SUPPORTING INFORMATION

Catalyst-free chemo/regio/stereo-selective amination of alk-3-ynones. Synthesis of 1,5-benzodiazepines and 3-amino-2-alkenones

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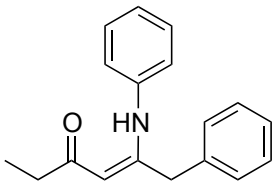
Experimental Section

General Methods. Triethylamine was distilled over CaH_2. *o*-Phenylenediamine (Mallickrodt), 4,5-dimethyl-1,2-phenylenediamine (Aldrich), ethanol (reagent grade 200 proof anhydrous, Pharmco Aaper), silica gel (Dynamic Adsorbents, 32-63 μ), and TLC plates (Whatman, hexanes/ethyl acetate 80:20) were used as received. Other materials not listed were used as received.

The microwave reactions were carried out using capped vials in a Biotage Initiator reactor equipped with a conventional temperature IR sensor. NMR spectra were obtained with a Bruker Avance III spectrometer (^{1}H of 400 MHz and ^{13}C of 100 MHz). The chemical shifts are reported in δ (ppm) values relative to TMS (0 ppm), C_6D_6, and $CDCl_3$ (7.16/7.26 ppm, 128.06/77.16 ppm respectively). Mass spectra were recorded on Agilent 6520 Q-TOF LCMS (HRMS) and Hewlett Packard 5973 GC/MS instruments. IR spectra were recorded on a Bruker Alpha-P ATR spectrometer. Melting points (closed capillaries) were recorded on a Mel Temp apparatus. All products were stored in a refrigerator.

(*Z*)-6-Phenyl-5-(phenylamino)hex-4-en-3-one (6a).



A 20 mL round bottom microwave vial equipped with a stir bar was charged with alkynone **1d** (0.174 g, 1.01 mmol), aniline (0.099 g, 1.05 mmol), and ethanol (10 mL). The vial was sealed and the reaction was irradiated in the microwave reactor for 2.5 h at 45°C. The solvent was removed by rotary evaporation. Silica gel column chromatography (15 × 2.5 cm; hexanes/ethyl acetate; 80:20) gave a yellowish fraction. The solvent was removed by rotary evaporation and the residue was dried by oil pump vacuum to afford **6a** as a reddish oil (0.249 g, 0.939 mmol, 93%). MS: (EI, m/z): 265 (M^+, 59%), 236 ($M - C_2H_5^+$, 100%), 174 ($M - C_7H_7^+$, 100%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{18}H_{20}NO$ 266.1545, found 266.1558. IR (cm^{-1}, film) 3398 br s, 1609 s, 1593 s, 1562 s, 1492 m, 1451 m, 753 s, 695 s. NMR ($CDCl_3$, δ): ^{1}H: 12.51 (br s, 1H), 7.28-7.13 (m, 6H) 7.23-7.11 (m, 4H), 7.03 (td, 4H, J = 7.4, 1.4 Hz), 5.12 (s, 1H), 3.61 (s, 2H), 2.34 (q, 2H, J = 7.6 Hz), 1.12 (t, 3H, J = 7.5 Hz); ^{13}C: 200.4, 162.3, 136.9, 129.1(2C), 128.9(2C), 128.6(2C), 126.8, 126.0, 125.6(2C), 97.3, 38.4, 35.4, 9.9.

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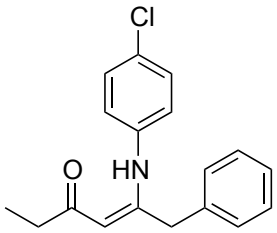
(*Z*)-6-Phenyl-5-(4-chlorophenylamino)hex-4-en-3-one (6b).

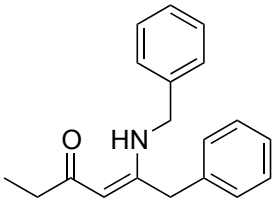


A 20 mL round bottom microwave vial equipped with a stir bar was charged with alkynone **1d** (0.174 g, 1.01 mmol), *p*-chloroaniline (0.134 g, 1.06 mmol), and ethanol (10 mL). The vial was sealed and the reaction was irradiated in the microwave reactor for 4 h at 45°C. The solvent was removed by rotary evaporation. Silica gel column chromatography (15 × 2.5 cm; hexanes/ethyl acetate; 80:20) gave a yellowish fraction. The solvent was removed by rotary evaporation and the residue was dried by oil pump vacuum to afford **6b** as a reddish solid (0.277 g, 0.929 mmol, 92%), mp 79-80°C. MS: (EI, m/z): 299 (M^+, 71%), 270($M - C_2H_5^+$, 86%), 208 ($M - C_7H_7^+$, 100%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{18}H_{19}ClNO$ 300.1155, found 300.1147. IR (cm^{-1}, film) 3385 br, 1582 s, 1560 s, 1493 m, 1449 m, 723 s, 695 s. NMR ($CDCl_3$, δ): ^1H: 12.44 (br s, 1H), 8.51(s, 1H), 7.26-7.16 (m, 5H), 7.04 (AA'XX', 1H, J = 8.0 Hz),[S1] 6.92 (AA'XX', 1H, J = 8.6 Hz), 5.16 (s, 1H), 3.58 (s, 2H), 2.35 (q, 2H, J = 7.5 Hz), 1.11 (t, 3H, J = 7.5 Hz); ^{13}C: 200.7, 161.7, 137.3, 136.6, 131.4, 129.2(2C), 128.7(2C), 128.6(2C), 126.9, 126.7(2C), 97.9, 38.4, 35.4, 9.7.

Synthesis of Enaminones 6c-e. **General Procedure**.

A 10 mL round bottom microwave vial equipped with a stir bar was charged with alkynone **1d** (~1.00 mmol), amine (~1.05 mmol), and ethanol (5 mL). The vial was sealed and the mixture was irradiated in the microwave reactor for 5 min at 50°C. The solvent was removed by rotary evaporation. The residue was dissolved in ether (ca. 40 mL), and washed with ice-water (ca. 25 mL; 100 mL separatory funnel). Water phase was extracted with ether (3 × 40 mL). The combined organic layers were dried over $MgSO_4$, filtered, through medium porosity frit funnel, concentrated by a rotary evaporation, and dried by oil pump vacuum to give **6c-e**.

(*Z*)-5-(Benzylamino)-6-phenylhex-4-en-3-one (6c).



From **1d** (0.190 g, 1.10 mmol), and benzylamine (0.124g, 1.16 mmol), afforded **6c** as a yellow solid (0.260 g, 0.935 mmol, 85%), mp 44-45°C. MS: (EI, m/z): 279 (M^+, 51%), 250 ($M - C_2H_5^+$, 89%), 91 ($M - C_6H_{14}NO^+$, 100%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{19}H_{22}NO$

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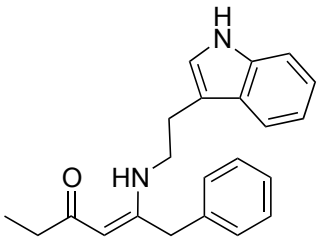
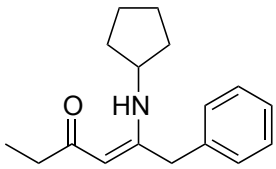
280.1701, found 280.1700. IR (cm^{-1}, film) 3248 m, 1613 s, 1573 s, 1493 m, 1434 m, 731 s, 693 s. NMR ($CDCl_3$, δ): ^1H: 11.20 (s, 1H), 7.37-7.21 (m, 8H), 7.17 (AA'XX', 2H, J = 7.2 Hz),[S1] 5.08 (s, 1H), 4.32 (d, 2H, J = 6.2 Hz) 3.55 (s, 2H), 2.34 (q, 2H, J = 6.5 Hz), 1.14 (t, 3H, J = 7.5 Hz); ^{13}C: 199.5, 163.7, 137.9, 136.1, 128.7(2C), 128.6(2C), 128.4(2C), 127.3, 126.8, 126.8(2C), 95.9, 46.6, 38.5, 34.9, 9.8

(*Z*)-5-{[2-(1*H*-indol-3-yl)ethyl]amino}-6-phenylhex-4-en-3-one (6d).



From **1d** (0.166 g, 0.965 mmol) and tryptamine (0.162 g, 1.03 mmol) afforded **6d** as a yellow solid (0.312 g, 0.939 mmol, 97%), mp 99-101°C. MS: (EI, m/z): 332 (M^+, 2%), 303 ($M - C_2H_5^+$, 2%), 202 ($C_{17}H_{16}NO^+$, 82%), 143 ($C_{10}H_9N^+$, 41%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{22}H_{25}N_2O$ 333.1967, found 333.1997. IR (cm^{-1}, film) 3272 br, 1599 s, 1547 s, 1491 m, 1454 m, 736 s, 697 s. NMR ($CDCl_3$, δ): ^1H: 10.88 (br s, 1H,), 8.51 (s, 1H), 7.30 (d, 1H, J = 7.9 Hz), 7.23-7.11 (m, 4H), 7.08-7.02 (m, 3H), 6.97 (dt, 1H, J = 7.5, 0.9 Hz), 6.89 (d, 1H, J = 2.3 Hz), 4.86 (s, 1H), 3.37 (t, 2H, J = 6.8 Hz), 3.36 (s, 2H,) 2.81 (t, 2H, J = 6.9 Hz), 2.19 (q, 2H, J = 7.6 Hz), 1.01 (t, 3H, J = 7.6 Hz); ^{13}C: 199.4, 164.3, 136.6, 136.4, 128.9(2C), 128.6(2C), 127.2, 127.0, 123.0, 122.0, 119.4, 118.5, 112.1, 111.5, 95.3, 43.8, 38.9, 35.2, 26.3, 10.5.

(*Z*)-5-(Cyclopentylamino)-6-phenylhex-4-en-3-one (6e).



From **1d** (0.190 g, 1.10 mmol) and cyclopentyl amine (0.099g, 1.16 mmol), afforded **6e** as a yellow oil (0.240 g, 0.935 mmol, 85%). MS: (EI, m/z): 257 (M^+, 50%), 228 ($M - C_2H_5^+$, 100%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{17}H_{23}NO$ 258.1858, found 258.1857. IR (cm^{-1}, film) 2960 m, 1602 s, 1570 s, 1509 m, 1494 m, 1444 m, 719 s, 698 s. NMR ($CDCl_3$, δ): ^1H 11.04 (s, 1H), 7.29 (tt, 2H, J = 7.1, 1.4 Hz), 7.24-7.17 (m, 3H), 4.90 (s, 1H), 3.86-3.74 (m, 1H) 3.56 (s, 2H), 2.25 (q, 2H, J = 7.6 Hz) 1.84-1.63 (m, 4H), 1.54-1.36 (m, 4H), 1.07 (t, 3H, J = 7.5 Hz); ^{13}C: 198.7, 163.3, 136.5, 129.7, 128.7(2C), 128.5(2C), 126.8, 94.9, 54.4, 38.7, 34.8, 34.2(2C), 23.8(2C), 9.9.

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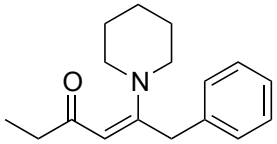
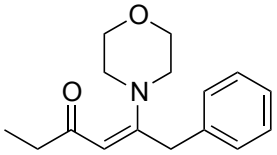
(*Z*)-5-(Piperidin-1-yl)-6-phenylhex-4-en-3-one (6f).



From **1d** (0.166 g, 0.965 mmol) and piperidine (0.086 g, 1.01 mmol) afforded **6f** as a colorless oil (0.230 g, 0.895 mmol, 93%). MS: (EI, m/z): 257 (M^+, 21%), 242 ($M - CH_3^+$, 33%), 228 ($M - C_2H_5^+$, 100%), 200 ($M - C_3H_5O^+$, 73%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{17}H_{24}NO$ 258.1858, found 258.1854. IR (cm^{-1}, film) 1636 m, 1527 s, 1493 m, 1441 s, 721 m, 667 m. NMR ($CDCl_3$, δ): ^1H: 7.25-7.16 (m, 2H), 7.16-7.06 (m, 3H), 5.35 (s, 1H), 4.55 (s, 2H), 3.24 (t, 4H, J = 5.3 Hz), 2.35 (q, 2H, J = 7.4 Hz), 1.55-1.46 (m, 2H), 1.40-1.28 (m, 4H), 1.07 (t, 3H, J = 7.5 Hz); ^{13}C: 198.1, 161.0, 138.1, 128.3(2C), 127.9(2C), 125.7(2C), 95.5, 47.6(2C), 37.5, 33.7, 25.4(2C), 24.2, 9.8.

(*Z*)-5-(Morpholino)-6-phenylhex-4-en-3-one (6g).



From **1d** (0.166 g, 0.965 mmol) and morpholine (0.90 g, 1.03 mmol) afforded **6g** as a reddish solid (0.242 g, 0.934 mmol, 97%), mp 46-48 °C. MS: (EI, m/z): 259 (M^+, 41%), 244 ($M - CH_3^+$, 35%), 230 ($M - C_2H_5^+$, 100%), 202 ($M - C_3H_5O^+$, 62%), 115 ($M - CH_3^+$, 41%), 91($C_7H_7^+$, 62%). HRMS (ESI-TOF) $[M + H]^+$ calcd for $C_{16}H_{22}NO_2$ 260.1651, found 260.1643. IR (cm^{-1}, film) 1639 m, 1599 m, 1527 s, 1493 m, 1441 s, 717 s, 698 m. NMR ($CDCl_3$, δ): ^1H: 7.27-7.21 (m, 2H), 7.18-7.12 (m, 3H), 5.41 (s, 1H), 4.54 (s, 2H) 3.51 (apparent t, 4H, J = 4.9 Hz), 3.24 (apparent t, 4H, J = 5.4 Hz), 2.41 (q, 2H, J = 7.4 Hz), 1.09 (t, 3H, J = 7.5 Hz); ^{13}C: 198.9, 161.3, 138.0, 128.8(2C), 128.1(2C), 126.2, 97.6, 66.4(2C), 46.6(2C), 37.9, 33.6, 9.7.

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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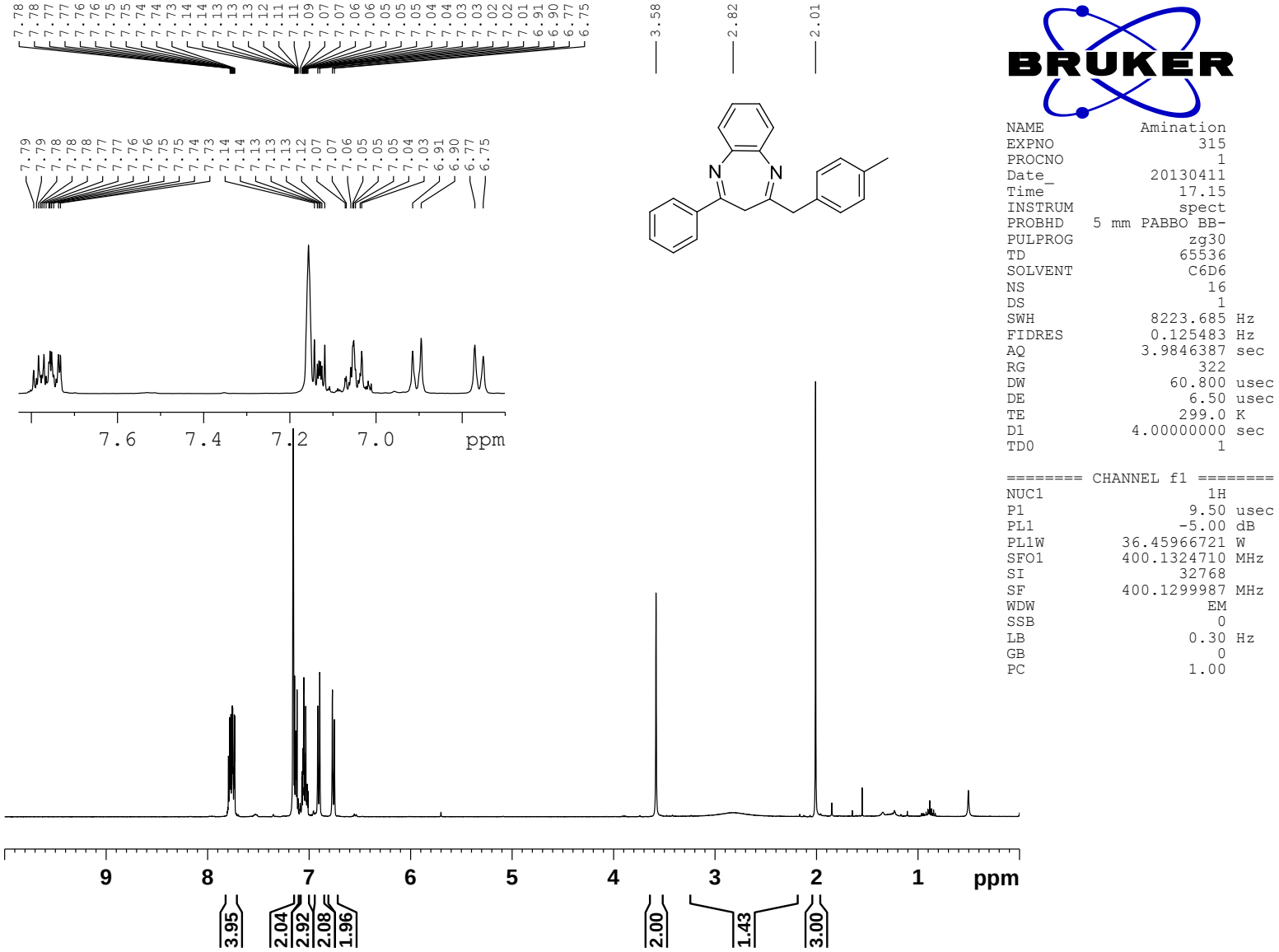
^{1}H NMR spectrum for **3aa** (C_6D_6)

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FIDRES             0.125483 Hz
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RG                      322
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DE                    6.50 usec
TE                   299.0 K
D1                 4.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
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PL1                   -5.00 dB
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SFO1            400.1324710 MHz
SI                      32768
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WDW                       EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
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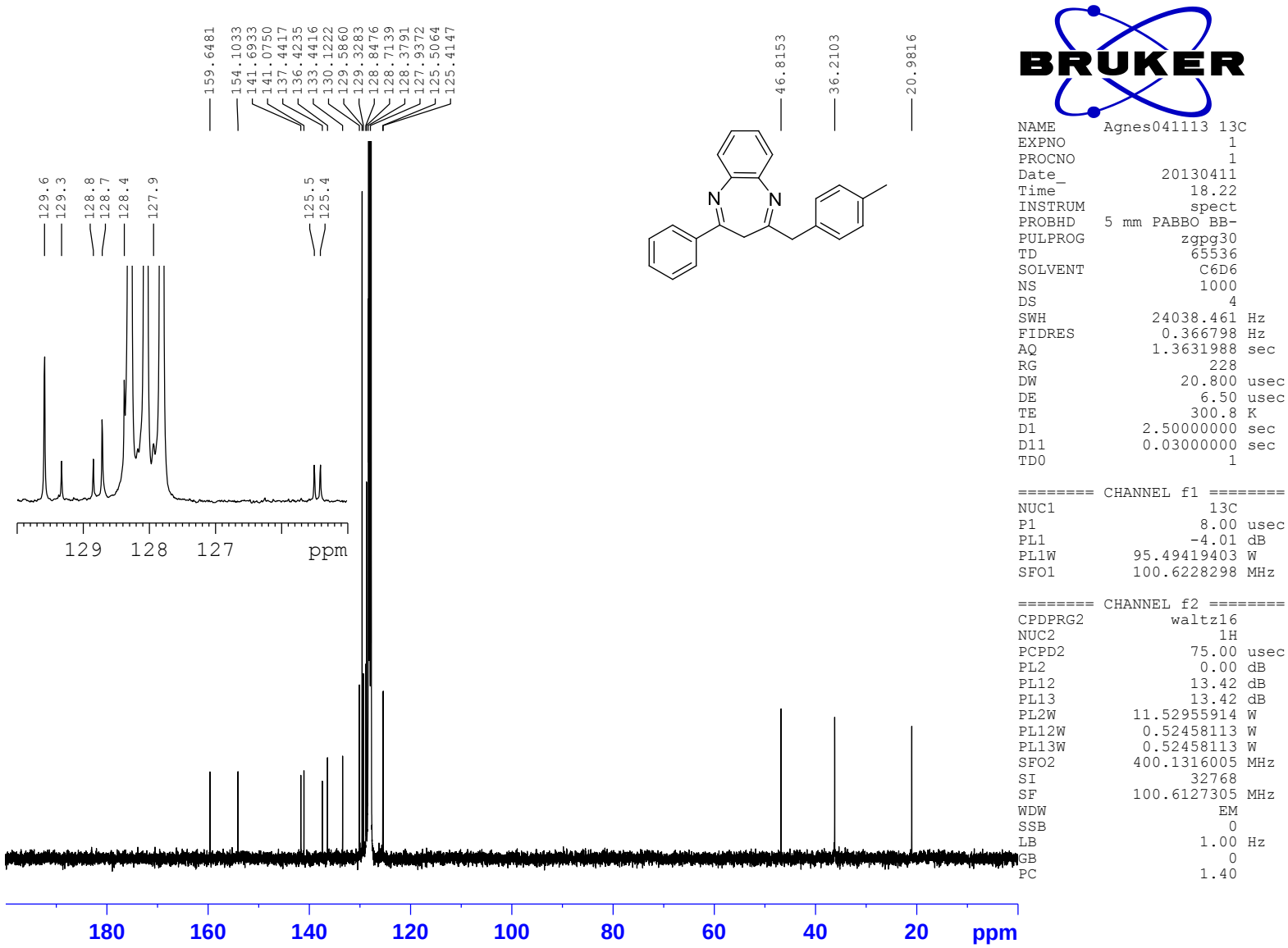
^{13}C NMR spectrum for **3aa** (C_6D_6)

