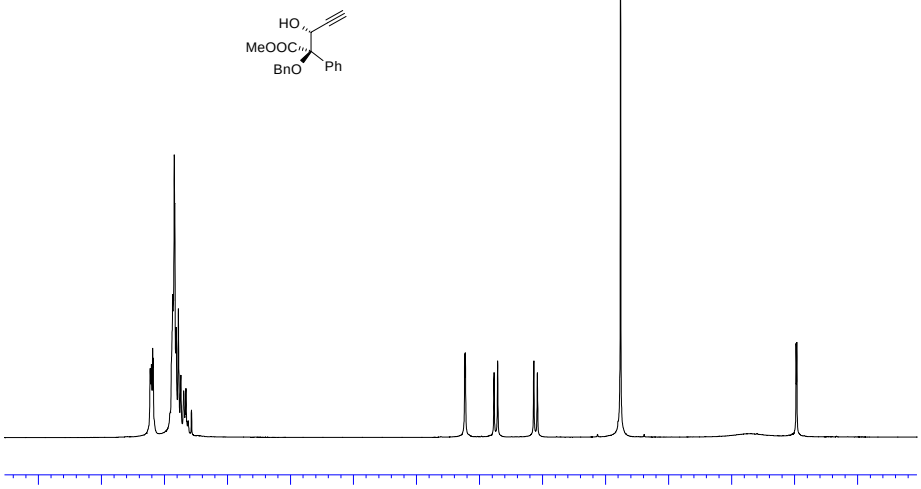


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PROCNO                 1
Date_           20101029
Time                16.40
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 6.50 usec
TE                299.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.50 usec
PL1               -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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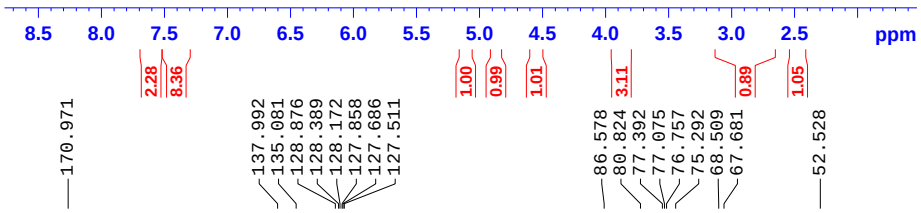

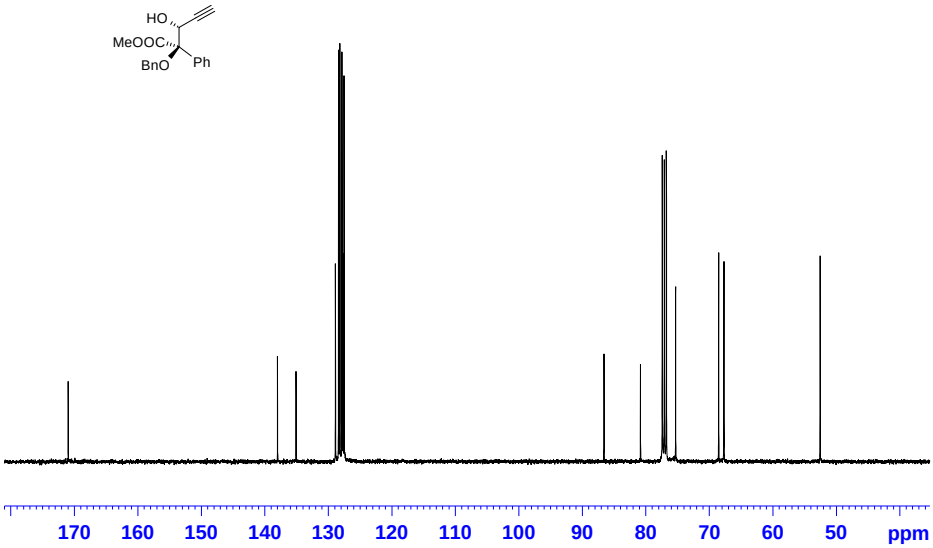


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EXPNO                  2
PROCNO                 1
Date_           20101029
Time                21.13
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 32768
DW                20.850 usec
DE                 6.50 usec
TE                299.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.20 usec
PL1               -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2               -4.00 dB
PL12              10.83 dB
PL13              10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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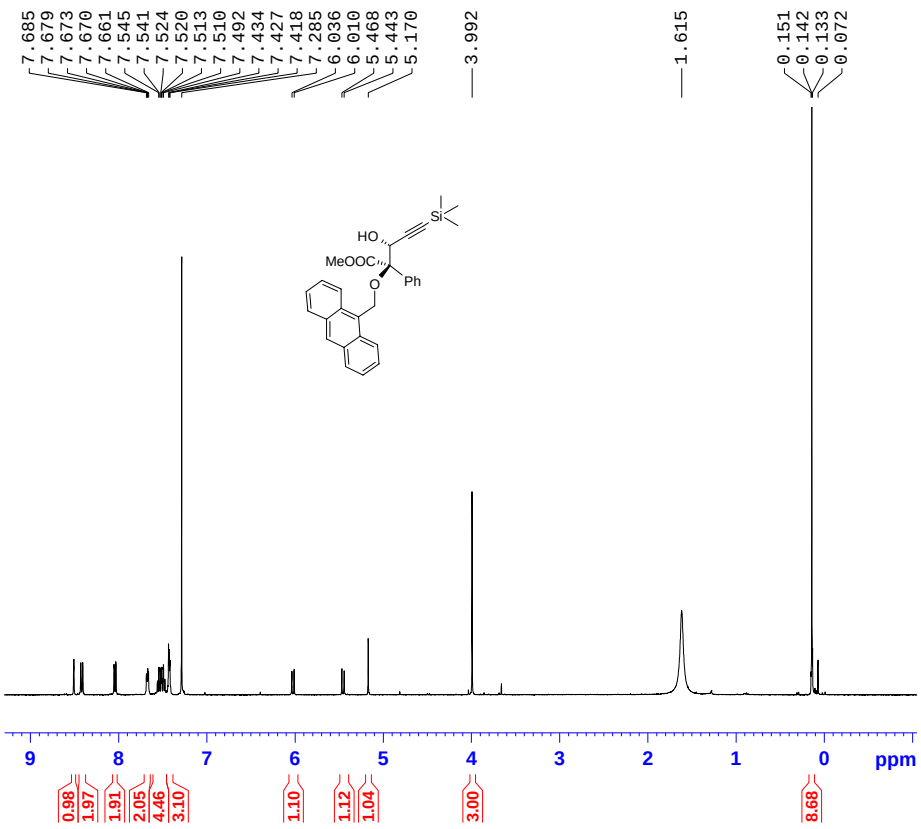


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Time                14.57
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 456.1
DW                60.400 usec
DE                 6.50 usec
TE                296.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.50 usec
PL1                -1.00 dB
PL1W         10.40389347 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```
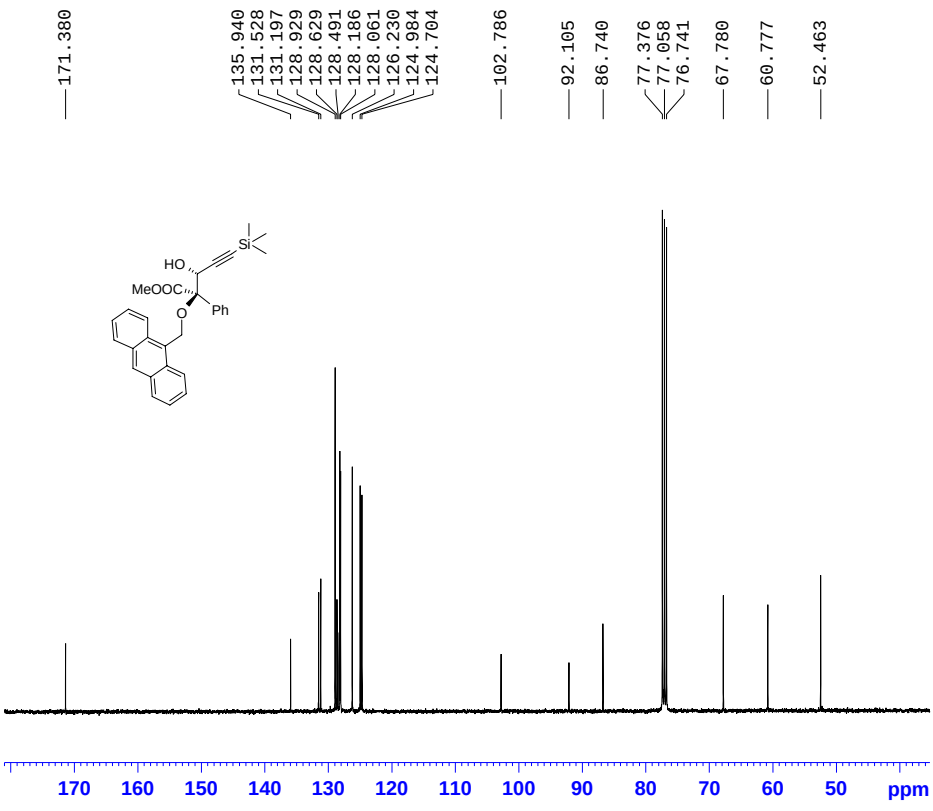
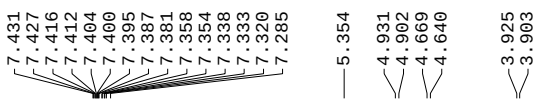
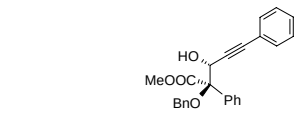
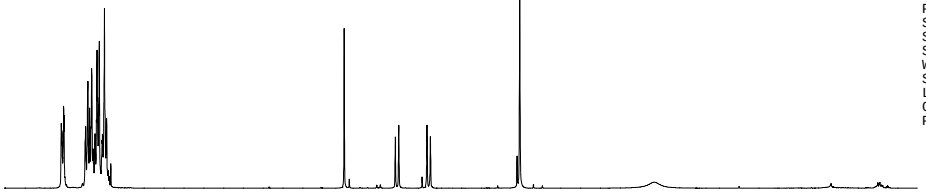
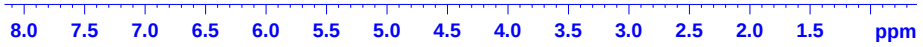




```
NAME      NHB1-2005005-151-02
EXPNO                  2
PROCNO                 1
Date_           20100929
Time                20.08
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 5792.6
DW                20.850 usec
DE                 6.50 usec
TE                297.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.46 dB
PL13               14.00 dB
PL2W         10.40389347 W
PL12W         0.29593471 W
PL13W         0.32900000 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

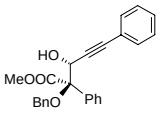


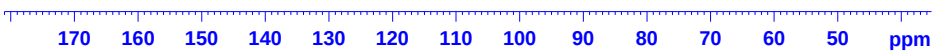




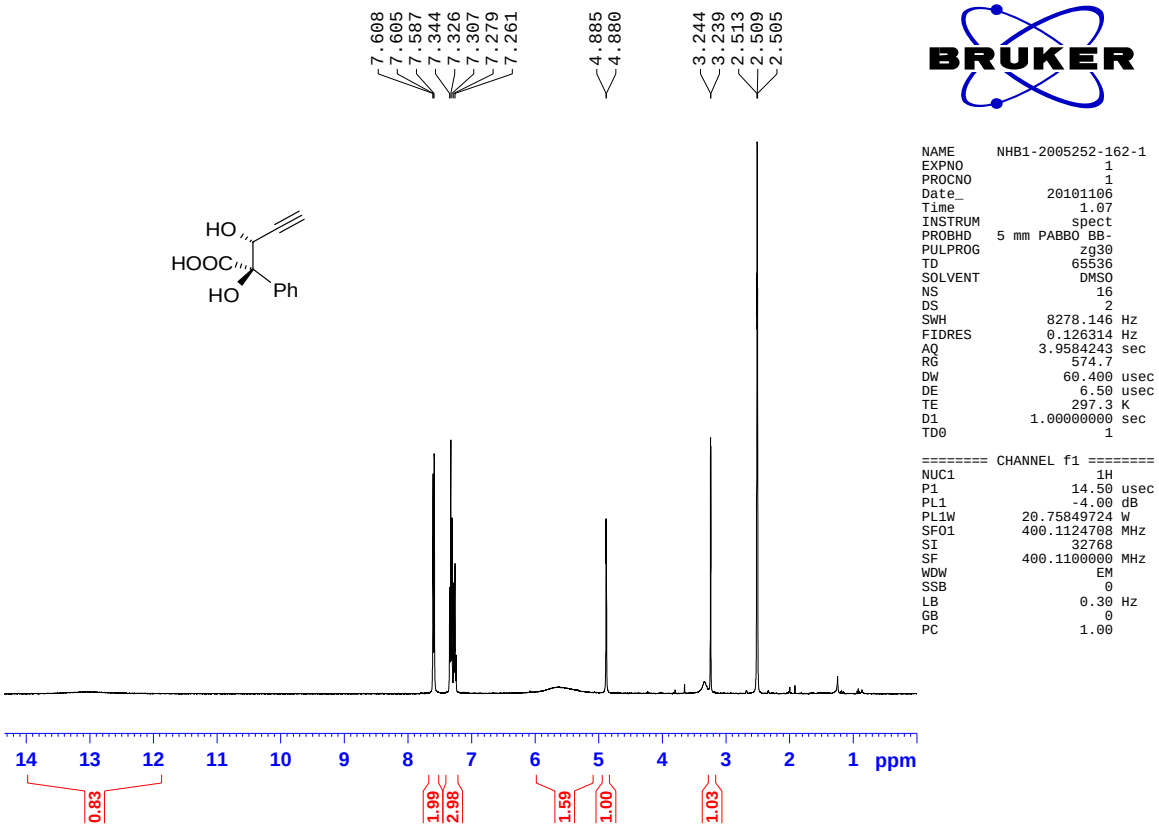



