Supporting Information

2-Pyridinyl-*β*-Ketones as New Ligands for Room-Temperature CuI-Catalyzed C-N Coupling Reaction

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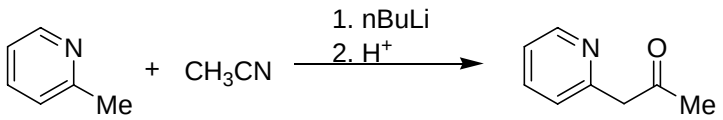
General Experimental Procedures

All reactions were carried out in 5 mL sealed tubes, under a pure and dry Argon atmosphere. DMF was distilled from CaH_2 and was stored on 4 Å activated molecular sieves. Cesium carbonate (Alfa Aesar) were stored in the presence of P_2O_5 in a benchtop desiccator at room temperature and weighed in the air quickly. CuI, Aryl iodides, aryl bromides and amines were purchased from commercial sources (Aldrich, Acros, 3B, Alfa Aesar) and in most cases used directly without further purification. The ligand **F** N-(pyridin-2-yl)acetamide was purchased from TCI and were used as received. Column chromatography was performed with QDHY 60 A C.C silica gel (35-70 mm). All products were characterized by their NMR, GC/MS or LC/MS. NMR spectra were recorded at 20°C on a Bruker AC 400 MHz spectrometer working respectively at 400 MHz for ^1H, at 100 MHz for ^{13}C. Optical rotations were obtained on a polarimeter, specific rotations ([α]D) are reported in deg/dm and the concentration (*c*) is given in g/100 mL in the specified solvent. Gas chromatography - mass spectra (GC/MS) were recorded on an Shimadzu Technologies QP2010 plus instrument with a mass detector (EI) and a Rtx-MS 30 m x 0.32 mm capillary apolar column (Stationary phase: 5 % diphenyldimethylpolysiloxane film, 0.25 μm). GC/MS method: Initial temperature: 80 °C; Initial time: 3 min; Ramp: 30 °C/min; Final temperature: 280 °C; Final time: 15 min. Gas chromatography - mass spectra (GC/MS) were recorded on an Agilent1200. FAB+ mass spectra and HRMS were recorded on a Thermo Finnigan MAT-95XP spectrometer (3 keV, xenon) in a m-nitrobenzylalcohol matrix.The firstorder peak patterns are indicated as s (singulet), d (doublet), t (triplet), q (quadruplet). Complex nonfirst-order signals are indicated as m (multiplet).

Preparation of Ligands: 2-Pyridinyl-*β*-Ketones

Preparation of 2-Acetonylpyridine (A) This procedure was adapted with minor modify from the synthesis reported by Kurashina Yoshikazu and coworkers.[1]

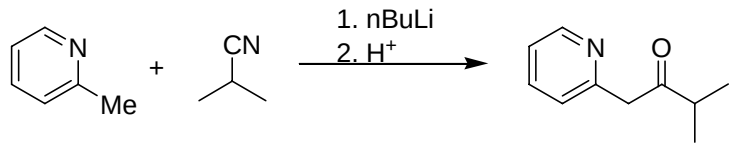


To a solution of 32 mmol of nBuLi in 60 ml THF was added dropwise 2.96 ml (32 mmol) of 2-methylpyridine at 0 °C under an Argon, the reaction mixture was stirred for 45 min at 0 °C. To the reaction mixture was added 1.56 ml (32 mmol) of acetonitrile and the mixture was stirred for 3 hours at room temperature, followed by extraction with 30

ml of 3N-sulfuric acid. The acidic solution was washed with 2*25 ml of diether ether, then made alkaline (PH=11~12) with a 2N-aqueous sodium hydroxide solution and the solution was extracted with dichloromethane. The organic layer was dried over anhydrous sodium sulfate and the solvent was evaporated under reduce pressure. The residual oil was distilled under vacuum to give the desired product as pale yellow oil 2.2 g (51%) *(90:10 ketone: enol, only data for the major ketone form is given)*.

^{1}H NMR($CDCl_3$): δ=8.52 (d, *J*=4.8 Hz, 1 H; Phenyl), 7.60 (td, *J*=8.0, 1.6 Hz, 1 H; Phenyl), 7.20-7.15 (m, 2 H; Phenyl), 3.89 (s, 2 H; CH_2), 2.19 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=205.3 (-C=O), 154.7, 149.5, 136.7, 124.1, 121.9 (Phenyl), 53.1 (CH_2), 29.9 ppm (CH_3); MS (APCI): M/Z: 136 [M-].

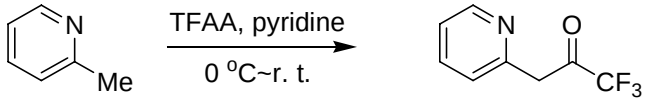
Preparation of 2-(Isobutyrylmethyl)pyridine (B)



This procedure was similar with the synthesis of **A** *(89:11 ketone: enol, only data for the major ketone form is given)***.**

^{1}H NMR($CDCl_3$): δ=8.52 (d, *J*=4.8 Hz, 1 H; Phenyl), 7.60 (td, *J*=7.6, 1.6 Hz, 1 H; Phenyl), 7.21-7.13 (m, 2 H; Phenyl), 3.96 (s, 2 H; CH_2), 2.80-2.74 (m, 1 H; CH), 1.11 ppm (d, *J*=7.2 Hz, 6 H; $2CH_3$); ^{13}C NMR($CDCl_3$) : δ=211.1 (-C=O), 155.1, 149.4, 136.5, 124.2, 121.9 (Phenyl), 49.9 (CH_2), 40.8 (CH), 18.1 ppm (CH_3); GC/MS: rt = 5.91 min, M/Z = 163.

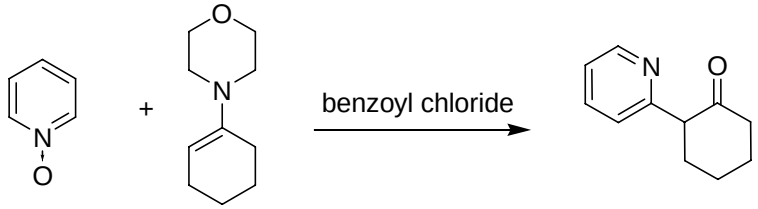
Preparation of 1,1,1-trifluoro-3-(2-pyridinyl)- 2-Propanone (C) This procedure was adapted from the synthesis reported by Masami Kawase and co-workers.[2]



TFAA (2.6 ml, 18 mmol) was added dropwise to a solution of a 2-methylpyridine (6 mmol) and pyridine (2.4 ml, 30 ml) in anhydrous benzene (15 ml) at 0 °C. The mixture was then stirred at room temperature for 24 hours, then diluted with 3% Na_2CO_3 (60 ml) and extracted with EtOAc (60 ml*2). The combined extracts were washed with brine 60 ml, dried over anhydrous Na_2SO_4, filtered and concentrated under vacuum. The residual oil was purified by chromatography (PE-EtOAc=2:1) affording 0.9g (80%) of the desired product*(1:99 ketone: enol, only data for the major enol form is given)*.

^{1}H NMR($CDCl_3$): δ=8.09 (d, *J*=5.6 Hz, 1 H; Phenyl), 7.69 (t, *J*=7.6 Hz, 1 H; Phenyl), 7.07 (d, *J*=8.4 Hz, 1 H; Phenyl), 7.015 (t, *J*=6.4 Hz, 1 H; Phenyl), 5.82 ppm (s, 1 H; -CH=); ^{13}C NMR($CDCl_3$) : δ=155.9 (=CH-OH), 139.2 , 139.1, 122.5, 120.5, 118.1 (Phenyl), 117.8 (CF_3), 90.3 ppm (-CH=); MS (APCI): M/Z: 190 [M-].

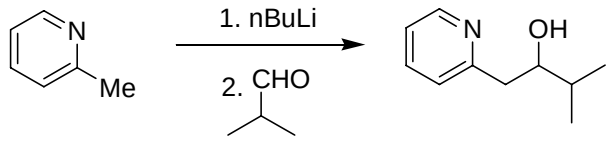
Preparation of 2-(2-Pyridyl)cyclohexanone (D) A slightly modified literature procedure has been used.[3]



To a solution of pyridine N-oxide (4.75 g, 0.05 mol) in CH_2Cl_2 (30 ml) at 0 °C was added dropwise with stirring 7.0 g (0.05 mol) of benzoyl chloride. A white precipitate formed and the mixture was stirred at 0 °C for 30 min. A solution of 4-cyclohexenylmorpholine (10.2 g, 0.06 mol) in CH_2Cl_2 (15 ml) was added slowly with stirring. The

mixture was allowed to warm to room temperature and then refluxed for 5 hours. The solvent was removed under reduced pressure and to the remaining orange oil was added 20% HC1 solution (60 ml). The mixture was washed several times with ether. The aqueous solution was adjusted to pH=8 with dilute NaOH solution and the oil which separated was extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and evaporated under vacuum to give 15.6 g of a brown oil. Distillation under reduce pressure gave 4.2g (48%) of pure desired product as a pale yellow liquid*(a mixture of ketone- enol 1:1)*; MS (APCI): M/Z: 176 [M-].

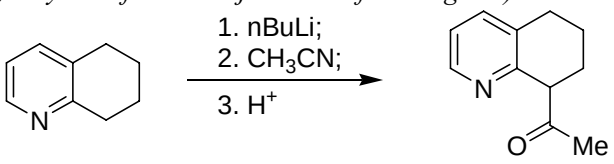
Preparation of 3-methyl-1-(2-pyridin)-2-butanol (E) This procedure was similar from the synthesis reported by Kurashina Yoshikazu and coworkers.[1]



To a solution of 32 mmol of nBuLi in 60 ml THF was added dropwise 2.96 ml (32 mmol) of 2-methylpyridine at 0 ^oC under an Argon, the reaction mixture was stirred for 45 min at 0 ^oC. To the reaction mixture was added 2.3g (32 mmol) of isobutyraldehyde and the mixture was stirred for 3 hours at room temperature, followed by addition with 30 ml of saturated aqueous of NH_4Cl. The mixture was extracted with dichloromethane. The organic layer was dried over anhydrous sodium sulfate and the solvent was evaporated under reduce pressure. The residual oil was distilled under vacuum to give the desired product as colourless oil 2.65g (racemic 50%).

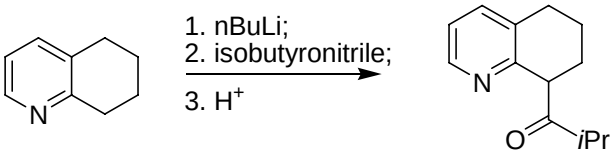
^1H NMR($CDCl_3$): δ=8.47 (d, *J*=4.8 Hz, 1 H; Phenyl), 7.59 (td, *J*=7.6, 1.2 Hz, 1 H; Phenyl), 7.17-7.13 (m, 2 H; Phenyl), 4.66 (br, 1H; OH), 3.79-3.75 (m, 1 H; -CH-O-), 2.94-2.80 (m, 2 H; CH_2), 1.79-1.71 (m, 1 H; CH), 1.03-0.98 ppm (m, 6 H; $2CH_3$); ^{13}C NMR($CDCl_3$) : δ=160.7, 148.5, 136.7, 123.8, 121.4 (Phenyl), 75.9 (-CH-O-), 40.3 (CH), 33.5 (CH), 18.6 (CH_3), 18.0 ppm (CH_3); GC/MS: rt = 6.12 min, M/Z = 164.

Preparation of 8-acetyl-5,6,7,8-tetrahydroquinoline (G) This procedure was similar with the synthesis of **A** with yield: 46%, *(81:19 ketone: enol, only data for the major ketone form is given)***.**



^1H NMR($CDCl_3$): δ=8.41 (s, 1 H; Phenyl), 7.42 (d, *J*=7.6 Hz, 1 H; Phenyl), 7.09 (t, *J*=7.2 Hz, 1 H; Phenyl), 4.01 (t, *J*=6.4 Hz, 1 H; CH), 2.882-2.78 (m, 2 H; CH_2), 2.25 (s, 3 H; CH_3), 2.16-2.12 (m, 2 H; CH_2), 1.81 ppm (br, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=209.8 (-C=O), 162.6, 154.6, 147.2, 137.1, 132.8, 121.9 (Phenyl), 55.5 (CH), 29.1, 28.4, 25.9, 20.3 ppm (CH_2); GC/MS: rt = 7.09 min, M/Z = 175; HRMS (EI): calcd for $C_{11}H_{13}NO$: 175.0992 [M+]; found: 175.0996.

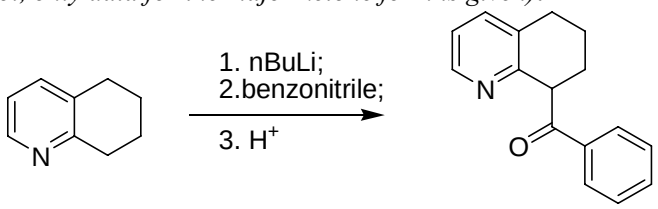
Preparation of 8-(2-methyl-1-propanone)-5,6,7,8-tetrahydroquinolin (H) This procedure was similar with the synthesis of **A** with yield: 42%, *(77:23 ketone: enol, only data for the major ketone form is given)***.**



^1H NMR($CDCl_3$): δ=8.36 (d, *J*=4.4 Hz, 1 H; Phenyl), 7.39 (d, *J*=8.0 Hz, 1 H; Phenyl), 7.06 (dd, *J*=7.6, 4.8 Hz, 1 H; Phenyl), 4.20 (t, *J*=6.8 Hz, 1 H; CH), 3.07-3.00 (m, 1 H; CH), 2.79-2.73 (m, 2 H; CH_2), 2.11-2.06 (m, 2 H; CH_2), 1.85-1.80 (m, 2 H; CH_2), 1.20-1.14 ppm (m, 6 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=215.8 (-C=O), 155.3, 147.1, 136.9,

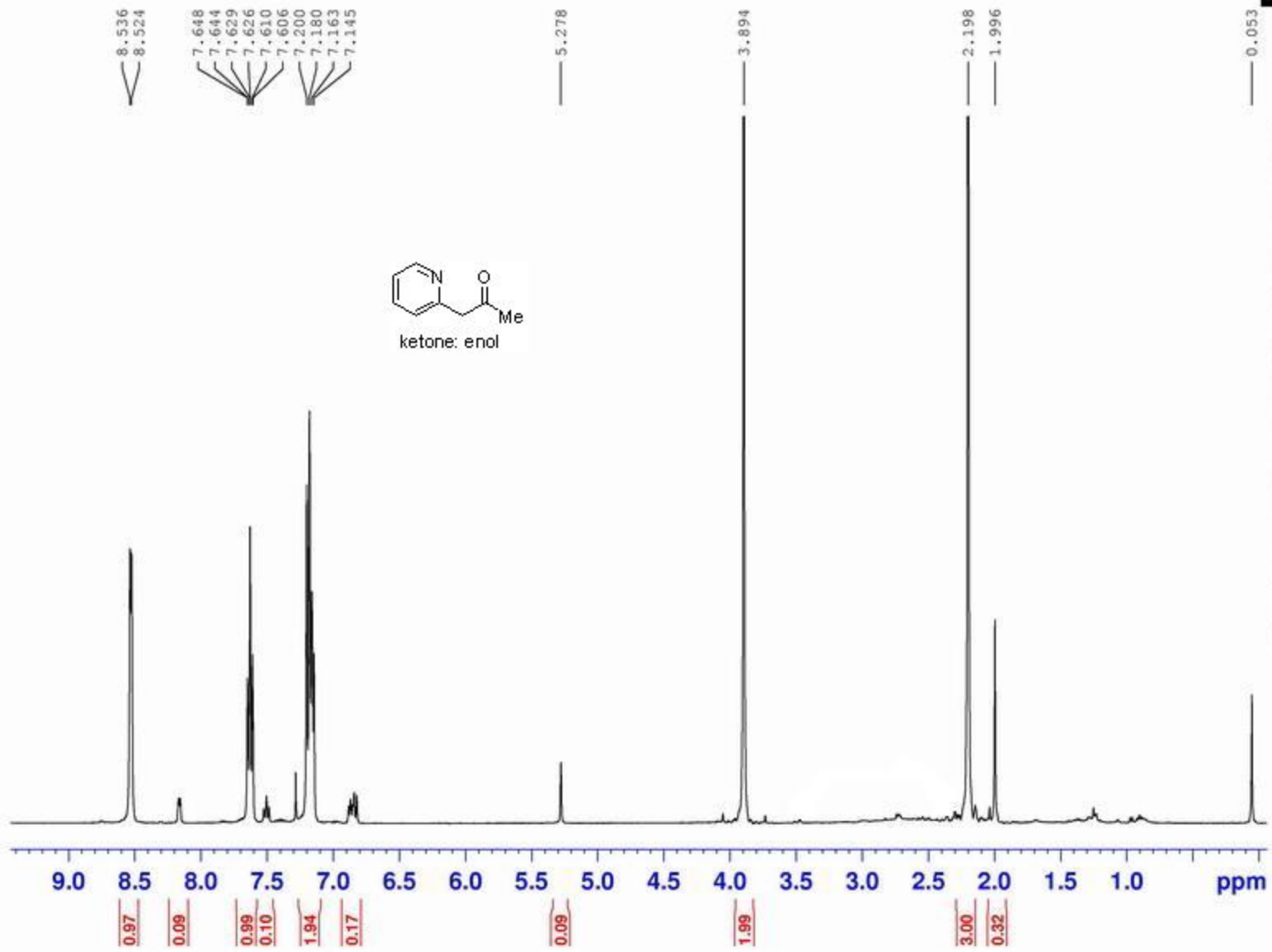
132.9, 121.7 (Phenyl), 53.1 (CH), 40.3 (CH), 28.4 (CH_2), 26.1 (CH_2), 20.2 (CH_2), 18.5 ppm (CH_3); GC/MS: rt = 7.57 min, M/Z = 203; HRMS (EI): calcd for $C_{13}H_{17}NO$: 203.1305 [M+]; found: 203.1301.

Preparation of 5,6,7,8-tetrahydro-8-benzoylquinolinyl (I) This procedure was similar with the synthesis of **A** with yield: 66%, *(68:32 ketone: enol, only data for the major ketone form is given)***.**



^{1}H NMR($CDCl_3$): δ=8.37 (d, *J*=4.0 Hz, 1 H; Phenyl), 8.07 (d, *J*=8.0 Hz, 2 H; Phenyl), 7.60-7.36 (m, 4 H; Phenyl), 7.09 (dd, *J*=7.6, 4.8 Hz, 1 H; Phenyl), 5.02 (t, *J*=6.4 Hz, 1 H; CH), 2.85-2.78 (m, 2 H; CH_2), 2.26-2.21 (m, 2 H; CH_2), 1.98-1.83 (m, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=202.0 (-C=O), 155.7, 147.3, 136.9, 133.3, 132.9, 128.9, 128.6, 127.9, 121.8 (Phenyl), 49.6 (CH), 28.4, 27.3, 19.9 ppm (CH_2); GC/MS: rt = 9.76 min, M/Z = 236.

125237



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SOLVENT CDCl3
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FIDRES 0.126314 Hz
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RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1

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PL1 0.00 dB
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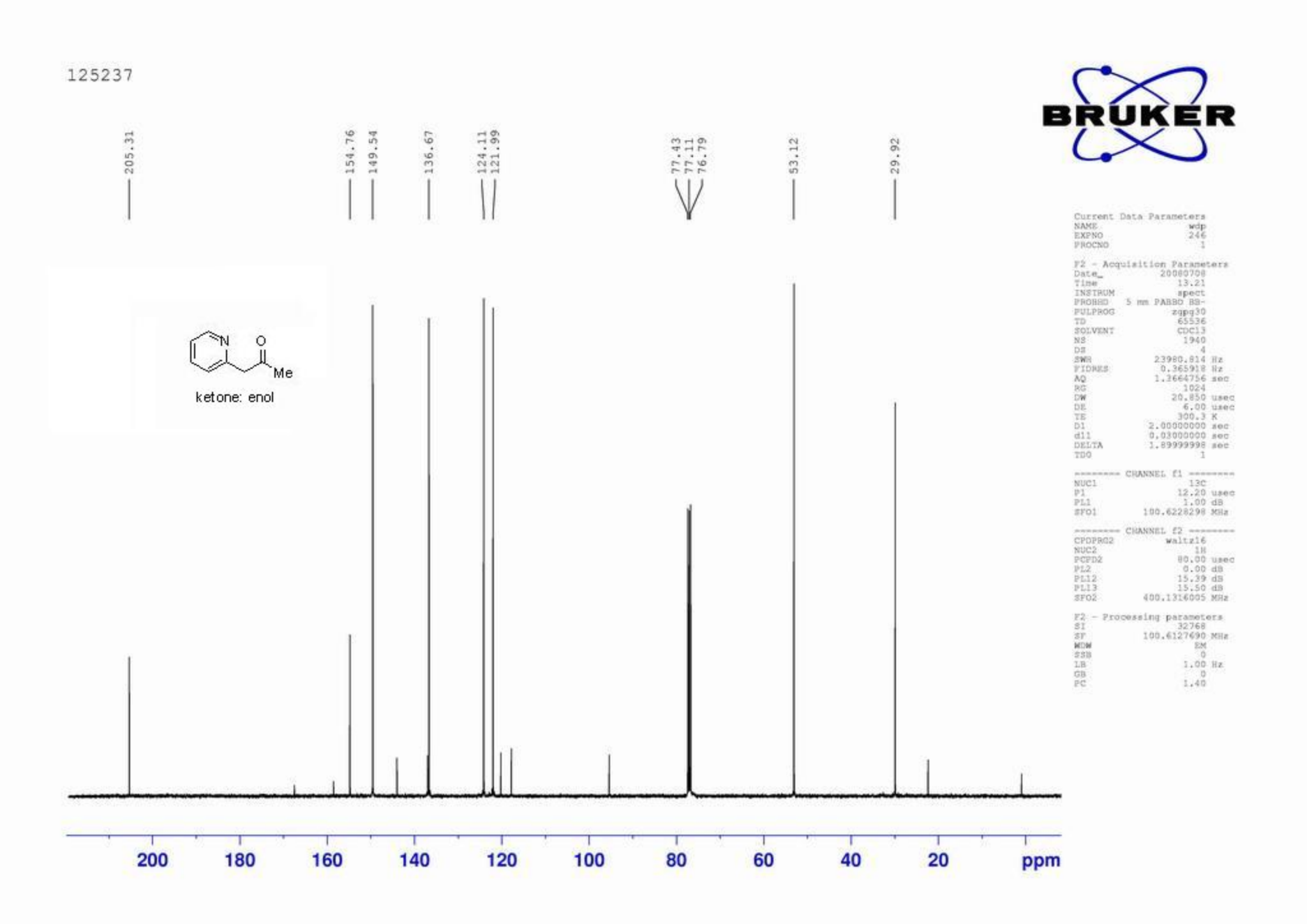
125237



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SOLVENT CDCl3
NS 1940
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1024
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DE 6.00 usec
TE 300.3 K
D1 2.00000000 sec
d11 0.03000000 sec
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TD0 1

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P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

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PL13 15.50 dB
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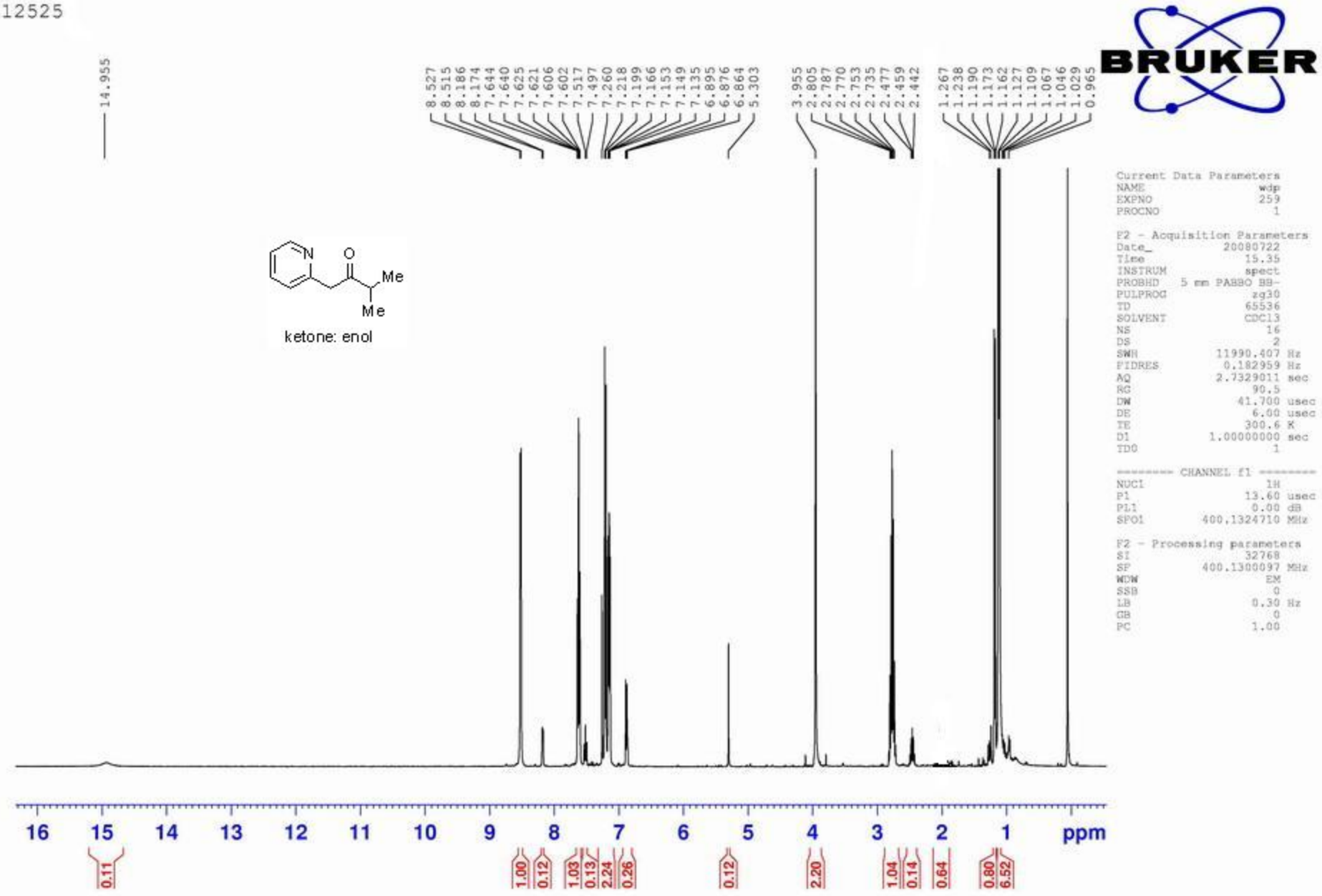
12525



BRUKER

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TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 11990.407 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 90.5
DW 41.700 usec
DE 6.00 usec
TE 300.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
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PL1 0.00 dB
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F2 - Processing parameters
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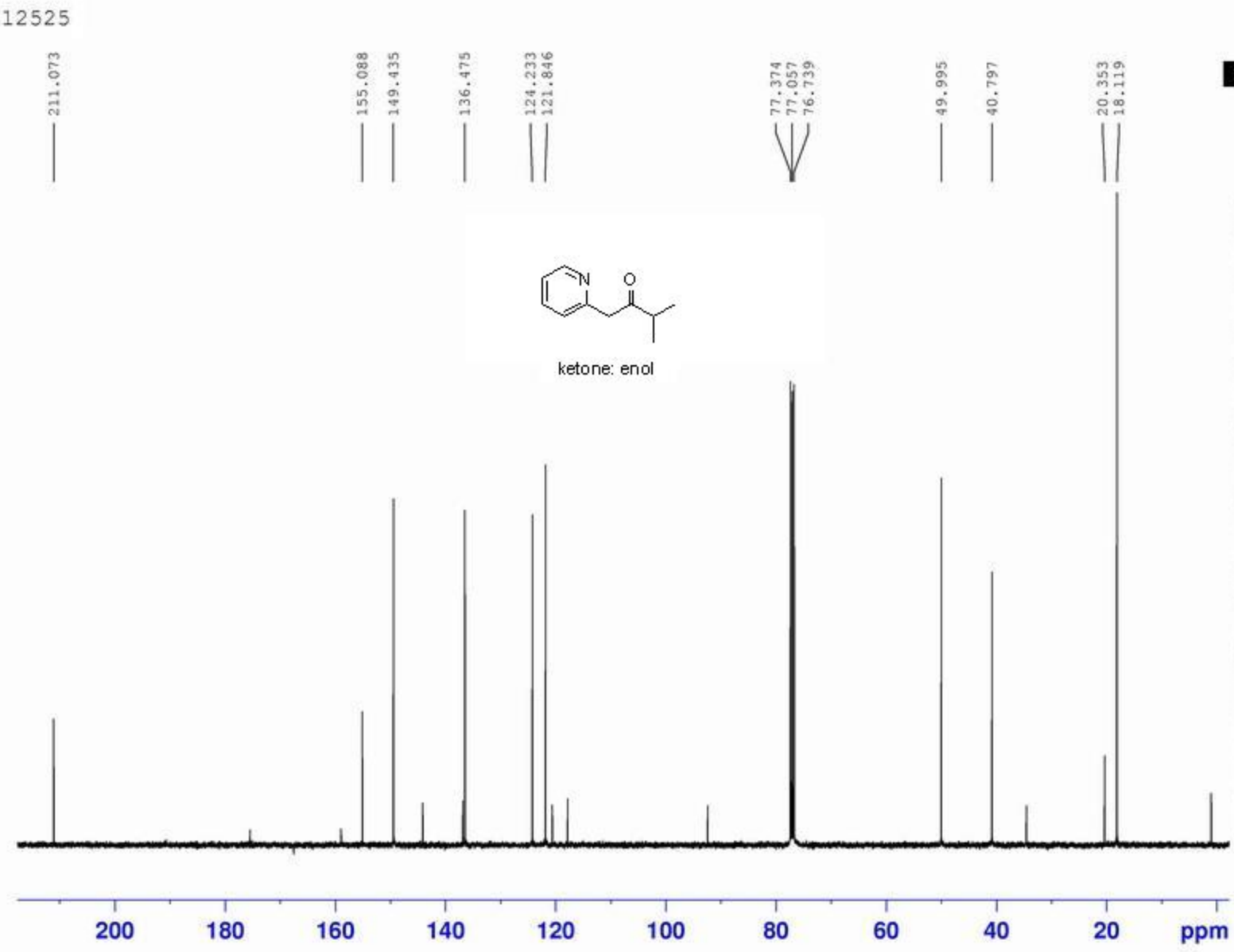
12525



BRUKER

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PROCNO                  1

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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                1824.6
DW               20.850 usec
DE                6.00 usec
TE               299.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
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WDW                  EM
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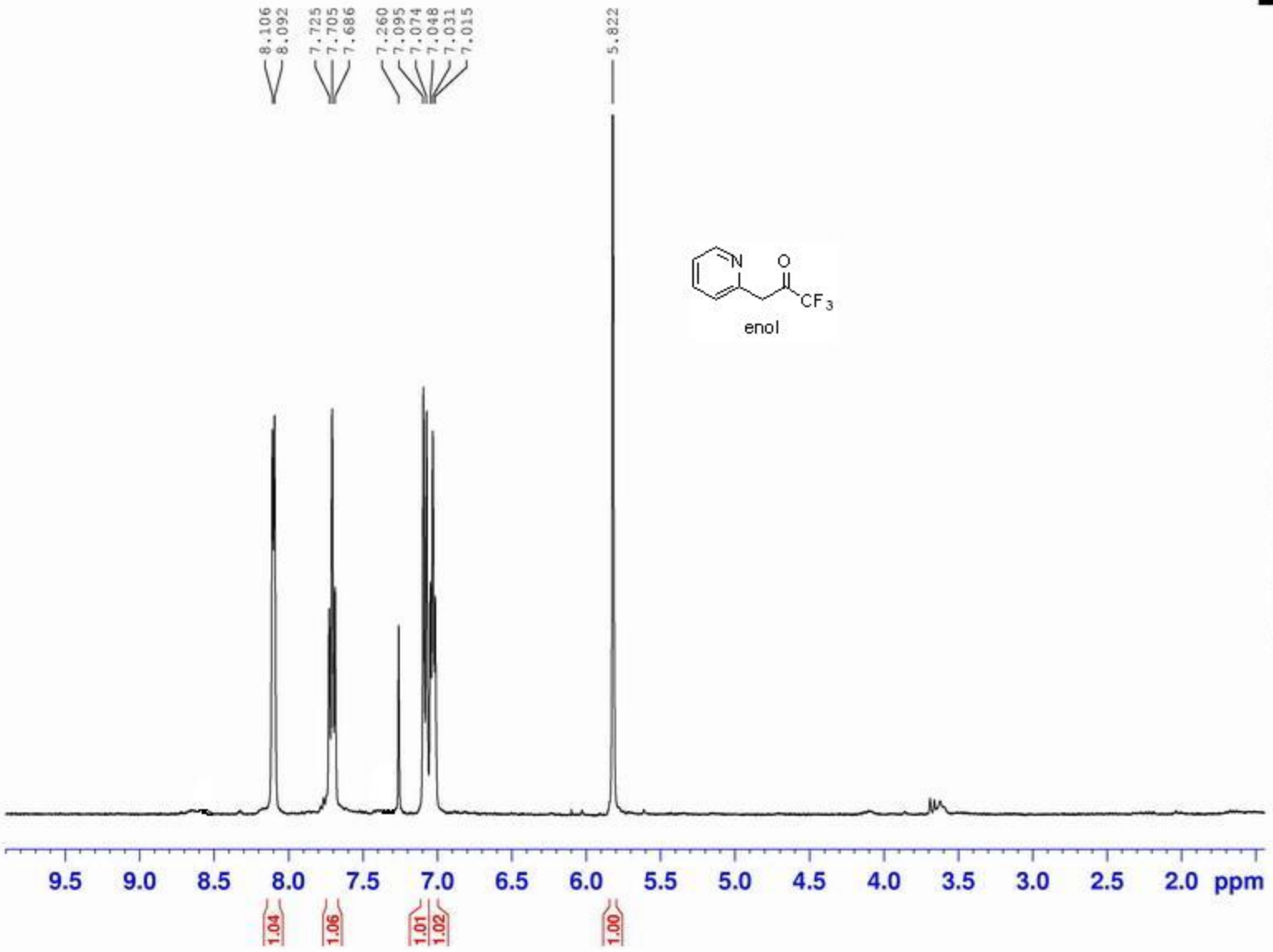
125234



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PROCNO	1

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PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	2
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	256
DW	60.400 usec
DE	6.00 usec
TE	300.2 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

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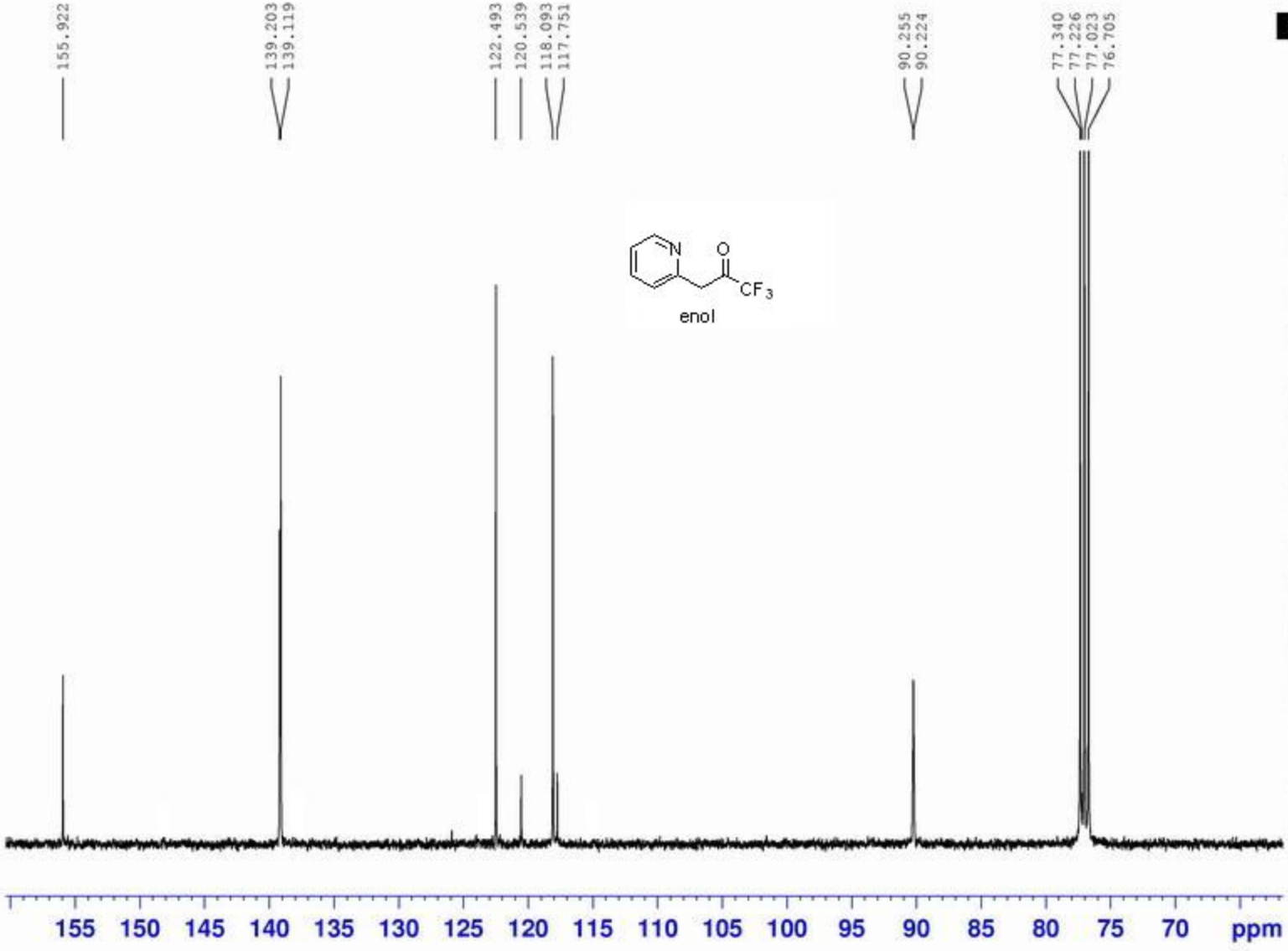
125234



