Supporting Information

Ligand-free reusable nano copper oxide-catalyzed synthesis of 3-amino-1,4-diynes

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1. General Information:

^{1}H NMR spectra were determined on 400 MHz spectrometer as solutions in $CDCl_3$. Chemical shifts are expressed in parts per million (δ) and are referenced to tetramethylsilane (TMS) as internal standard and the signals were reported as s (singlet), d (doublet), t (triplet), dd (double doublet), m (multiplet) and coupling constants *J* were given in Hz. ^{13}C NMR spectra were recorded at 100 MHz in $CDCl_3$. TLC was done on TLC Silica gel 60 F_{254} coated on aluminium sheets (Merck). Silica gel (60-120 mesh) was used for column chromatography. Petroleum ether refers to the fraction boiling in the range of 60-80 °C unless otherwise mentioned. All solvents were dried and distilled before use. Solvents, reagents and chemicals were purchased from Aldrich and Merck. All reactions involving moisture sensitive reactants were executed using oven dried glassware.

2. General procedure for the synthesis of *N,N*-dimethyl-1,5-diphenylpenta-1,4-diyn-3-amine (3a):

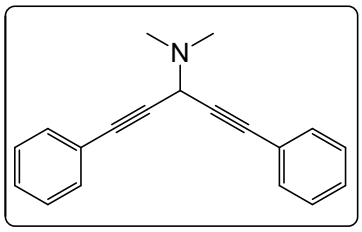
A mixture of N,N-dimethyl formamide dimethyl acetal (70 μL, 0.5 mmol), and phenylacetylene (160 μL, 1.5 mmol) was stirred in presence of CuO nano (Sigma-Aldrich, nanopowder, <50 nm particle size, 10 mol%) in 1,2-DCE (3 mL) under refluxed conditions for 10 h. After completion of the reaction (TLC), DCM (10 mL) was added to the reaction mixture. Then the insoluble CuO nanoparticles were filtered by Teflon membrane (PTFE, 0.2 mm pore size). The CuO nanoparticles was thoroughly washed with the DCM, dried and reused for the next cycle. Water was added to the filtrate and extracted with DCM (10 mL) followed by washing with brine (5 mL) and dried over Na_2SO_4. The crude residue was obtained after evaporation of the solvent in vacuum and purified by column chromatography on silica gel (60−120 mesh) using petroleum ether–ethyl acetate (9:1) as the eluent to afford pure **3a** as Yellow liquid.

3. General procedure for the synthesis of *N,N*-dimethyl-1,5-diphenylpenta-1,4-diyn-3-amine (3n):

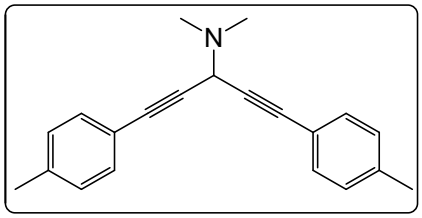
A mixture of N,N-dimethyl formamide dimethyl acetal (70 μL, 0.5 mmol), phenylacetylene (80 μL, 0.75 mmol), and 4-ethynyltolune (95 μL, 0.75 mmol) was stirred in presence of CuO nano (Sigma-Aldrich, nanopowder, <50 nm particle size, 10 mol%) in 1,2-DCE (3 mL) under refluxed conditions for 10 h. After completion of the reaction (TLC), DCM (10 mL) was added to the reaction mixture. Then the insoluble CuO nanoparticles were filtered by Teflon

membrane (PTFE, 0.2 mm pore size). The CuO nanoparticles was thoroughly washed with the DCM, dried and reused for the next cycle. Water was added to the filtrate and extracted with DCM (10 mL) followed by washing with brine (5 mL) and dried over Na_2SO_4. The crude residue was obtained after evaporation of the solvent in vacuum and purified by column chromatography on silica gel (60-120 mesh) using petroleum ether–ethyl acetate (6:1) as the eluent to afford pure **3n** as Yellow liquid.

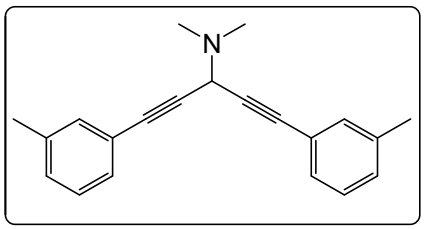
4. Characterization Data of the Synthesized Products:



***N,N*-Dimethyl-1,5-diphenylpenta-1,4-diyn-3-amine** (**3a**): Yellow liquid (87%, 112 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.48 (m, 4H), 7.32-7.30 (m, 6H), 4.79 (s, 1H), 2.49 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.0, 128.5, 128.3, 122.6, 84.7, 83.5, 50.2, 41.5; Anal calcd for $C_{19}H_{17}N$: C, 87.99; H, 6.61; N, 5.40%; Found: C, 87.82; H, 6.70; N, 5.28%.

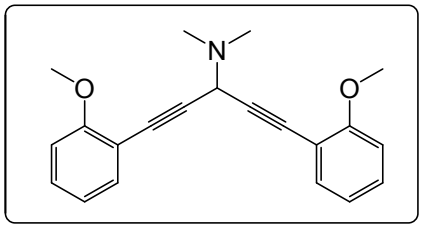


***N,N*-Dimethyl-1,5-di-*p*-tolylpenta-1,4-diyn-3-amine** (**3b**): Yellow liquid (92%, 132 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, J = 8.4 Hz, 4H), 7.12 (d, J = 8.0 Hz, 4H), 4.77 (s, 1H), 2.48 (s, 6H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.5, 131.8, 129.0, 119.6, 84.6, 83.0, 50.2, 41.4, 21.5; Anal calcd for $C_{21}H_{21}N$: C, 87.76; H, 7.36; N, 4.87%; Found: C, 87.66; H, 7.49; N, 4.72%.

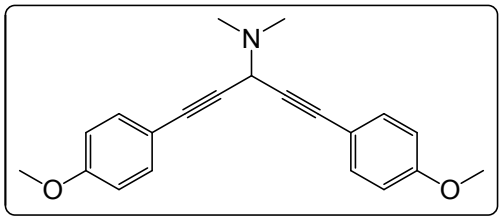


***N,N*-Dimethyl-1,5-di-*m*-tolylpenta-1,4-diyn-3-amine** (**3c**): Yellow liquid (90%, 129 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.30 (m, 4H), 7.21 (t, J = 7.6 Hz, 2H), 7.13 (d, J = 7.6 Hz, 2H), 4.78 (s, 1H), 2.49 (s, 6H), 2.33 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9,

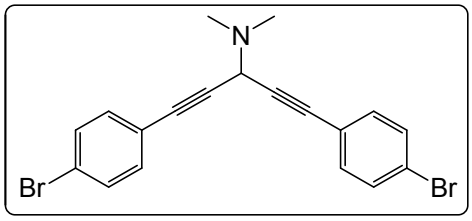
132.5, 129.3, 129.0, 128.2, 122.4, 84.7, 83.3, 50.1, 41.4, 21.2; Anal calcd for $C_{21}H_{21}N$: C, 87.76; H, 7.36; N, 4.87 %; Found: C, 87.64; H, 7.46; N, 4.78%.



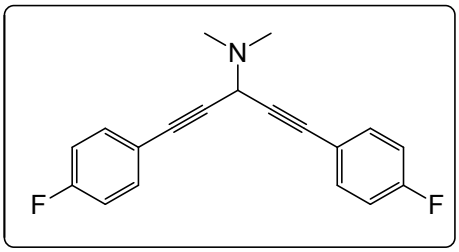
1,5-Bis(2-methoxyphenyl)-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3d**): Yellow liquid (88%, 140 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, *J* = 9.2 Hz, 2H), 7.22-7.17 (m, 2H), 6.82-6.77 (m, 4H), 4.79 (s, 1H), 3.78 (s, 6H), 2.43 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 133.8, 129.8, 120.4, 112.0, 110.8, 87.7, 80.8, 55.8, 50.5, 41.2. Anal calcd for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.63; N, 4.39%; Found: C, 78.85; H, 6.72; N, 4.27%.



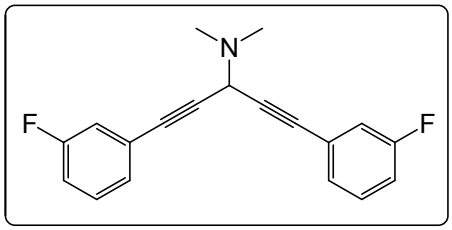
1,5-Bis(4-methoxyphenyl)-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3e**): Yellow liquid (90%, 143 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.4 Hz, 4H), 6.83 (d, *J* = 8.8 Hz, 4H), 4.74 (s, 1H), 3.79 (s, 6H), 2.46 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.7, 133.4, 114.8, 113.9, 84.3, 82.4, 55.3, 50.2, 41.4; Anal calcd for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.63; N, 4.39%; Found: C, 78.82; H, 6.74; N, 4.30%.



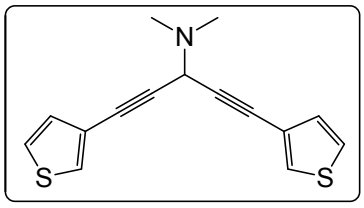
1,5-Bis(4-bromophenyl)-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3f**): Yellow solid (82%, 169 mg), m.p. 80-81 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, *J* = 8.4 Hz, 4H), 7.34 (d, *J* = 8.4 Hz, 4H), 4.74 (s, 1H), 2.46 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.4, 131.6, 122.9, 121.5, 84.5, 83.8, 50.2, 41.5; Anal calcd for $C_{19}H_{15}Br_2N$: C, 54.71; H, 3.62; N, 3.36%; Found: C, 54.54; H, 3.73; N, 3.26%.



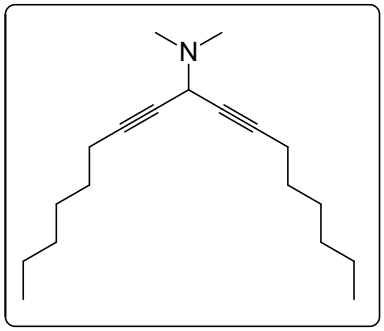
1,5-Bis(4-fluorophenyl)-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3g**): Yellow liquid (91%, 134 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.44 (m, 4H), 7.02-6.97 (m, 4H), 4.76 (s, 1H), 2.47 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.7 ($J_{C\text{-}F}$ = 248 Hz), 133.9 ($J_{C\text{-}F}$ = 9 Hz), 118.6 ($J_{C\text{-}F}$ = 4 Hz), 115.6 ($J_{C\text{-}F}$ = 22 Hz), 83.7, 83.0, 49.9, 41.3; Anal calcd for $C_{19}H_{15}F_2N$: C, 77.27; H, 5.12; N, 4.74%; Found: C, 77.13; H, 5.26; N, 4.63%.



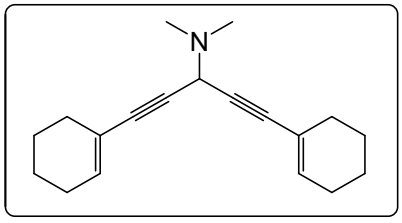
1,5-Bis(3-fluorophenyl)-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3h**): Yellow liquid (85%, 125 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22-7.18 (m, 4H), 7.13-7.09 (m, 2H), 6.98-6.95 (m, 2H), 4.69 (s, 1H), 2.39 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.4 ($J_{C\text{-}F}$ = 245 Hz), 130.0 ($J_{C\text{-}F}$ = 9 Hz), 127.9 ($J_{C\text{-}F}$ = 3 Hz), 124.4 ($J_{C\text{-}F}$ = 10 Hz), 118.8 ($J_{C\text{-}F}$ = 22 Hz), 116.0 ($J_{C\text{-}F}$ = 21 Hz), 84.4, 83.6 ($J_{C\text{-}F}$ = 2 Hz), 50.1, 41.5; Anal calcd for $C_{19}H_{15}F_2N$: C, 77.27; H, 5.12; N, 4.74%; Found: C, 77.15; H, 5.22; N, 4.61%.



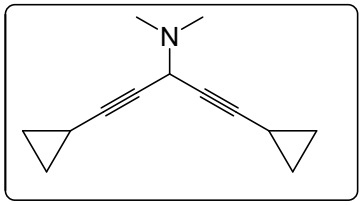
***N,N*-Dimethyl-1,5-di(thiophen-3-yl)penta-1,4-diyn-3-amine** (**3i**): Yellow liquid (90%, 122 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.40 (m, 2H), 7.17 (d, J = 8.0 Hz, 2H), 7.07-7.05 (m, 2H), 4.65 (s, 1H), 2.37 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 130.1, 129.3, 125.3, 121.6, 83.1, 79.7, 50.1, 41.5. Anal calcd for $C_{15}H_{13}NS_2$: C, 66.38; H, 4.83; N, 5.16%; Found: C, 66.22; H, 4.95; N, 5.08%.



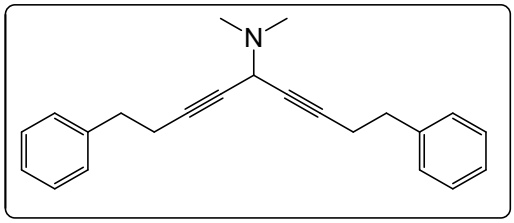
***N,N*-Dimethylheptadeca-7,10-diyn-9-amine** (**3j**): Yellow liquid (85%, 116 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.30 (s, 6H), 2.23-2.18 (m, 4H), 1.54-1.46 (m, 4H), 1.39-1.35 (m, 4H), 1.30-1.25 (m, 8H), 0.89-0.85 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 84.7, 74.9, 49.1, 41.1, 31.4, 28.8, 28.6, 22.6, 18.8, 14.1; Anal calcd for $C_{19}H_{33}N$: C, 82.84; H, 12.07; N, 5.08%; Found: C, 82.65; H, 12.17; N, 4.95%.



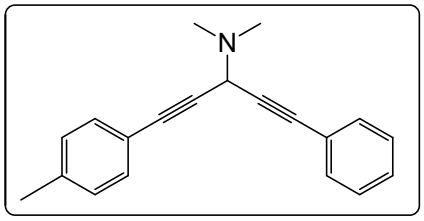
1,5-Di(cyclohex-1-en-1-yl)-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3k**): Brown liquid (90%, 120 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.11-6.09 (m, 2H), 4.52 (s, 1H), 2.33 (s, 6H), 2.12-2.05 (m, 8H), 1.61-1.53 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.2, 120.2, 86.1, 81.0, 49.8, 41.1, 29.3, 25.6, 22.3, 21.5; Anal calcd for $C_{19}H_{25}N$: C, 85.34; H, 9.42; N, 5.24%; Found: C, 85.23; H, 9.47; N, 5.15%.



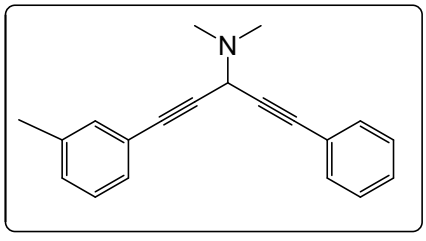
1,5-Dicyclopropyl-*N,N*-dimethylpenta-1,4-diyn-3-amine (**3l**): Yellow liquid (65%, 60 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.21 (s, 1H), 2.27 (s, 6H), 1.26-1.23 (m, 2H), 0.76-0.74 (m, 4H), 0.73-0.69 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 87.7, 70.0, 49.1, 41.1, 8.3, -0.4; Anal calcd for $C_{13}H_{17}N$: C, 83.37; H, 9.15; N, 7.48%; Found: C, 83.21; H, 9.29; N, 7.34%.



N,N-Dimethyl-1,9-diphenylnona-3,6-diyn-5-amine (**3m**): Brown liquid (90%, 141 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.27 (m, 4H), 7.23-7.20 (m, 6H), 4.26 (s, 1H), 2.85 (t, *J* = 7.6 Hz, 4H), 2.53 (t, *J* = 7.6 Hz, 4H), 2.23 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.7, 128.6, 128.4, 126.3, 83.9, 75.5, 49.1, 41.1, 35.2, 21.0; Anal calcd for $C_{23}H_{25}N$: C, 87.57; H, 7.99; N, 4.44%; Found: C, 87.35; H, 8.11; N, 4.32%.

