SUPPORTING INFORMATION

for

Synthesis of diversely 1,3,5-substituted pyrazoles via 5-*exo*-*dig* cyclization

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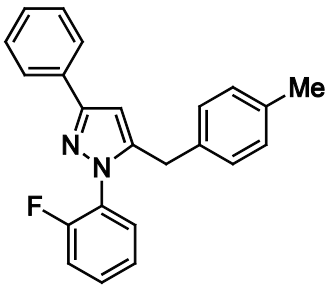
Experimental Section

General Methods.

Microwave-accelerated reactions were carried out using a Discover Benchmate (CEM) reactor. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance III 400 spectrometer. HRMS were recorded on Agilent 6520 Q-TOF LCMS instrument. The chemical shifts are reported in δ (ppm) values (^{1}H and ^{13}C relative to chloroform-d_6 (7.26 ppm, 77.16 ppm respectively). Preparative TLC was carried out on silica gel plates (Analtech, Silica Gel G, 2000 μ).

Representative Procedure for Synthesis of Pyrazoles: 1-(2-Fluorophenyl)-5-(4-methylbenzyl)-3-phenyl-1*H*-pyrazole (3e).



A 50 mL round-bottom flask was charged with 1-phenyl-4-(p-tolyl)but-3-yn-1-one (0.058 g, 0.25 mmol), 4-(2-fluorophenyl)hydrazine (0.047 g, 0.37 mmol), and dichloromethane (1 mL). The mixture was manually swirled at ambient temperature (22°C) until the ketone and hydrazine completelly dissolved in the solution. To the solution, the K-10 catalyst (0.25 g) was added and the mixture was agitated for about 5 min on a vortex mixer. The heterogenous mixture was transferred to a microwave vial and the solvent was removed under reduced pressure using rotary evaporator. The dry solid was microwaved without stirring for 10 min at 100°C. The solid was washed with dichloromethane (3 × 8 mL). The filtrate was concentrated on a rotary evaporator and dried on oil pump vacuum. Preparative TLC (hexane/ethyl acetate, usually 80:20 or 60:40) gave analytically pure sample of **3e** (0.070 g, 0.21 mmol, 82%) as a solid. MS (EI, m/z): 342 (100%, M^+). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{23}H_{19}FN_2$343.1611, found 343.1678**.** NMR ($CDCl_3$, δ): ^{1}H 7.88-7.81 (AA'XX', J = 7.0 Hz, 2H),[S1] 7.50-7.37 (m, 4H), 7.35-7.31 (m, 1H), 7.28 (s, 1H), 7.09 (AB, J = 7.9 Hz, 2H), 7.02 (AB, J = 8.0, 2H), 6.44 (s, 1H), 3.89 (s, 2H), 2.34 (s, 3H); ^{13}C ($CDCl_3$) 157.3 (d, J = 251.8 Hz), 152.5, 146.1, 136.4, 134.7, 133.2, 130.7 (d, J = 7.9 Hz), 129.8, 129.3(2C), 128.8(2C), 128.7(2C), 128.0, 127.8 (d, J = 12.1 Hz), 125.9(2C), 124.9 (d, J = 4.0 Hz), 116.7 (d, J = 19.9 Hz), 103.9, 31.9 (d, J = 3.2 Hz), 21.2.

References

S1) Six peaks exhibiting a splitting pattern of pseudo triplet of doublet were observed. The estimated values were determined as $\nu_A = ½ (\delta_2 + \delta_5)$, $J_{AX} \approx \delta_2 - \delta_5$.

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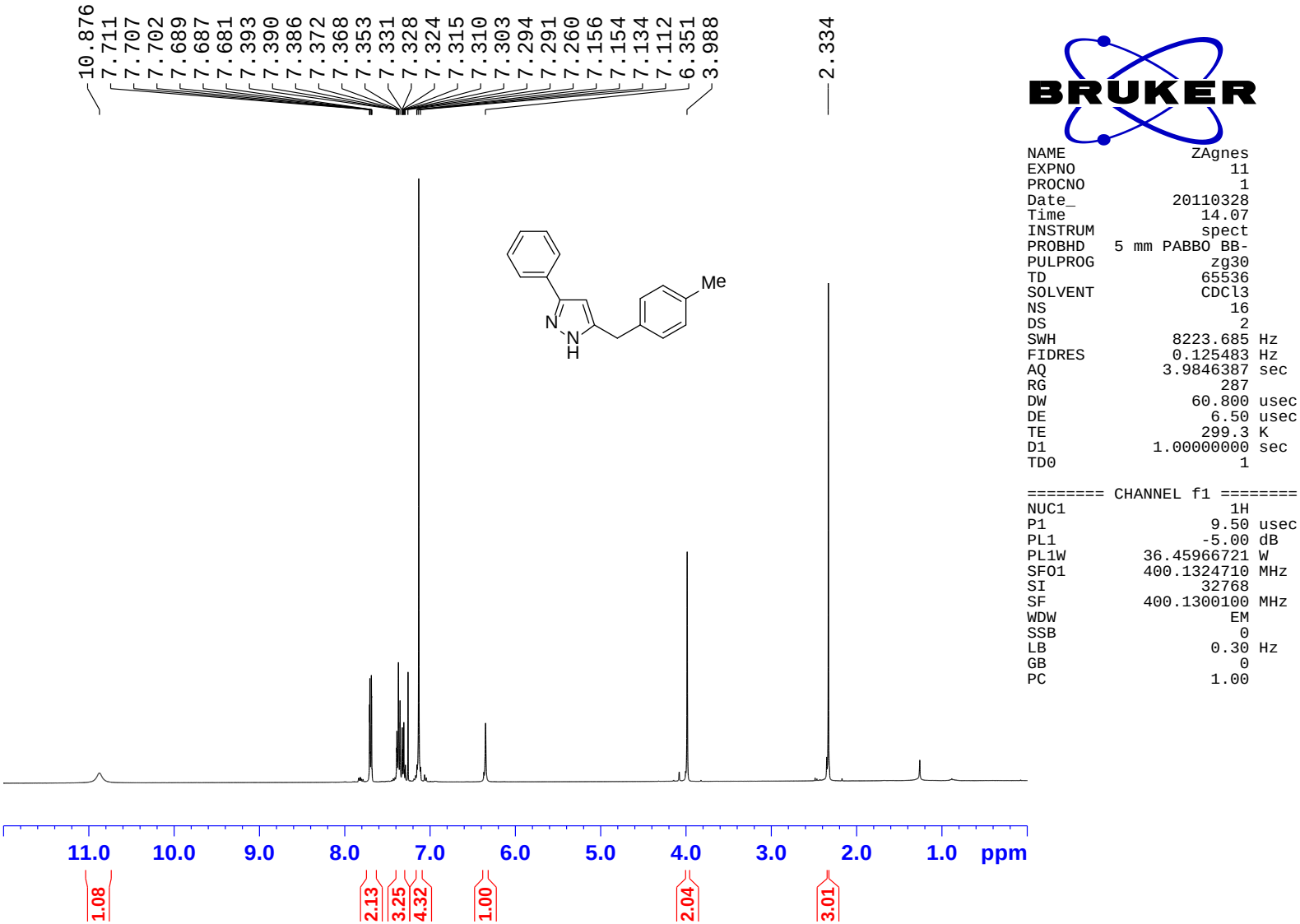
^{1}H NMR spectrum for **3a** ($CDCl_3$)

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FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     287
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DE                   6.50 usec
TE                  299.3 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                 -5.00 dB
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SFO1          400.1324710 MHz
SI                   32768
SF            400.1300100 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                1.00
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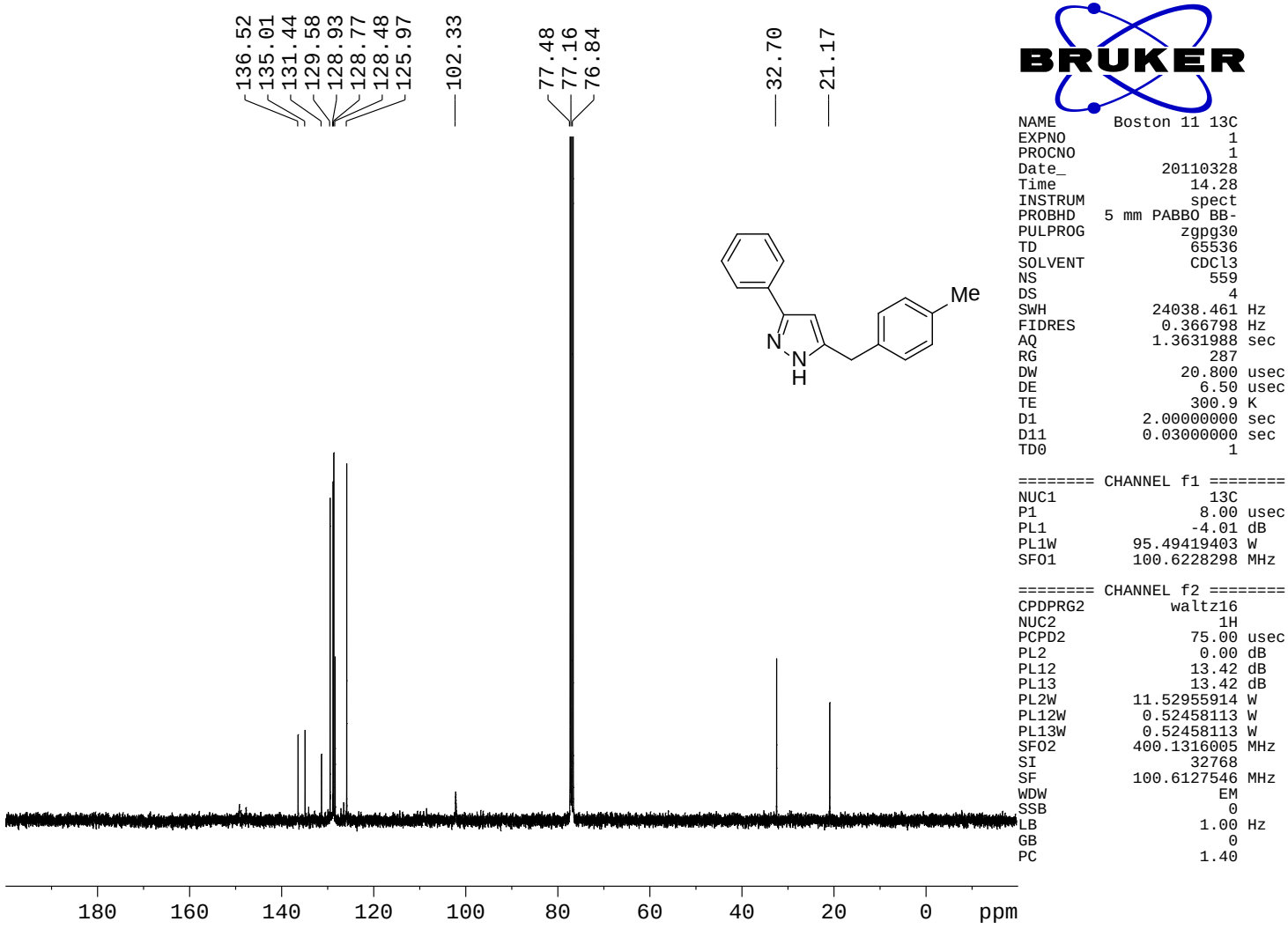
^{13}C NMR spectrum for **3a** ($CDCl_3$)

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DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     287
DW                  20.800 usec
DE                   6.50 usec
TE                  300.9 K
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D11              0.03000000 sec
TD0                      1

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SFO1          100.6228298 MHz