```
NAME      NHB1-2005252-146-1
EXPNO                  1
PROCNO                 1
Date_           20101129
Time               14.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  40.3
DW                60.400 usec
DE                 6.50 usec
TE                297.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.70 usec
PL1                -1.00 dB
PL1W         10.40389347 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

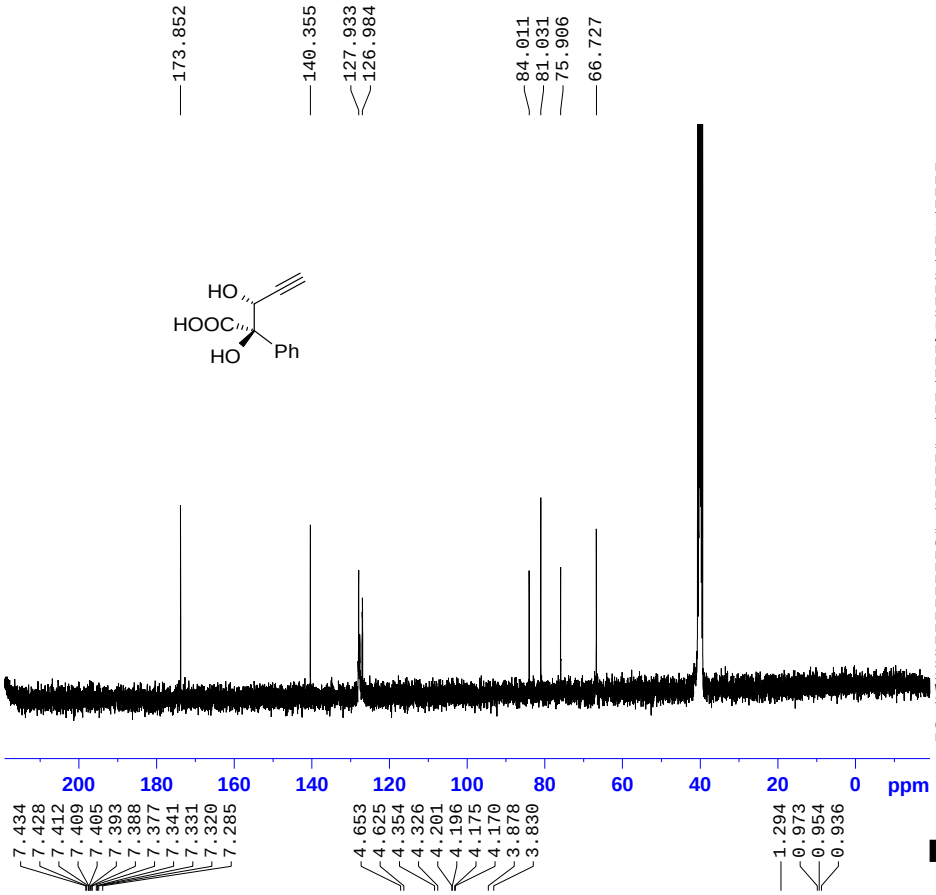




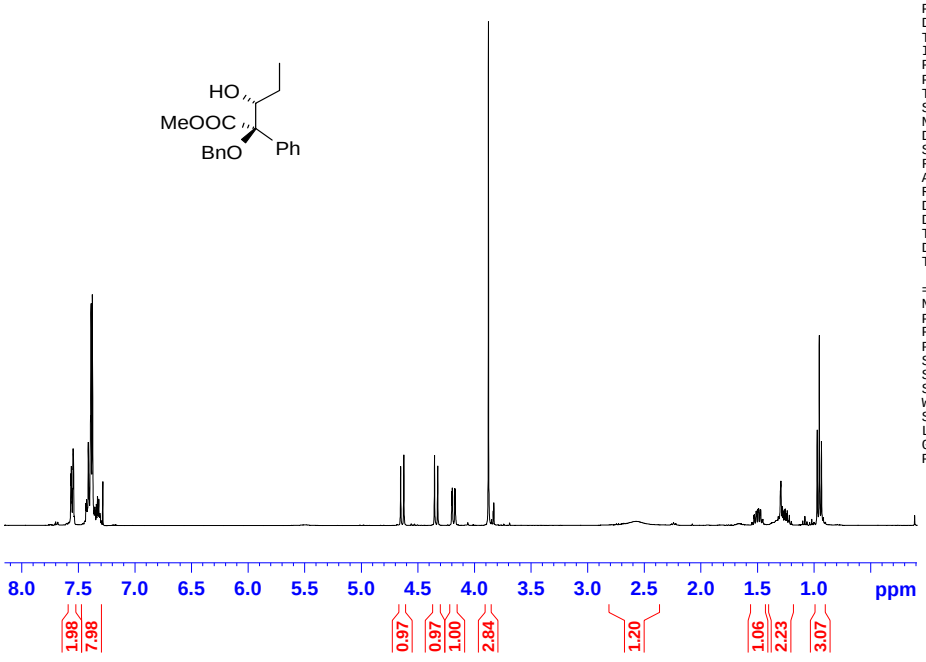

```
NAME      NHB1-2005252-146-1
EXPNO                  2
PROCNO                 1
Date_           20101129
Time               15.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 32768
DW                20.850 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.20 usec
PL1                -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.33 dB
PL13               14.00 dB
PL2W         10.40389347 W
PL12W         0.30492702 W
PL13W         0.32900000 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



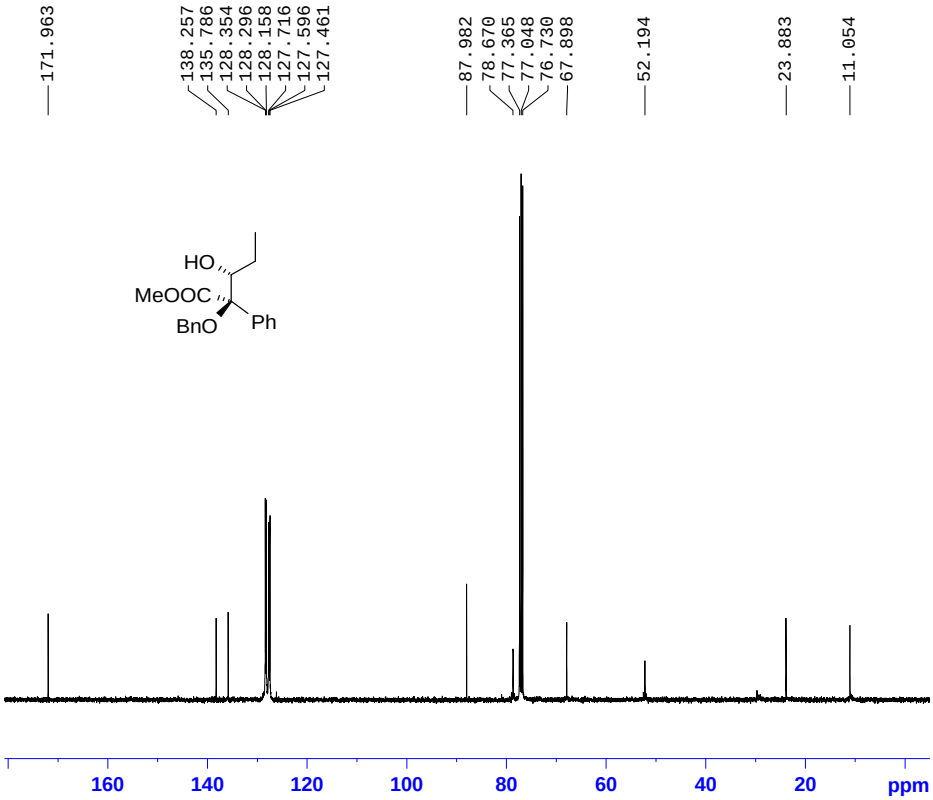