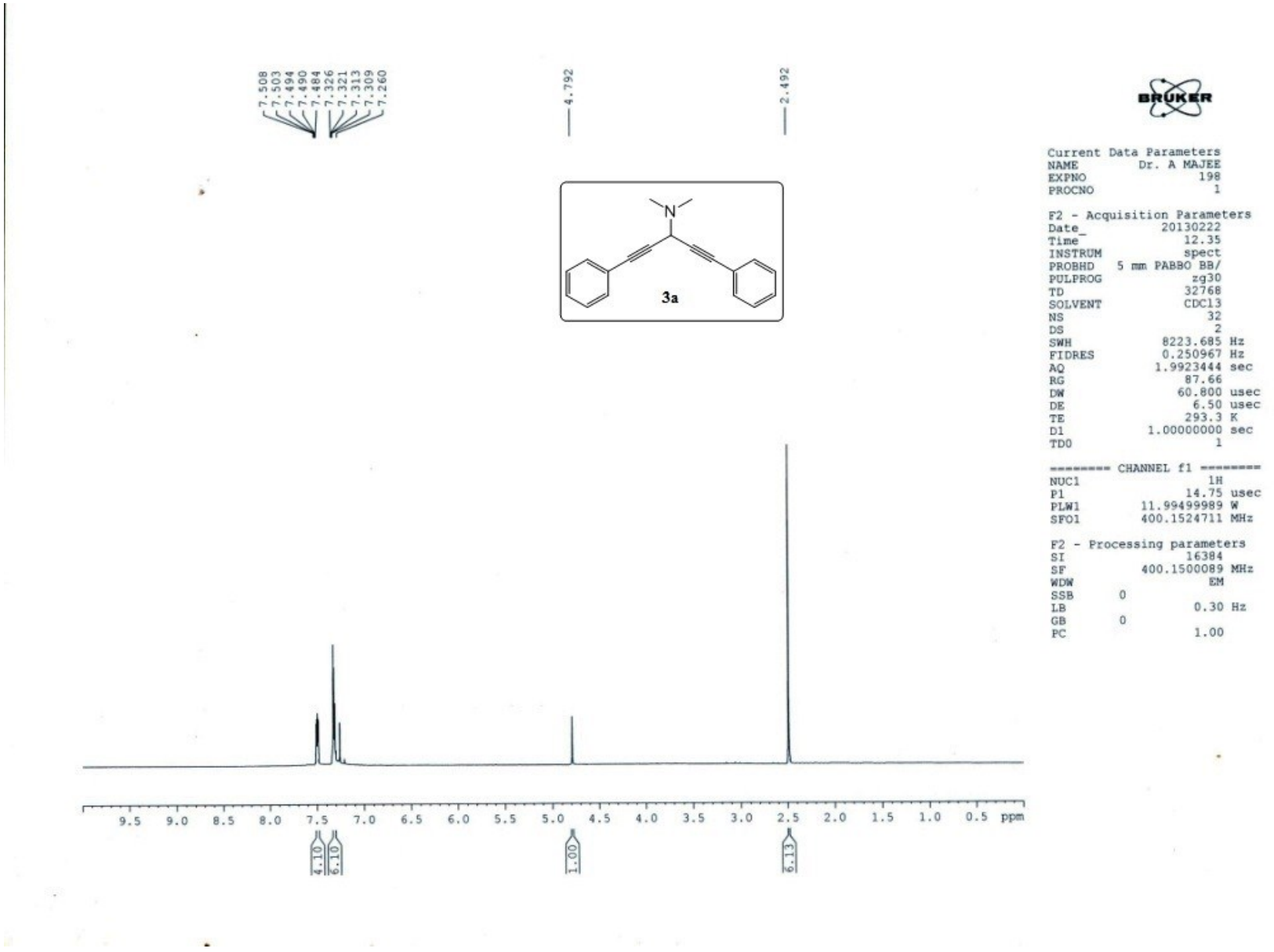


***N,N*-Dimethyl-1-phenyl-5-(*p*-tolyl)penta-1,4-diyn-3-amine** (**3n**): Yellow liquid (85%, 116 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.48 (m, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.33-7.30 (m, 3H), 7.11 (d, *J* = 6.8 Hz, 2H), 4.76 (s, 1H), 2.47 (s, 6H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6, 132.0, 131.9, 129.1, 128.5, 128.3, 122.8, 119.6, 84.6, 84.5, 83.7, 82.9, 50.2, 41.5, 21.6; Anal calcd for $C_{20}H_{19}N$: C, 87.87; H, 7.01; N, 5.12%; Found: C, 87.69; H, 7.12; N, 5.01%.



***N,N*-Dimethyl-1-phenyl-5-(*m*-tolyl)penta-1,4-diyn-3-amine** (**3o**): Yellow liquid (88%, 120 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.48 (m, 2H), 7.32-7.29 (m, 5H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.6 Hz, 1H), 4.77 (s, 1H), 2.48 (s, 6H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.0, 132.6, 132.0, 129.4, 129.0, 128.5, 128.3, 128.2, 122.7, 122.5, 84.8, 84.6, 83.7, 83.2, 50.2, 41.5, 21.3; Anal calcd for $C_{20}H_{19}N$: C, 87.87; H, 7.01; N, 5.12%; Found: C, 87.68; H, 7.15; N, 4.99%.

5. ^{1}H and ^{13}C NMR Spectra for the Synthesized Products



BRUKER

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PROCNO 1

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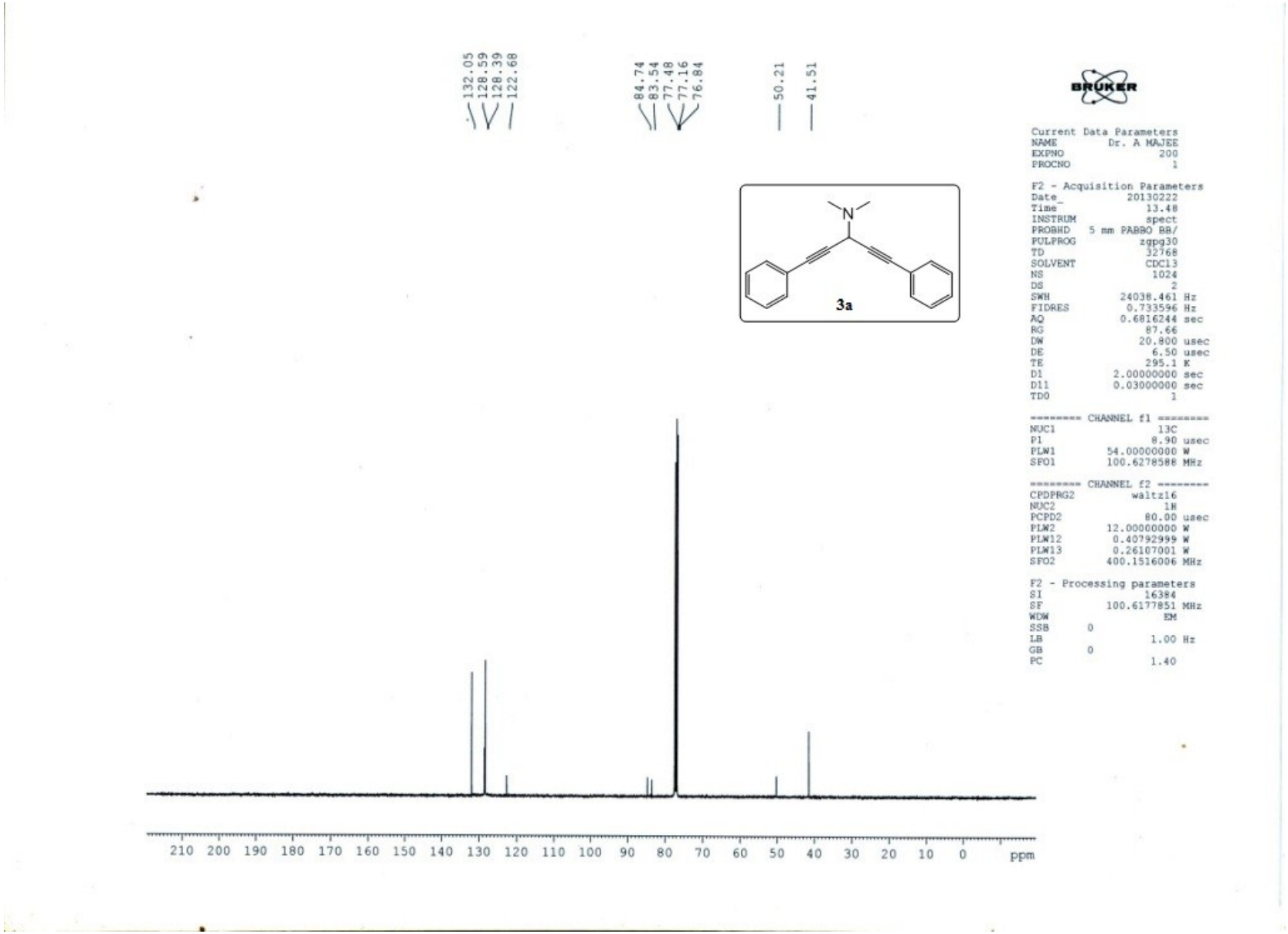

BRUKER

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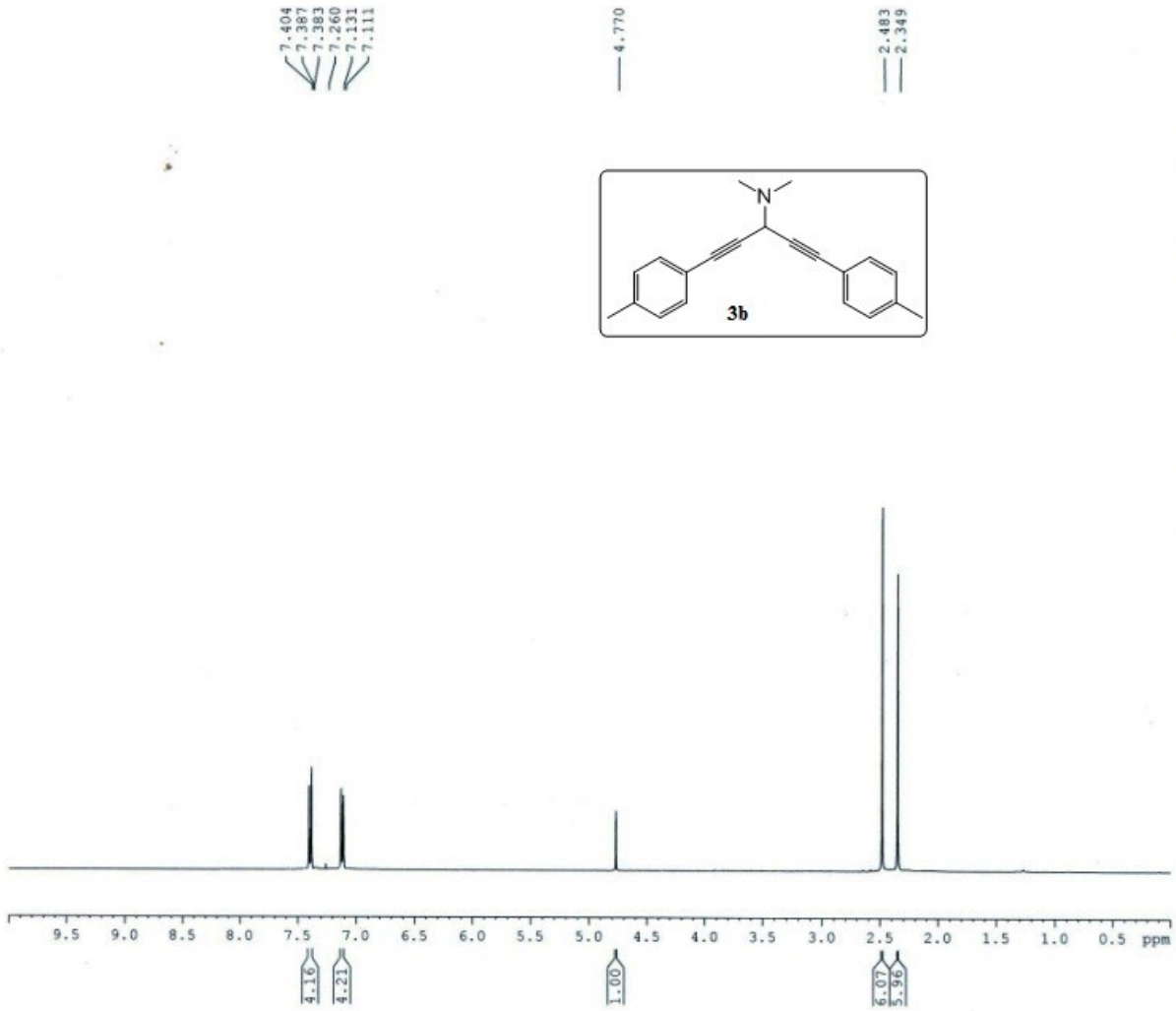


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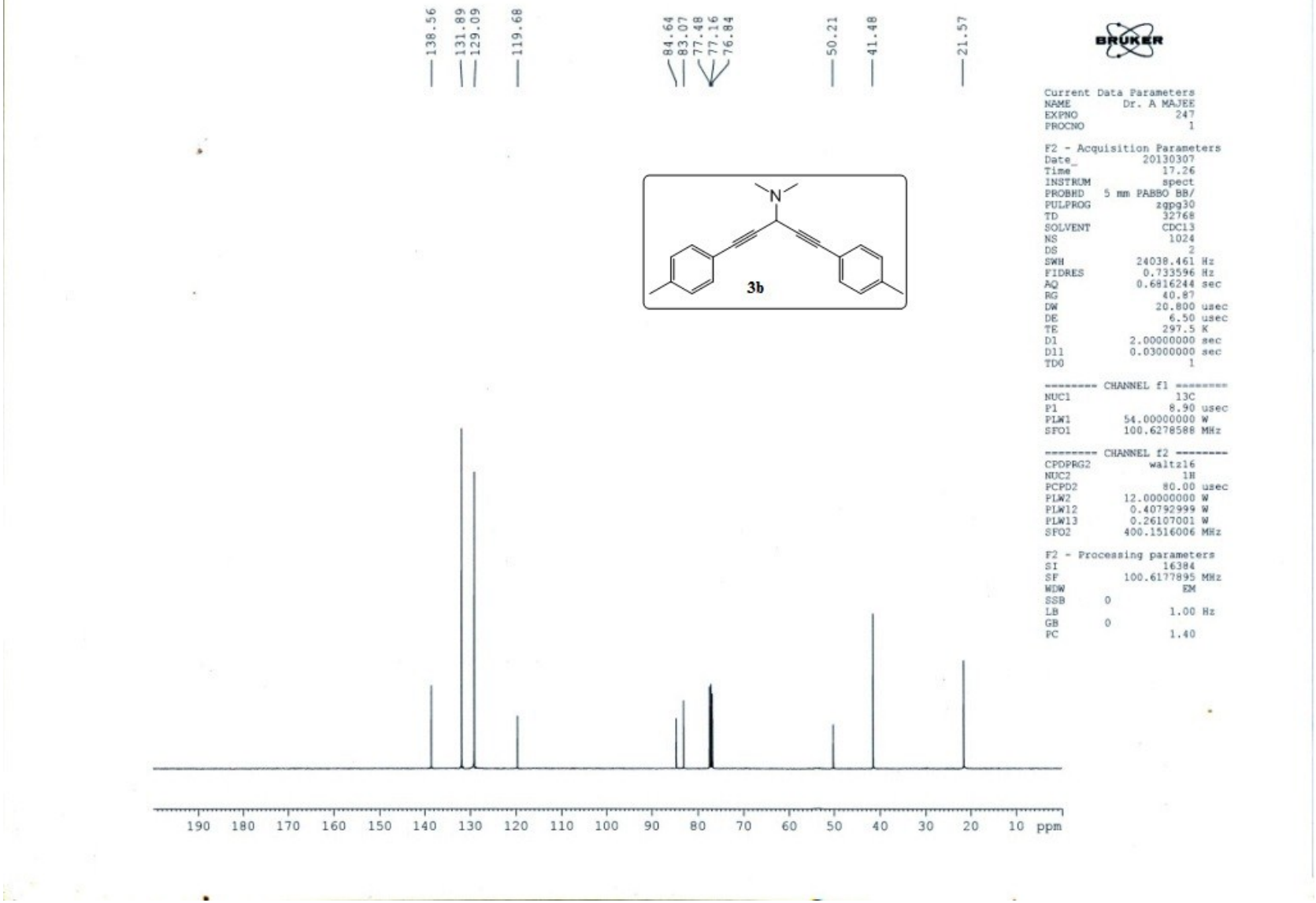

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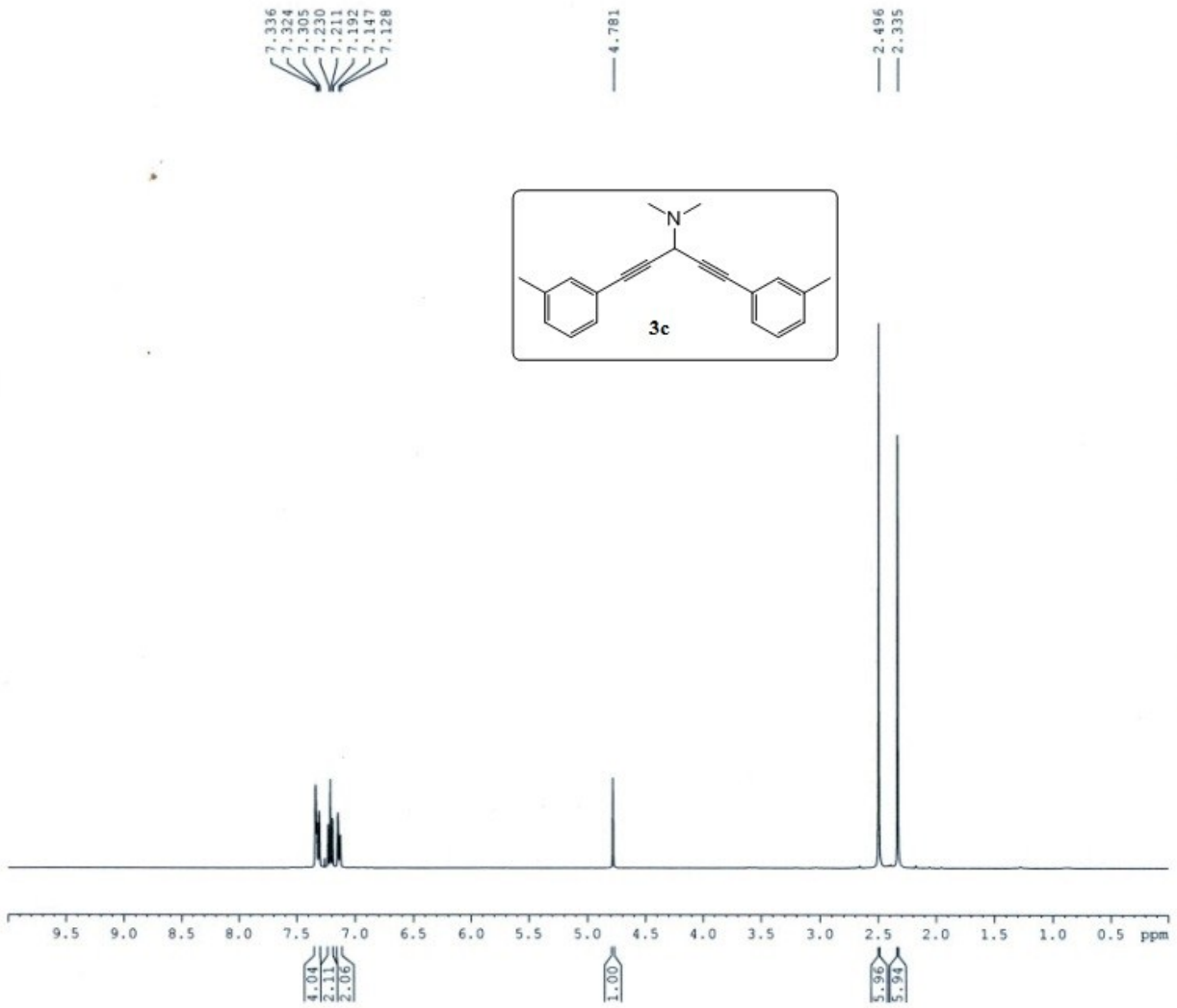
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 247
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130307
Time 17.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1024
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 40.87
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177895 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