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NAME                  wdp
EXPNO                 242
PROCNO                  1

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Time                 13.37
INSTRUM              spect
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TD                  65536
SOLVENT             CDCl3
NS                   2048
DS                      4
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                   724.1
DW                  20.850 usec
DE                   6.00 usec
TE                  303.2 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.20 usec
PL1                  1.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                  0.00 dB
PL12                15.39 dB
PL13                15.50 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
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SF            100.6127690 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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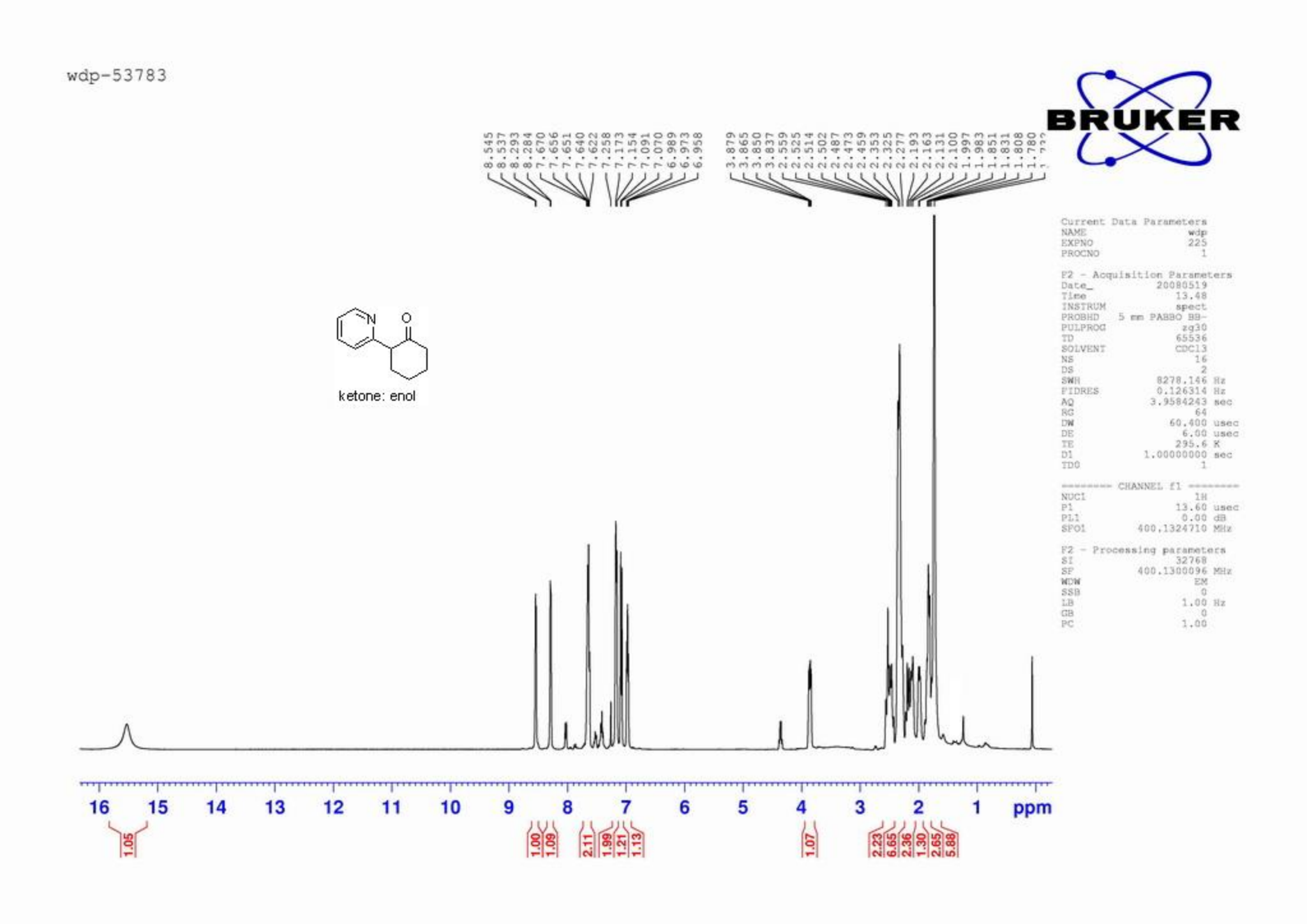
wdp-53783



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SOLVENT	CDCl3
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DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	64
DW	60.400 usec
DE	6.00 usec
TE	295.6 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

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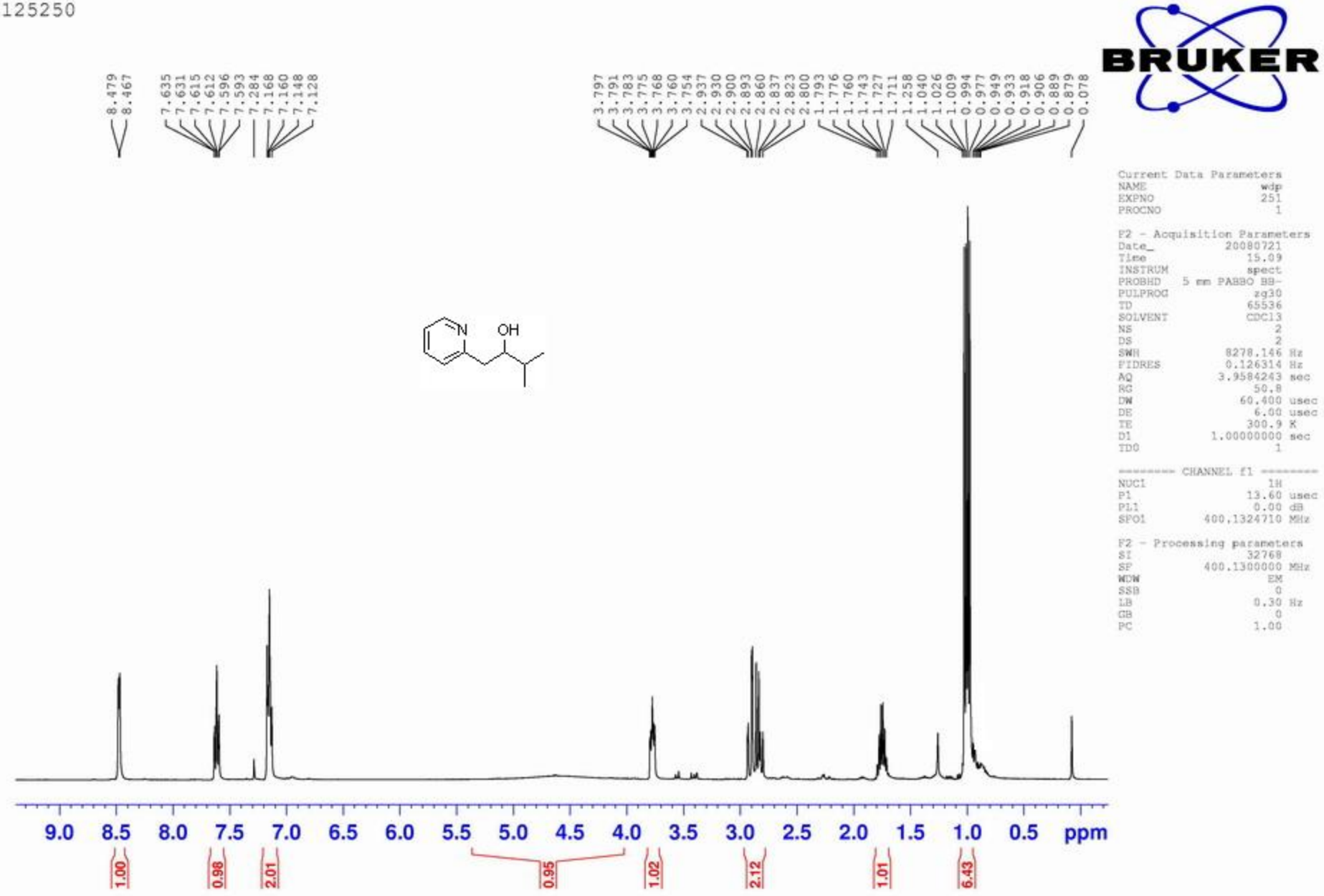
125250



BRUKER

Current Data Parameters
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TD 65536
SOLVENT CDCl3
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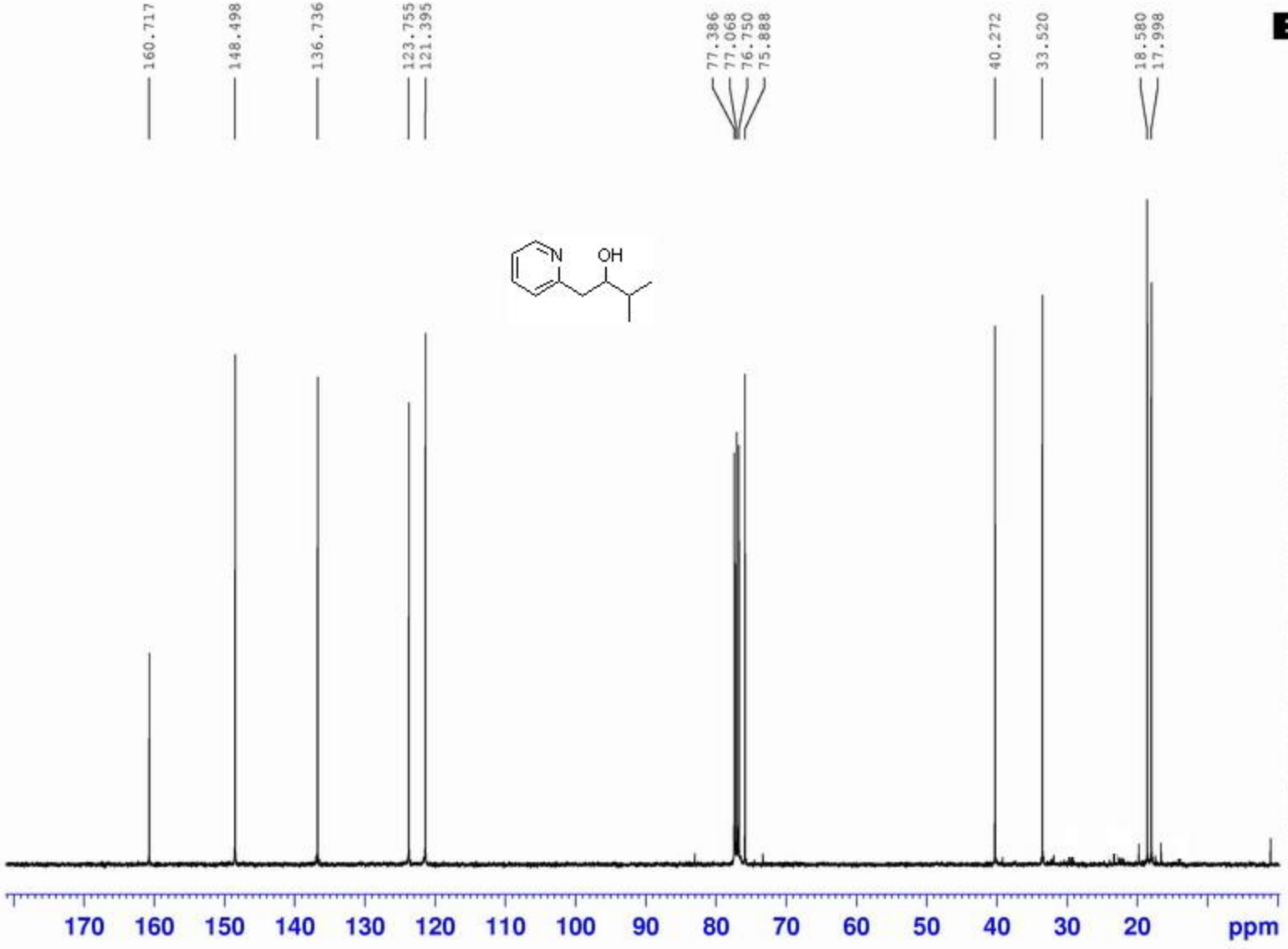
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 usec
TE 300.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125250



BRUKER

Current Data Parameters
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EXPNO 258
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1955
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SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 299.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

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P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
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NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
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F2 - Processing parameters
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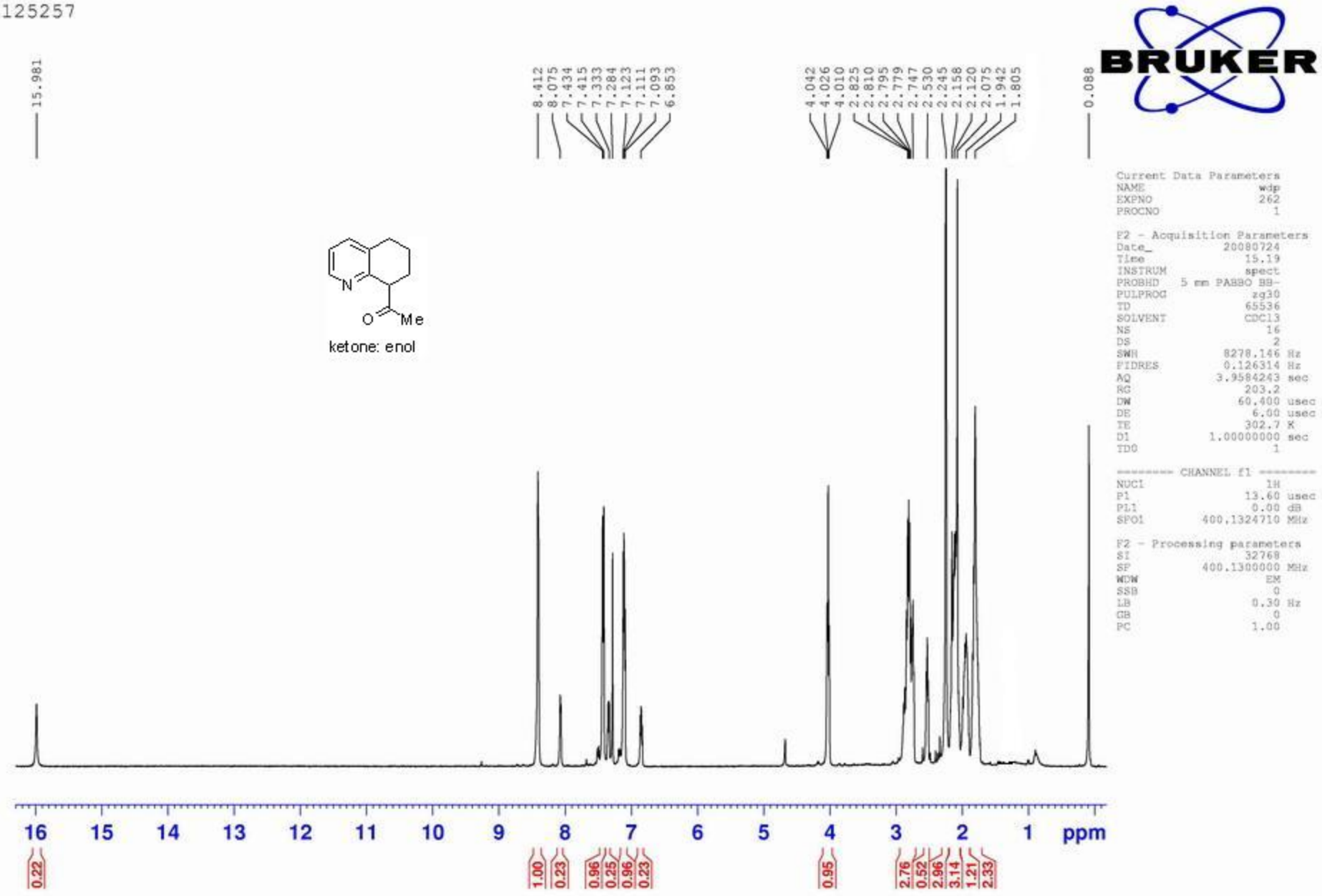
125257

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EXPNO                 262
PROCNO                1

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TD               65536
SOLVENT          CDCl3
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DS                   2
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FIDRES        0.126314 Hz
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TE               302.7 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
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PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
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WDW                  EM
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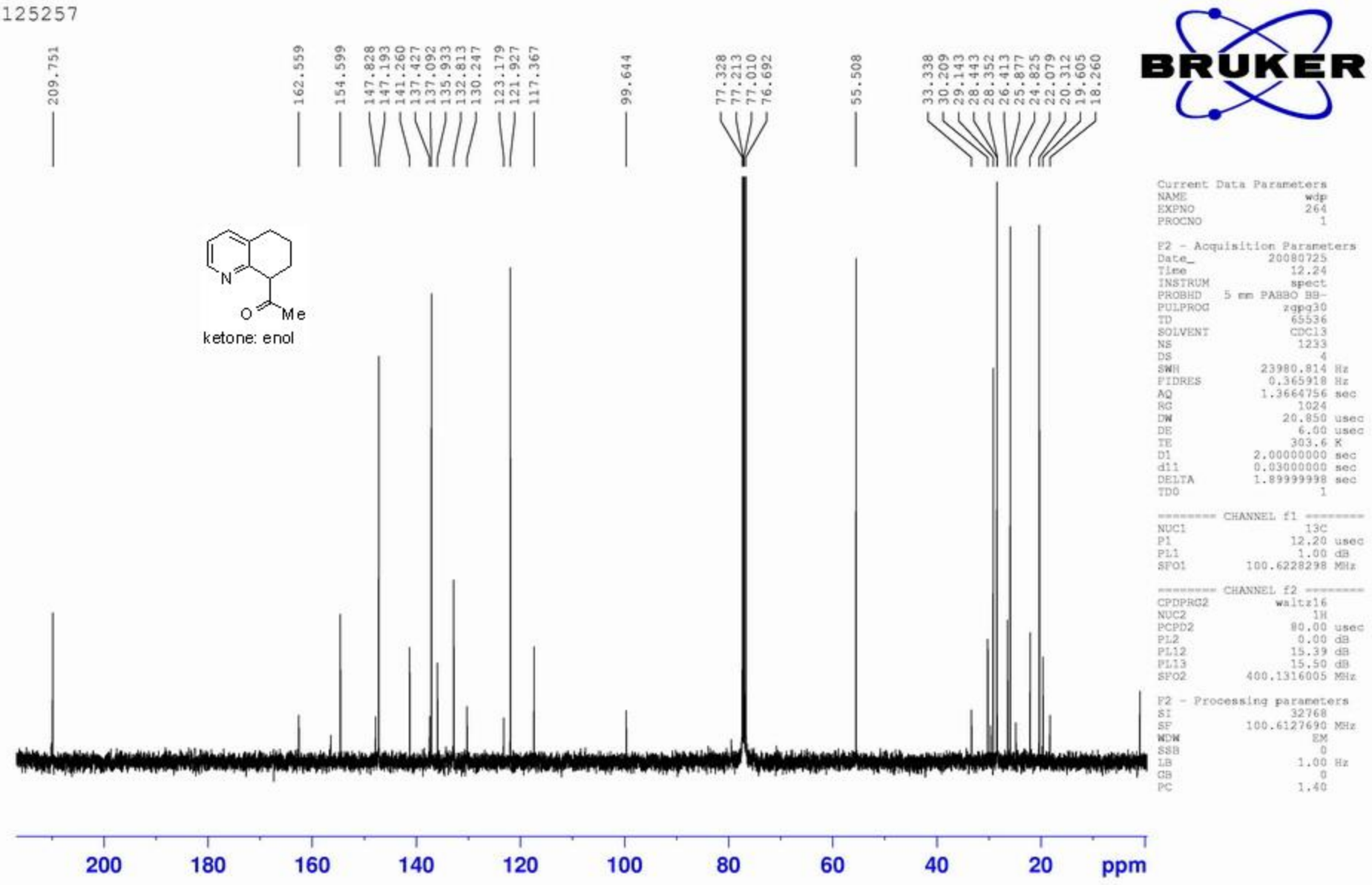
125257



BRUKER

Current Data Parameters
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PROCNO 1

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SOLVENT CDCl3
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FIDRES 0.365918 Hz
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DW 20.850 usec
DE 6.00 usec
TE 303.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
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SFO1 100.6228298 MHz

======== CHANNEL f2 ========
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NUC2 1H
PCPD2 80.00 usec
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PL13 15.50 dB
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F2 - Processing parameters
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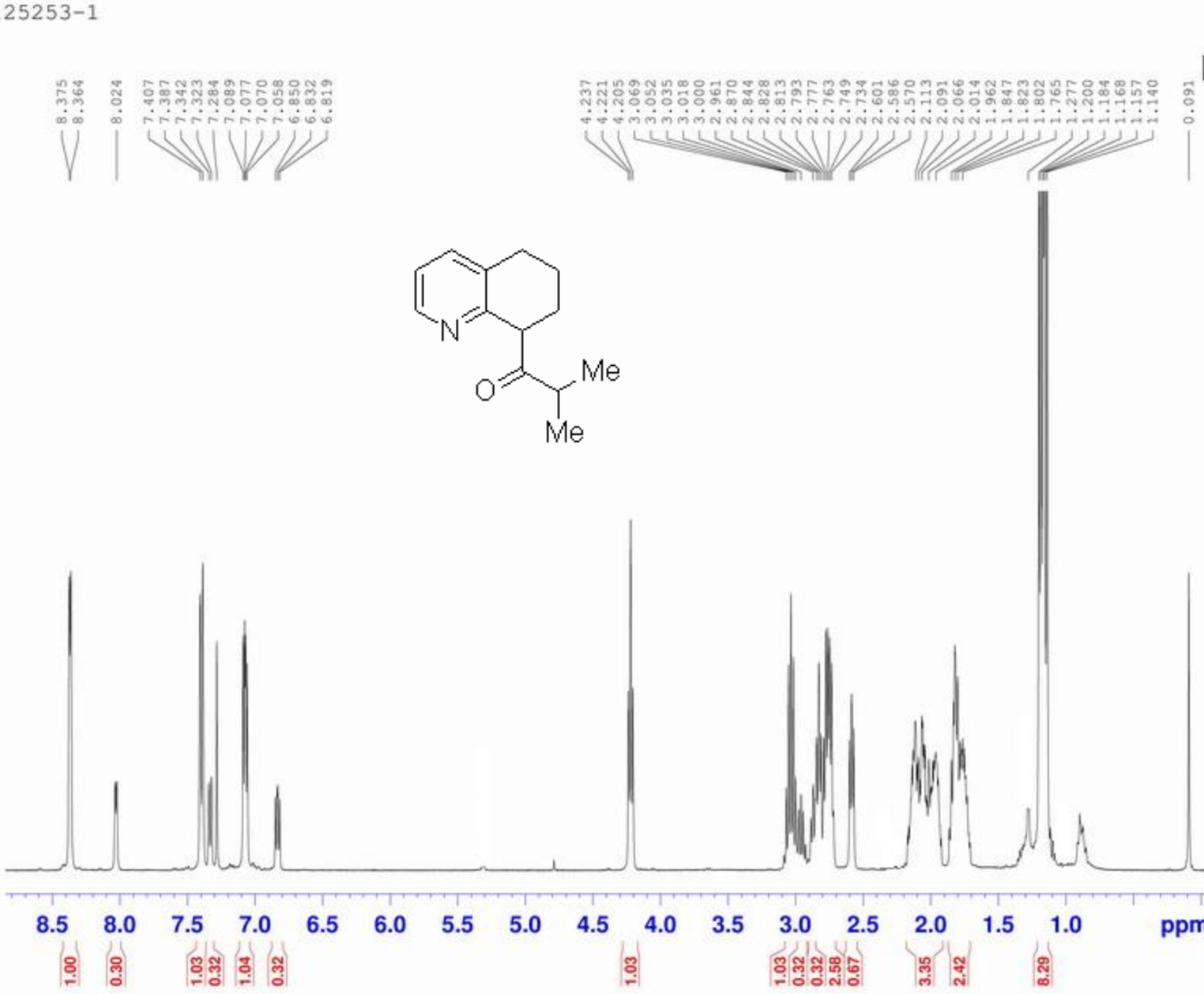
GB 0
PC 1.40

125253-1



BRUKER

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EXPNO                 254
PROCNO                  1

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TD               65536
SOLVENT          CDCl3
NS                   9
DS                   2
SWH            8278.146 Hz
FIDRES            0.126314 Hz
AQ             3.9584243 sec
RG                 101.6
DW                60.400 usec
DE                 6.00 usec
TE                299.1 K
D1             1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.60 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
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SF           400.1300000 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
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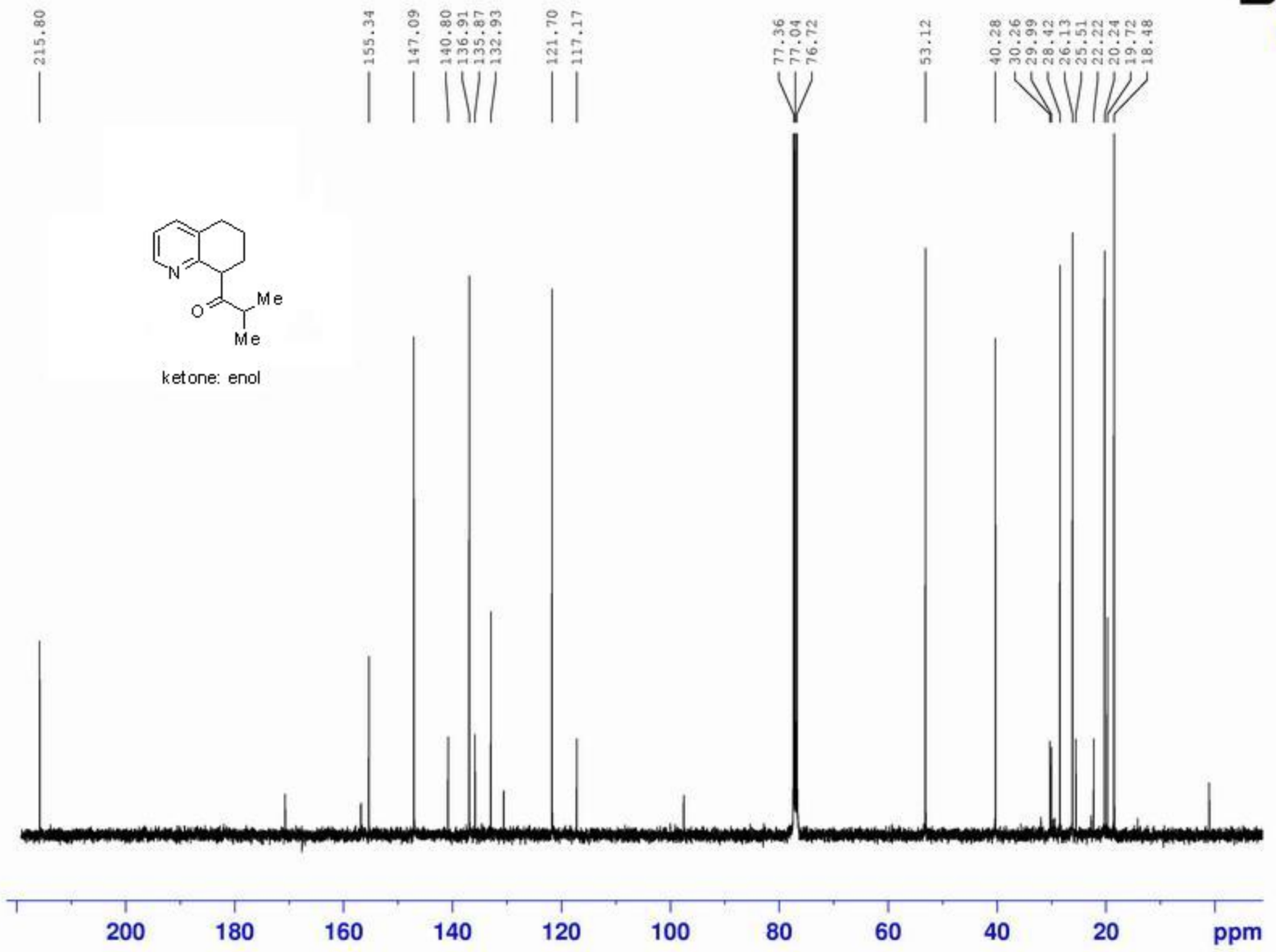
125253-1



BRUKER

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EXPNO                 255
PROCNO                  1

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INSTRUM              spect
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PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1786
DS                     4
SWH            23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                1149.4
DW               20.850 usec
DE                6.00 usec
TE                297.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
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WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
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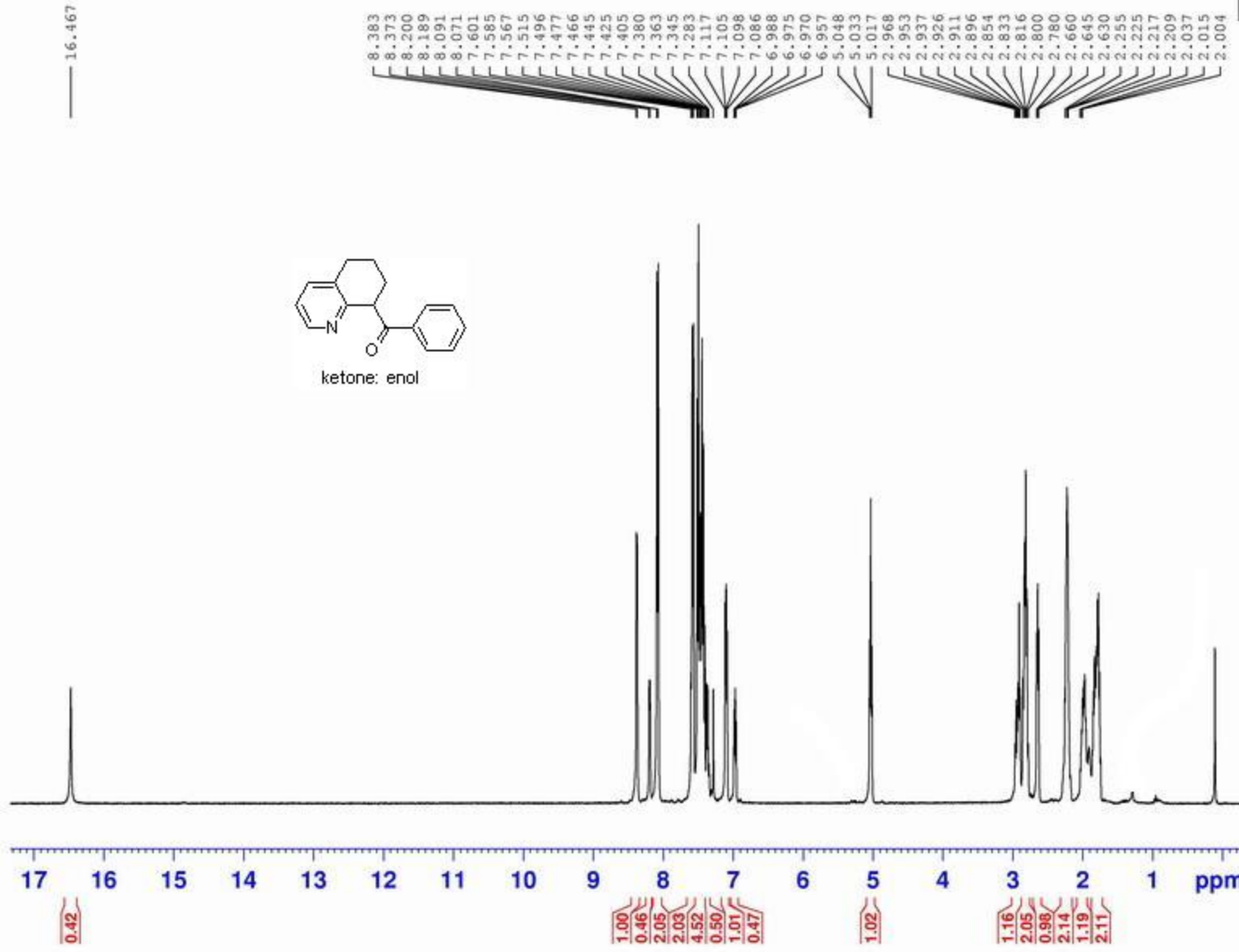
125258



BRUKER

Current Data Parameters

NAME	wdp
EXPNO	263
PROCNO	1

F2 - Acquisition Parameters

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Time	10.35
INSTRUM	spect
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PULPROG	zg30
TD	65536
SOLVENT	CDCl3
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DS	2
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AQ	2.7329011 sec
RG	80.6
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DE	6.00 usec
TE	302.6 K
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TD0	1

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

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

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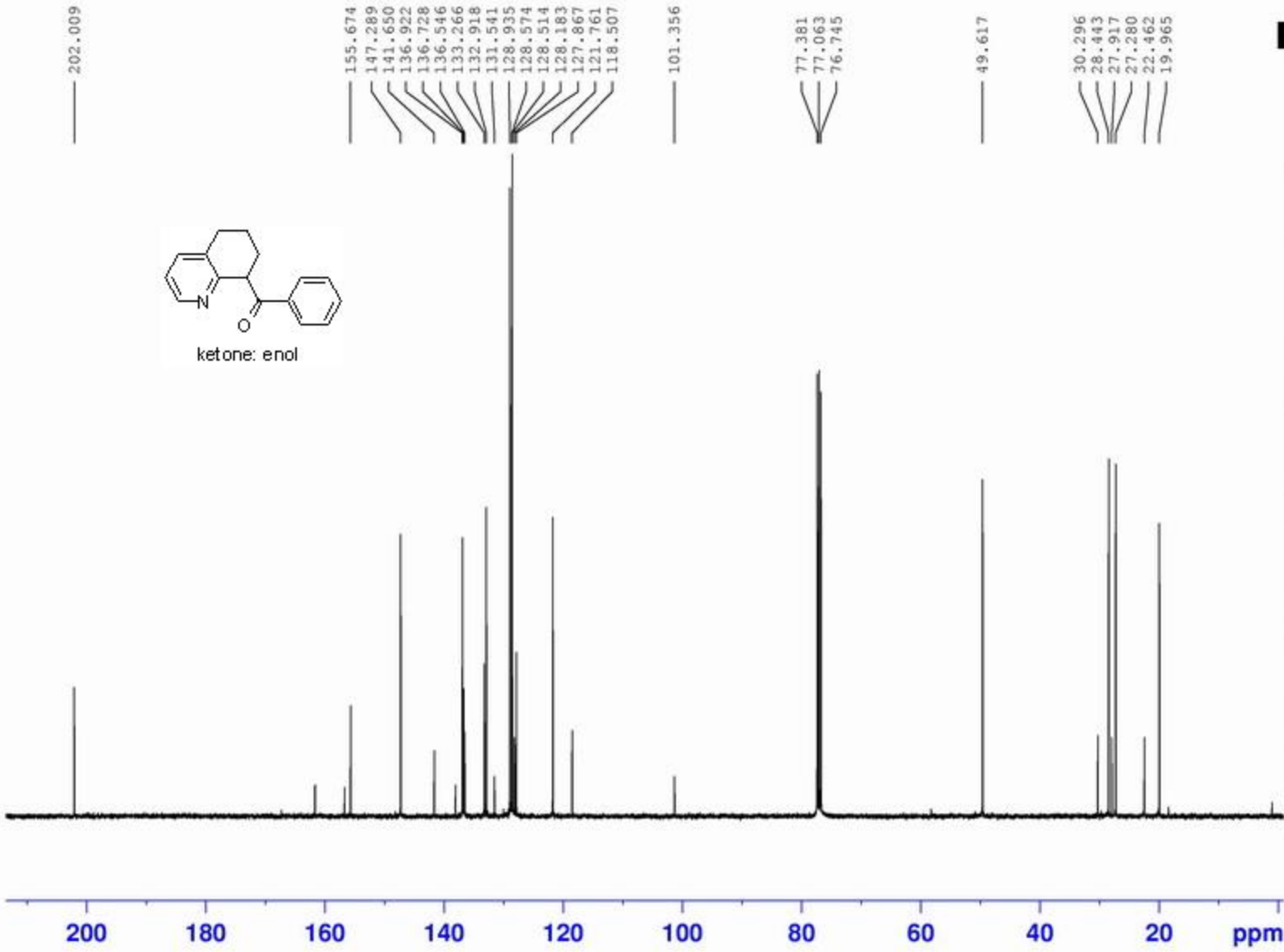
PC	1.00

125258



BRUKER

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EXPNO                  265
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TD                    65536
SOLVENT               CDCl3
NS                     2048
DS                        4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                   1290.2
DW                  20.850 usec
DE                   6.00 usec
TE                  304.0 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.20 usec
PL1                   1.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   0.00 dB
PL12                 15.39 dB
PL13                 15.50 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF            100.6127690 MHz
WDW                       EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
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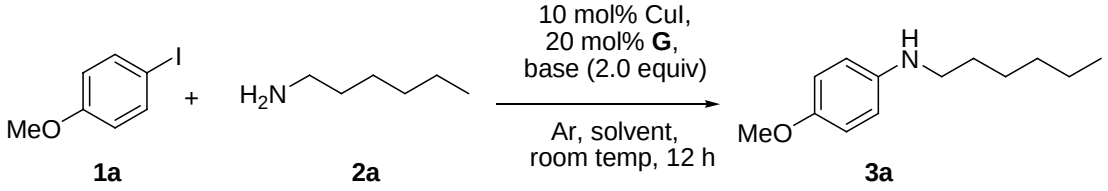
Experimental Procedure for optimization of the Catalytic Conditions.

