Supporting Information

An efficient approach to homocoupling of terminal alkynes: Solvent-free synthesis of 1,3-diynes using catalytic Cu(II) and base

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1. General Remarks:

Flash column chromatography was performed using silica gel (300–400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 200–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). NMR spectra were recorded in $CDCl_3$ on a Varian Inova-300 or 400 MHz NMR spectrometer with TMS as an internal reference. Copies of 1H NMR and 13C NMR spectra of the unsymmetric 1,3-diynes are provided.

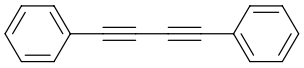
2. General procedure for the preparation of unsymmetric 1,3-diynes. Synthesis of 1-methoxy-4-(phenylbuta-1,3-diynyl)benzene (3a):

A typical reaction procedure: to a mixture of $CuCl_2$ (5.0 mol%) and Et_3N (5.0 mol%), phenylacetylene (3 mmol) and p-methoxyphenylacetylene (0.5mmol) were added. The mixture was stirred at 60 ^oC in air for 10 hours. After cooling to room temperature, the mixture was diluted with ethyl acetate and filtered. The filtrate was removed under reduced pressure to get the crude product, which was further purified by silica gel chromatography (petroleum ether as eluent) to yield corresponding unsymmetric 1,3-diynes.

3. General procedure of catalyst reactivation

The reaction mixture was cooled to room temperature, diluted with dichloromethane, and then filtered. The filtration residue was placed in a beaker, where 2mL HCl of 0.01M was added for every 10 mg filtration residue. The mixture was dried in vacuum for 6 h at 100°C to produce the recycled catalyst.

4. Spectral data of the compounds:



2a

1,4-diphenyl buta-1,3-diyne (2a) [1] : **2a** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield >96%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.52 (d, J = 6.87 Hz, 4 H), 7.28-7.40 (m, 6 H). ^{13}C NMR (100MHz, $CDCl_3$): δ_C 132.4, 129.2, 128.4, 121.7, 81.5, 73.8.

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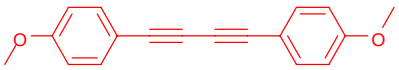
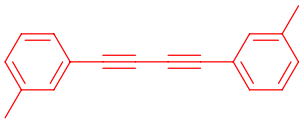


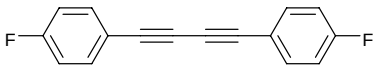
2b

1,4-bis(p-methoxyphenyl)buta-1,3-diyne (2b) [2, 3]**: 2b** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield 99%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.46 (d, J = 8.4 Hz, 4H), 6.85 (d, J = 8.4 Hz, 4H), 3.81 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 160.42, 134.23, 114.32, 114.11, 81.42, 73.14, 55.52.



2c

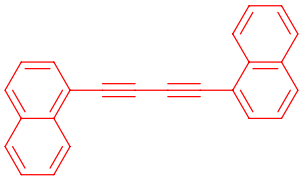
1,4-bis(m-methylphenyl)buta-1,3-diyne (2c) [4] **: 2c** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield >98%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.33 (m, 4H), 7.24-7.18 (m, 4H), 2.33 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 138.1, 133.0, 130.1, 129.6, 128.3, 121.6, 81.6, 73.6, 21.2.



2d

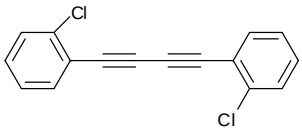
1,4-bis(p-fluorophenyl)buta-1,3-diyne (2d) [4] **: 2d** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield 80%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.53-7.48 (m, 4H), 7.06-7.00 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C

164.9, 161.2, 134.6, 134.5, 117.84, 117.78, 116.1, 115.7, 80.4, 73.5.



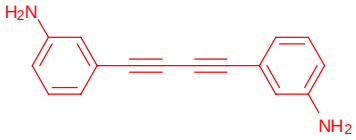
2e

1,4-dinaphthyl buta-1,3-diyne (2e) [5] **: 2e** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as yellow solid (yield 88%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 8.44 (d, *J* = 8.3 Hz, 2H), 7.90 (d, *J* = 8.3 Hz, 2H), 7.85 (d, *J* = 8.3 Hz, 2H), 7.84 (dd, *J* = 7.2, 1.3 Hz, 2H), 7.64 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 2H), 7.56 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 2H), 7.47 (dd, *J* = 8.3, 7.2 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 133.8, 133.1, 132.0, 129.7, 128.4, 127.2, 126.6, 126.1, 125.2, 119.5, 80.9, 78.6.



2f

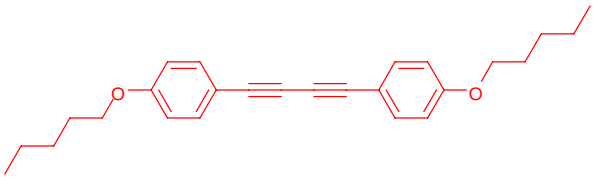
1,4-bis(o-chlorophenyl)buta-1,3-diyne (2f) [6] **: 2f** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield 85%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.57 (d, *J*=7.6Hz, 2H), 7.42 (d, *J*=7.6Hz, 2H), 7.31 (t, *J*=7.6Hz, 2H), 7.24 (t, *J*=7.6Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 137.38, 134.79, 130.70, 129.87, 126.98, 122.21, 79.82, 78.78.



2g

1,4-bis(m-aminophenyl)buta-1,3-diyne (2g) [7] **: 2g** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as yellow liquid (yield 50%).

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.23 (t, 2H), 7.11-7.05 (t, 2H), 6.90-6.87 (t, 2H), 6.65-6.62 (t, 2H), 3.59(s, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 146.1, 129.2, 122.6, 122.3, 118.2, 115.7, 83.8, 76.5.

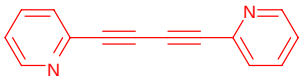


2h

1,4-bis(p-n-pentyloxyphenyl)buta-1,3-diyne (2h) [8] **: 2h** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield >98%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.42 (d, *J*=5.9Hz, 2H), 6.81 (d, *J*=5.9Hz, 2H), 3.93 (t, *J*=5.9Hz, 2H), 1.80-1.73 (m, 2H), 1.50-1.33 (m, 4H), 0.91 (t, *J*=5.9Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 159.9, 134.1, 114.7, 113.7, 81.4, 72.9, 68.2, 29.0, 28.3, 22.6, 14.2.