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PL13                 13.42 dB
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PL12W           0.52458113 W
PL13W           0.52458113 W
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WDW                    EM
SSB                     0
LB                   1.00 Hz
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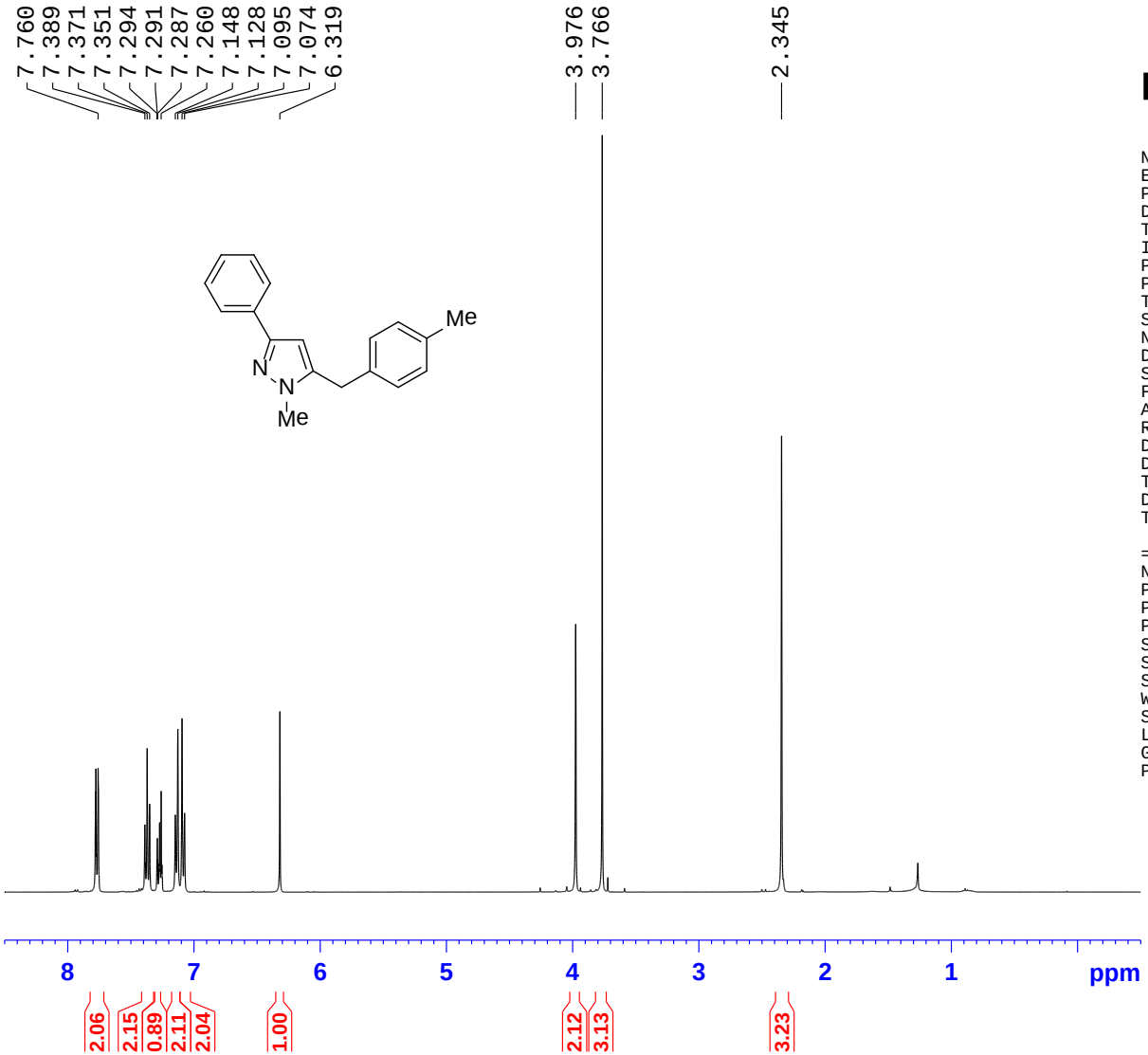
^{1}H NMR spectrum for **3b** ($CDCl_3$)

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TE                  299.0 K
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TD0                      1

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SI                   32768
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SSB                      0
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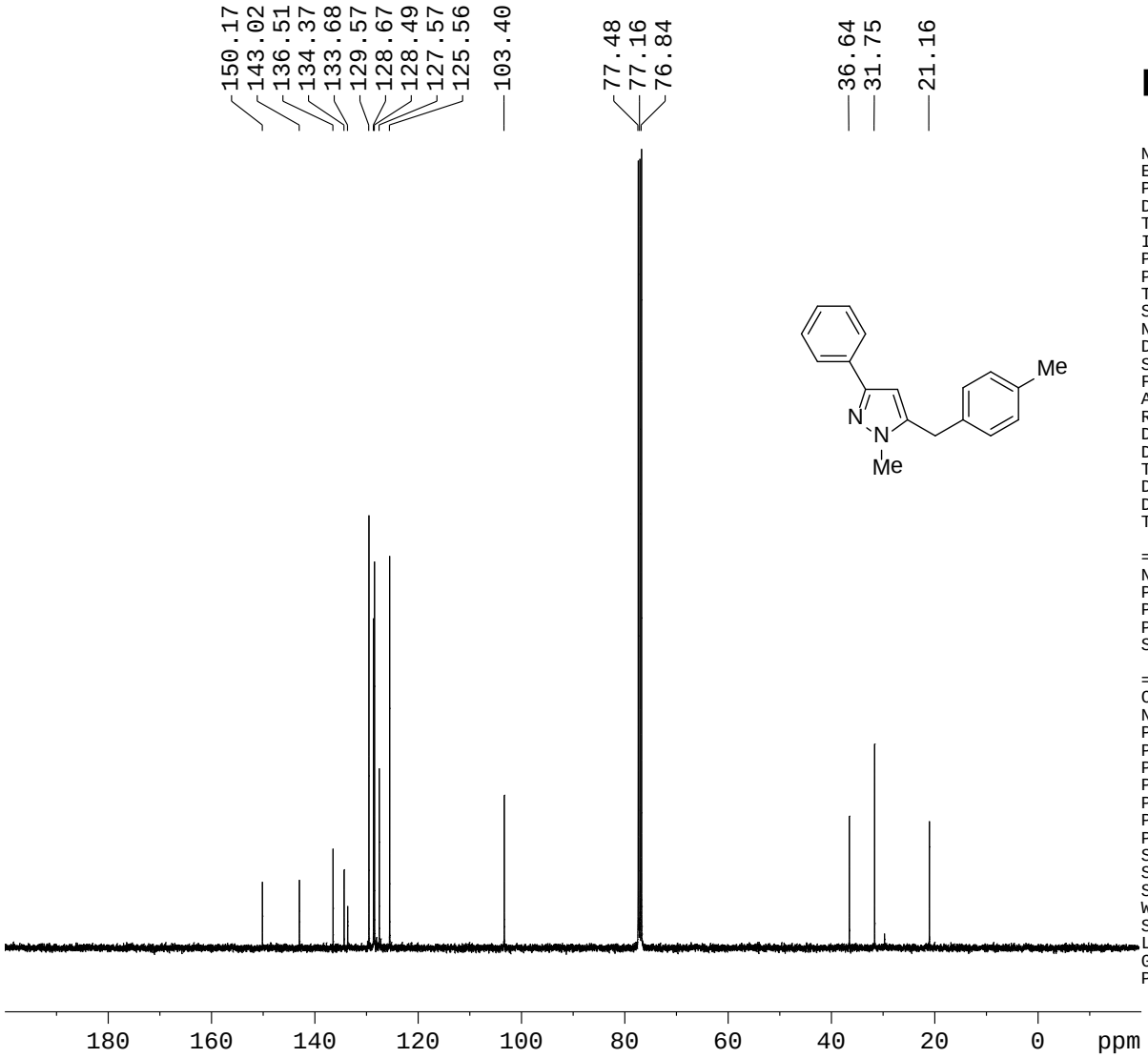
^{13}C NMR spectrum for **3b** ($CDCl_3$)

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RG                  287
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TE                300.4 K
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D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
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PL1               -4.01 dB
PL1W        95.49419403 W
SFO1       100.6228298 MHz

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PL2                 0.00 dB
PL12               13.42 dB
PL13               13.42 dB
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PL12W        0.52458113 W
PL13W        0.52458113 W
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SI                 32768
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SSB                    0
LB                  1.00 Hz
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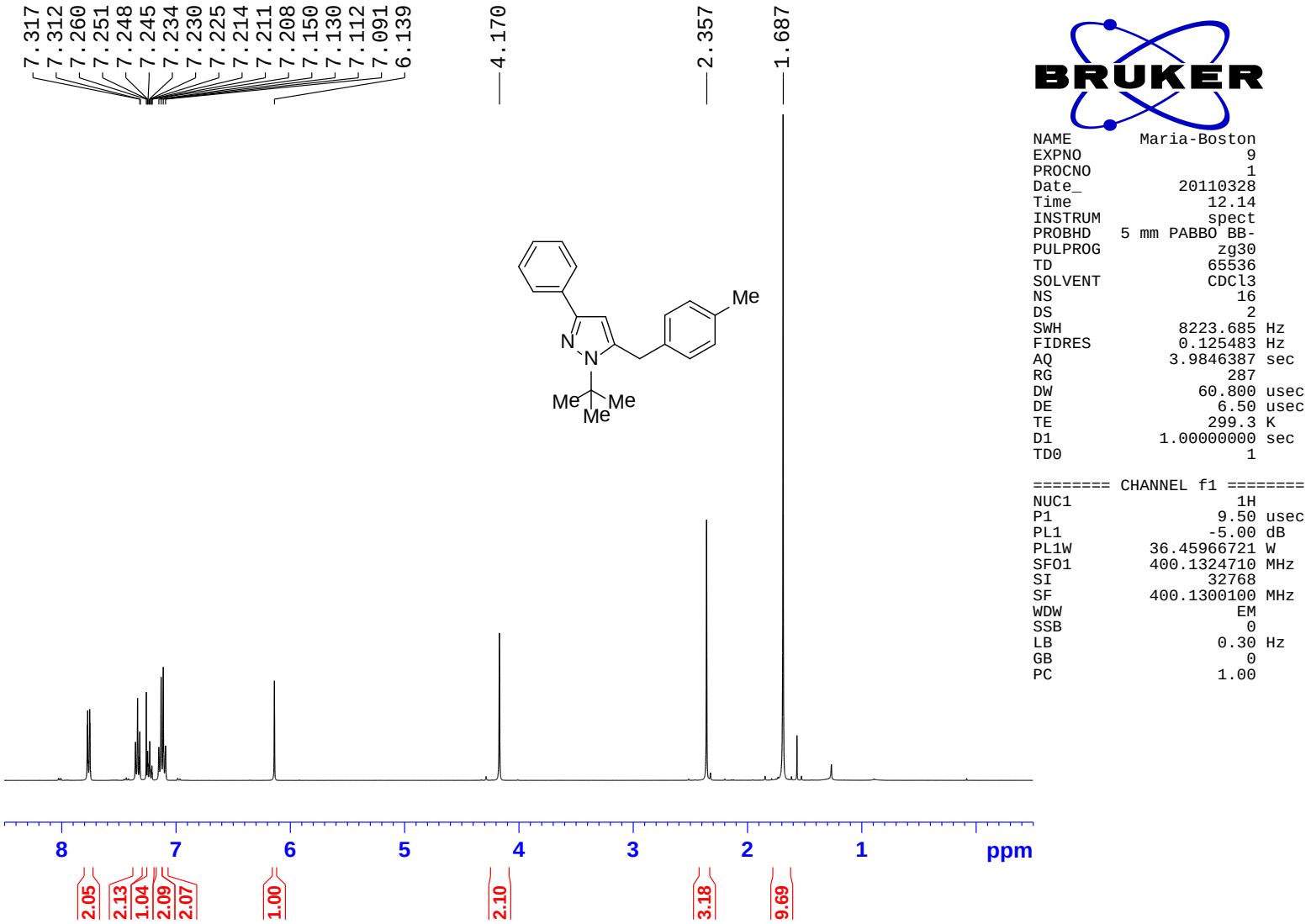
^{1}H NMR spectrum for **3c** ($CDCl_3$)

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TD                   65536
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DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
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RG                     287
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DE                   6.50 usec
TE                  299.3 K
D1              1.00000000 sec
TD0                      1

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SI                   32768
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WDW                    EM
SSB                     0
LB                   0.30 Hz
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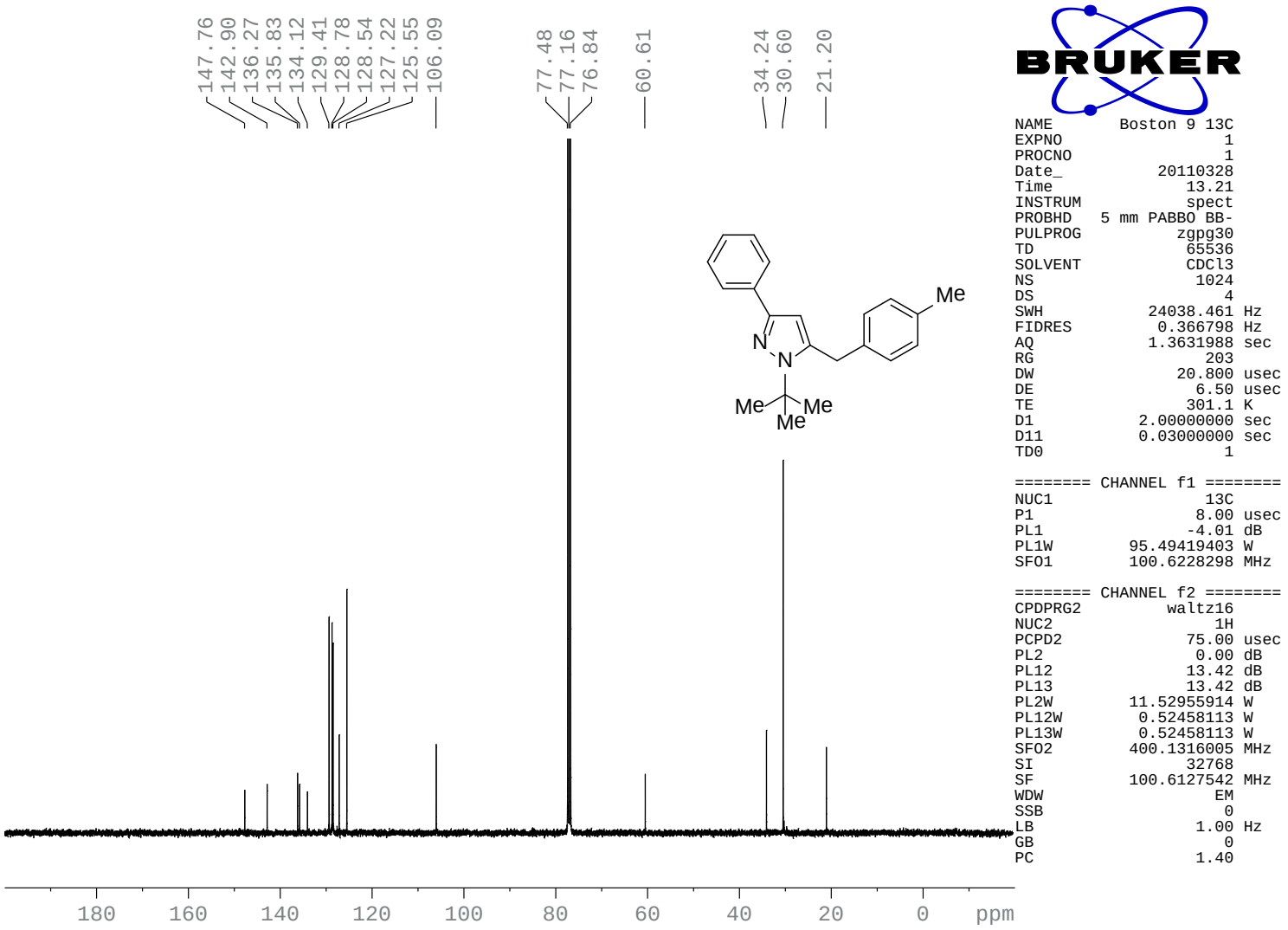
^{13}C NMR spectrum for **3c** ($CDCl_3$)