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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                   300.8 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           100.6127305 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
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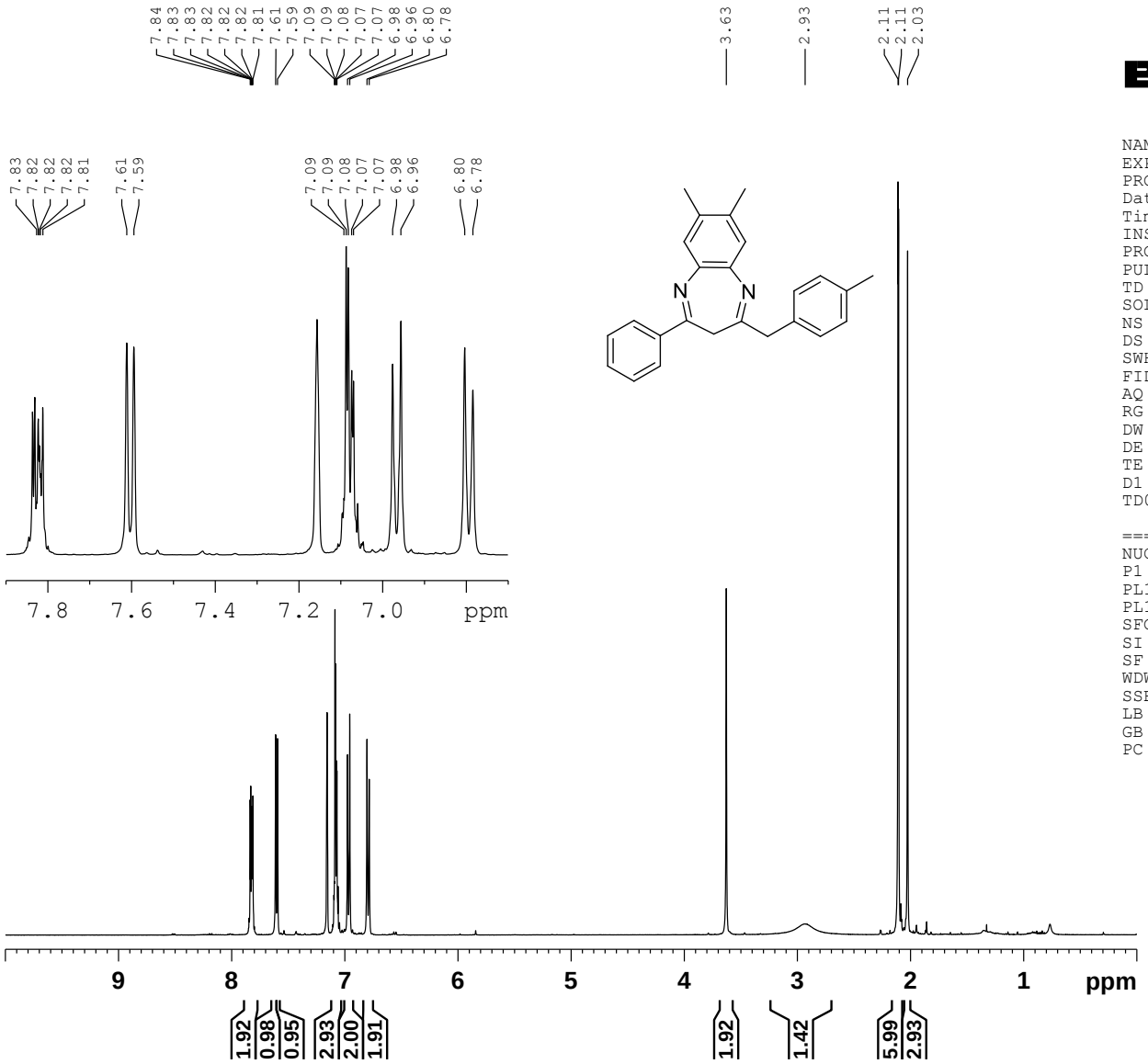
^{1}H NMR spectrum for **3ab** (C_6D_6)

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PROCNO                    1
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TD                    65536
SOLVENT                C6D6
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DS                        1
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                3.9846387 sec
RG                      114
DW                   60.800 usec
DE                    6.50 usec
TE                   298.7 K
D1               4.00000000 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             400.1299987 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
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PC                     1.00
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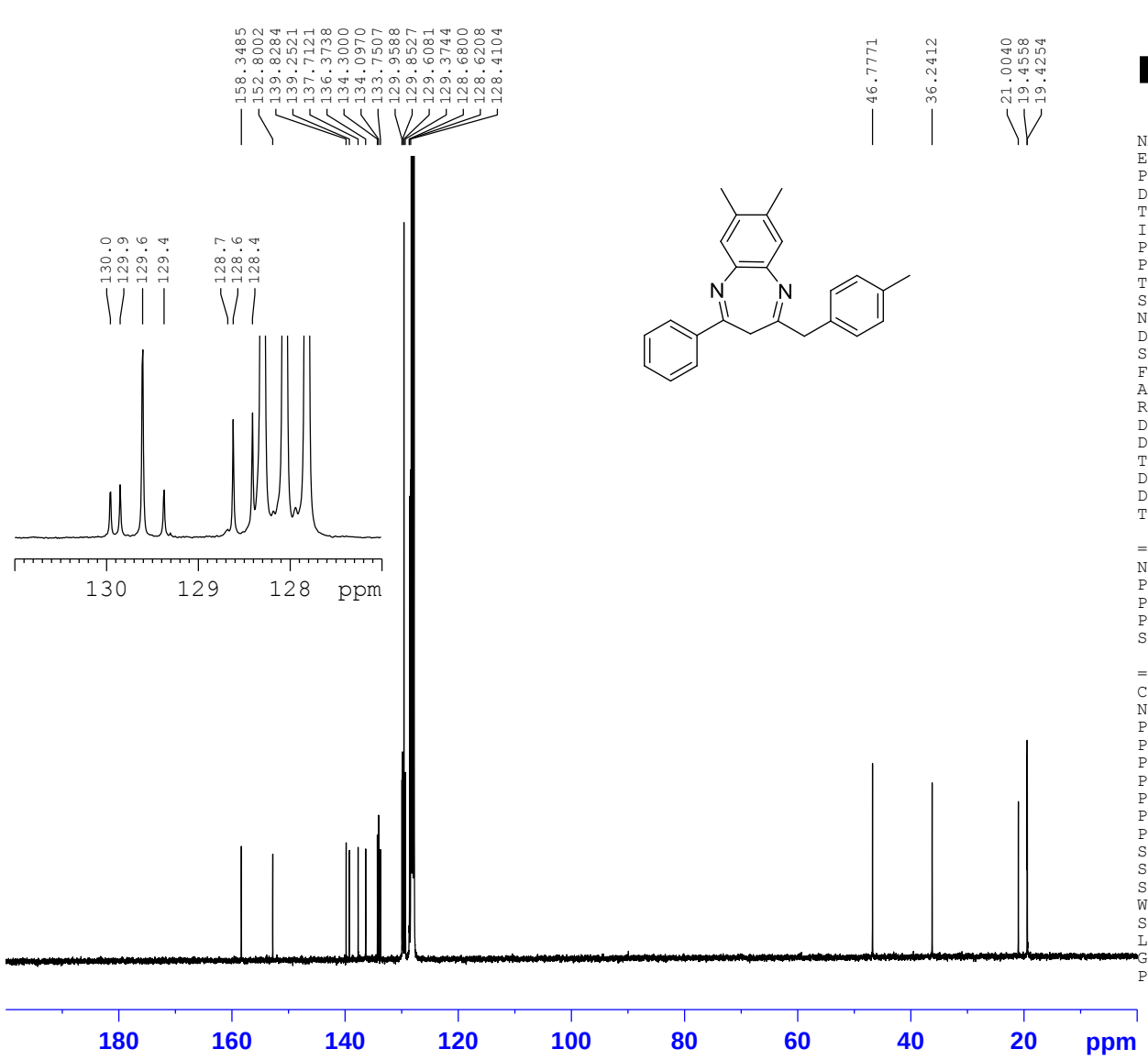
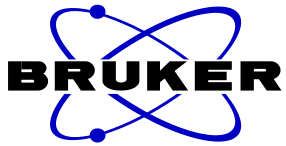
^{13}C NMR spectrum for **3ab** (C_6D_6)





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PROCNO	1
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TD	65536
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SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	228
DW	20.800 usec
DE	6.50 usec
TE	300.5 K
D1	2.50000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
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PL1W	95.49419403 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
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NUC2	1H
PCPD2	75.00 usec
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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	100.6127305 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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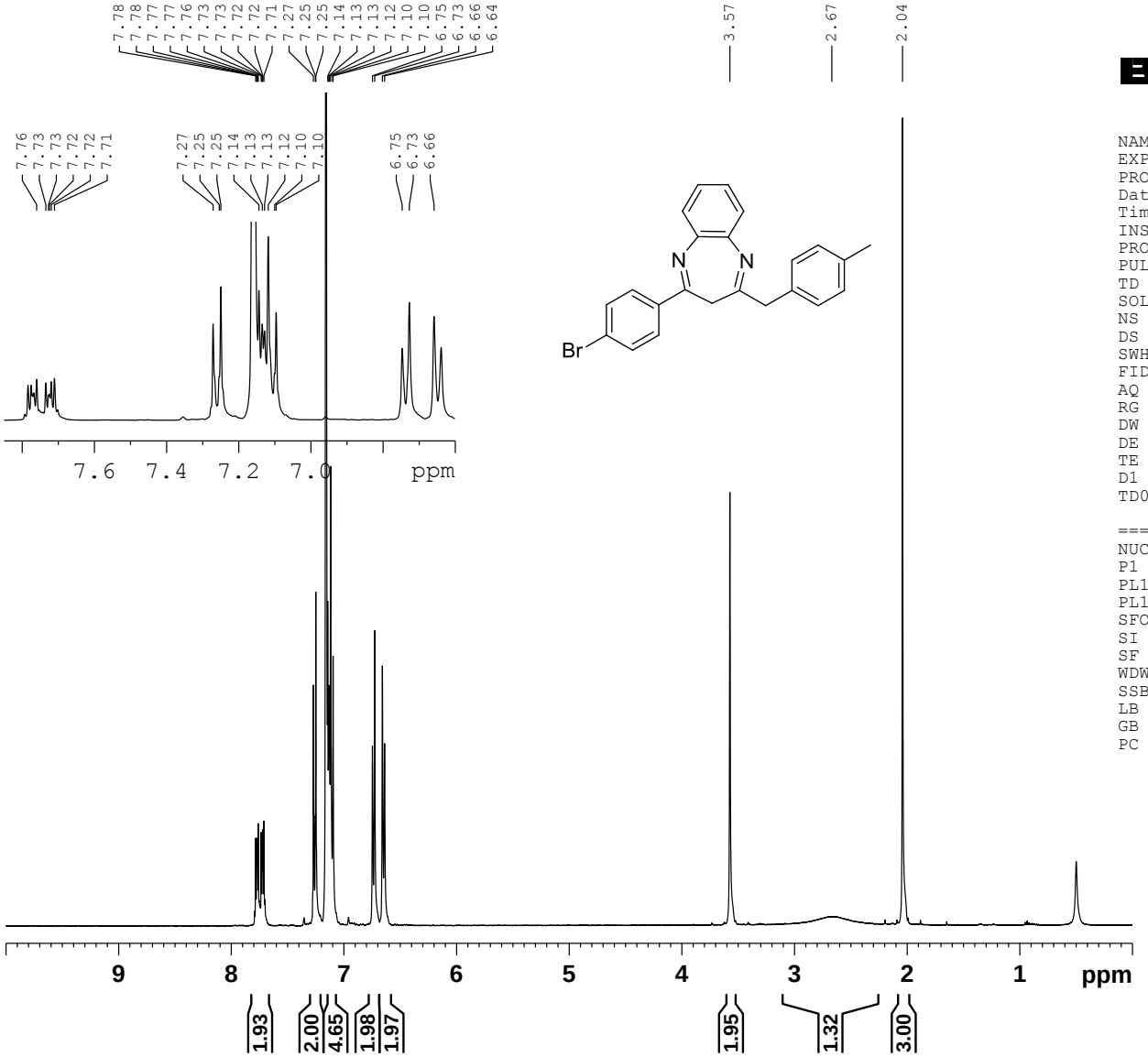
^{1}H NMR spectrum for **3ba** (C_6D_6)



BRUKER

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FIDRES	0.125483 Hz
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RG	287
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TE	297.3 K
D1	4.00000000 sec
TD0	1
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NUC1	1H
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SI	32768
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WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

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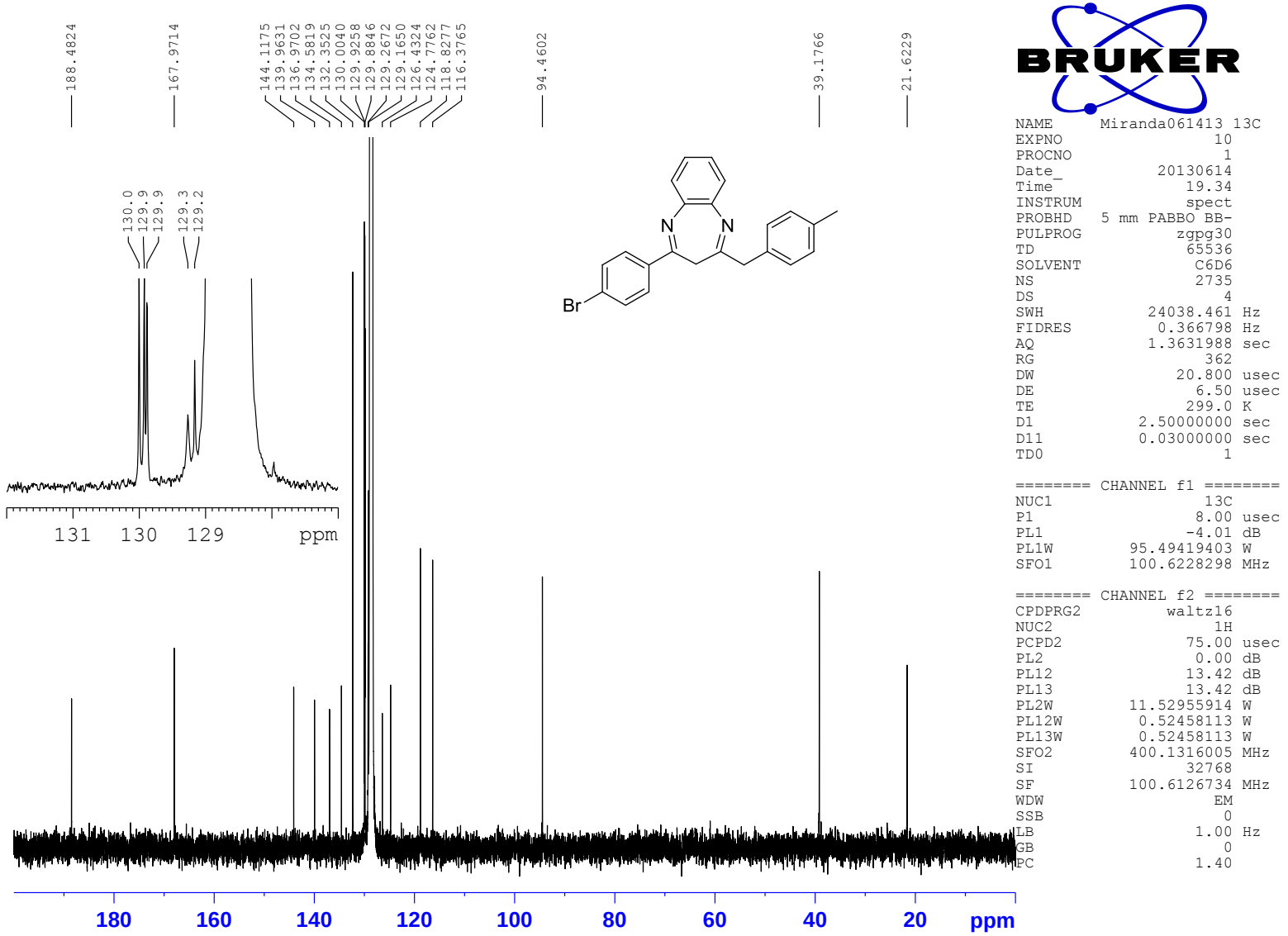
^{13}C NMR spectrum for **3ba** (C_6D_6)



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PROCNO	1
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SWH	24038.461 Hz
FIDRES	0.366798 Hz
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RG	362
DW	20.800 usec
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TE	299.0 K
D1	2.50000000 sec
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
======== CHANNEL f2 ========	
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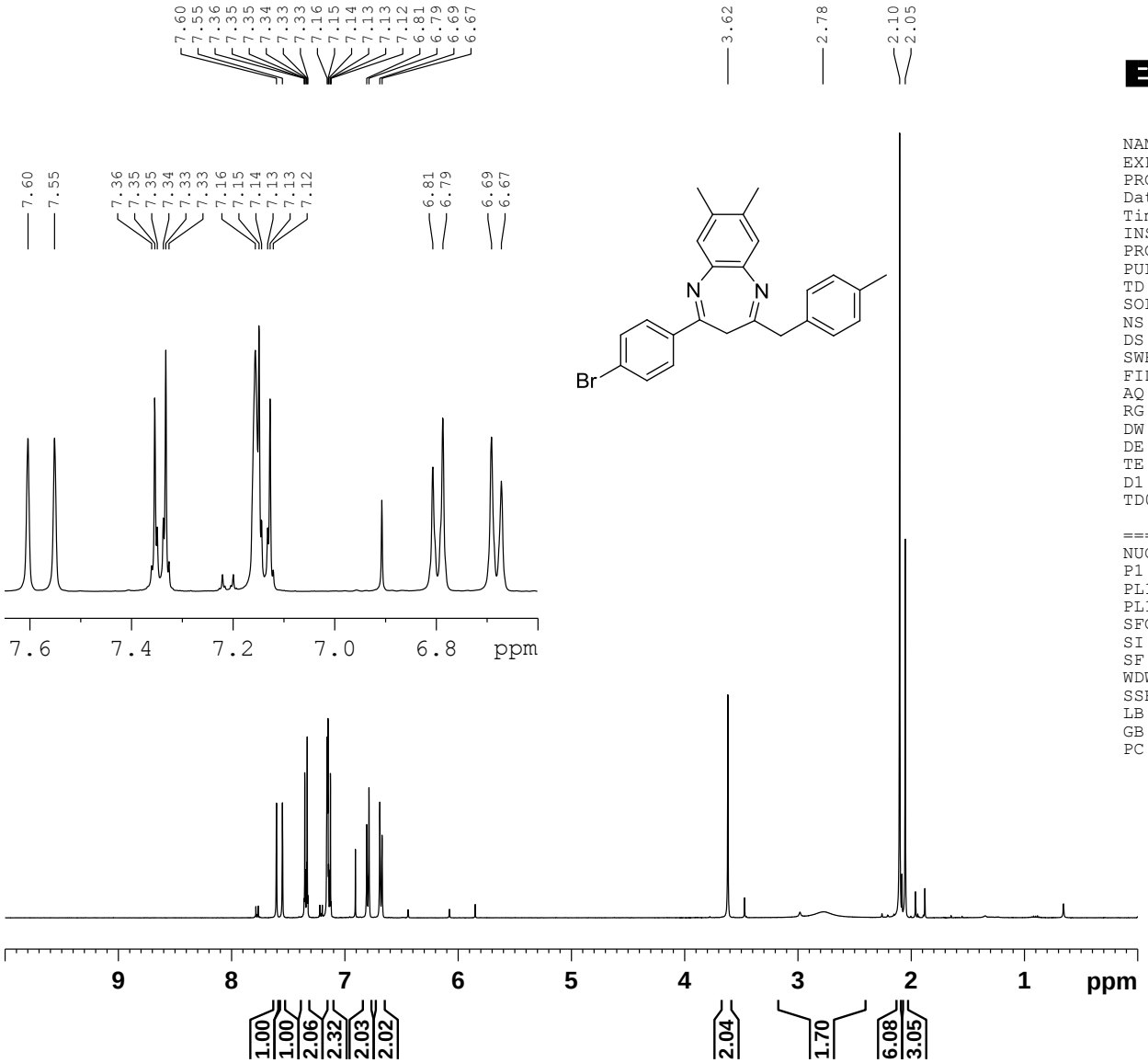
PCPD2	75.00 usec
PL2	0.00 dB
PL12	13.42 dB
PL13	13.42 dB
PL2W	11.52955914 W
PL12W	0.52458113 W
PL13W	0.52458113 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6126734 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **3bb** (C_6D_6)