TABLE I. Effects of the base and solvent on the coupling reaction between 4-methoxy iodobenzene and *n*-hexylamine [a]



1a **2a** **3a**

Entry	Base	Solvent	Yield (%)[b]
1	Cs_2CO_3	DMF	97
2	K_2CO_3	DMF	31
3	K_3PO_4	DMF	72
4	DABCO	DMF	trace
5	Cs_2CO_3	DMSO	95
6	Cs_2CO_3	CH_3CN	47
7	Cs_2CO_3	toluene	19
8	Cs_2CO_3	DMF	63[c]
9	Cs_2CO_3	DMF	92[d]

[a] **1a** (1.0 mmol), **2a** (1.5 mmol), 10 mol% CuI, 20 mol% **G**, base (2.0 mmol), solvent (0.5 mL), Ar, 25 °C, 12 h. [b] Isolated yield. [c] CuI (5 mol%), **G** (10 mol%). [d] **1a** (50 mmol), **2a** (75 mmol), 10 mol% CuI, 20 mol% **G**, Cs_2CO_3 (100 mmol), DMF (25 mL), Ar, 25 °C, 12 h.

General Procedure A for Room-Temperature Amination of Aryl Iodides and Heteroaryl Iodides (1.0 mmol scale).

An oven-dried Schlenk tube equipped with a Teflon valve was charged with a magnetic stir bar, CuI (19 mg, 0.10 mmol, 10 mol %), Cs_2CO_3 (650 mg, 2 mmol) and any remaining solids (amine and/or aryl halide). The tube was evacuated and backfilled with argon (this procedure was repeated three times). Under a counter flow of argon, 1.5 mmol amine (if liquid), 1 mmol aryl halide (if liquid), DMF (0.5 mL) and ligand **G** (35 mg, 0.2 mmol, 20 mol%) were added by syringe. The tube was evacuated and backfilled with argon again (this procedure was repeated three times) and sealed. The reaction mixture was allowed to stir under argon at ambient temperature (25 °C) for the required time period. Upon completion of the reaction, the mixture was diluted with ethyl acetate (10 ml), passed through a fritted glass filter to remove the inorganic salts and the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel and the product was dried under high vacuum for at least 1 h.

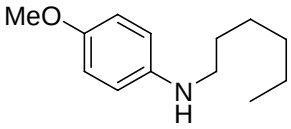
General Procedure B for Coupling of Aryl Bromides with Amines (1.0 mmol scale).

An oven-dried Schlenk tube equipped with a Teflon valve was charged with a magnetic stir bar, CuI (19 mg, 0.10 mmol, 10 mol %), Cs_2CO_3 (650 mg, 2 mmol) and any remaining solids (amine and/or aryl halide). The tube was evacuated and backfilled with argon (this procedure was repeated three times). Under a counter flow of argon, 1.5 mmol amine (if liquid), 1 mmol aryl halide (if liquid), DMF (0.5 mL) and ligand **G** (35 mg, 0.2 mmol, 20 mol%) were added by syringe. The tube was evacuated and backfilled with argon again (this procedure was repeated three times) and sealed. The reaction mixture heated to the indicated temperature (60-100 °C) for the required time period. After cooling to room temperature, the mixture was diluted with ethyl acetate (10 ml) and filtered through a plug of celite® to remove the inorganic salts, the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel and the product was dried under high vacuum for at least 1 h.

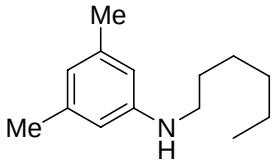
General Procedure C for Coupling of Aryl aryl halide with Amino Acids (1.0 mmol scale).

The reaction procedure is similar to the general procedure **A** or **B** except use of 3 mmol Cs_2CO_3. After completion of the reaction, 5% NaOH aqueous (10 ml) and Et_2O (10 ml) were added to the solution. The resulting solution was partitioned into two phases, the aqueous layer was separated, and the organic fraction was extracted with 5% NaOH aqueous (3 × 5 ml). The combined aqueous phase was neutralized to pH=4 with 20% HCl, the precipitation was filtered and dried by high vacuum at least 24 hours affording the desired products.

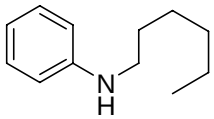
Room Temperature Amination of Aryl Iodides: Table 2.



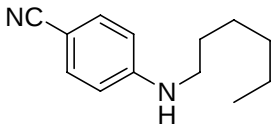
N-*n*-hexyl-4-methoxybenzenamine (3a). Following Procedure A, 1-iodo-4-methoxybenzene (234 mg, 1.0 mmol) was allowed to react with *n*-hexylamine (152 mg, 1.5 mmol) for 12 h. The crude brown oil was purified by flash chromatography on silica gel eluent (15:1 petroleum ether: ethyl acetate) to provide 97 % yield of the desired product as a pale-yellow oil. ^{1}H NMR($CDCl_3$): δ=6.82 (d, *J*=8.8 Hz, 2 H; Phenyl), 6.62 (d, *J*=8.8 Hz, 2 H; Phenyl), 3.79 (s, 3 H; OCH_3), 3.34 (s, 1 H; NH), 3.08 (t, *J*=7.2 Hz, 2 H; -N-CH_2-), 1.63 (m, 2 H; CH_2), 1.47-1.35 (m, 6 H; CH_2), 0.94 ppm (t, *J*=6.8 Hz, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=152.1, 142.8, 114.9, 114.1 (Phenyl), 55.8 (-N-CH-), 45.1, 31.7, 29.7, 26.9, 22.7 (CH_2), 14.1 ppm (CH_3); GC/MS: rt = 8.11 min, M/Z = 207.



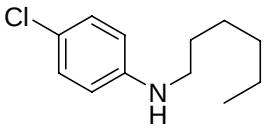
N-*n*-hexyl-3,5-dimethylbenzenamine (3b). Following Procedure A, 1-iodo-3,5-dimethylbenzene (232 mg, 1.0 mmol) was allowed to react with *n*-hexylamine (152 mg, 1.5 mmol) for 12 h. The crude brown oil was purified by flash chromatography on silica gel eluent (25:1 petroleum ether: ethyl acetate) to provide 97 % yield of the desired product as a pale-yellow oil. ^{1}H NMR($CDCl_3$): δ=6.5 (s, 1 H; Phenyl), 6.38 (s, 2 H; Phenyl), 3.56 (s, 1 H; NH), 3.19 (t, *J*=7.2, 2 H; -N-CH_2), 2.39 (s, 6 H; CH_3), 1.77-1.69 (m, 2H; CH_2), 1.57-1.43 (m, 6 H; CH_2), 1.04 ppm (t, *J*=6.8 Hz, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=148.8, 138.9, 119.3, 110.9 (Phenyl), 44.2 (-N-CH_2-), 31.8 (Aryl-CH_3), 29.8, 27.0, 22.8, 21.6 (CH_2), 14.2 ppm (CH_3); GC/MS: rt = 7.82 min, M/Z = 205.



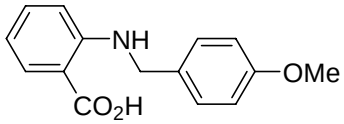
N-*n*-hexylbenzenamine (3c). Following Procedure A, iodobenzene (204 mg, 1.0 mmol) was allowed to react with *n*-hexylamine (152 mg, 1.5 mmol) for 10 h. The crude brown oil was purified by flash chromatography on silica gel eluent (25:1 petroleum ether: ethyl acetate) to provide 97 % yield of the desired product as a pale-yellow oil. ^{1}H NMR($CDCl_3$): δ=7.21-7.17 (m, 2 H; Phenyl), 6.69 (t, *J*=7.6 Hz, 1 H; Phenyl), 6.63 (d, *J*=7.6 Hz, 2 H; Phenyl), 3.73 (s, 1 H; NH), 3.10 (t, *J*=7.2, 2 H; -N-CH_2-), 1.67-1.60 (m, 2H; CH_2), 1.46-1.29 (m, 6 H; CH_2), 0.91 ppm (t, *J*=6.8 Hz, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=148.3, 129.2, 117.3, 112.9 (Phenyl), 44.2 (-NH-CH_2), 31.7, 29.5, 26.9, 22.7 (CH_2), 14.1 ppm (CH_3); GC/MS: rt = 7.06 min, M/Z = 177.



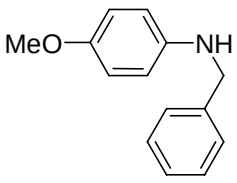
N-*n*-hexyl-4-benzonitrile (3d). Following Procedure A, 4-iodobenzonitrile (229 mg, 1.0 mmol) was allowed to react with *n*-hexylamine (152 mg, 1.5 mmol) for 10 h. The crude oil was purified by flash chromatography on silica gel eluent (20:1 petroleum ether: ethyl acetate) to provide 95 % yield of the desired product as a pale yellow oil. ^{1}H NMR($CDCl_3$): δ=7.39 (d, *J*=8.8 Hz, 2 H; Phenyl), 6.55 (d, *J*=8.4 Hz, 2 H; Phenyl), 4.51 (s, 1 H; NH), 3.11 (t, *J*=8.8 Hz, 2 H; -N-CH_2-), 1.66-1.59 (m, 2 H; CH_2), 1.43-1.25 (m, 6 H; CH_2), 0.88 ppm (t, *J*=6.8 Hz, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=151.3, 133.7, 120.5, 112.3 (Phenyl), 98.5 (CN), 43.5 (-N-CH_2), 31.5, 29.1, 26.7, 22.6 (CH_2), 14.0 ppm (CH_3); GC/MS: rt = 9.02 min, M/Z = 202.



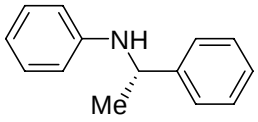
N-*n*-hexyl-4-chloroaniline (3e). Following Procedure A, 1-chloro-4-iodobenzene (238 mg, 1.0 mmol) was allowed to react with *n*-hexylamine (152 mg, 1.5 mmol) for 12 h. The crude oil was purified by flash chromatography on silica gel eluent (20:1 petroleum ether: ethyl acetate) to provide 91 % yield of the desired product as a pale purple oil. ^{1}H NMR($CDCl_3$): δ=7.11 (dd, *J*=6.8, 2.4 Hz, 2 H; Phenyl), 6.51 (dd, *J*=6.8, 2.0 Hz, 2 H; Phenyl), 3.59 (s, 1 H; NH), 3.06 (t, *J*=7.2 Hz, 2 H; -N-CH_2-), 1.65-1.58 (m, 2 H; CH_2), 1.45-1.30 (m, 6 H; CH_2), 0.92 ppm (t, *J*=7.2 Hz, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=147.1, 129.0, 121.6, 113.8 (Phenyl), 44.2 (-N-CH_2), 31.7, 29.4, 26.8, 22.7 (CH_2), 14.1 ppm (CH_3); GC/MS: rt = 8.07 min, M/Z = 257.



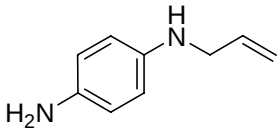
2-(4-methoxybenzylamino)benzoic acid (3f). Following Procedure A, 2-iodobenzoic acid (248 mg, 1.0 mmol) was allowed to react with 4-methoxybenzylamine (206 mg, 1.5 mmol) for 10 h. The crude oil was purified by flash chromatography on silica gel eluent (20:1 petroleum ether: ethyl acetate) to provide 92 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.79 (d, *J*=7.6 Hz, 1 H; Phenyl), 7.33-7.27 (m, 3 H; Phenyl), 6.90 (d, *J*=8.4 Hz, 2 H; Phenyl), 6.69 (d, *J*=8.4, 1 H; Phenyl), 6.54 (t, *J*=7.2, 1 H; Phenyl), 4.36 (s, 2 H; CH_2), 3.73 ppm (s, 3 H; -OCH_3); ^{13}C NMR($CDCl_3$) : δ=158.8, 151.1, 134.8, 132.1, 131.5, 128.9, 114.9, 114.4, 112.1, 111.5, 55.5, 45.9 ppm; MS (APCI): m/z: 258 [M-];.



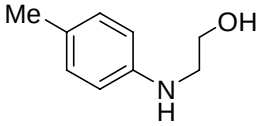
N-benzyl-4-methoxybenzenamine (3g). Following Procedure A, 1-iodo-4-methoxybenzene (234 mg, 1.0 mmol) was allowed to react with benzylamine (161 mg, 1.5 mmol) for 13 h. The crude oil was purified by flash chromatography on silica gel eluent (20:1 petroleum ether: ethyl acetate) to provide 91 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.43-7.33 (m, 5 H; Phenyl), 6.84 (d, *J*=8.8 Hz, 2 H; Phenyl), 6.65 (d, *J*=8.8 Hz, 2 H; Phenyl), 4.33 (s, 2 H; CH_2), 3.79 ppm (s, 3 H; -OCH_3); ^{13}C NMR($CDCl_3$) : δ=152.3, 142.5, 139.8, 128.7, 127.7, 127.3, 115.0, 114.3 (Phenyl), 55.7 (CH_2), 49.3 ppm (CH_3); GC/MS: rt = 8.92 min, M/Z = 213.



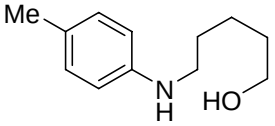
(S)-N-Phenyl-*α*-methylbenzylamine (3h)[4]. Following Procedure A, iodobenzene (204 mg, 1.0 mmol) was allowed to react with (S)-1-phenylethanamine (181 mg, 1.5 mmol) for 15 h. The crude oil was purified by flash chromatography on silica gel eluent (20:1 petroleum ether: ethyl acetate) to provide 85 % yield of the desired product as a pale yellow oil. ^{1}H NMR($CDCl_3$): δ=7.49-7.41 (m, 4 H; Phenyl), 7.32 (t, *J*=7.2 Hz, 1 H; Phenyl), 7.19 (t, *J*=8.0 Hz, 2 H; Phenyl) 6.76 (t, *J*=7.6 Hz, 1 H; Phenyl), 6.63 (d, *J*=8.0 Hz, 2 H; Phenyl), 4.58 (m, 1 H; CH), 4.19 (s, 1 H; NH), 1.62 ppm (d, *J*=6.8 Hz, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=147.3, 145.3, 129.2, 128.8, 126.9, 126.9, 117.4, 113.5 (Phenyl), 53.6 (CH), 25.1 ppm (CH_3); $[\alpha]_D^{18}$ = +16.9^o (c 1.0, methanol), literature: +16.9^o; GC/MS: rt = 7.85 min, M/Z = 197.



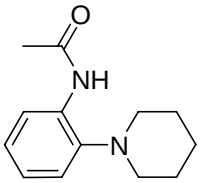
N-Allyl-*p*-phenylenediamine (3k). Following Procedure A, 4-iodobenzenamine (219 mg, 1.0 mmol) was allowed to react with allylamine (115 mg, 2 mmol) for 16 h. The crude oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 80 % yield of the desired product as a brown oil. ^{1}H NMR($CDCl_3$): δ=6.66-6.55 (m, 4 H; Phenyl), 6.01-5.92 (m, 1 H; -CH_2-C*H*=), 5.24 (m, 1 H; -CH=C*H*H), 5.13 (m, 1 H; -CH=CH*H*), 3.72 (br s, 2 H; CH2), 4.58 ppm (br s, 3 H; NH and NH_2); ^{13}C NMR($CDCl_3$) : δ=140.9, 137.9, 135.8, 116.9 (Phenyl), 116.3 (-CH=), 115.1 (=CH_2), 47.9 ppm (-CH_2-); GC/MS: rt = 6.93 min, M/Z = 148.



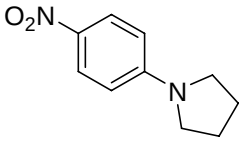
2-*p*-Tolylaminoethanol (3i). Following Procedure A, 1-iodo-4-methylbenzene (218 mg, 1.0 mmol) was allowed to react with 2-aminoethanol (92 mg, 1.5 mmol) for 8 h. The crude oil was purified by flash chromatography on silica gel eluent (5:1 petroleum ether: ethyl acetate) to provide 98 % yield of the desired product as a pale yellow oil. ^{1}H NMR($CDCl_3$): δ=7.03 (d, *J*=8.0 Hz, 2 H; Phenyl), 6.60 (d, *J*=8.4 Hz, 2 H; Phenyl), 3.78 (t, *J*=5.2 Hz, 2 H; -O-CH_2-), 3.43 (br s, 2 H; NH and OH), 3.24 (t, *J*=5.2 Hz, 2 H; -N-CH_2), 2.30 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=145.8, 129.8, 127.3, 113.7 (Phenyl), 61.1 (-O-CH_2-), 46.7 (-N-CH_2-), 20.4 ppm (CH_3); GC/MS: rt = 6.82 min, M/Z = 151.



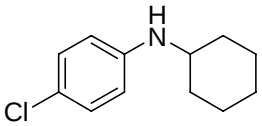
5-[(4-methylphenyl)amino]-1-Pentanol (3j). Following Procedure A, 1-iodo-4-methylbenzene (218 mg, 1.0 mmol) was allowed to react with 5-aminopentan-1-ol (155 mg, 1.5 mmol) for 12 h. The crude oil was purified by flash chromatography on silica gel eluent (5:1 petroleum ether: ethyl acetate) to provide 93 % yield of the desired product as a yellow oil. ^{1}H NMR($CDCl_3$): δ=7.01 (d, *J*=8.0 Hz, 2 H; Phenyl), 6.56 (d, *J*=8.0 Hz, 2 H; Phenyl), 3.61 (t, *J*=6.8 Hz, 2 H; -O-CH_2-), 3.09 (t, *J*=6.8 Hz, 2 H; -N-CH_2), 2.92 (br s, 2 H; NH and OH), 2.27 (s, 3 H; CH_3), 1.68-1.57 (m, 4 H; CH_2), 1.51-1.45 ppm (m, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=146.2, 129.8, 126.6, 113.2 (Phenyl), 62.6 (-O-CH_2-), 44.4 (-N-CH_2-), 32.5 (CH_3), 29.4, 23.4, 20.4 ppm (CH_2); GC/MS: rt = 8.31 min, M/Z = 193.



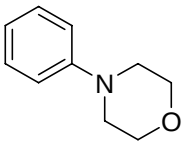
N-[2-(1-piperidinyl)phenyl]-acetamide (3o). Following Procedure A, N-(2-iodophenyl)acetamide (261 mg, 1.0 mmol) was allowed to react with piperidine (128 mg, 1.5 mmol) for 15 h. The crude oil was purified by flash chromatography on silica gel eluent (8:1 petroleum ether: ethyl acetate) to provide 83 % yield of the desired product as a pale yellow oil. ^{1}H NMR($CDCl_3$): δ=8.54 (br s, 1 H; NH), 8.33 (d, *J*=7.6 Hz, 1 H; Phenyl), 7.14-7.02 (m, 3 H; Phenyl), 2.79 (br s, 4 H; -N-CH_2-), 2.21 (s, 3 H; CH_3), 1.74 (br s, 4 H; CH_2), 1.62 ppm (br s, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=168.0 (-C=O), 142.4, 133.5, 124.9, 123.5, 120.3, 119.3 (Phenyl), 53.7 (-N-CH_2-), 26.9, 24.9, 24.0 ppm (CH_2); GC/MS: rt = 8.67 min, M/Z = 218.



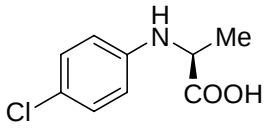
4-pyrrolidinonitrobenzene (3m). Following Procedure A, 1-iodo-4-nitrobenzene (249 mg, 1.0 mmol) was allowed to react with pyrrolidine (107 mg, 1.5 mmol) for 24 h. The crude oil was purified by flash chromatography on silica gel eluent (8:1 petroleum ether: ethyl acetate) to provide 95 % yield of the desired product as a yellow solid. ^{1}H NMR(*d*-DMSO): δ=8.02 (d, *J*=9.6 Hz, 2 H; Phenyl), 6.56 (d, *J*=9.2 Hz, 2 H; Phenyl), 3.34 (t, *J*=5.6 Hz, 4 H; -N-CH_2-), 1.96 ppm (t, *J*=6.8 Hz, 4 H; CH_2); ^{13}C NMR(*d*-DMSO) : δ=152.3, 135.7, 126.4, 111.3 (Phenyl), 48.2 (-N-CH_2-), 25.4 ppm (CH_2); GC/MS: rt = 9.56 min, M/Z = 192.



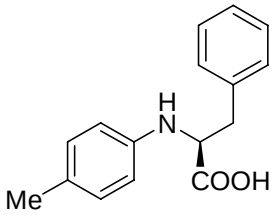
N-cyclohexyl-4-chloro-benzenamine (3l). Following Procedure A, 1-chloro-4-iodobenzene (238 mg, 1.0 mmol) was allowed to react with cyclohexanamine (149 mg, 1.5 mmol) for 19 h. The crude oil was purified by flash chromatography on silica gel eluent (15:1 petroleum ether: ethyl acetate) to provide 86 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.28-7.10 (m, 2 H; Phenyl), 6.53 (m, 2 H; Phenyl), 3.61 (br s, 1 H; NH), 3.27-3.19 (m, 1 H; -N-CH-), 2.08-2.05 (m, 2 H; CH_2), 1.82-1.77 (m, 2 H; CH_2), 1.71-1.66 (m, 1 H; CH_2), 1.45-1.24 ppm (m, 5 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=145.9, 129.1, 121.4, 114.3 (Phenyl), 51.9 (-N-CH-), 33.3, 25.9, 24.9 ppm (CH_2); GC/MS: rt = 8.31 min, M/Z = 209.



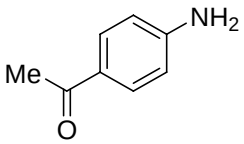
(4-morpholinyl)benzene (3n). Following Procedure A, iodobenzene (204 mg, 1.0 mmol) was allowed to react with morpholine (131 mg, 1.5 mmol) for 24 h. The crude oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 90 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.34-7.29 (m, 2 H; Phenyl), 6.97-6.91 (m, 3 H; Phenyl), 3.89 (t, *J*=4.8 Hz, 4 H; -O-CH_2-), 3.18 ppm (t, *J*=4.8 Hz, 4 H; -N-CH_2-); ^{13}C NMR($CDCl_3$) : δ=151.2, 129.2, 120.2, 115.8 (Phenyl), 66.9 (-O-CH_2-), 49.5 ppm (-N-CH_2-); GC/MS: rt = 6.59 min, M/Z = 163.



(S)-2-(4-chlorophenylamino)propanoic acid[5] **(3p)**. Following Procedure C, 1-chloro-4-iodobenzene (238 mg, 1.0 mmol) was allowed to react with (S)-2-aminopropanoic acid (134 mg, 1.5 mmol) for 11 h. yield: 89 %. the desired product was a gray solid. ^{1}H NMR(*d*-DMSO): δ=7.08 (dd, *J*=6.8, 2.0 Hz, 2 H; Phenyl), 6.55 (dd, *J*=6.8, 2.0 Hz, 2 H; Phenyl), 3.92 (m, 1 H; CH), 1.36 ppm (d, *J*=7.2 Hz, 3 H; CH_3); ^{13}C NMR(*d*-DMSO) : δ=176.0 (COOH), 147.2, 128.9, 119.9, 114.2 (Phenyl), 51.5 (CH), 18.5 ppm (CH_3); $[\alpha]_D^{18}$ = -73.3^o (c 0.33, MeOH), literature: -73.5^o; MS (APCI): M/Z: 199 [M-].

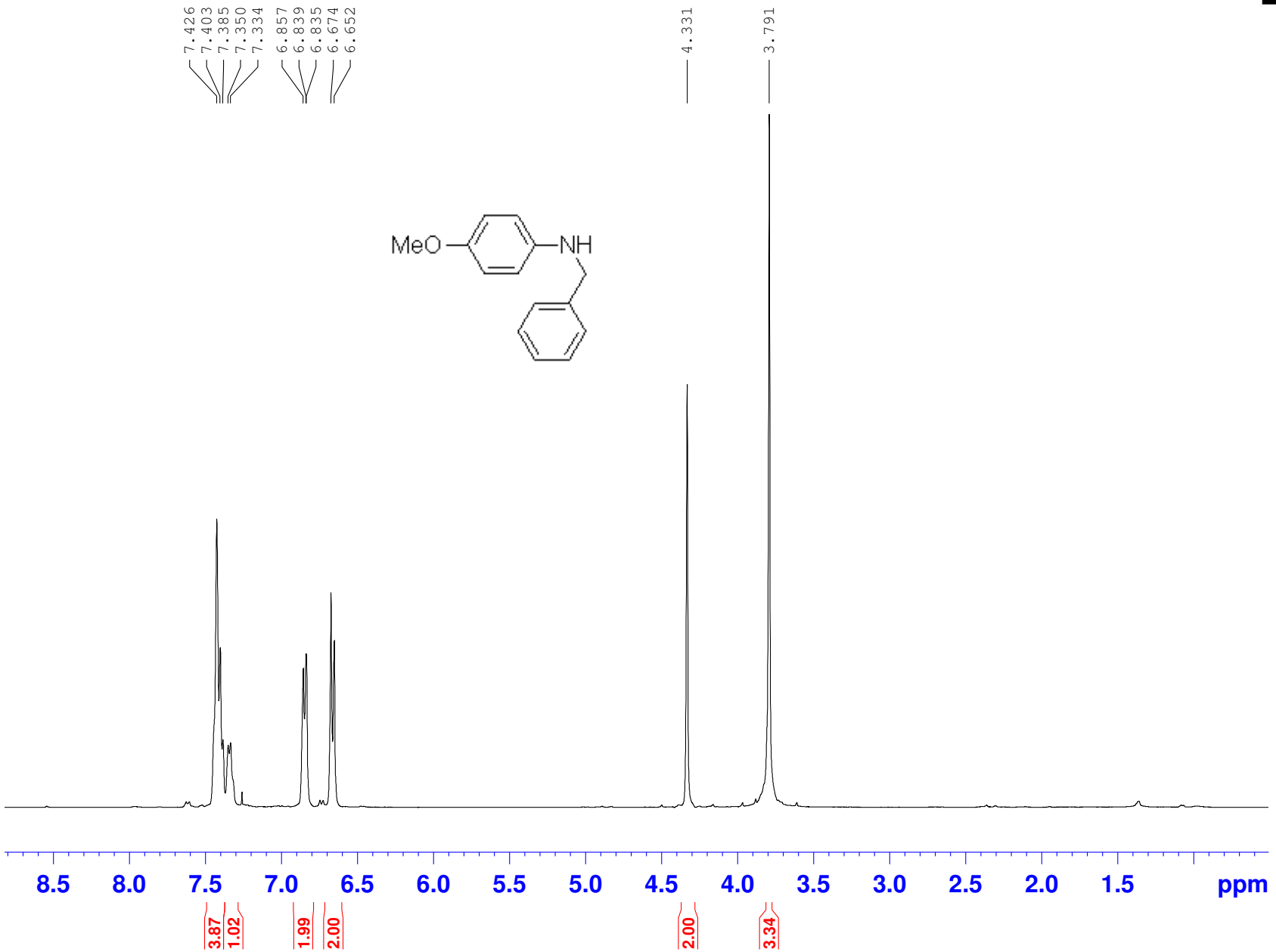


(S)-2-(*p*-toluidino)-3-phenylpropanoic acid (3q). Following Procedure C, 1-iodo-4-methylbenzene (218 mg, 1.0 mmol) was allowed to react with (S)-2-amino-3-phenylpropanoic acid (248 mg, 1.5 mmol) for 12 h. yield: 85 %. the desired product was a gray solid. ^{1}H NMR(*d*-DMSO): δ=7.29-7.20 (m, 5 H; Phenyl), 6.87 (d, *J*=7.6 Hz, 2 H; Phenyl), 6.49 (d, *J*=7.6 Hz, 2 H; Phenyl), 4.09 (br s, 1 H; NH), 3.35 (br s, 1 H; CH), 3.08-2.94 (m, 2 H; CH_2), 2.13 ppm (s, 3 H; CH_3); ^{13}C NMR(*d*-DMSO) : δ=175.2 (COOH), 145.9, 138.4, 129.8, 129.6, 128.6, 126.8, 125.3, 113.2 (Phenyl), 58.2 (CH), 38.3 (CH_2), 20.5 ppm (CH_3); $[\alpha]_D^{18}$ = +1.6^o (c 0.20, acetone); MS (APCI): M/Z: 254 [M-].



1-(4-aminophenyl)ethanone (3r). Following Procedure A, a mixture of 4'-iodoacetophenone (246 mg, 1.0 mmol), CuI (20%), L7 (40%), NH_4Cl (2 mmol), Cs_2CO_3 (2 mmol), DMF (1 mL), H_2O (50 uL) was stirred under Argon at room temperature for 20 h. The crude oil was purified by flash chromatography on silica gel eluent (1:2 petroleum ether: ethyl acetate) to provide 92 % yield of the desired product as a pale yellow solid. ^{1}H NMR($CDCl_3$): δ=7.76 (d, *J*=8.4 Hz, 2 H; Phenyl), 6.60 (d, *J*=8.0 Hz, 2 H; Phenyl), 4.27 (s, 2 H; -NH_2), 2.47 ppm (s, 3 H; -CH_3); ^{13}C NMR($CDCl_3$) : δ=196.7 (-C=O), 151.5, 130.8, 127.5, 113.7 (Phenyl), 26.1 ppm (-CH_3); GC/MS: rt = 6.93 min, M/Z = 135.

wdp-53793

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RG                  35.9
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DE                  6.00 usec
TE                 299.0 K
D1            1.00000000 sec
TD0                    1

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F2 - Processing parameters
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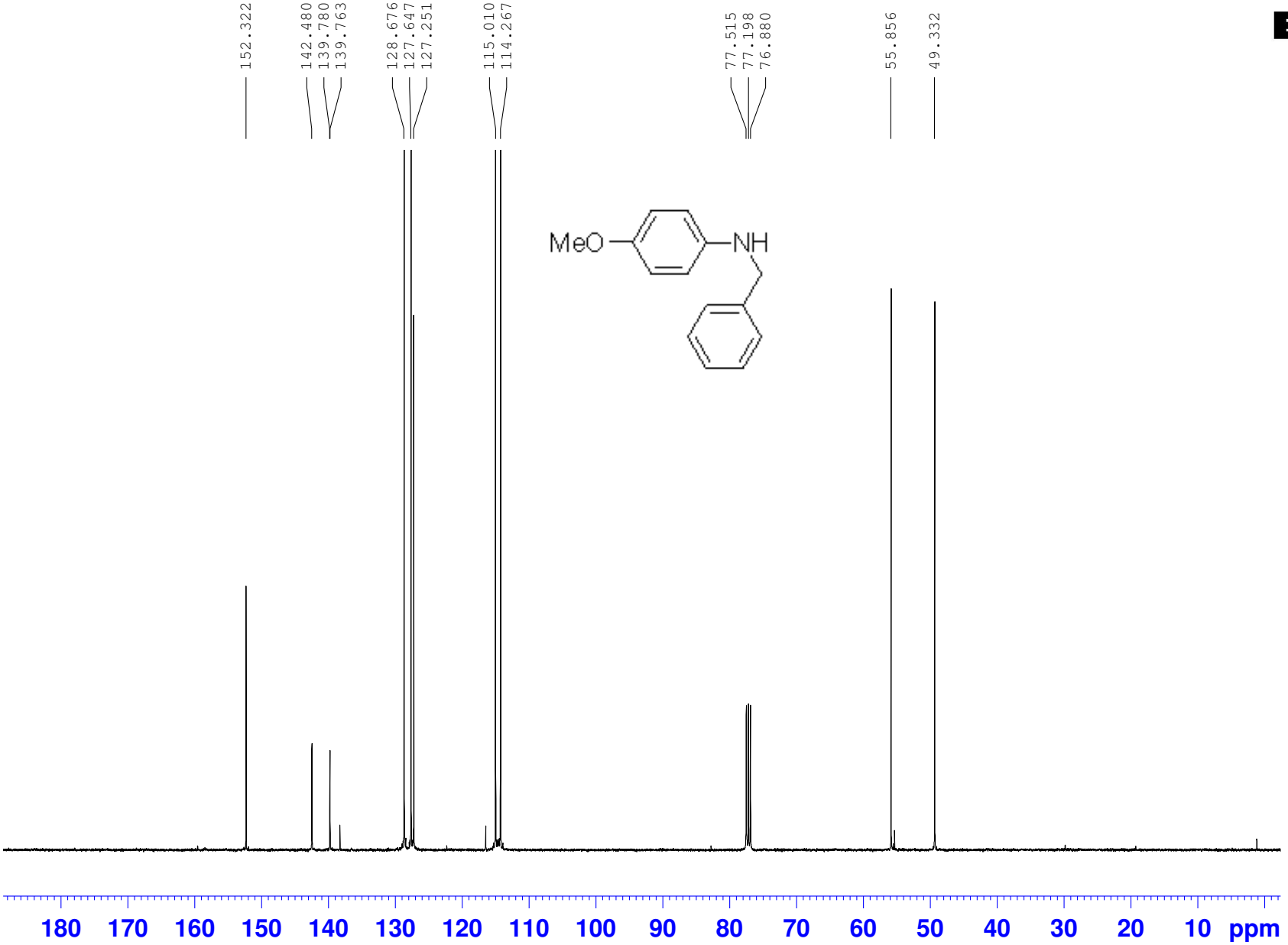
wdp-53793

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SOLVENT            CDCl3
NS                  2048
DS                     4
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PCPD2             80.00 usec
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SFO2         400.1316005 MHz

F2 - Processing parameters
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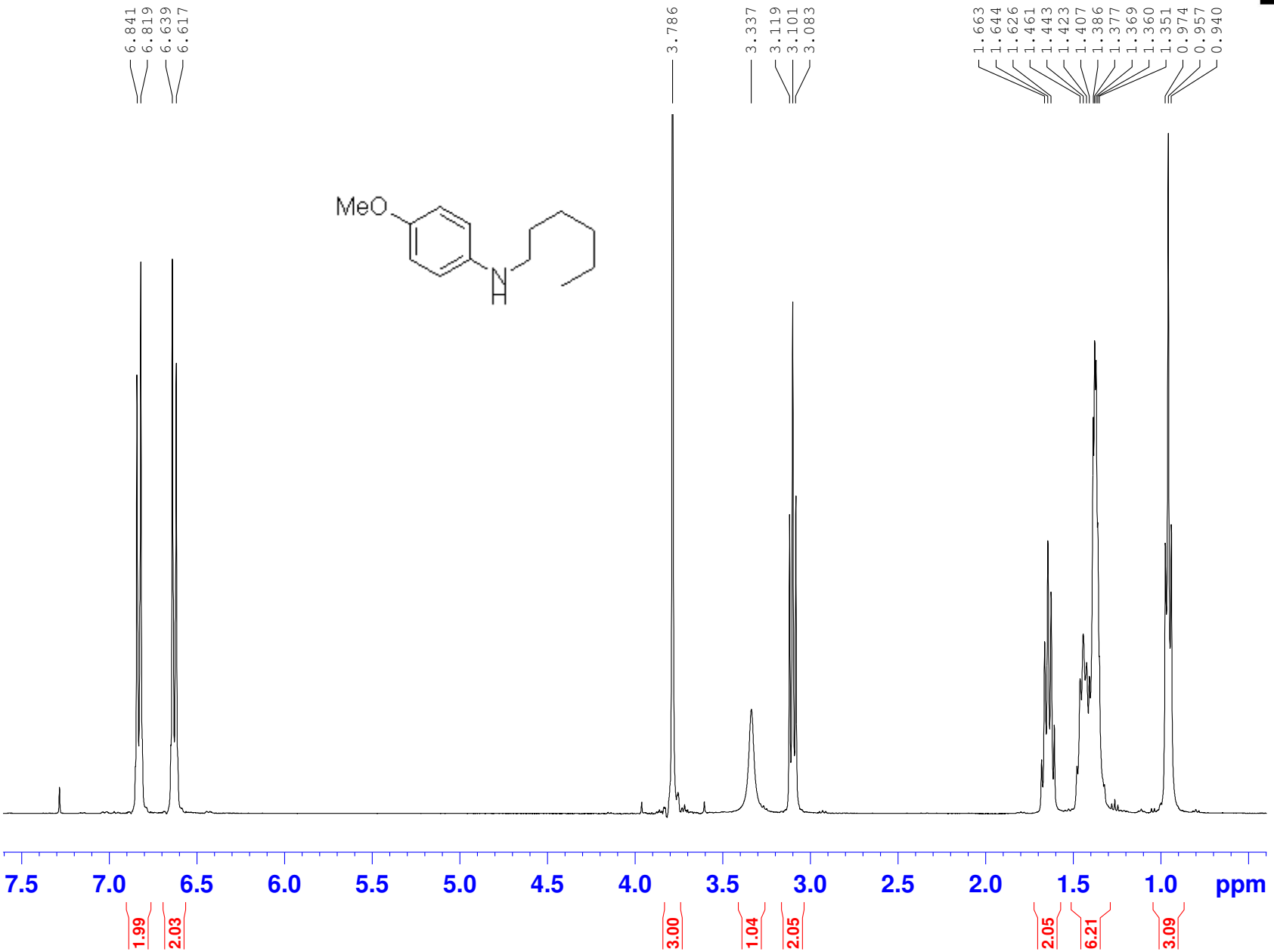
wdp125202