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SFO1           100.6228298 MHz

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PL12W           0.52458113 W
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SSB                     0
LB                   1.00 Hz
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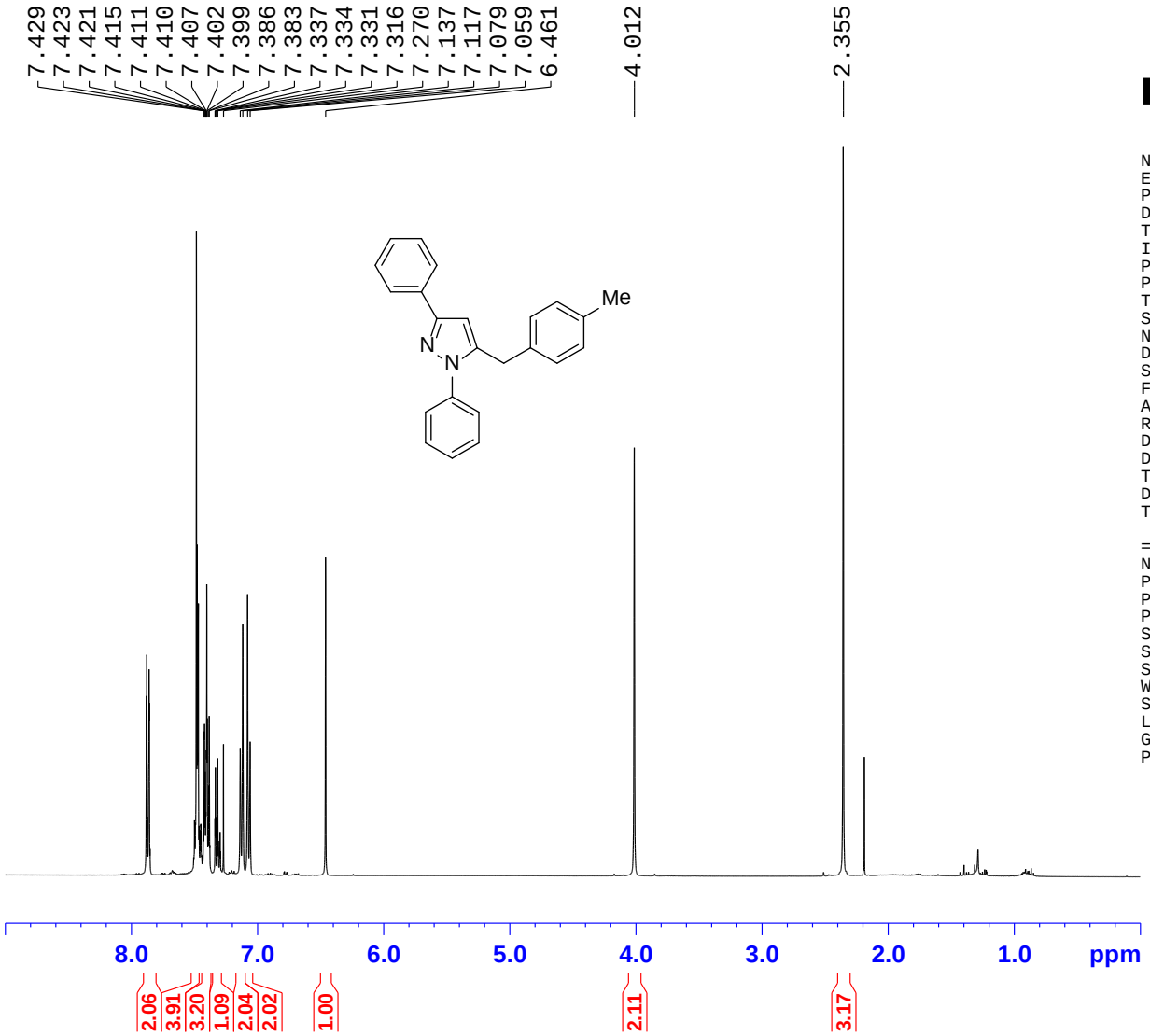
^{1}H NMR spectrum for **3d** ($CDCl_3$)

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RG                       144
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TE                    297.9 K
D1                 1.00000000 sec
TD0                       1

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SI                    32768
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SSB                       0
LB                    0.30 Hz
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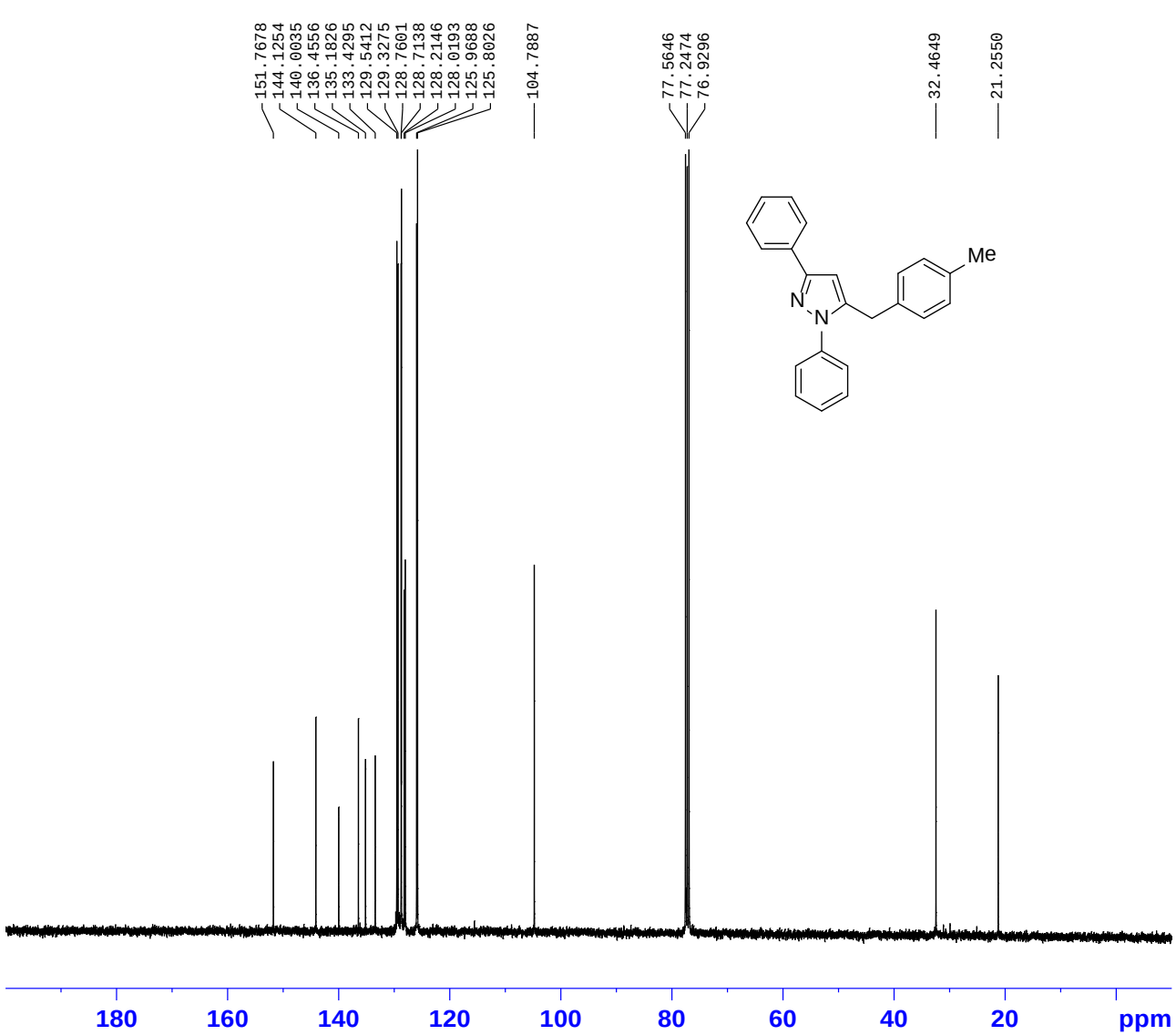
^{13}C NMR spectrum for **3d** ($CDCl_3$)

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DE                 6.50 usec
TE                299.9 K
D1            2.50000000 sec
D11           0.03000000 sec
TD0                      1

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PL1                  -4.01 dB
PL1W         95.49419403 W
SFO1         100.6228298 MHz

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PL13                 13.42 dB
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PL13W         0.52458113 W
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SI                   32768
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SSB                     0
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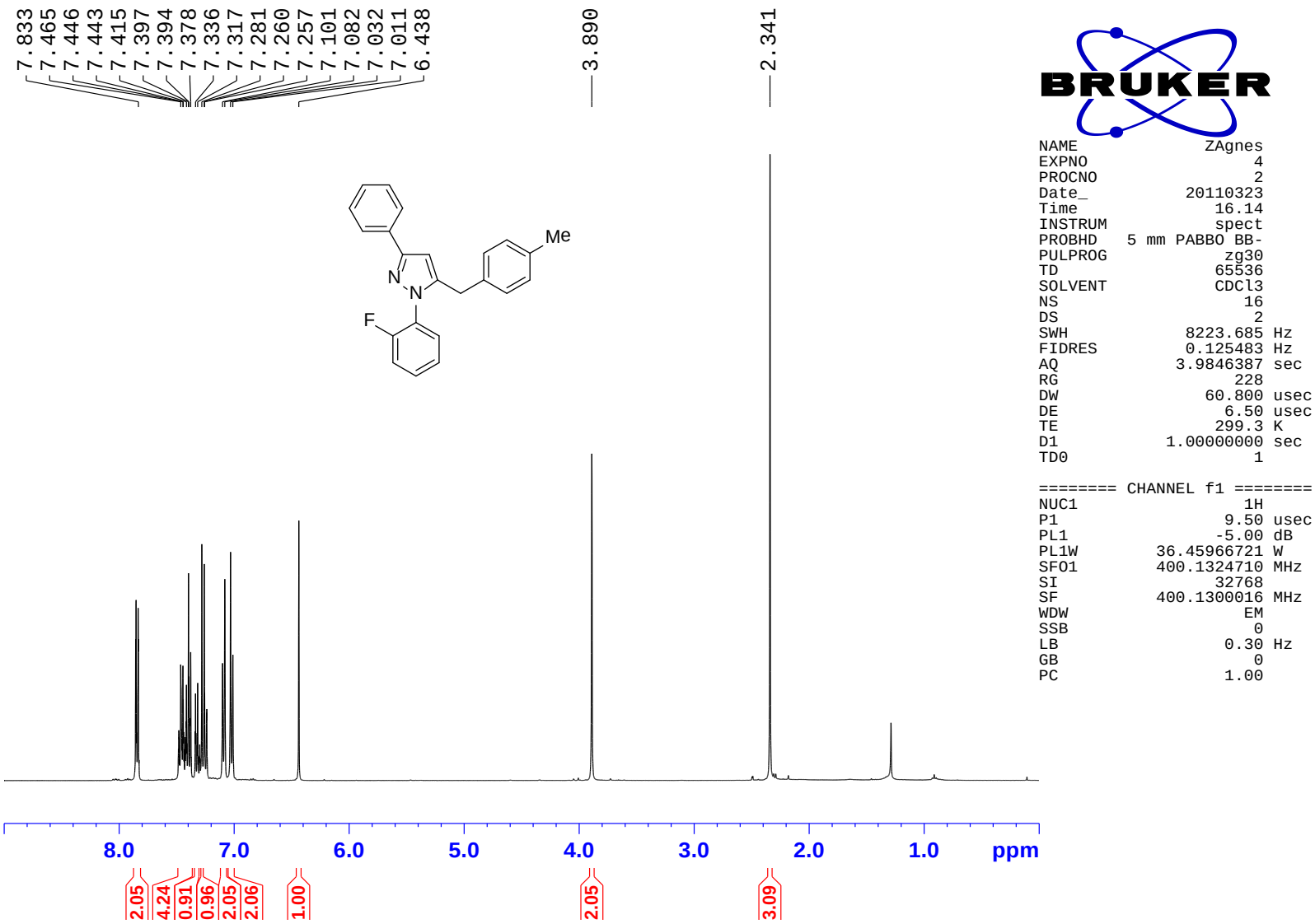
^{1}H NMR spectrum for **3e** ($CDCl_3$)