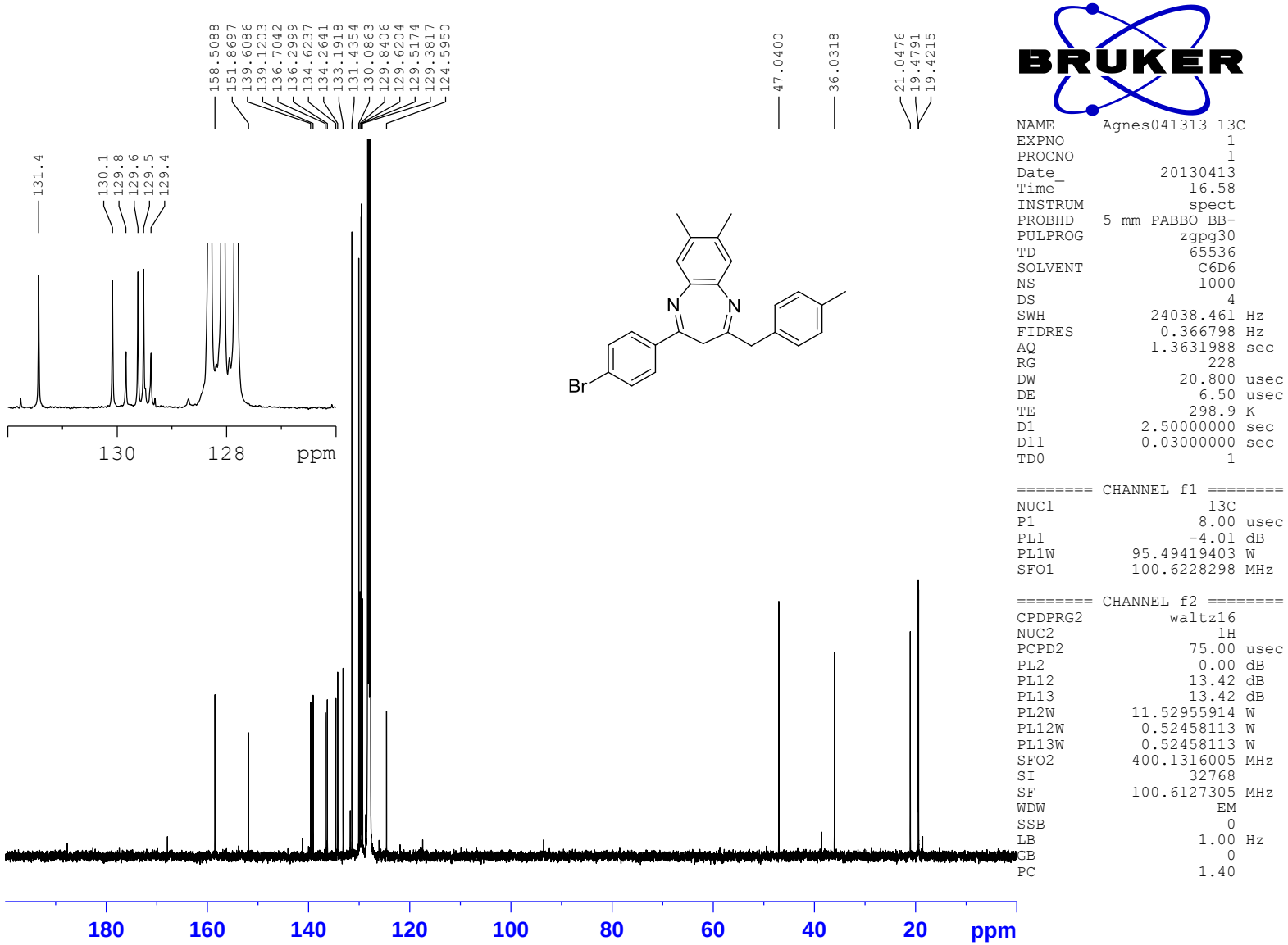



```
NAME            Agnes041313
EXPNO               317
PROCNO                1
Date_          20130413
Time              15.49
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            C6D6
NS                   16
DS                    1
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  161
DW               60.800 usec
DE                6.50 usec
TE                297.1 K
D1           4.00000000 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         400.1299987 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **3bb** (C_6D_6)



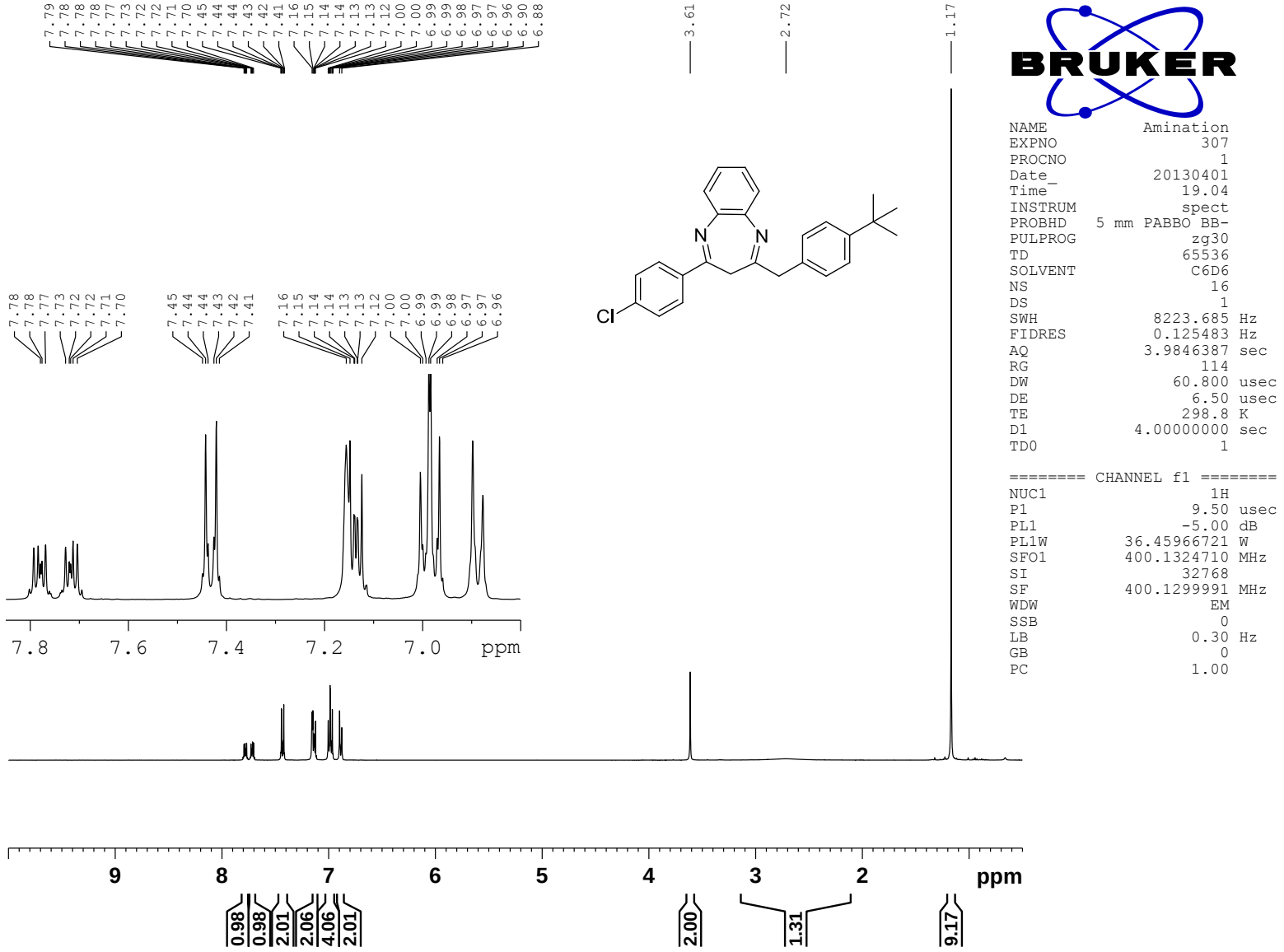

```
NAME        Agnes041313 13C
EXPNO                    1
PROCNO                   1
Date_             20130413
Time                 16.58
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              C6D6
NS                   1000
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                   228
DW                20.800 usec
DE                 6.50 usec
TE                298.9 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          100.6127305 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **3ca** (C_6D_6)

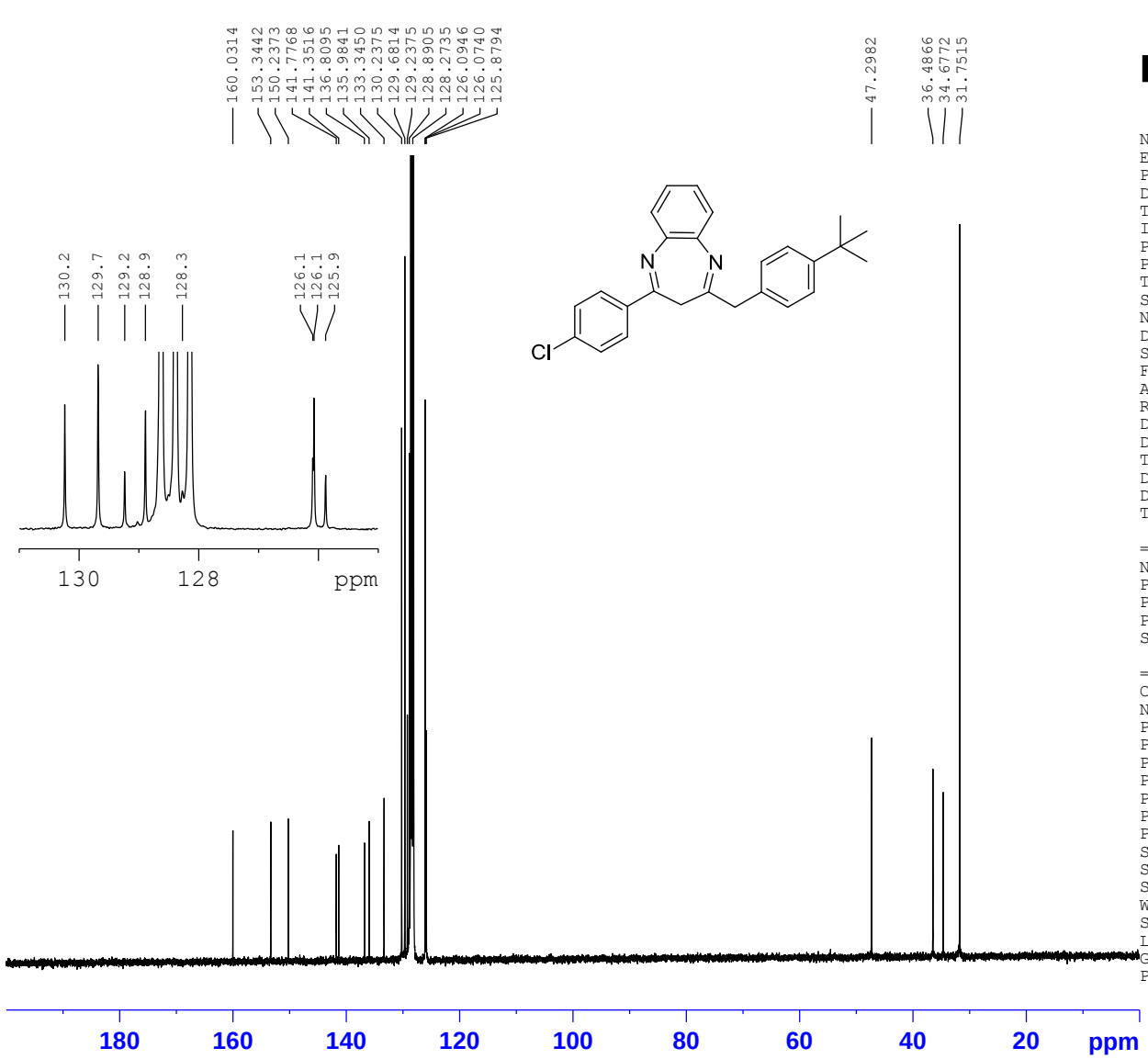


BRUKER

NAME	Amination
EXPNO	307
PROCNO	1
Date_	20130401
Time	19.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	C6D6
NS	16
DS	1
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	114
DW	60.800 usec
DE	6.50 usec
TE	298.8 K
D1	4.00000000 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	400.1299991 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **3ca** (C_6D_6)



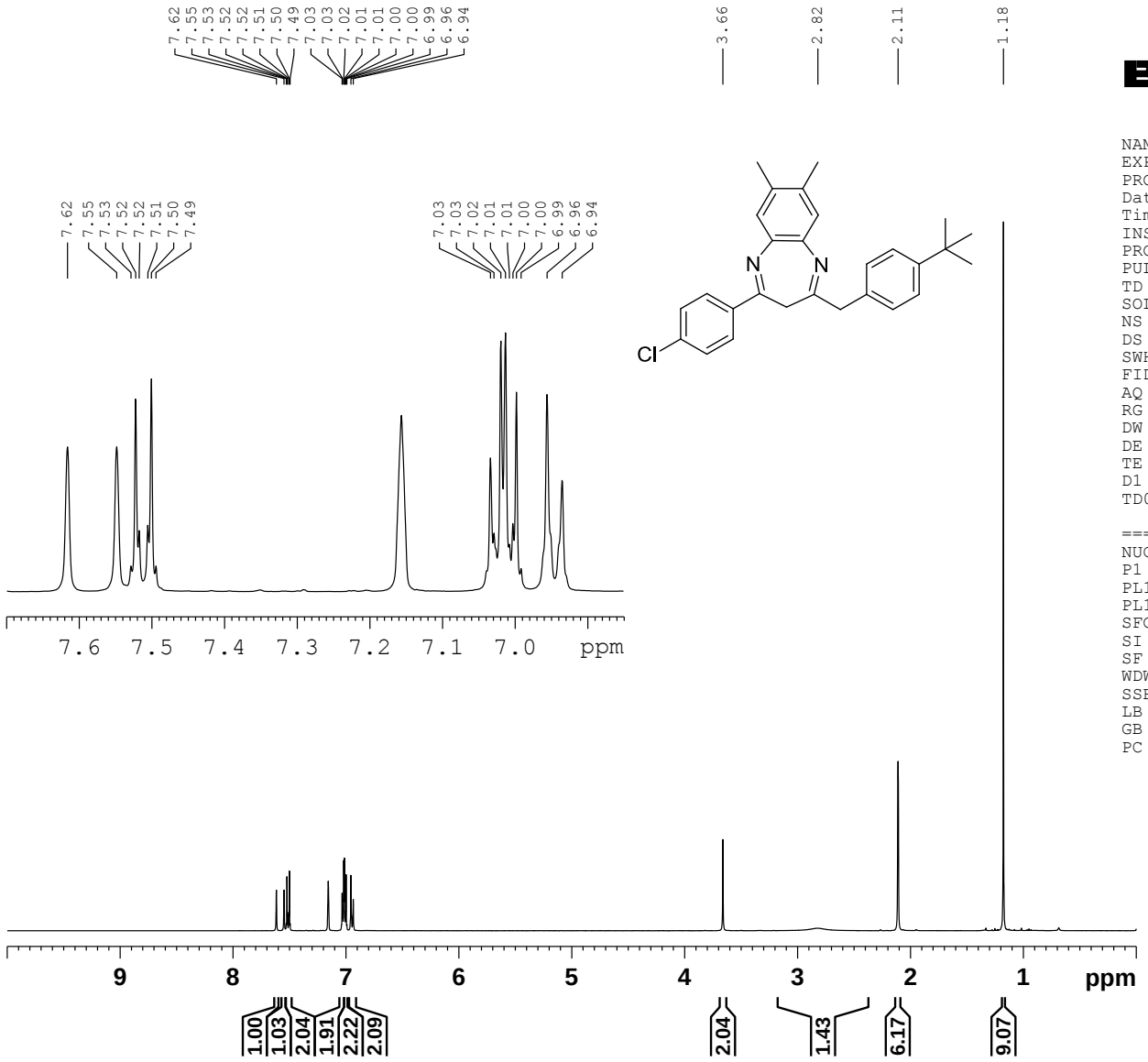

```
NAME        Agnes040113 13C
EXPNO                    1
PROCNO                   1
Date_             20130401
Time                 20.03
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                C6D6
NS                      872
DS                        1
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                      228
DW                  20.800 usec
DE                   6.50 usec
TE                  300.7 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            100.6126977 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **3cb** (C_6D_6)

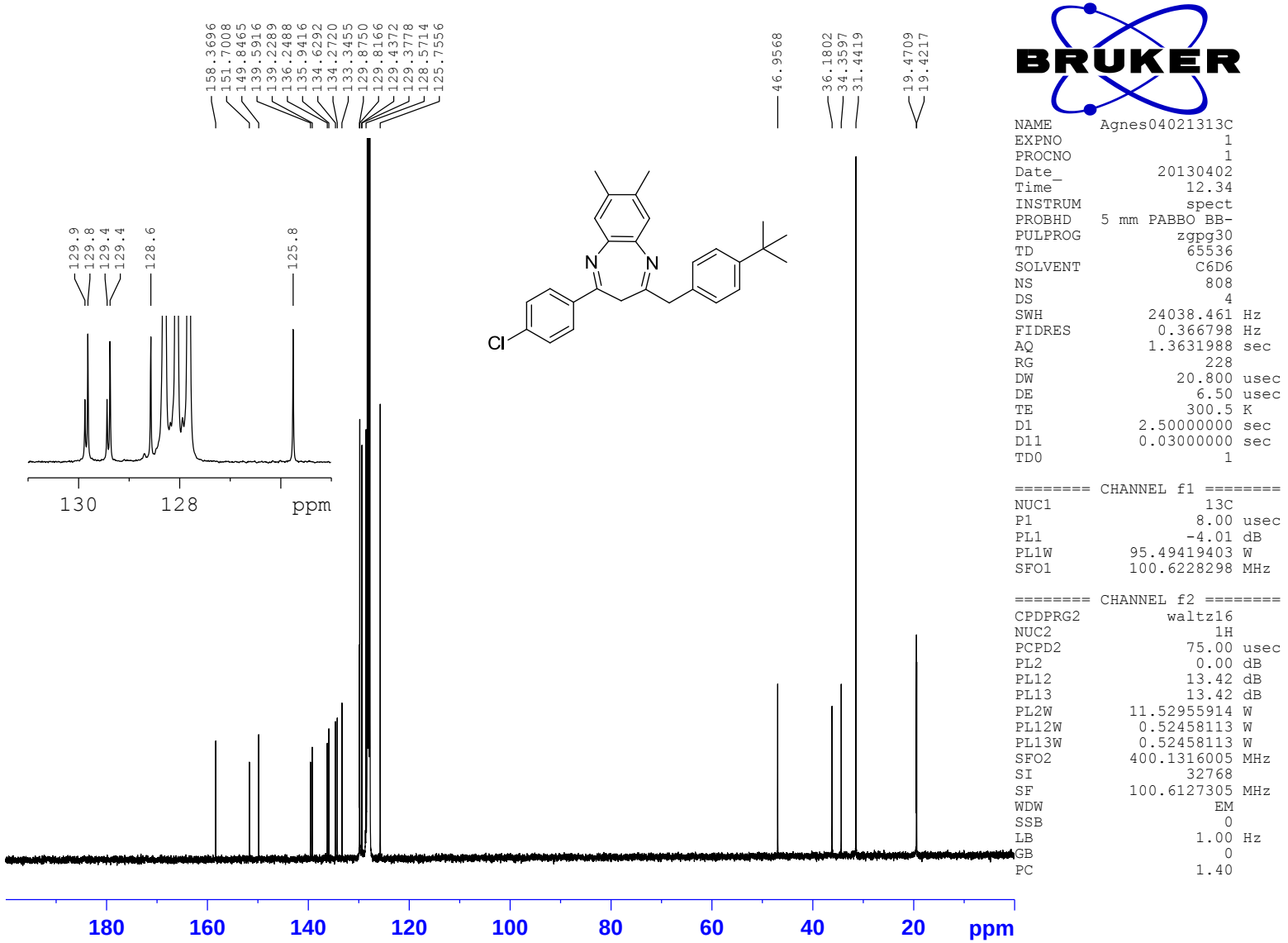



```
NAME          Agnes040213
EXPNO             309
PROCNO              1
Date_          20130402
Time              12.42
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT           C6D6
NS                  16
DS                   1
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                 114
DW                60.800 usec
DE                 6.50 usec
TE                299.2 K
D1             4.00000000 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          400.1299986 MHz
WDW                EM
SSB                 0
LB                0.30 Hz
GB                  0
PC                1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **3cb** (C_6D_6)



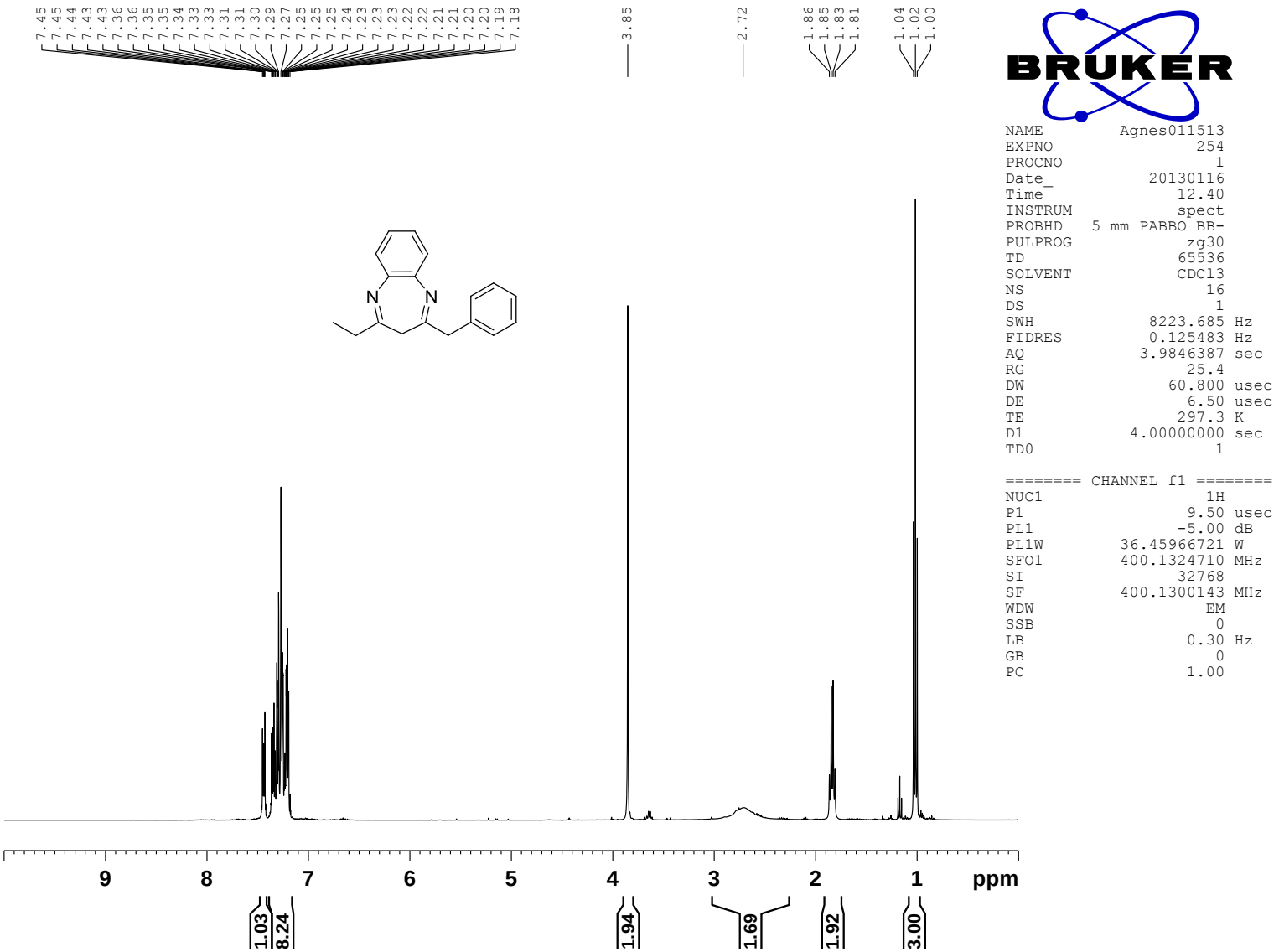

```
NAME        Agnes04021313C
EXPNO                    1
PROCNO                   1
Date_             20130402
Time                 12.34
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                C6D6
NS                      808
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                   300.5 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           100.6127305 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

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

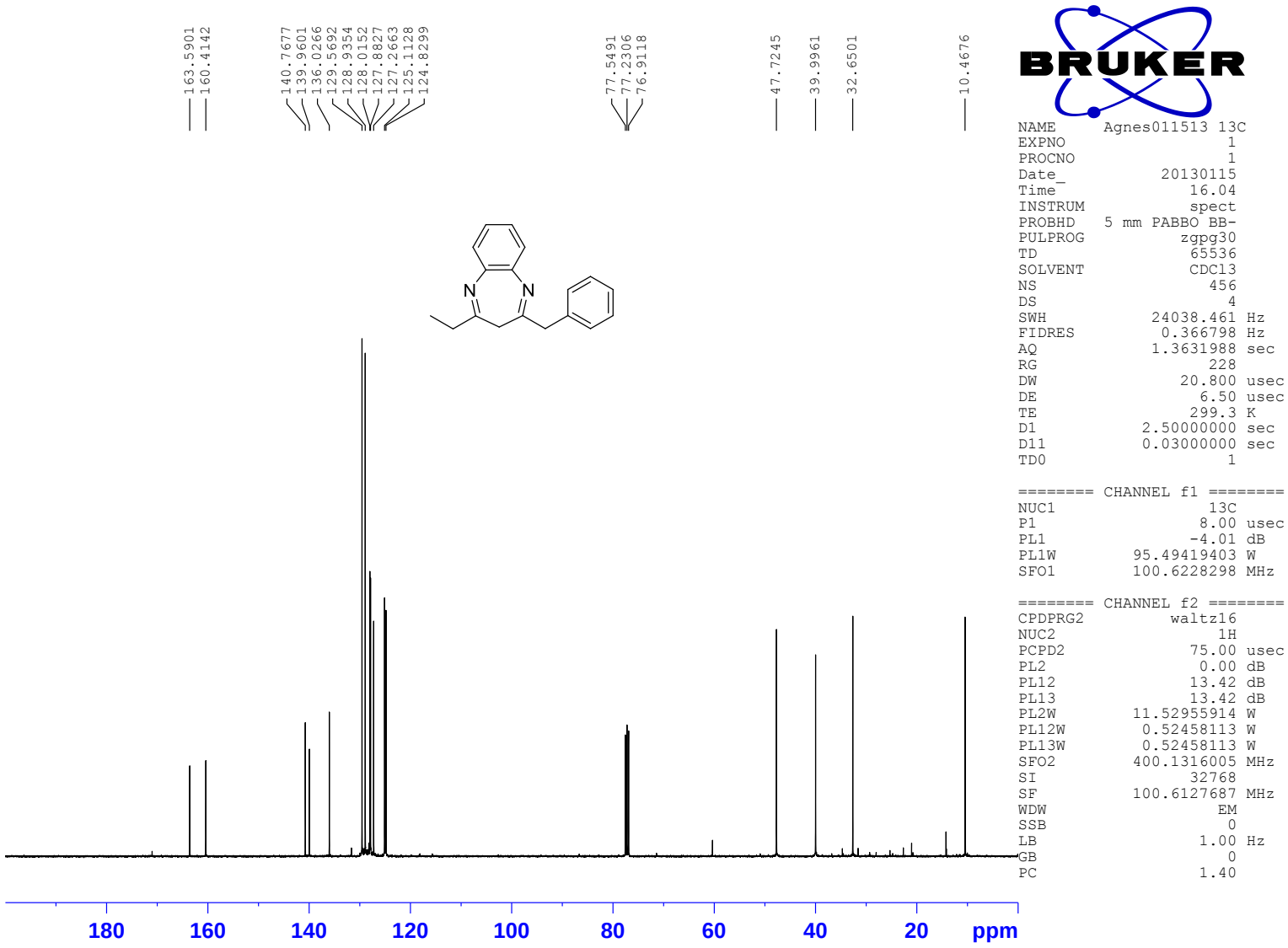


BRUKER

NAME	Agnes011513
EXPNO	254
PROCNO	1
Date_	20130116
Time	12.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	1
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	25.4
DW	60.800 usec
DE	6.50 usec
TE	297.3 K
D1	4.00000000 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	400.1300143 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

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



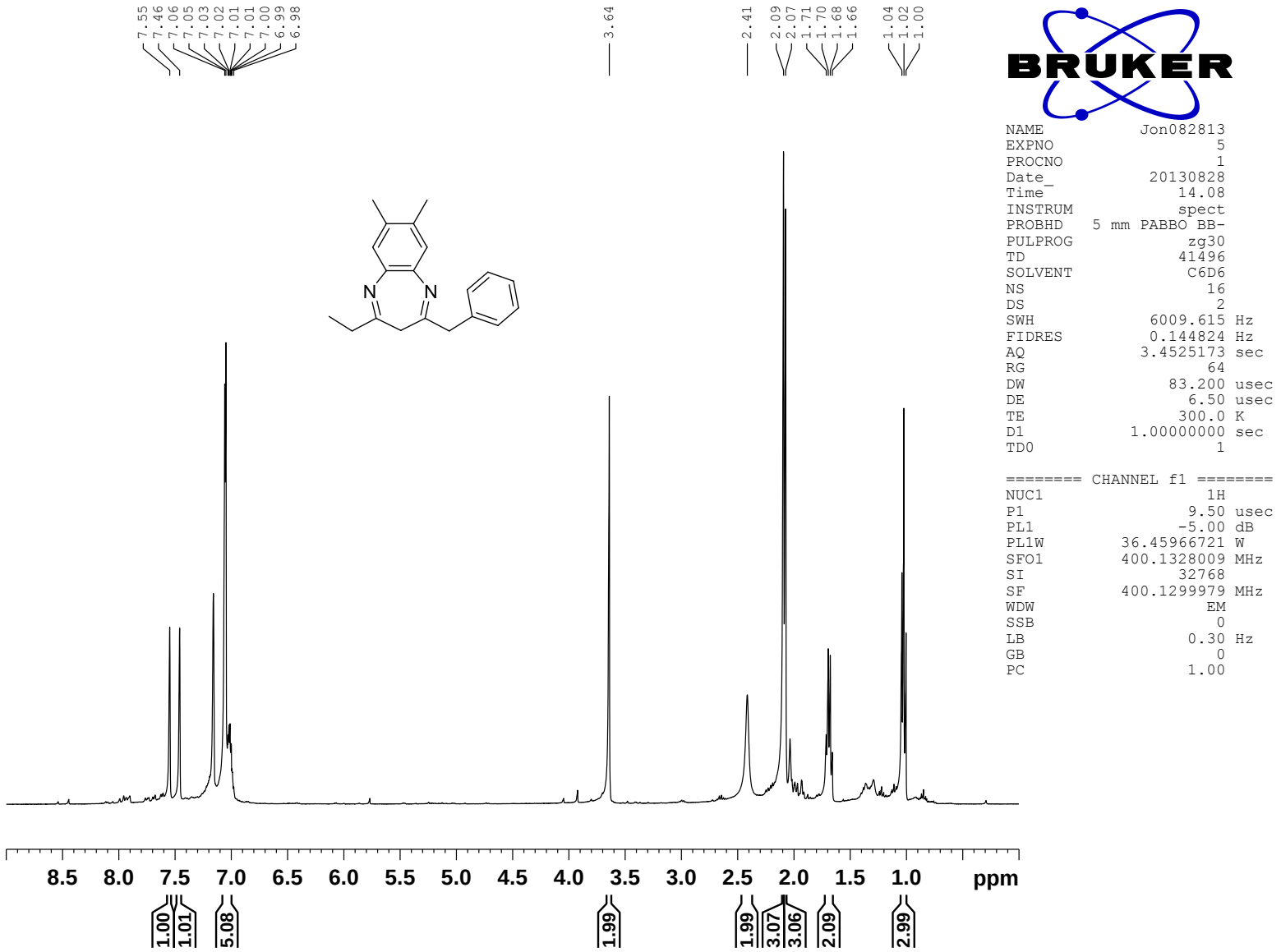