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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 25.4
DW 60.400 usec
DE 6.00 usec
TE 299.8 K
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TD0 1

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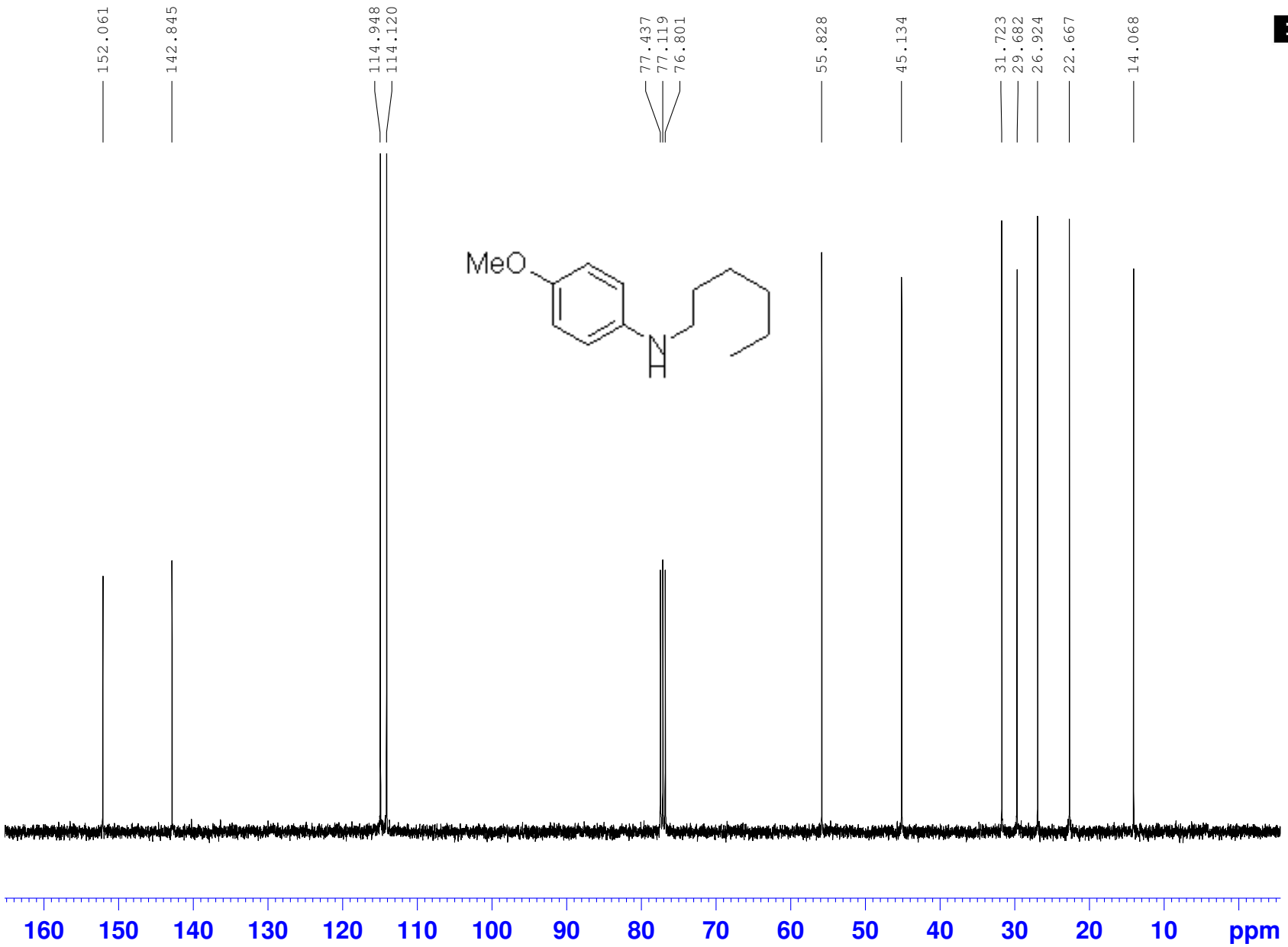
wdp125202

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PROCNO                  1

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NS                   122
DS                     4
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RG                 912.3
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TD0                    1

======== CHANNEL f1 ========
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SFO1         100.6228298 MHz

======== CHANNEL f2 ========
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NUC2                 1H
PCPD2             80.00 usec
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PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
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WDW                 EM
SSB                    0
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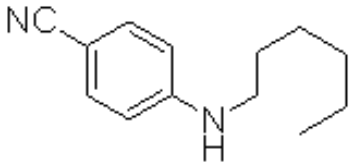
125275



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TD 65536
SOLVENT CDCl3
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DS 2
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FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
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D1 1.00000000 sec
TD0 1

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F2 - Processing parameters
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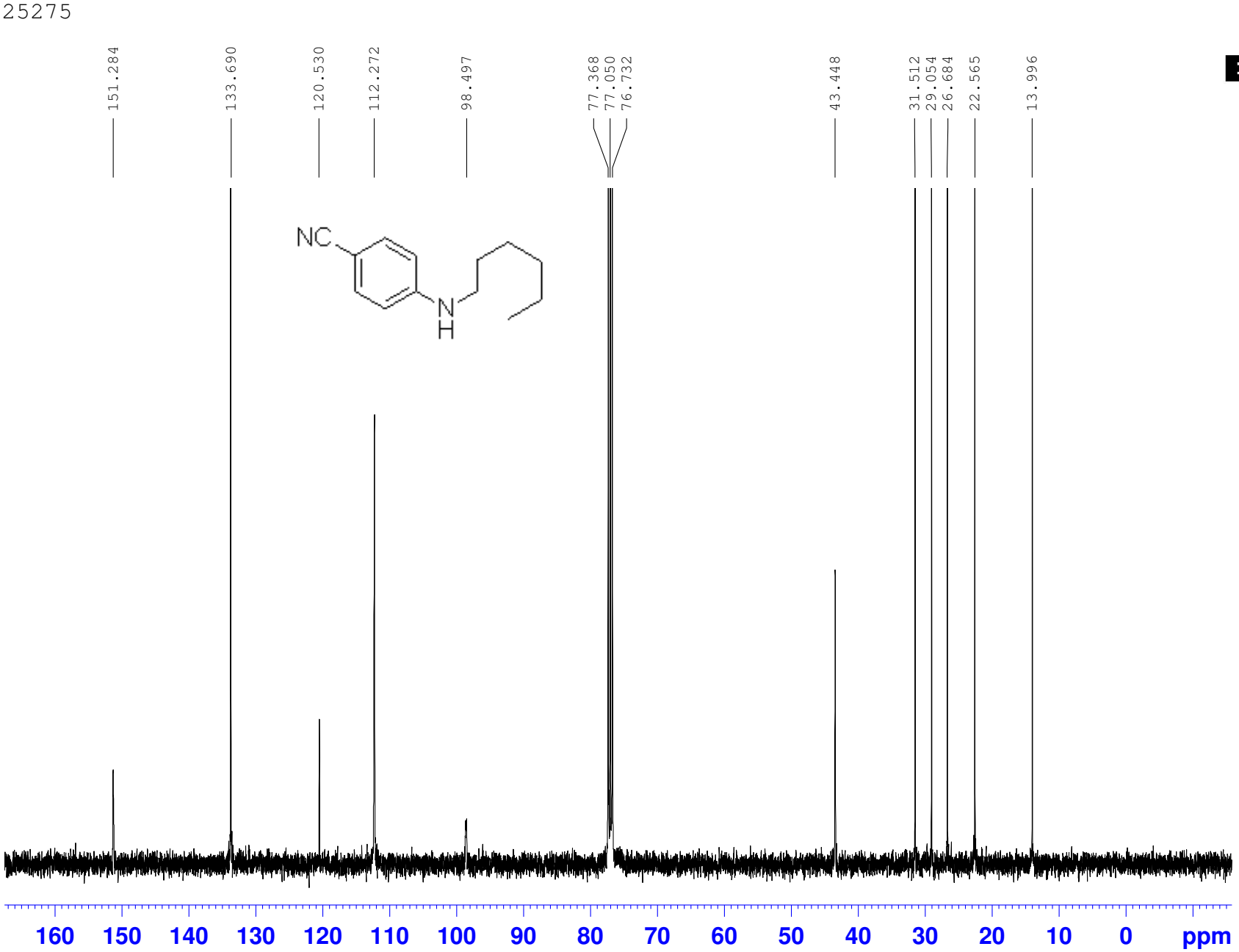
125275



Current Data Parameters

NAME	wdp
EXPNO	268
PROCNO	1

F2 - Acquisition Parameters

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RG	1024
DW	20.850 usec
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d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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P1	12.20 usec
PL1	1.00 dB
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======== CHANNEL f2 ========

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PL12	15.39 dB
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SFO2	400.1316005 MHz

F2 - Processing parameters

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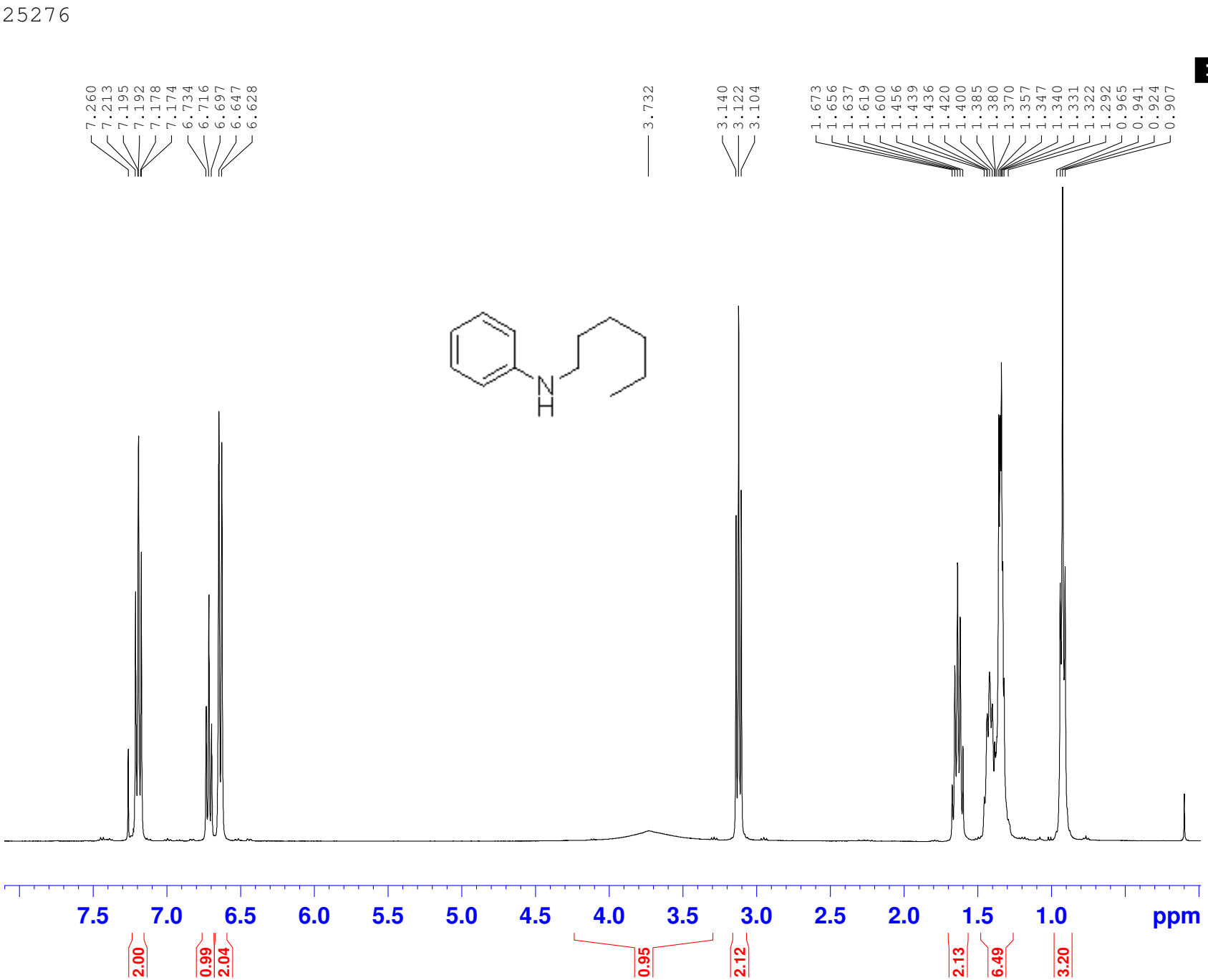
125276



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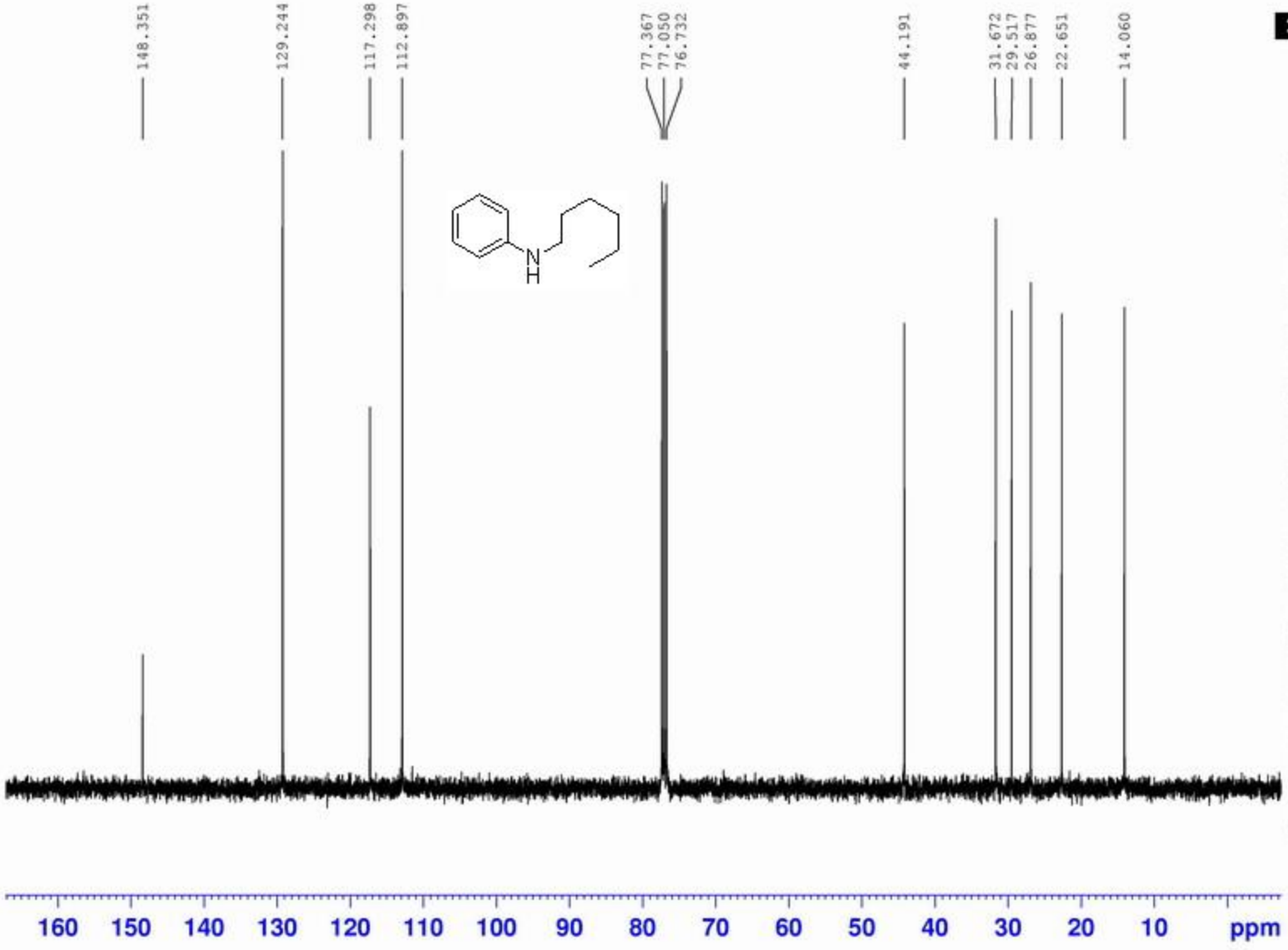
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125276




```
Current Data Parameters
NAME                  wdp
EXPNO                 269
PROCNO                  1

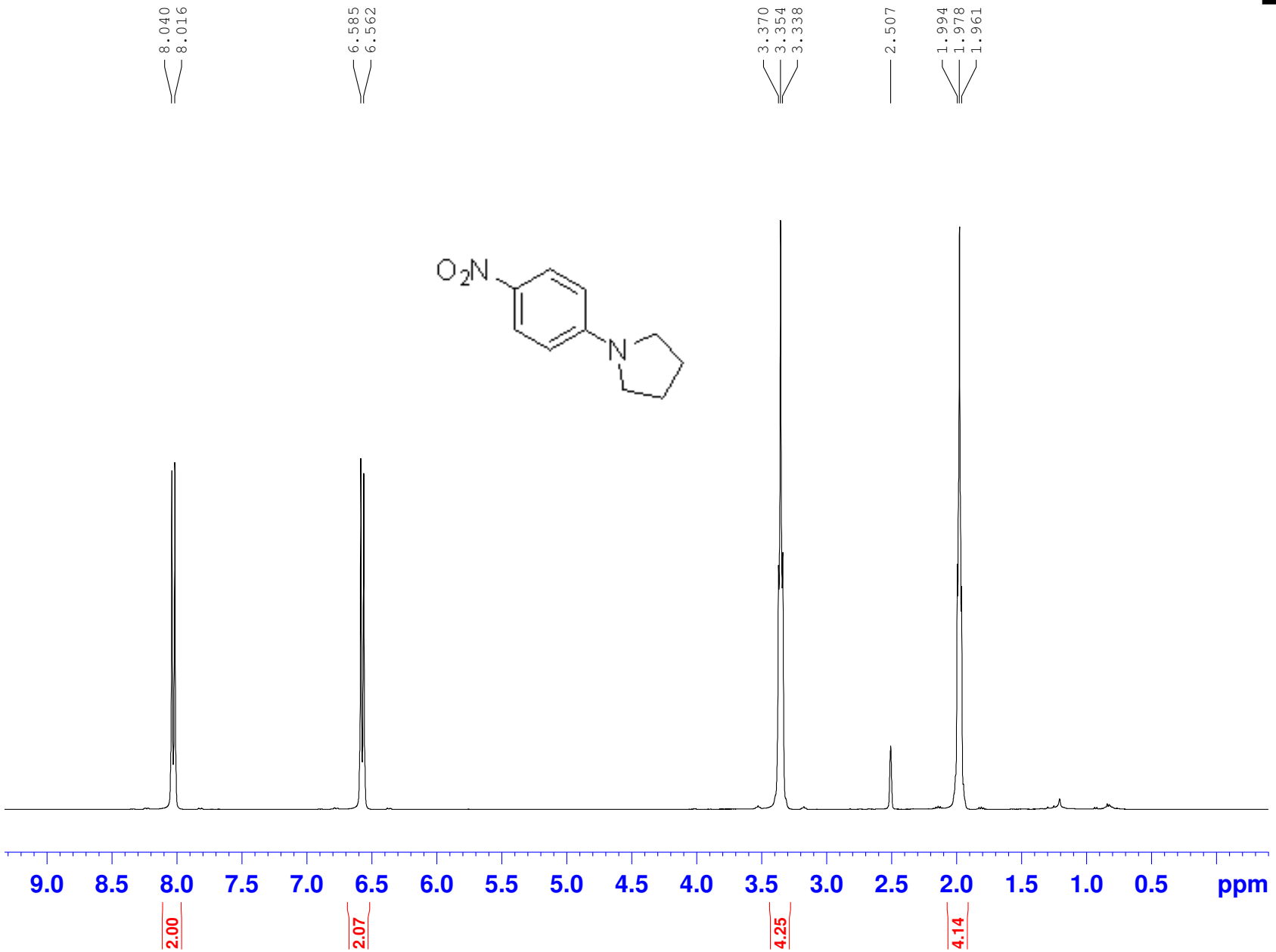
F2 - Acquisition Parameters
Date_            20080809
Time                14.27
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   126
DS                     4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                812.7
DW               20.850 usec
DE                6.00 usec
TE               299.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                12.20 usec
PL1                1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

125279



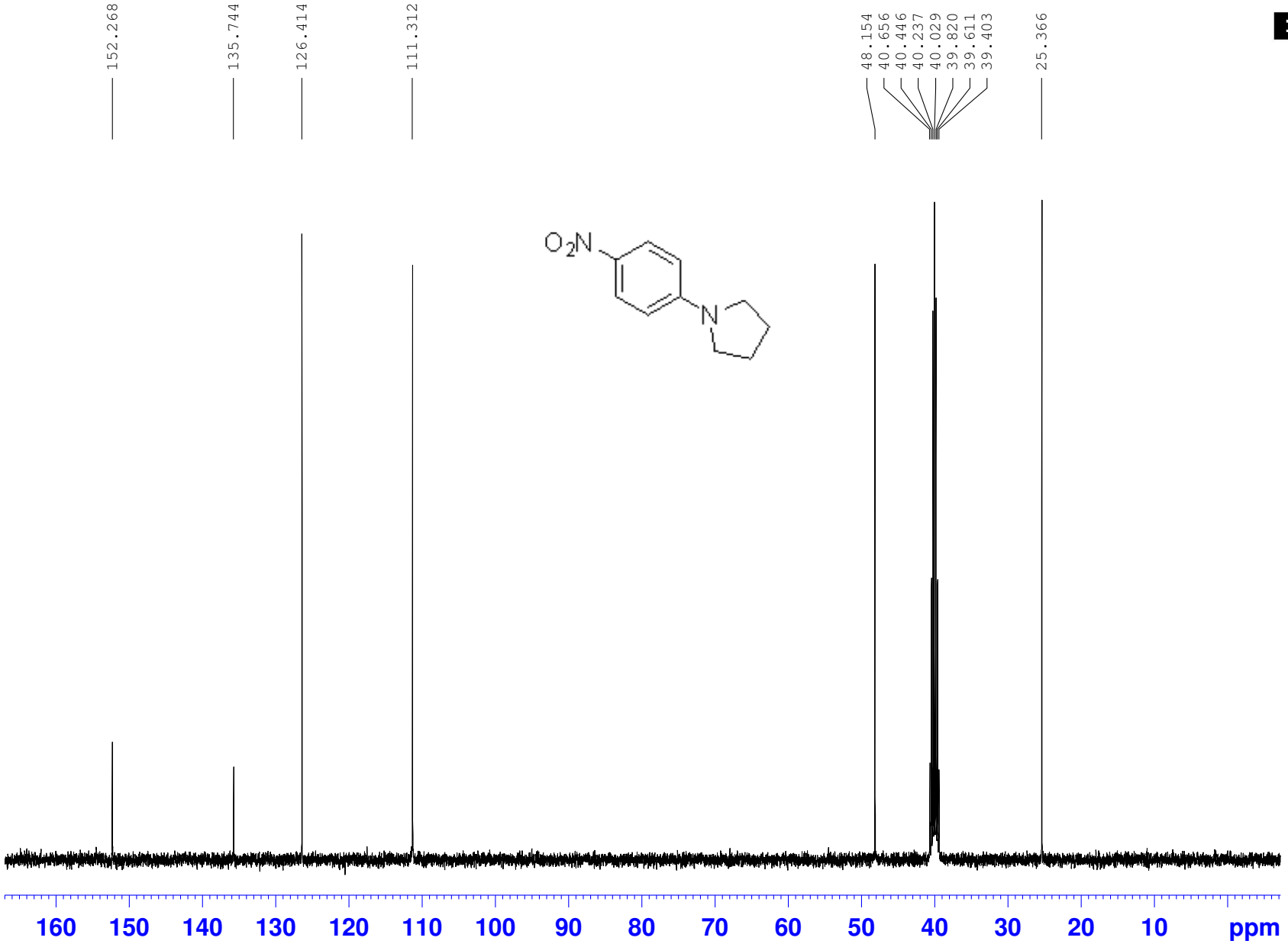
Current Data Parameters
NAME wdp
EXPNO 270
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080810
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 80.6
DW 60.400 usec
DE 6.00 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125279




```
Current Data Parameters
NAME                   wdp
EXPNO                  273
PROCNO                   1

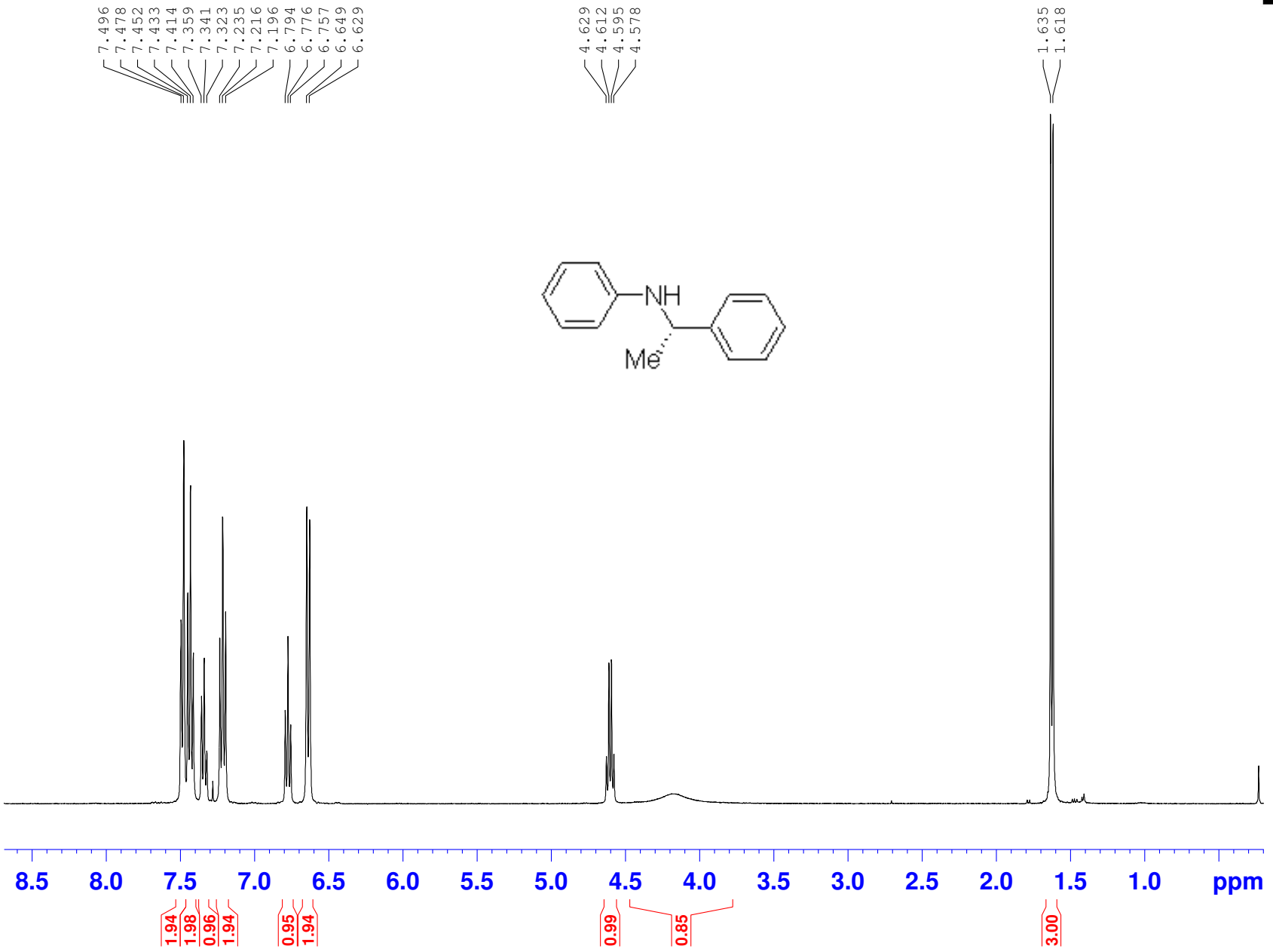
F2 - Acquisition Parameters
Date_           20080810
Time                14.30
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                    102
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                1290.2
DW                20.850 usec
DE                 6.00 usec
TE                301.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                 1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

125280



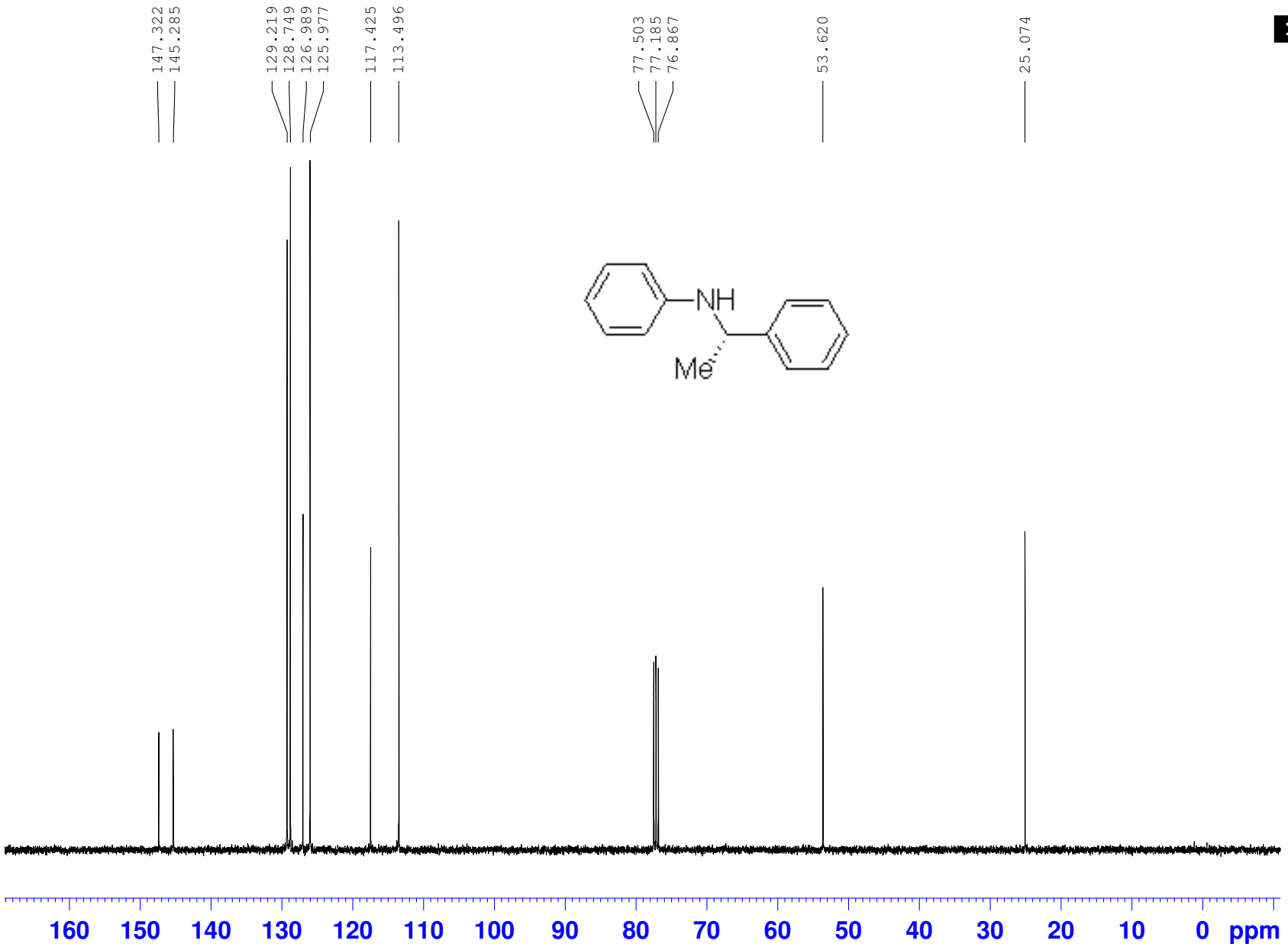

```
Current Data Parameters
NAME                   wdp
EXPNO                  275
PROCNO                   1

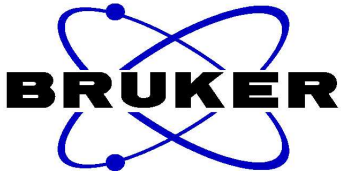
F2 - Acquisition Parameters
Date_           20080812
Time               10.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     2
DS                     2
SWH             8278.146 Hz
FIDRES             0.126314 Hz
AQ              3.9584243 sec
RG                     4
DW                 60.400 usec
DE                  6.00 usec
TE                 299.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.60 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

125280






```
Current Data Parameters
NAME                  wdp
EXPNO                 276
PROCNO                  1

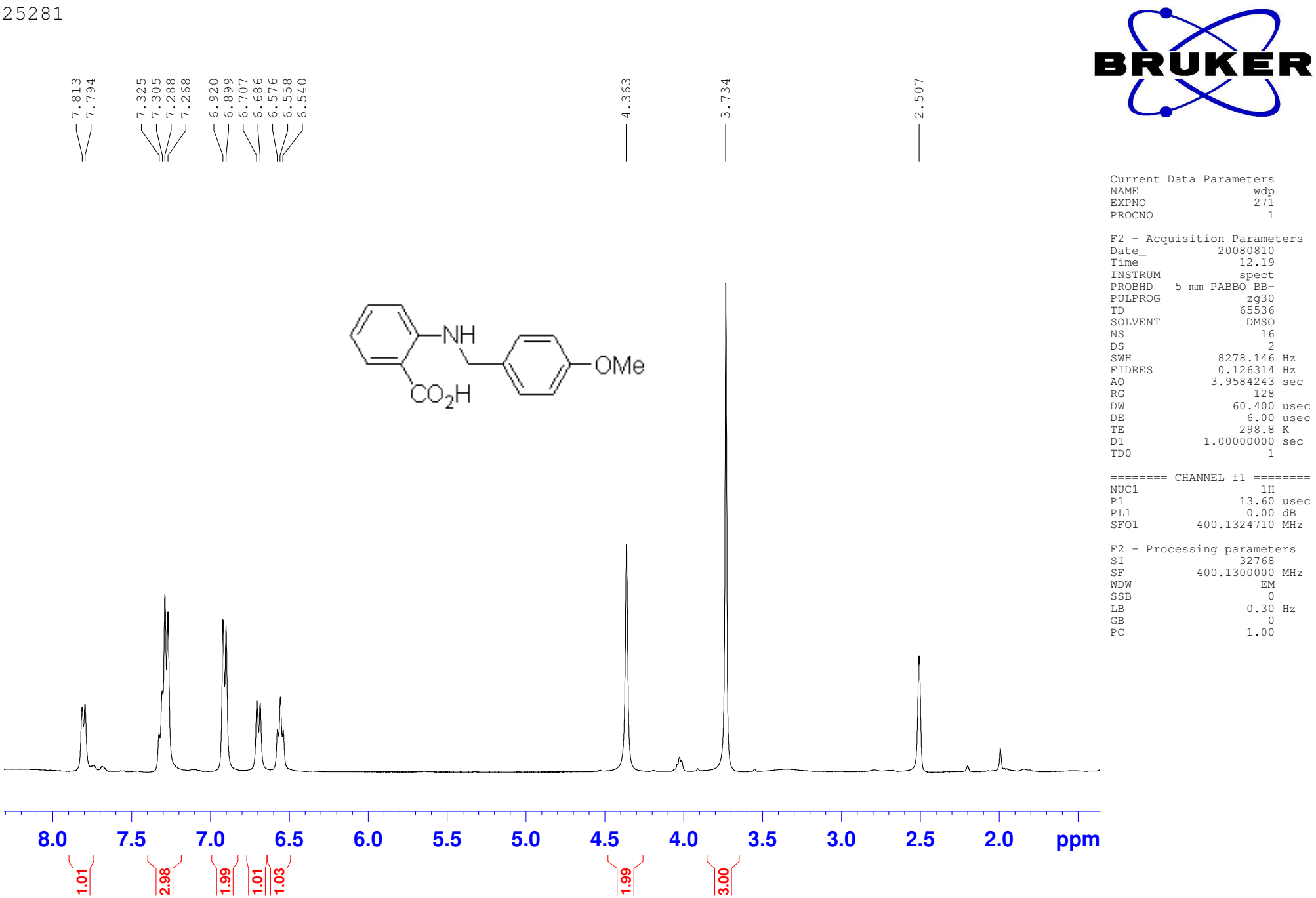
F2 - Acquisition Parameters
Date_           20080812
Time               10.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   63
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                912.3
DW               20.850 usec
DE                6.00 usec
TE                300.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