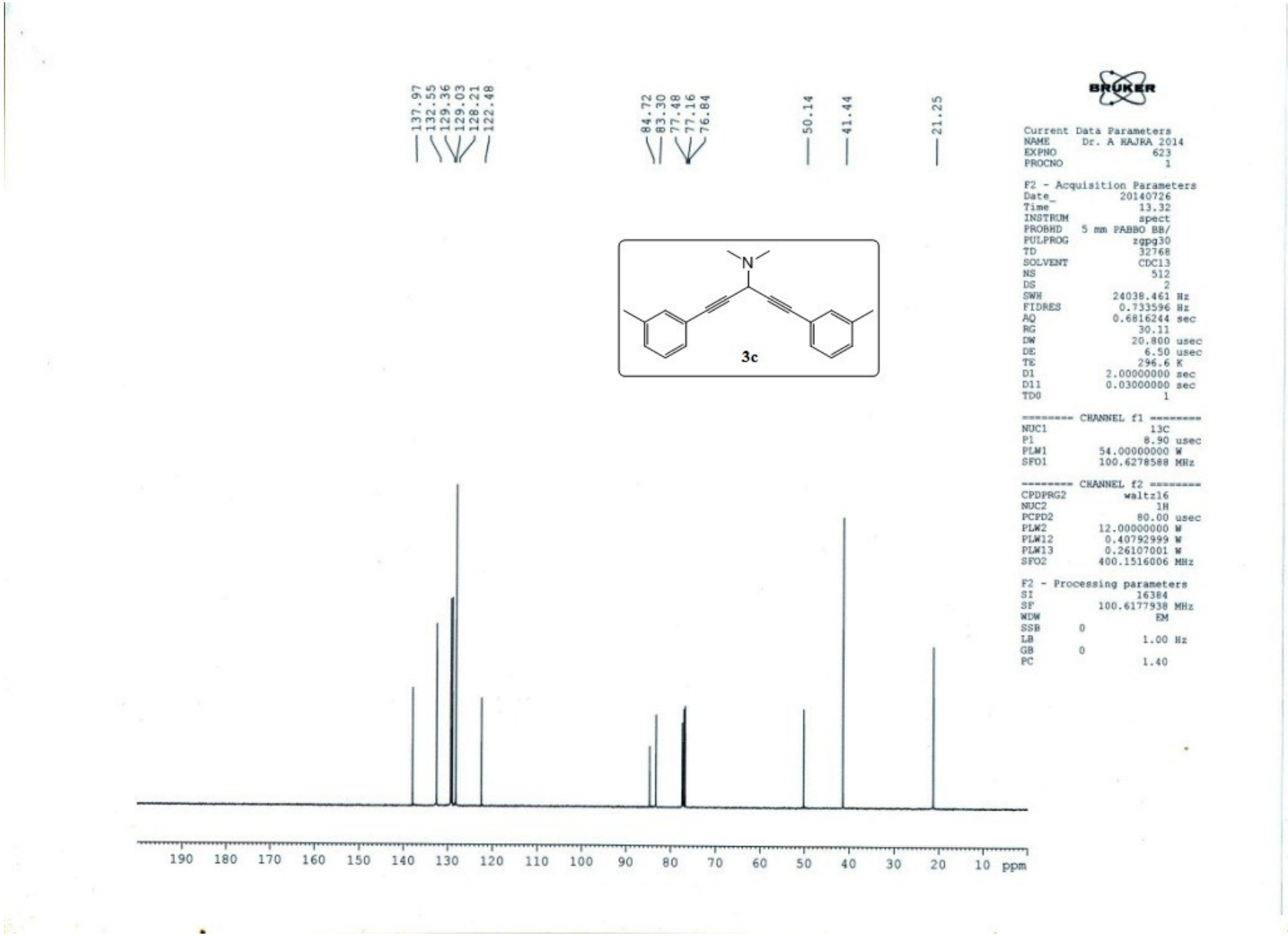
BRUKER

```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO              622
PROCNO               1

F2 - Acquisition Parameters
Date_          20140726
Time               13.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   24
DS                    2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9923444 sec
RG                30.11
DW               60.800 usec
DE                6.50 usec
TE                295.5 K
D1             1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1         11.99499989 W
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                16384
SF          400.1500090 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



BRUKER

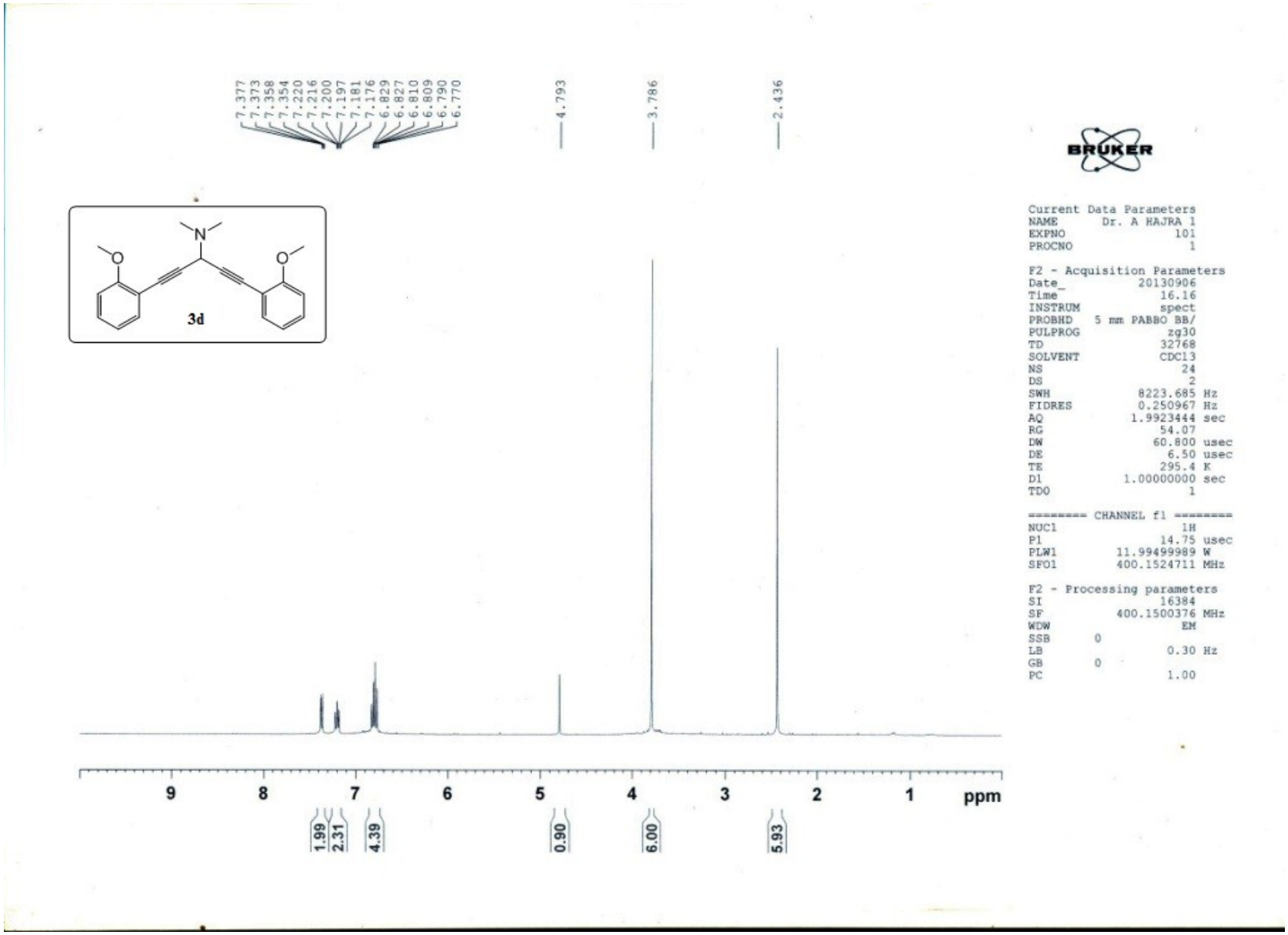
Current Data Parameters
NAME Dr. A HAJRA 2014
EXPNO 623
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140726
Time 13.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 30.11
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177938 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



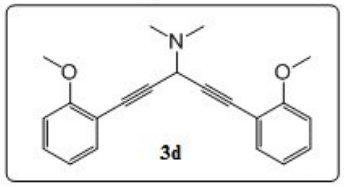
BRUKER

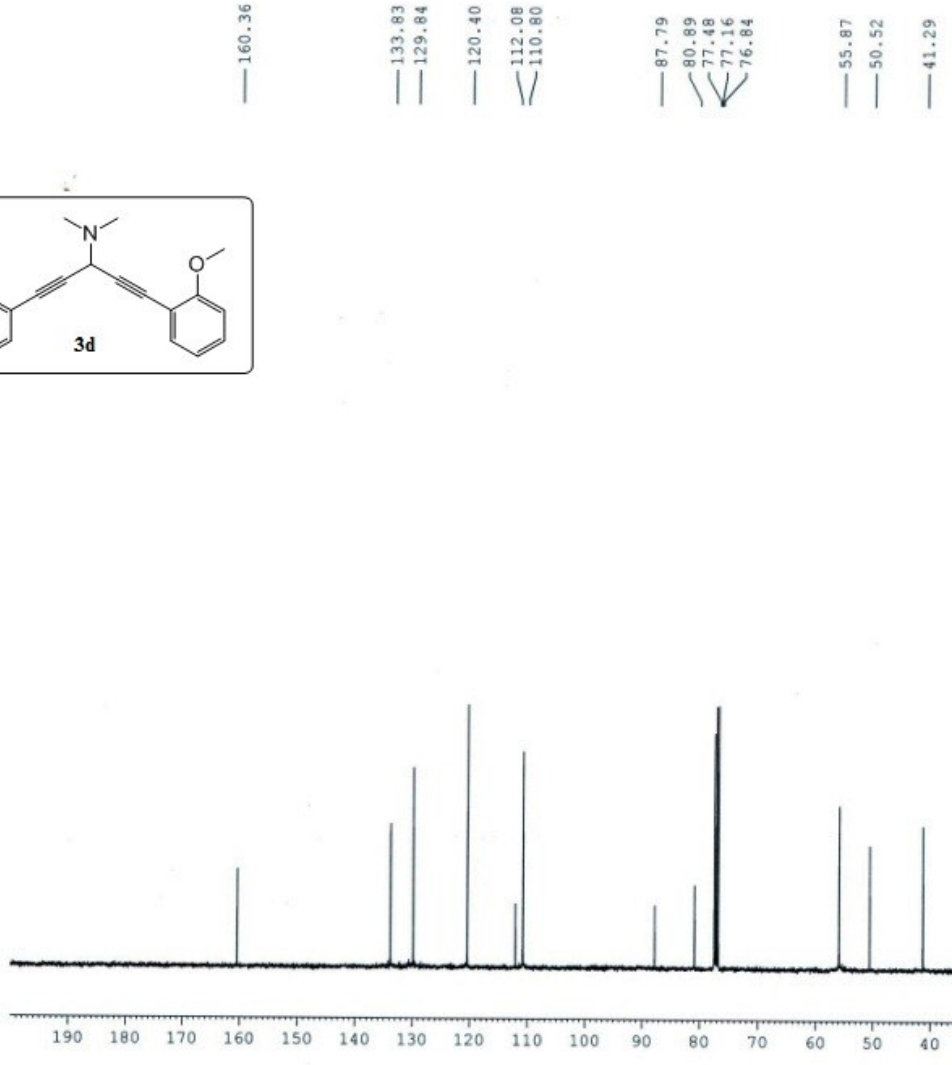
```
Current Data Parameters
NAME        Dr. A HAJRA 1
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130906
Time              16.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  24
DS                   2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                54.07
DW               60.800 usec
DE                6.50 usec
TE               295.4 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1        11.99499989 W
SFO1        400.1524711 MHz

F2 - Processing parameters
SI                16384
SF          400.1500376 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