2i

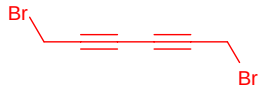
1,4-dithienyl buta-1,3-diyne (2i) [9] **: 2i** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as white solid (yield 90%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.57 (d, *J*=6.1Hz, 2H), 7.27 (s, 2H), 7.16 (d, *J*=5.4Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 131.2, 130.1, 128.4, 125.5, 120.8, 73.5.



2j

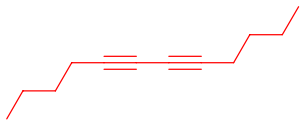
1,4-Bis(2-pyridine)buta-1,3-diyne (2j) [10] **: 2j** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as colorless oil (yield 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 8.63 (d, *J* = 4.4 Hz, 2H), 7.71 (t, *J* = 7.6 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.31 (t, *J* = 4.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 150.3, 141.8, 134.3, 128.4,

123.8, 80.8, 73.2.



2k

1,6-dibromo buta-2,4-diyne (2k) [11] **: 2k** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 5/1) as pale yellow oil (yield 60%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 3.99 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 75.27, 70.29, 13.96

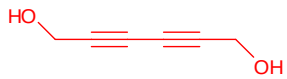


2l

5,7-dodecadiyne (2l) [1] **: 2l** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 40/1) as pale yellow oil (yield 75%). ^{1}H NMR (400 MHz, $CDCl_3$): 2.18 (t, J = 7.3 Hz, 4 H), 1.43 (m, 4 H), 1.35 (m, 4 H), 0.83 (t, J = 7.3 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 76.6, 65.1, 30.3, 21.8, 18.8, 13.5.

2m

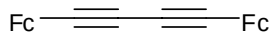
2,7-dimethyl octa-3,5-diyne-2,7-diol (2m) [4] **: 2m** was purified by flash chromatography (ethyl acetate) as white solid (yield 40%). ^{1}H NMR (270 MHz, $CDCl_3$): δ_H 1.99(s, 2H), 1.56(s, 12H). ^{13}C NMR (67.8 MHz, $CDCl_3$): δ_C 84.0, 66.3, 65.6, 31.0.



2n

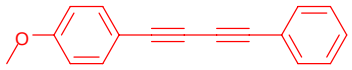
hexa-2,4-diyne-1,6-diol (2n) [4] **: 2n** was purified by flash chromatography (ethyl acetate)

as white solid (yield 45%). ^{1}H NMR (270 MHz, DMSO-D_6): δ_H 5.16 (s, 2H), 4.17 (s, 4H). ^{13}C NMR (67.8 MHz, DMSO-D_6): δ_C 79.5, 67.9, 49.3.



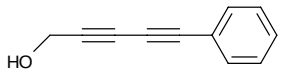
2o

1,4-differrocenyl buta-1,3-diyne (2o) [12] **: 2o** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 5/1) as red solid (yield 75%). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H 4.51 (s, 4H), 4.26 (s, 14H). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 82.6, 73.5, 71.7, 70.0, 68.7, 63.8.



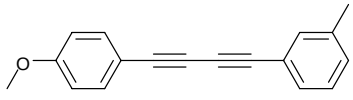
3a

1-methoxy-4-(phenylbuta-1,3-diynyl)benzene(3a) [13] **: 3a** was purified by flash chromatography (petroleum ether) as white solid (yield 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.46-7.52(m, 4H), 7.25-7.35(m, 3H), 6.85(d, *J*=8.0 Hz, 2H), 3.81(s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 160.36, 134.10, 132.40, 129.00, 128.39, 122.00, 114.15, 113.69, 81.81, 81.01, 74.17, 72.73, 55.31.



3b

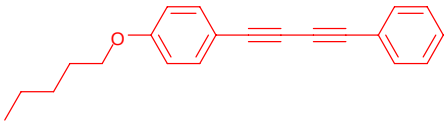
5-phenylpenta-2,4-diyn-1-ol(3b) [14] **: 3b** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 10/1) as yellow oil (yield 35%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.48(t, *J*=4.0 Hz, 2H), 7.30-7.37(m, 3H), 4.42(s, 2H), 2.09(s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 132.58, 129.34, 128.40, 121.35, 80.41, 78.58, 73.14, 70.45, 51.64.



3c

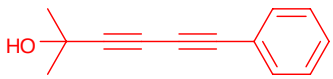
1-((4-methoxyphenyl)buta-1,3-diynyl)-3-methylbenzene(3c): 3c was purified by flash

chromatography (petroleum ether) as white solid (yield 55%). mp : 59-60°C. IR (cm-1): 2957.09, 2929.93, 2837.43, 2217.48, 2143.21, 1602.42, 1508.33, 1251.67, 1173.54, 1032.20, 830.79. ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.45-7.49(m, 2H), 7.32(d, *J*=8.4 Hz, 2H), 7.16-7.26(m, 2H), 6.84-6.87(m, 2H), 3.82(s, 3H), 2.33(s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 160.33, 138.13, 134.10, 132.91, 129.97, 129.54, 128.29, 121.79, 114.14, 113.78, 81.59, 81.26, 73.80, 72.80, 55.33, 21.19. MS: m/z: 246(M^+, 100%), 231(45), 203(26), 123(35), 101(27), 88(24), 71(19), 57(39), 43(39). Elemental analysis: found: C, 87.70; H, 5.81. Calc. for $C_{18}H_{14}O$: C, 87.78; H, 5.73%).



3d

1-(pentyloxy)-4-(phenylbuta-1,3-diynyl)benzene(3d): 3d was purified by flash chromatography (petroleum ether) as white solid (yield 72%). mp : 60-61°C. IR (cm-1): 2953.42, 2865.73, 2206.74, 2138.96, 1597.76, 1506.69, 1249.22, 1170.96, 1019.28, 830.05, 749.45. ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.50-7.53(m, 2H), 7.45(d, *J*=8.0 Hz, 2H), 7.32-7.36(m, 3H), 6.84(d, *J*=8.8 Hz, 2H), 3.96(t, *J*=6.4 Hz, 2H), 1.75-7.82(m, 2H), 1.34-1.47(m, 4H), 0.93(t, *J*=7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 160.02, 134.10, 132.43, 128.98, 128.40, 122.10, 114.09, 113.44, 81.91, 80.97, 74.24, 72.65, 68.15, 28.83, 28.15, 22.42, 13.98. MS: m/z: 288(M^+, 25%), 218(100), 203(26), 189(18), 43(42). Elemental analysis: found: C, 87.30; H, 6.85. Calc. for $C_{21}H_{20}O$: C, 87.46; H, 6.99%).