125281



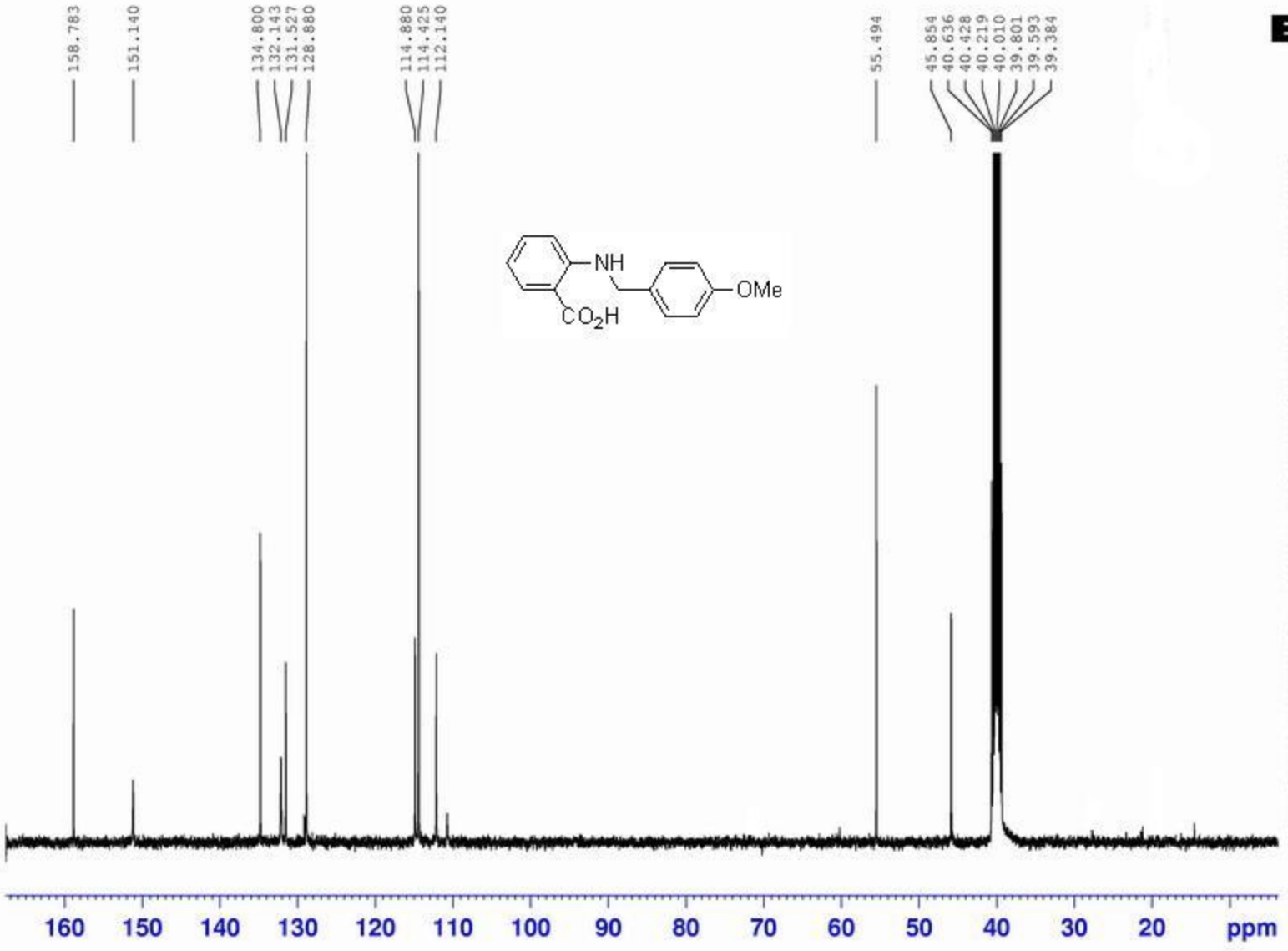
Current Data Parameters
NAME wdp
EXPNO 271
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080810
Time 12.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125281



BRUKER

```
Current Data Parameters
NAME                  wdp
EXPNO                 272
PROCNO                  1

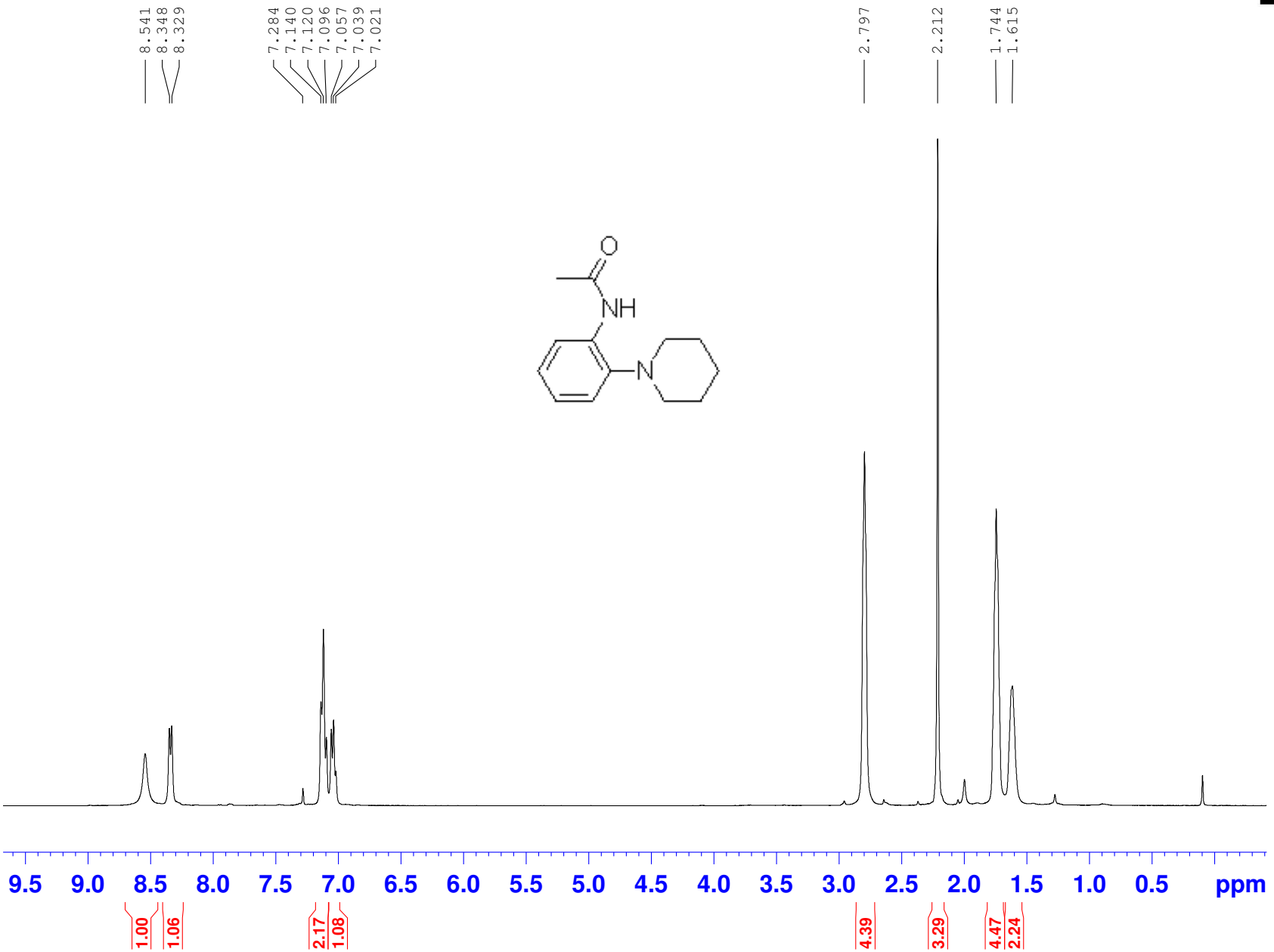
F2 - Acquisition Parameters
Date_              20080810
Time                  12.30
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                     2036
DS                        4
SWH               23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                   1149.4
DW                  20.850 usec
DE                   6.00 usec
TE                  299.5 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.20 usec
PL1                   1.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   0.00 dB
PL12                 15.39 dB
PL13                 15.50 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF            100.6127690 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```

125283



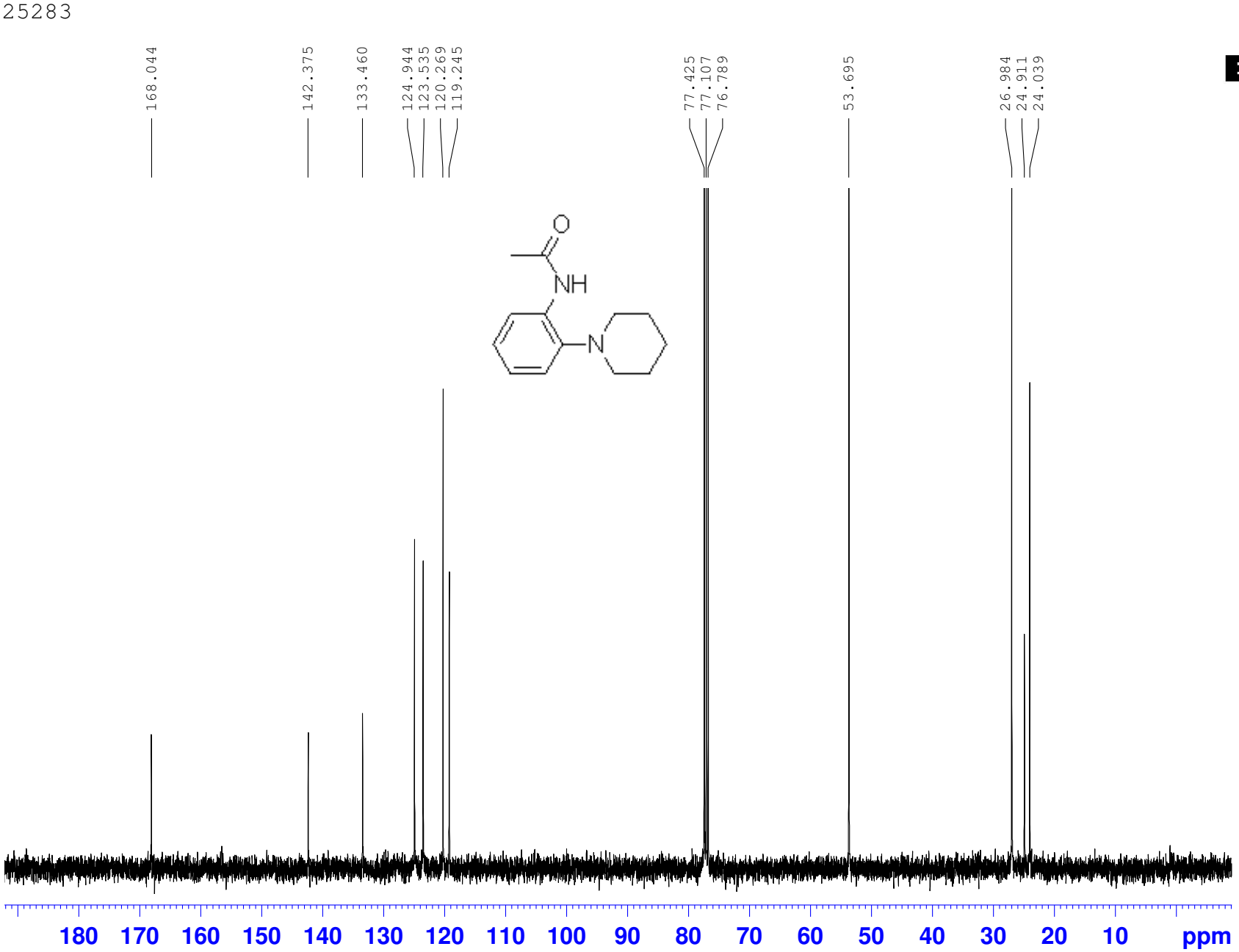
Current Data Parameters
NAME wdp
EXPNO 274
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080812
Time 10.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125283




```
Current Data Parameters
NAME                  wdp
EXPNO                 277
PROCNO                  1

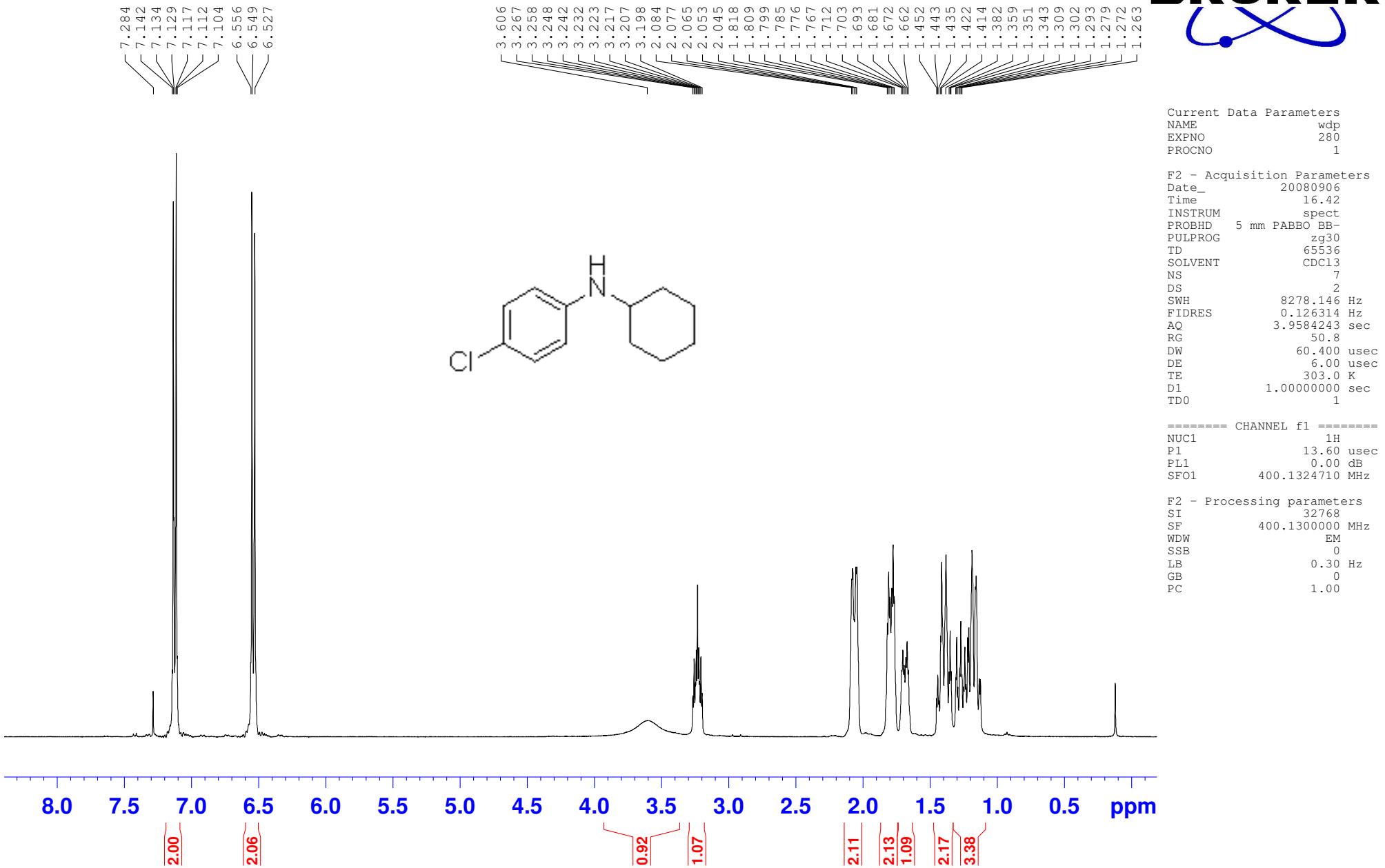
F2 - Acquisition Parameters
Date_            20080812
Time                10.18
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   106
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                1290.2
DW                20.850 usec
DE                 6.00 usec
TE                300.4 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

125287



Current Data Parameters

NAME	wdp
EXPNO	280
PROCNO	1

F2 - Acquisition Parameters

Date_	20080906
Time	16.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	7
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	50.8
DW	60.400 usec
DE	6.00 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

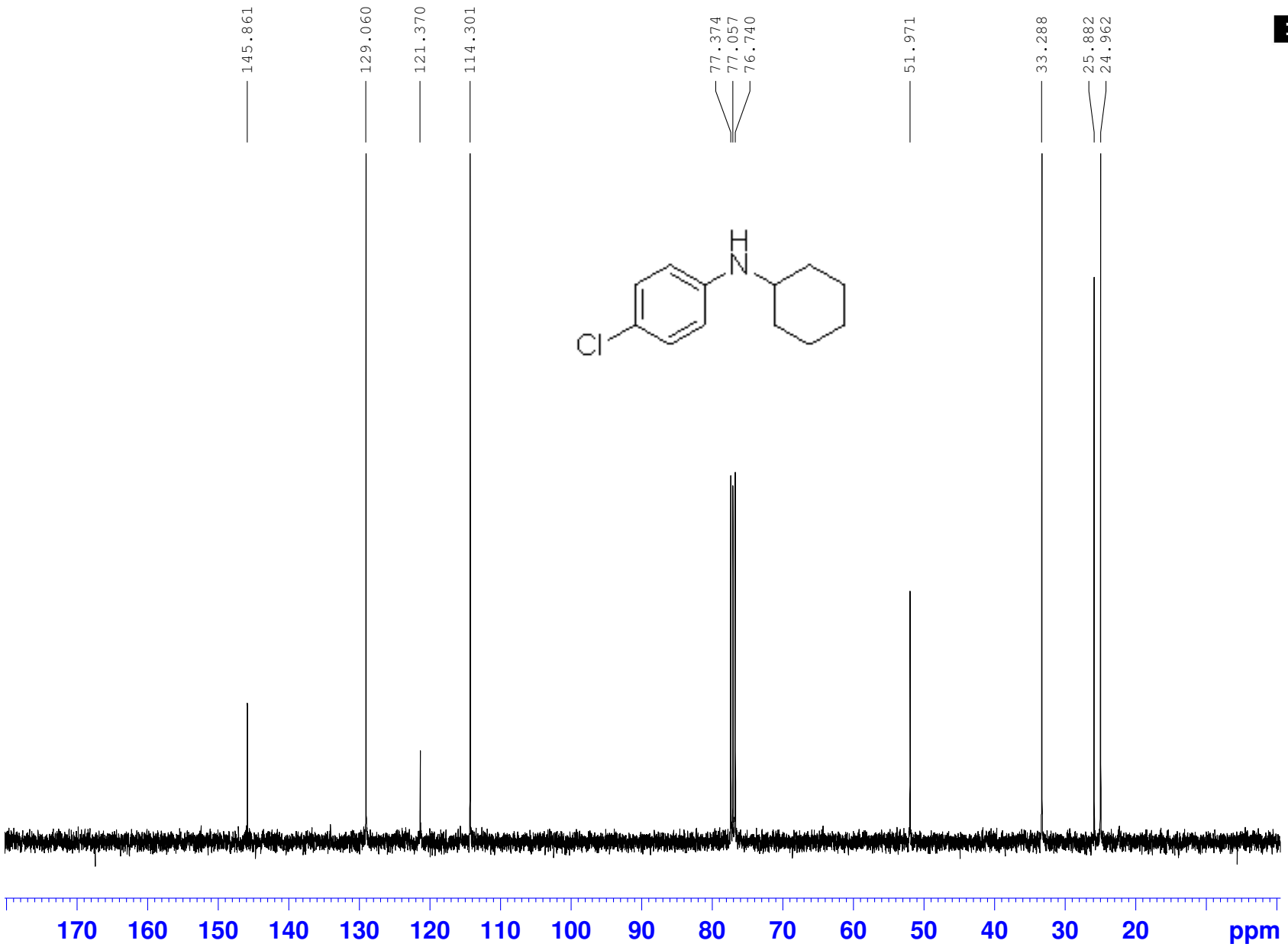
======== CHANNEL f1 ========

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

125287



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Current Data Parameters
NAME wdp
EXPNO 281
PROCNO 1

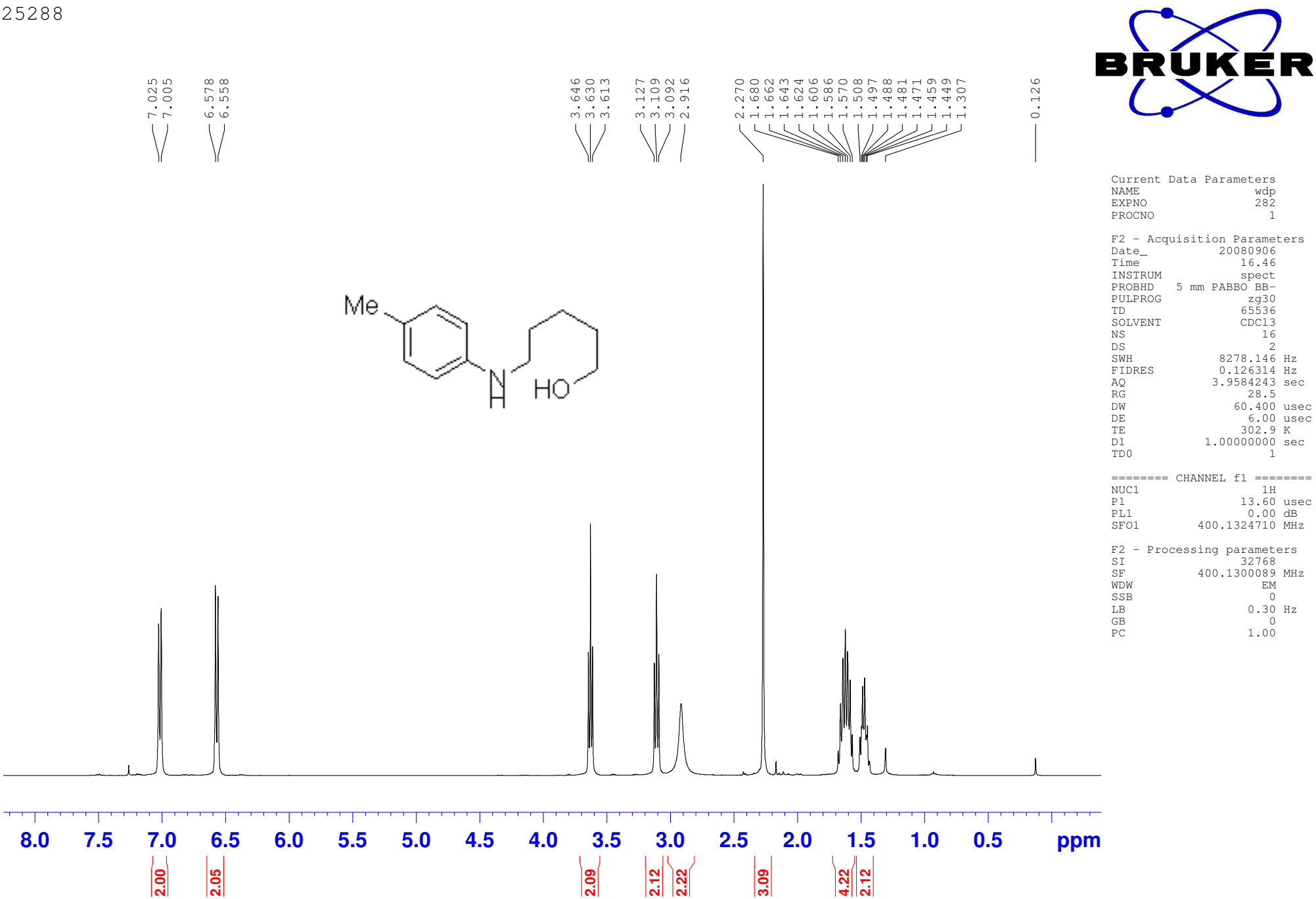
F2 - Acquisition Parameters
Date_ 20080906
Time 16.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 83
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 812.7
DW 20.850 usec
DE 6.00 usec
TE 303.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125288



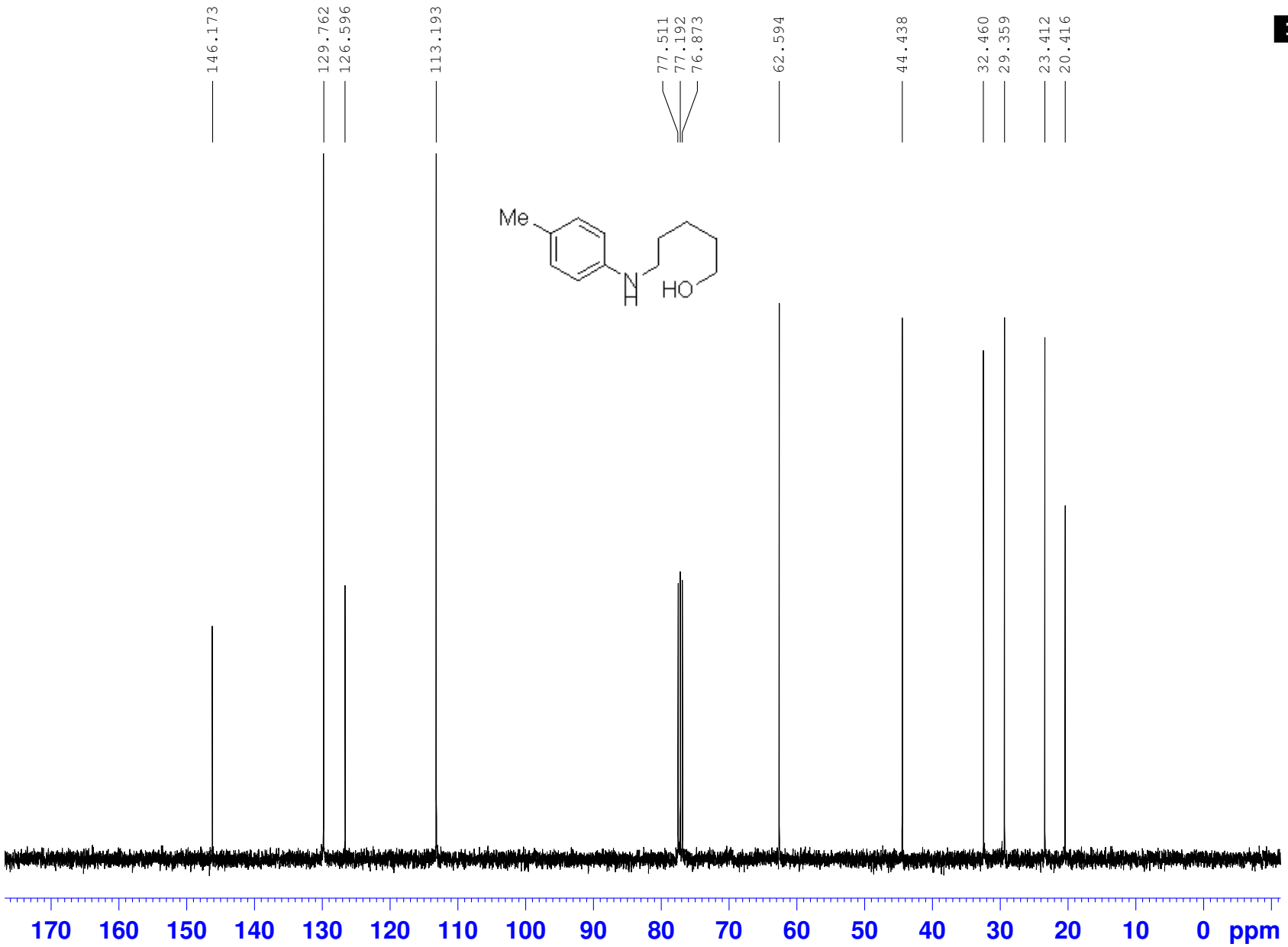

```
Current Data Parameters
NAME                  wdp
EXPNO                 282
PROCNO                  1

F2 - Acquisition Parameters
Date_           20080906
Time               16.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  28.5
DW                60.400 usec
DE                 6.00 usec
TE                302.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.60 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300089 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

125288



Current Data Parameters
NAME wdp
EXPNO 283
PROCNO 1

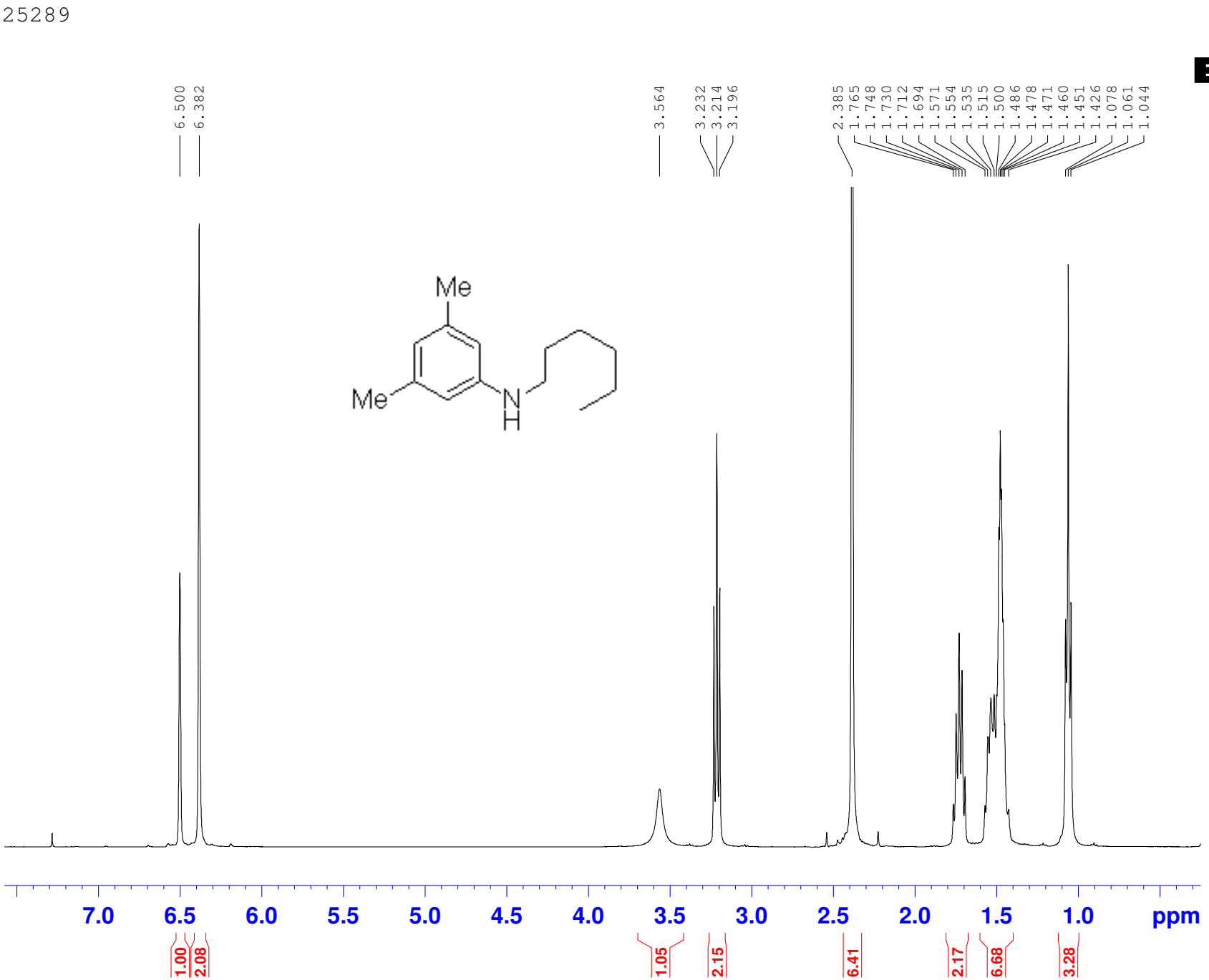
F2 - Acquisition Parameters
Date_ 20080906
Time 16.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 45
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 724.1
DW 20.850 usec
DE 6.00 usec
TE 303.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125289



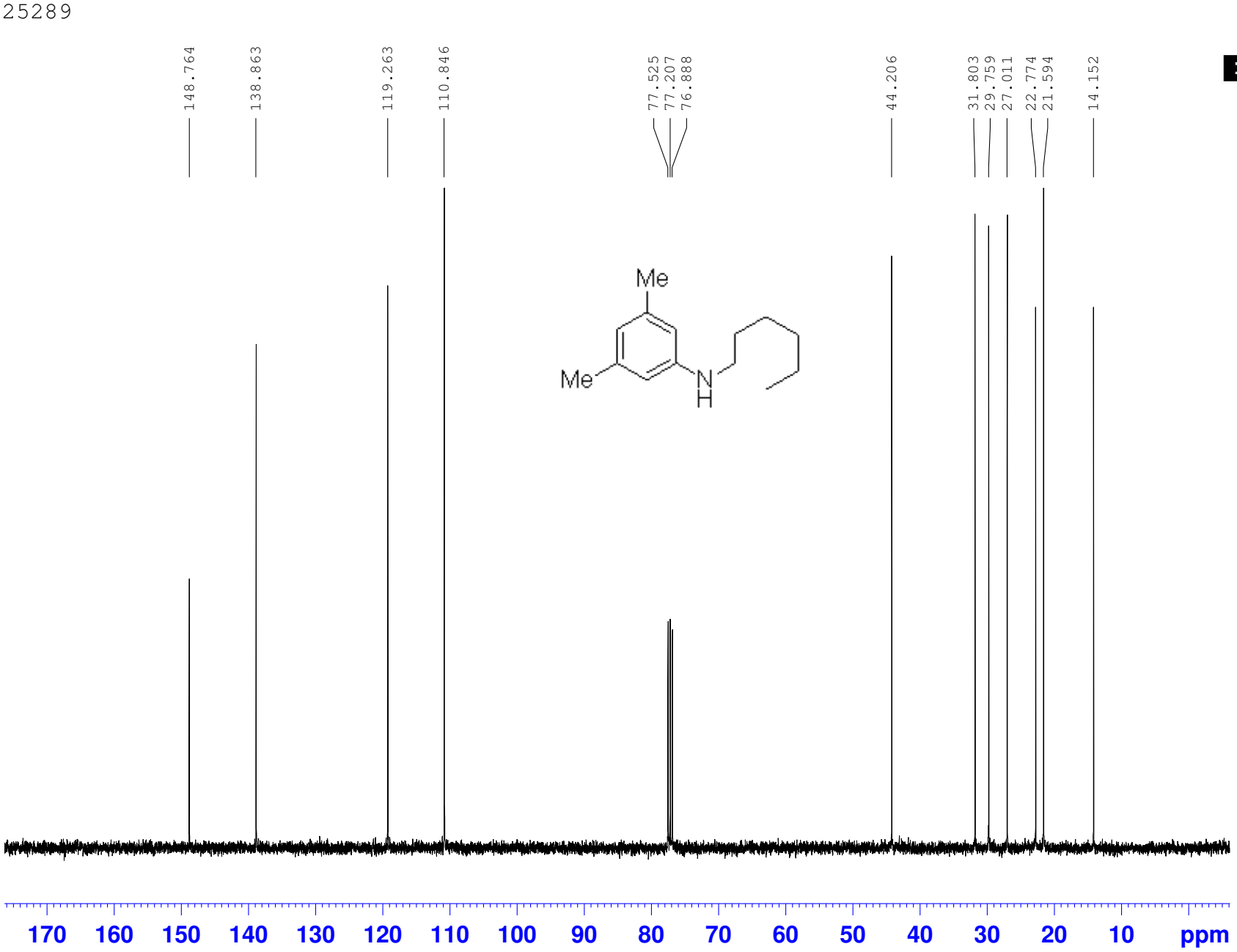
Current Data Parameters
NAME wdp
EXPNO 284
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080906
Time 16.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 18
DW 60.400 usec
DE 6.00 usec
TE 302.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125289



BRUKER

```
Current Data Parameters
NAME                  wdp
EXPNO                 285
PROCNO                  1

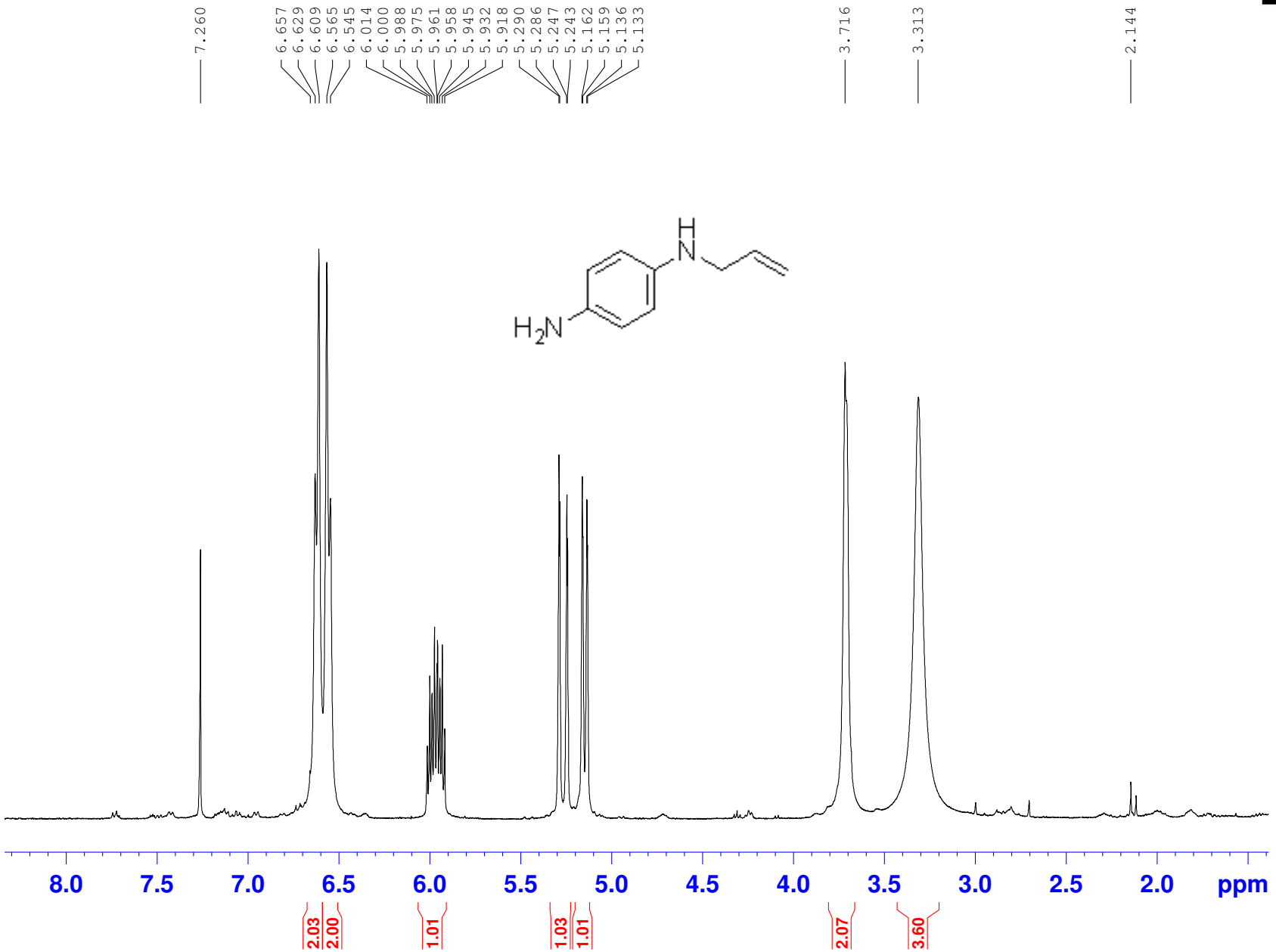
F2 - Acquisition Parameters
Date_           20080906
Time               17.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   44
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                 1024
DW               20.850 usec
DE                6.00 usec
TE                303.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