BRUKER

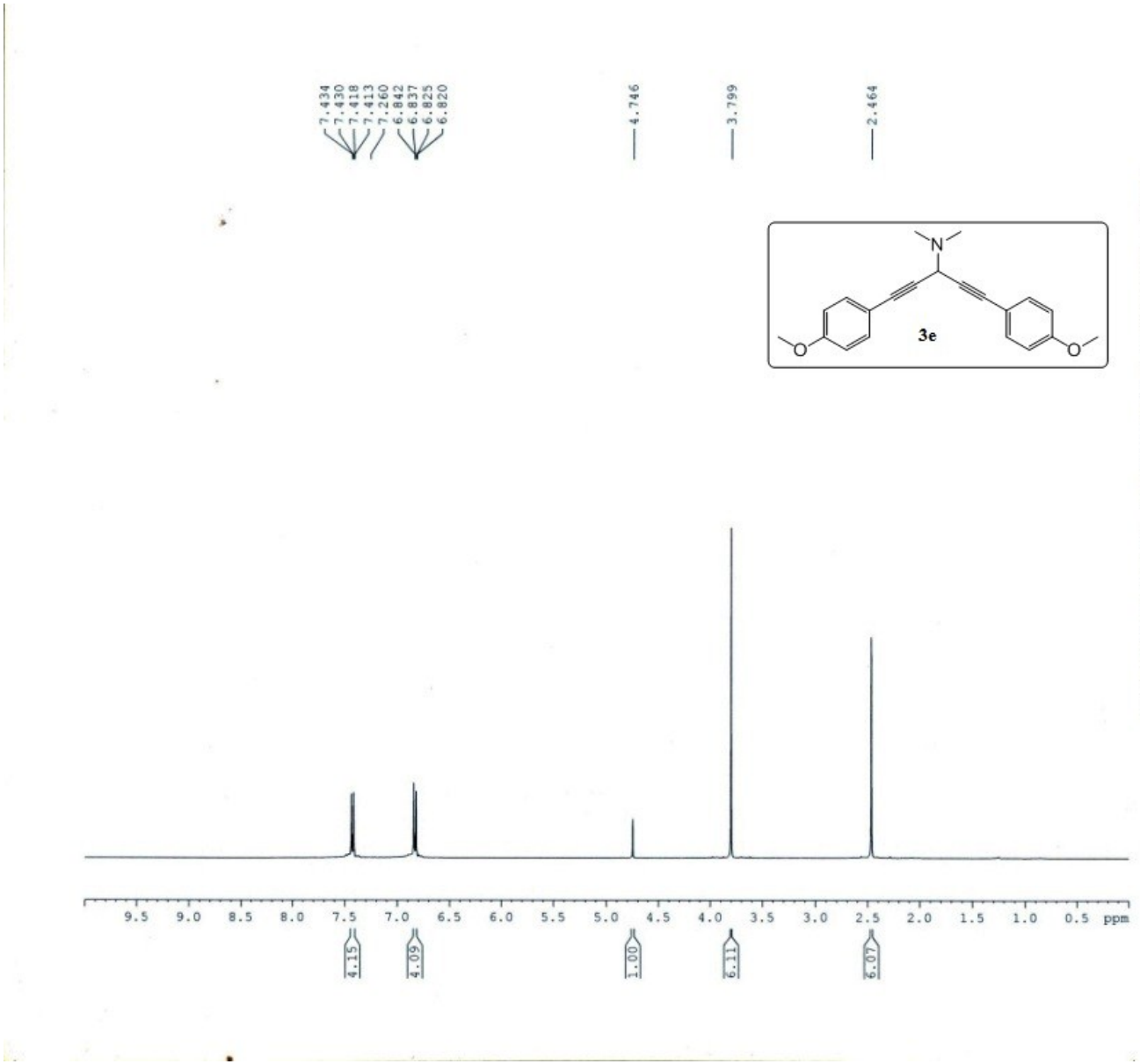
```
Current Data Parameters
NAME        Dr. A HAJRA 1
EXPNO                 108
PROCNO                  1

F2 - Acquisition Parameters
Date_          20130906
Time              18.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 320
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                54.07
DW               20.800 usec
DE                6.50 usec
TE               297.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.90 usec
PLW1         54.00000000 W
SFO1        100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2         12.00000000 W
PLW12         0.40792999 W
PLW13         0.26107001 W
SFO2        400.1516006 MHz

F2 - Processing parameters
SI                 16384
SF          100.6177895 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

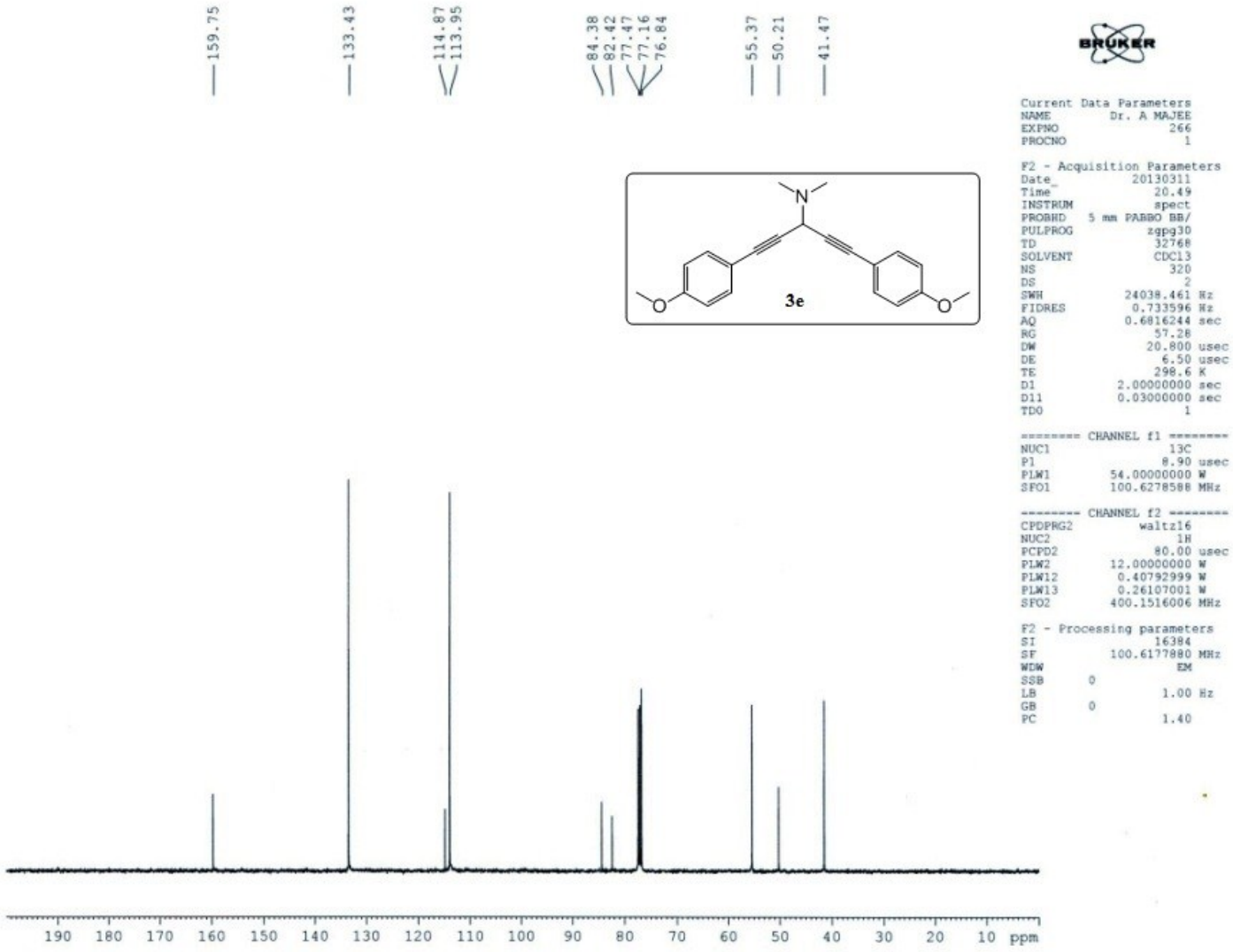



```
Current Data Parameters
NAME          Dr. A MAJEE
EXPNO                 260
PROCNO                  1

F2 - Acquisition Parameters
Date_              20130309
Time                 16.26
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       2
SWH                8223.685 Hz
FIDRES             0.250967 Hz
AQ                 1.9923444 sec
RG                     57.28
DW                    60.800 usec
DE                     6.50 usec
TE                    295.8 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.75 usec
PLW1           11.99499989 W
SFO1           400.1524711 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500090 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



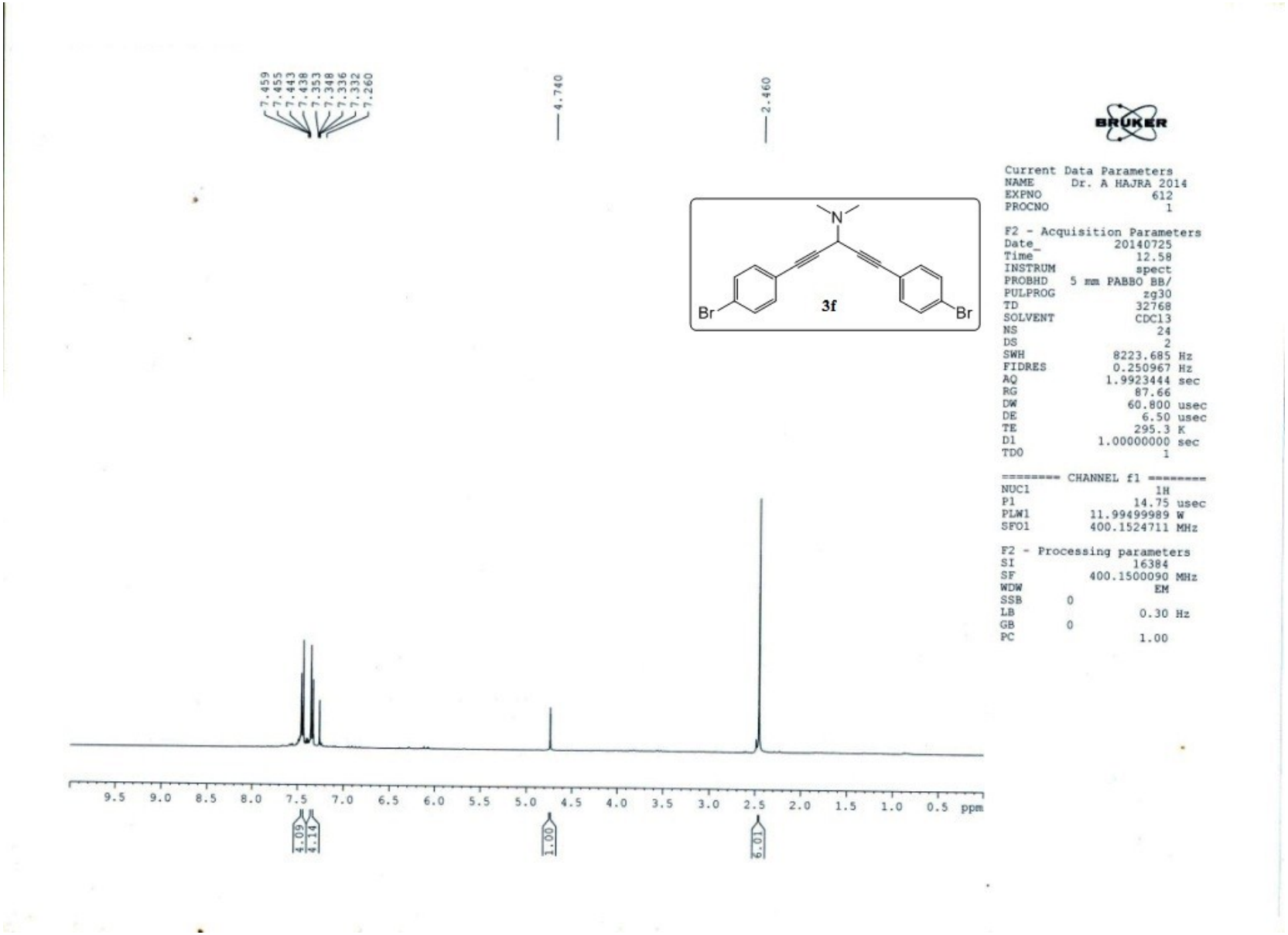

```
Current Data Parameters
NAME         Dr. A MAJEE
EXPNO                266
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130311
Time              20.49
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 320
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                57.28
DW               20.800 usec
DE                6.50 usec
TE              298.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                 16384
SF         100.6177880 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



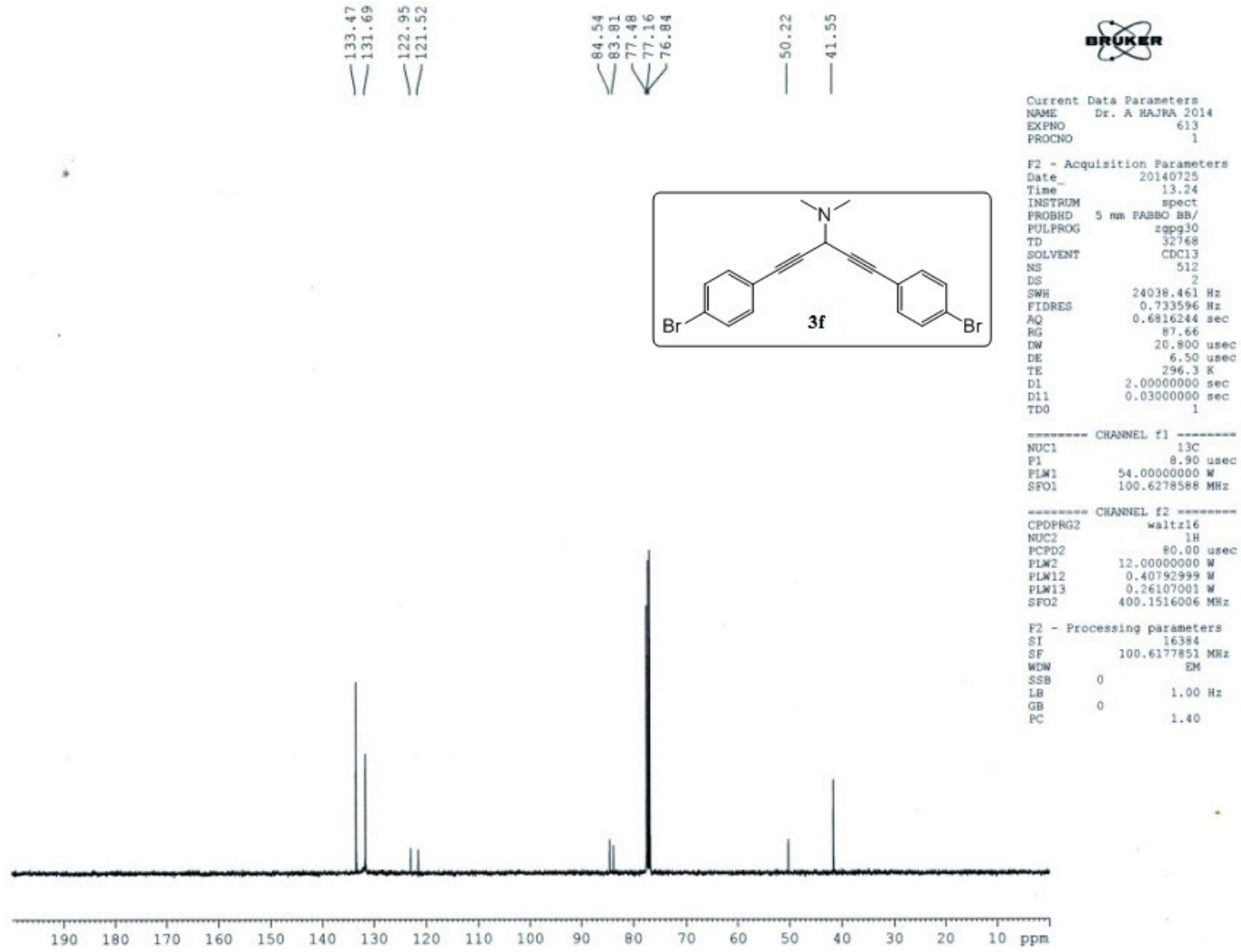
BRUKER

```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO              612
PROCNO               1

F2 - Acquisition Parameters
Date_          20140725
Time              12.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  24
DS                   2
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                87.66
DW               60.800 usec
DE                6.50 usec
TE               295.3 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1         11.99499989 W
SFO1        400.1524711 MHz

F2 - Processing parameters
SI                16384
SF          400.1500090 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



BRUKER

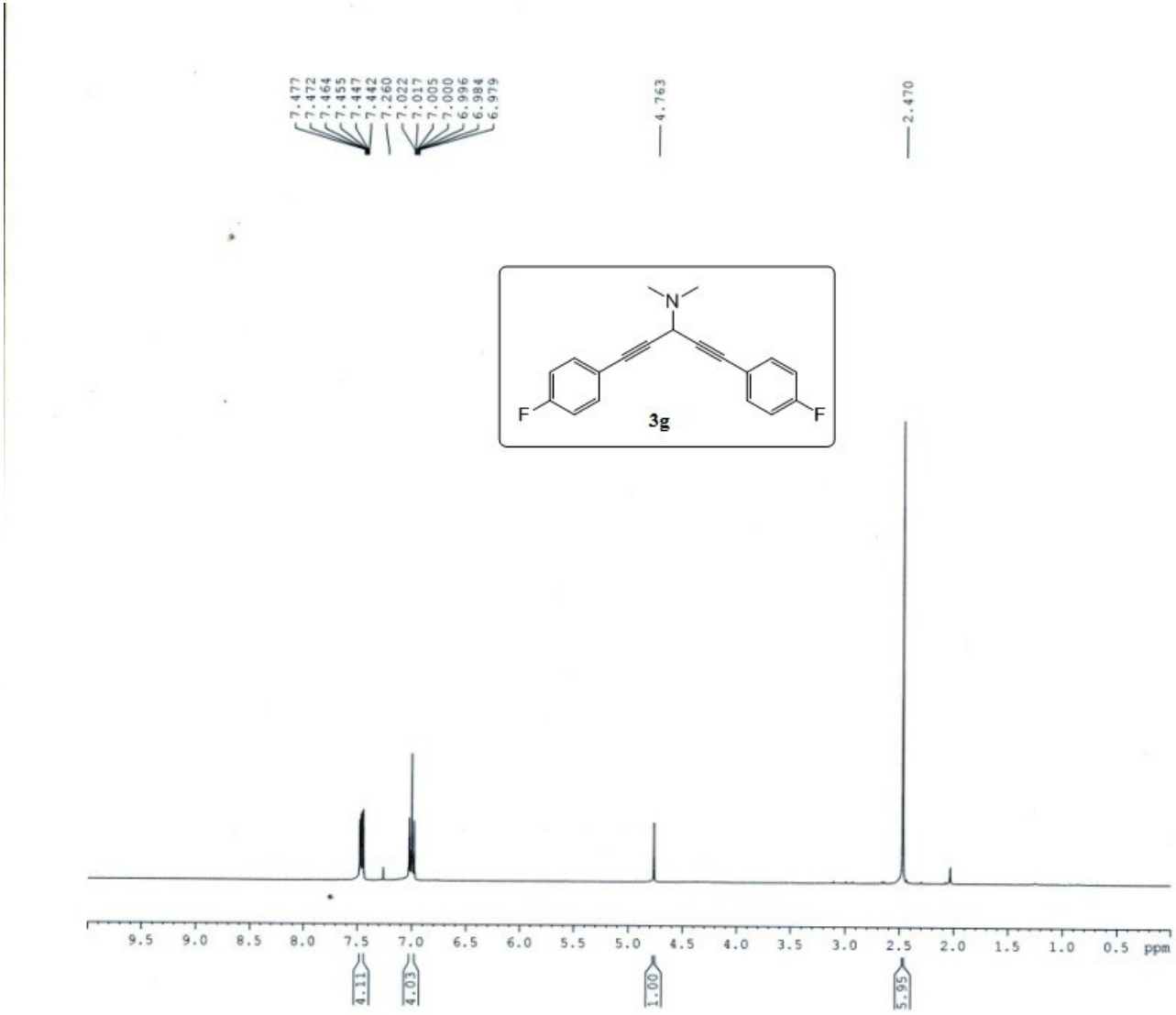
```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO              613
PROCNO               1

F2 - Acquisition Parameters
Date_          20140725
Time             13.24
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 512
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                87.66
DW               20.800 usec
DE                6.50 usec
TE               296.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                8.90 usec
PLW1       54.00000000 W
SFO1      100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2       12.00000000 W
PLW12       0.40792999 W
PLW13       0.26107001 W
SFO2      400.1516006 MHz

F2 - Processing parameters
SI               16384
SF        100.6177851 MHz
WDW                EM
SSB      0
LB               1.00 Hz
GB       0
PC               1.40
```