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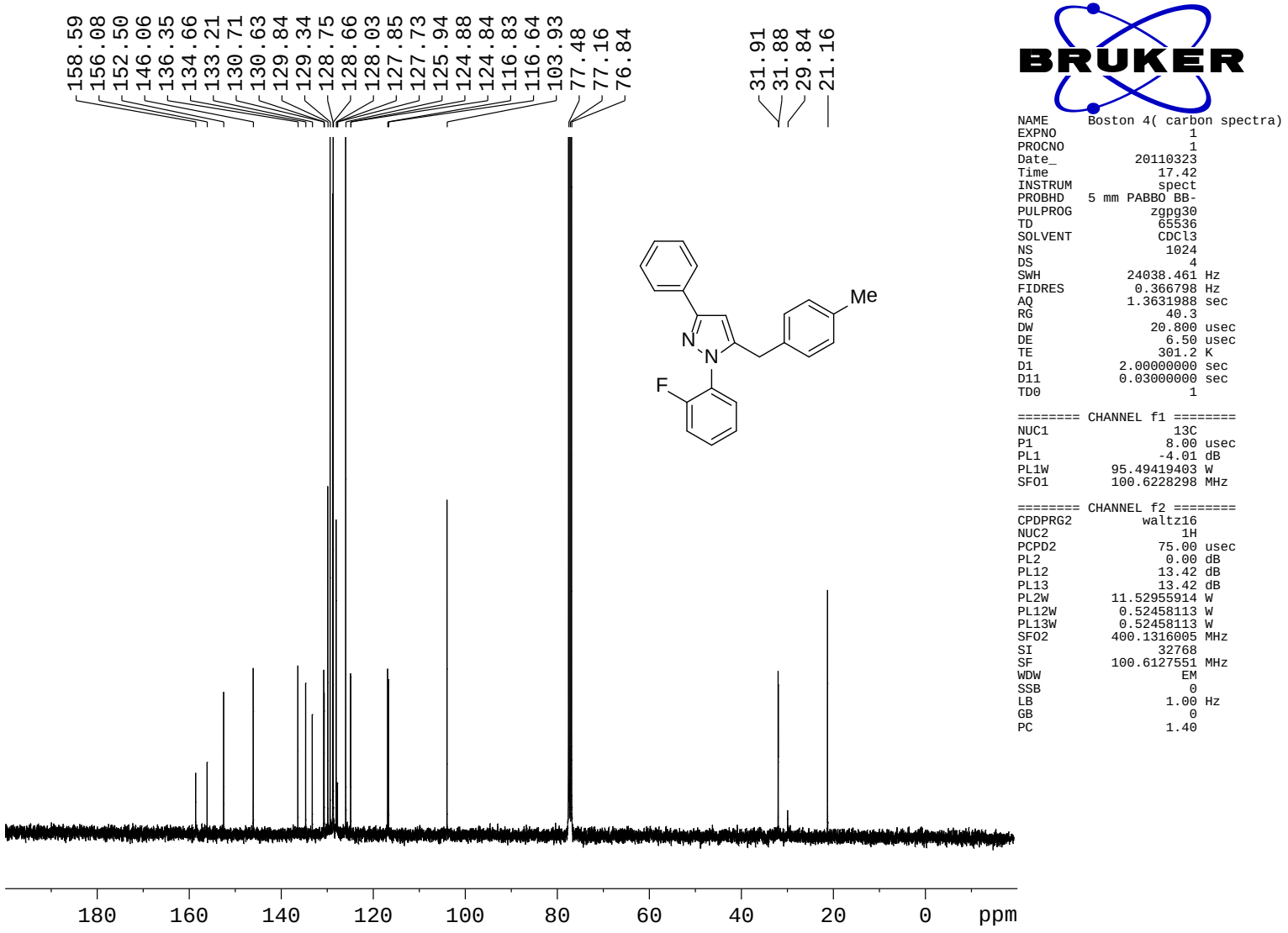
^{13}C NMR spectrum for **3e** ($CDCl_3$)

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PL13                13.42 dB
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PL13W        0.52458113 W
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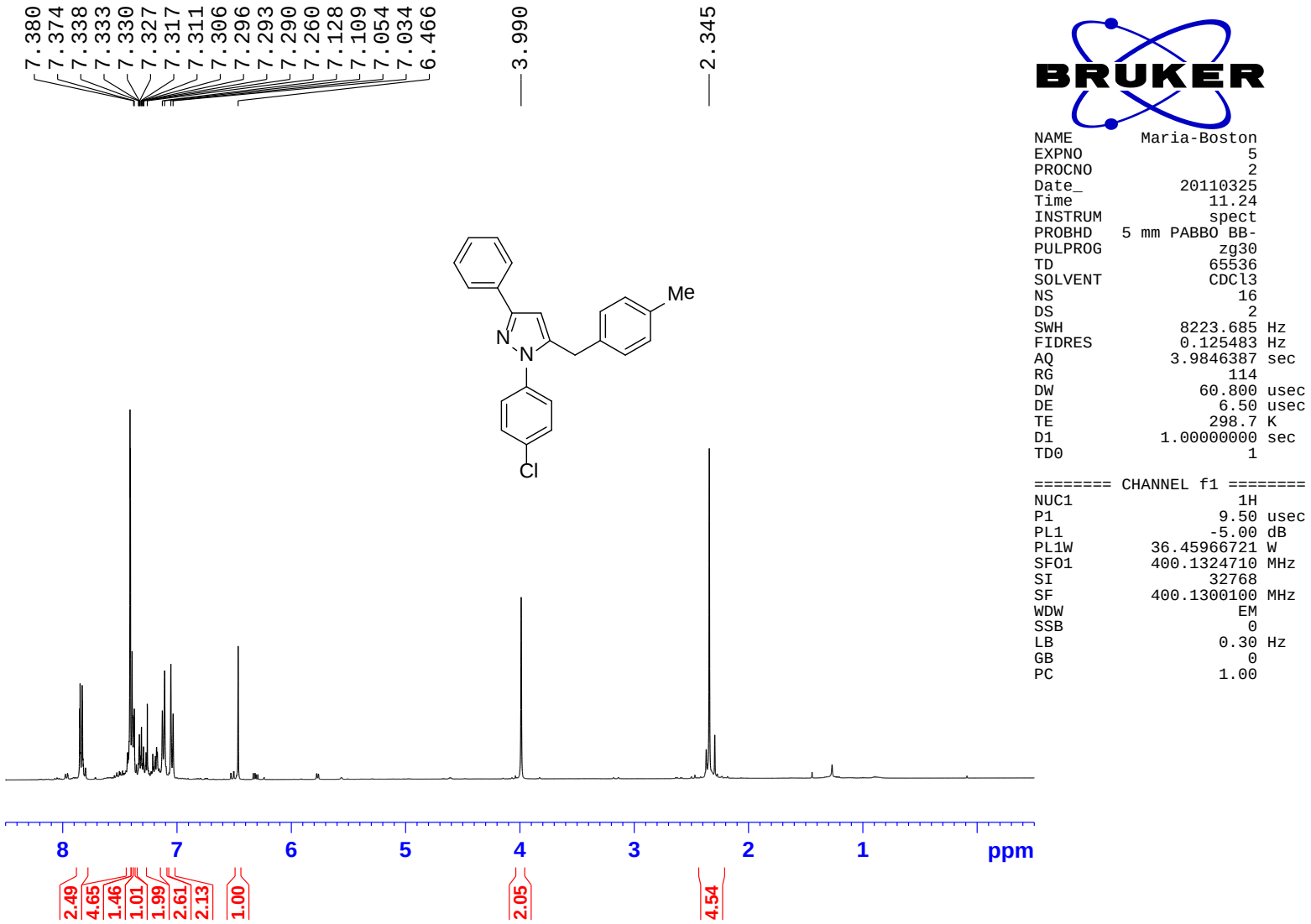
^{1}H NMR spectrum for **3f** ($CDCl_3$)

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RG                     114
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TE                  298.7 K
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SI                   32768
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WDW                    EM
SSB                     0
LB                   0.30 Hz
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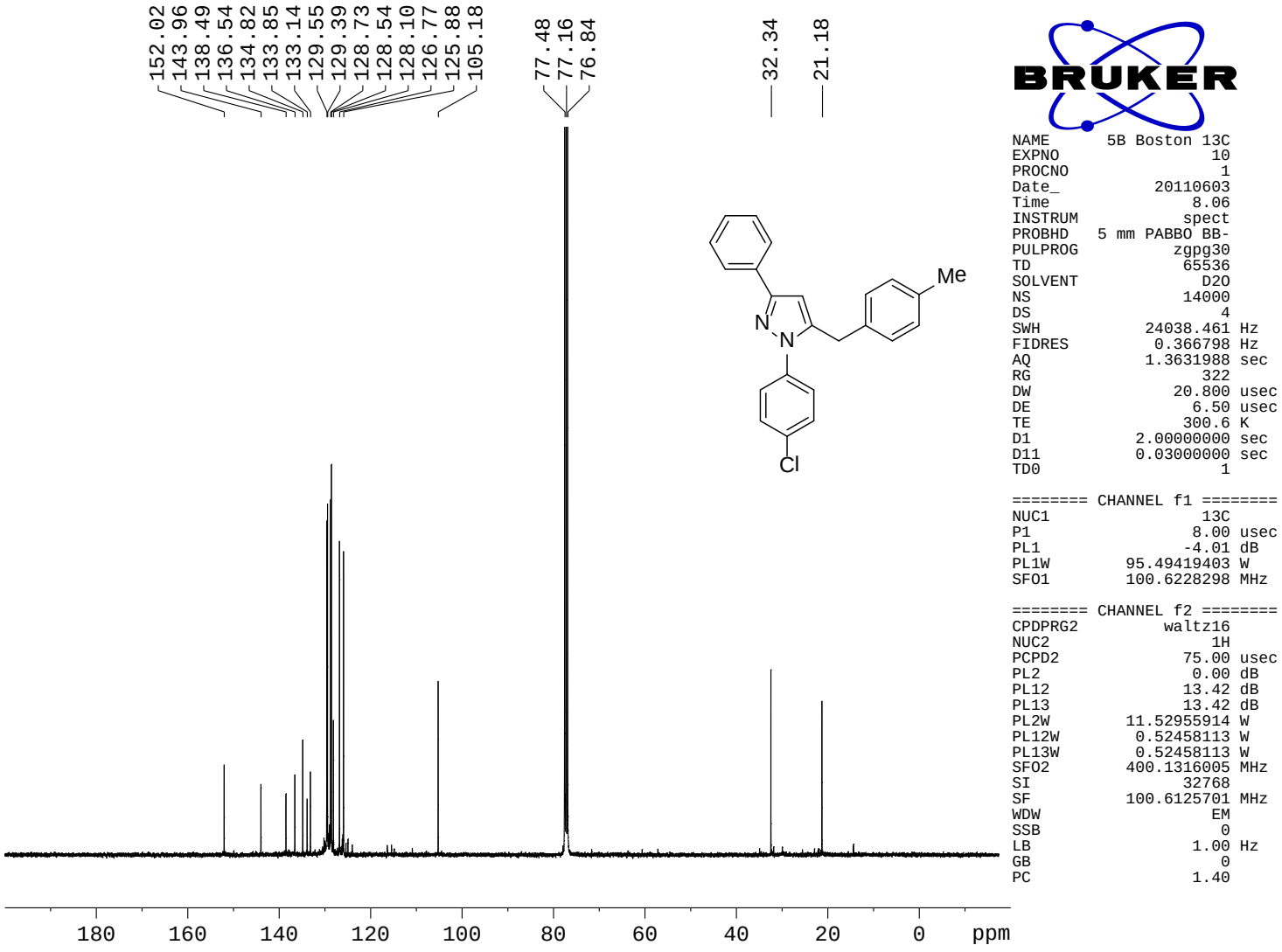
^{13}C NMR spectrum for **3f** ($CDCl_3$)