```
NAME        Agnes011513 13C
EXPNO                     1
PROCNO                    1
Date_              20130115
Time                  16.04
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      456
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           100.6127687 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **3db** (C_6D_6)

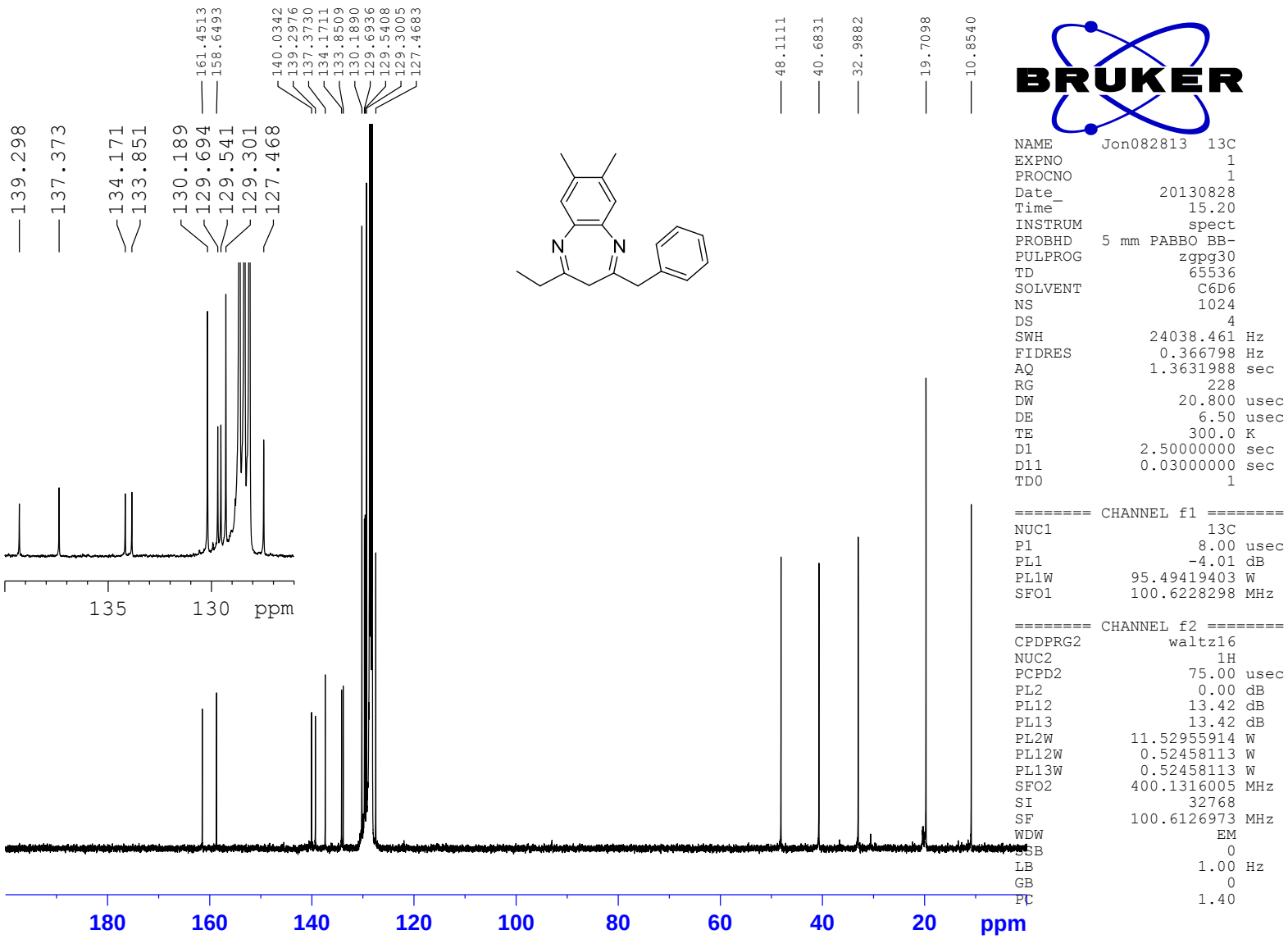


BRUKER

NAME	Jon082813
EXPNO	5
PROCNO	1
Date_	20130828
Time	14.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	41496
SOLVENT	C6D6
NS	16
DS	2
SWH	6009.615 Hz
FIDRES	0.144824 Hz
AQ	3.4525173 sec
RG	64
DW	83.200 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.50 usec
PL1	-5.00 dB
PL1W	36.45966721 W
SFO1	400.1328009 MHz
SI	32768
SF	400.1299979 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **3db** (C_6D_6)




```
NAME      Jon082813  13C
EXPNO                 1
PROCNO                1
Date_          20130828
Time_             15.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT             C6D6
NS                 1024
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               300.0 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         100.6126973 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

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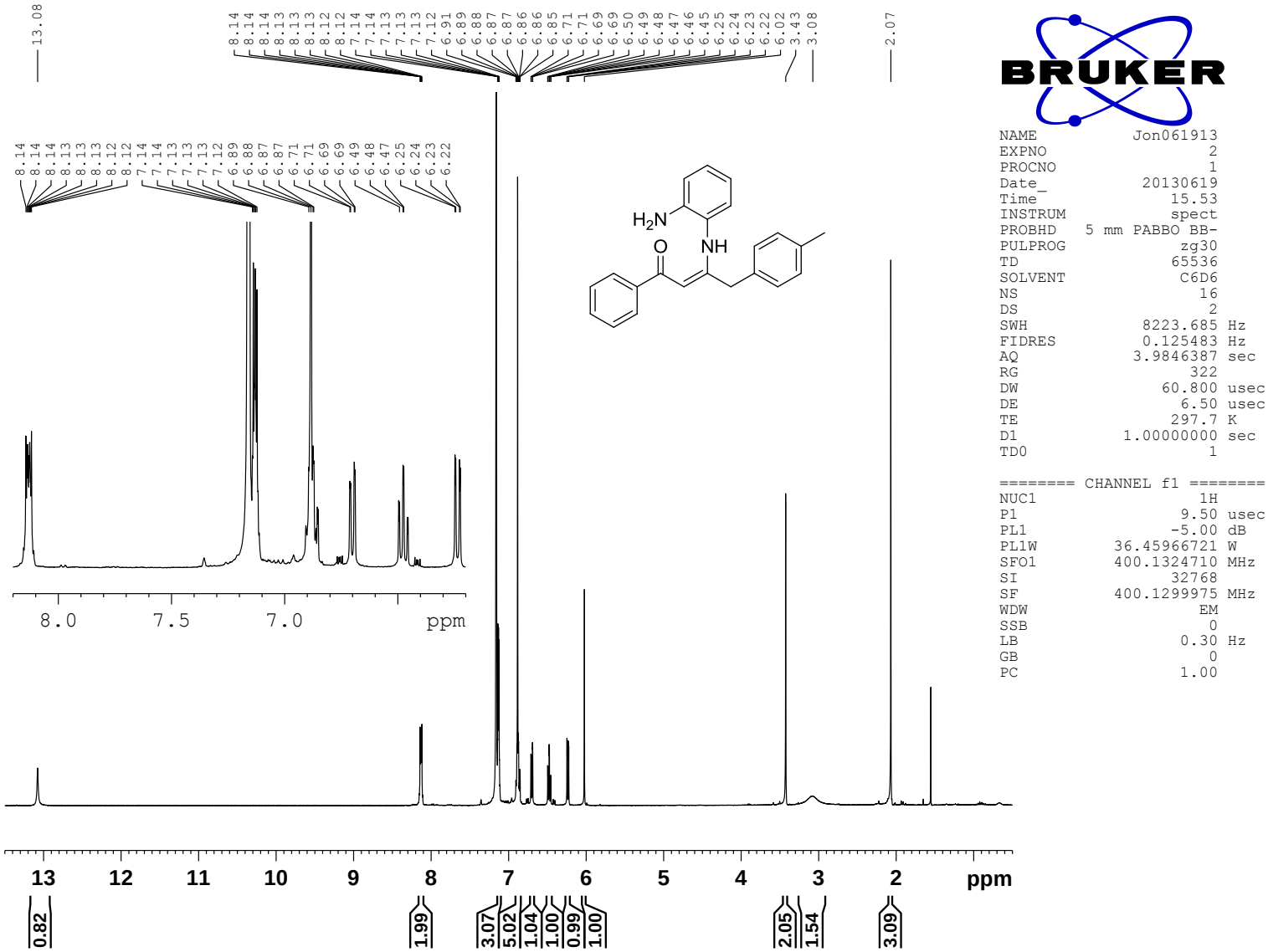
^{1}H NMR spectrum for **4aa** (C_6D_6)

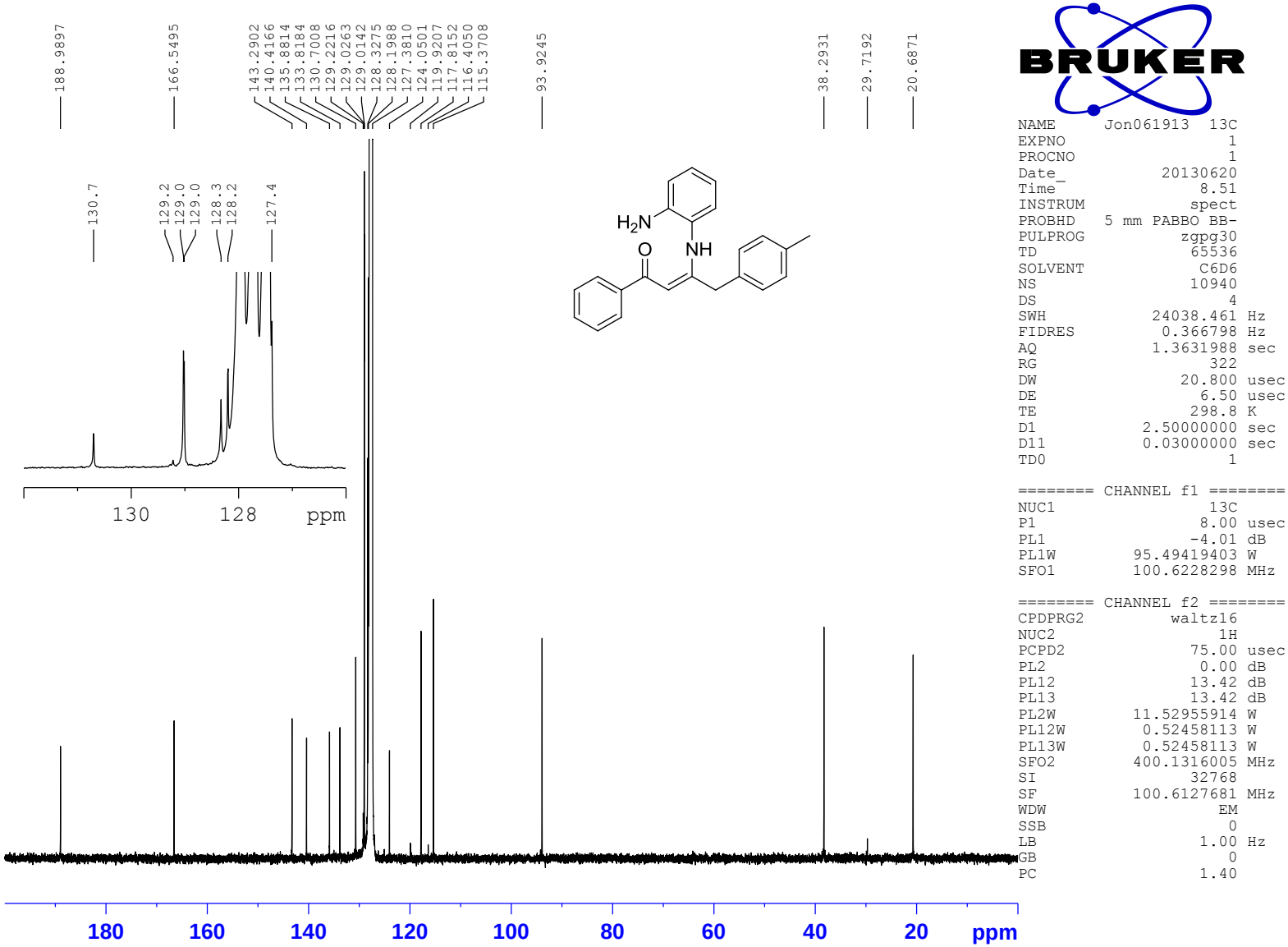



```
NAME                Jon061913
EXPNO                       2
PROCNO                      1
Date_                20130619
Time                    15.53
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                C6D6
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                       322
DW                  60.800 usec
DE                   6.50 usec
TE                   297.7 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               400.1299975 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **4aa** (C_6D_6)




```
NAME        Jon061913  13C
EXPNO                  1
PROCNO                 1
Date_           20130620
Time                8.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             C6D6
NS                10940
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                298.8 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          100.6127681 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

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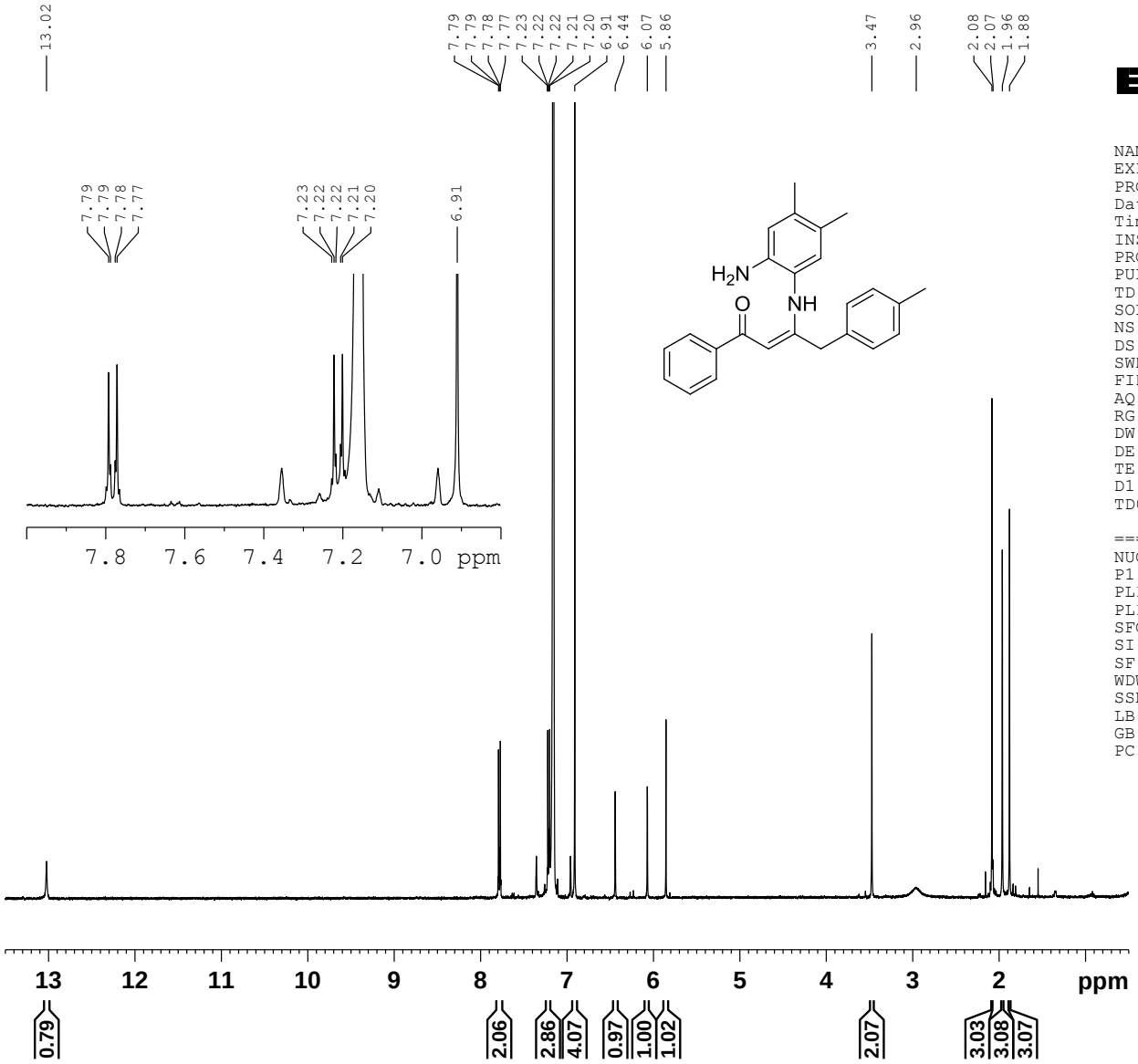
^{1}H NMR spectrum for **4ab** (C_6D_6)

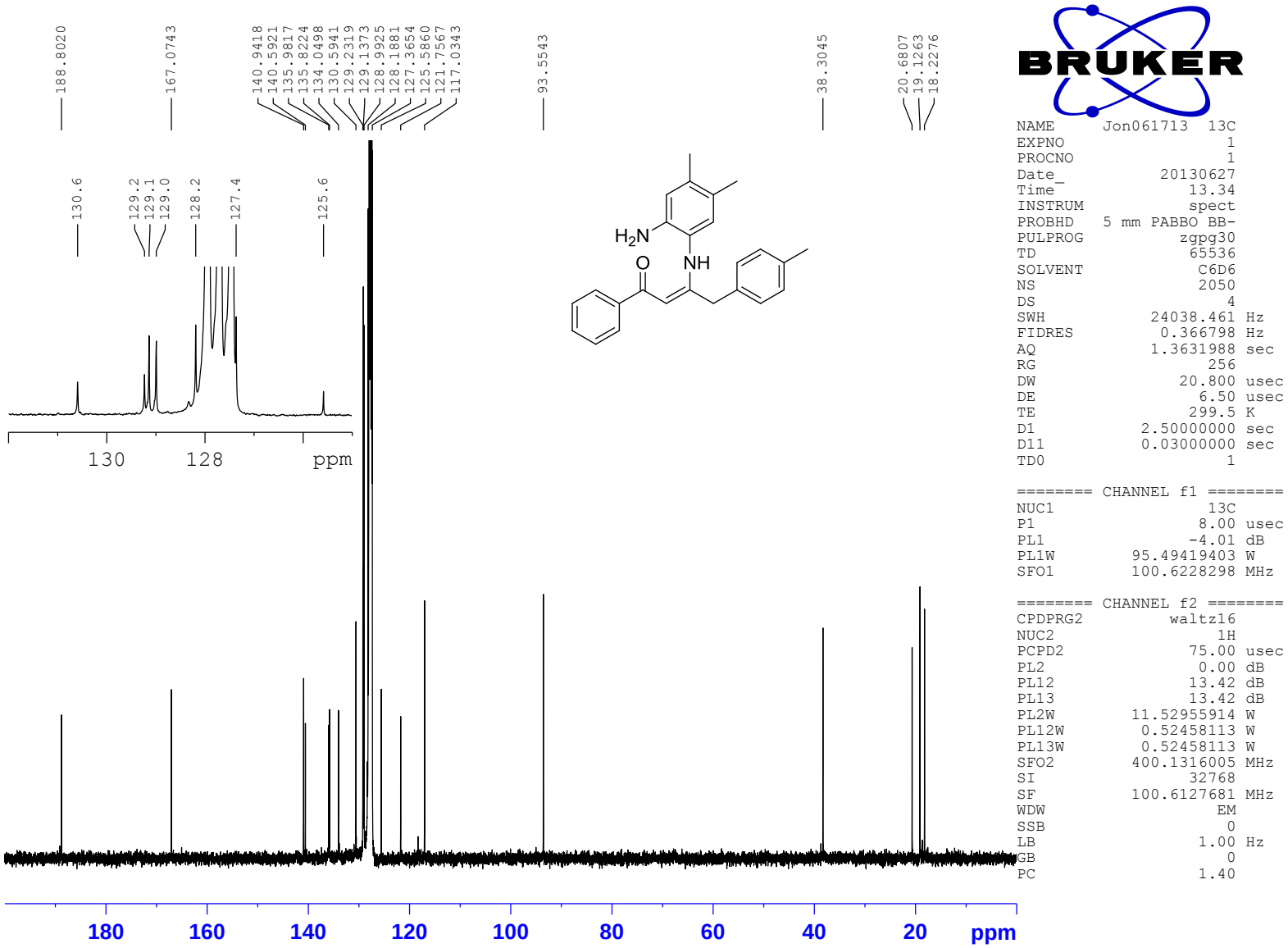