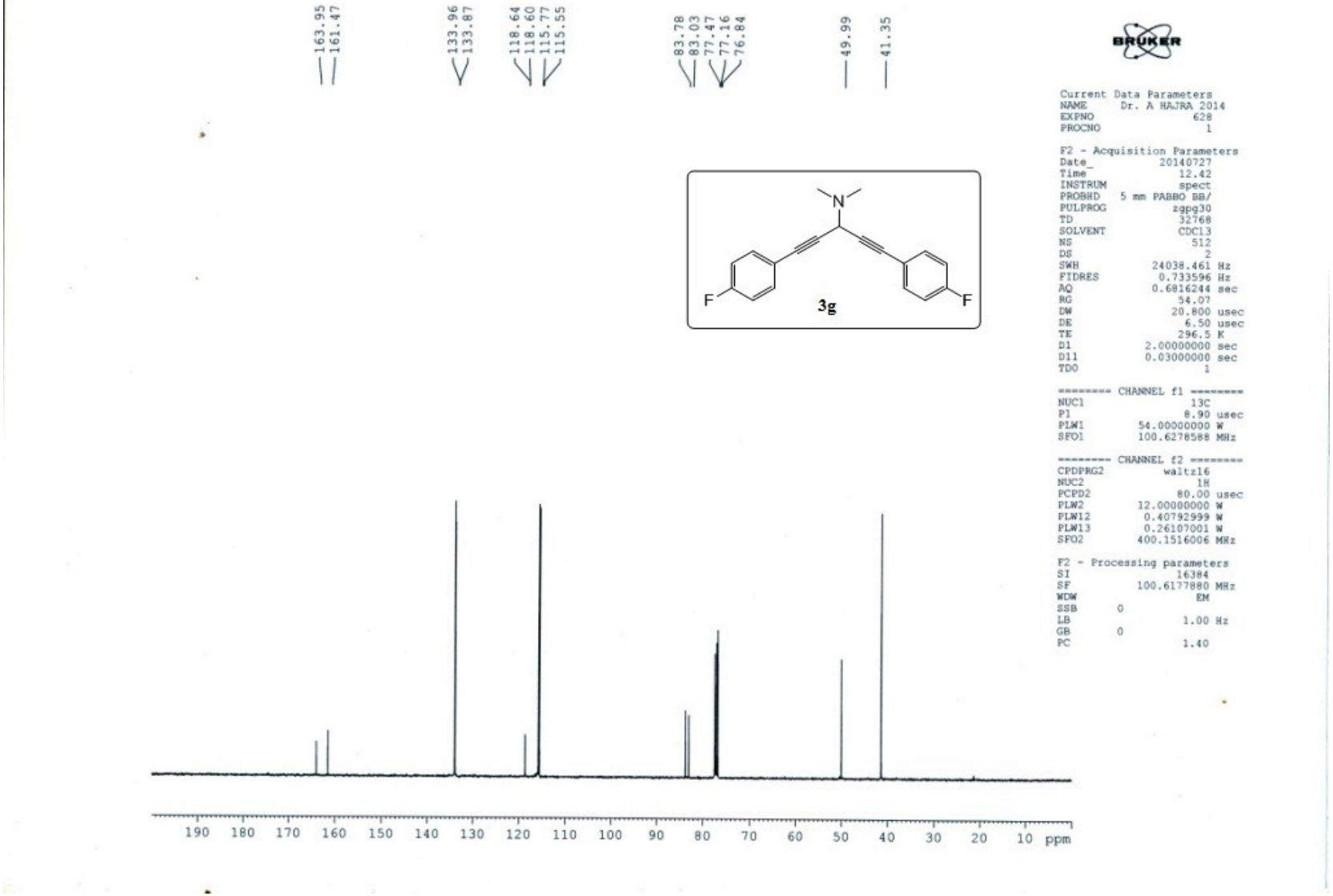
BRUKER

```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO                  627
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140727
Time                 12.13
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       2
SWH               8223.685 Hz
FIDRES            0.250967 Hz
AQ                1.9923444 sec
RG                    54.07
DW                   60.800 usec
DE                    6.50 usec
TE                   295.5 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.75 usec
PLW1          11.99499989 W
SFO1          400.1524711 MHz

F2 - Processing parameters
SI                   16384
SF            400.1500090 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



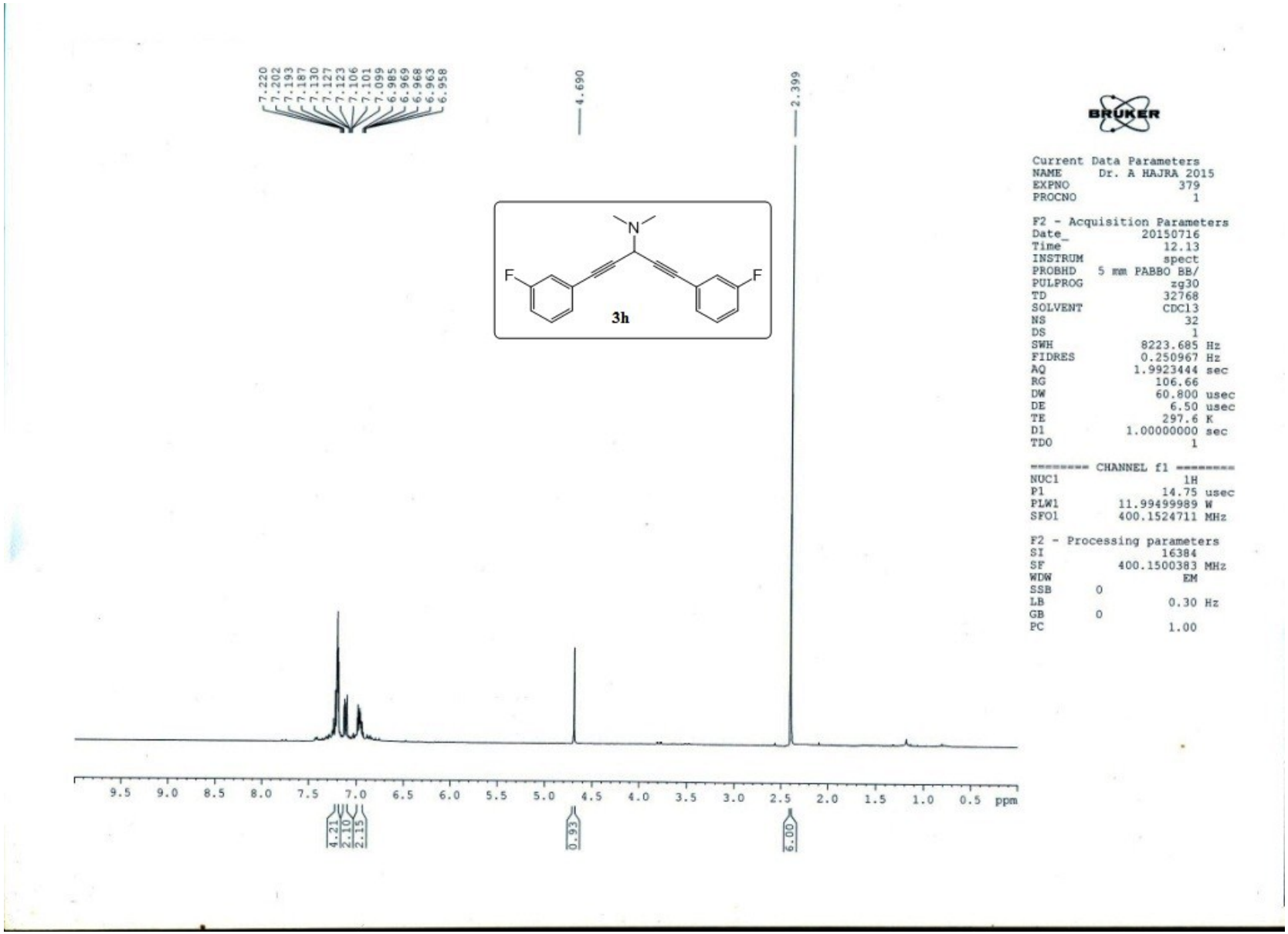

```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO              628
PROCNO               1

F2 - Acquisition Parameters
Date_          20140727
Time             12.42
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD               32768
SOLVENT          CDCl3
NS                 512
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                54.07
DW               20.800 usec
DE                6.50 usec
TE               296.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                8.90 usec
PLW1       54.00000000 W
SFO1      100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2             80.00 usec
PLW2       12.00000000 W
PLW12       0.40792999 W
PLW13       0.26107001 W
SFO2      400.1516006 MHz

F2 - Processing parameters
SI                16384
SF        100.6177880 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



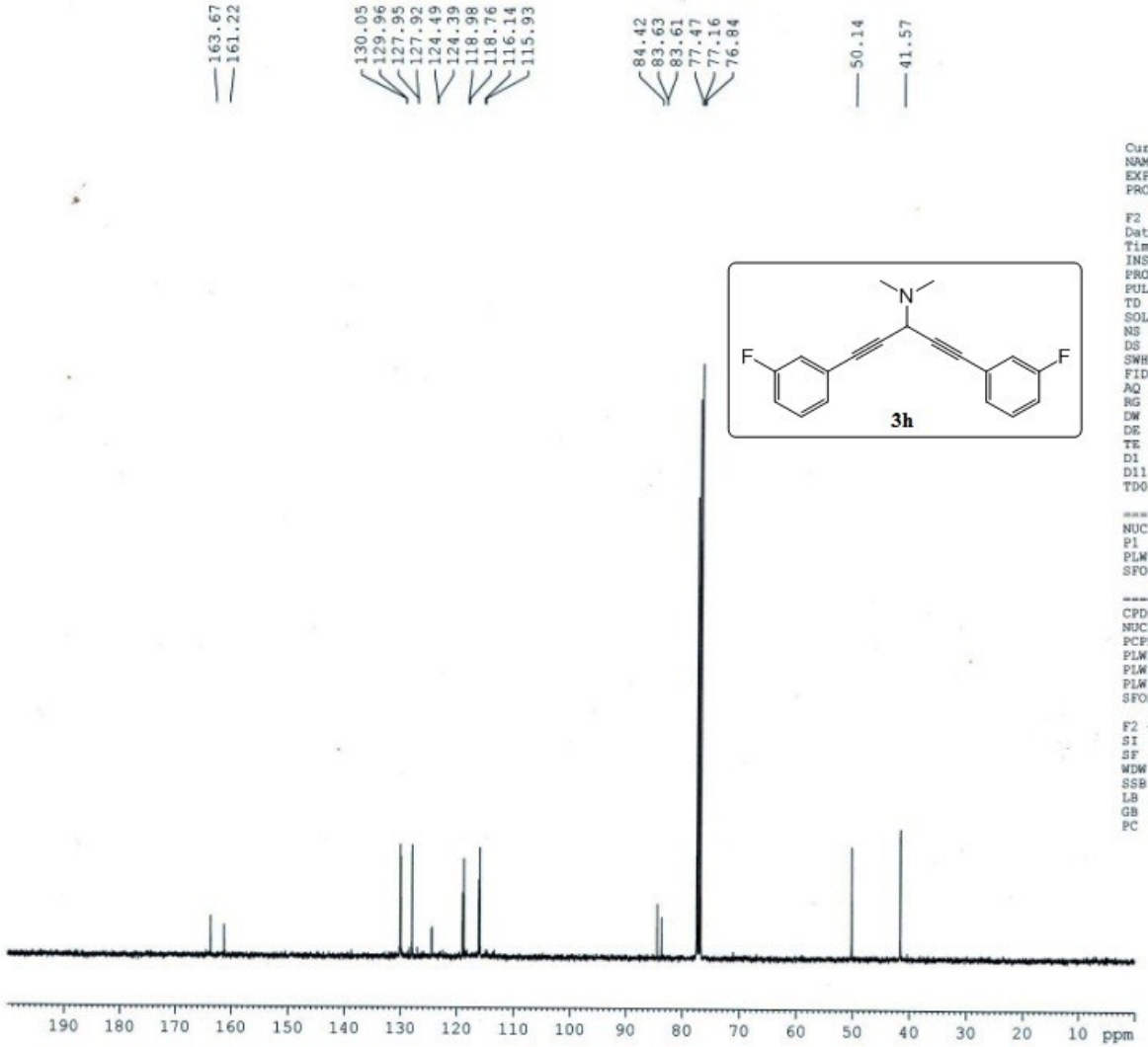
BRUKER

Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 379
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150716
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 106.66
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500383 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

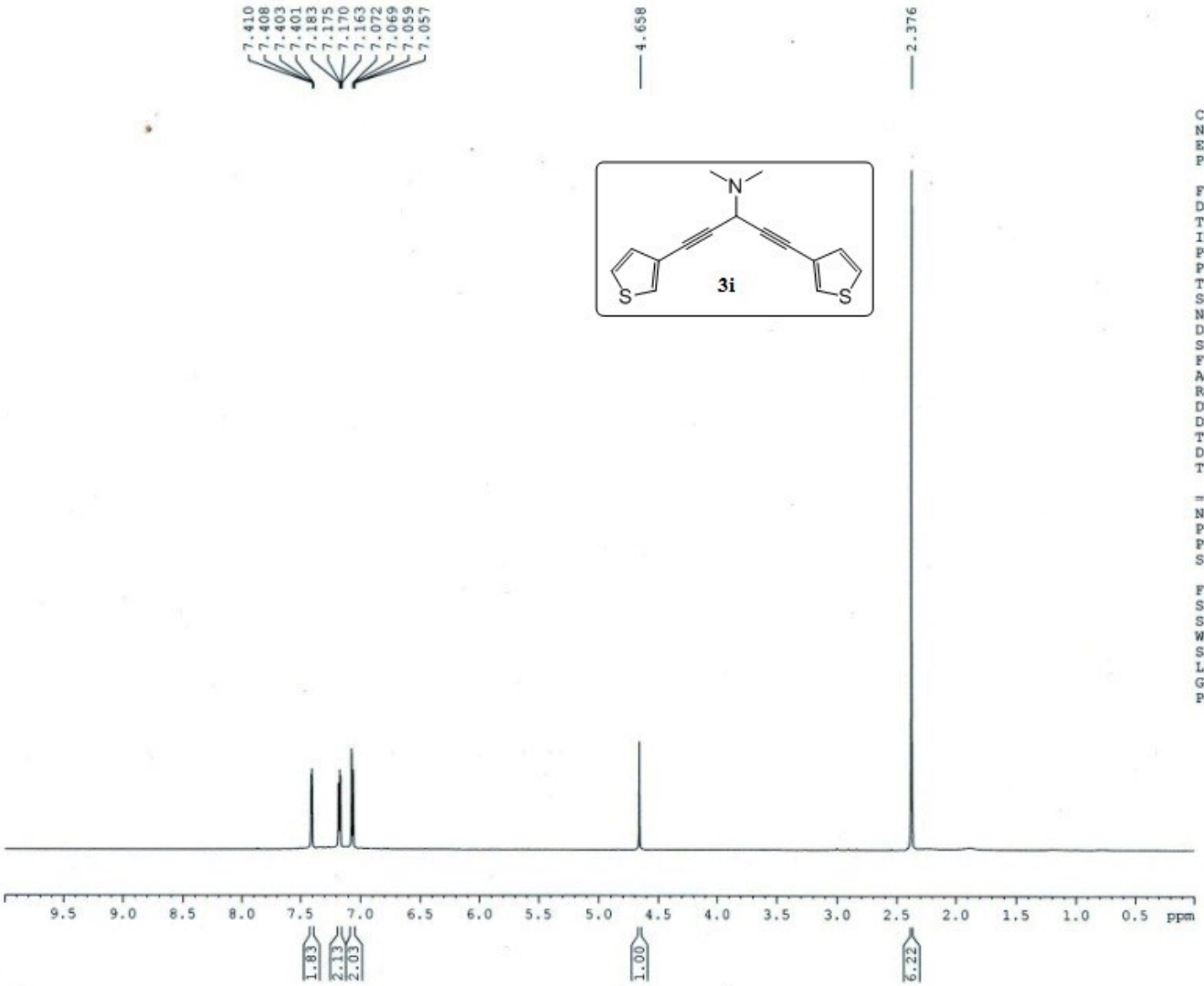
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 380
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150716
Time 13.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 106.66
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177836 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