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FIDRES            0.366798 Hz
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RG                      322
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DE                    6.50 usec
TE                   300.6 K
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D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
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P1                    8.00 usec
PL1                  -4.01 dB
PL1W          95.49419403 W
SFO1         100.6228298 MHz

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NUC2                   1H
PCPD2                75.00 usec
PL2                   0.00 dB
PL12                 13.42 dB
PL13                 13.42 dB
PL2W          11.52955914 W
PL12W          0.52458113 W
PL13W          0.52458113 W
SFO2         400.1316005 MHz
SI                    32768
SF           100.6125701 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
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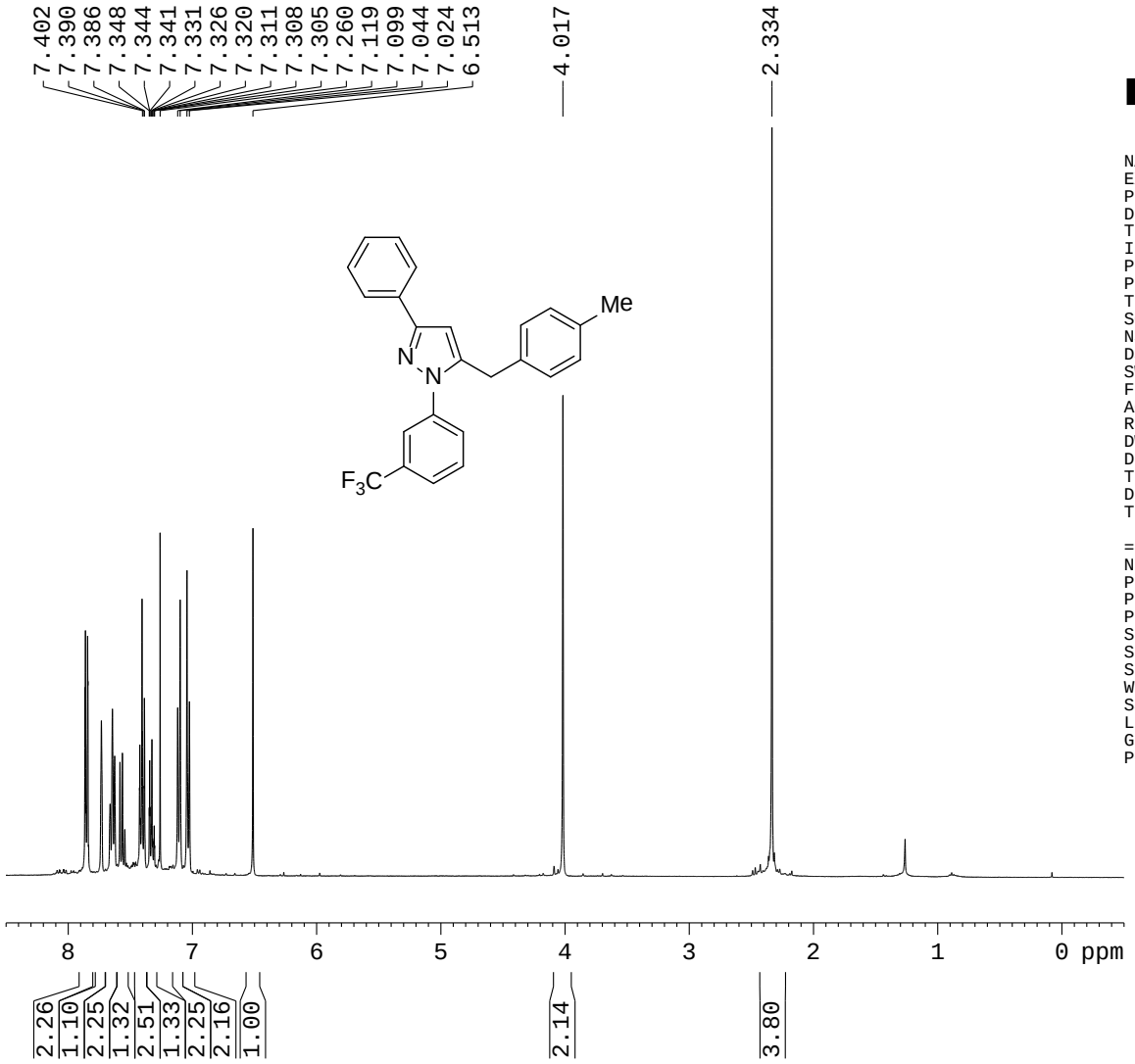
^{1}H NMR spectrum for **3g** ($CDCl_3$)

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FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     322
DW                  60.800 usec
DE                   6.50 usec
TE                  298.9 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                 -5.00 dB
PL1W           36.45966721 W
SFO1           400.1324710 MHz
SI                   32768
SF             400.1300100 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
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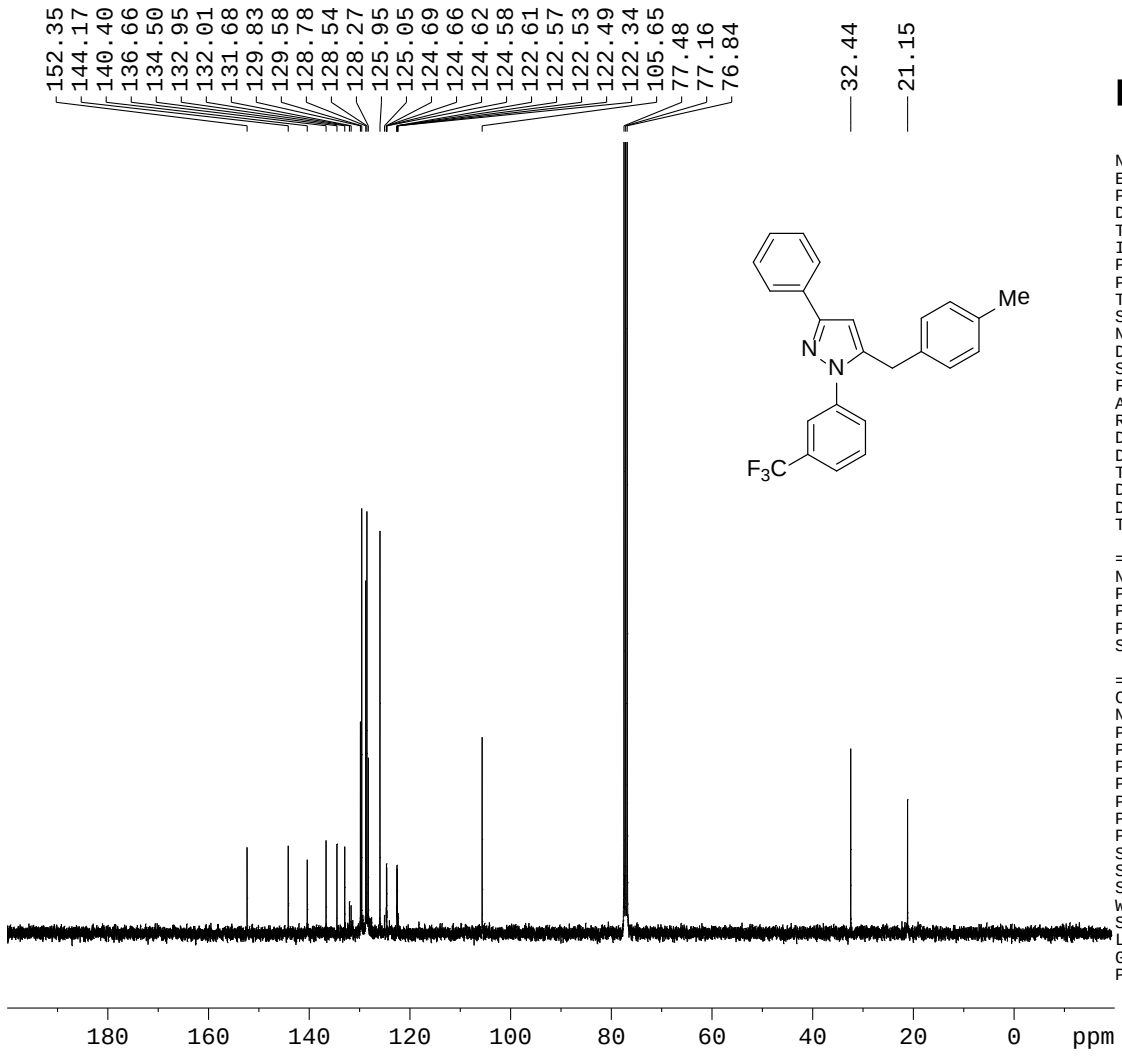
Organic & Biomolecular Chemistry **2012**, *10*, doi: 10.1039/c2ob25580d

^{13}C NMR spectrum for **3g** ($CDCl_3$)

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PROCNO                1
Date_          20110324
Time              16.07
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 40.3
DW               20.800 usec
DE                6.50 usec
TE               300.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.00 usec
PL1                -4.01 dB
PL1W        95.49419403 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              75.00 usec
PL2                 0.00 dB
PL12               13.42 dB
PL13               13.42 dB
PL2W        11.52955914 W
PL12W        0.52458113 W
PL13W        0.52458113 W
SFO2       400.1316005 MHz
SI               32768
SF         100.6127539 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