3e

2-methyl-6-phenylhexa-3,5-diyn-2-ol(3e) [15] **: 3e** was purified by flash chromatography (petroleum ether – ethyl acetate, v/v 10/1) as white solid (yield 32%). ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.47(t, *J*=4.0 Hz, 2H), 7.26-7.38(m, 3H), 2.09(s, 1H), 1.58(s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 132.50, 129.23, 128.40, 121.53, 86.65, 78.79, 73.11, 67.05, 65.75, 31.10.

3f

3-((4-methoxyphenyl)buta-1,3-diynyl)thiophene (3f): **3f** was purified by flash chromatography (petroleum ether) as pale yellow solid (yield 56%). mp : 87-88^{o}C. IR (cm-1): 3385.71, 3108.54, 2921.31, 2141.38, 1599.18, 1503.40, 1249.39, 1029.90, 830.88, 786.12. ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.56-7.57(m, 1H), 7.46(d, *J*=8.8 Hz, 2H), 7.26-7.28(m, 1H), 7.16-7.17(m, 1H), 6.85(d, *J*=9.2 Hz, 2H), 3.82(s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_C 160.38, 134.10, 130.92, 130.17, 125.51, 121.13, 114.17, 113.74, 81.61, 76.18, 73.80, 72.69, 55.34. MS: m/z: 238(M^+, 100%), 223(55), 195(29). Elemental analysis: found: C, 75.61; H, 4.30. Calc. for $C_{15}H_{10}OS$: C, 75.60; H, 4.23%).

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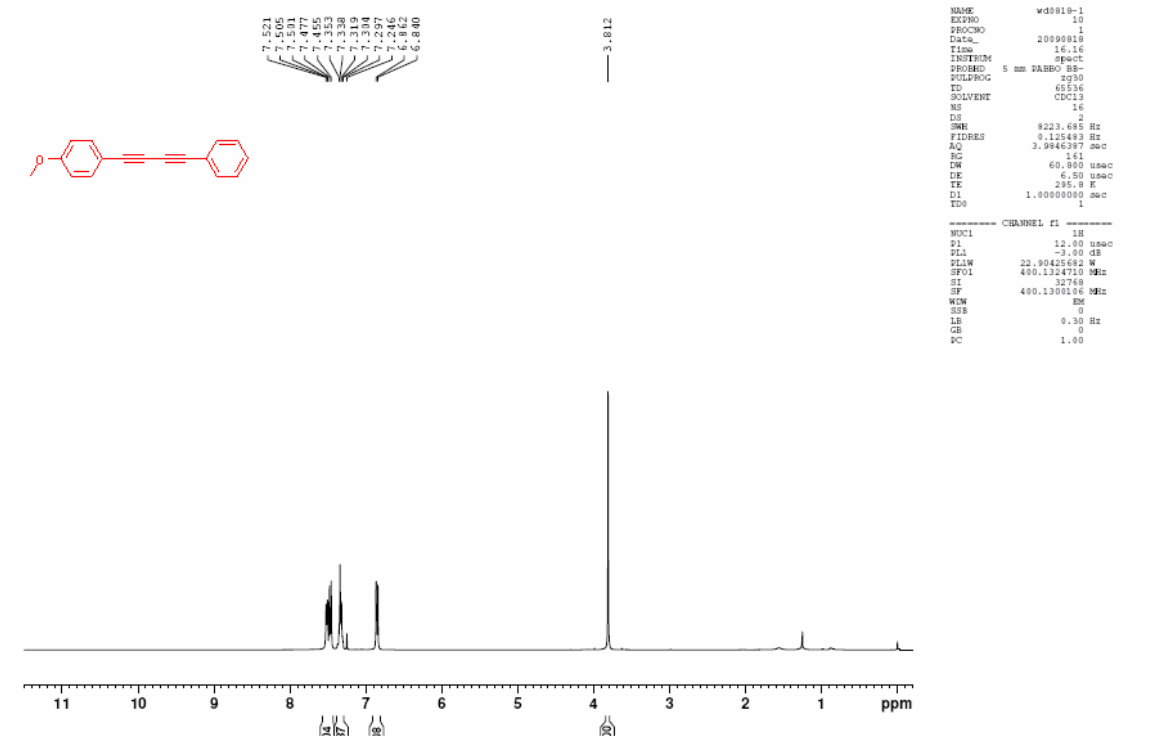
3a:

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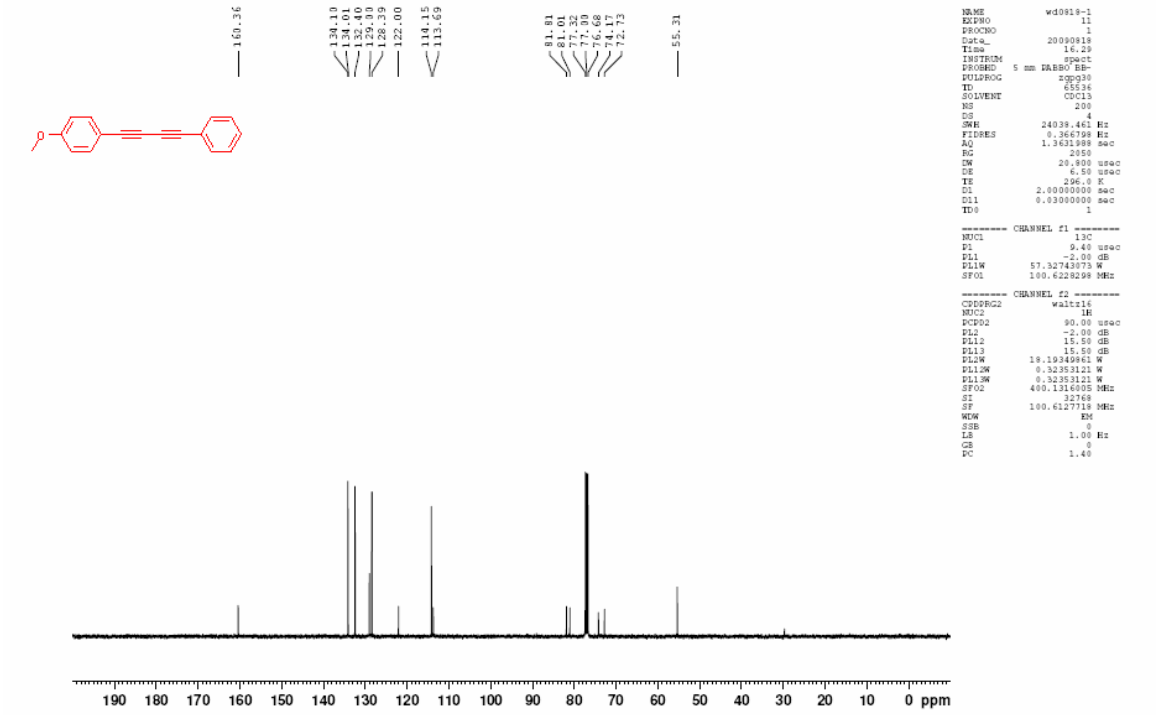
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     161
DW                  60.800 usec
DE                   6.50 usec
TE                  295.8 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   12.00 usec
PL1                  -3.00 dB
PL1W            22.90425682 W
SFO1           400.1324710 MHz
SI                   32768
SF             400.1300106 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