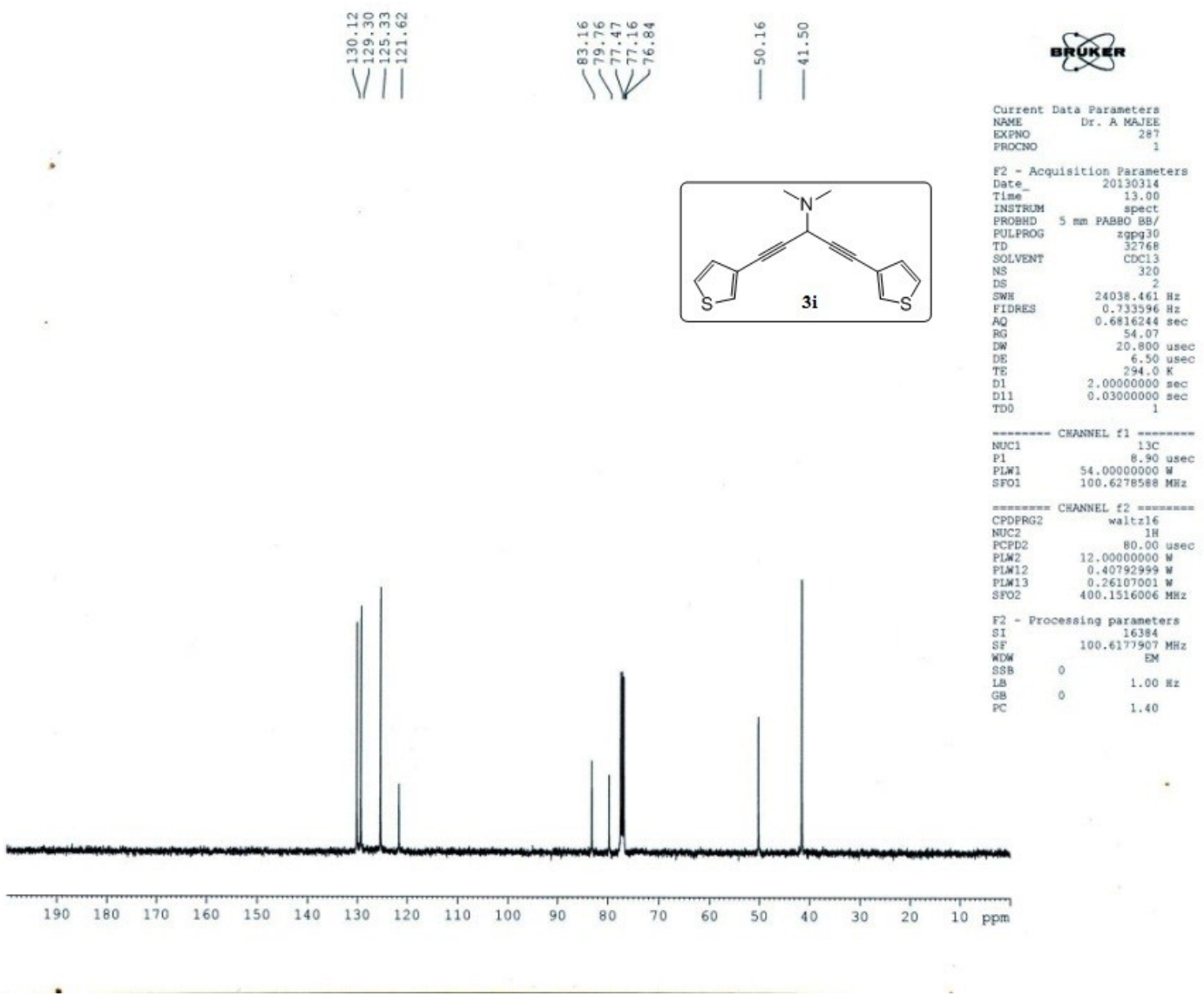
BRUKER

```
Current Data Parameters
NAME         Dr. A MAJEE
EXPNO               275
PROCNO                1

F2 - Acquisition Parameters
Date_          20130312
Time              20.58
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  24
DS                   2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            1.9923444 sec
RG                54.07
DW               60.800 usec
DE                6.50 usec
TE               297.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.75 usec
PLW1         11.99499989 W
SFO1         400.1524711 MHz

F2 - Processing parameters
SI                16384
SF           400.1500407 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



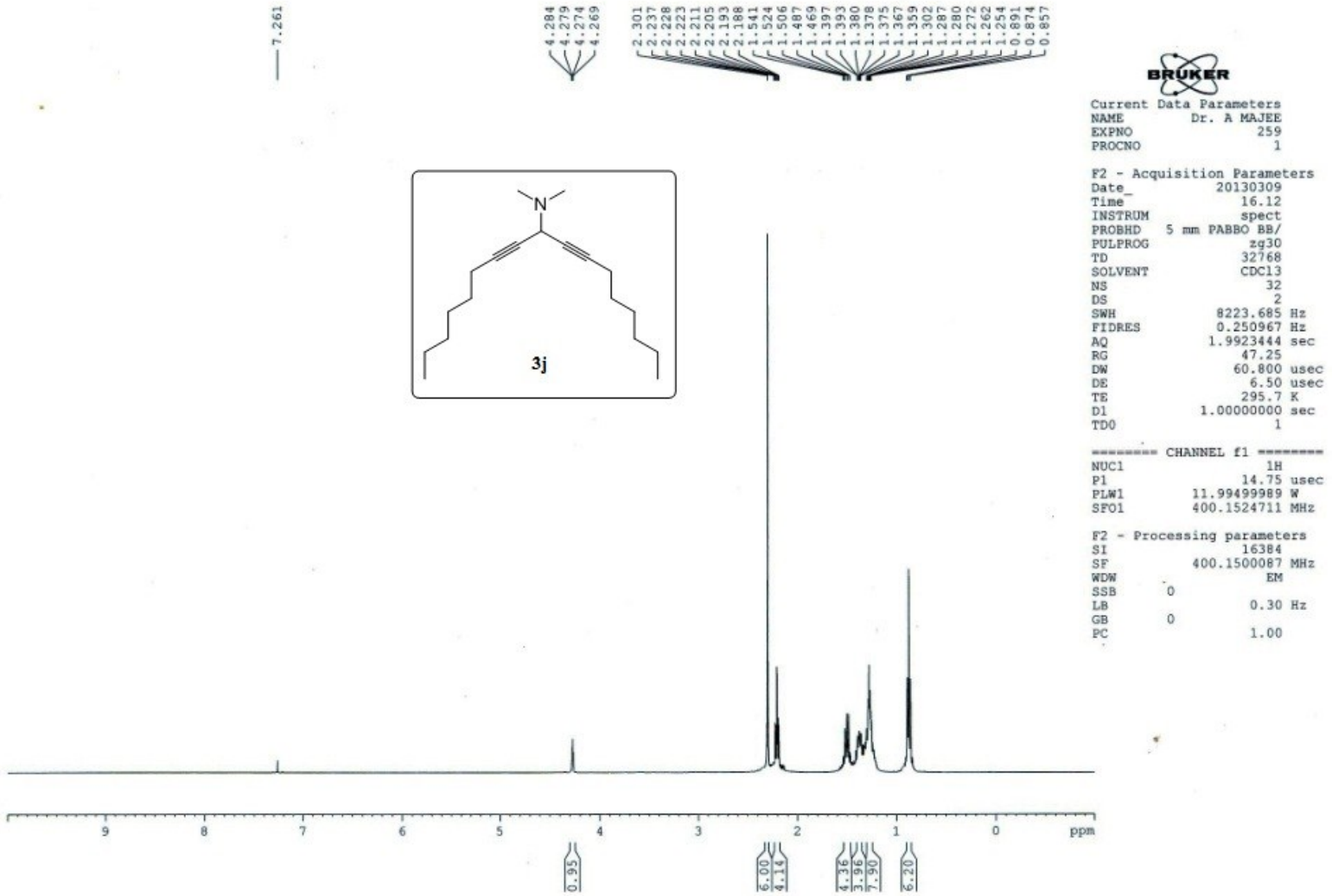

```
Current Data Parameters
NAME         Dr. A MAJEE
EXPNO                287
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130314
Time              13.00
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 320
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                54.07
DW               20.800 usec
DE                6.50 usec
TE               294.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                 8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                 16384
SF         100.6177907 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



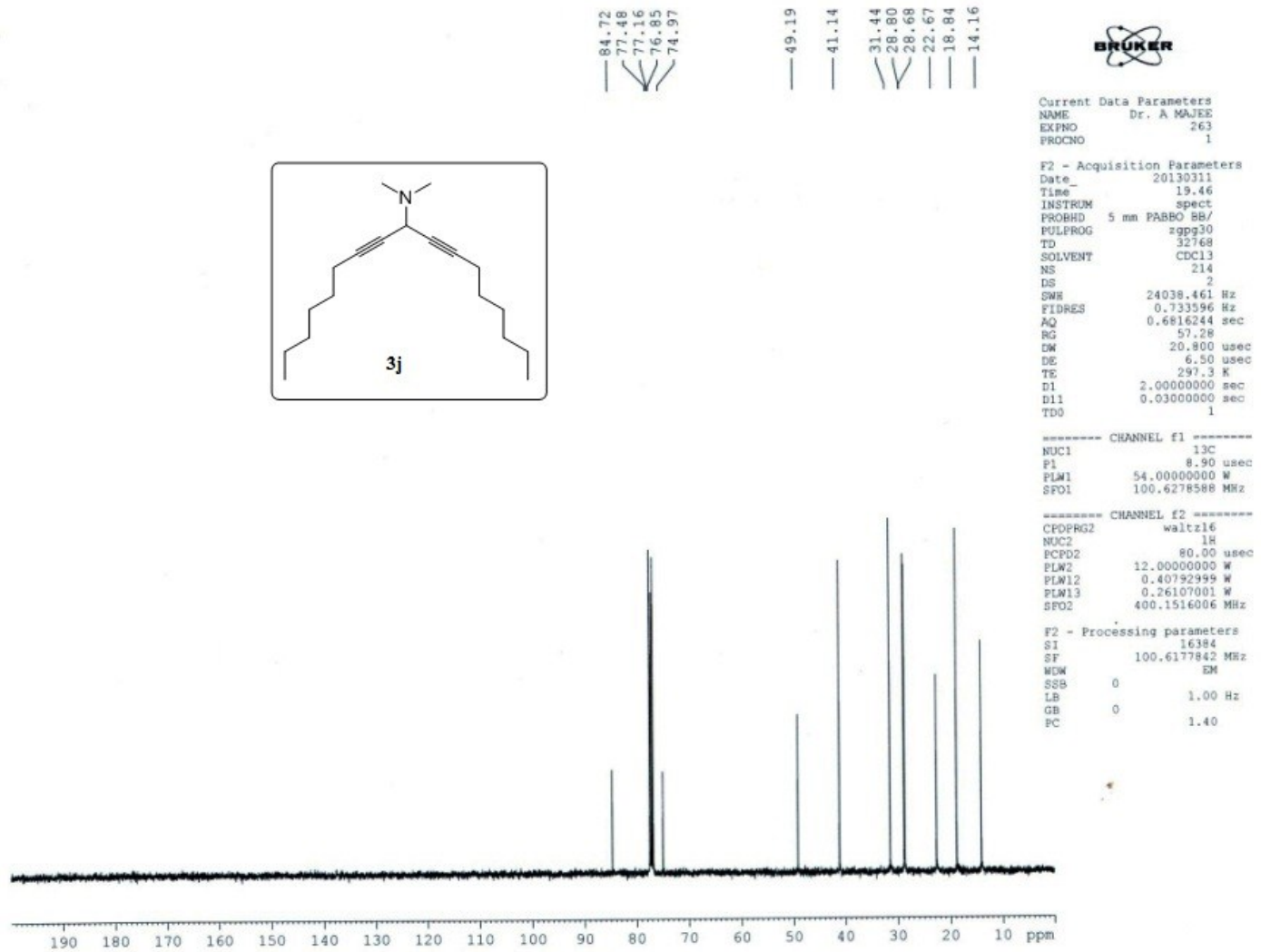
BRUKER

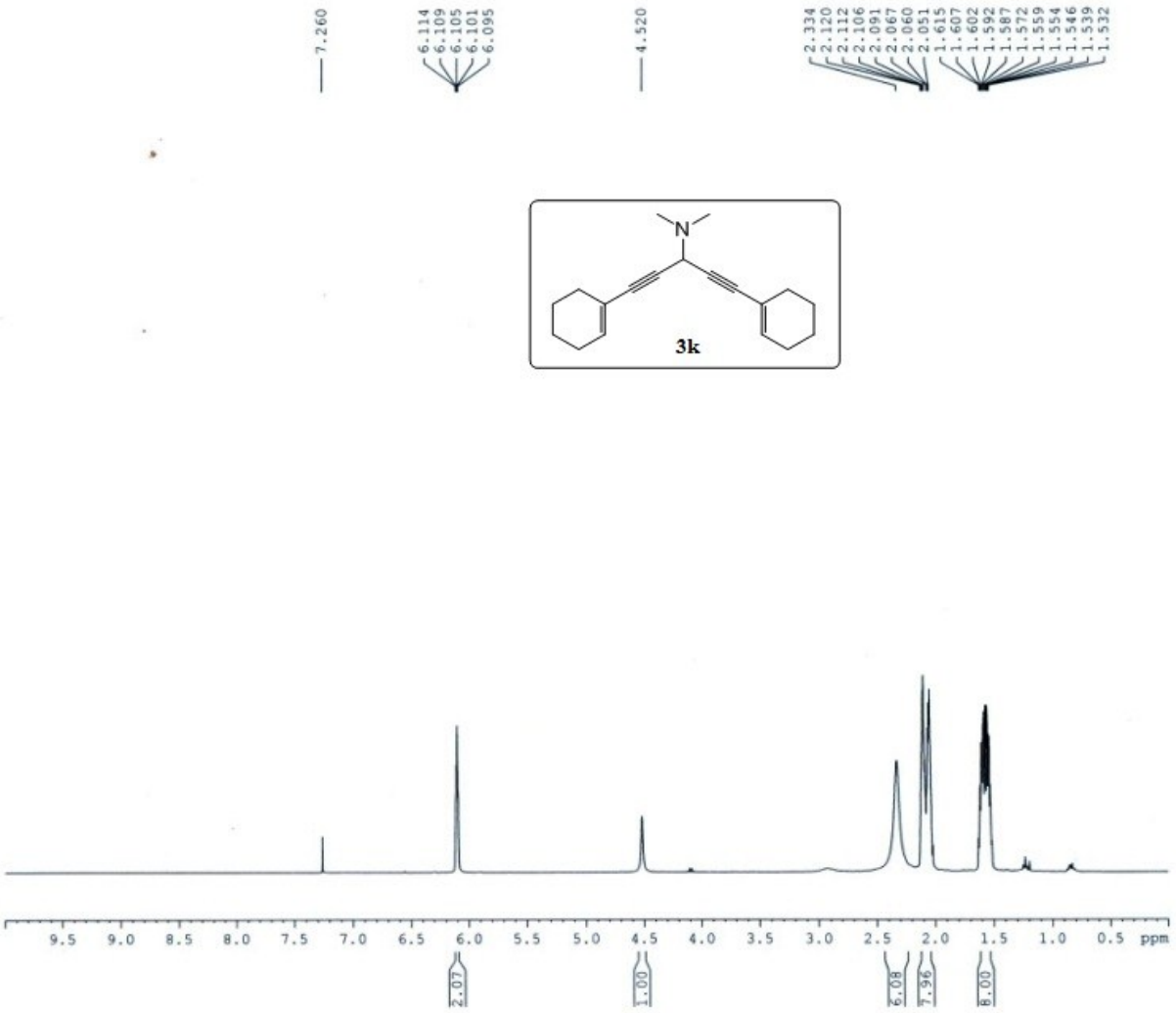
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 259
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130309
Time 16.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 47.25
DW 60.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


84.72
77.48
77.16
76.85
74.97
49.19
41.14
31.44
28.80
28.68
22.67
18.84
14.16
N
3j
BRUKER
Current Data Parameters
NAME Dr. A MAJEE
EXPNO 263
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130311
Time 19.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 214
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 57.28
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz
F2 - Processing parameters
SI 16384
SF 100.6177842 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