```
NAME      NHB1-2005252-162-1
EXPNO                  1
PROCNO                 1
Date_           20101106
Time                1.07
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 574.7
DW                60.400 usec
DE                 6.50 usec
TE                297.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



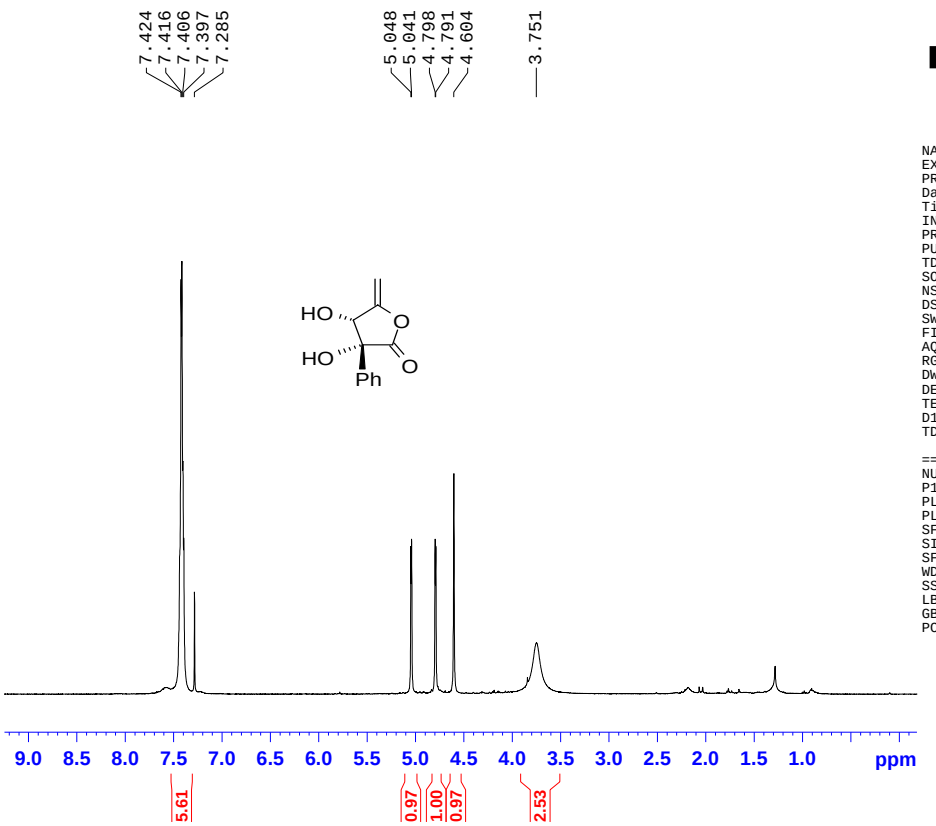

```
NAME      NHB1-2005252-162-1
EXPNO                  2
PROCNO                 1
Date_           20101106
Time                2.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                5160.6
DW                20.850 usec
DE                 6.50 usec
TE                297.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W        61.30850601 W
SFO1        100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W        20.75849724 W
PL12W        0.68264657 W
PL13W        0.68264657 W
SFO2        400.1116004 MHz
SI                 32768
SF          100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



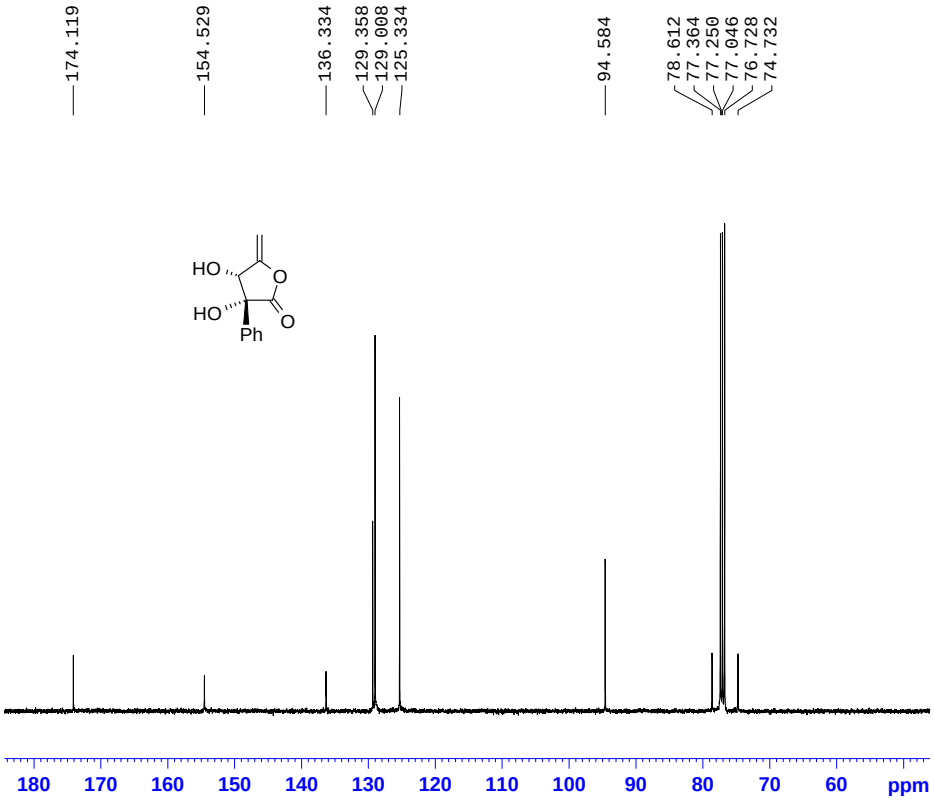

```
NAME      NHB1-2005252-163-1
EXPNO                  1
PROCNO                 1
Date_           20101103
Time               18.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    16
DS                     2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                  90.5
DW                60.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1                -4.00 dB
PL1W        20.75849724 W
SFO1        400.1124708 MHz
SI                 32768
SF          400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



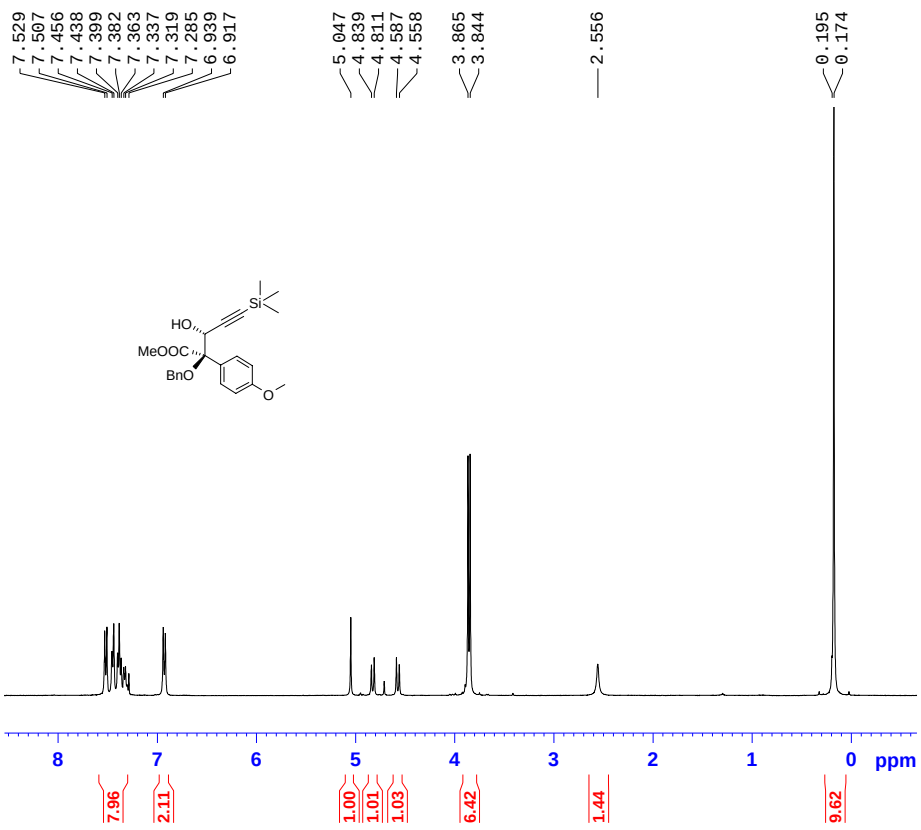

```
NAME      NHB1-2005252-163-1
EXPNO                  2
PROCNO                 1
Date_           20101104
Time                20.03
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG               11585.2
DW                20.850 usec
DE                 6.50 usec
TE                298.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.20 usec
PL1                 -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                10.83 dB
PL13                10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



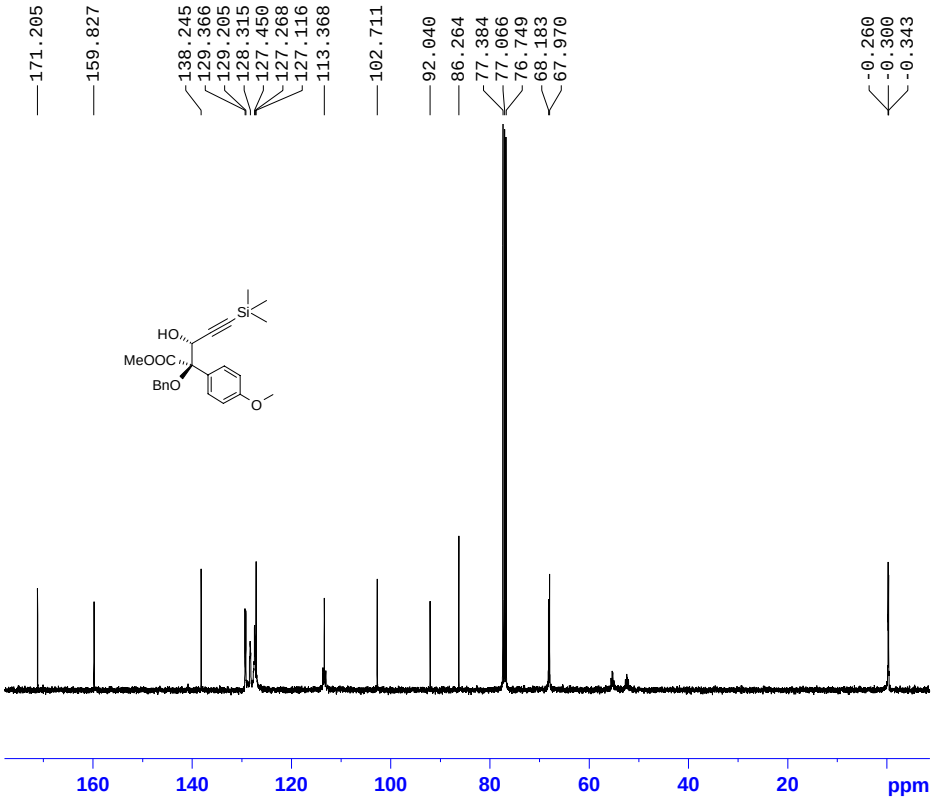