```
NAME                Jon061413
EXPNO                       1
PROCNO                      1
Date_                20130614
Time                    12.42
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                C6D6
NS                       16
DS                        2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                3.9846387 sec
RG                       645
DW                    60.800 usec
DE                     6.50 usec
TE                    296.6 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              400.1299979 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **4ab** (C_6D_6)



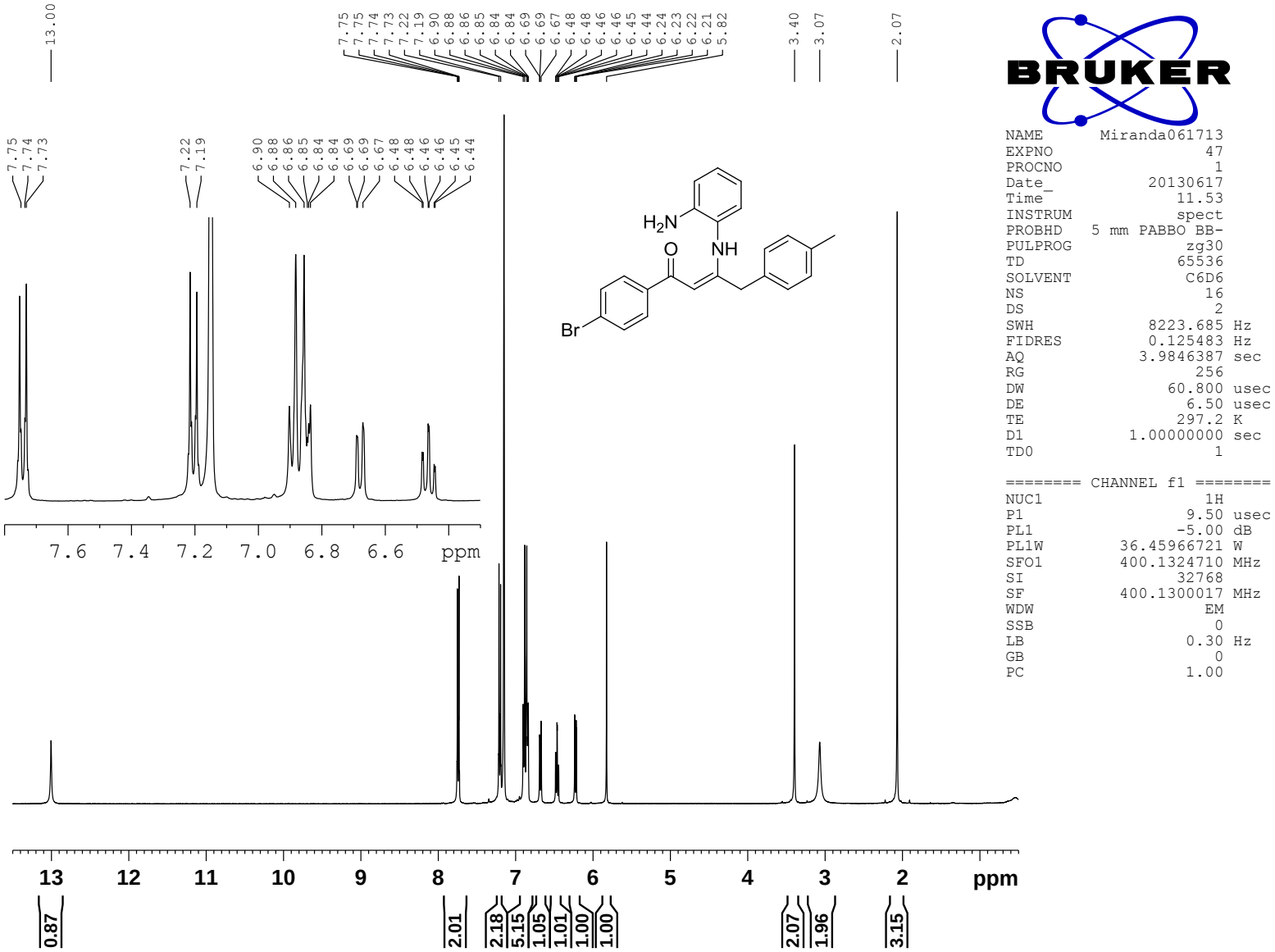

```
NAME          Jon061713  13C
EXPNO                    1
PROCNO                   1
Date_             20130627
Time                 13.34
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                C6D6
NS                     2050
DS                        4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                     256
DW                  20.800 usec
DE                   6.50 usec
TE                  299.5 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            100.6127681 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **4ba** (C_6D_6)

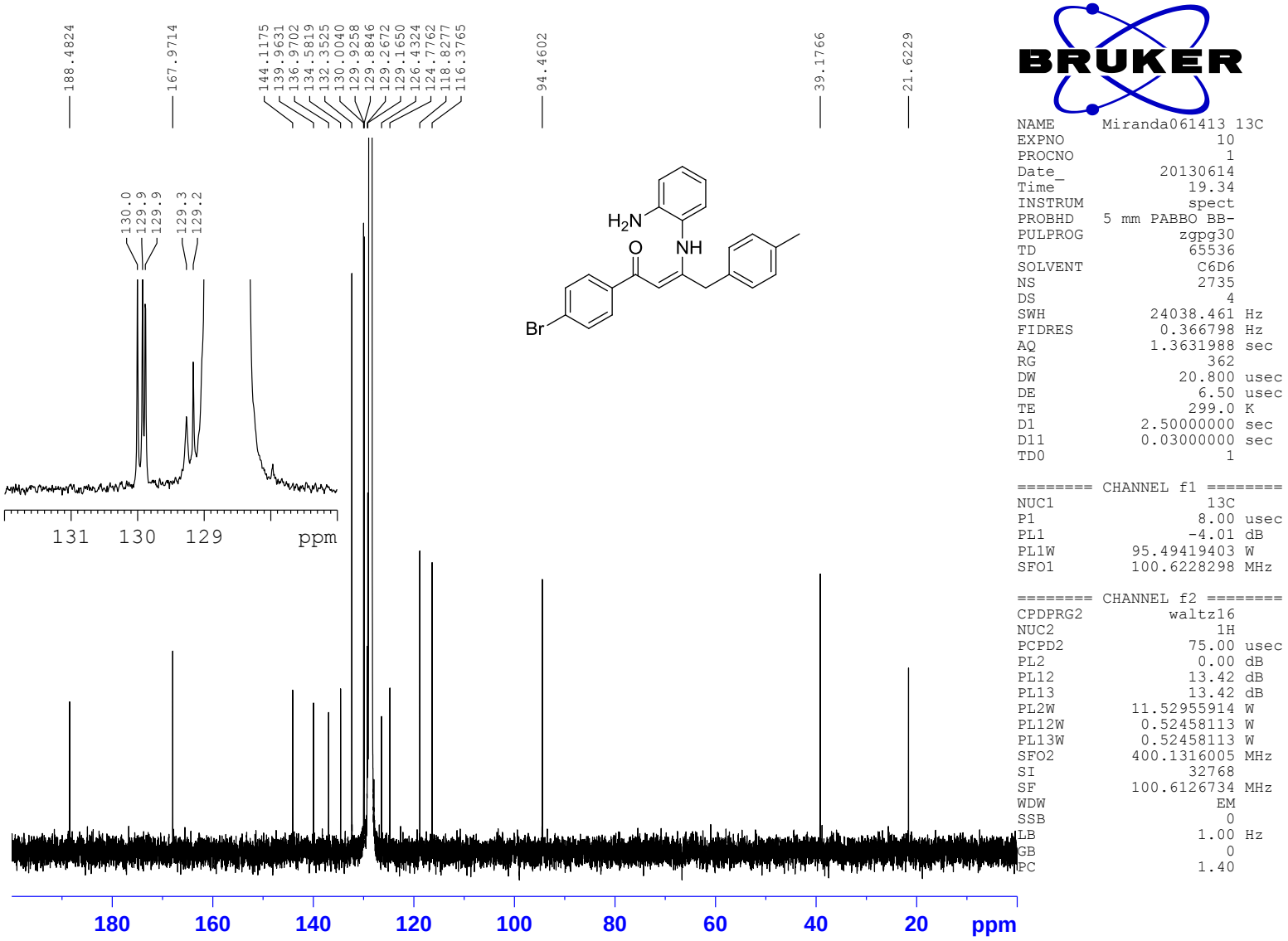



```
NAME          Miranda061713
EXPNO                    47
PROCNO                    1
Date_              20130617
Time_                 11.53
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                C6D6
NS                       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                   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             400.1300017 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **4ba** (C_6D_6)



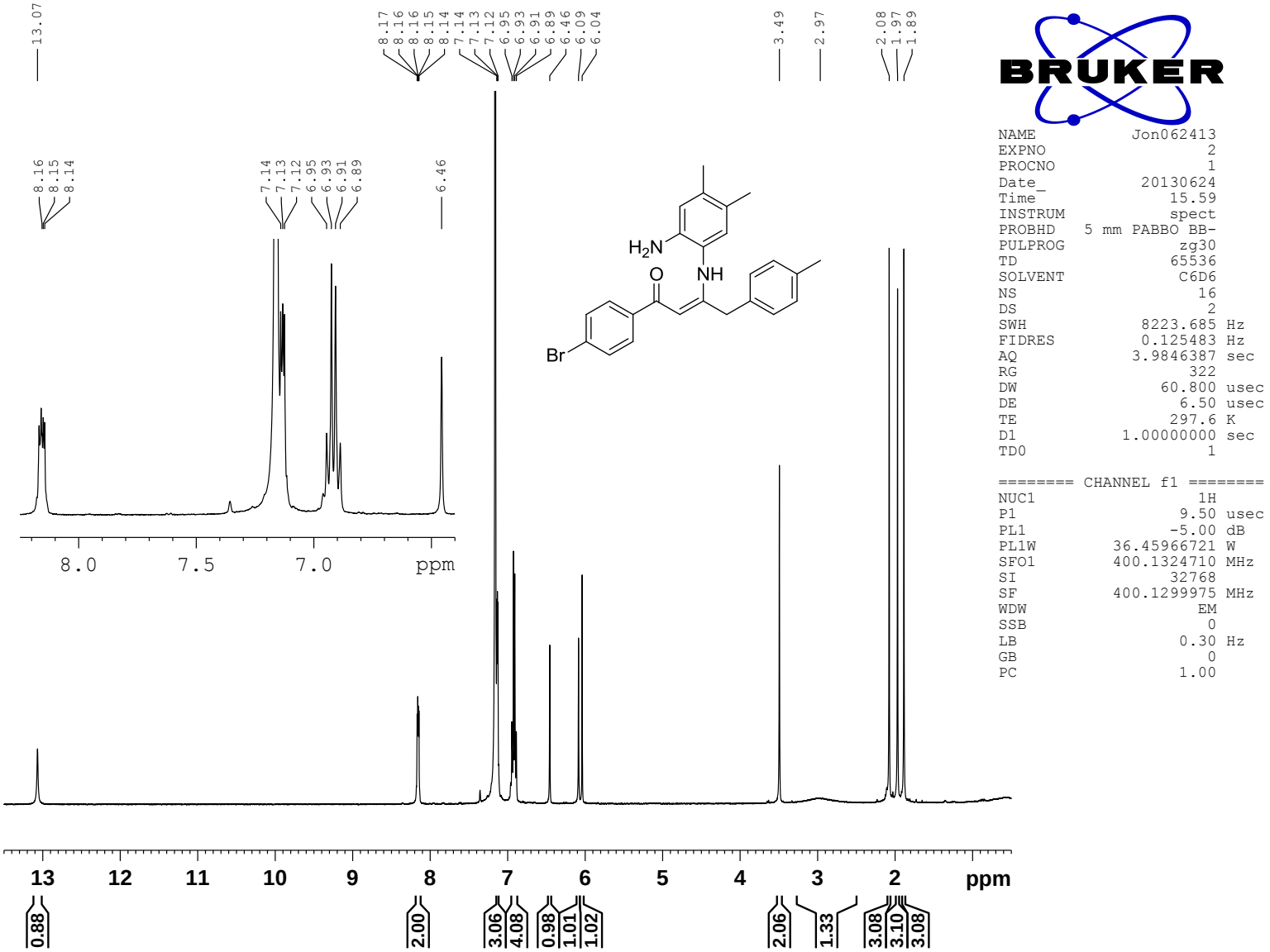

```
NAME        Miranda061413 13C
EXPNO                  10
PROCNO                  1
Date_            20130614
Time                19.34
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              C6D6
NS                   2735
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    362
DW                 20.800 usec
DE                  6.50 usec
TE                 299.0 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           100.6126734 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **4bb** (C_6D_6)

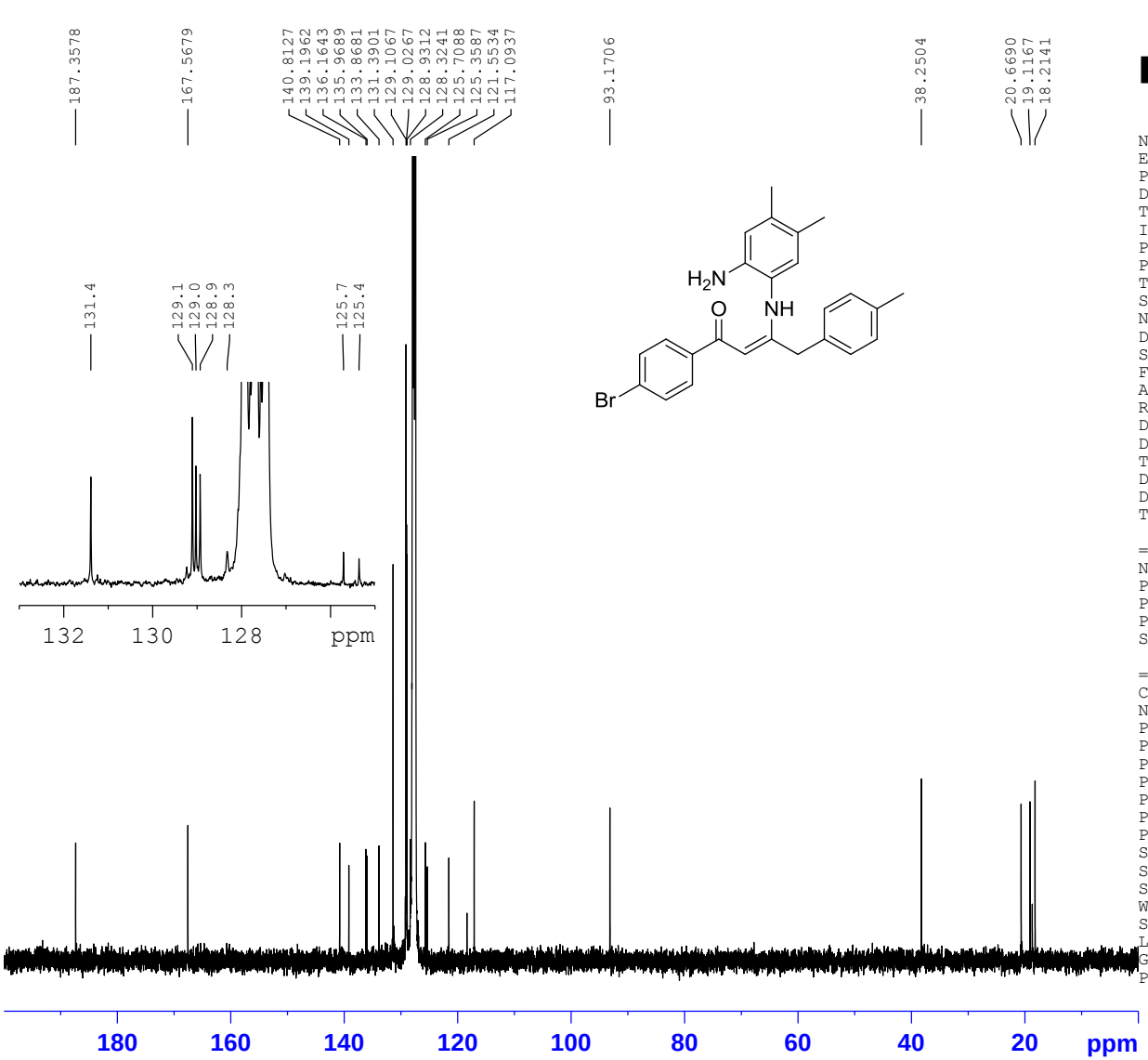



```
NAME                Jon062413
EXPNO                       2
PROCNO                      1
Date_                20130624
Time_                   15.59
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                C6D6
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                       322
DW                  60.800 usec
DE                   6.50 usec
TE                   297.6 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               400.1299975 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **4bb** (C_6D_6)




```
NAME        Jon062813  13C
EXPNO                1
PROCNO               1
Date_         20130628
Time_            12.14
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT           C6D6
NS                2050
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              300.0 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        100.6127681 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

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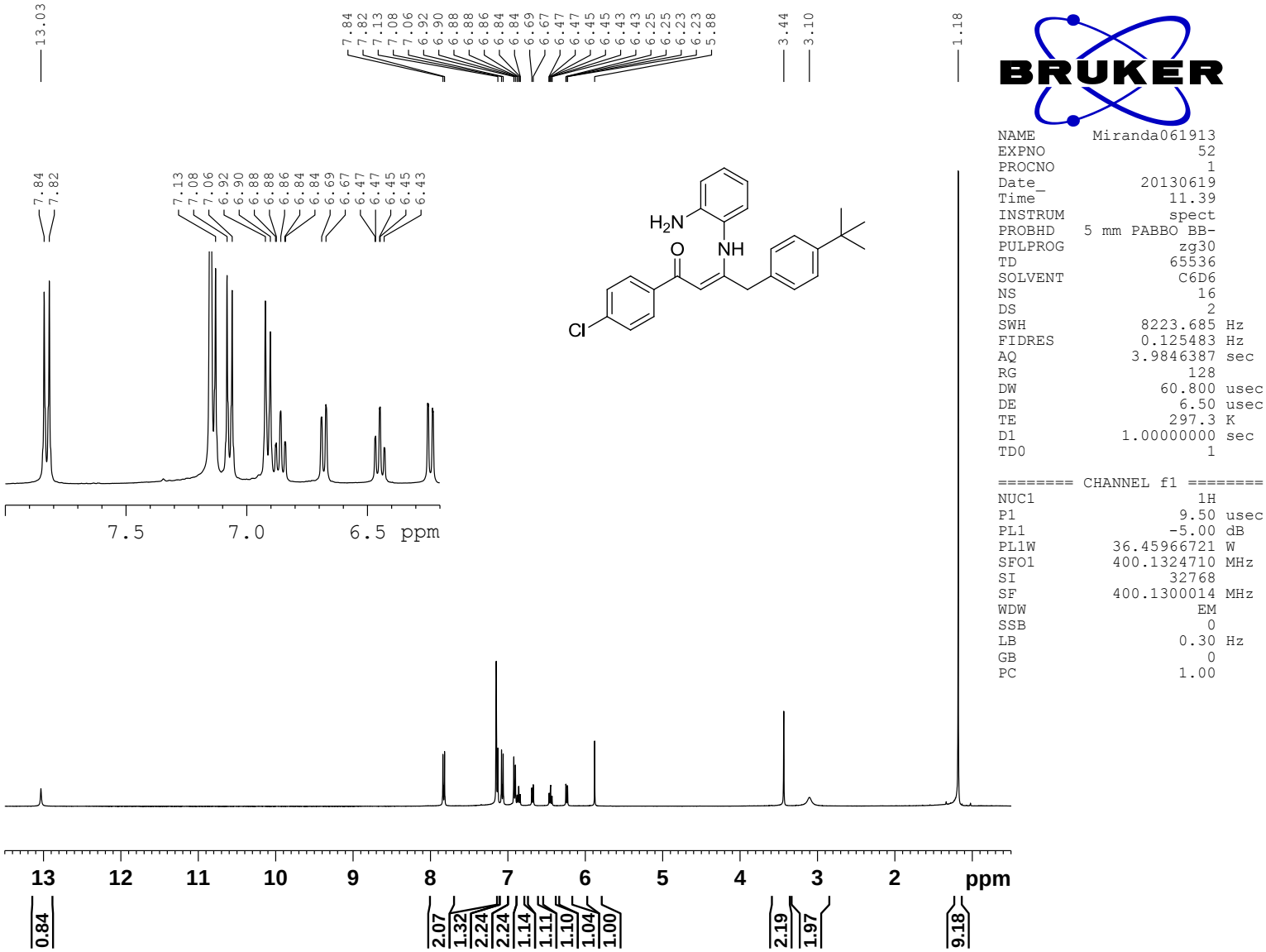
^{1}H NMR spectrum for **4ca** (C_6D_6)