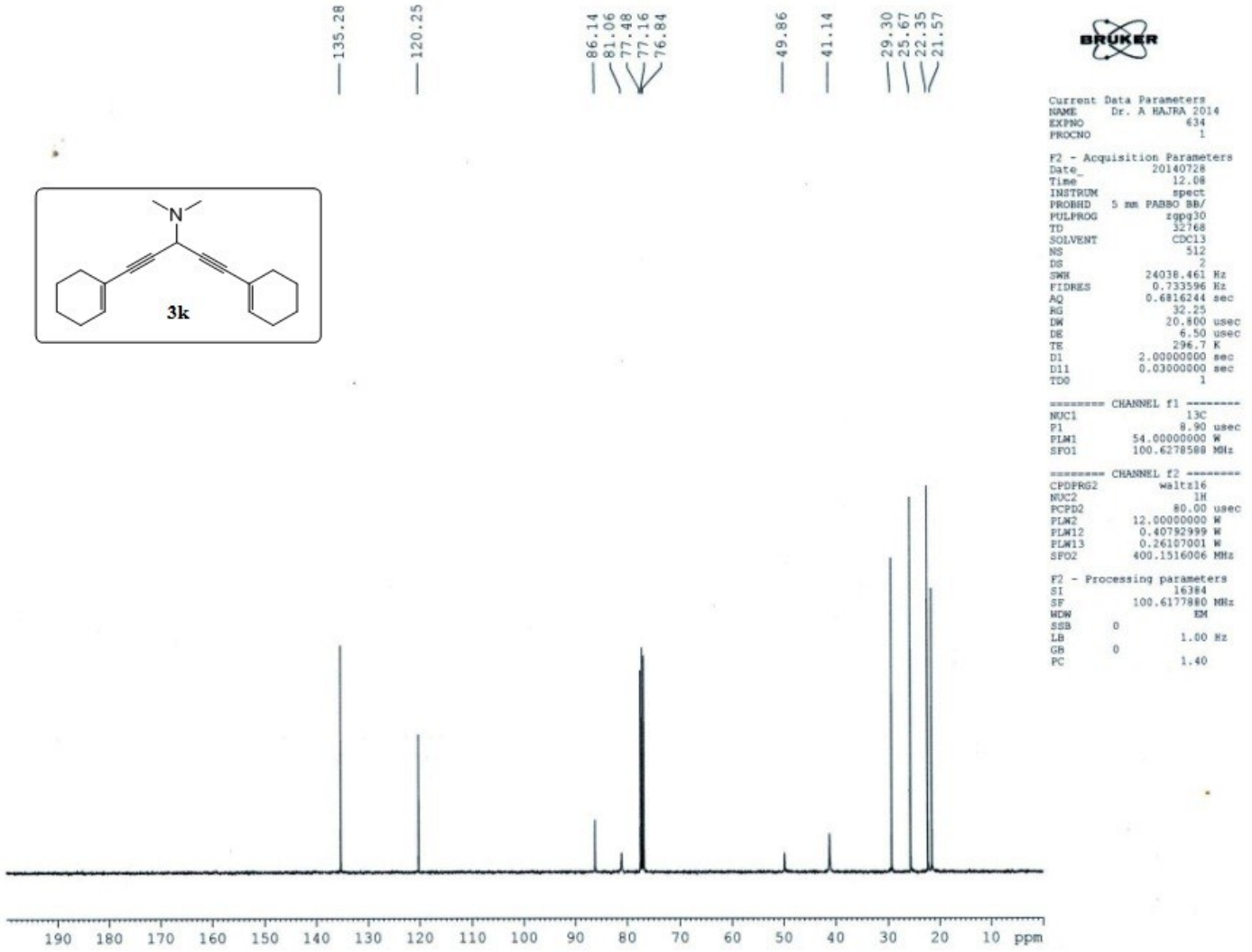



```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO                633
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140728
Time               11.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9923444 sec
RG                 32.25
DW                60.800 usec
DE                 6.50 usec
TE                295.6 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.75 usec
PLW1         11.99499989 W
SFO1        400.1524711 MHz

F2 - Processing parameters
SI                 16384
SF          400.1500090 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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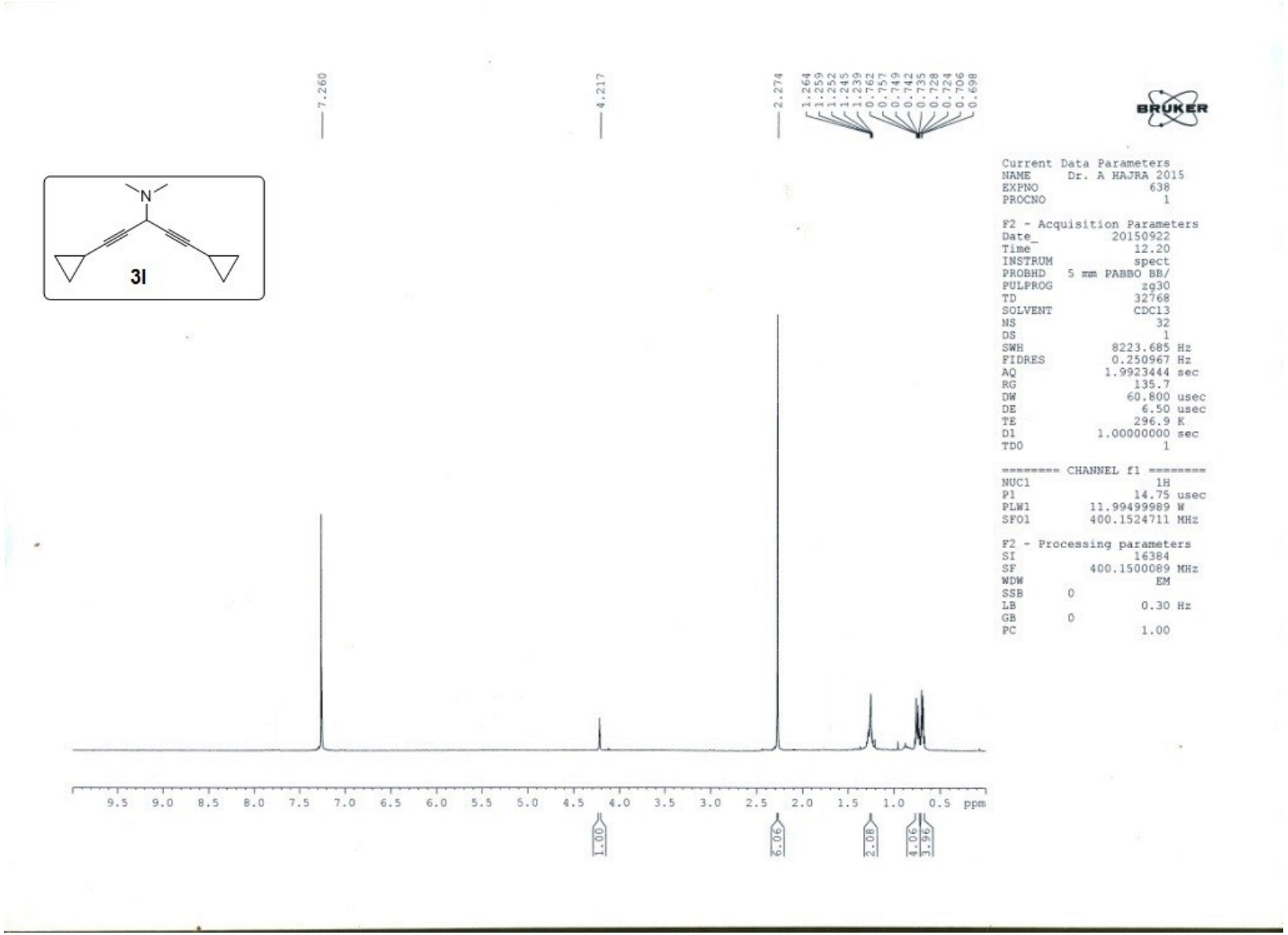
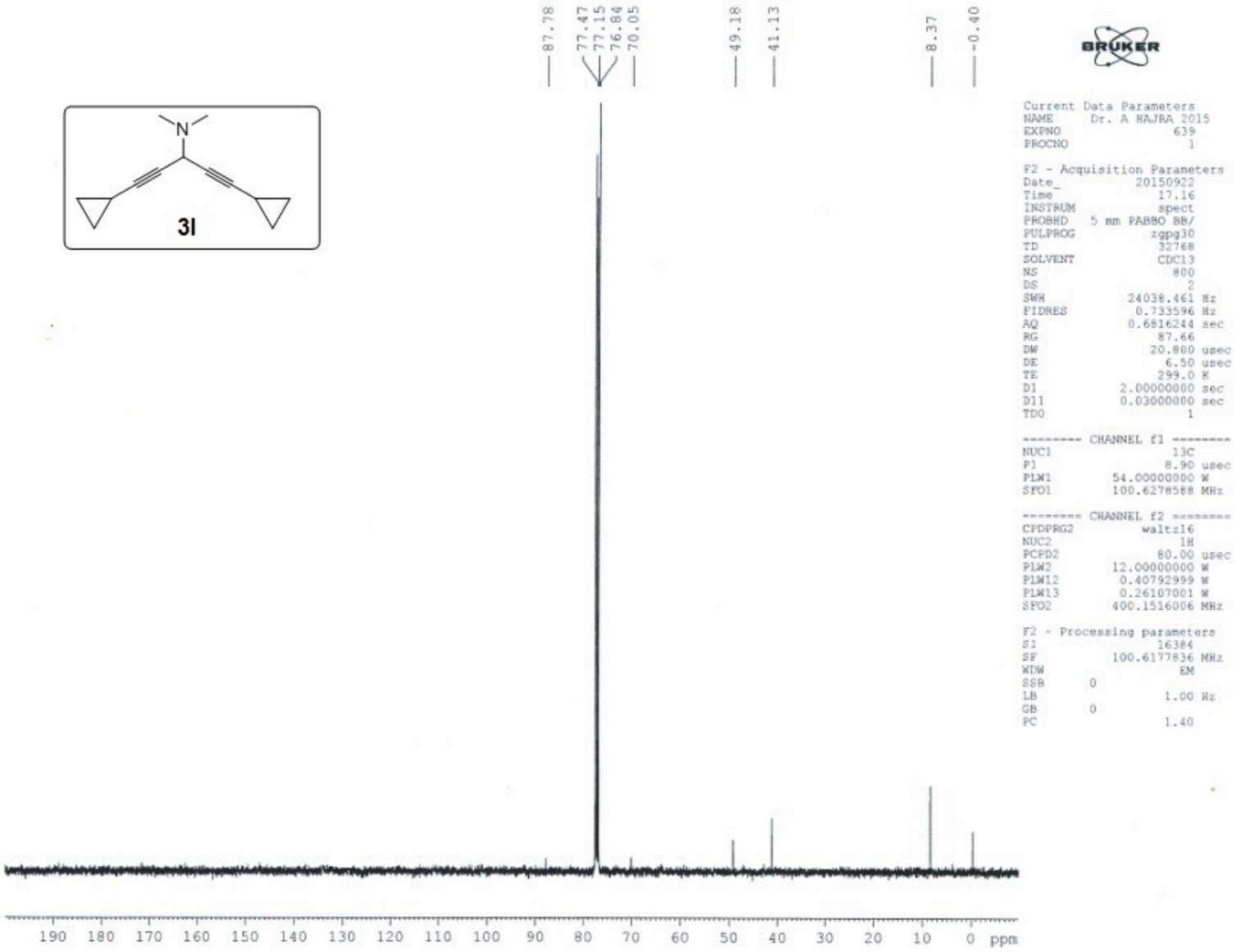
```
Current Data Parameters
NAME      Dr. A HAJRA 2014
EXPNO              634
PROCNO               1

F2 - Acquisition Parameters
Date_          20140728
Time             12.08
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD             32768
SOLVENT          CDCl3
NS                 512
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                32.25
DW               20.800 usec
DE                6.50 usec
TE              296.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                8.90 usec
PLW1        54.00000000 W
SFO1       100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2        12.00000000 W
PLW12        0.40792999 W
PLW13        0.26107001 W
SFO2       400.1516006 MHz

F2 - Processing parameters
SI                16384
SF         100.6177880 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```


7.260
4.217
2.274
1.264
1.259
1.252
1.245
1.239
0.762
0.757
0.749
0.742
0.735
0.728
0.724
0.706
0.698
N
3l
BRUKER
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 638
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150922
Time 12.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 135.7
DW 60.800 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz
F2 - Processing parameters
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
6.06
2.08
4.06
3.96



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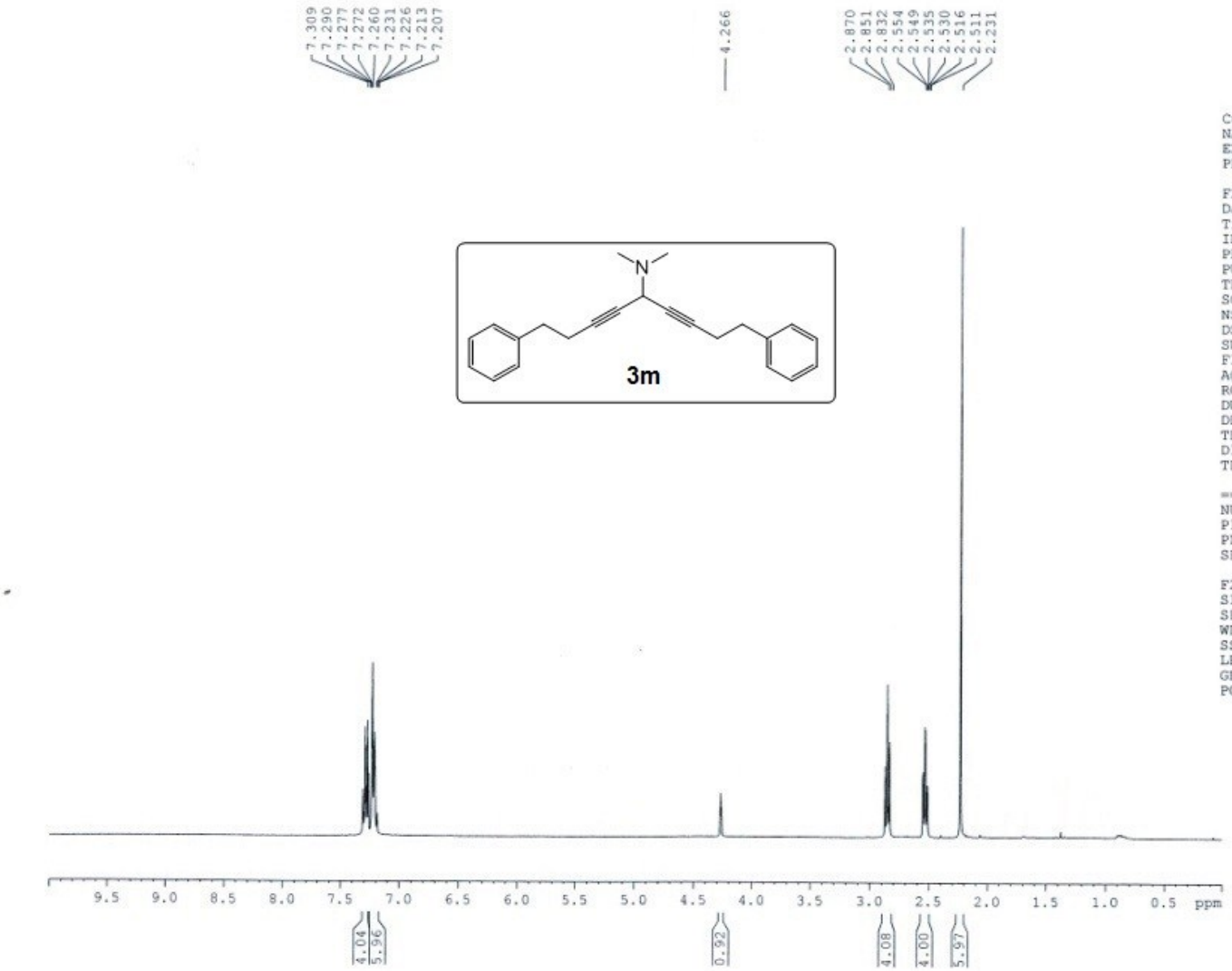
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 639
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150922
Time 17.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 800
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 87.66
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177836 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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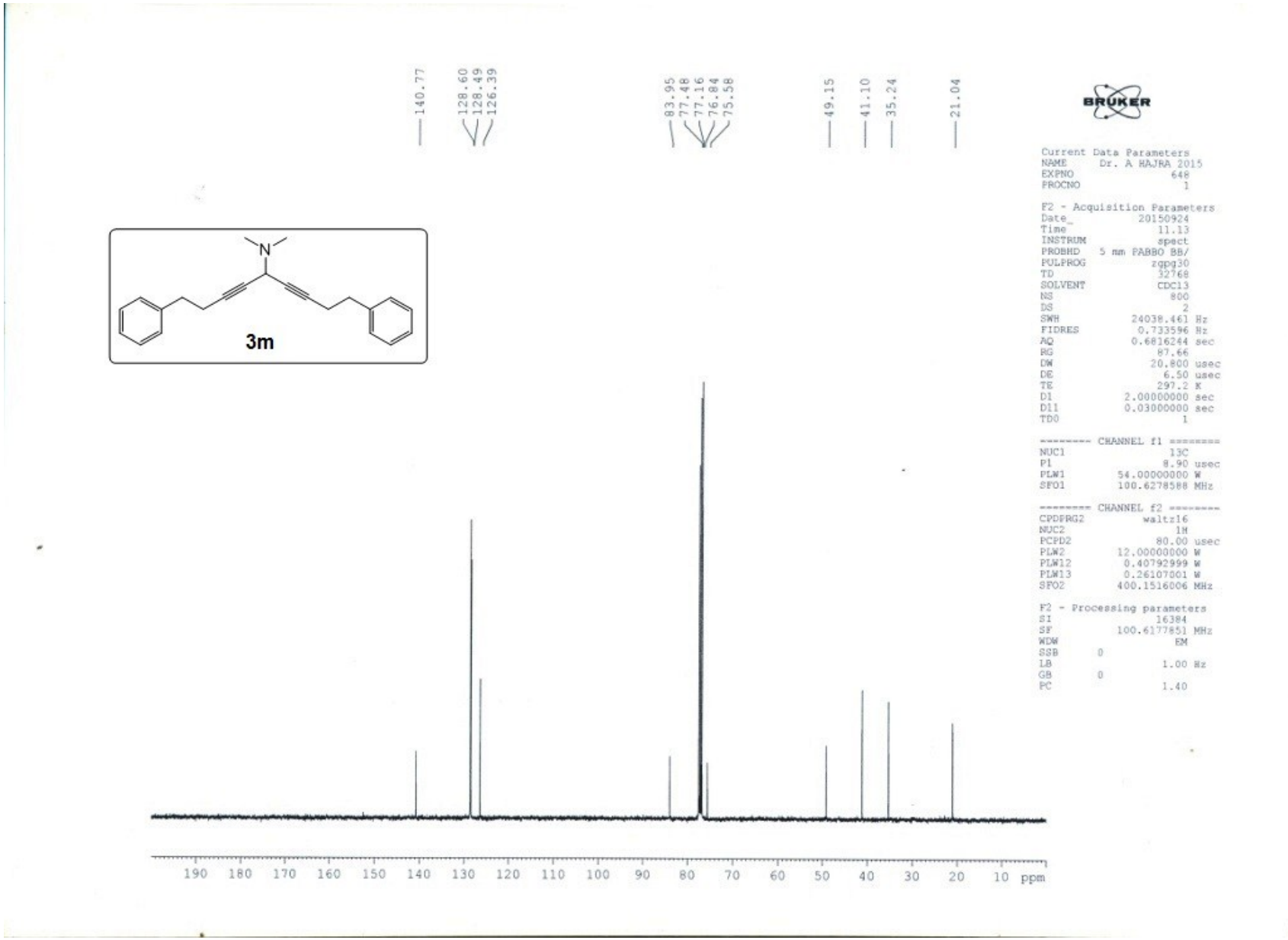
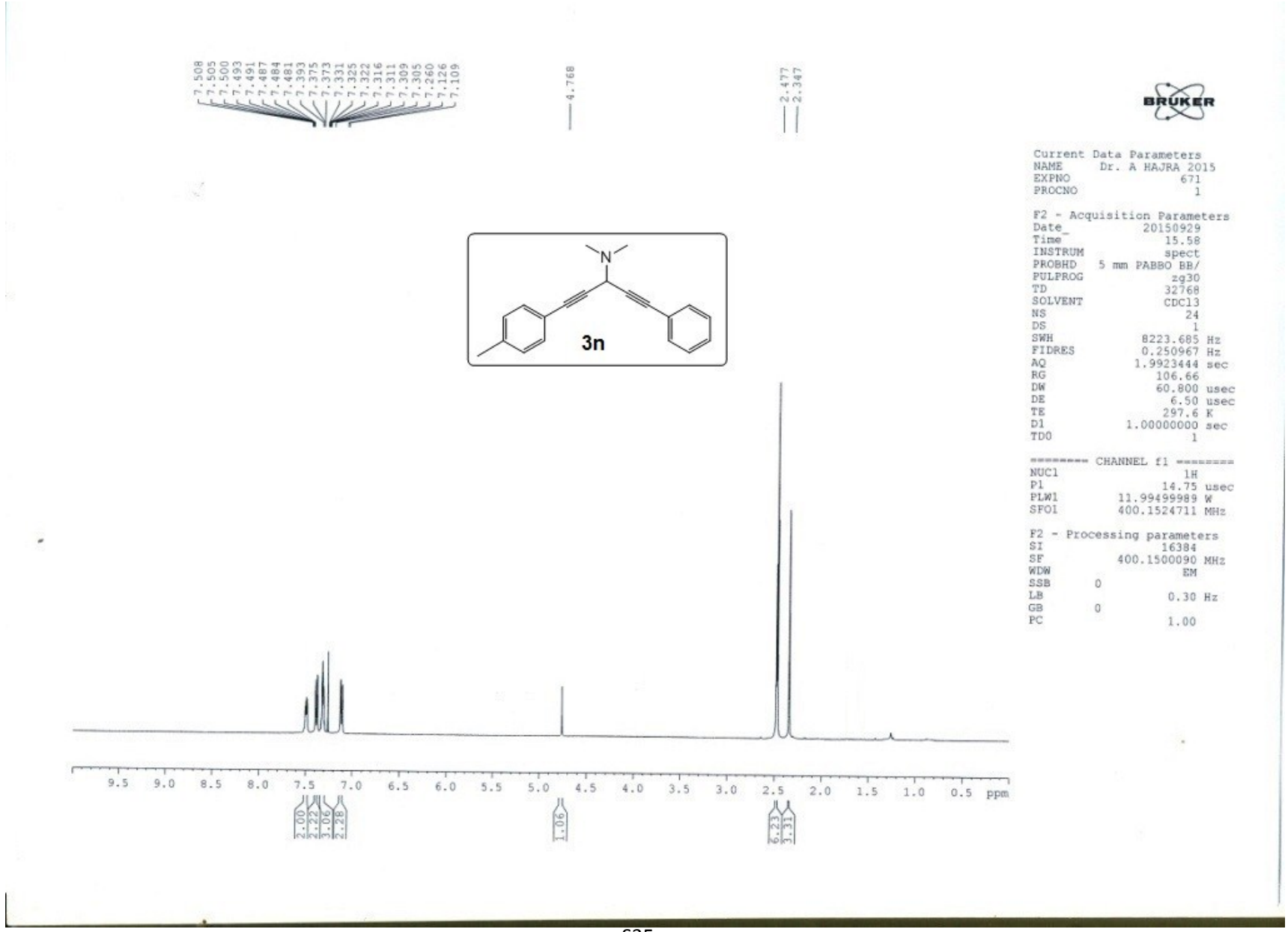
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 647
PROCNO 1