```
NAME      NHB1-2005252-164-1
EXPNO                  4
PROCNO                 1
Date_           20101105
Time                13.58
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                   256
DW                60.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



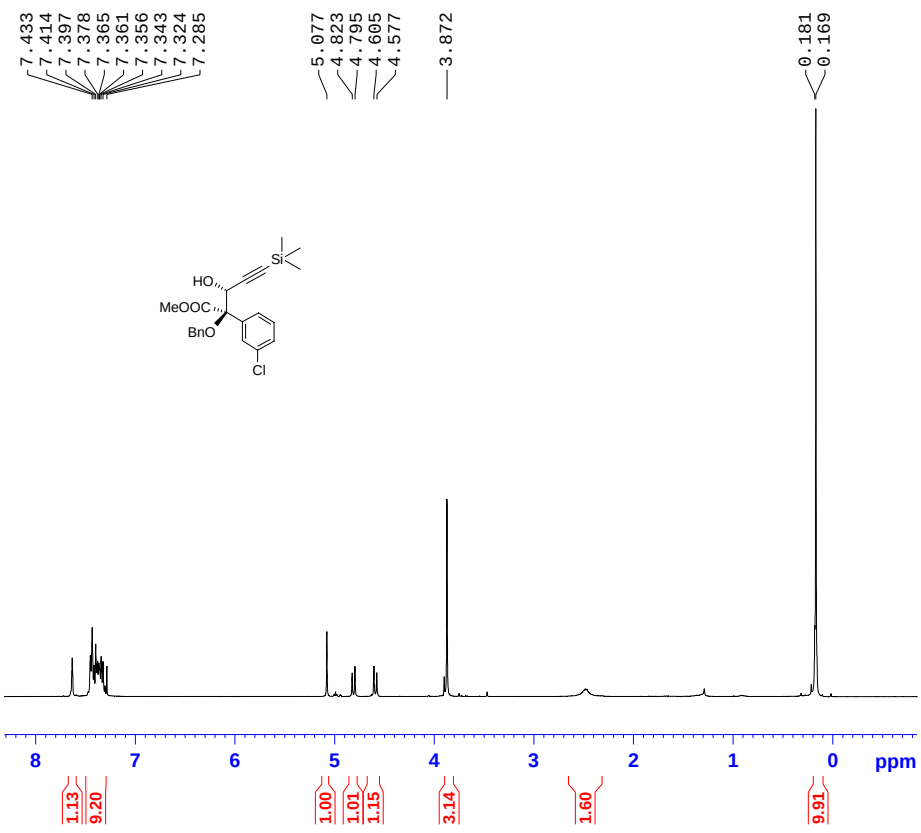

```
NAME      NHB1-2005252-164-1
EXPNO                  2
PROCNO                 1
Date_           20101104
Time                16.07
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                  32768
DW                 20.850 usec
DE                  6.50 usec
TE                 298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.20 usec
PL1                 -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                10.83 dB
PL13                10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                  32768
SF           100.6077400 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



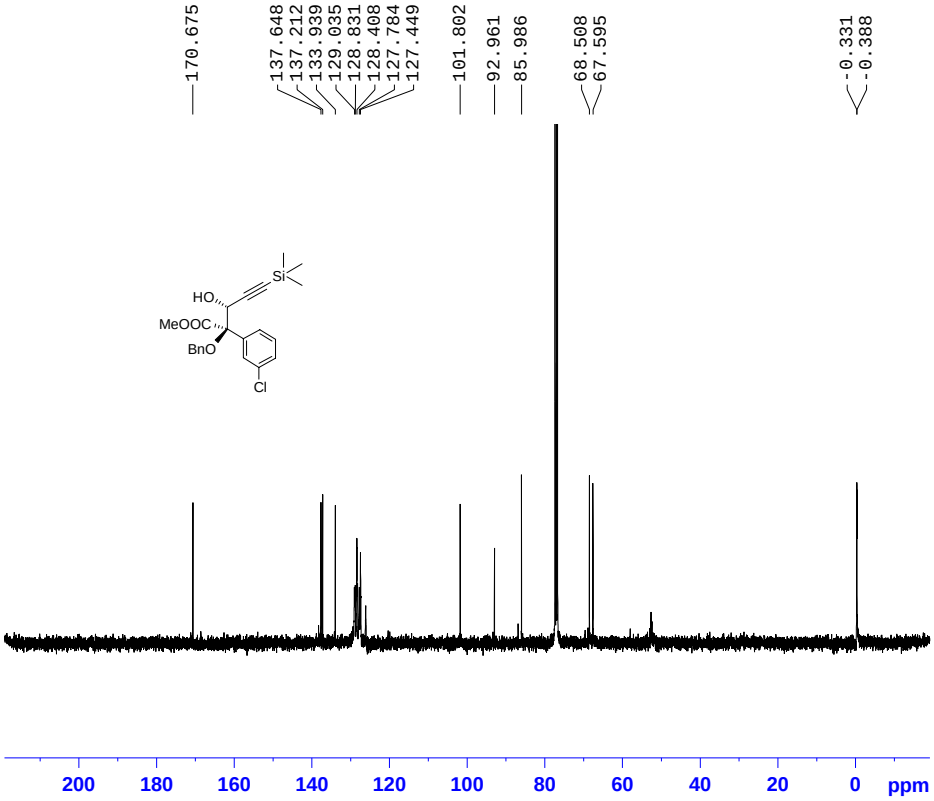

```
NAME      NHB1-2005252-166-1
EXPNO                  1
PROCNO                 1
Date_           20101105
Time                21.50
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                    128
DW                 60.400 usec
DE                  6.50 usec
TE                 297.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                  32768
SF           400.1100000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



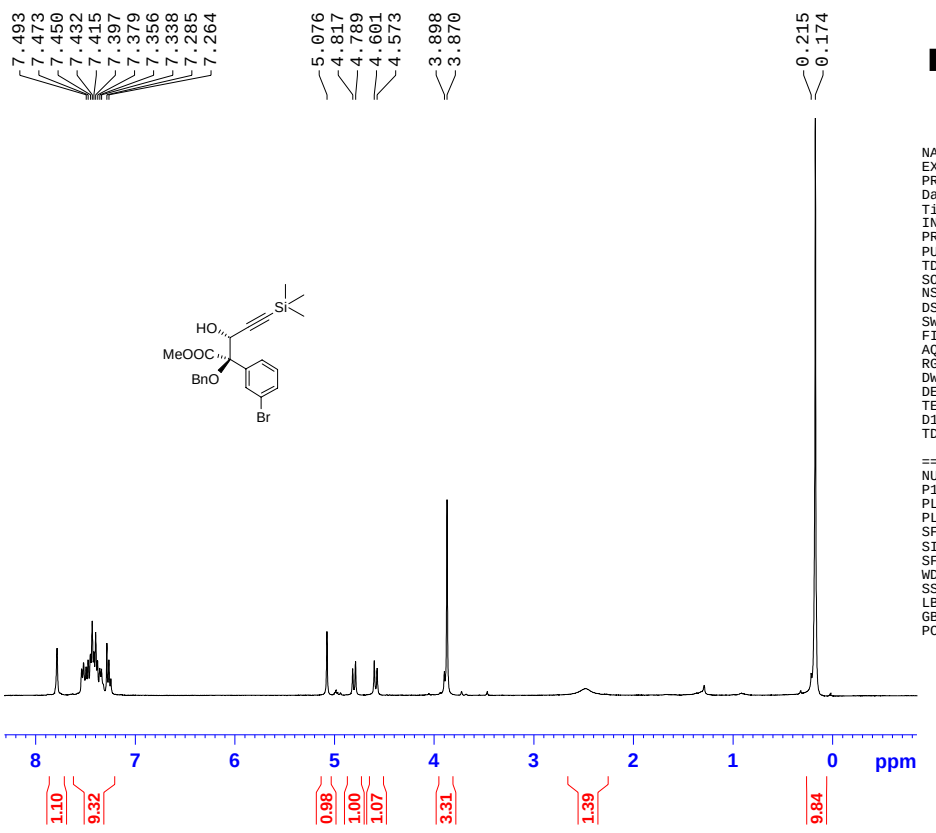

```
NAME      NHB1-2005252-166-1
EXPNO                  2
PROCNO                 1
Date_           20101105
Time               22.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                32768
DW               20.850 usec
DE                6.50 usec
TE                297.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.20 usec
PL1                -2.00 dB
PL1W        61.30850601 W
SFO1        100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              10.83 dB
PL13              10.83 dB
PL2W        20.75849724 W
PL12W        0.68264657 W
PL13W        0.68264657 W
SFO2        400.1116004 MHz
SI                32768
SF          100.6077400 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



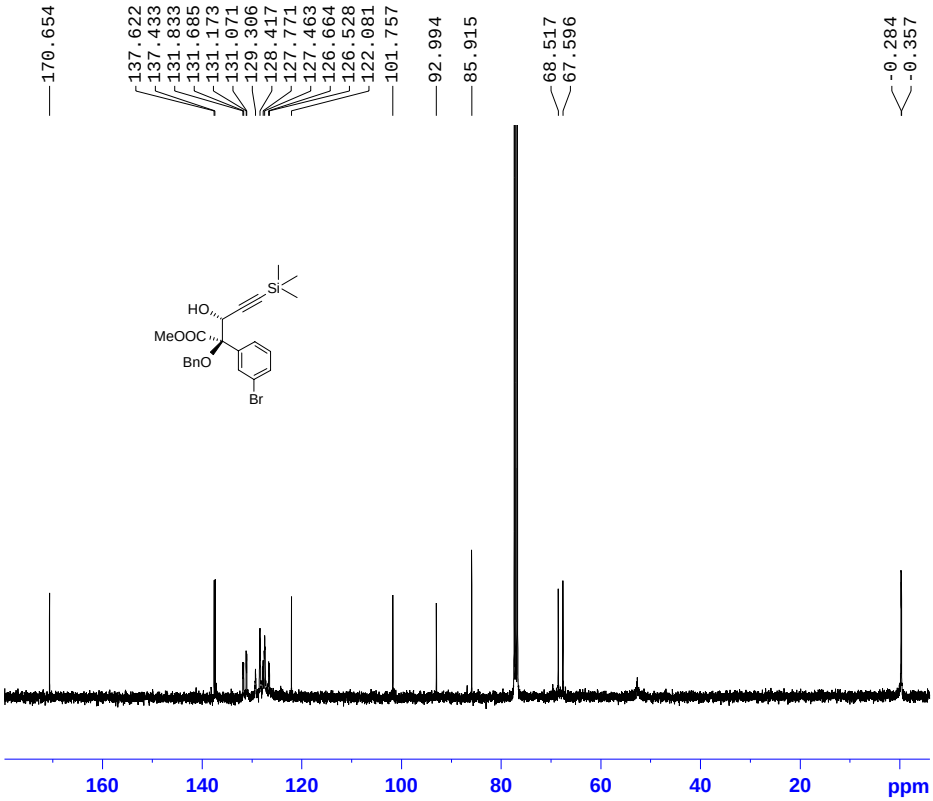