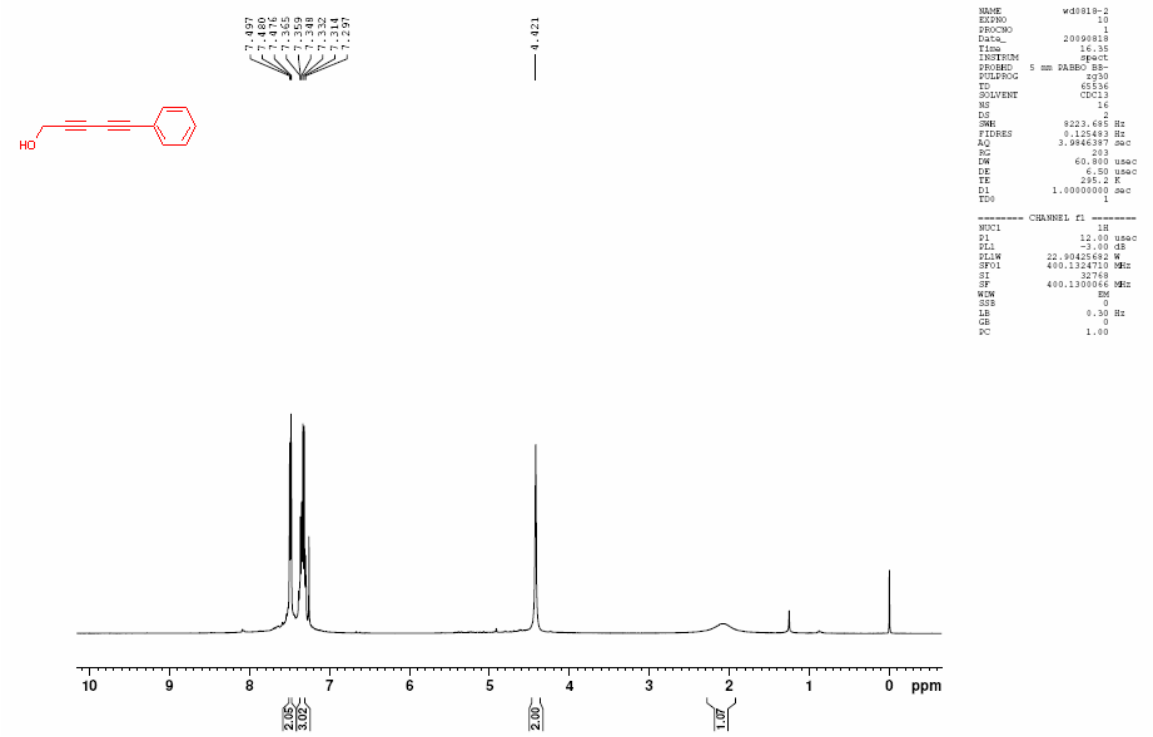



```
NAME              wd0818-1
EXPNO                   11
PROCNO                   1
Date_             20090818
Time                 16.29
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT              CDCl3
NS                     200
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                    2050
DW                  20.800 usec
DE                   6.50 usec
TE                  296.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.40 usec
PL1                  -2.00 dB
PL1W            57.32743873 W
SFO1           100.6228298 MHz

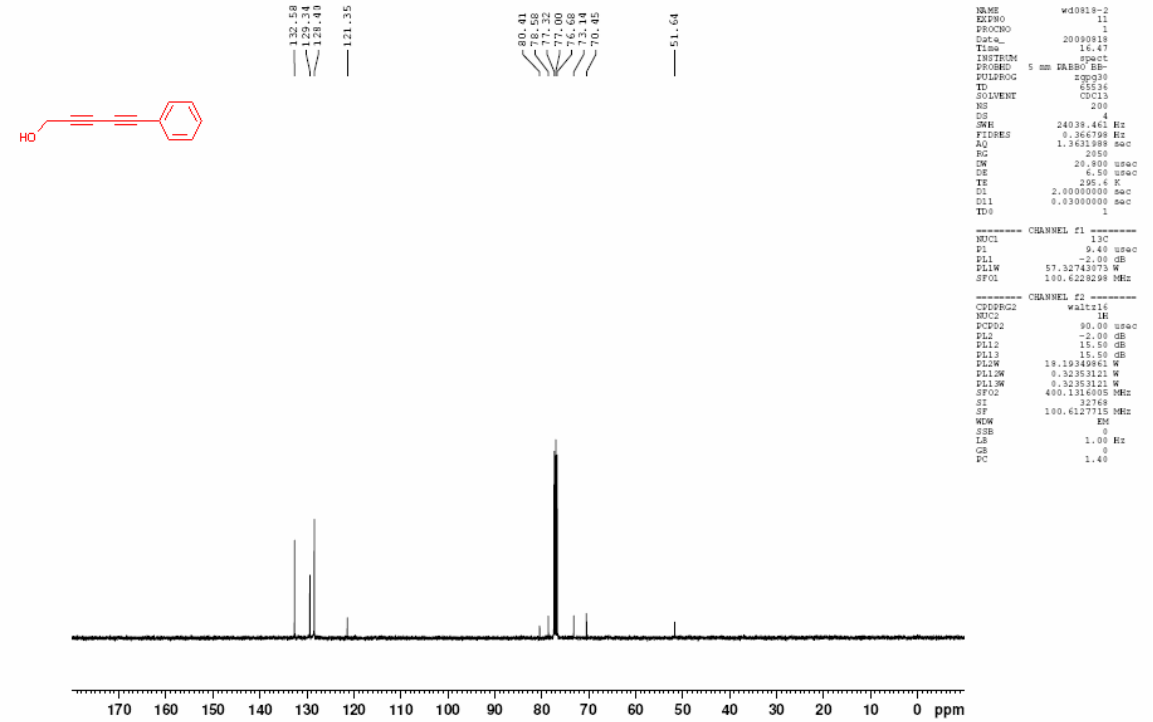
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -2.00 dB
PL12                 15.50 dB
PL13                 15.50 dB
PL2W            18.19349861 W
PL12W            0.32353121 W
PL13W            0.32353121 W
SFO2           400.1316005 MHz
SI                   32768
SF             100.6127718 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

3b:




```
NAME                wd0818-2
EXPNO                     10
PROCNO                     1
Date_               20090818
Time                   16.35
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125493 Hz
AQ                 3.9846387 sec
RG                      203
DW                   60.800 usec
DE                    6.50 usec
TE                   295.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     12.00 usec
PL1                    -3.00 dB
PL1W             22.90425682 W
SFO1             400.1324710 MHz
SI                      32768
SF               400.1300066 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

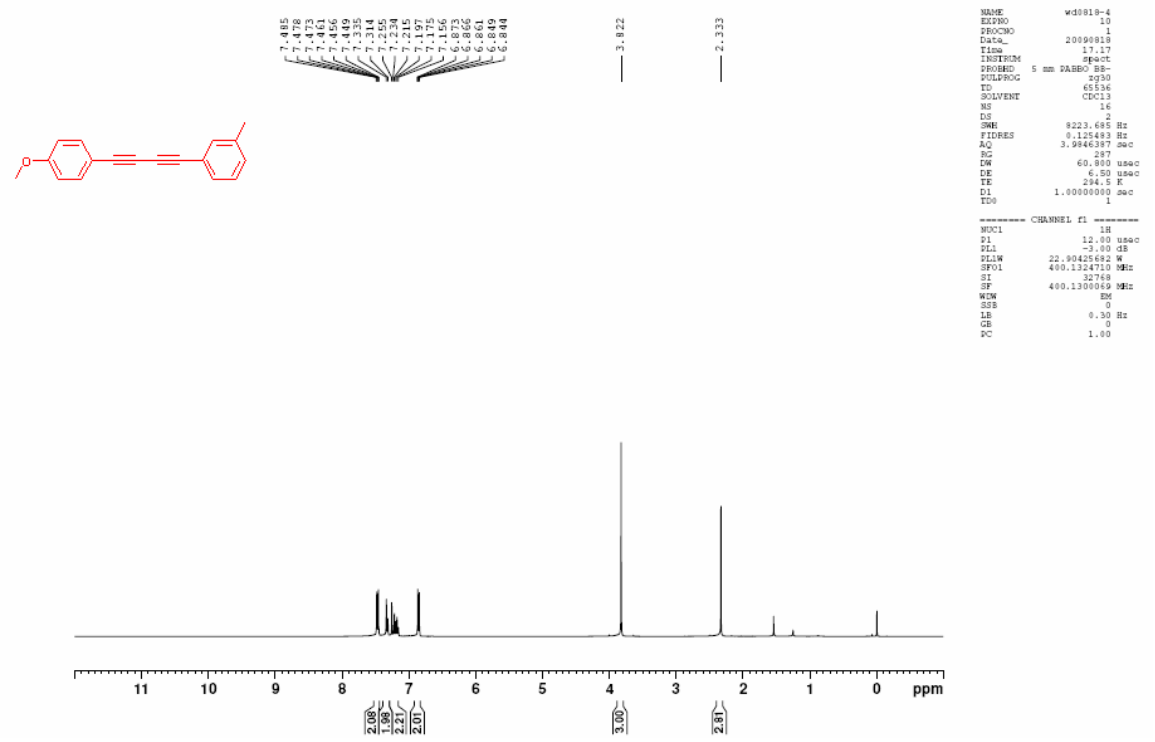


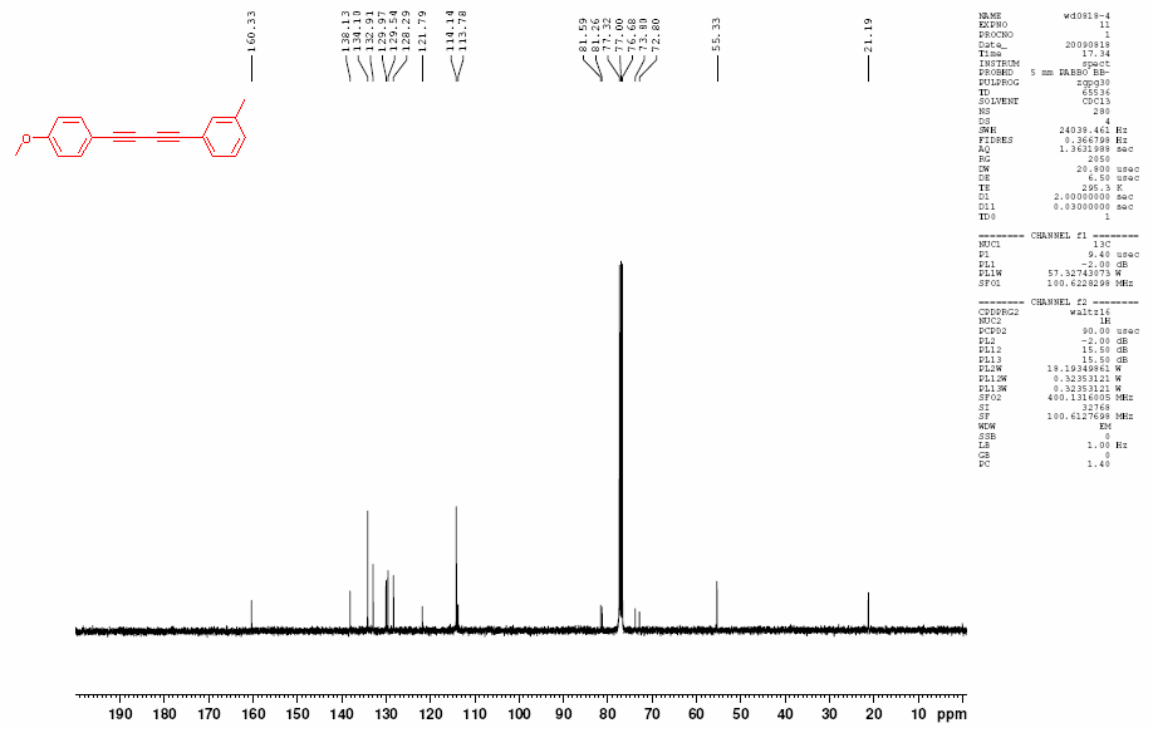

```
NAME                wd0818-2
EXPNO                     11
PROCNO                     1
Date_               20090818
Time                   16.47
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      200
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                     2050