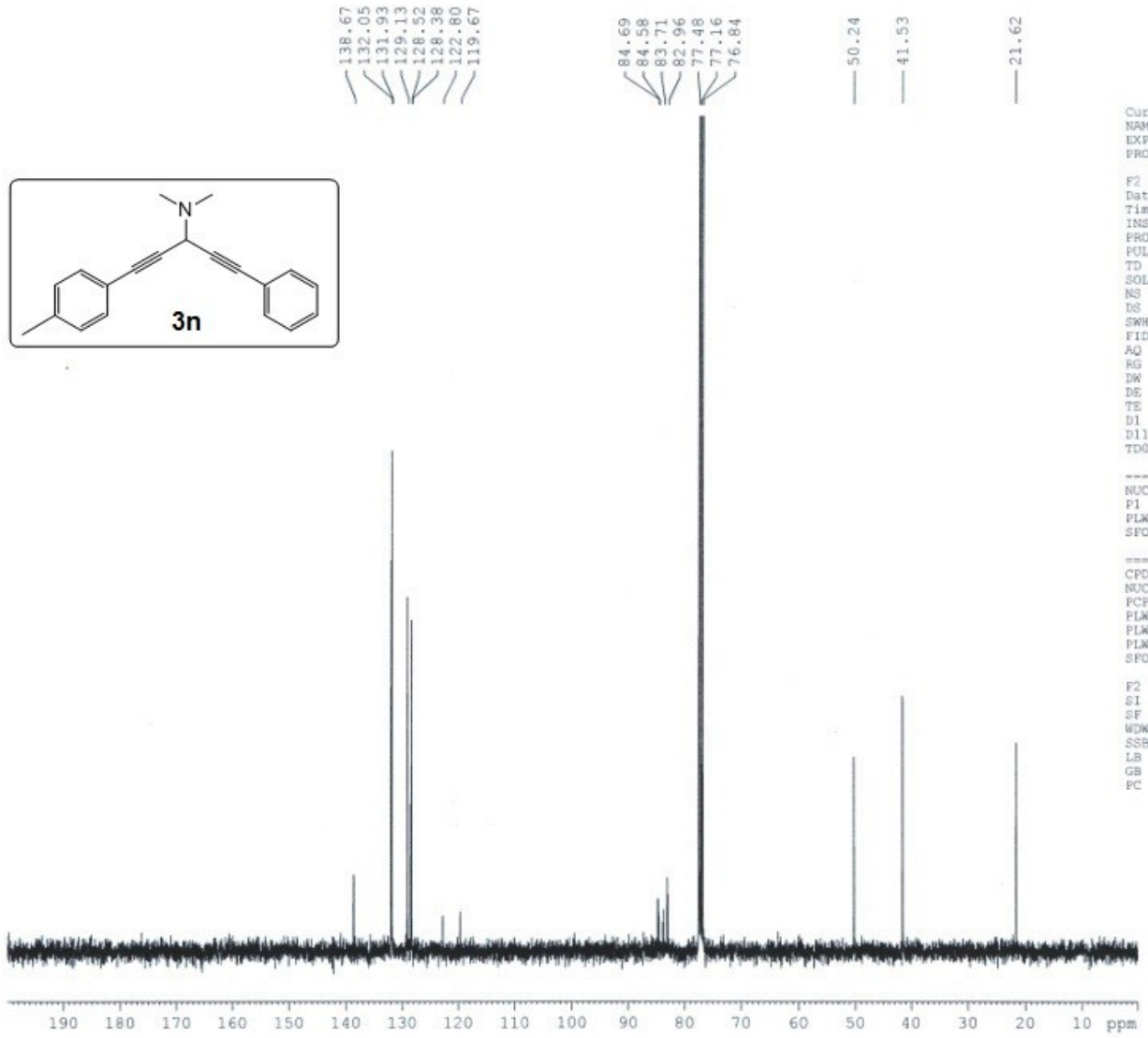
F2 - Acquisition Parameters
Date_ 20150924
Time 10.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 93.46
DW 60.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


140.77
128.60
128.49
126.39
83.95
77.48
77.16
76.84
75.58
49.15
41.10
35.24
21.04
N
3m
BRUKER
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 648
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150924
Time 11.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 800
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 87.66
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz
F2 - Processing parameters
SI 16384
SF 100.6177851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.508
7.505
7.500
7.493
7.491
7.487
7.484
7.481
7.393
7.375
7.373
7.331
7.325
7.322
7.316
7.311
7.309
7.305
7.260
7.126
7.109
4.768
2.477
2.347
3n
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 671
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150929
Time 15.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 1
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 106.66
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz
F2 - Processing parameters
SI 16384
SF 400.1500090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.22
3.06
2.28
1.06
6.23
3.31



BRUKER

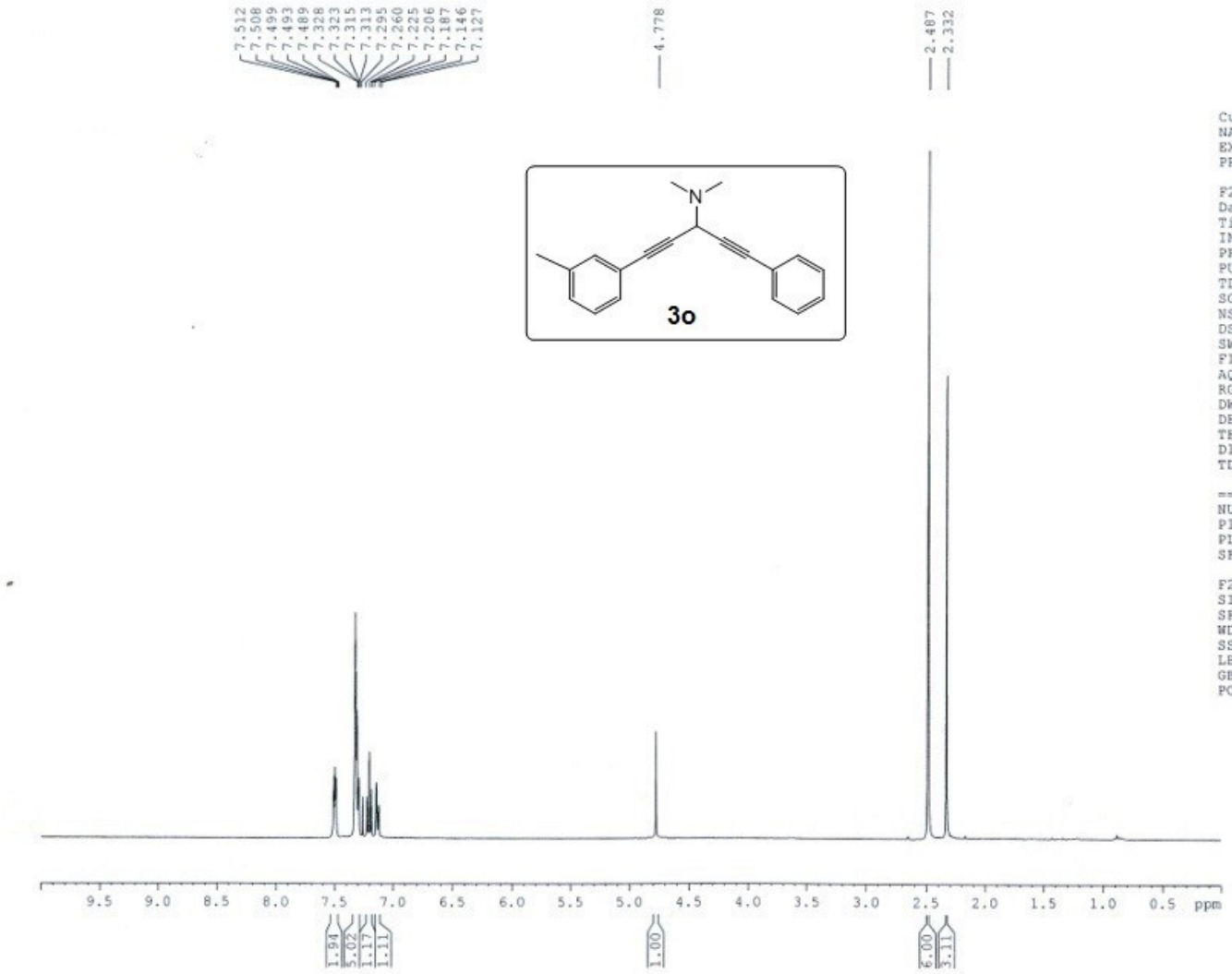
Current Data Parameters
NAME Dr. A HAJRA 2015
EXPNO 672
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150929
Time 16.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 106.66
DW 20.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W
SFO1 100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.40792999 W
PLW13 0.26107001 W
SFO2 400.1516006 MHz

F2 - Processing parameters
SI 16384
SF 100.6177835 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



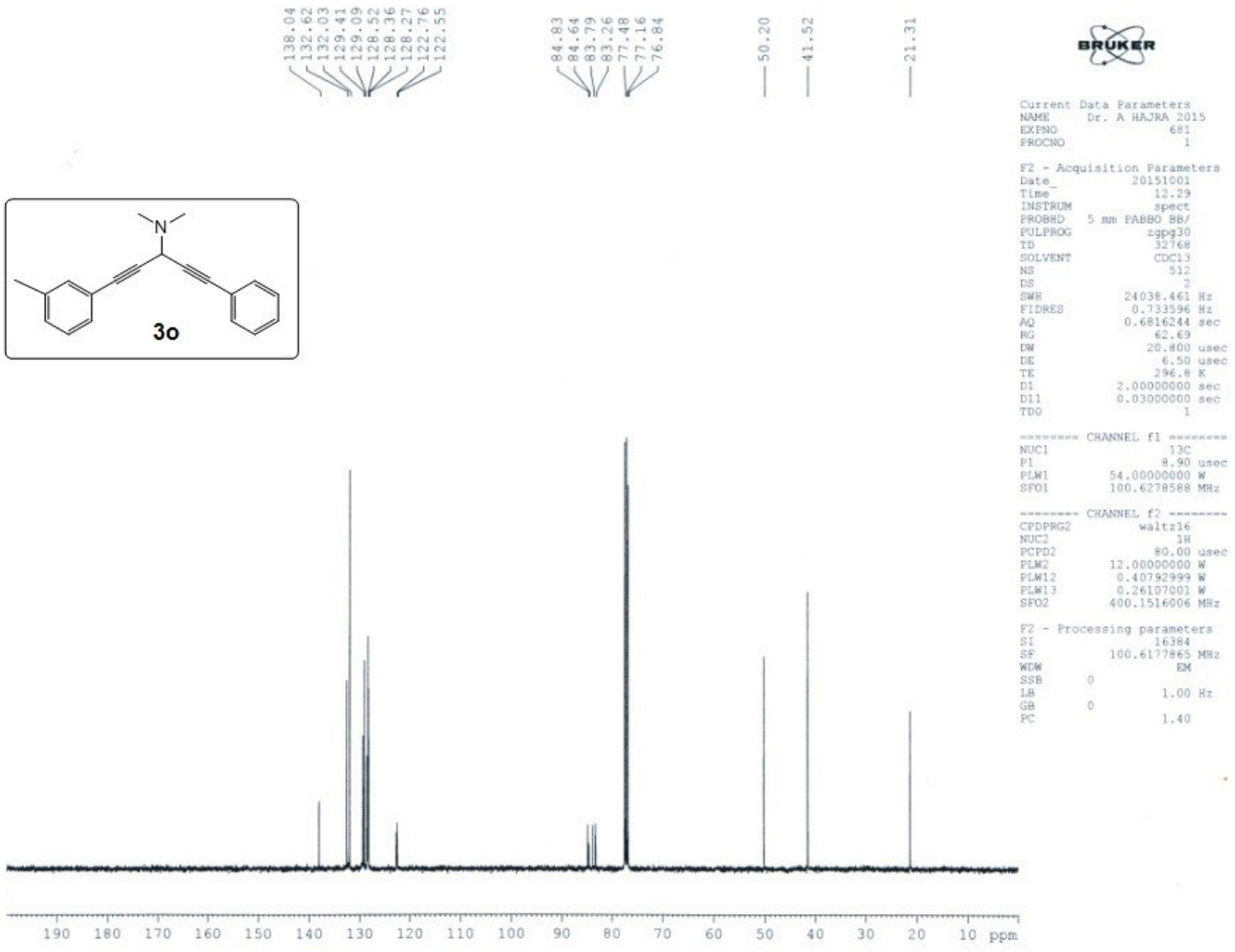
BRUKER

```
Current Data Parameters
NAME      Dr. A HAJRA 2015
EXPNO                  680
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151001
Time                 12.05
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     24
DS                      1
SWH               8223.685 Hz
FIDRES            0.250967 Hz
AQ                1.9923444 sec
RG                    62.69
DW                  60.800 usec
DE                   6.50 usec
TE                  295.5 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.75 usec
PLW1          11.99499989 W
SFO1          400.1524711 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500090 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



BRUKER

```
Current Data Parameters
NAME      Dr. A HAJRA 2015
EXPNO                  681
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151001
Time              12.29
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              32768
SOLVENT           CDCl3
NS                  512
DS                    2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6816244 sec
RG                62.69
DW                20.800 usec
DE                 6.50 usec
TE                296.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.90 usec
PLW1         54.00000000 W
SFO1        100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2         12.00000000 W
PLW12         0.40792999 W
PLW13         0.26107001 W
SFO2        400.1516006 MHz

F2 - Processing parameters
SI                  16384
SF          100.6177865 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```