```
NAME      NHB1-2005252-167-1
EXPNO                  1
PROCNO                 1
Date_           20101105
Time               22.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                  256
DW               60.400 usec
DE                6.50 usec
TE                297.6 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -4.00 dB
PL1W        20.75849724 W
SFO1        400.1124708 MHz
SI                32768
SF          400.1100000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



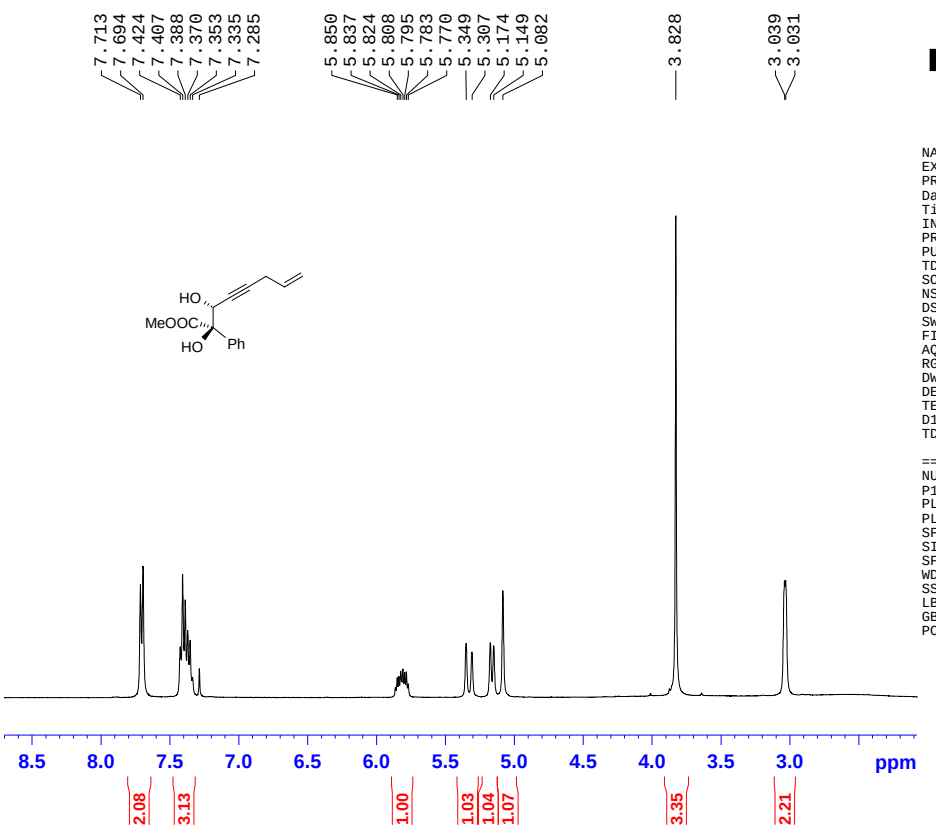

```
NAME      NHB1-2005252-167-1
EXPNO                  2
PROCNO                 1
Date_           20101105
Time               23.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                  8192
DW                20.850 usec
DE                 6.50 usec
TE                297.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.20 usec
PL1                 -2.00 dB
PL1W         61.30850601 W
SFO1        100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                10.83 dB
PL13                10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2        400.1116004 MHz
SI                 32768
SF          100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



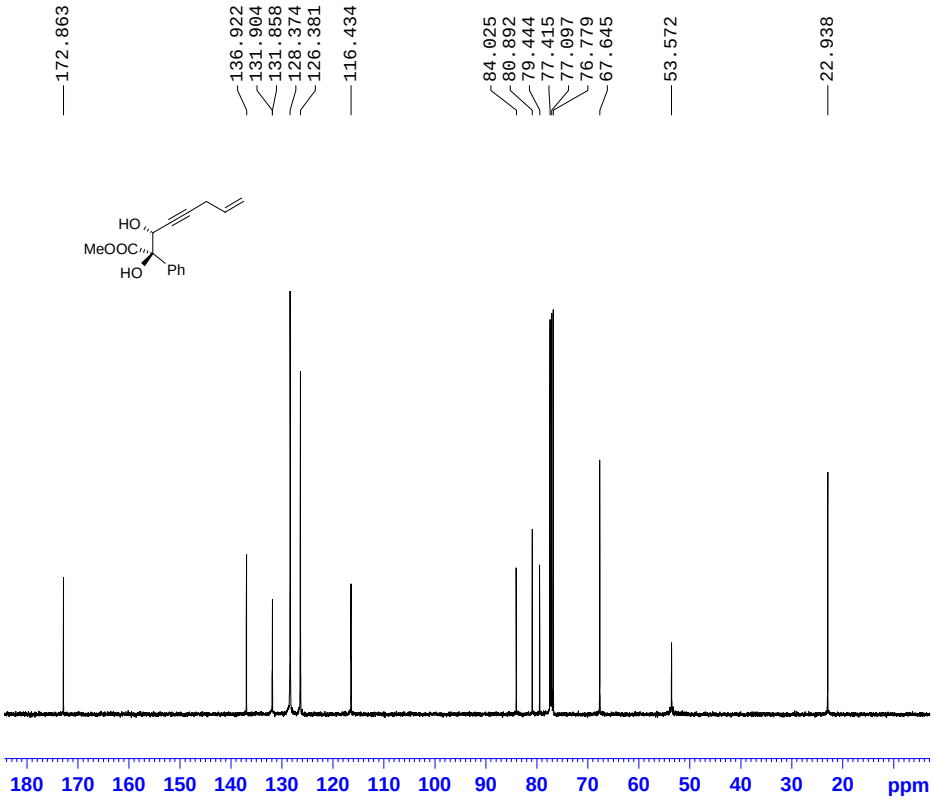

```
NAME      NHB1-2005252-168-1
EXPNO                  1
PROCNO                 1
Date_           20101106
Time               0.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 usec
DE                 6.50 usec
TE                297.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1        400.1124708 MHz
SI                 32768
SF          400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