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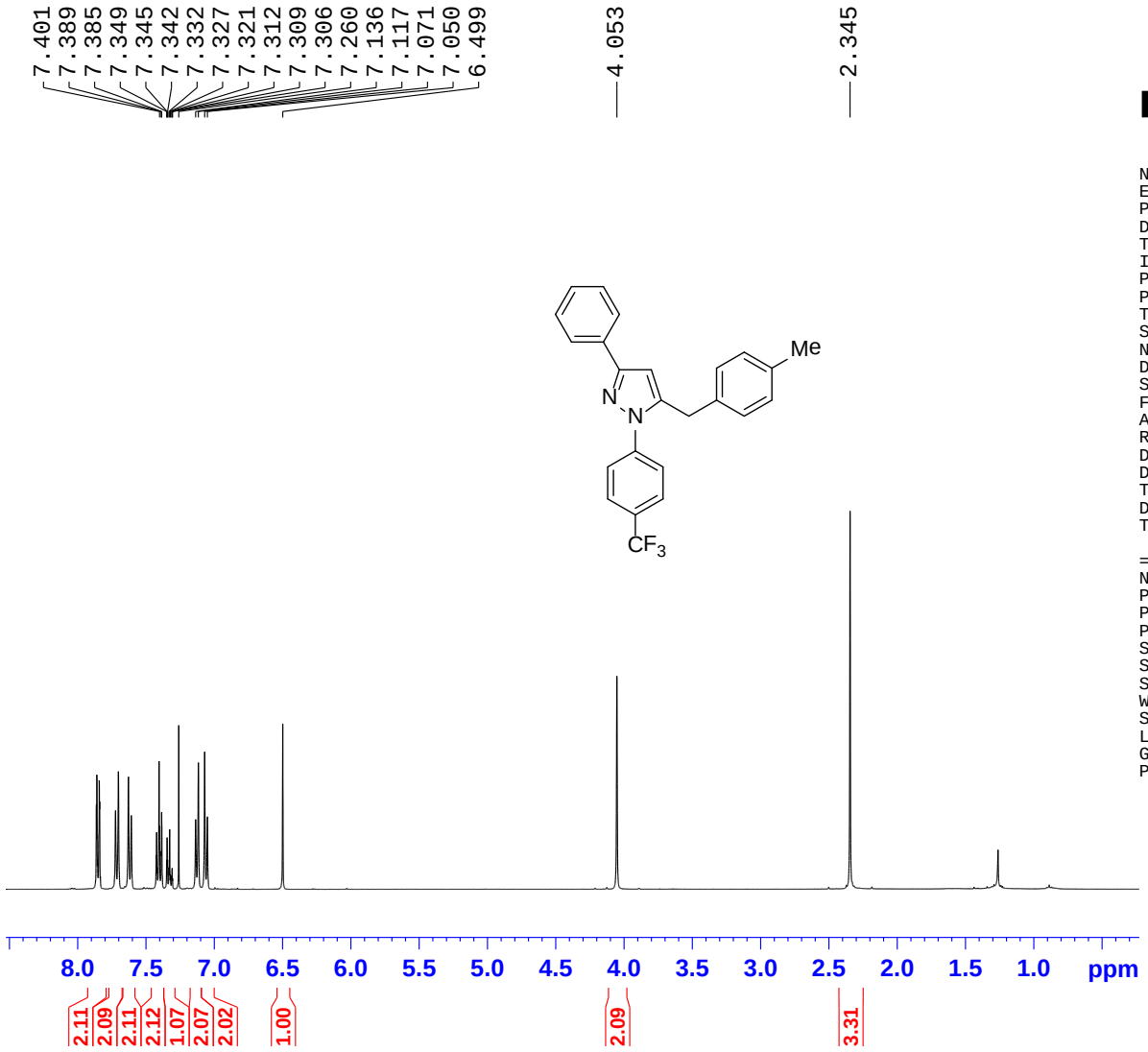
^{1}H NMR spectrum for **3h** ($CDCl_3$)




```
NAME          Maria-Boston
EXPNO                    3
PROCNO                   2
Date_             20110325
Time                 12.42
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       2
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                  298.8 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                 -5.00 dB
PL1W          36.45966721 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300100 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

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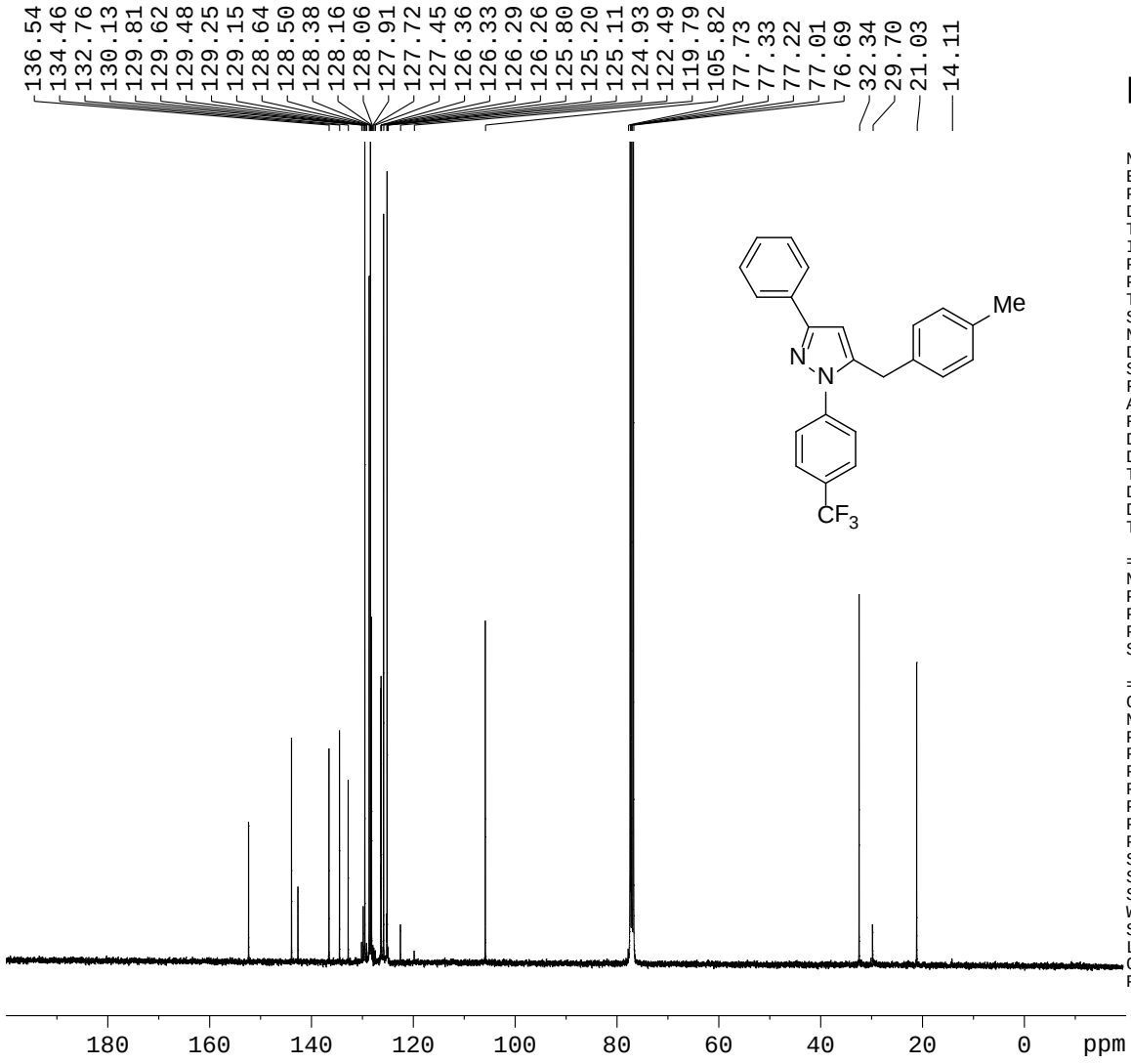
^{13}C NMR spectrum for **3h** ($CDCl_3$)




```
NAME      Boston 3 (14000SC)
EXPNO                  2
PROCNO                 1
Date_           20110504
Time               9.21
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                14000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  287
DW                20.800 usec
DE                 6.50 usec
TE                300.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.00 usec
PL1                -4.01 dB
PL1W        95.49419403 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              75.00 usec
PL2                 0.00 dB
PL12               13.42 dB
PL13               13.42 dB
PL2W        11.52955914 W
PL12W        0.52458113 W
PL13W        0.52458113 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

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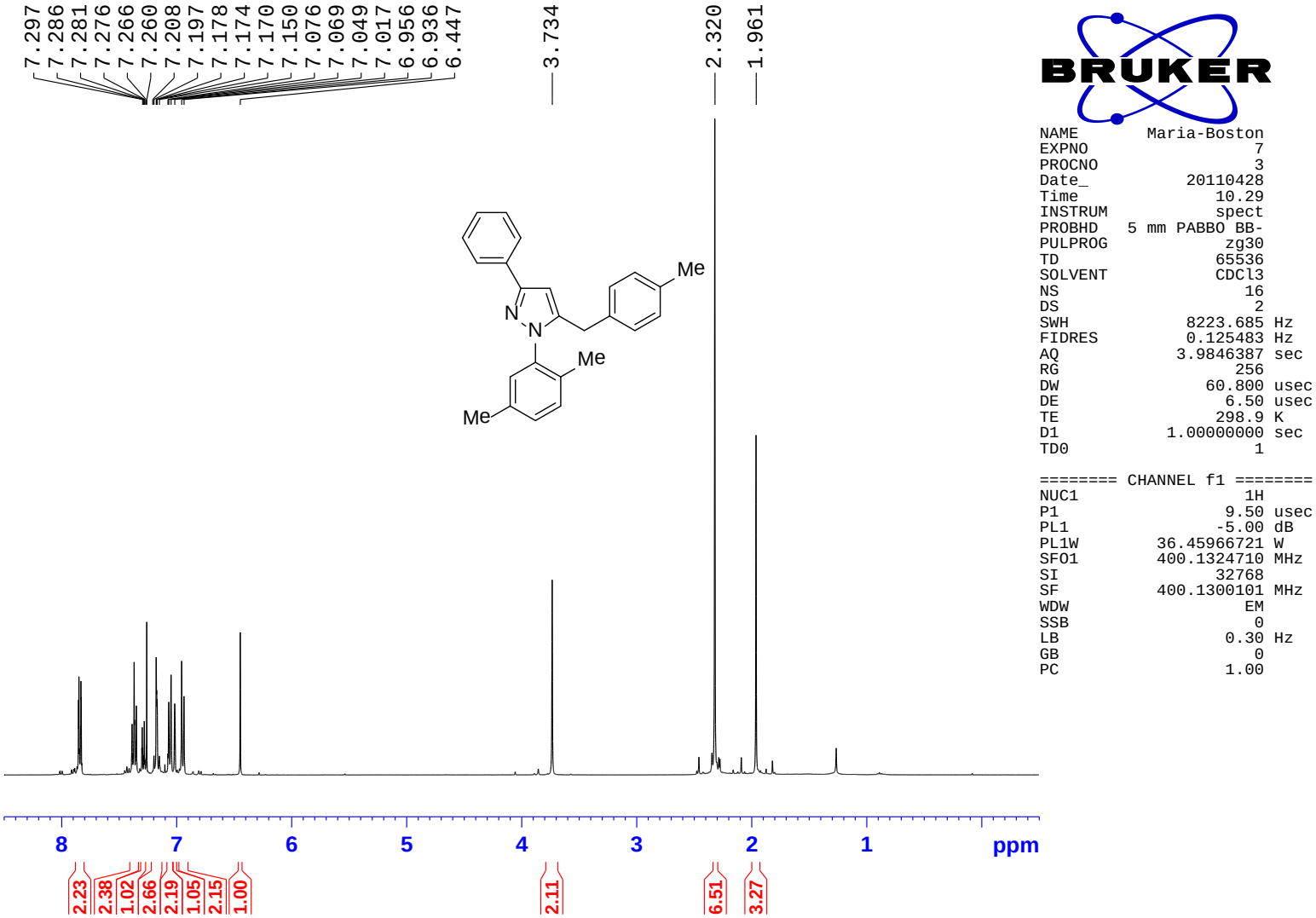
^{1}H NMR spectrum for **3i** ($CDCl_3$)




```
NAME          Maria-Boston
EXPNO                    7
PROCNO                   3
Date_             20110428
Time                 10.29
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                     256
DW                  60.800 usec
DE                   6.50 usec
TE                  298.9 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.50 usec
PL1                  -5.00 dB
PL1W           36.45966721 W
SFO1           400.1324710 MHz
SI                   32768
SF             400.1300101 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

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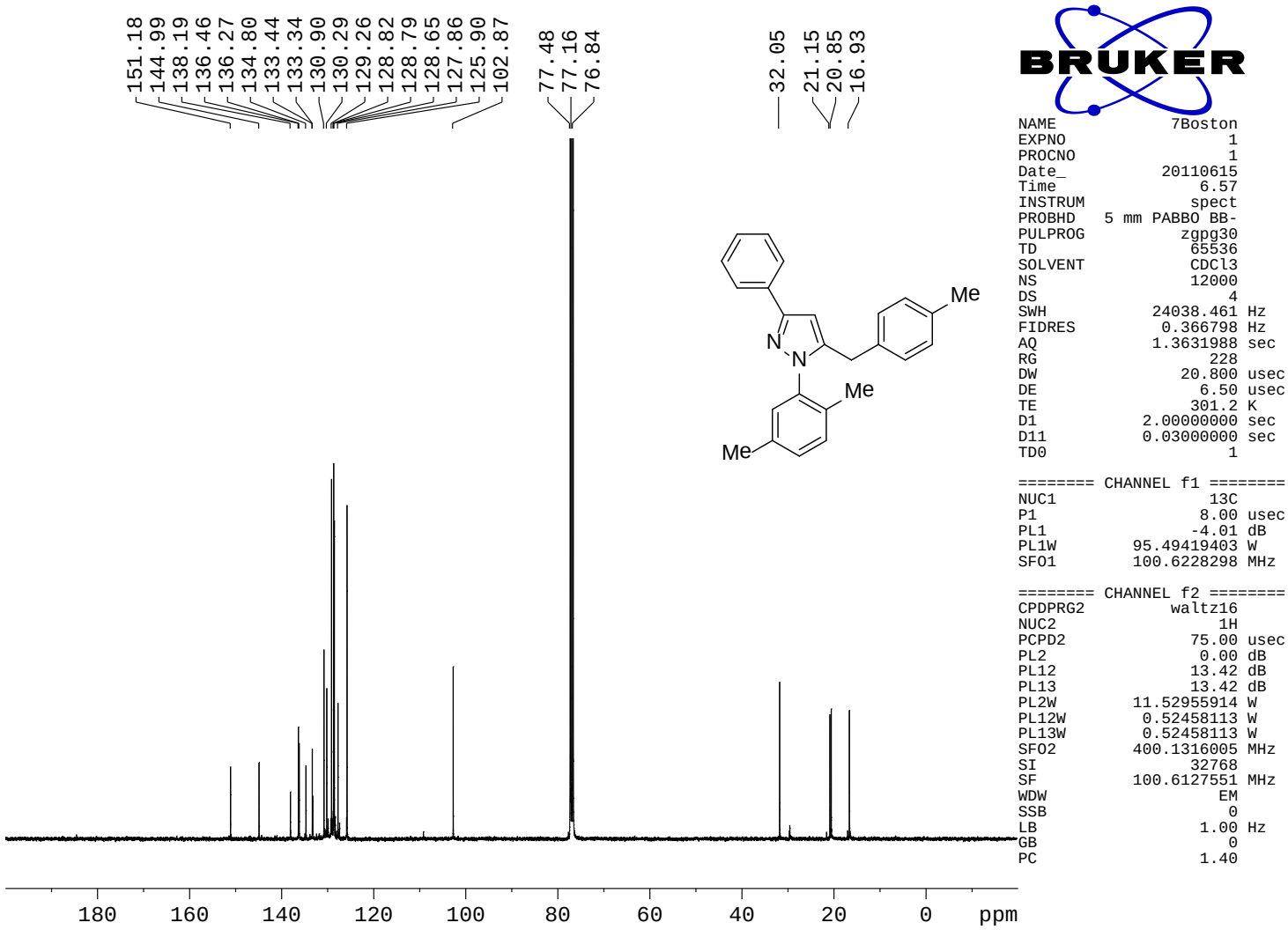
^{13}C NMR spectrum for **3i** ($CDCl_3$)