```
NAME          Miranda061913
EXPNO                    52
PROCNO                    1
Date_              20130619
Time                  11.39
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                C6D6
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                   297.3 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             400.1300014 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

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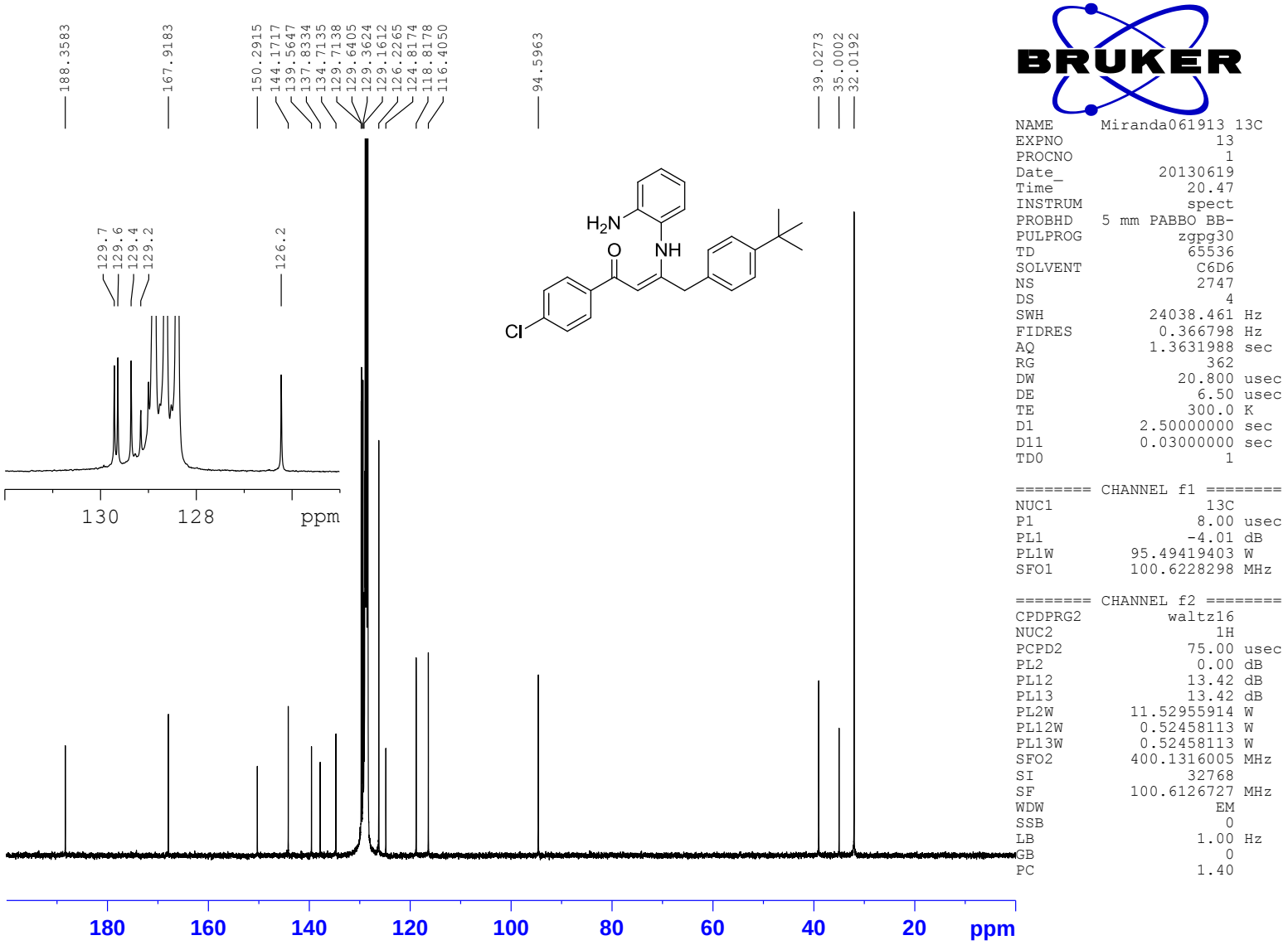
^{13}C NMR spectrum for **4ca** (C_6D_6)



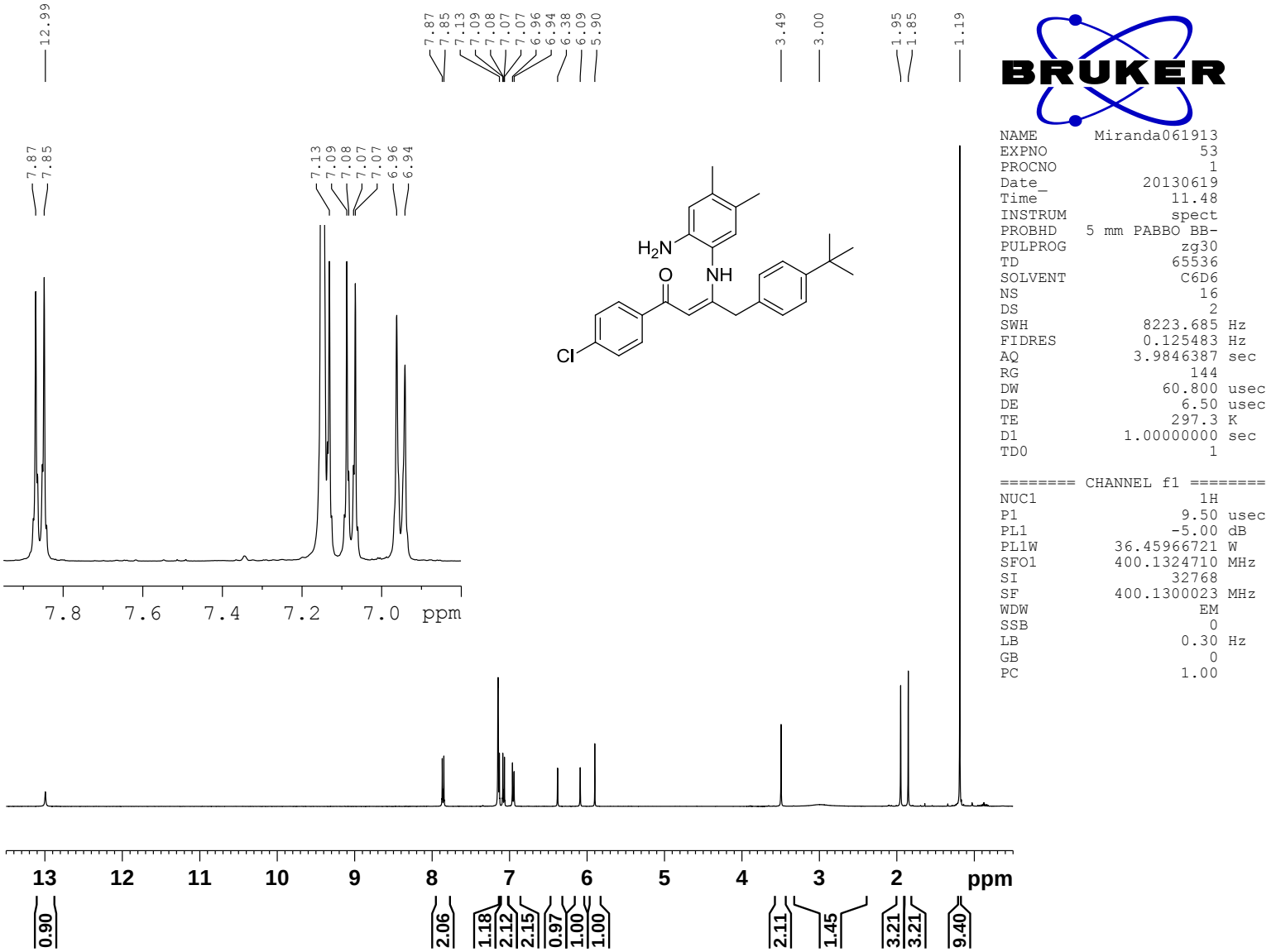

```
NAME        Miranda061913 13C
EXPNO                    13
PROCNO                    1
Date_              20130619
Time                  20.47
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                C6D6
NS                     2747
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631988 sec
RG                      362
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 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            100.6126727 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **4cb** (C_6D_6)




```
NAME        Miranda061913
EXPNO              53
PROCNO             1
Date_          20130619
Time_             11.48
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT           C6D6
NS                16
DS                 2
SWH          8223.685 Hz
FIDRES       0.125483 Hz
AQ           3.9846387 sec
RG               144
DW              60.800 usec
DE               6.50 usec
TE              297.3 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         400.1300023 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00
```

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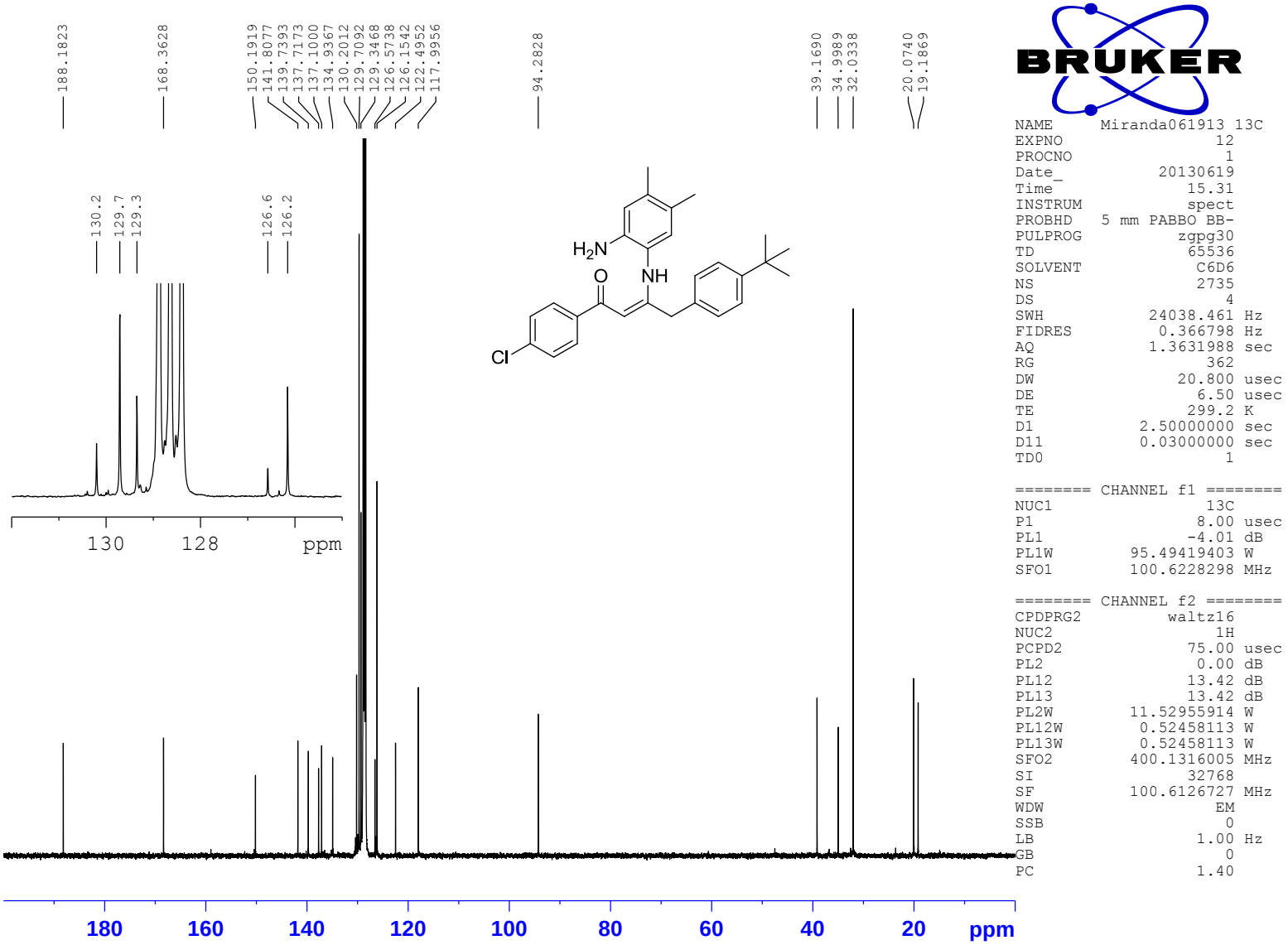
^{13}C NMR spectrum for **4cb**(C_6D_6)



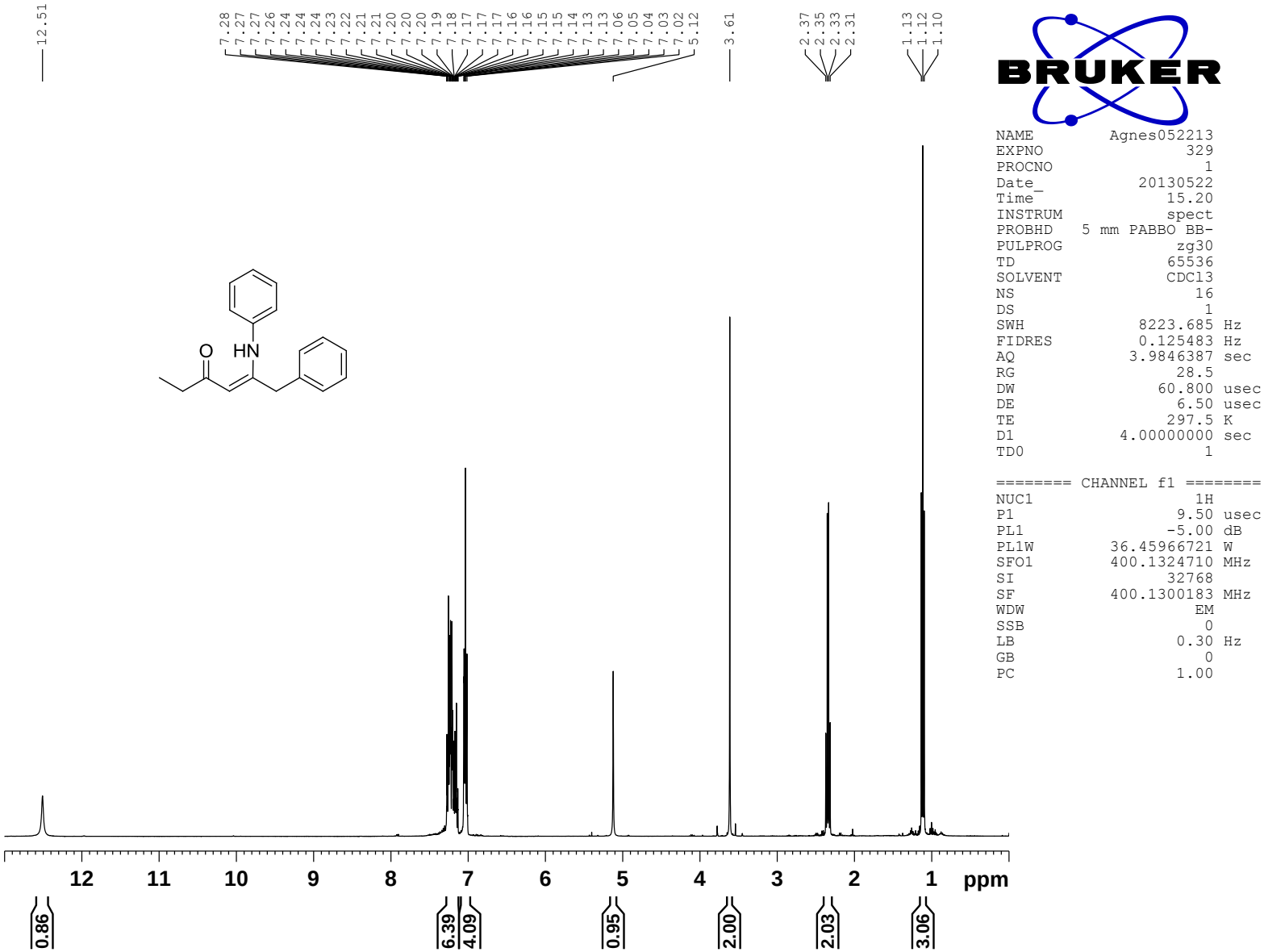

```
NAME        Miranda061913 13C
EXPNO                     12
PROCNO                     1
Date_               20130619
Time                   15.31
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 C6D6
NS                      2735
DS                         4
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                       362
DW                    20.800 usec
DE                     6.50 usec
TE                    299.2 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               100.6126727 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                     1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **6a** ($CDCl_3$)



BRUKER

NAME	Agnes052213
EXPNO	329
PROCNO	1
Date_	20130522
Time	15.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	1
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	28.5
DW	60.800 usec
DE	6.50 usec
TE	297.5 K
D1	4.00000000 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	400.1300183 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

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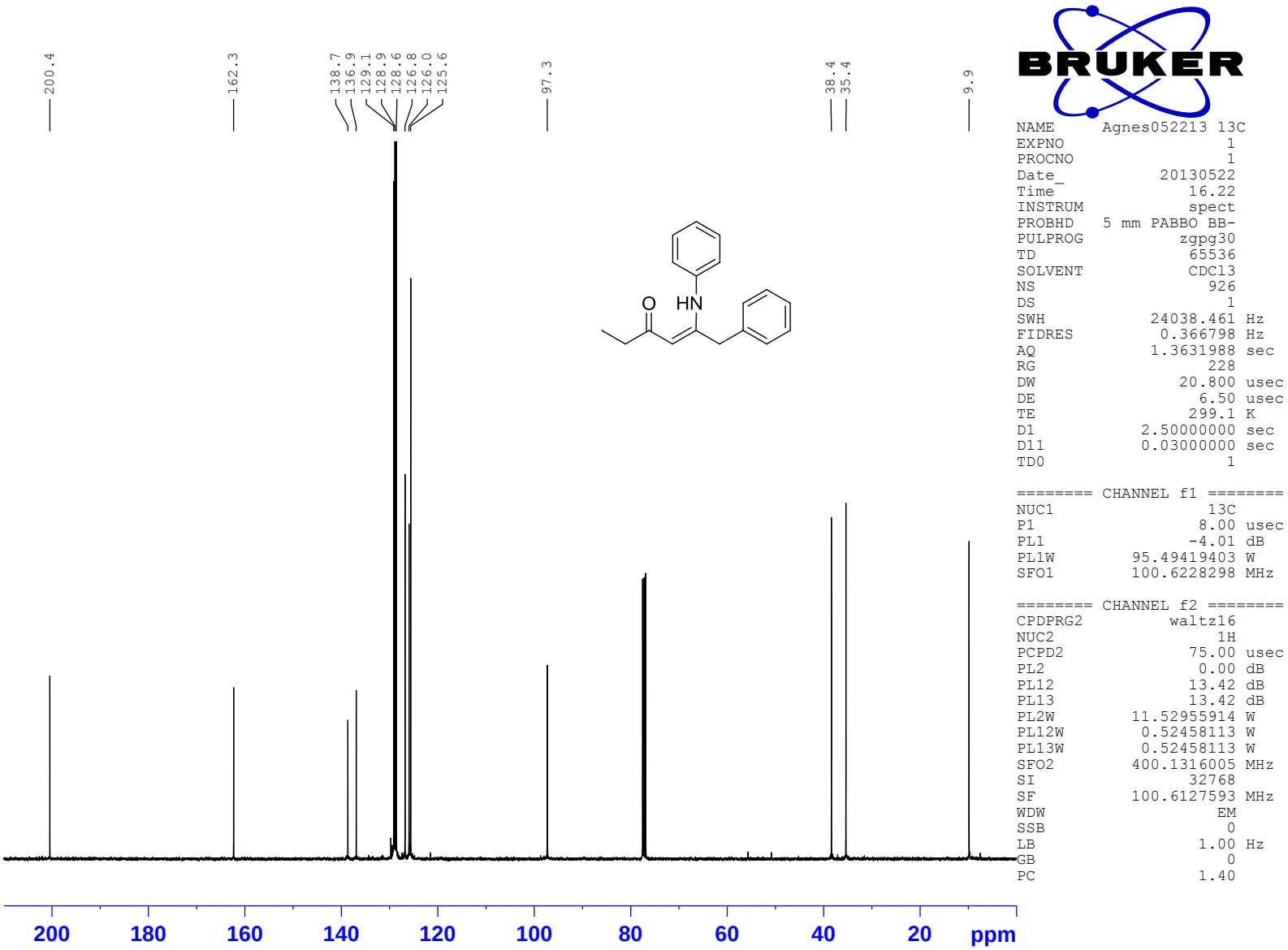
^{13}C NMR spectrum for **6a** ($CDCl_3$)