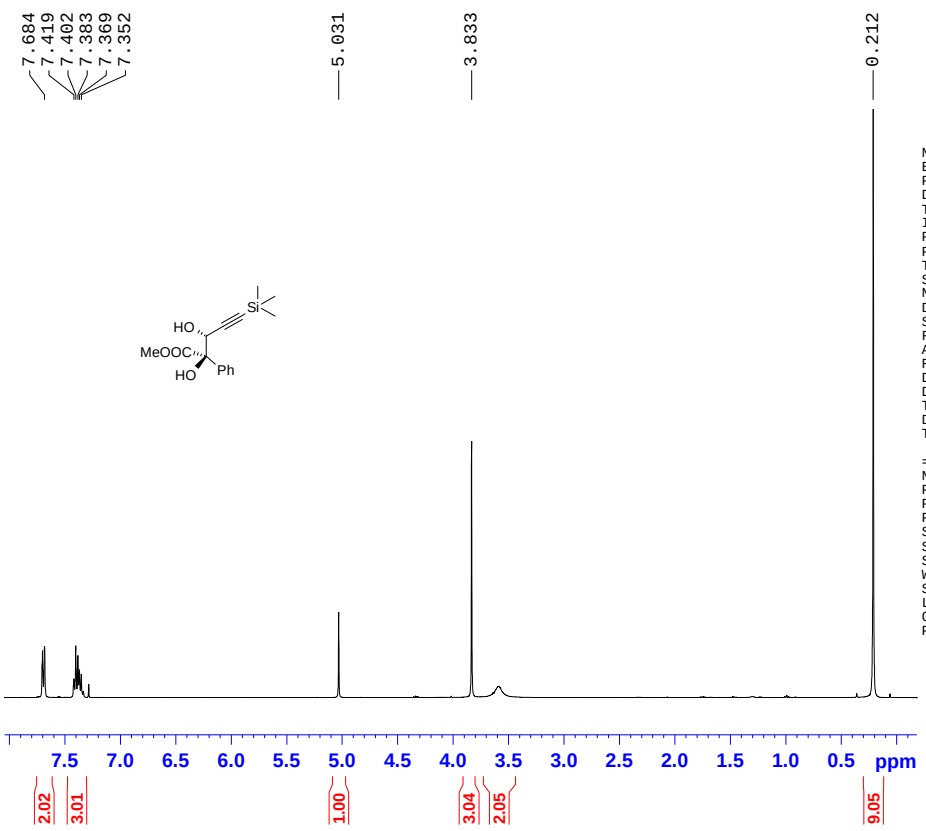

```
NAME      NHB1-2005252-168-1
EXPNO                  2
PROCNO                 1
Date_           20101106
Time                1.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               9195.2
DW              20.850 usec
DE               6.50 usec
TE              297.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.20 usec
PL1               -2.00 dB
PL1W        61.30850601 W
SFO1        100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -4.00 dB
PL12              10.83 dB
PL13              10.83 dB
PL2W        20.75849724 W
PL12W        0.68264657 W
PL13W        0.68264657 W
SFO2        400.1116004 MHz
SI                 32768
SF          100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



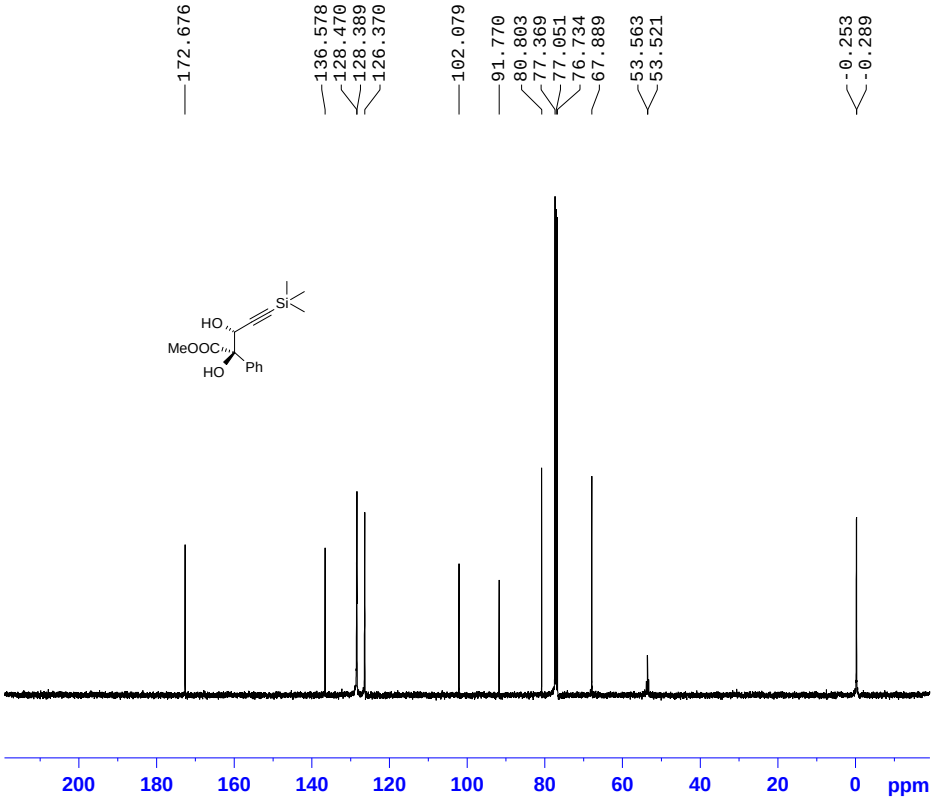

```
NAME      NHB1-2005252-172-1
EXPNO                  3
PROCNO                 1
Date_           20101105
Time               14.45
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.50 usec
PL1               -4.00 dB
PL1W        20.75849724 W
SFO1        400.1124708 MHz
SI                 32768
SF          400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



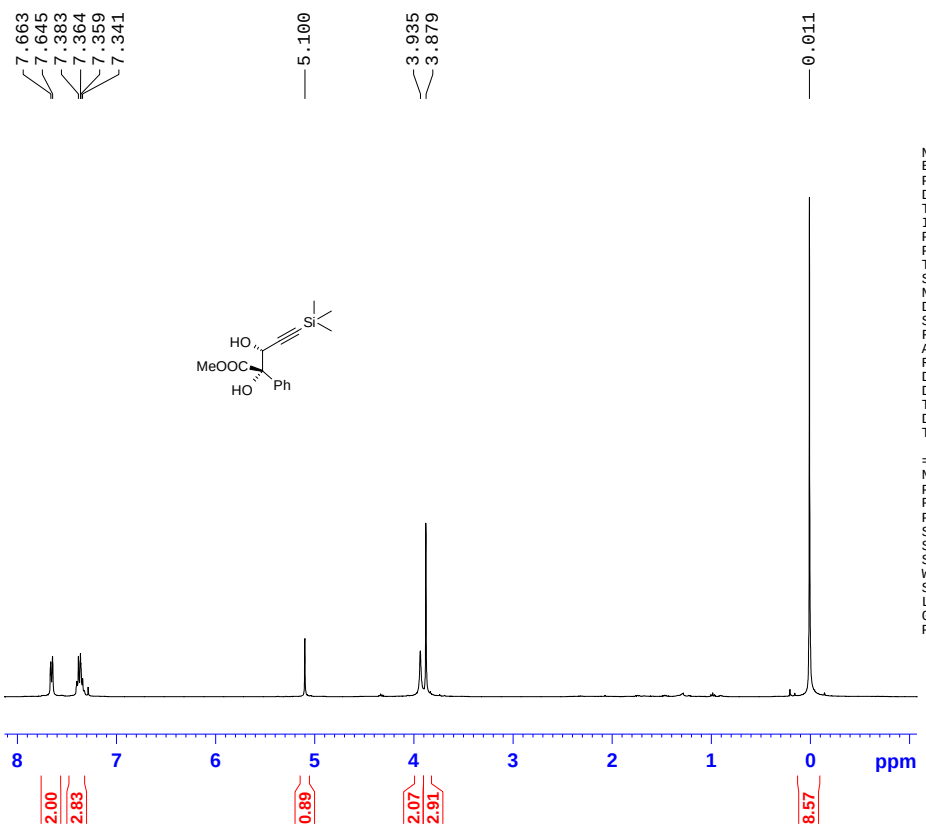

```
NAME      NHB1-2005252-172-1
EXPNO                  2
PROCNO                 1
Date_           20101104
Time                21.11
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                  32768
DW                 20.850 usec
DE                  6.50 usec
TE                 298.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.20 usec
PL1                 -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                10.83 dB
PL13                10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                  32768
SF           100.6077400 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



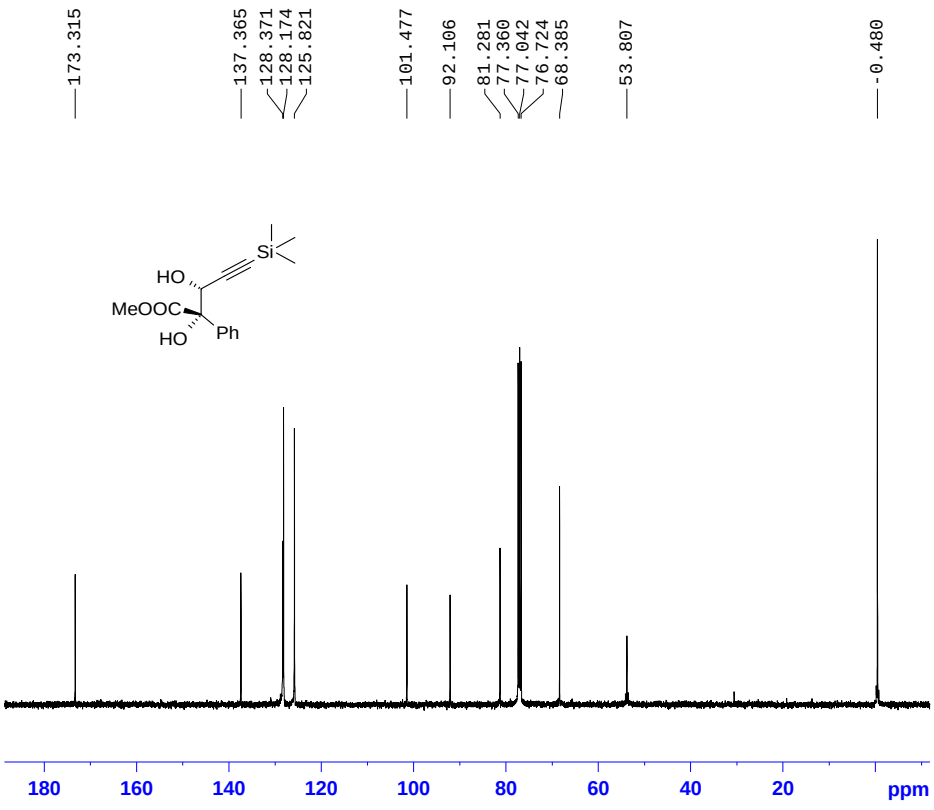

```
NAME      NHB1-2005252-178-A
EXPNO                   1
PROCNO                  1
Date_            20101111
Time                20.01
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                    128
DW                 60.400 usec
DE                  6.50 usec
TE                 299.2 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.50 usec
PL1                 -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                  32768
SF           400.1100000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