```
NAME                 7Boston
EXPNO                      1
PROCNO                     1
Date_               20110615
Time                    6.57
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                      12000
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                        228
DW                     20.800 usec
DE                      6.50 usec
TE                     301.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     8.00 usec
PL1                   -4.01 dB
PL1W              95.49419403 W
SFO1             100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  75.00 usec
PL2                     0.00 dB
PL12                   13.42 dB
PL13                   13.42 dB
PL2W              11.52955914 W
PL12W              0.52458113 W
PL13W              0.52458113 W
SFO2             400.1316005 MHz
SI                      32768
SF               100.6127551 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

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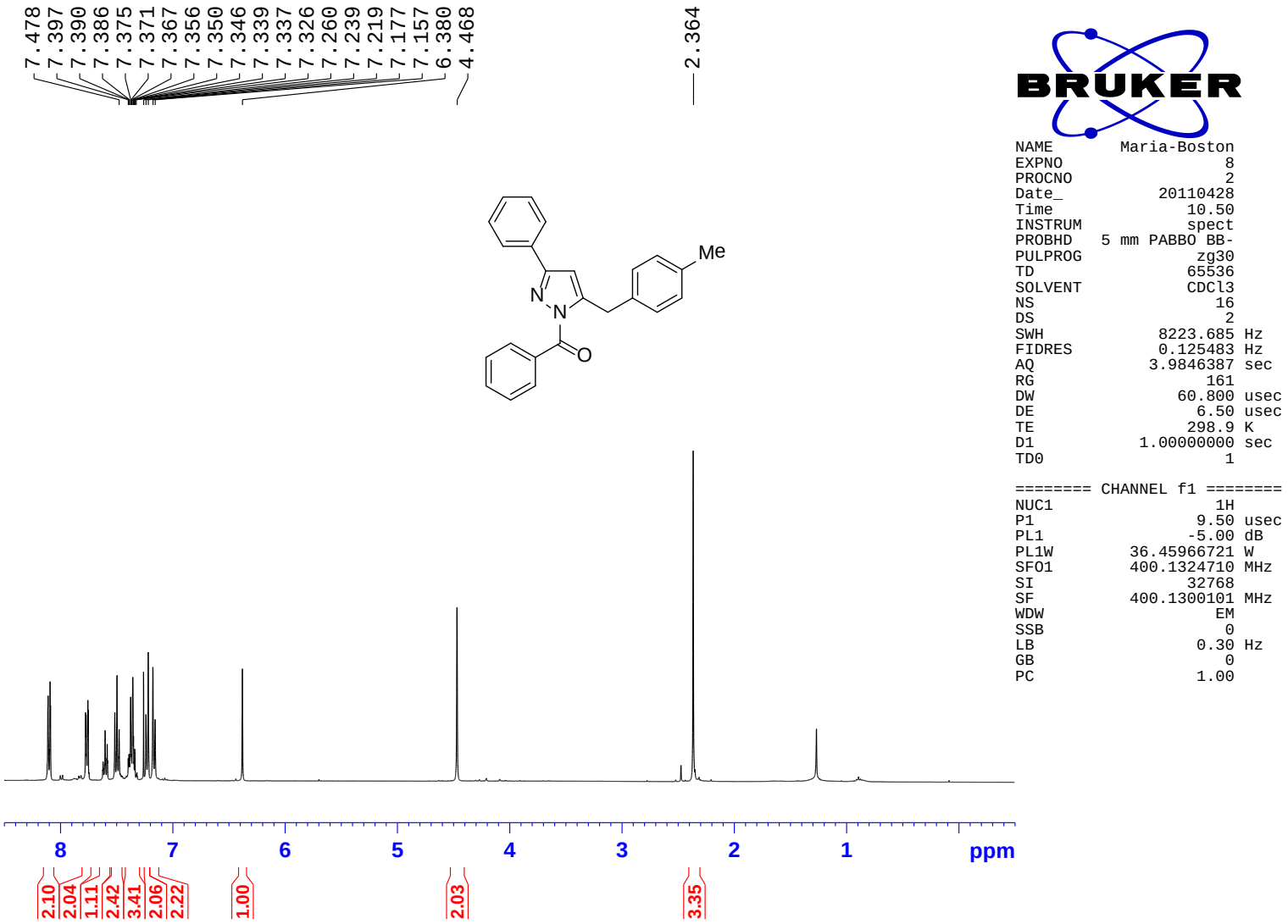
^{1}H NMR spectrum for **3j** ($CDCl_3$)




```
NAME          Maria-Boston
EXPNO                    8
PROCNO                   2
Date_             20110428
Time                 10.50
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                  298.9 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                 -5.00 dB
PL1W           36.45966721 W
SFO1           400.1324710 MHz
SI                   32768
SF             400.1300101 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

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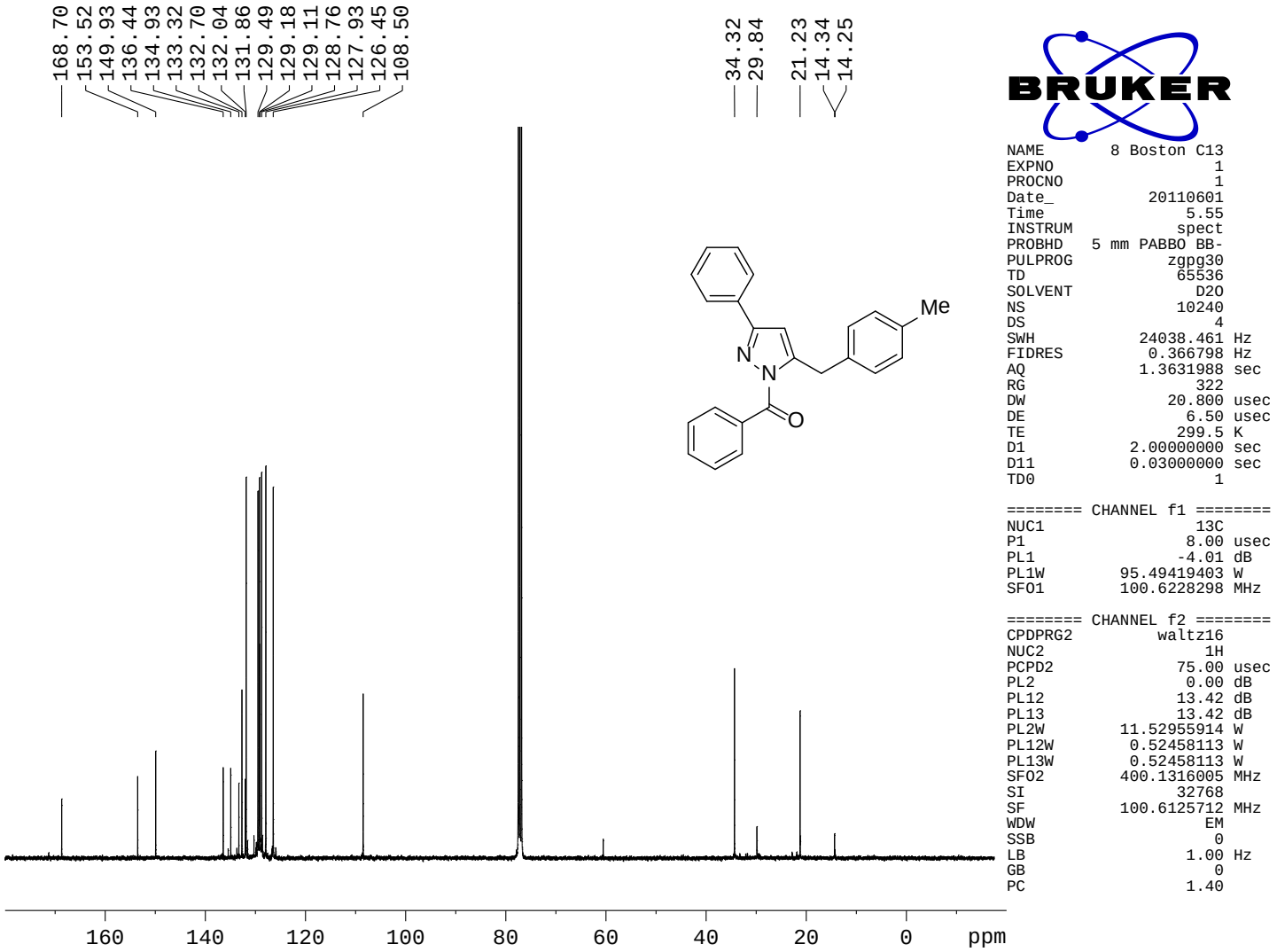
^{13}C NMR spectrum for **3j** ($CDCl_3$)




```
NAME          8 Boston C13
EXPNO                    1
PROCNO                   1
Date_             20110601
Time                  5.55
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                D2O
NS                   10240
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     322
DW                  20.800 usec
DE                   6.50 usec
TE                  299.5 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.00 usec
PL1                  -4.01 dB
PL1W           95.49419403 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                75.00 usec
PL2                   0.00 dB
PL12                 13.42 dB
PL13                 13.42 dB
PL2W           11.52955914 W
PL12W           0.52458113 W
PL13W           0.52458113 W
SFO2           400.1316005 MHz
SI                   32768
SF             100.6125712 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