125290



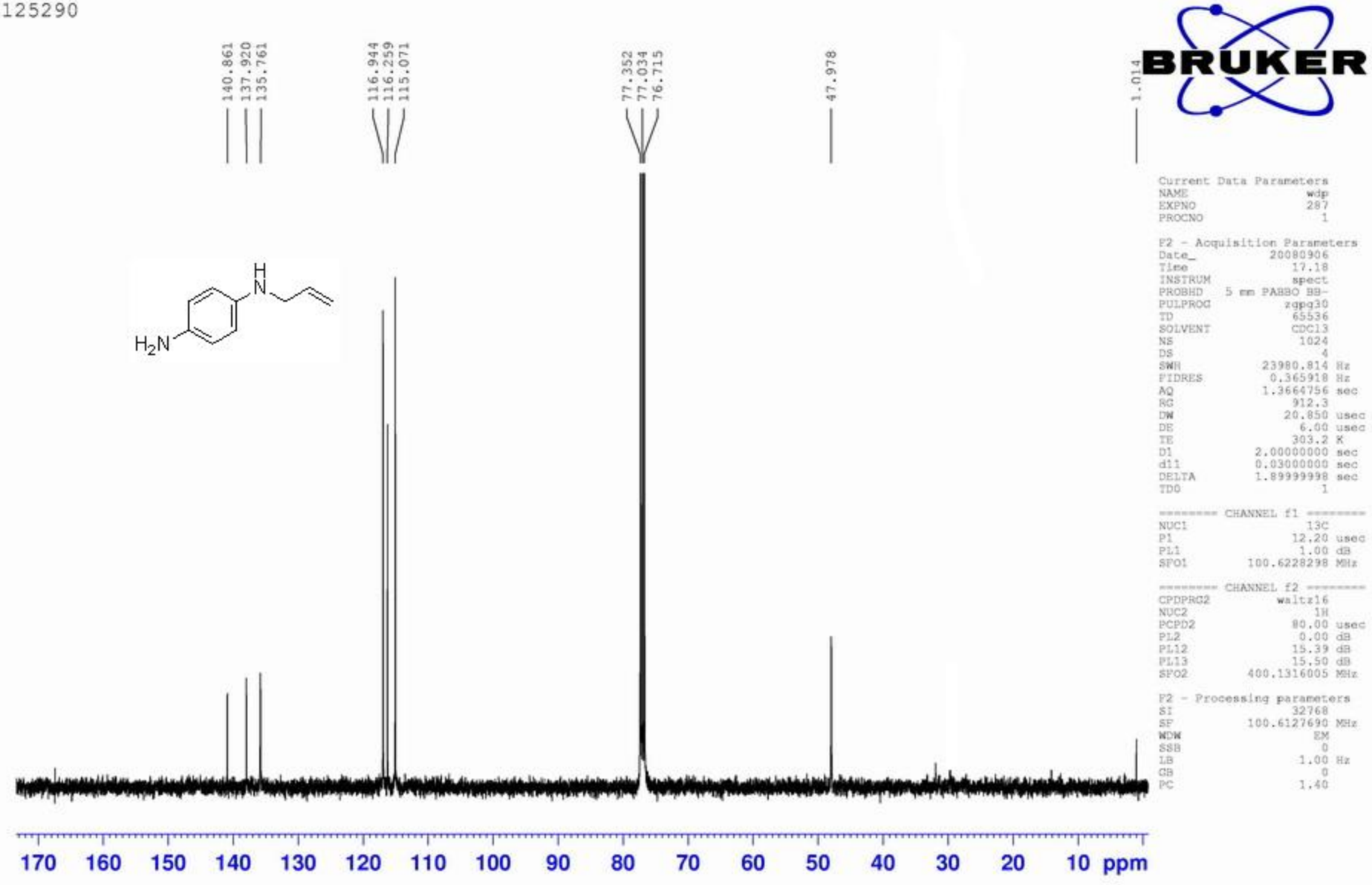

```
Current Data Parameters
NAME                  wdp
EXPNO                 286
PROCNO                  1

F2 - Acquisition Parameters
Date_           20080906
Time               18.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   181
DW                60.400 usec
DE                 6.00 usec
TE                301.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.60 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300085 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

125290



Current Data Parameters

NAME	wdp
EXPNO	287
PROCNO	1

F2 - Acquisition Parameters

Date_	20080906
Time	17.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	912.3
DW	20.850 usec
DE	6.00 usec
TE	303.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

NUC1	13C
P1	12.20 usec
PL1	1.00 dB
SFO1	100.6228298 MHz

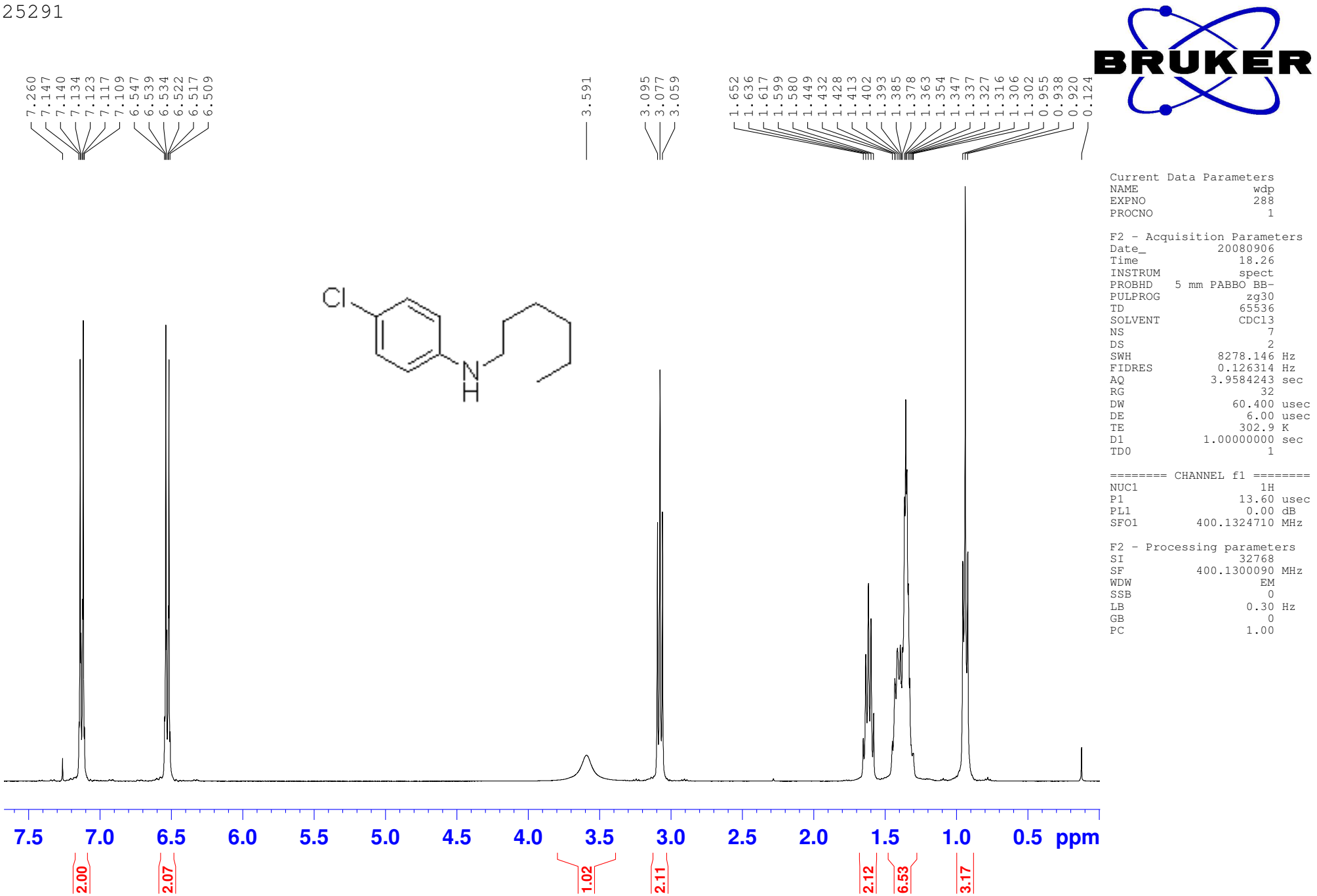
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.39 dB
PL13	15.50 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

125291



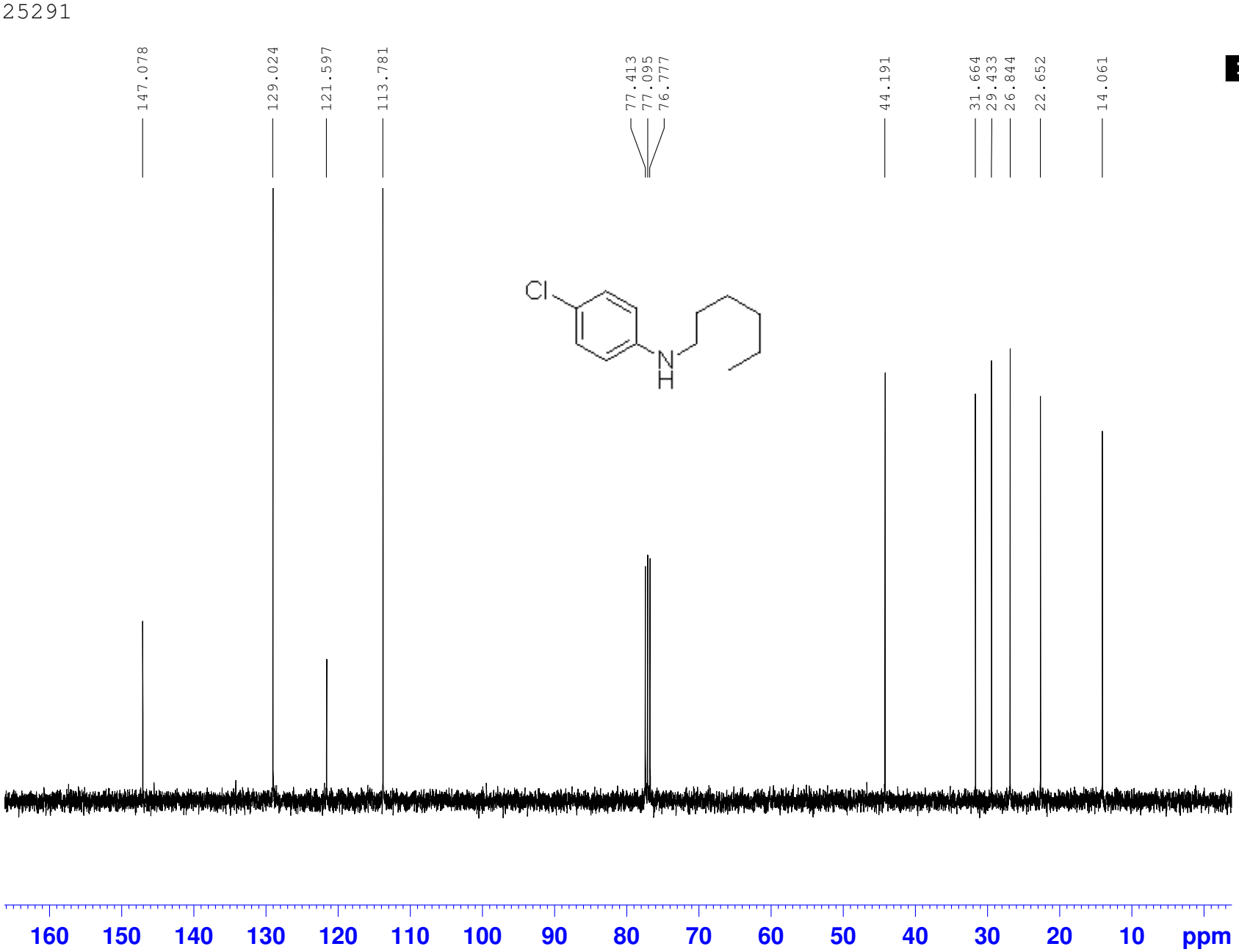
Current Data Parameters
NAME wdp
EXPNO 288
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080906
Time 18.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 32
DW 60.400 usec
DE 6.00 usec
TE 302.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125291

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Current Data Parameters
NAME                  wdp
EXPNO                 289
PROCNO                  1

F2 - Acquisition Parameters
Date_           20080906
Time               18.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   18
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                912.3
DW               20.850 usec
DE                6.00 usec
TE                302.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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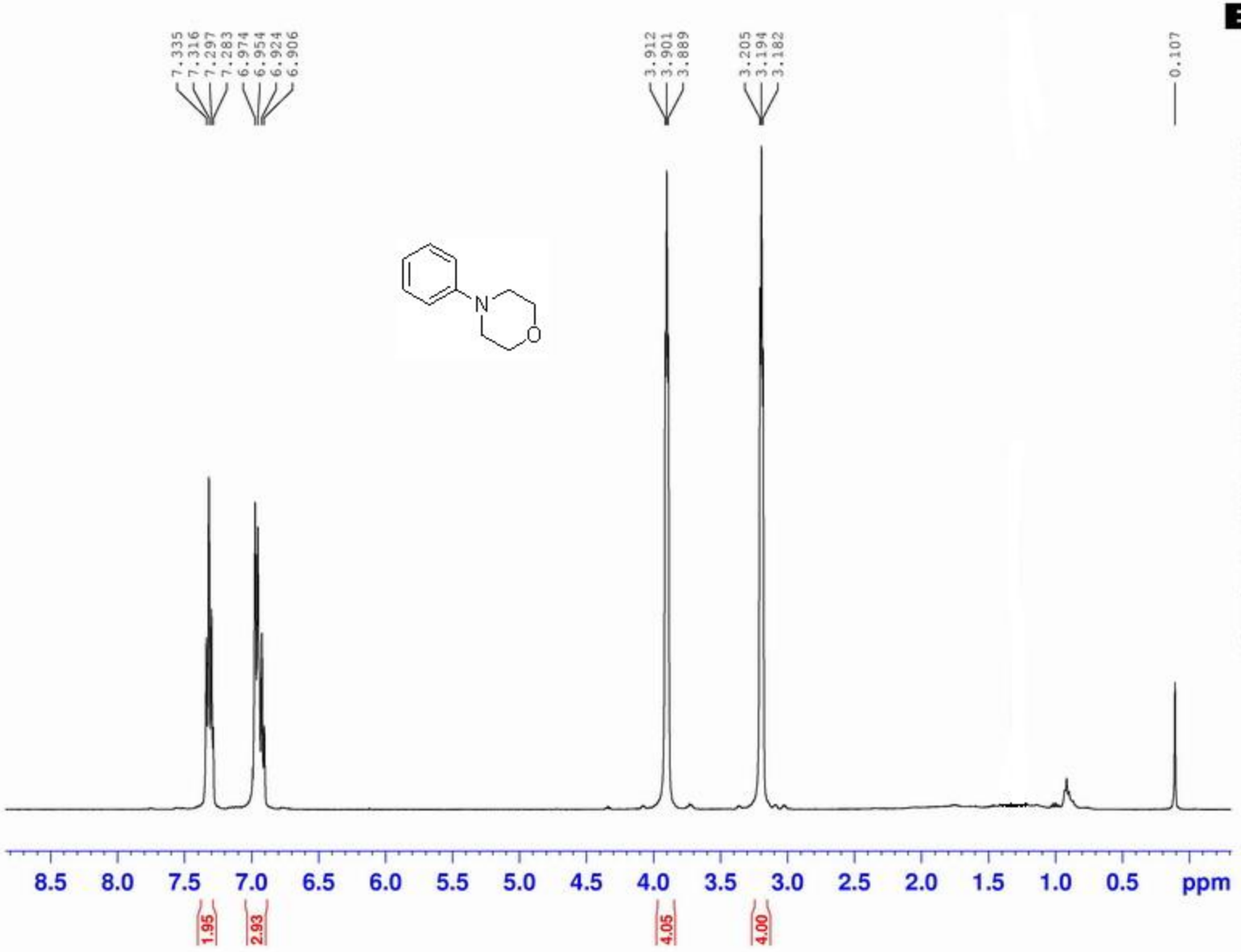
125292



BRUKER

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Current Data Parameters
NAME                  wdp
EXPNO                 290
PROCNO                1

F2 - Acquisition Parameters
Date_          20080906
Time               18.52
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                143.7
DW               60.400 usec
DE                6.00 usec
TE               301.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.60 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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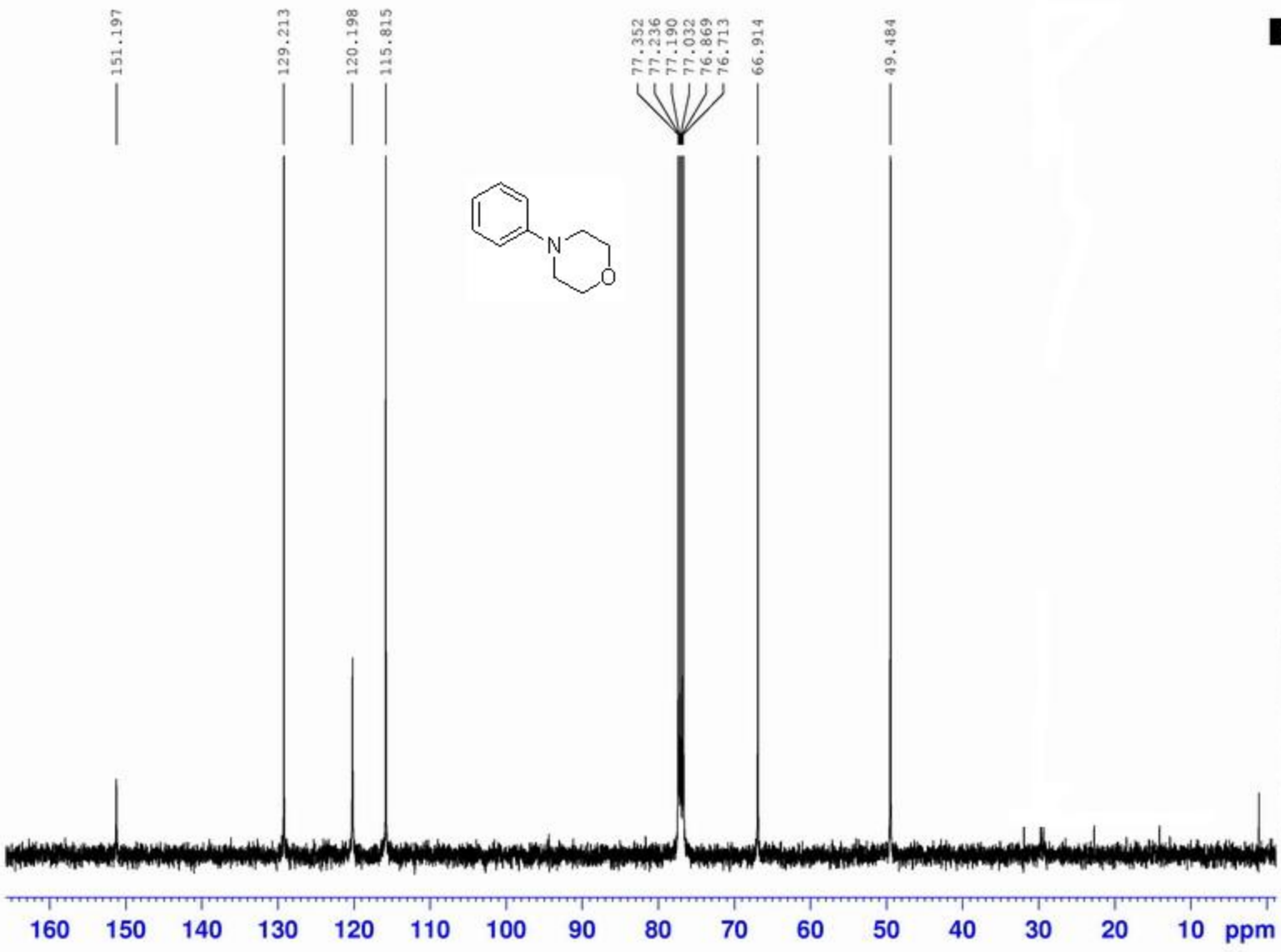
125292



Current Data Parameters

NAME	wdp
EXPNO	291
PROCNO	1

F2 - Acquisition Parameters

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Time	18.59
INSTRUM	spect
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PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	1024
DW	20.850 usec
DE	6.00 usec
TE	301.7 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

NUC1	13C
P1	12.20 usec
PL1	1.00 dB
SFO1	100.6228298 MHz

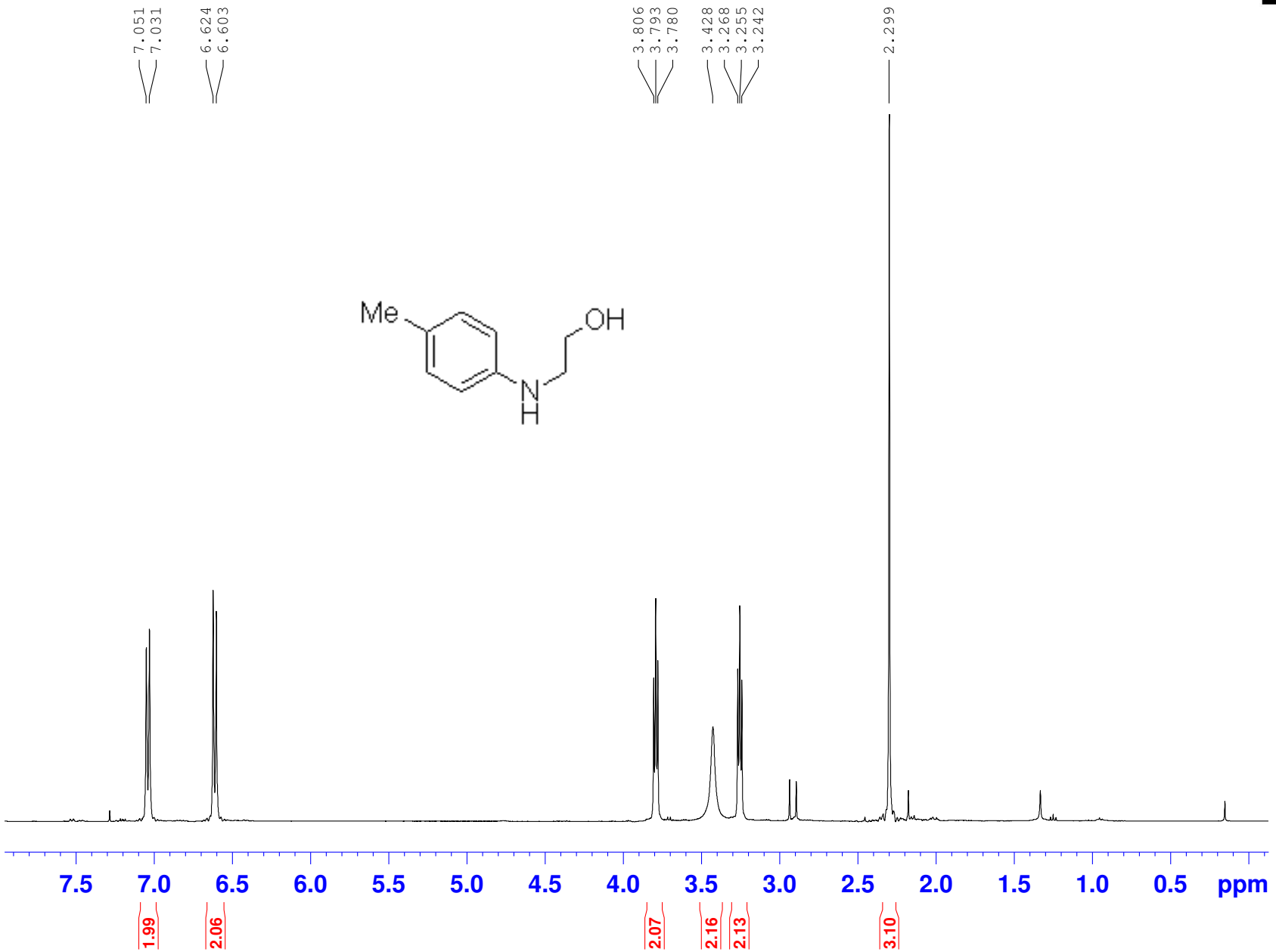
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	15.39 dB
PL13	15.50 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

125293



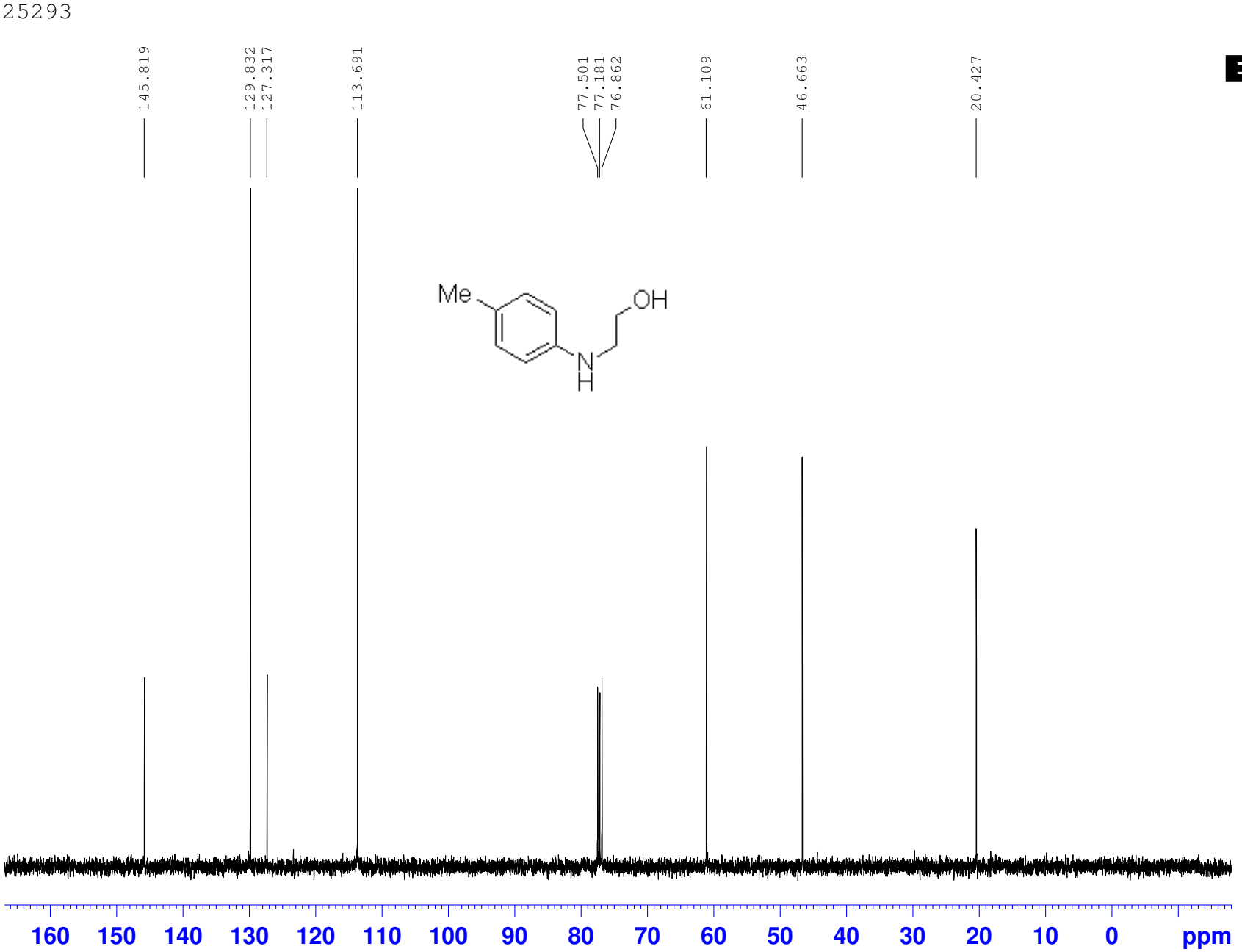
Current Data Parameters
NAME wdp
EXPNO 293
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080906
Time 21.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 32
DW 60.400 usec
DE 6.00 usec
TE 302.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125293

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Current Data Parameters
NAME                  wdp
EXPNO                 292
PROCNO                  1

F2 - Acquisition Parameters
Date_           20080906
Time               21.12
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                812.7
DW               20.850 usec
DE                6.00 usec
TE                303.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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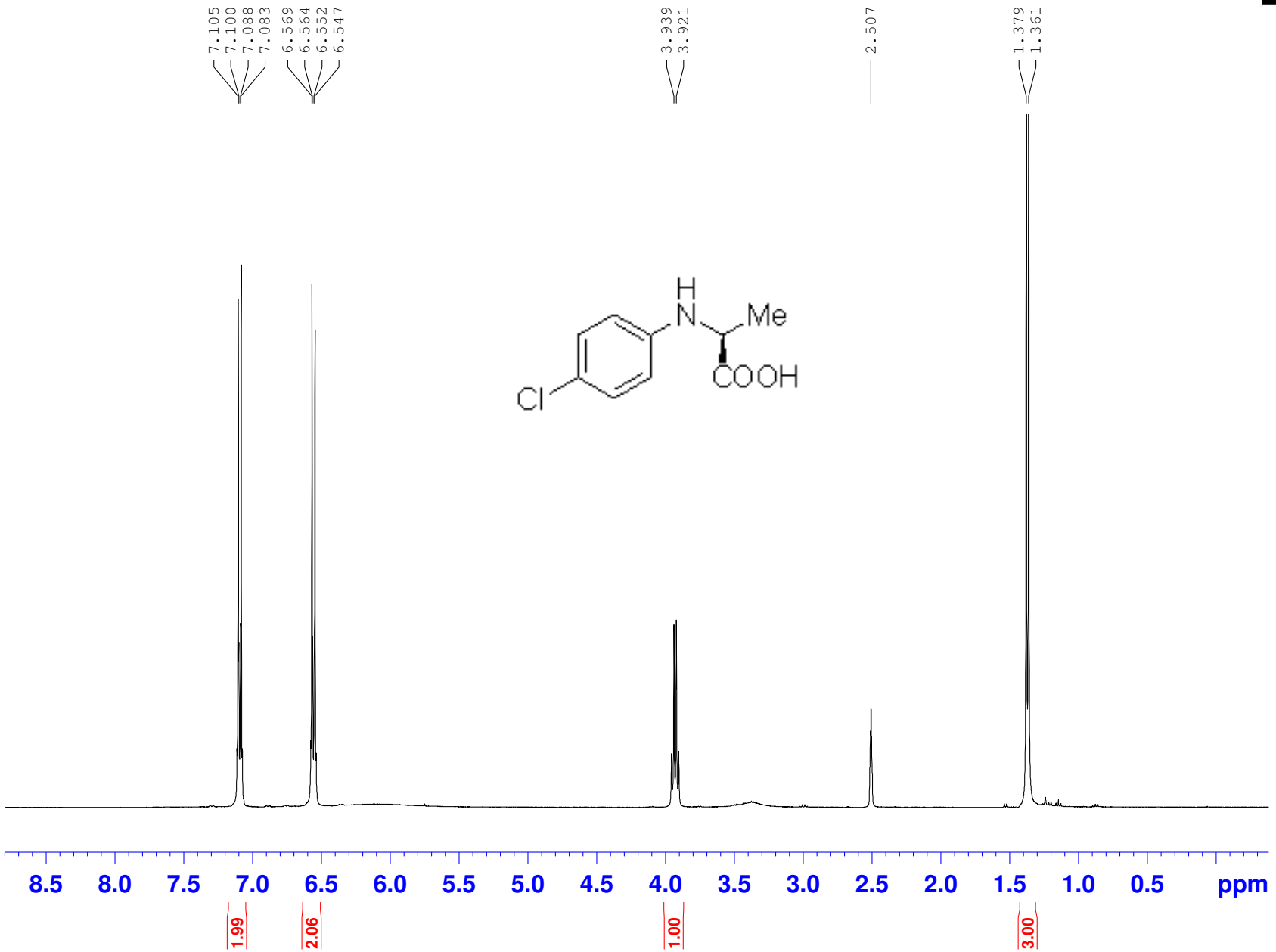
125318



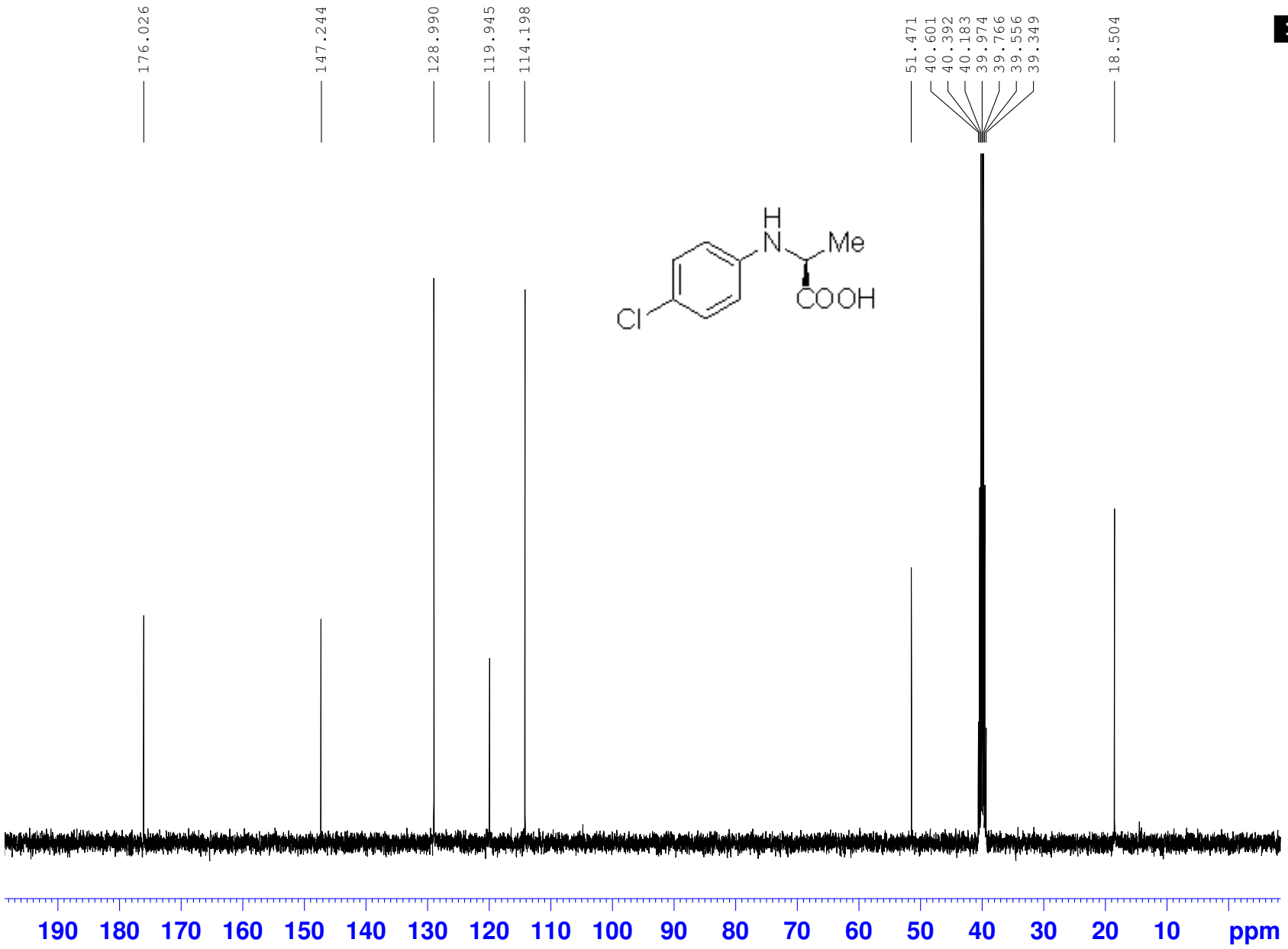
Current Data Parameters
NAME wdp
EXPNO 336
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081012
Time 11.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 10
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 8.00 usec
TE 299.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125318

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Current Data Parameters
NAME                   wdp
EXPNO                  337
PROCNO                   1

F2 - Acquisition Parameters
Date_           20081012
Time                11.31
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                     74
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG               23170.5
DW                20.850 usec
DE                 8.00 usec
TE                300.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                 1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL12               15.39 dB
PL13               15.50 dB
PL2                 0.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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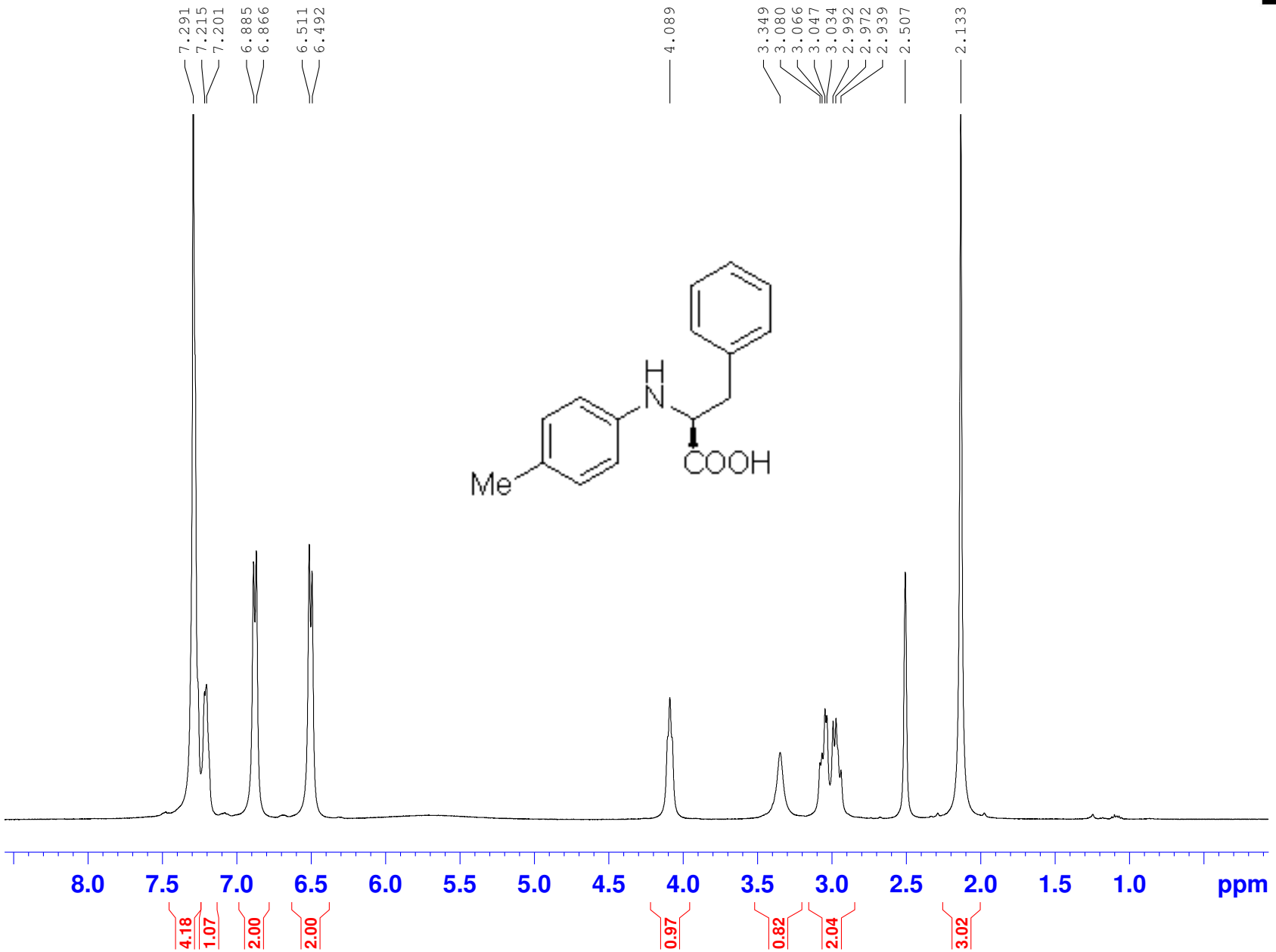
125322



Current Data Parameters
NAME wdp
EXPNO 338
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081012
Time 11.52
INSTRUM spect
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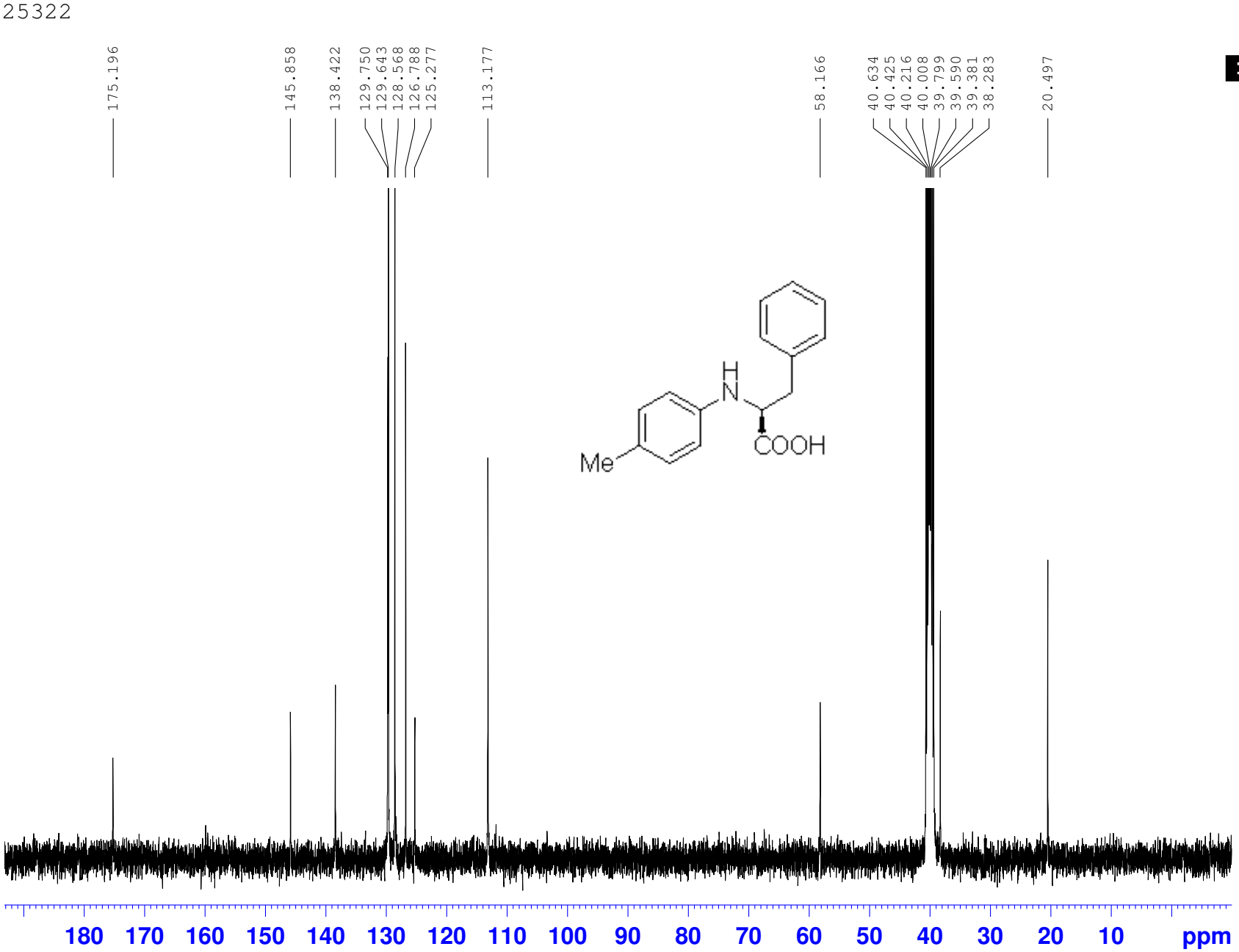
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 15
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 8.00 usec
TE 299.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125322

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Current Data Parameters
NAME                  wdp
EXPNO                 339
PROCNO                  1

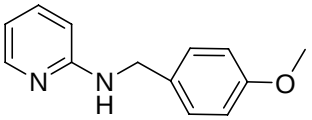
F2 - Acquisition Parameters
Date_           20081012
Time               11.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                    537
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                  26008
DW                 20.850 usec
DE                  8.00 usec
TE                 300.4 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                 1.00 dB
SFO1         100.6228298 MHz

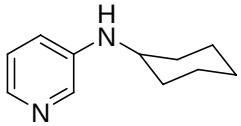
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL12               15.39 dB
PL13               15.50 dB
PL2                 0.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                  1.00 Hz
GB                      0
PC                  1.40
```