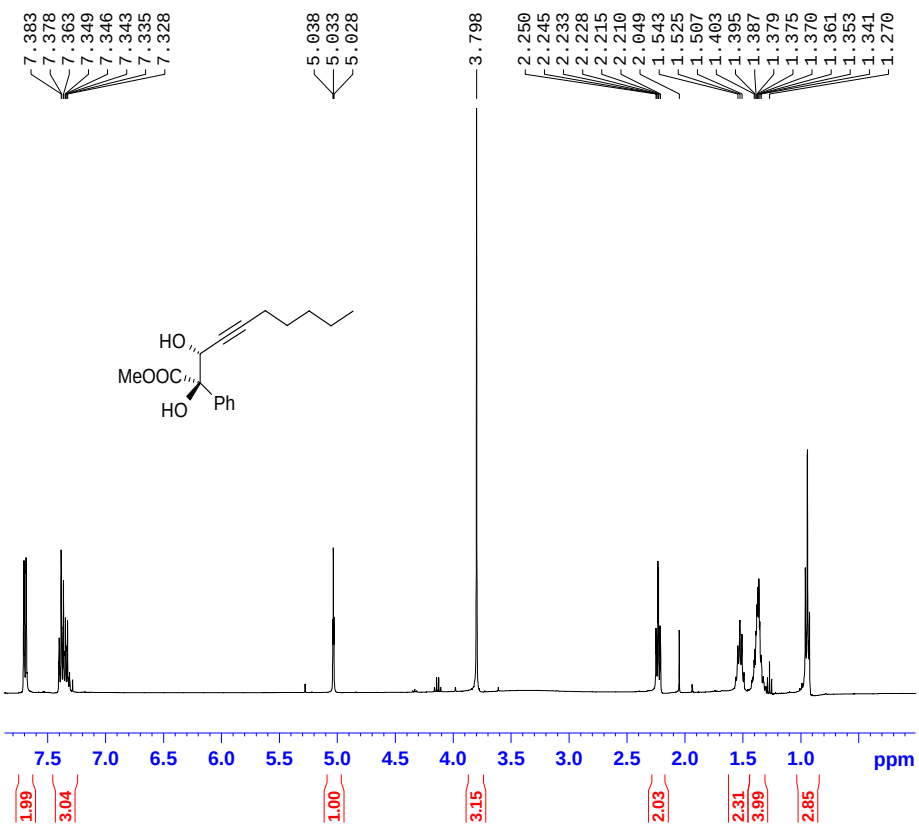

```
NAME      NHB1-2005252-178-A
EXPNO                  2
PROCNO                 1
Date_           20101111
Time              21.14
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                9195.2
DW                20.850 usec
DE                 6.50 usec
TE                299.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W        61.30850601 W
SFO1        100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W        20.75849724 W
PL12W        0.68264657 W
PL13W        0.68264657 W
SFO2        400.1116004 MHz
SI                32768
SF          100.6077400 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



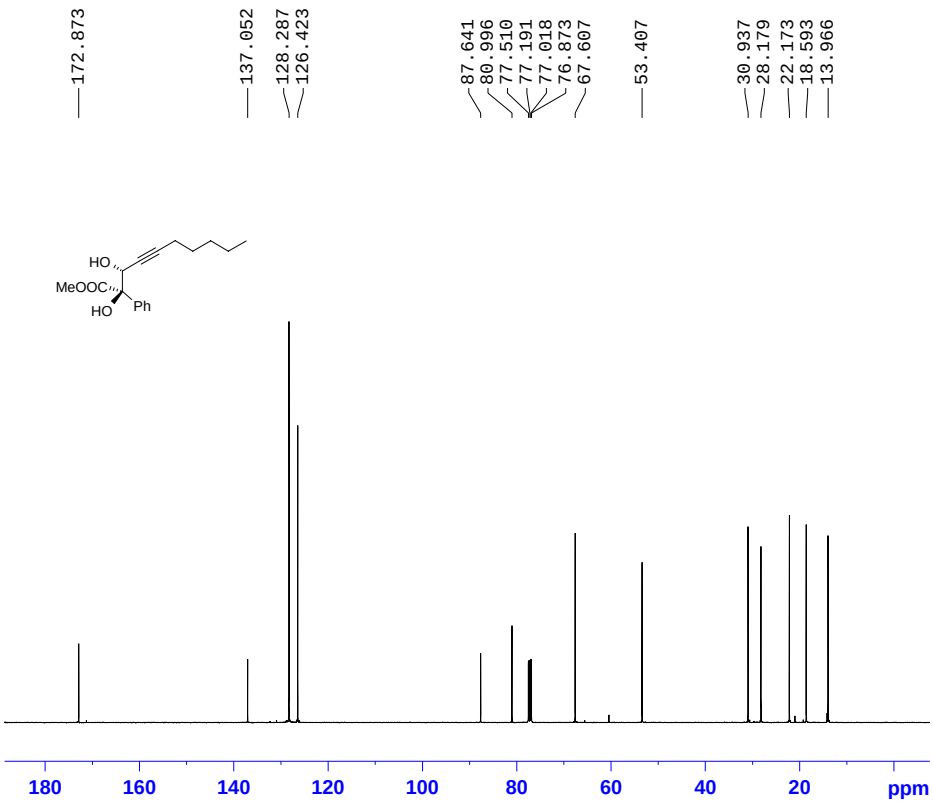

```
NAME      NHB1-2005252-178-B
EXPNO                  1
PROCNO                 1
Date_           20101111
Time               20.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                  90.5
DW                 60.400 usec
DE                  6.50 usec
TE                 299.3 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W          20.75849724 W
SFO1          400.1124708 MHz
SI                 32768
SF            400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



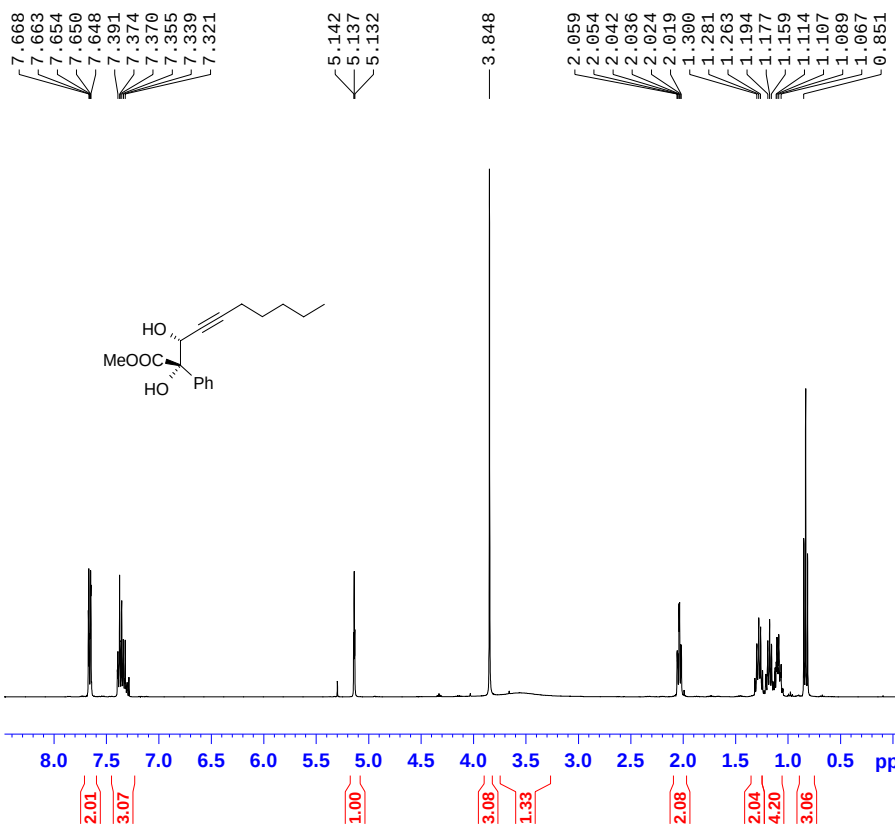

```
NAME      NHB1-2005252-178-B
EXPNO                  2
PROCNO                 1
Date_           20101111
Time               22.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                 9195.2
DW                 20.850 usec
DE                  6.50 usec
TE                 299.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W          61.30850601 W
SFO1          100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W          20.75849724 W
PL12W          0.68264657 W
PL13W          0.68264657 W
SFO2          400.1116004 MHz
SI                 32768
SF            100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



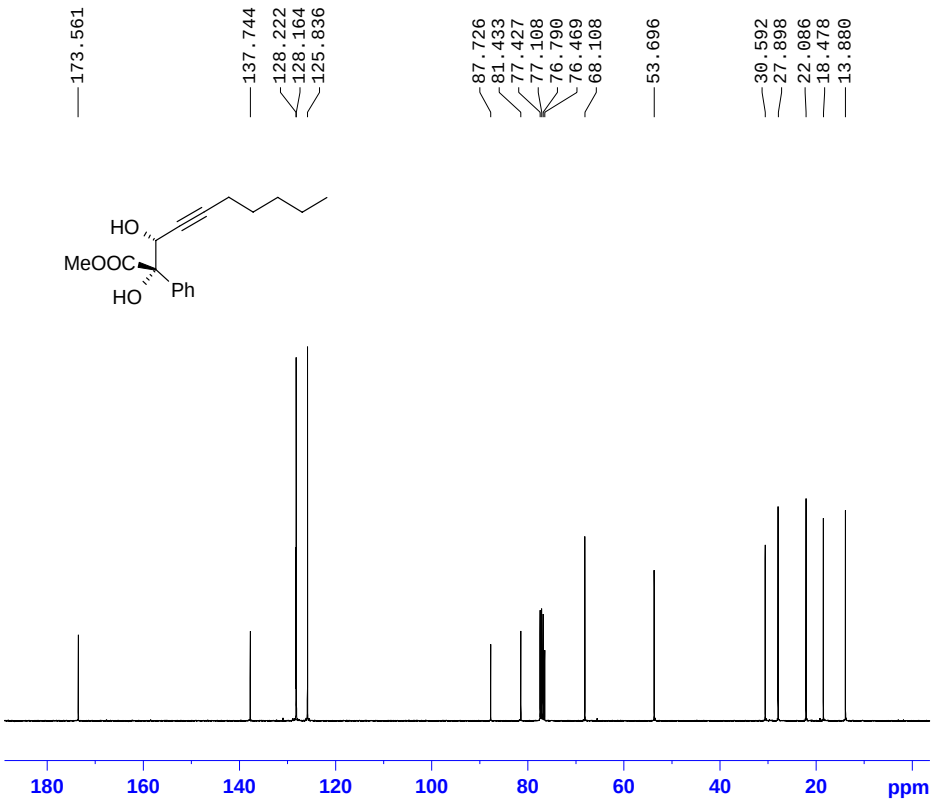

```
NAME      NHB1-2005252-179-1-A
EXPNO                1
PROCNO               1
Date_         20101115
Time              1.32
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                22.6
DW              60.400 usec
DE               6.50 usec
TE              297.3 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                14.50 usec
PL1               -4.00 dB
PL1W        20.75849724 W
SFO1        400.1124708 MHz
SI               32768
SF          400.1100000 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