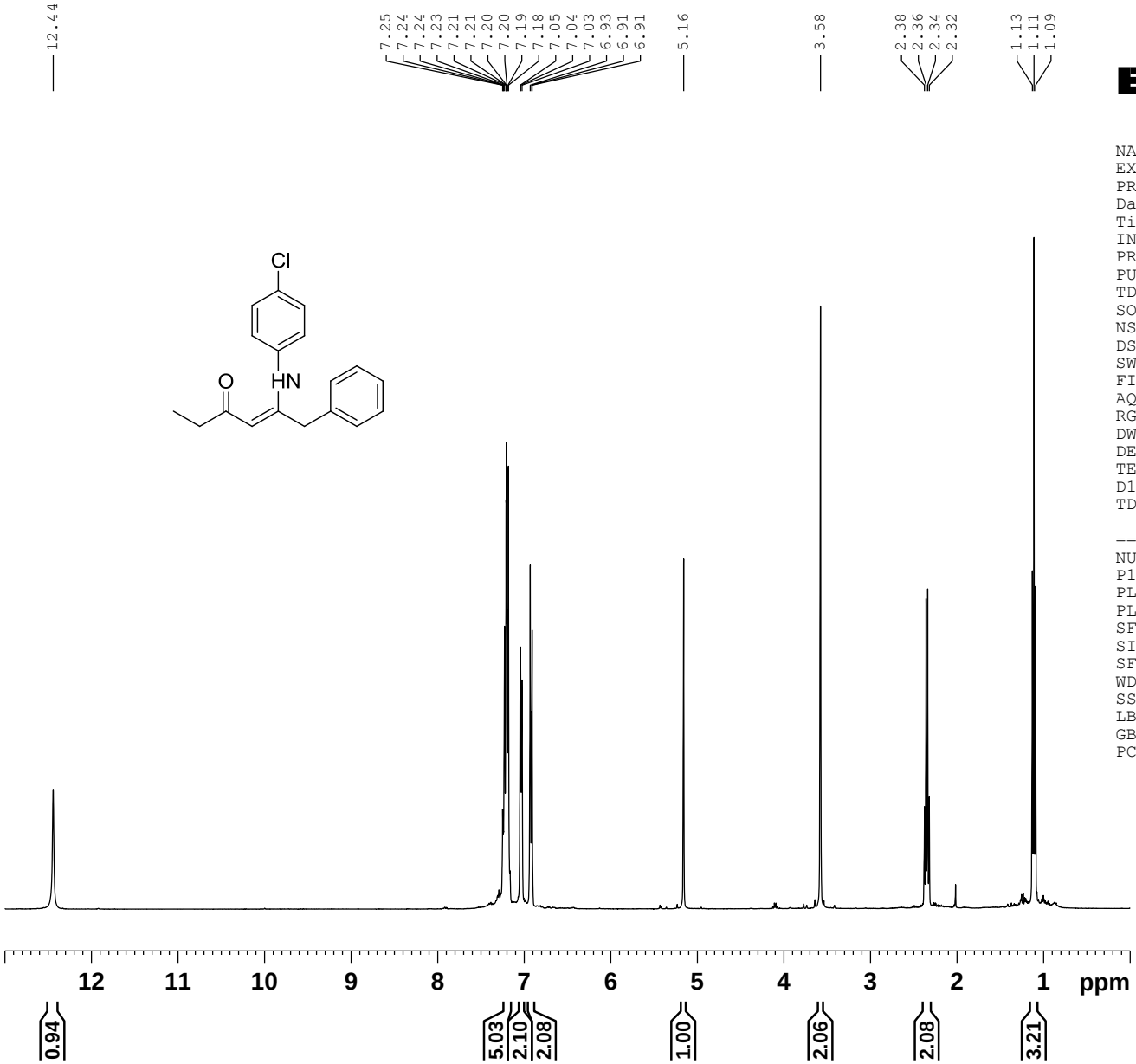

```
NAME      Agnes052213 13C
EXPNO                  1
PROCNO                 1
Date_           20130522
Time               16.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  926
DS                    1
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  228
DW               20.800 usec
DE                6.50 usec
TE               299.1 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         100.6127593 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **6b** ($CDCl_3$)

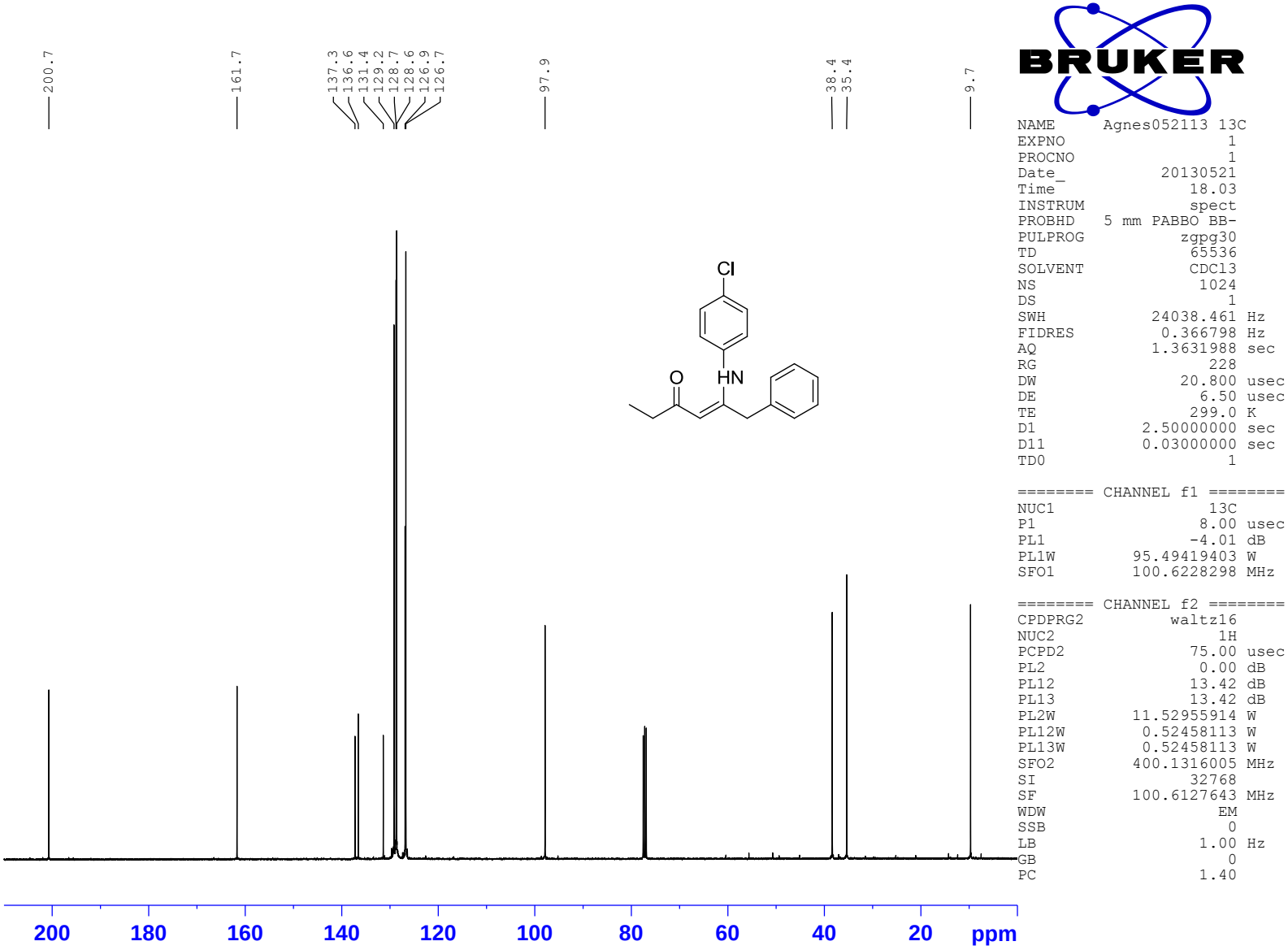



```
NAME            Amination
EXPNO                 328
PROCNO                  1
Date_            20130521
Time                16.54
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       1
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    28.5
DW                  60.800 usec
DE                   6.50 usec
TE                  297.4 K
D1             4.00000000 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            400.1300145 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **6b** ($CDCl_3$)



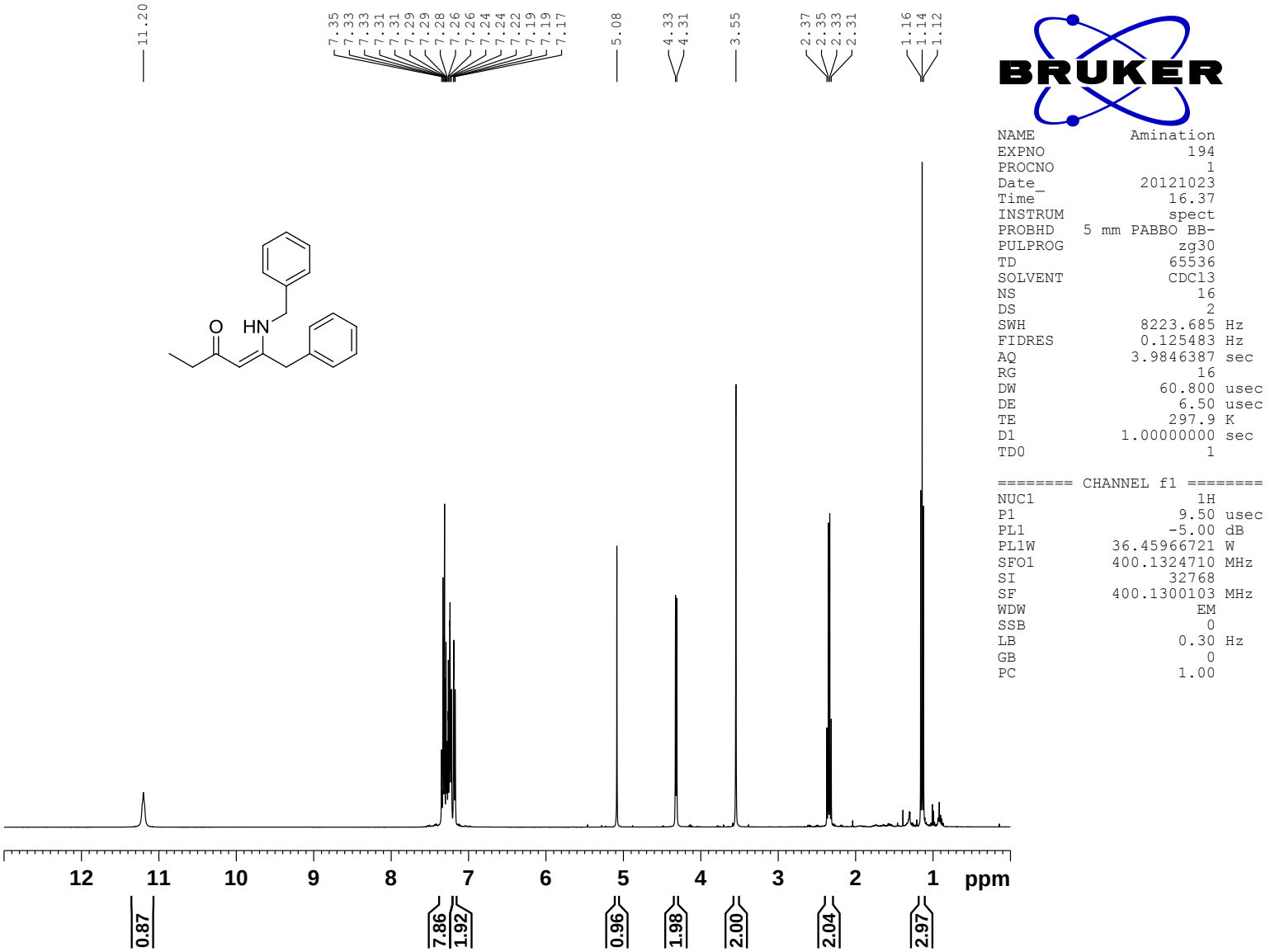

```
NAME        Agnes052113 13C
EXPNO                    1
PROCNO                   1
Date_             20130521
Time                 18.03
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       1
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                     228
DW                 20.800 usec
DE                  6.50 usec
TE                 299.0 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          100.6127643 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **6c** ($CDCl_3$)

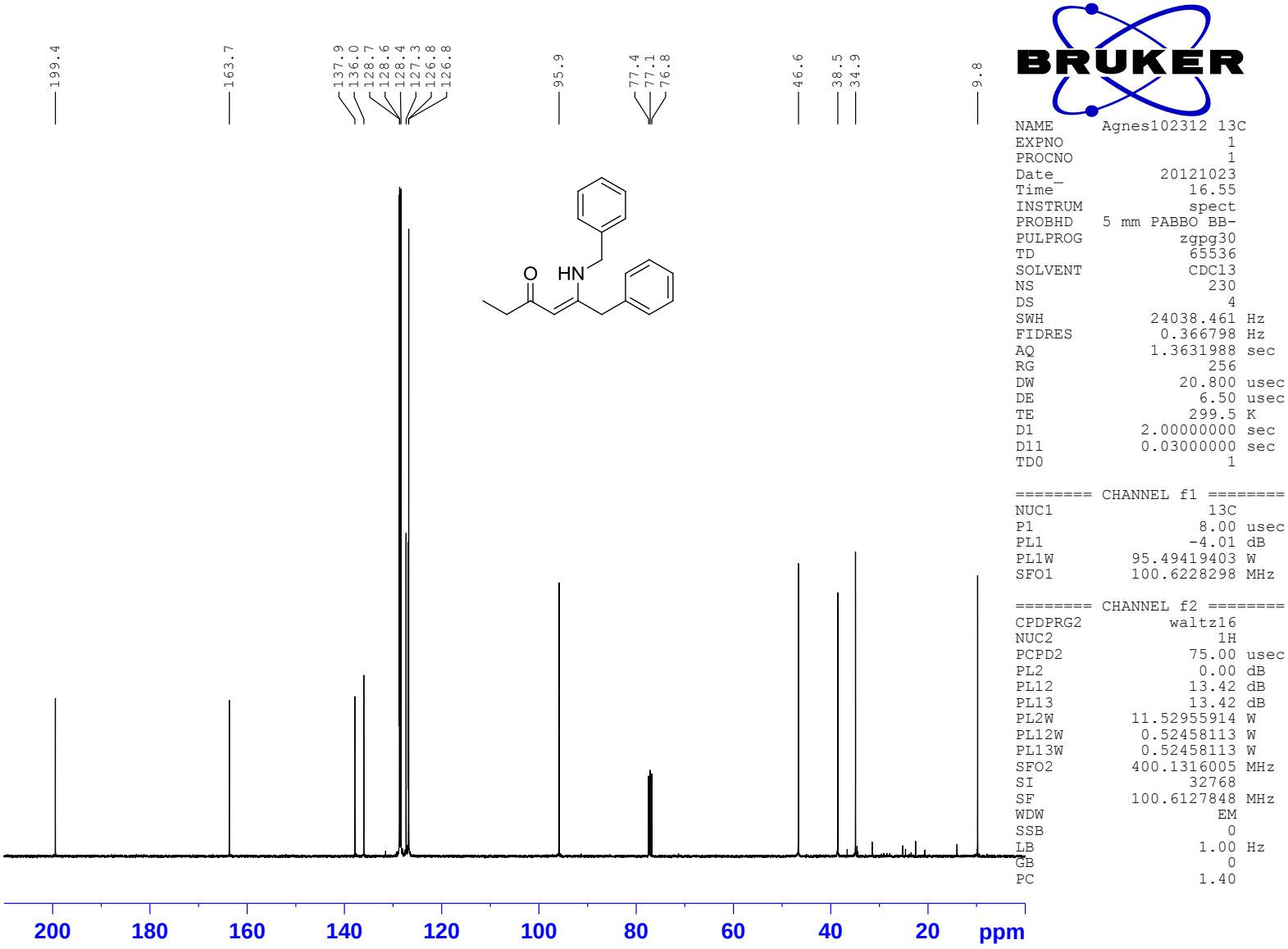



```
NAME              Amination
EXPNO                   194
PROCNO                    1
Date_              20121023
Time                  16.37
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       16
DW                   60.800 usec
DE                    6.50 usec
TE                   297.9 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             400.1300103 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **6c** ($CDCl_3$)



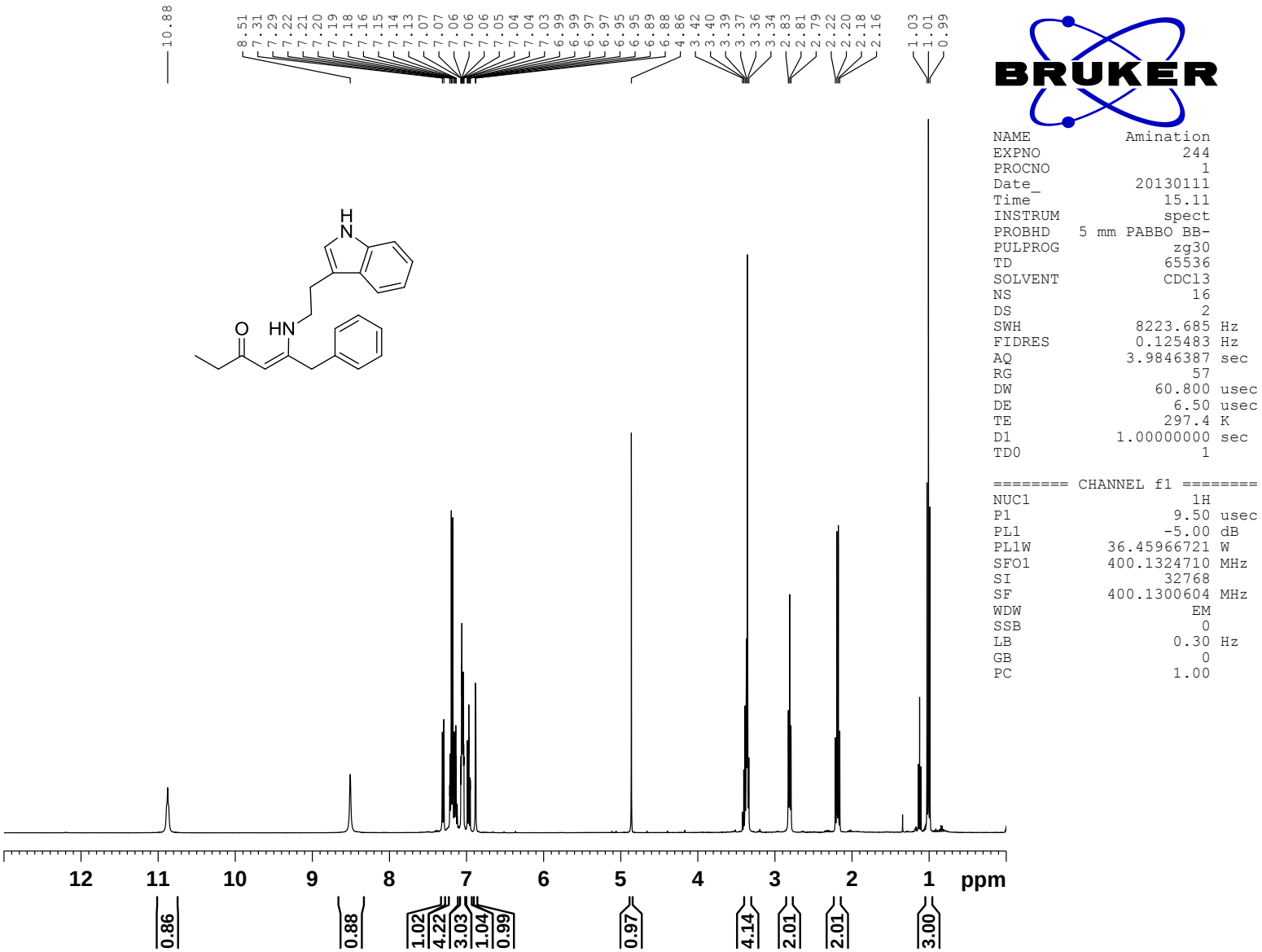

```
NAME        Agnes102312 13C
EXPNO                    1
PROCNO                   1
Date_             20121023
Time                 16.55
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   230
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   256
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          100.6127848 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **6d** ($CDCl_3$)



BRUKER

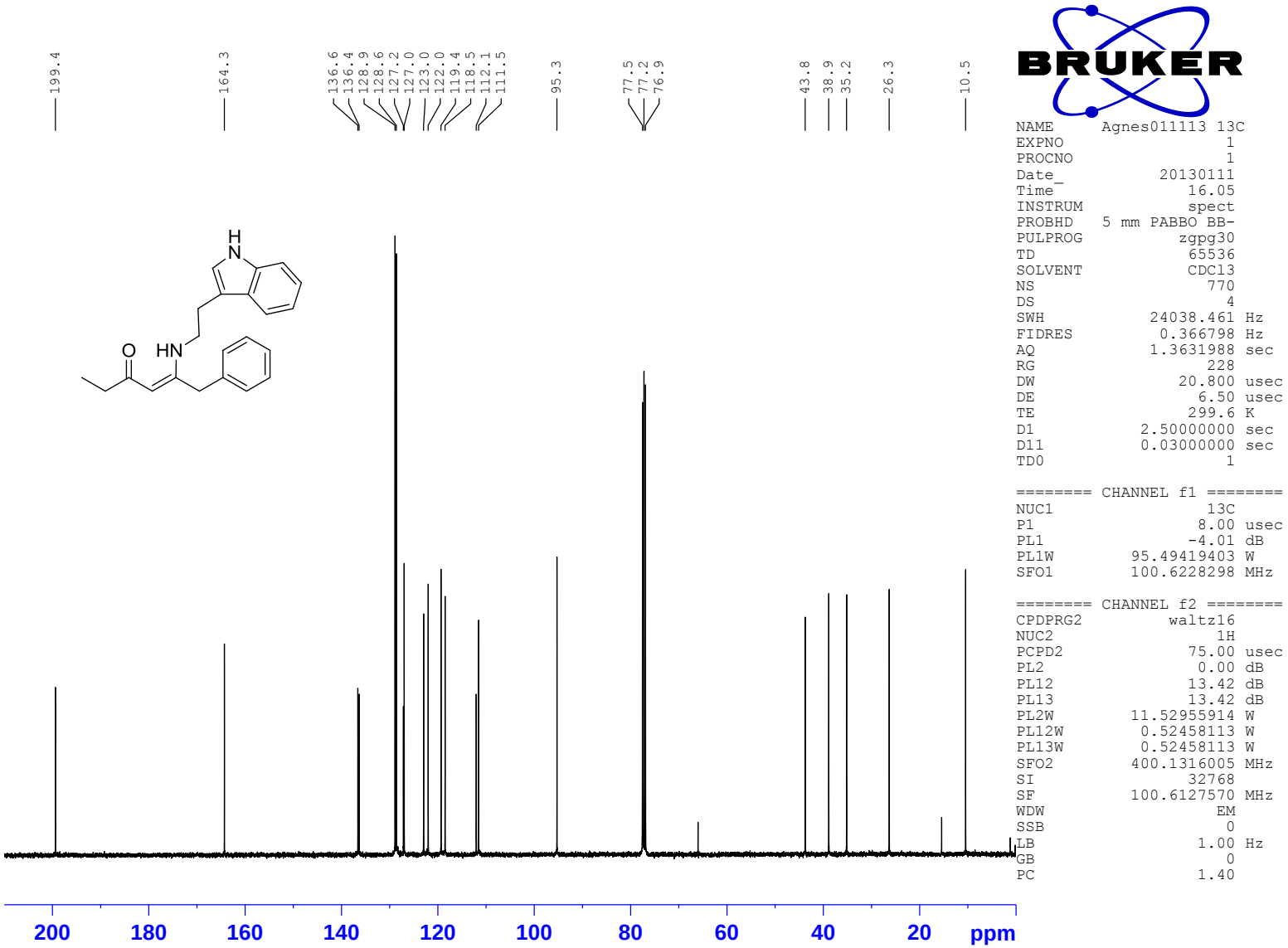
NAME	Amination
EXPNO	244
PROCNO	1
Date_	20130111
Time	15.11
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	57
DW	60.800 usec
DE	6.50 usec
TE	297.4 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	9.50 usec
PL1	-5.00 dB
PL1W	36.45966721 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300604 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **6d** ($CDCl_3$)



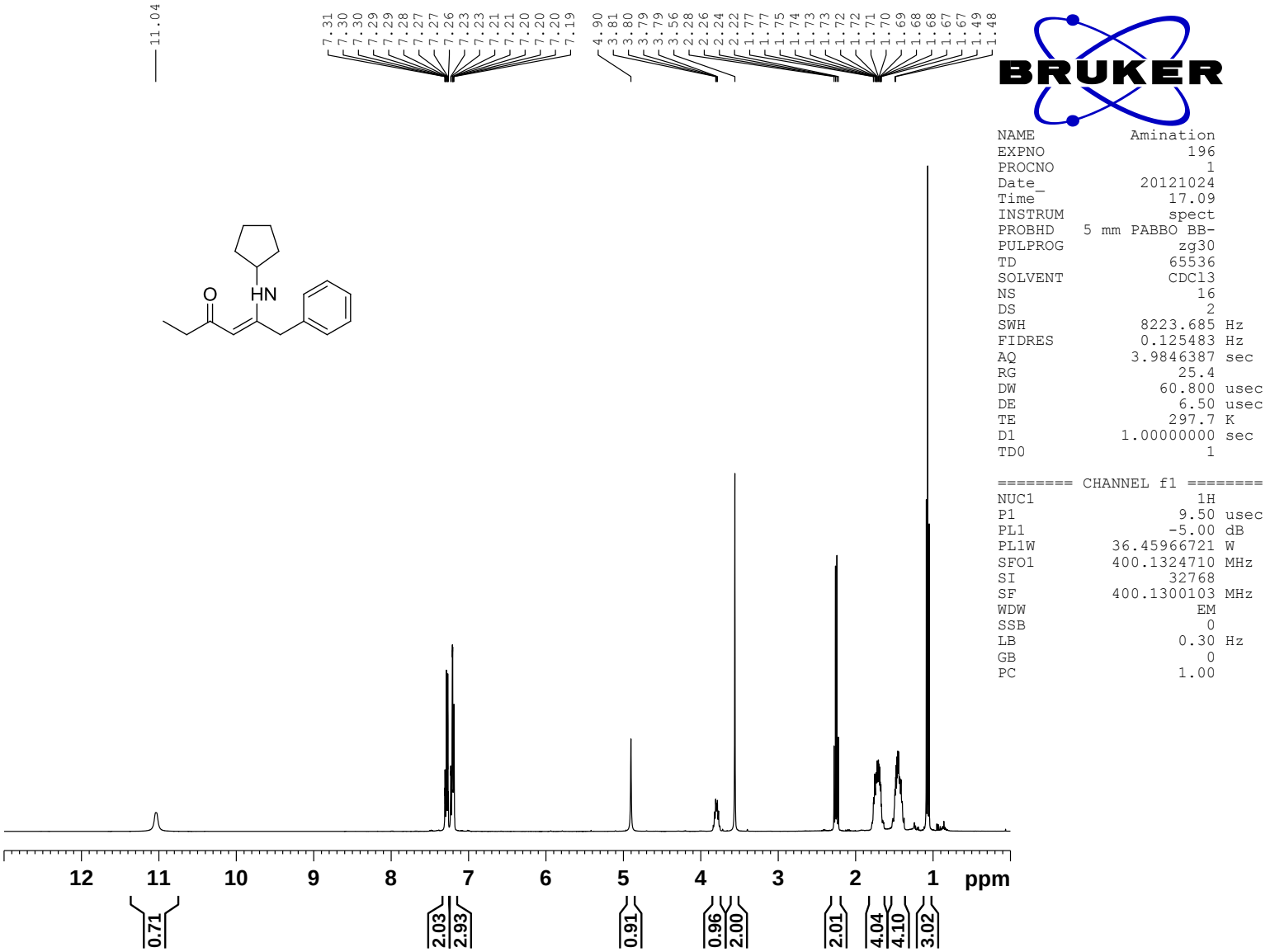