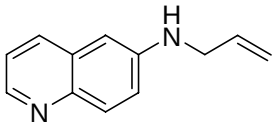
Room Temperature Amination of Heteroaryl Iodides: Table 3.



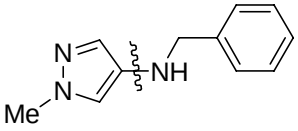
2-(*p*-methoxybenzylamino)pyridine (6a). Following Procedure A, 2-iodopyridine (205 mg, 1.0 mmol) was allowed to react with 4-methoxybenzylamine (206 mg, 1.5 mmol) for 17 h. The crude brown oil was purified by flash chromatography on silica gel eluent (6:1 petroleum ether: ethyl acetate) to provide 96 % yield of the desired product as a whit solid. ^{1}H NMR($CDCl_3$): δ=8.09 (d, *J*=5.2 Hz, 1 H; Phenyl), 7.39 (td, *J*=6.8, 2.0 Hz, 2 H; Phenyl), 7.29 (d, *J*=8.8 Hz, 2 H; Phenyl), 6.88 (d, *J*=8.4 Hz, 2 H; Phenyl), 6.57 (m, 1 H; Phenyl), 6.37 (d, *J*=8.4 Hz, 1 H; Phenyl), 5.06 (br s, 1 H; NH), 4.43 (d, *J*=5.6 Hz, 2 H; CH_2), 3.81 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=158.9, 158.7, 148.1, 137.5, 131.2, 128.7, 114.1, 113.0, 106.8 (Phenyl), 55.3 (CH_2), 45.8 ppm (CH_3); GC/MS: rt = 9.06 min, M/Z = 214.



3-(cyclohexylamino)pyridine (6b). Following Procedure A, 3-iodopyridine (205 mg, 1.0 mmol) was allowed to react with cyclohexanamine (149 mg, 1.5 mmol) for 24 h. The crude brown oil was purified by flash chromatography on silica gel eluent (5:1 petroleum ether: ethyl acetate) to provide 88 % yield of the desired product as a whit solid. ^{1}H NMR($CDCl_3$): δ=8.01 (br s, 1 H; Phenyl), 7.91 (br s, 1 H; Phenyl), 7.05 (br s, 1 H; Phenyl), 6.84 (d, *J*=7.6 Hz, 1 H; Phenyl), 3.70 (br s, 1 H; NH), 3.28-3.21 (m, 1 H; -N-CH-), 2.05-2.02 (m, 2 H; CH_2), 1.68-1.63 (m, 1 H; CH_2), 1.43-1.26 ppm (m, 5 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=143.5, 137.9, 136.1, 123.8, 118.8 (Phenyl), 51.4 (-N-CH-), 33.2, 25.8, 24.9 ppm (CH_2); GC/MS: rt = 7.85 min, M/Z = 176.

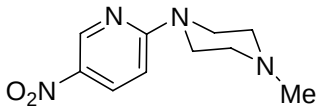


6-(allylamino)-quinoline (6f). Following Procedure A, 6-iodoquinoline (255 mg, 1.0 mmol) was allowed to react with allylamine (115 mg, 2 mmol) for 19 h. The crude brown oil was purified by flash chromatography on silica gel eluent (5:1 petroleum ether: ethyl acetate) to provide 87 % yield of the desired product as a brown oil. ^{1}H NMR($CDCl_3$): δ=8.61 (d, *J*=4.0 Hz, 1 H; Phenyl), 7.91 (m, 2 H; Phenyl), 7.29-7.26 (m, 1 H; Phenyl), 7.12 (m, 1 H; Phenyl), 6.72 (d, *J*=2.0 Hz, 1 H; Phenyl), 6.05-5.98 (m, 1 H; -CH_2-C*H*=), 5.33 (m, 1 H; -CH=C*H*H), 5.22 (m, 1 H; -CH=CH*H*), 4.22 (br s, 1 H; NH), 3.89 ppm (d, *J*=4.4 Hz, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=167.5, 146.1, 145.8, 134.6, 134.2, 130.2, 129.9, 121.6, 121.3 (Phenyl), 116.7 (-C*H*=), 103.3 (-CH=*C*HH), 46.4 ppm (CH_2); GC/MS: rt = 8.54 min, M/Z = 184.

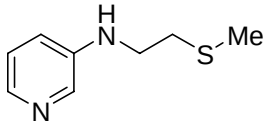


4-(N-benzyl)-1-methyl-1H- pyrazolamine (6g). Following Procedure A, 4-iodo-1-methyl-1H-pyrazole (208 mg, 1.0 mmol) was allowed to react with benzylamine (161 mg, 1.5 mmol) for 24 h. The crude brown oil was purified by flash chromatography on silica gel eluent (4:1 petroleum ether: ethyl acetate) to provide 83 % yield of the desired product as a brown oil. ^{1}H NMR($CDCl_3$): δ=7.39-7.28 (m, 5 H; Phenyl), 7.14 (s, 1 H; Phenyl), 6.86 (s, 1 H; Phenyl), 4.16 (s, 2

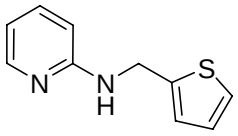
H; CH_2), 3.78 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=139.6, 134.1, 128.7, 128.5, 127.7, 127.2, 116.7 (Phenyl), 52.2 (CH_2), 39.1 ppm (CH_3); GC/MS: rt = 8.08 min, M/Z = 187.



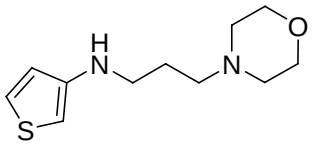
2-(N-Methylpiperazino)-5-nitropyridine (6c). Following Procedure A, 2-iodo-5-nitropyridine (250 mg, 1.0 mmol) was allowed to react with 1-methylpiperazine (150 mg, 1.5 mmol) for 11 h. The crude brown oil was purified by flash chromatography on silica gel eluent (1:2 petroleum ether: ethyl acetate) to provide 82 % yield of the desired product as a yellow solid. ^1H NMR($CDCl_3$): δ=9.03 (t, *J*=2.4 Hz, 1 H; Phenyl), 8.19 (m, 1 H; Phenyl), 6.57 (dd, *J*=9.6 0.8 Hz, 1 H; Phenyl), 3.83 (d, *J*=4.4 Hz, 4 H; Aryl-N-CH_2-), 2.57 (d, *J*=4.0 Hz, 4 H; CH_3-N-C*H*$_2$-), 2.39 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=160.3, 146.4, 135.1, 133.0, 104.6 (Phenyl), 54.5 (Aryl-N-CH_2-), 45.9 (CH_3-N-C*H*$_2$-), 44.7 ppm (CH_3); GC/MS: rt = 9.63 min, M/Z = 222.



3-[N-(2-(methylthio)ethyl)]-pyridineamine (6d). Following Procedure A, 3-iodopyridine (205 mg, 1.0 mmol) was allowed to react with 2-(methylthio)ethanamine (138 mg, 1.5 mmol) for 20 h. The crude brown oil was purified by flash chromatography on silica gel eluent (5:1 petroleum ether: ethyl acetate) to provide 95 % yield of the desired product as a yellow oil. ^1H NMR($CDCl_3$): δ=8.04 (s, 1 H; Phenyl), 7.94 (d, *J*=3.6 Hz, 1 H; Phenyl), 7.08-7.05 (m, 1 H; Phenyl), 6.87 (d, *J*=8.0 Hz, 1 H; Phenyl), 4.28 (br s, 1 H; NH), 3.29 (t, *J*=6.4 Hz, 2 H; -N-CH_2-), 2.73 (t, *J*=6.4 Hz, 2 H; -S-CH_2-), 2.09 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=143.9, 138.8, 135.9, 123.8, 118.9 (Phenyl), 41.5 (-N-CH_2-), 33.4 (-S-CH_2-), 15.0 ppm (CH_3); GC/MS: rt = 7.72 min, M/Z = 168; HRMS (EI): calcd for $C_8H_{12}N_2S$: 168.0716 [M+]; found: 168.0719.



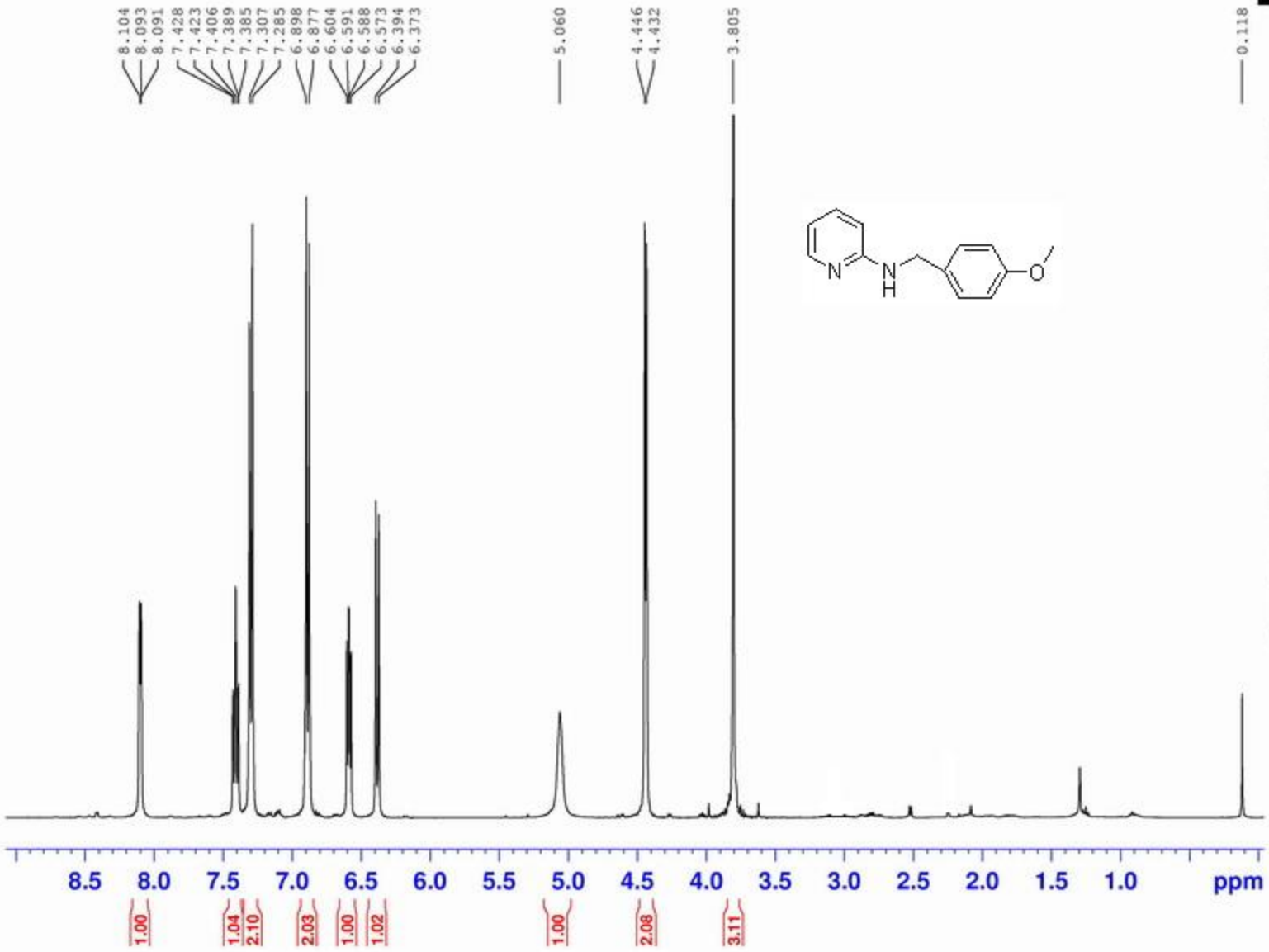
2-[N-(2-thiophenmethyl)]-pyridinamine (6e). Following Procedure A, 2-iodopyridine (205 mg, 1.0 mmol) was allowed to react with thiophen-2-ylmethanamine (170 mg, 1.5 mmol) for 14 h. The crude brown oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 97 % yield of the desired product as a white solid. ^1H NMR($CDCl_3$): δ=8.13 (m, 1 H; Phenyl), 7.46-7.41 (m, 1 H; Phenyl), 7.21 (dd, *J*=5.2 1.2 Hz, 1 H; Phenyl), 7.02 (m, 1 H; Phenyl), 6.96 (m, 1 H; Phenyl), 6.61 (td, *J*=6.0, 0.4 Hz, 1 H; Phenyl), 6.44 (d, *J*=8.0 Hz, 1 H; Phenyl), 5.08 (br s, 1 H; NH), 4.71 ppm (d, *J*=6.0 Hz, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=158.1, 147.9, 142.6, 137.5, 126.8, 125.2, 124.6, 113.5, 107.4 (Phenyl), 41.3 ppm (CH_2); GC/MS: rt = 8.01 min, M/Z = 190.



3-[N-(3-morpholinopropyl)]thiophenamine (6h). Following Procedure A, 3-iodothiophene (210 mg, 1.0 mmol) was allowed to react with 3-morpholinopropan-1-amine (216 mg, 1.5 mmol) for 14 h. The crude brown oil was purified by flash chromatography on silica gel eluent (40:1 CH_2Cl_2: MeOH) to provide 90 % yield of the desired product as a

brown oil. ^{1}H NMR($CDCl_3$): δ=7.15 (dd, *J*=4.8 2.8 Hz, 1 H; Phenyl), 6.62 (dd, *J*=5.2 1.6 Hz, 1 H; Phenyl), 5.95 (m, 1 H; Phenyl), 3.73 (m, 4 H; -O-CH_2-), 3.16 (t, *J*=6.4 Hz, 2 H; -NH-C*H*$_2$-), 2.51 (m, 6 H; -N-CH_2-), 1.87-1.81 ppm (m, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=148.9, 125.1, 119.9, 95.2 (Phenyl), 66.9 (-O-CH_2-), 57.6 (-NH-CH_2-), 53.7 (-N-CH_2-), 45.5 (-N-CH_2-), 25.5 ppm (CH_2); GC/MS: rt = 8.79 min, M/Z = 226; HRMS (EI): calcd for $C_{11}H_{18}ON_2S$: 226.1134 [M+]; found: 226.1133.

125294



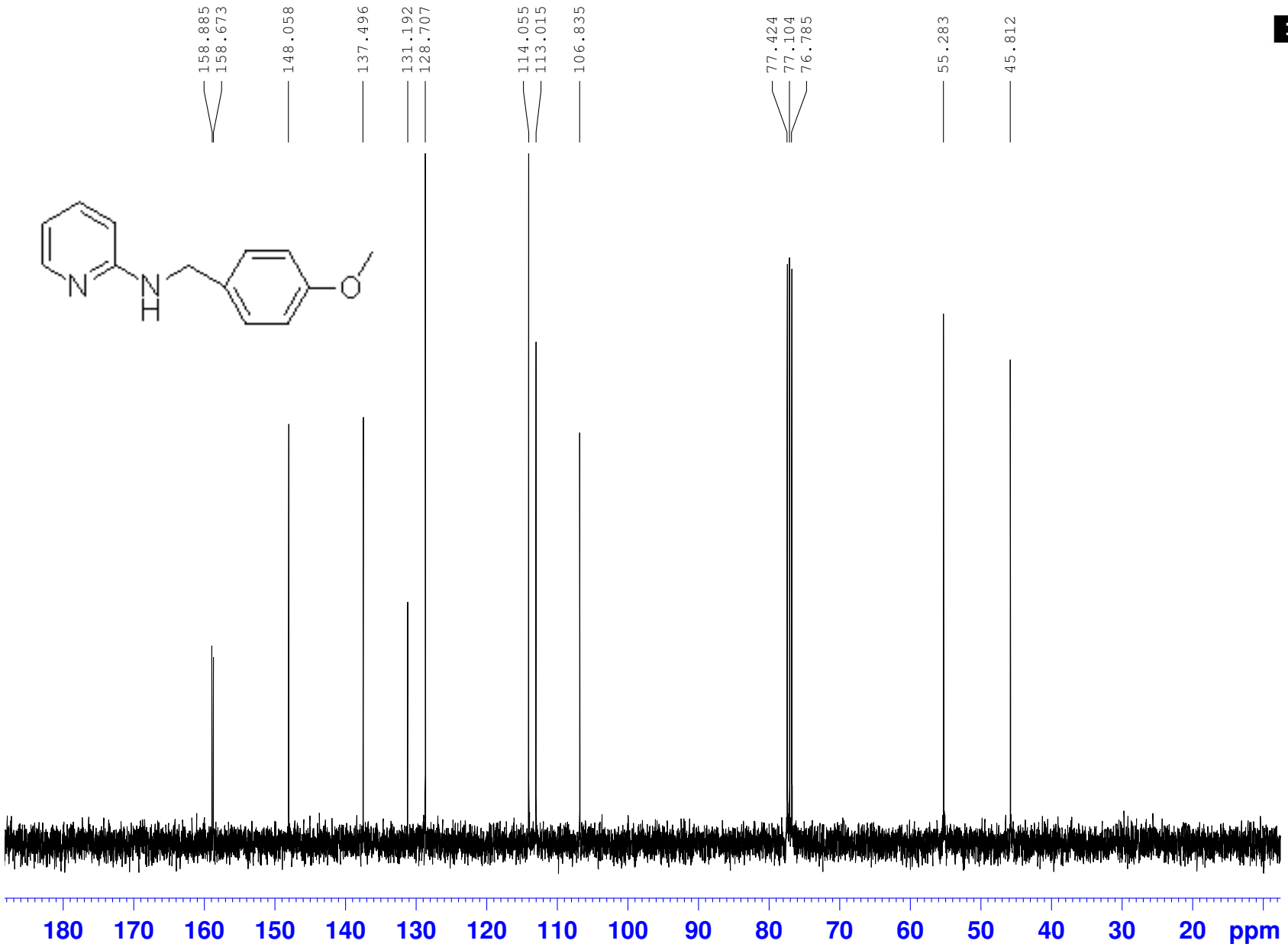
Current Data Parameters
NAME wdp
EXPNO 294
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080906
Time 21.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 302.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125294



Current Data Parameters
NAME wdp
EXPNO 295
PROCNO 1

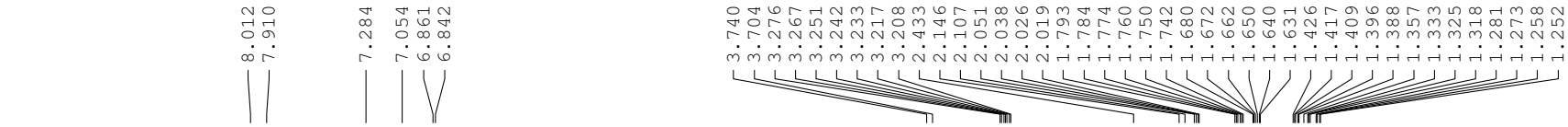
F2 - Acquisition Parameters
Date_ 20080906
Time 21.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 812.7
DW 20.850 usec
DE 6.00 usec
TE 302.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

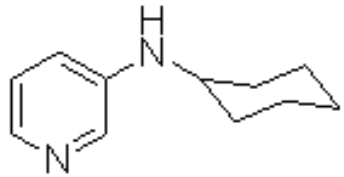
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

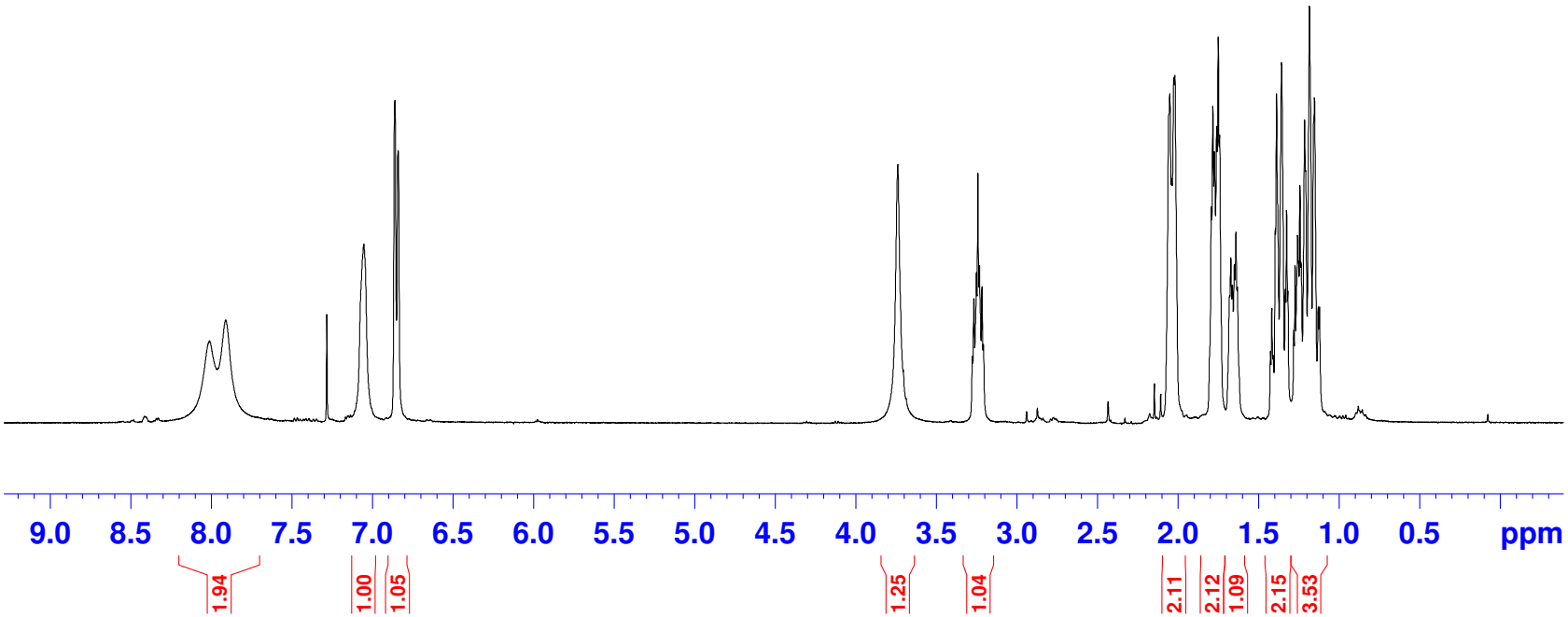
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125295







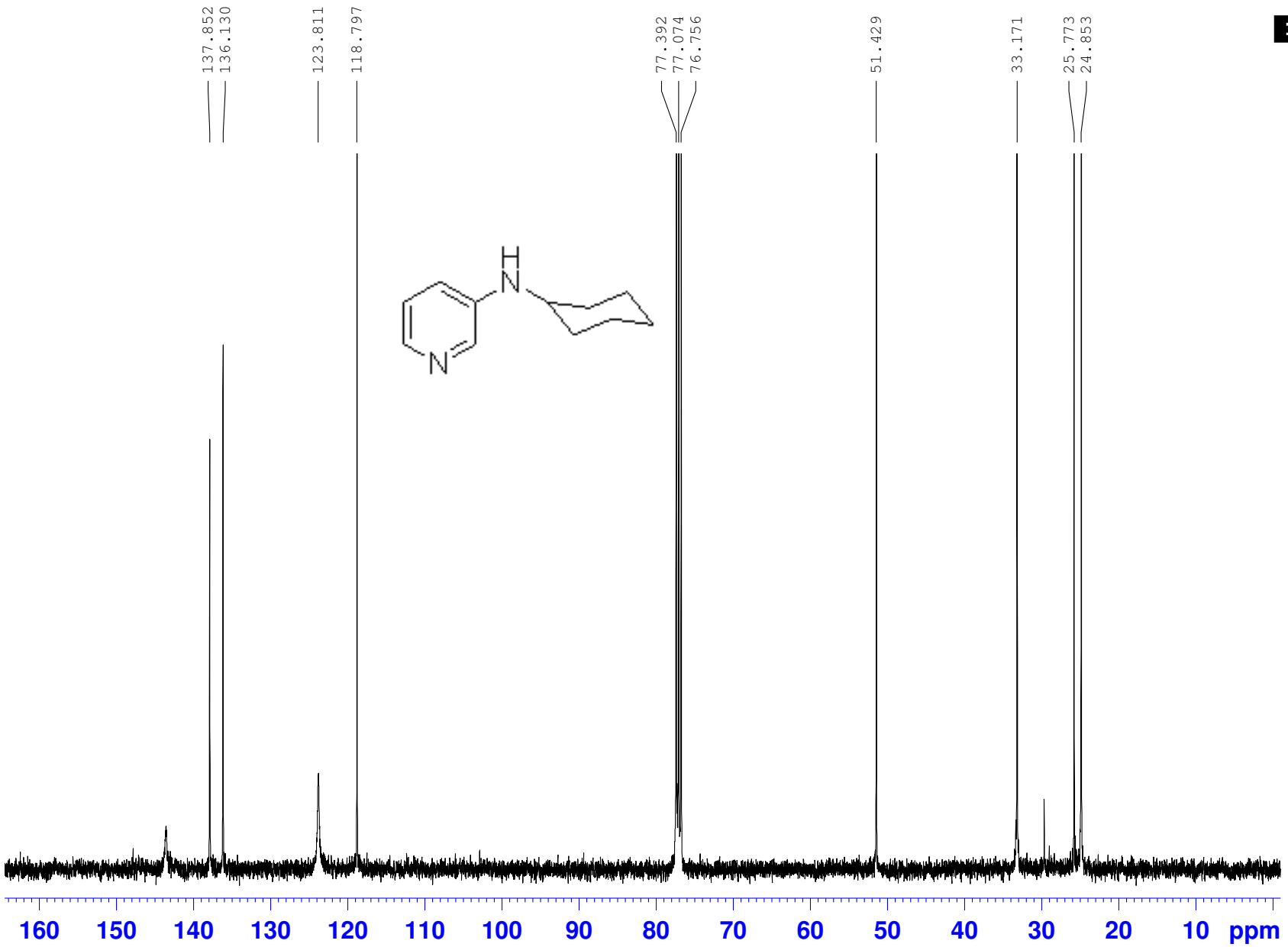
Current Data Parameters
NAME wdp
EXPNO 307
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080927
Time 18.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 301.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125295



BRUKER

Current Data Parameters
NAME wdp
EXPNO 306
PROCNO 1

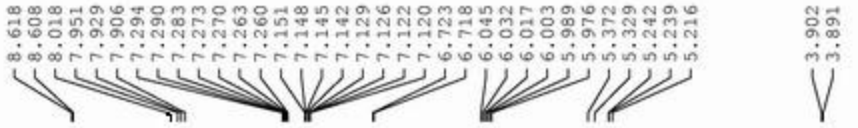
F2 - Acquisition Parameters
Date_ 20080927
Time 17.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 912.3
DW 20.850 usec
DE 6.00 usec
TE 302.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

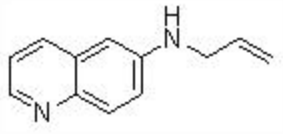
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

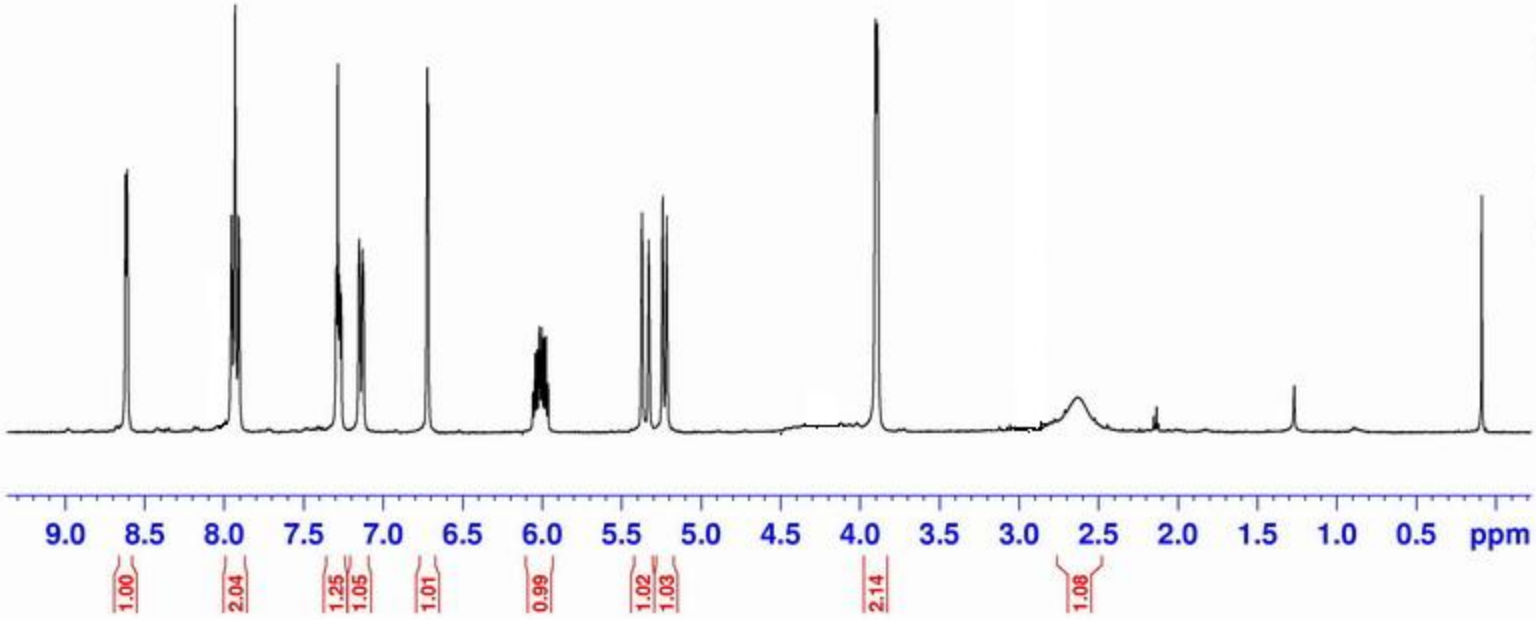
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125298