DW                   20.800 usec
DE                    6.50 usec
TE                   295.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      13C
P1                     9.40 usec
PL1                   -2.00 dB
PL1W             57.32743073 W
SFO1             100.6228298 MHz

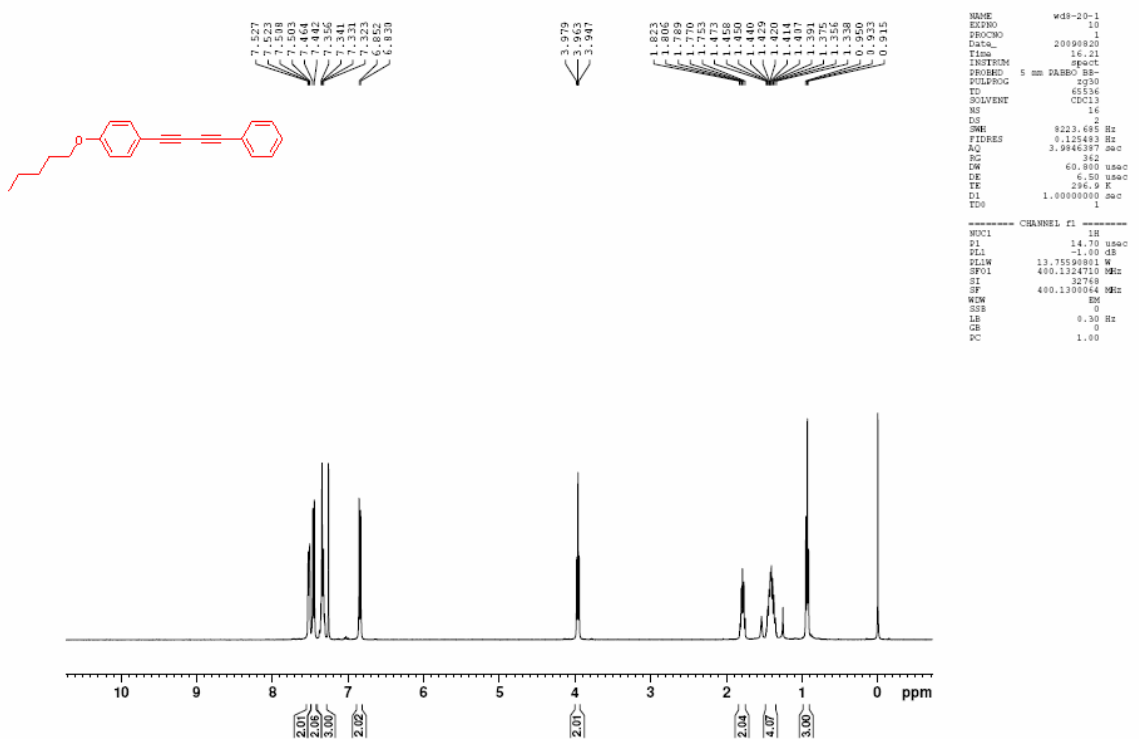
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 90.00 usec
PL2                   -2.00 dB
PL12                  15.50 dB
PL13                  15.50 dB
PL2W             18.19349861 W
PL12W             0.32353121 W
PL13W             0.32353121 W
SFO2             400.1316005 MHz
SI                      32768
SF               100.6127715 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

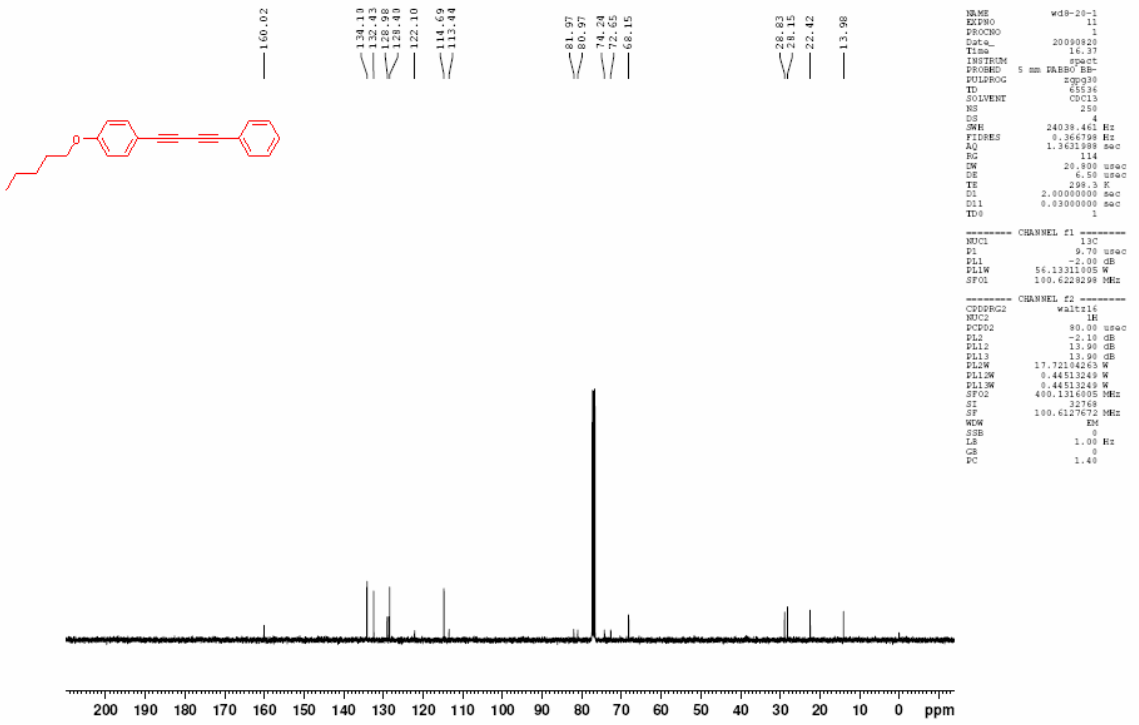
3c:


7.485
7.478
7.473
7.461
7.456
7.449
7.335
7.314
7.255
7.234
7.215
7.197
7.175
7.156
6.873
6.866
6.861
6.849
6.844
3.822
2.333
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.08
1.98