```
NAME        Agnes011113 13C
EXPNO                    1
PROCNO                   1
Date_             20130111
Time                 16.05
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     770
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                     228
DW                  20.800 usec
DE                   6.50 usec
TE                  299.6 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           100.6127570 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{1}H NMR spectrum for **6e** ($CDCl_3$)

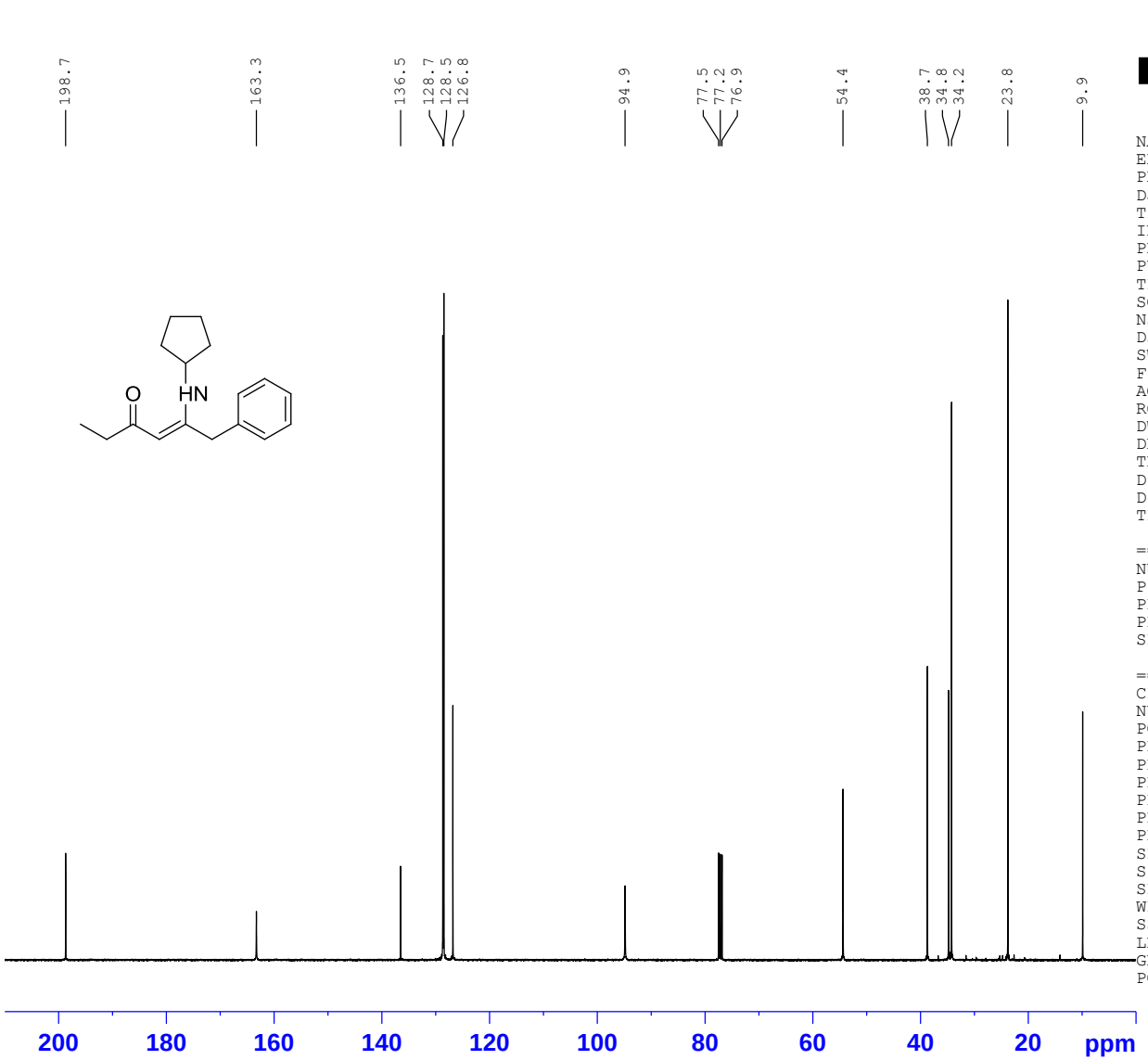



```
NAME              Amination
EXPNO                   196
PROCNO                    1
Date_              20121024
Time                  17.09
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      25.4
DW                    60.800 usec
DE                     6.50 usec
TE                    297.7 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             400.1300103 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **6e** ($CDCl_3$)




```
NAME        Agnes102412 13C
EXPNO                    1
PROCNO                   1
Date_             20121024
Time                 18.10
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                     256
DW                  20.800 usec
DE                   6.50 usec
TE                  299.7 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           100.6127676 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

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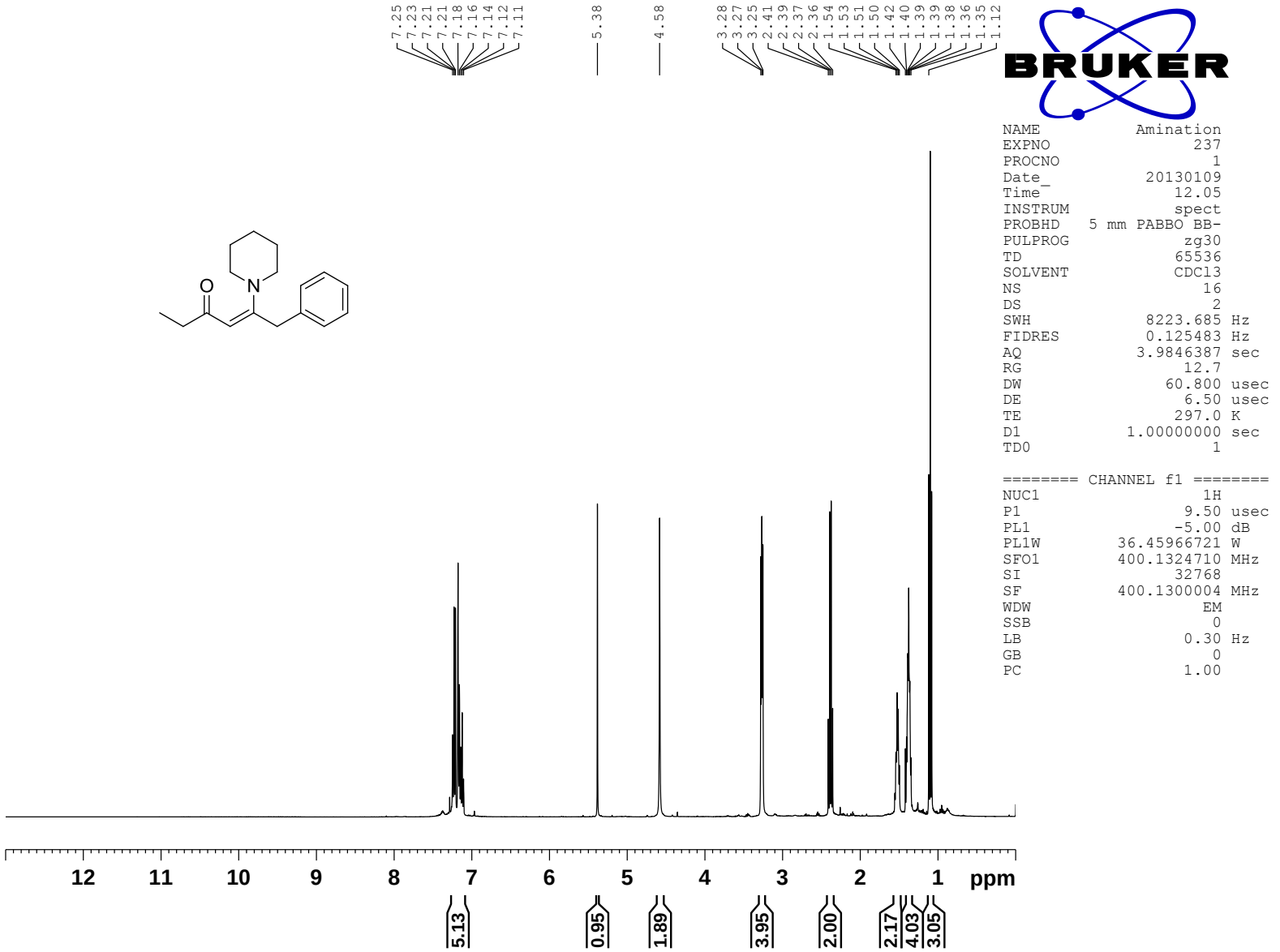
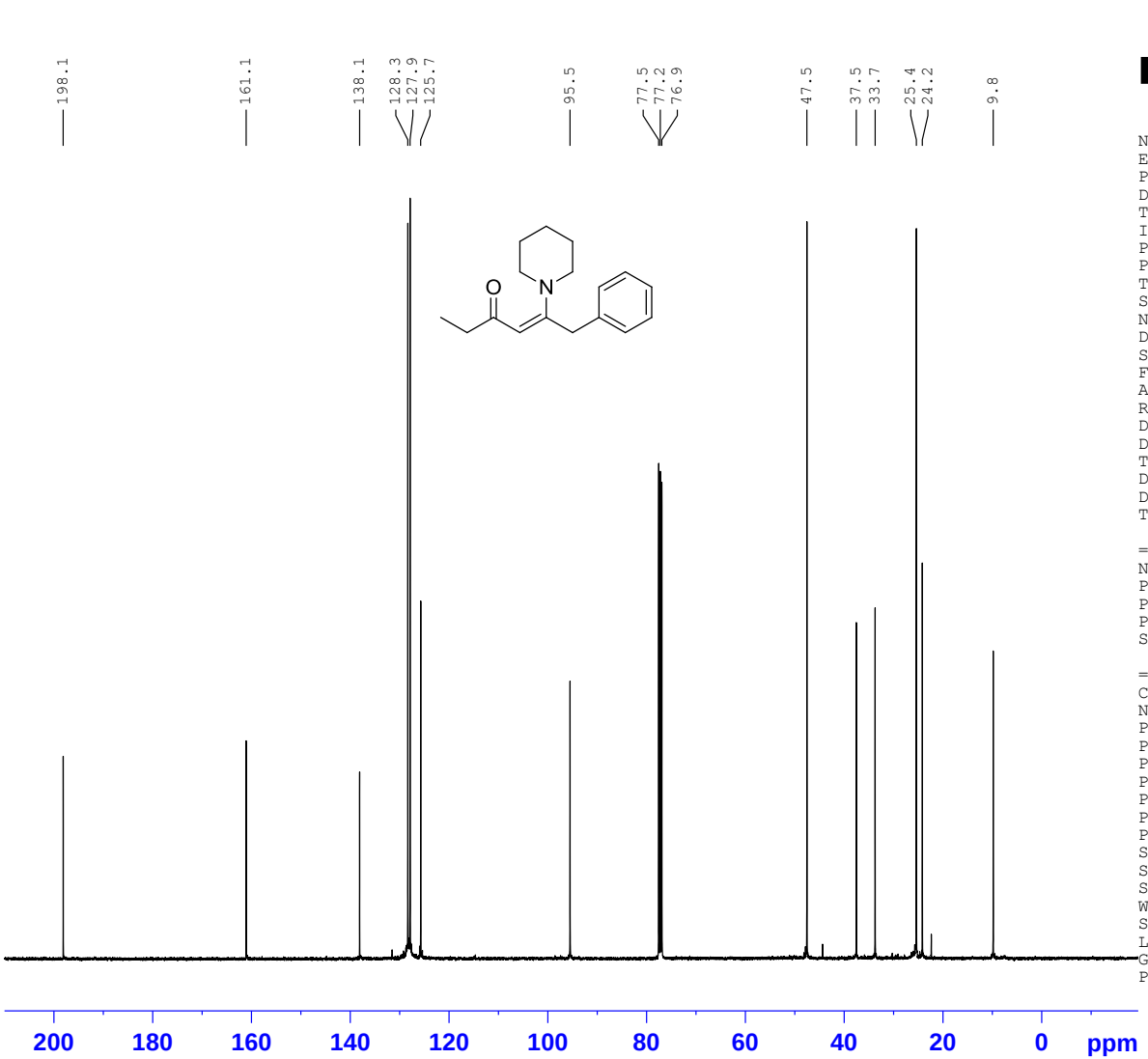
^{1}H NMR spectrum for **6f** ($CDCl_3$)



Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **6f** ($CDCl_3$)




```
NAME        Agnes110512 13C
EXPNO                    1
PROCNO                   1
Date_             20121105
Time                 17.13
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   556
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   256
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                   8.00 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           100.6127756 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

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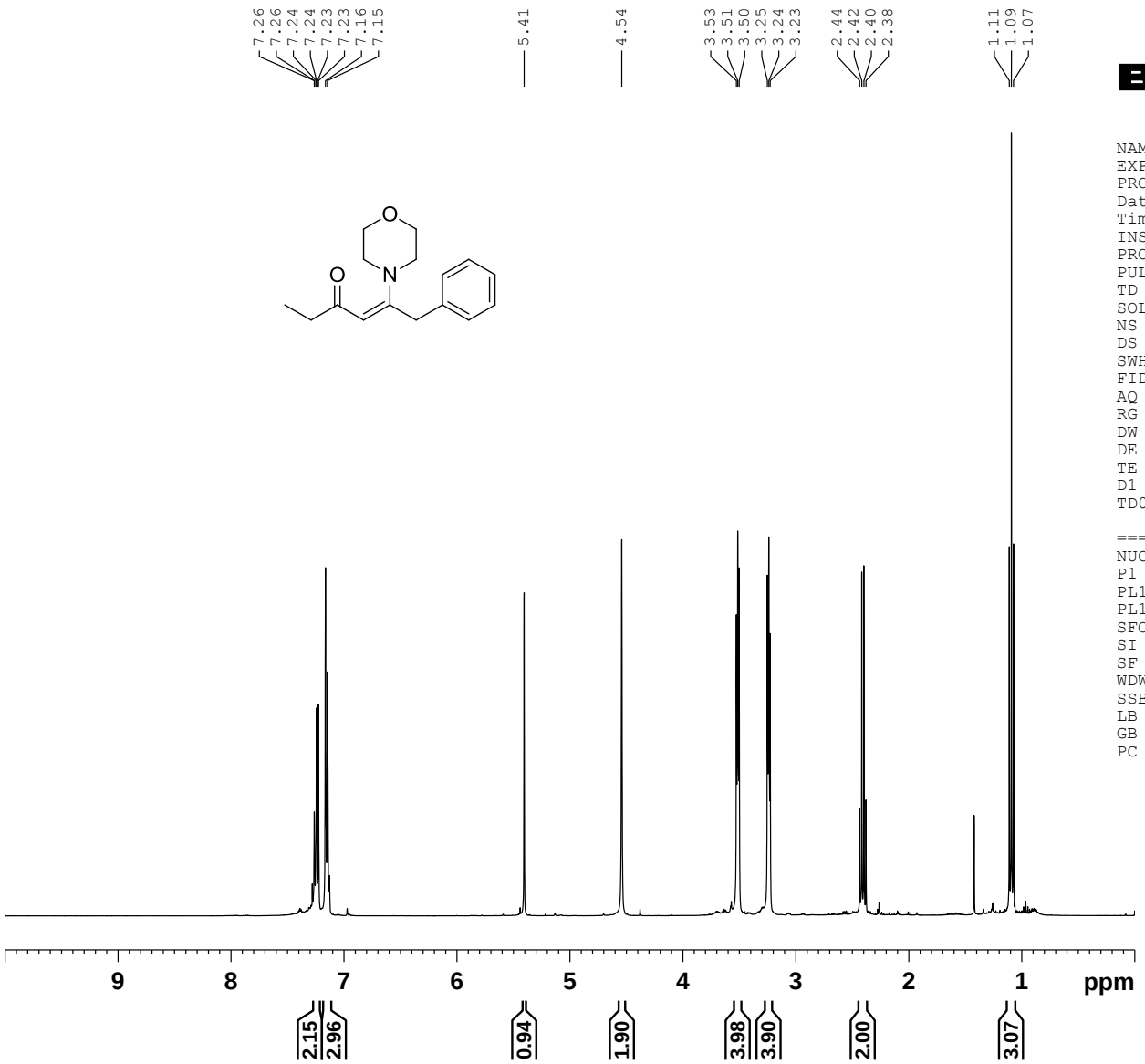
^{1}H NMR spectrum for **6g** ($CDCl_3$)

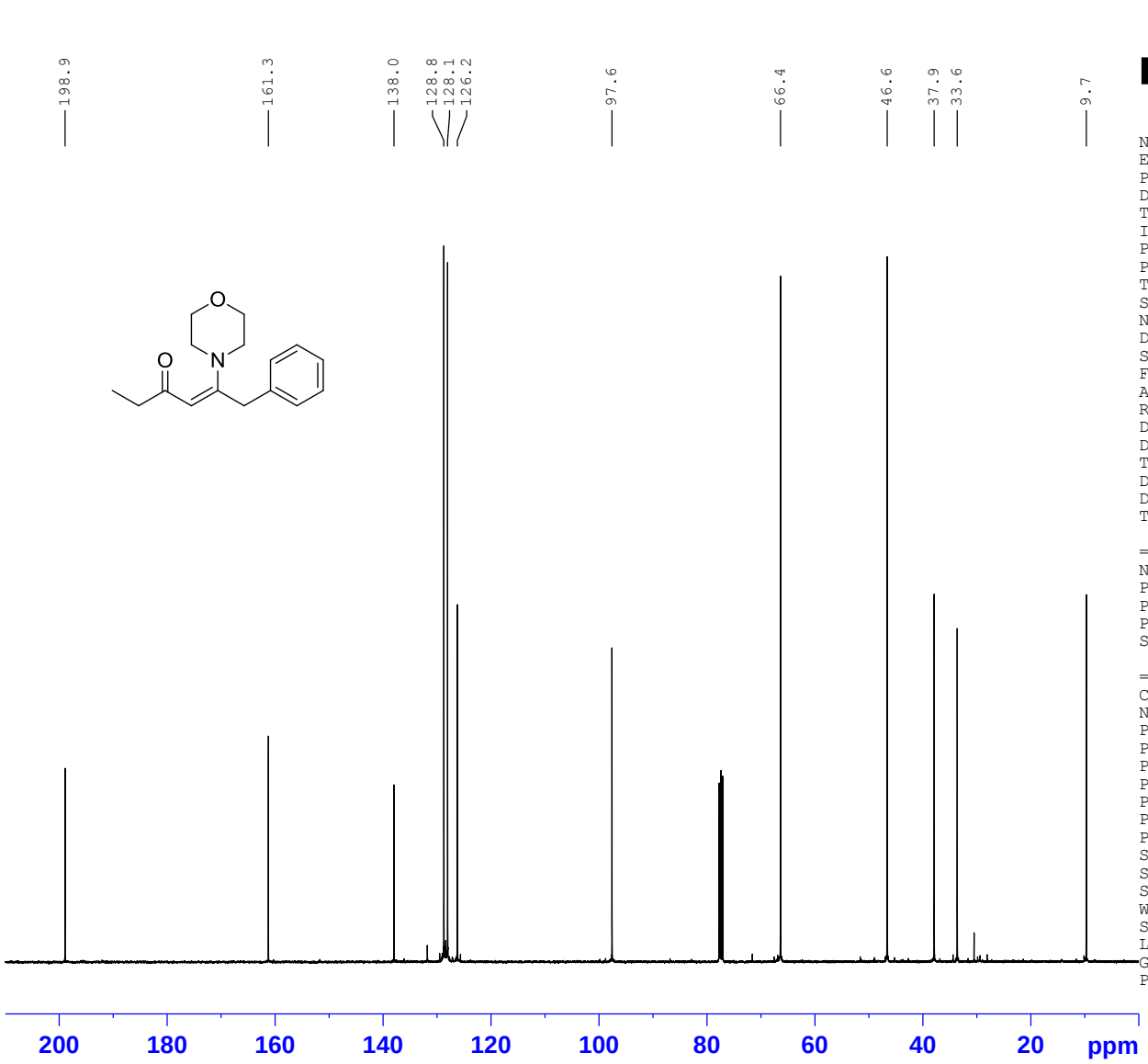



```
NAME            Amination
EXPNO                 239
PROCNO                  1
Date_            20130109
Time                16.12
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     18
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           400.1300021 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

Green Chemistry **2014**, *16*, doi: 10.1039/c3gc41898g

^{13}C NMR spectrum for **6g** ($CDCl_3$)




```
NAME        Agnes010913 13C
EXPNO                    1
PROCNO                   1
Date_             20130109
Time                 17.07
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    807
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           100.6127494 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```