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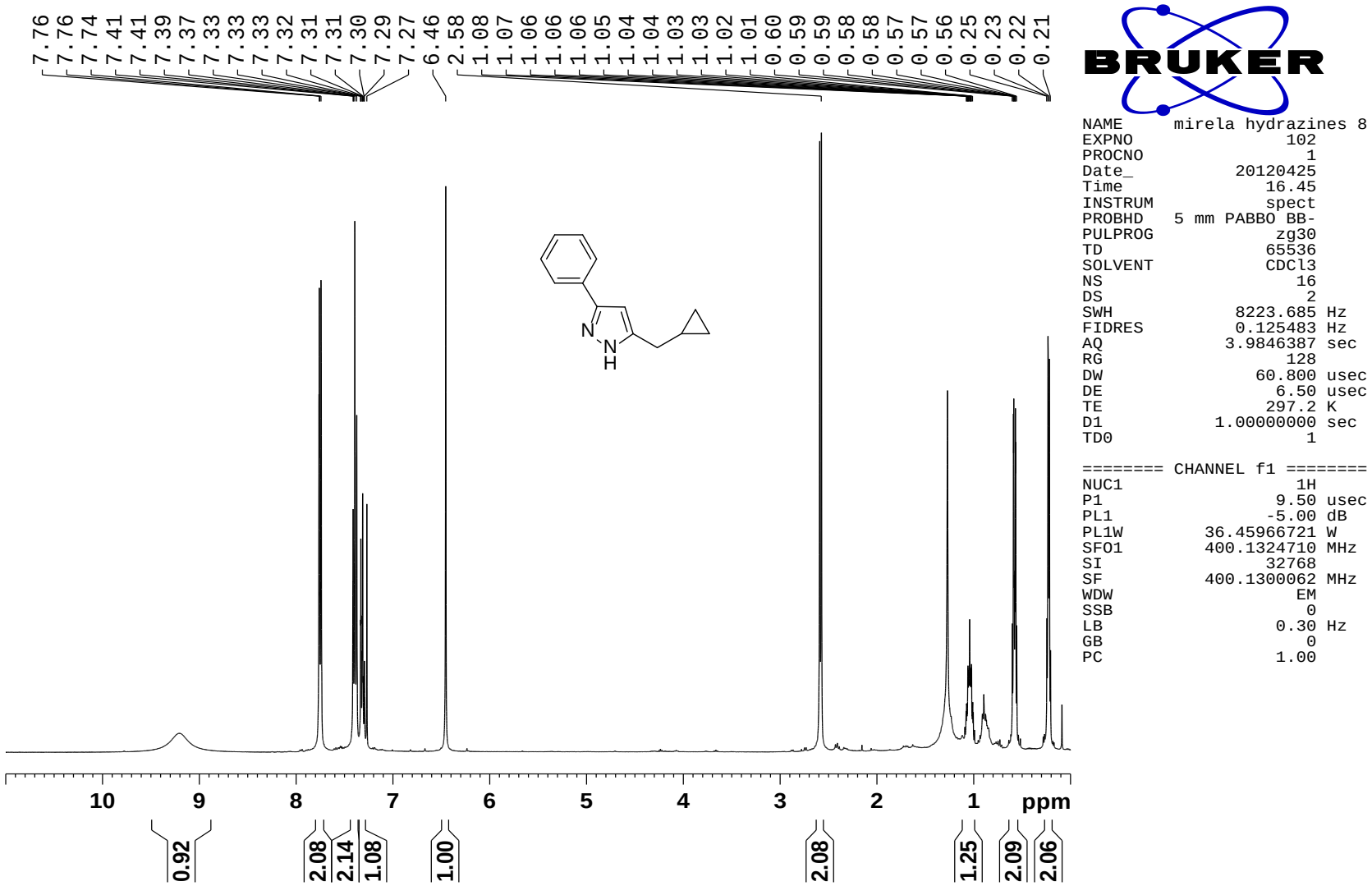
^{1}H NMR spectrum for **5-(cyclopropylmethyl)-3-phenyl-1*H*-pyrazole** ($CDCl_3$)




```
NAME      mirela hydrazines 8
EXPNO                 102
PROCNO                  1
Date_          20120425
Time              16.45
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                  128
DW                60.800 usec
DE                 6.50 usec
TE                297.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.50 usec
PL1                -5.00 dB
PL1W       36.45966721 W
SFO1       400.1324710 MHz
SI               32768
SF         400.1300062 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

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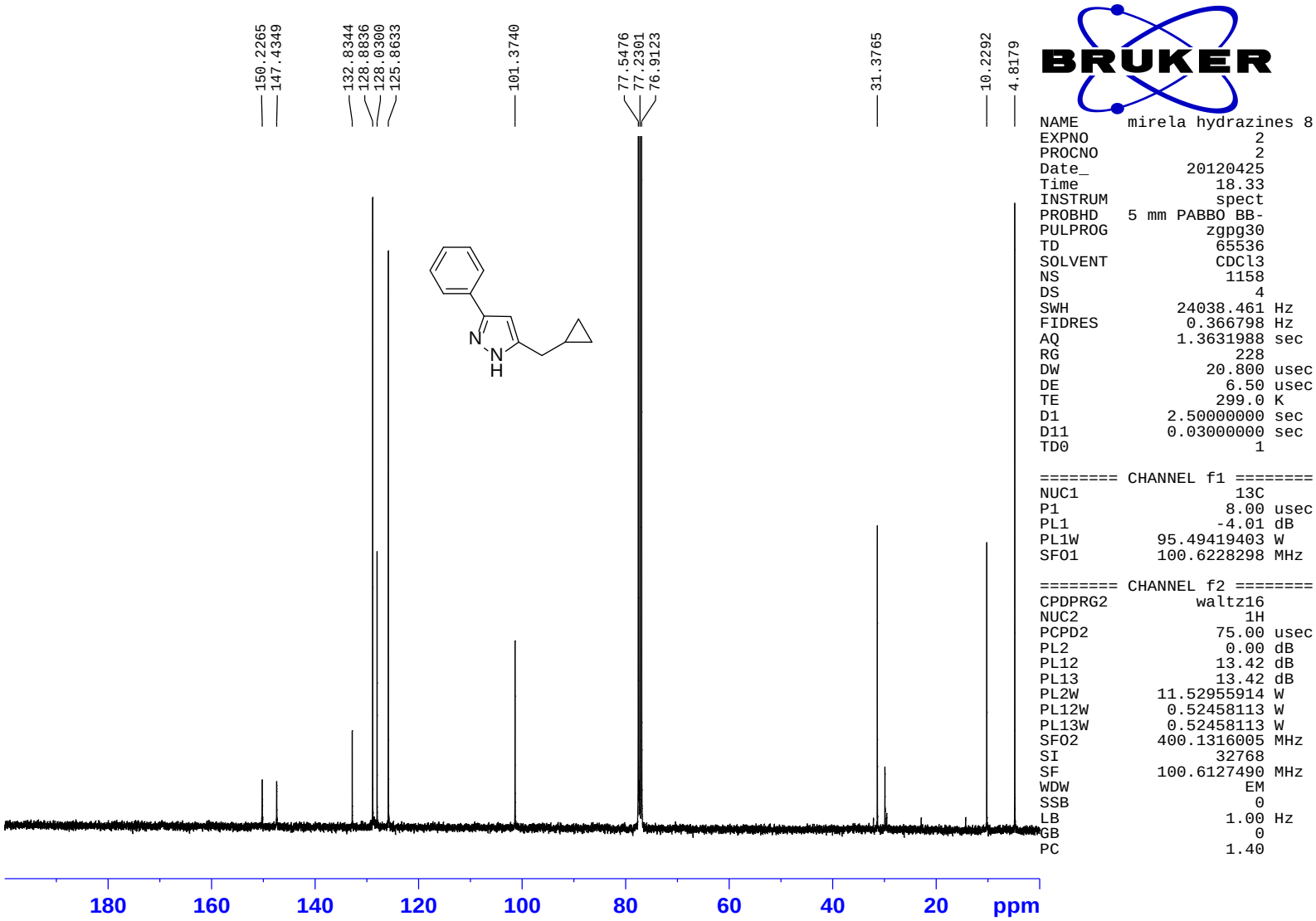
^{13}C NMR spectrum for **5-(cyclopropylmethyl)-3-phenyl-1*H*-pyrazole** ($CDCl_3$)



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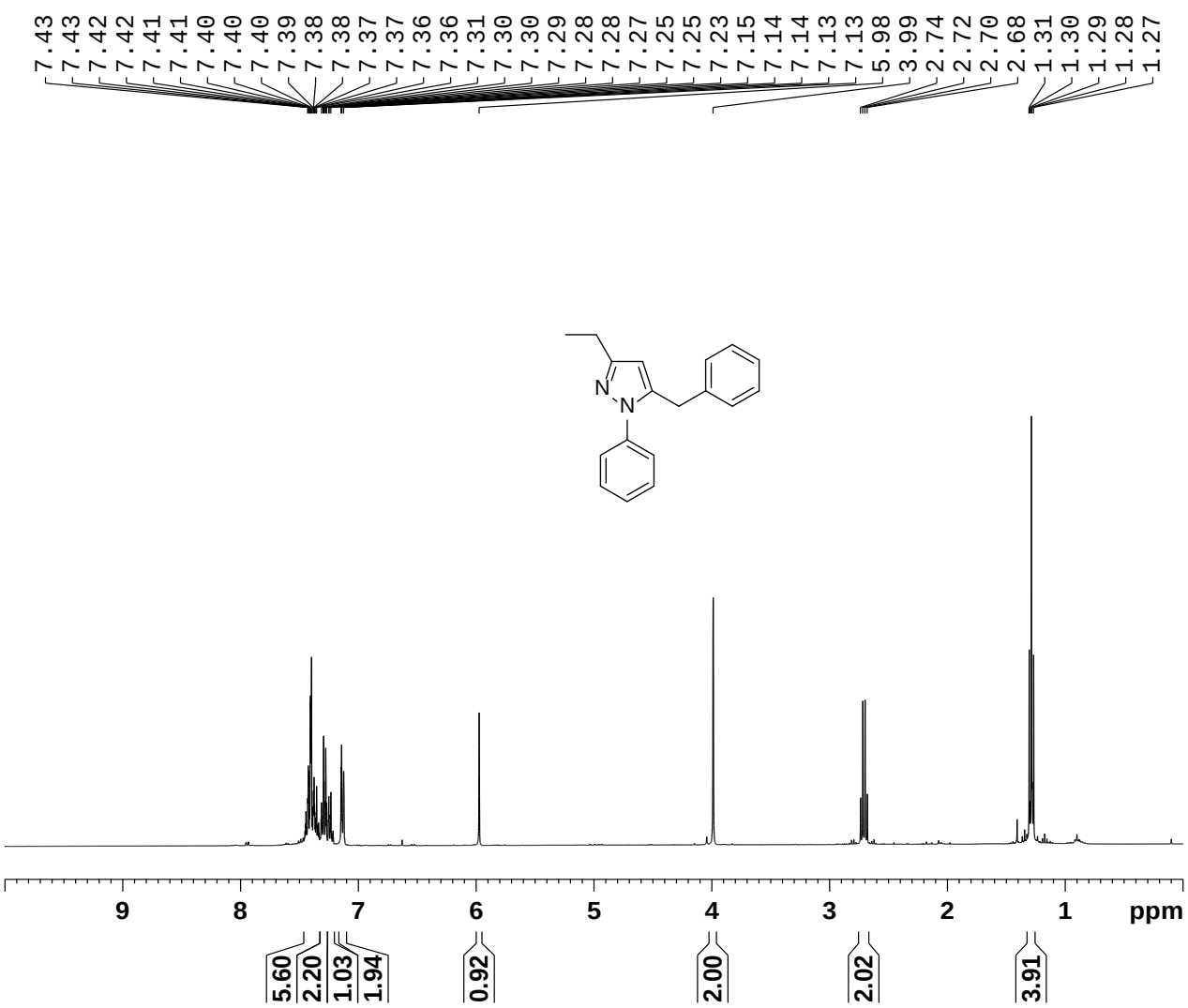
^{1}H NMR spectrum for **5-benzyl-3-ethyl-1-phenyl-1*H*-pyrazole** ($CDCl_3$)




```
NAME          Agnes041912
EXPNO                  98
PROCNO                  1
Date_           20120419
Time               15.01
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                   101
DW                60.800 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.50 usec
PL1                -5.00 dB
PL1W         36.45966721 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300064 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

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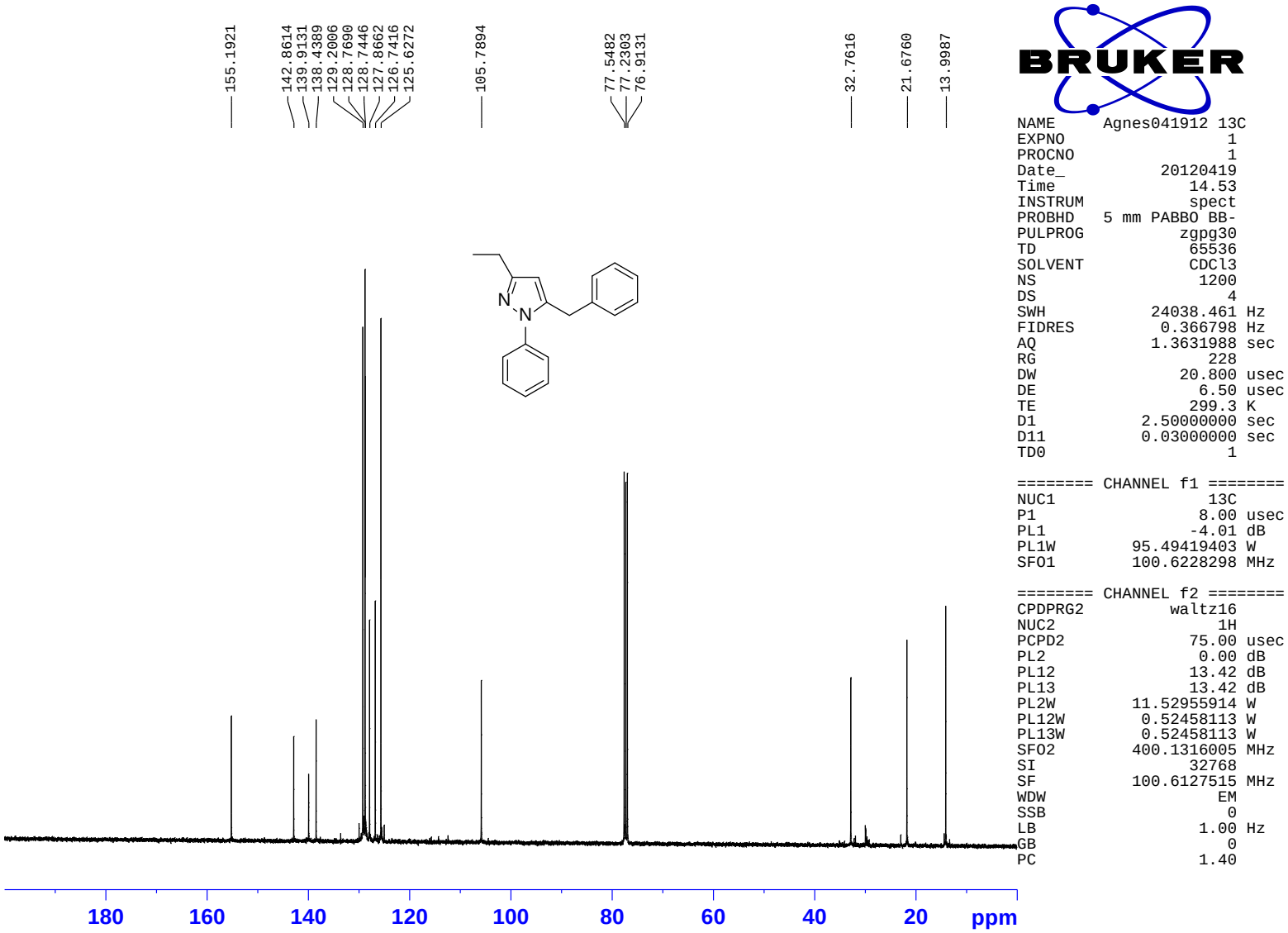
^{13}C NMR spectrum for **5-benzyl-3-ethyl-1-phenyl-1*H*-pyrazole** ($CDCl_3$)




```
NAME        Agnes041912 13C
EXPNO                    1
PROCNO                   1
Date_             20120419
Time                 14.53
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1200
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631988 sec
RG                    228
DW                20.800 usec
DE                 6.50 usec
TE                299.3 K
D1             2.50000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.00 usec
PL1                 -4.01 dB
PL1W          95.49419403 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               75.00 usec
PL2                  0.00 dB
PL12                13.42 dB
PL13                13.42 dB
PL2W          11.52955914 W
PL12W          0.52458113 W
PL13W          0.52458113 W
SFO2          400.1316005 MHz
SI                 32768
SF            100.6127515 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```