2.21
2.01
3.00
2.81

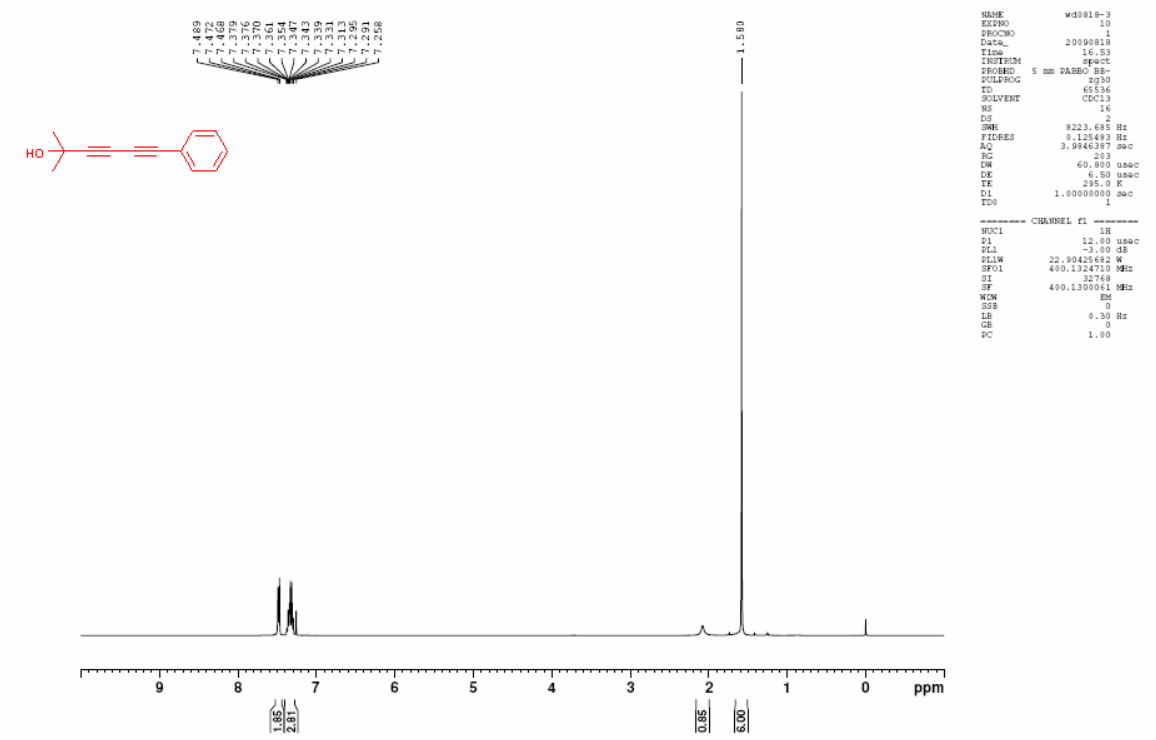

160.33
138.13
134.10
132.91
129.97
129.54
128.29
121.79
114.14
113.78
81.59
81.26
77.32
77.00
76.68
73.80
72.80
55.33
21.19
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3d:


7.527
7.523
7.518
7.503
7.464
7.442
7.356
7.341
7.331
7.323
6.852
6.839
3.979
3.963
3.947
1.823
1.806
1.789
1.770
1.753
1.473
1.458
1.450
1.440
1.429
1.420
1.414
1.407
1.391
1.375
1.356
1.338
0.950
0.933
0.915
NAME wd8-20-1
EXPNO 10
PROCNO 1
Date_ 20090820
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
2.01 2.06 3.00 2.02 2.01 2.04 4.07 3.00

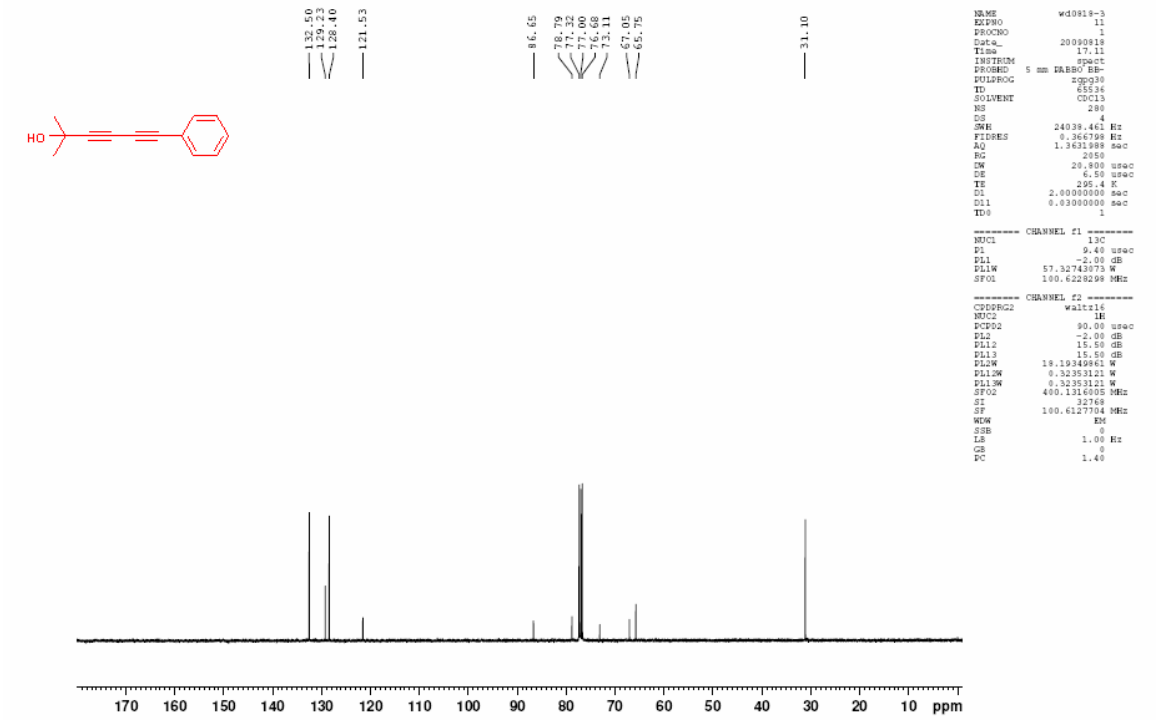

160.02
134.10
132.43
128.98
128.40
122.10
114.69
113.44
81.97
80.97
74.24
72.65
68.15
28.83
28.15
22.42
13.98
NAME wd8-20-1
EXPNO 11
PROCNO 1
Date_ 20090820
Time 16.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 250
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127672 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3e:




```
NAME            wd0818-3
EXPNO                 10
PROCNO                 1
Date_           20090818
Time                16.53
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT              CDCl3
NS                    16
DS                     2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846397 sec
RG                   203
DW                60.800 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -3.00 dB
PL1W         22.90425682 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300061 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

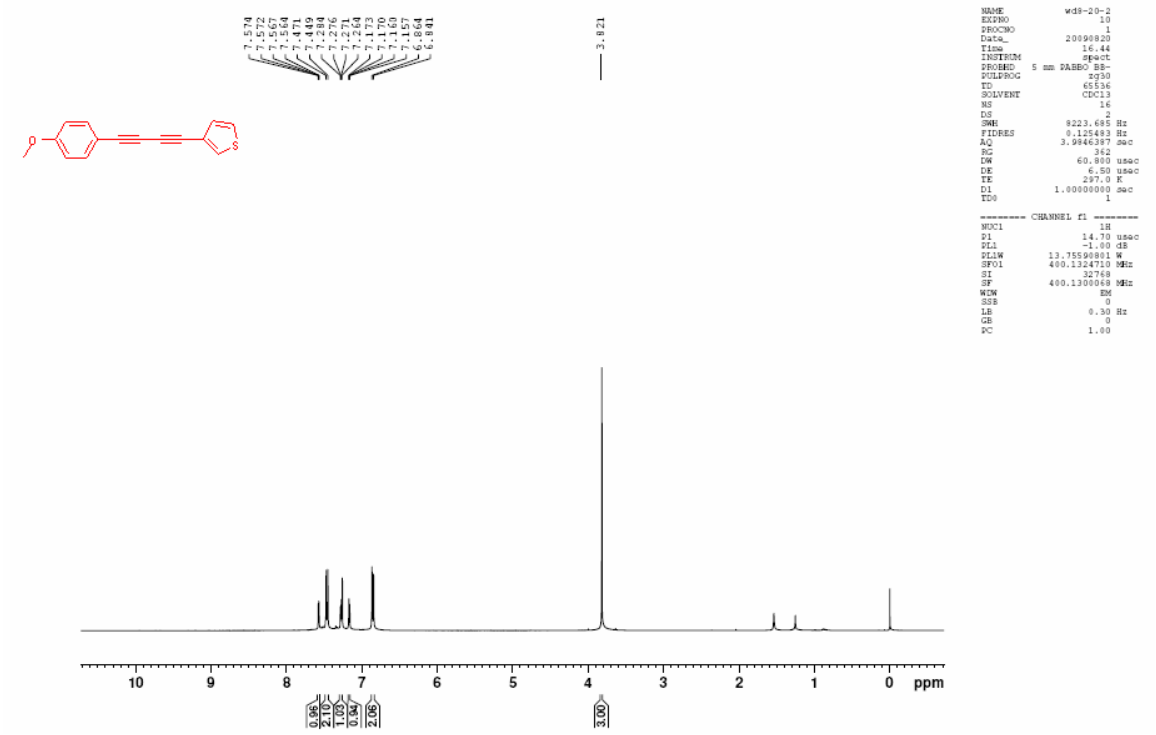



```
NAME            wd0819-3
EXPNO                 11
PROCNO                 1
Date_           20090819
Time                17.11
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT              CDCl3
NS                   288
DS                     4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                295.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.40 usec
PL1                -2.00 dB
PL1W         57.32743073 W
SFO1         100.6228298 MHz

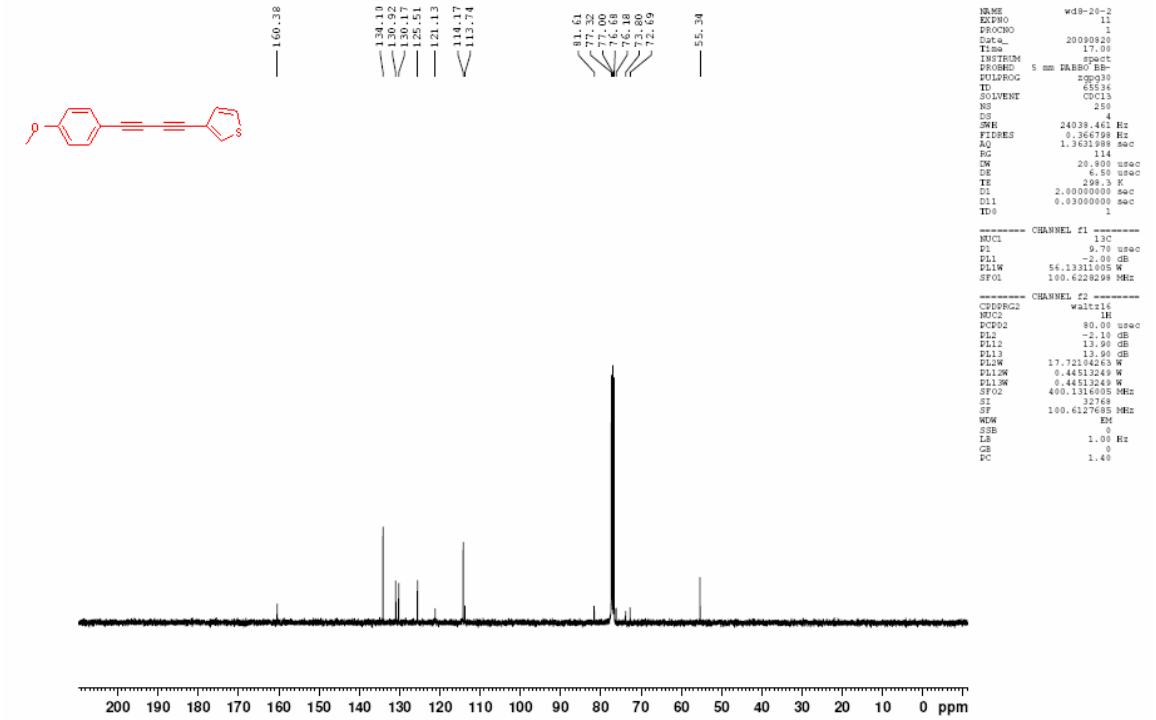
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.00 dB
PL12               15.50 dB
PL13               15.50 dB
PL2W         18.19349861 W
PL12W         0.32353121 W
PL13W         0.32353121 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127704 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

3f:




```
NAME                wd9-20-2
EXPNO                     10
PROCNO                     1
Date_               20090820
Time                   16.44
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125493 Hz
AQ                  3.9846287 sec
RG                       362
DW                    60.800 usec
DE                     6.50 usec
TE                    297.0 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.70 usec
PL1                    -1.00 dB
PL1W                13.75590801 W
SFO1               400.1324710 MHz
SI                        32768
SF                 400.1300068 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```




```
NAME                wd9-20-2
EXPNO                     11
PROCNO                     1
Date_               20090820
Time                   17.00
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      250
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631989 sec
RG                       114
DW                    20.800 usec
DE                     6.50 usec
TE                    298.3 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.70 usec
PL1                    -2.00 dB
PL1W               56.13311005 W
SFO1               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                    -2.10 dB
PL12                   13.90 dB
PL13                   13.90 dB
PL2W               17.72104263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127685 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```