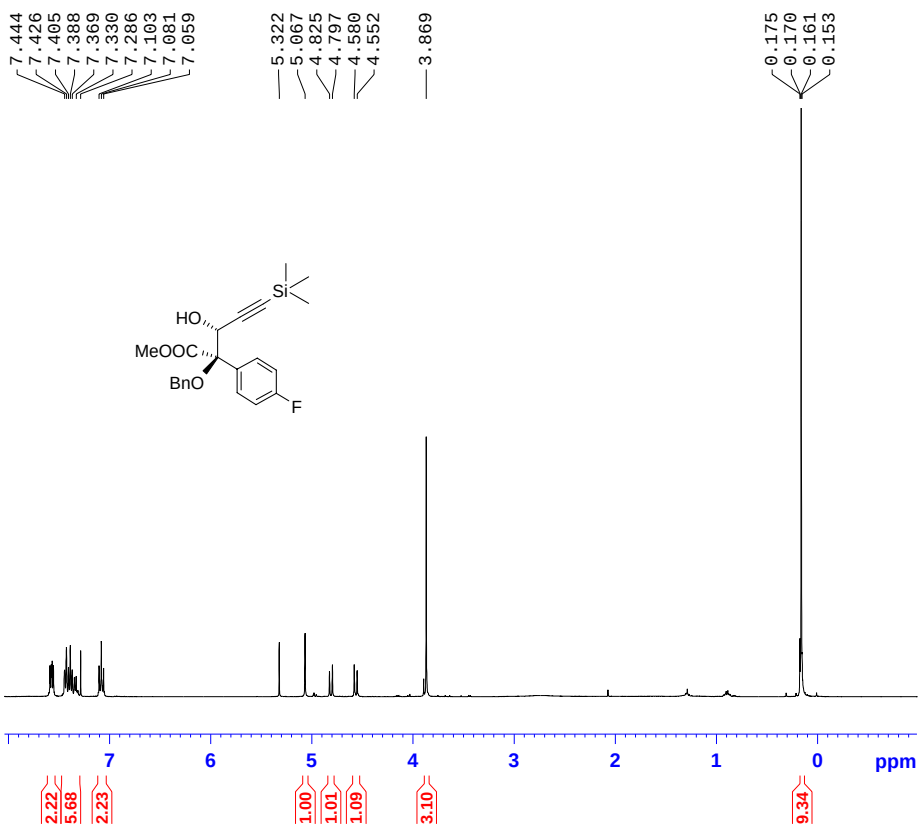

```
NAME      NHB1-2005252-179-1-A
EXPNO                2
PROCNO               1
Date_         20101115
Time              2.32
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG               9195.2
DW              20.850 usec
DE               6.50 usec
TE              298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                9.20 usec
PL1               -2.00 dB
PL1W        61.30850601 W
SFO1       100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               -4.00 dB
PL12              10.83 dB
PL13              10.83 dB
PL2W        20.75849724 W
PL12W        0.68264657 W
PL13W        0.68264657 W
SFO2       400.1116004 MHz
SI               32768
SF          100.6077400 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



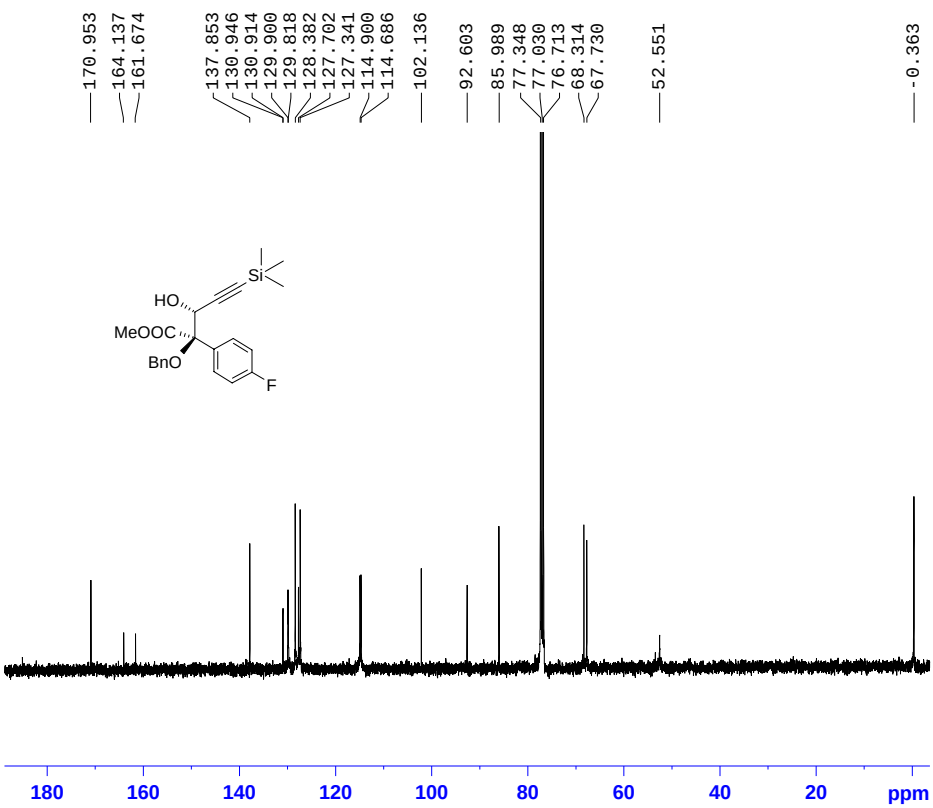

```
NAME      NHB1-2005252-179-2
EXPNO                  1
PROCNO                 1
Date_           20101115
Time                3.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES             0.126314 Hz
AQ              3.9584243 sec
RG                  35.9
DW                 60.400 usec
DE                  6.50 usec
TE                297.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W         20.75849724 W
SFO1         400.1124708 MHz
SI                 32768
SF           400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



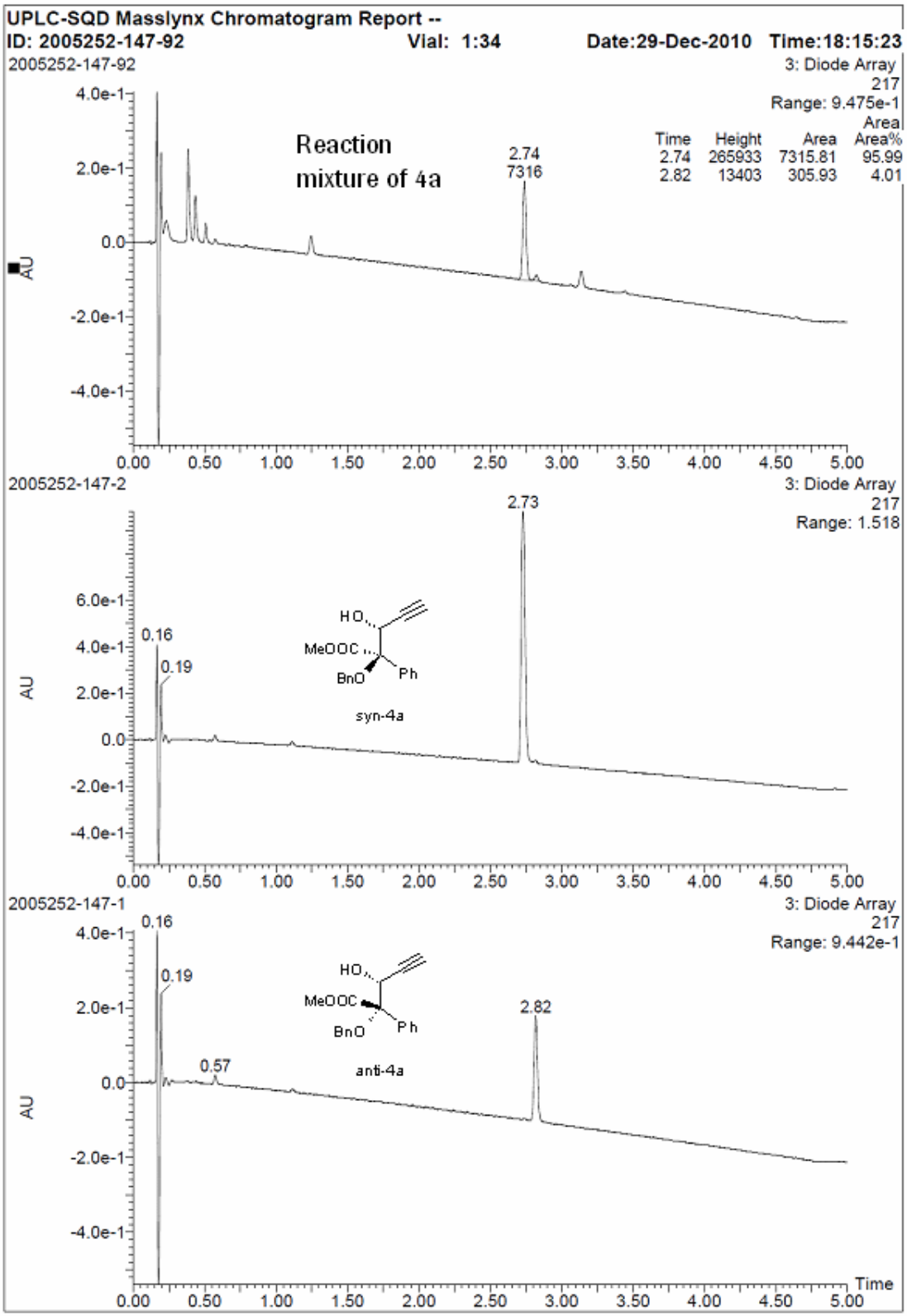

```
NAME      NHB1-2005252-179-2
EXPNO                  2
PROCNO                 1
Date_           20101115
Time                4.45
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES             0.365918 Hz
AQ              1.3664756 sec
RG                  32768
DW                 20.850 usec
DE                  6.50 usec
TE                297.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W         61.30850601 W
SFO1         100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W         20.75849724 W
PL12W         0.68264657 W
PL13W         0.68264657 W
SFO2         400.1116004 MHz
SI                 32768
SF           100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME      NHB1-2005252-183-1
EXPNO                  1
PROCNO                 1
Date_           20101116
Time               20.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                   362
DW                  60.400 usec
DE                   6.50 usec
TE                  297.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.50 usec
PL1                -4.00 dB
PL1W          20.75849724 W
SFO1          400.1124708 MHz
SI                 32768
SF            400.1100000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
NAME      NHB1-2005252-183-1
EXPNO                  2
PROCNO                 1
Date_           20101116
Time               21.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                  9195.2
DW                  20.850 usec
DE                   6.50 usec
TE                  297.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.20 usec
PL1                -2.00 dB
PL1W          61.30850601 W
SFO1          100.6178003 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               10.83 dB
PL13               10.83 dB
PL2W          20.75849724 W
PL12W          0.68264657 W
PL13W          0.68264657 W
SFO2          400.1116004 MHz
SI                 32768
SF            100.6077400 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


UPLC-SQD Masslynx Chromatogram Report --
ID: 2005252-147-92 Vial: 1:34 Date:29-Dec-2010 Time:18:15:23
2005252-147-92
3: Diode Array
217
Range: 9.475e-1
Reaction mixture of 4a
Time Height Area Area%
2.74 265933 7315.81 95.99
2.82 13403 305.93 4.01
2.74
7316
AU
2005252-147-2
3: Diode Array
217
Range: 1.518
2.73
0.16
0.19
HO
MeOOC
BnO
Ph
syn-4a
2005252-147-1
3: Diode Array
217
Range: 9.442e-1
0.16
0.19
0.57
2.82
anti-4a
Time