Current Data Parameters

NAME	wdp
EXPNO	311
PROCNO	1

F2 - Acquisition Parameters

Date_	20080927
Time	20.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	6
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	161.3
DW	60.400 usec
DE	6.00 usec
TE	301.3 K
D1	1.00000000 sec
TD0	1

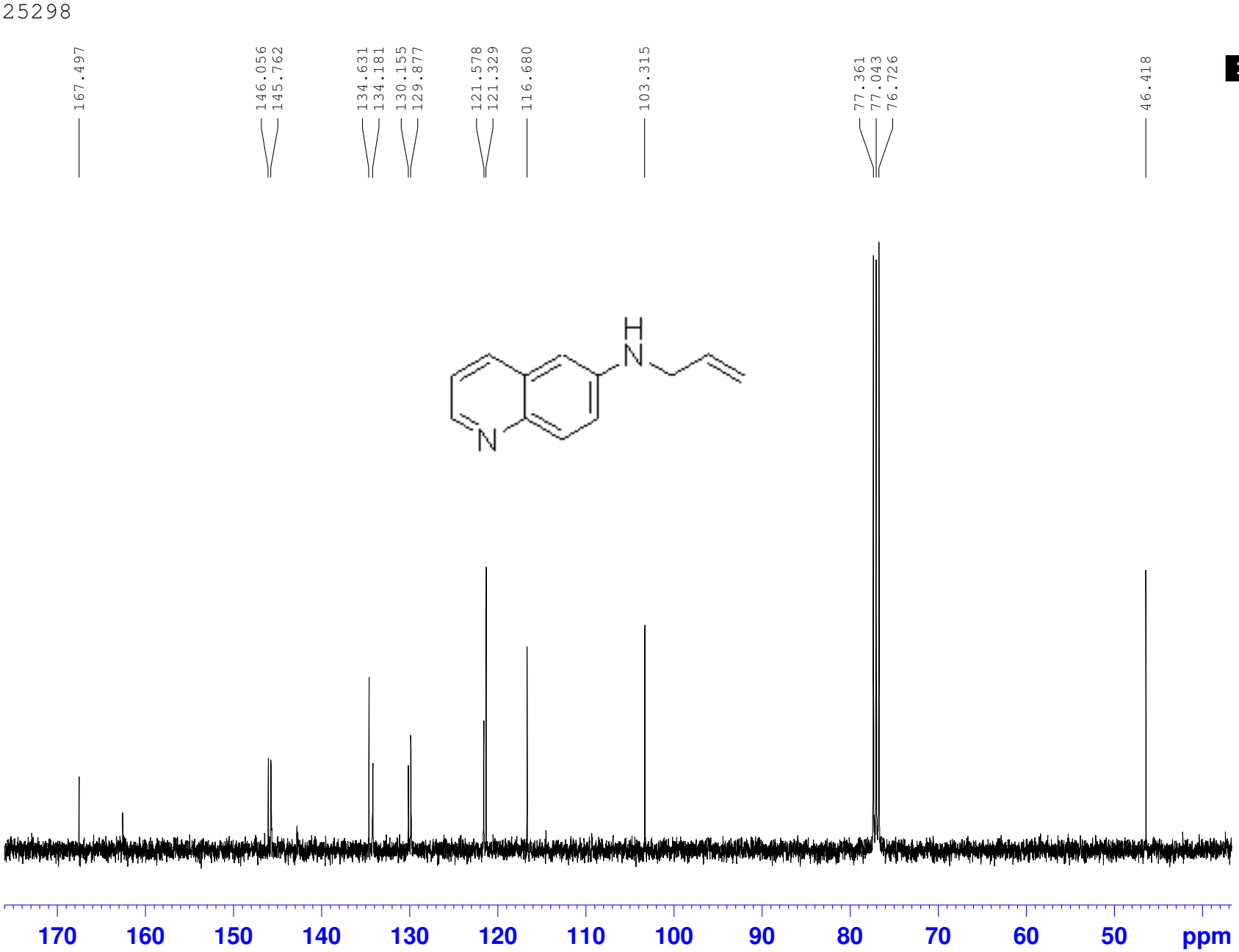
======== CHANNEL f1 ========

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

125298




```
Current Data Parameters
NAME                  wdp
EXPNO                 312
PROCNO                  1

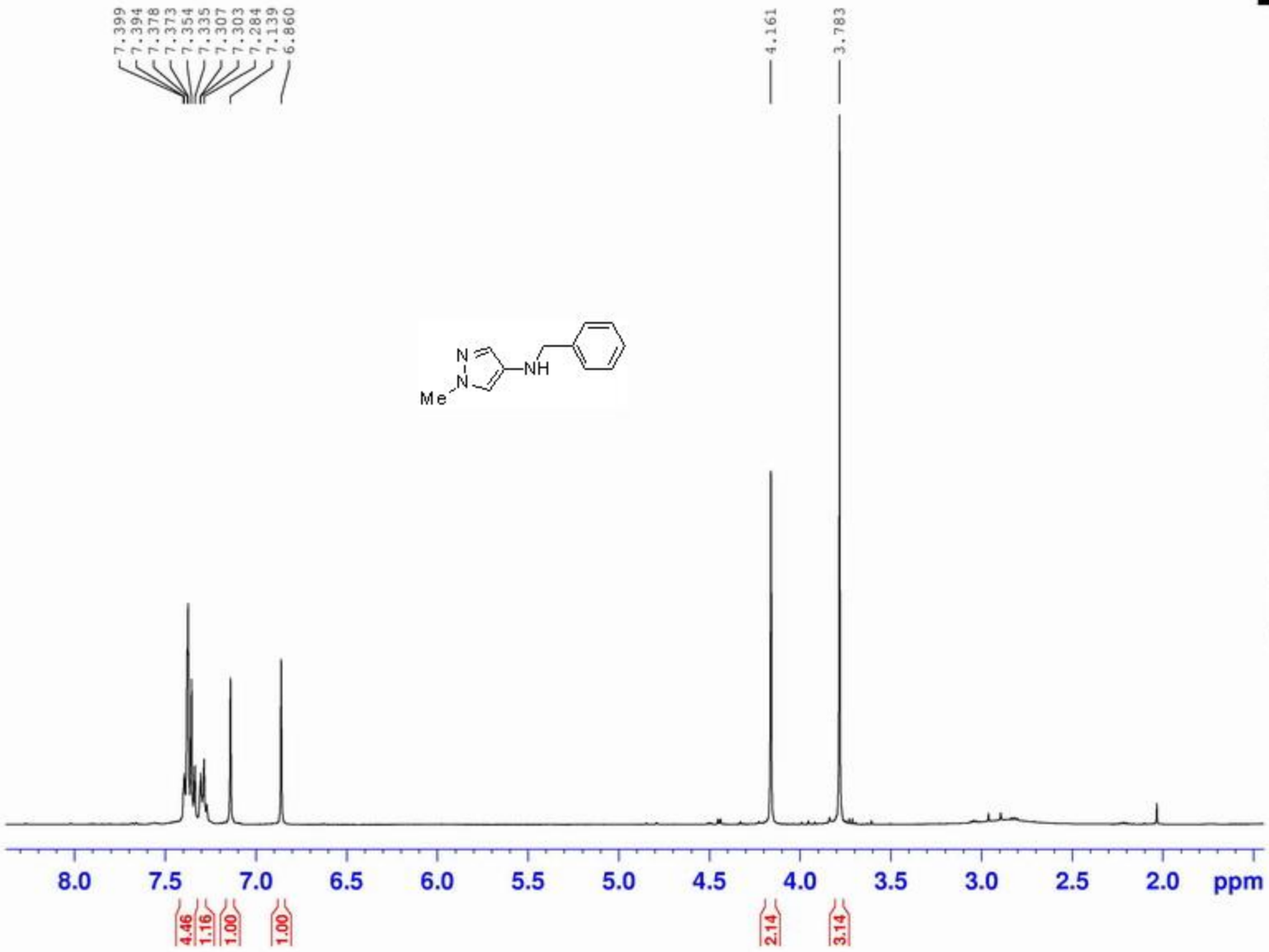
F2 - Acquisition Parameters
Date_           20080927
Time               20.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  157
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               1290.2
DW               20.850 usec
DE                6.00 usec
TE               301.9 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

125299



BRUKER

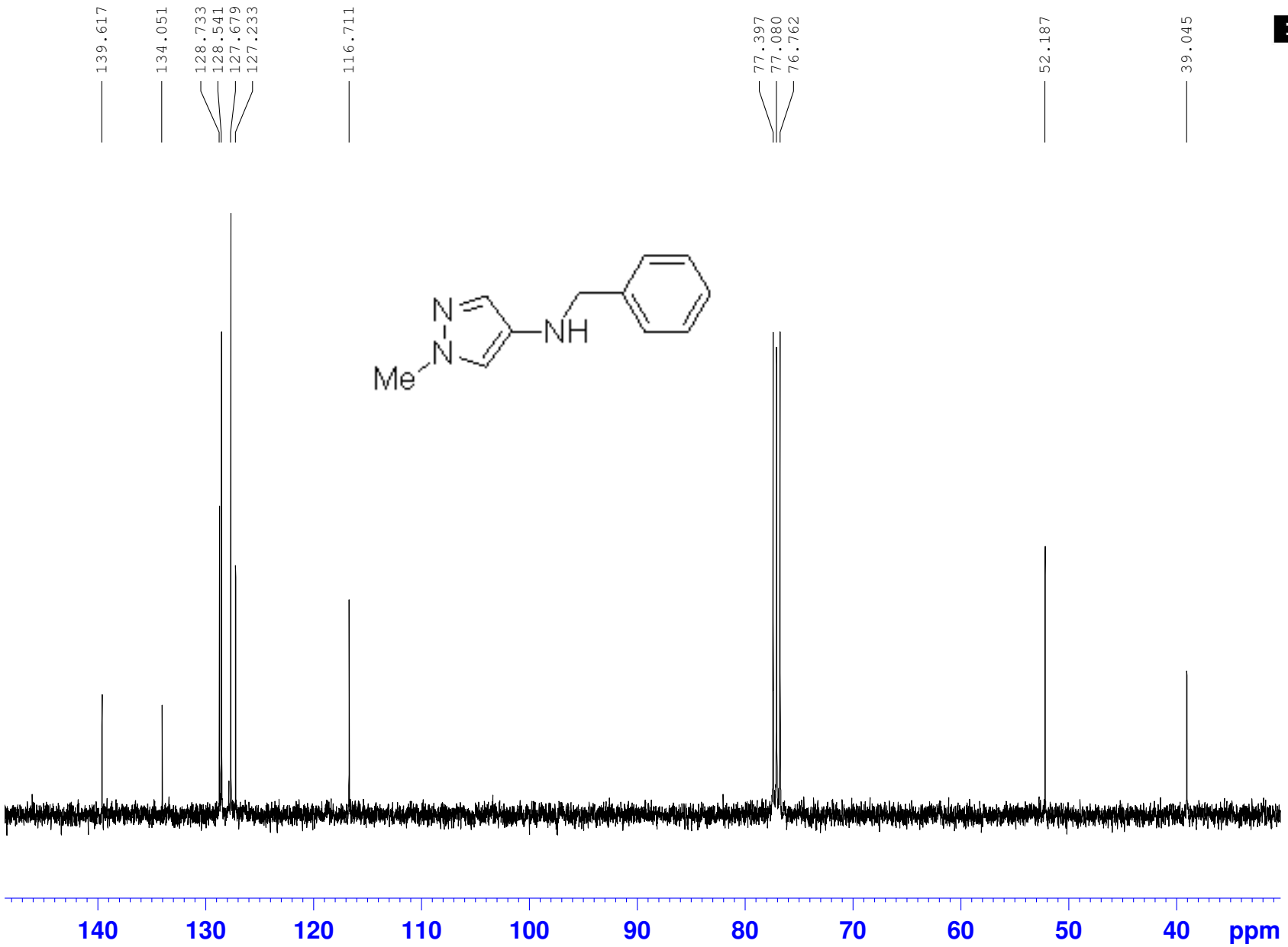
Current Data Parameters
NAME wdp
EXPNO 303
PROCNO 1

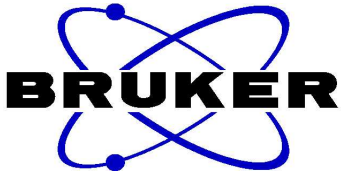
F2 - Acquisition Parameters
Date_ 20080927
Time 16.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 80.6
DW 60.400 usec
DE 6.00 usec
TE 301.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125299






```
Current Data Parameters
NAME                   wdp
EXPNO                  304
PROCNO                   1

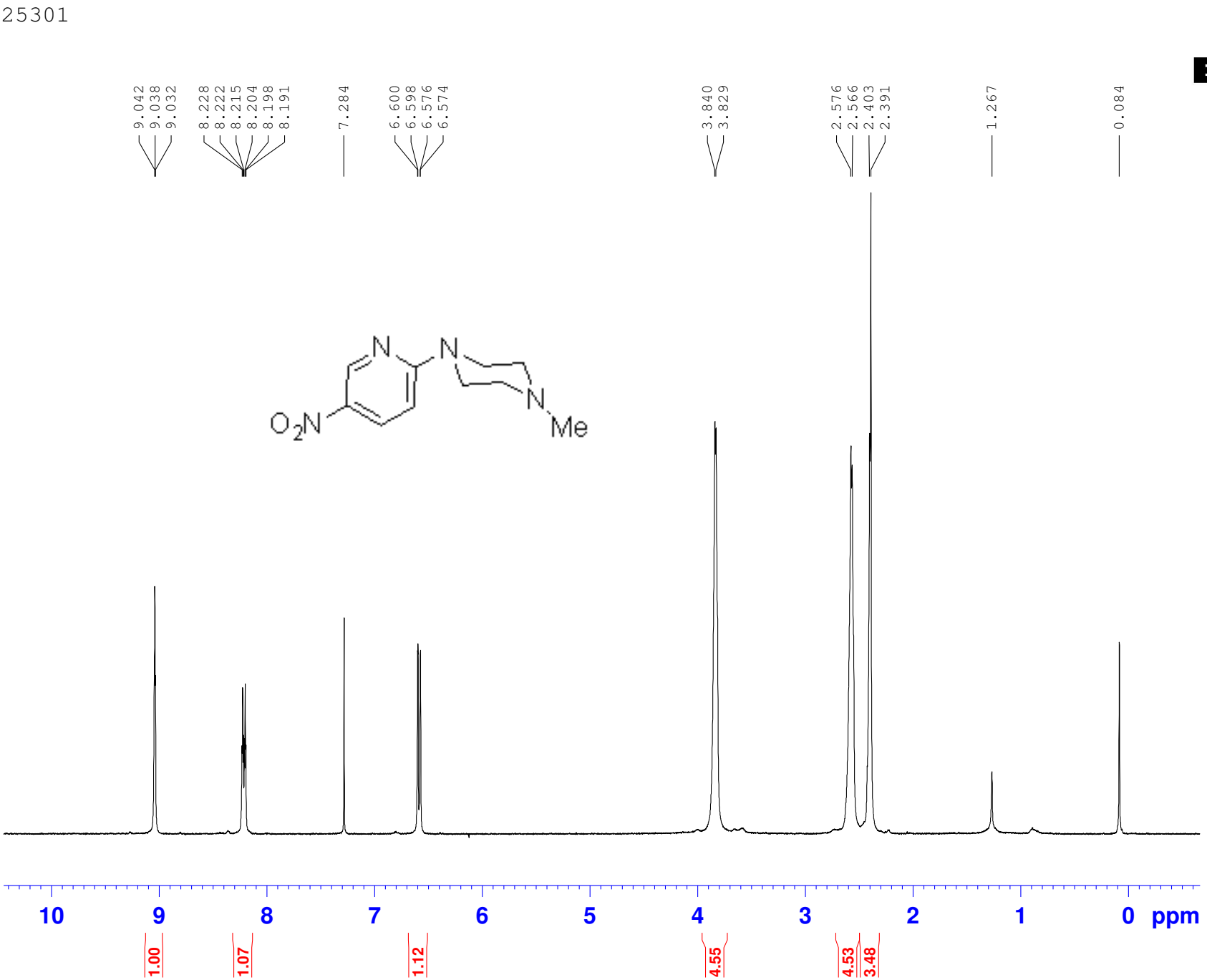
F2 - Acquisition Parameters
Date_           20080927
Time               16.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    49
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 1448.2
DW                20.850 usec
DE                 6.00 usec
TE                301.9 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

125301



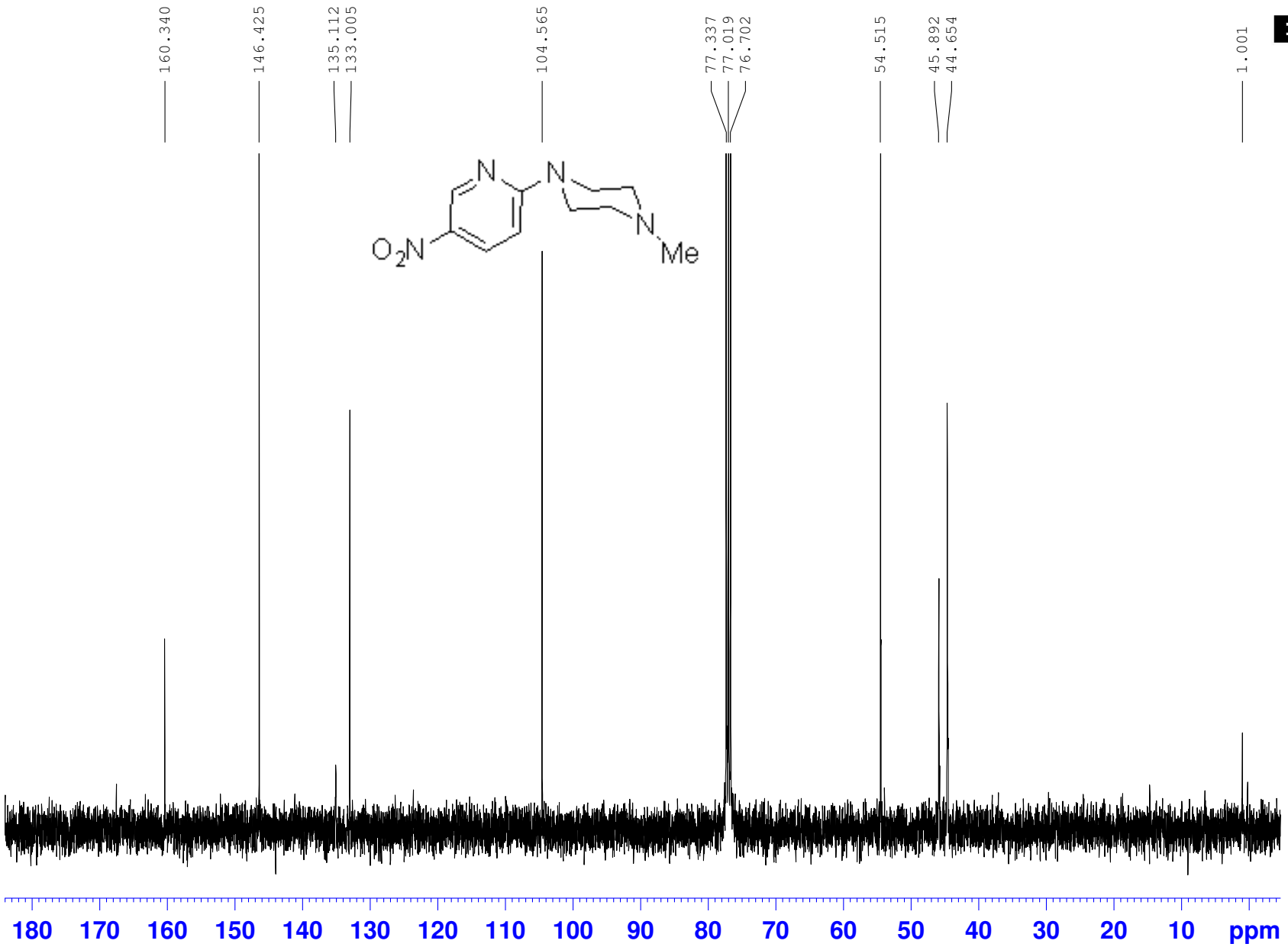
Current Data Parameters
NAME wdp
EXPNO 313
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080927
Time 20.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 301.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125301



BRUKER

Current Data Parameters
NAME wdp
EXPNO 314
PROCNO 1

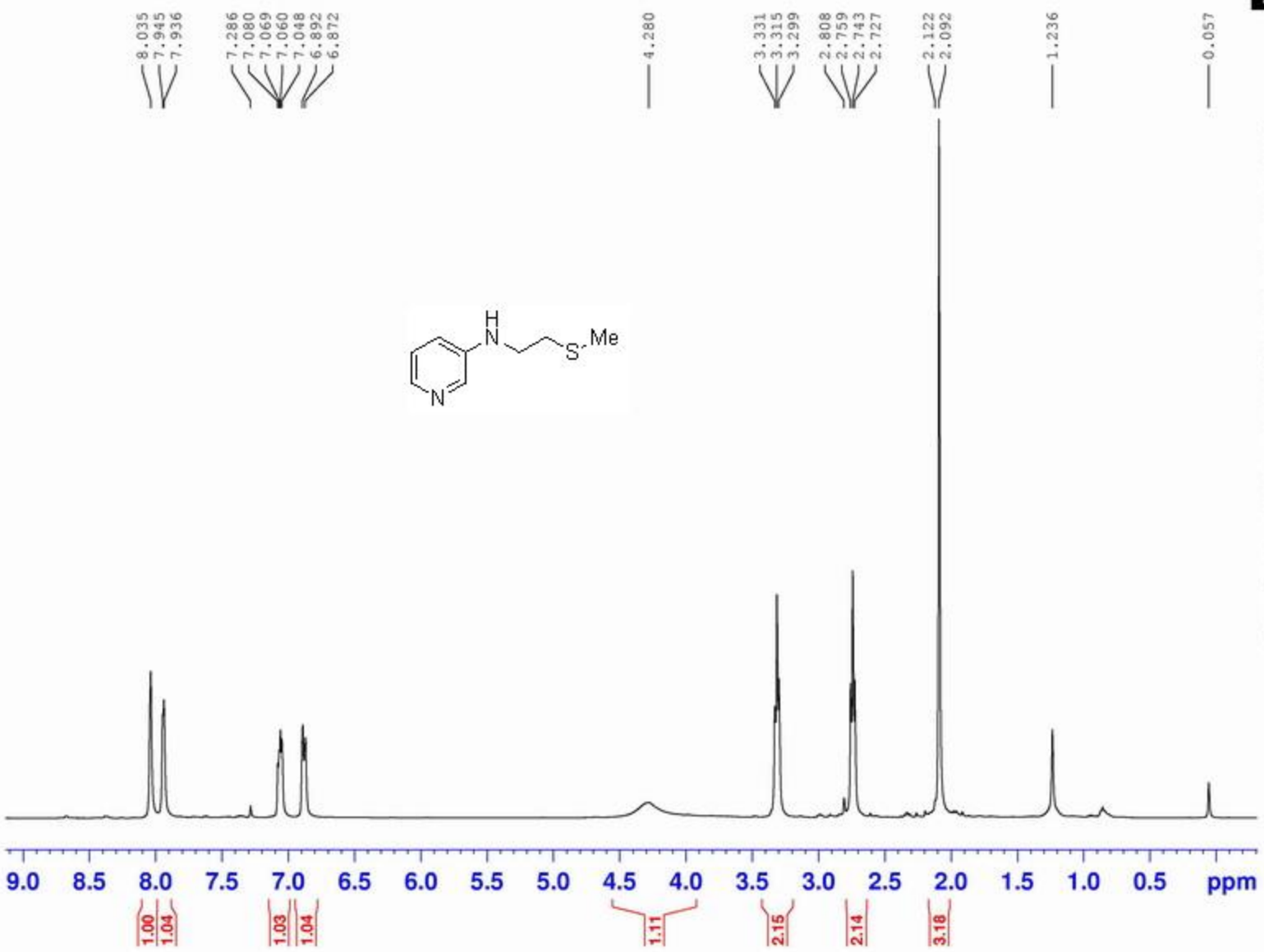
F2 - Acquisition Parameters
Date_ 20080927
Time 20.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 472
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.00 usec
TE 302.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125302



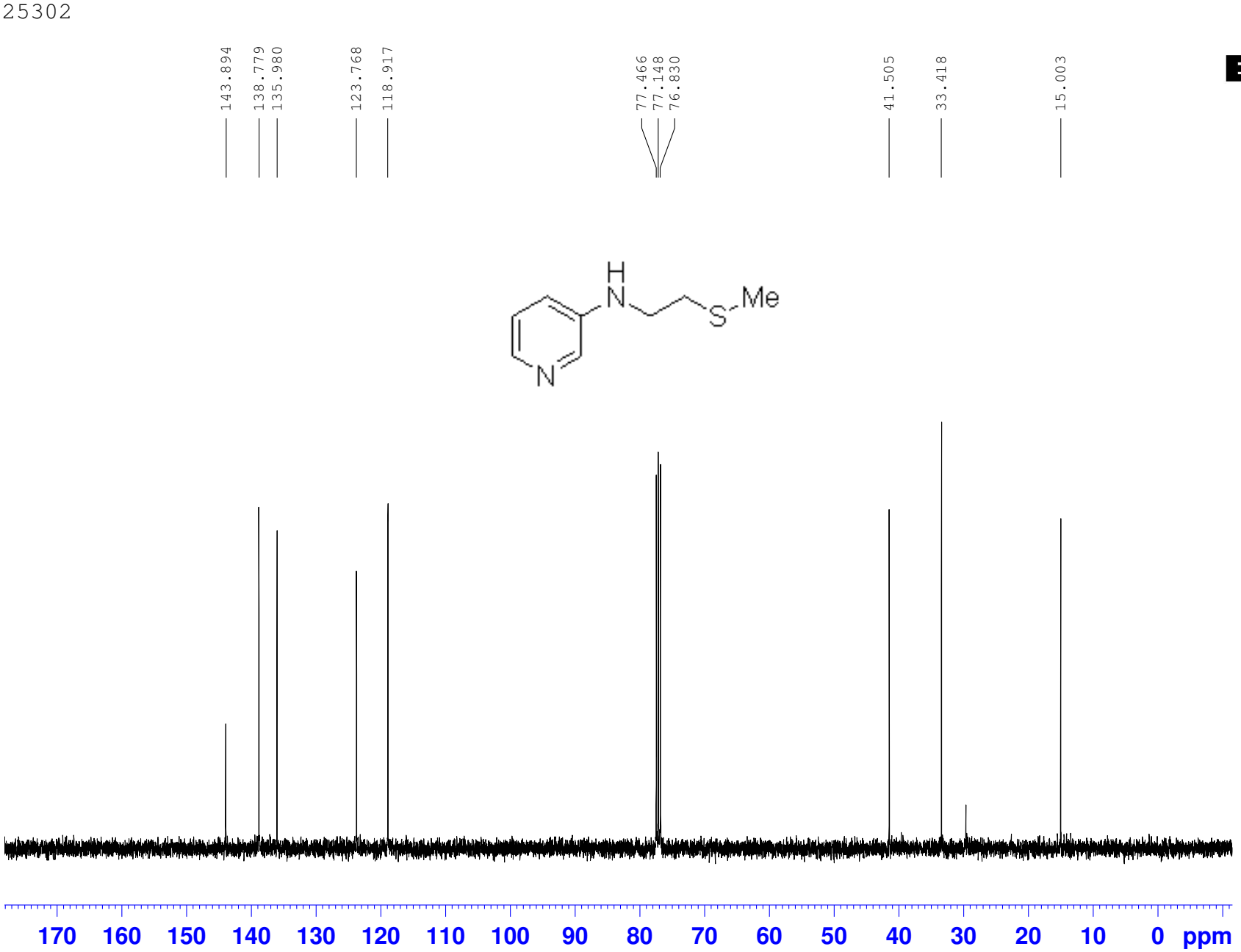

```
Current Data Parameters
NAME                  wdp
EXPNO                 296
PROCNO                1

F2 - Acquisition Parameters
Date_              20080910
Time                 13.21
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     12
DS                      2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                    45.3
DW                  60.400 usec
DE                   6.00 usec
TE                  303.4 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.60 usec
PL1                  0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300000 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00
```

125302




```
Current Data Parameters
NAME                   wdp
EXPNO                  297
PROCNO                   1

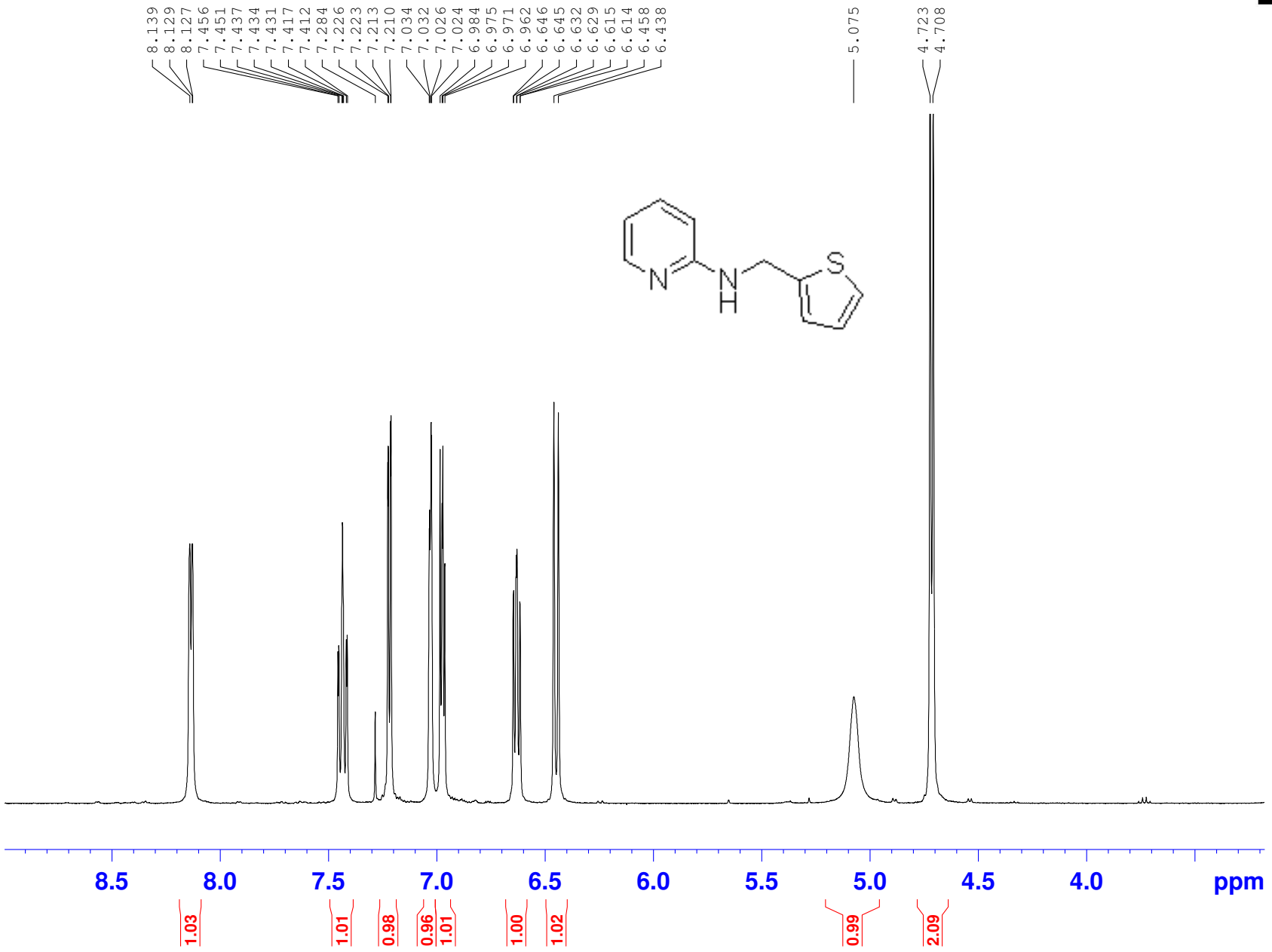
F2 - Acquisition Parameters
Date_           20080910
Time                13.28
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    42
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 912.3
DW                20.850 usec
DE                 6.00 usec
TE                303.5 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                 1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

125309



Current Data Parameters

NAME	wdp
EXPNO	319
PROCNO	1

F2 - Acquisition Parameters

Date_	20080927
Time	22.14
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	5
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	101.6
DW	60.400 usec
DE	6.00 usec
TE	300.9 K
D1	1.00000000 sec
TD0	1

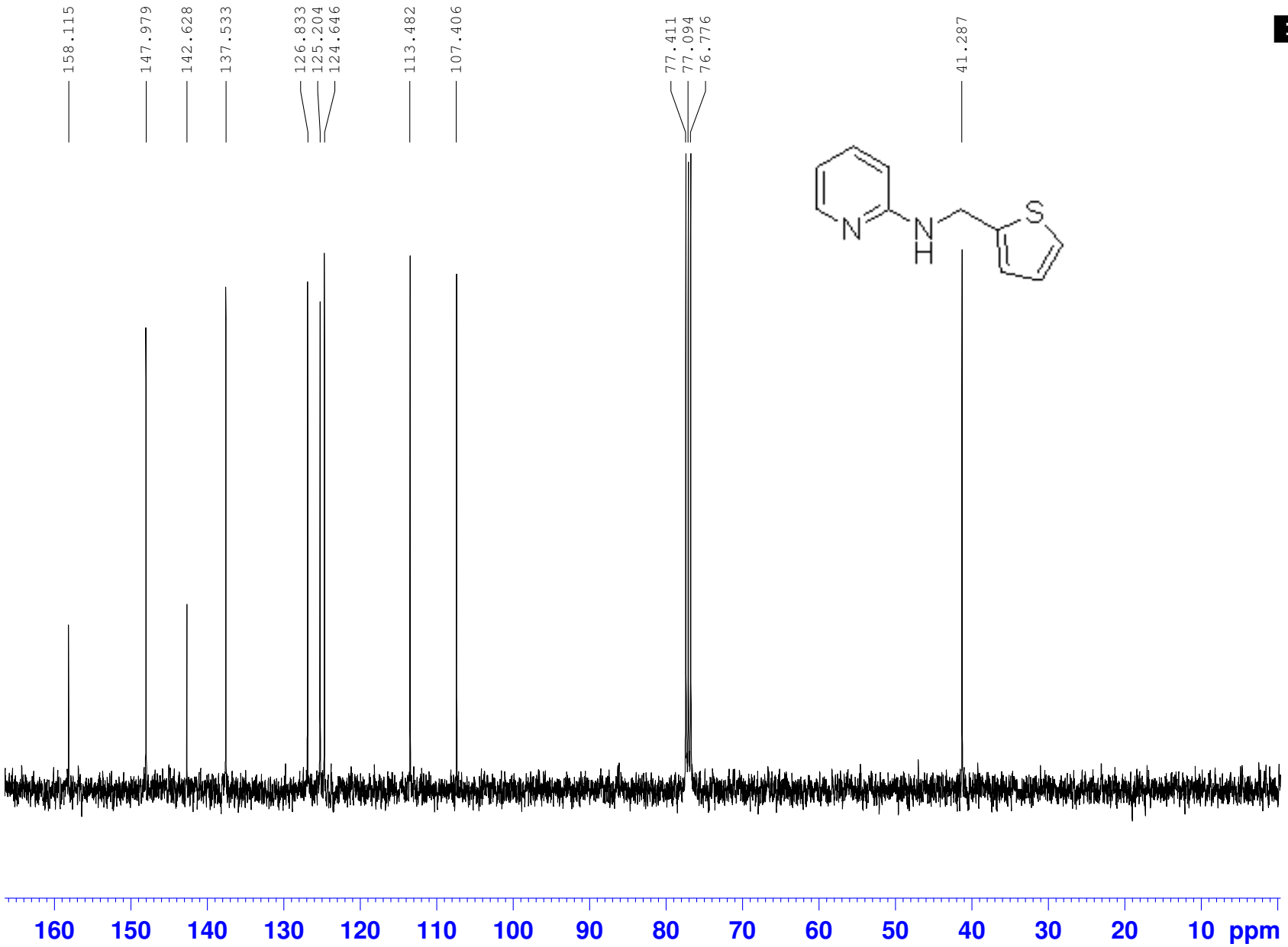
======== CHANNEL f1 ========

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

125309




```
Current Data Parameters
NAME                  wdp
EXPNO                 320
PROCNO                  1

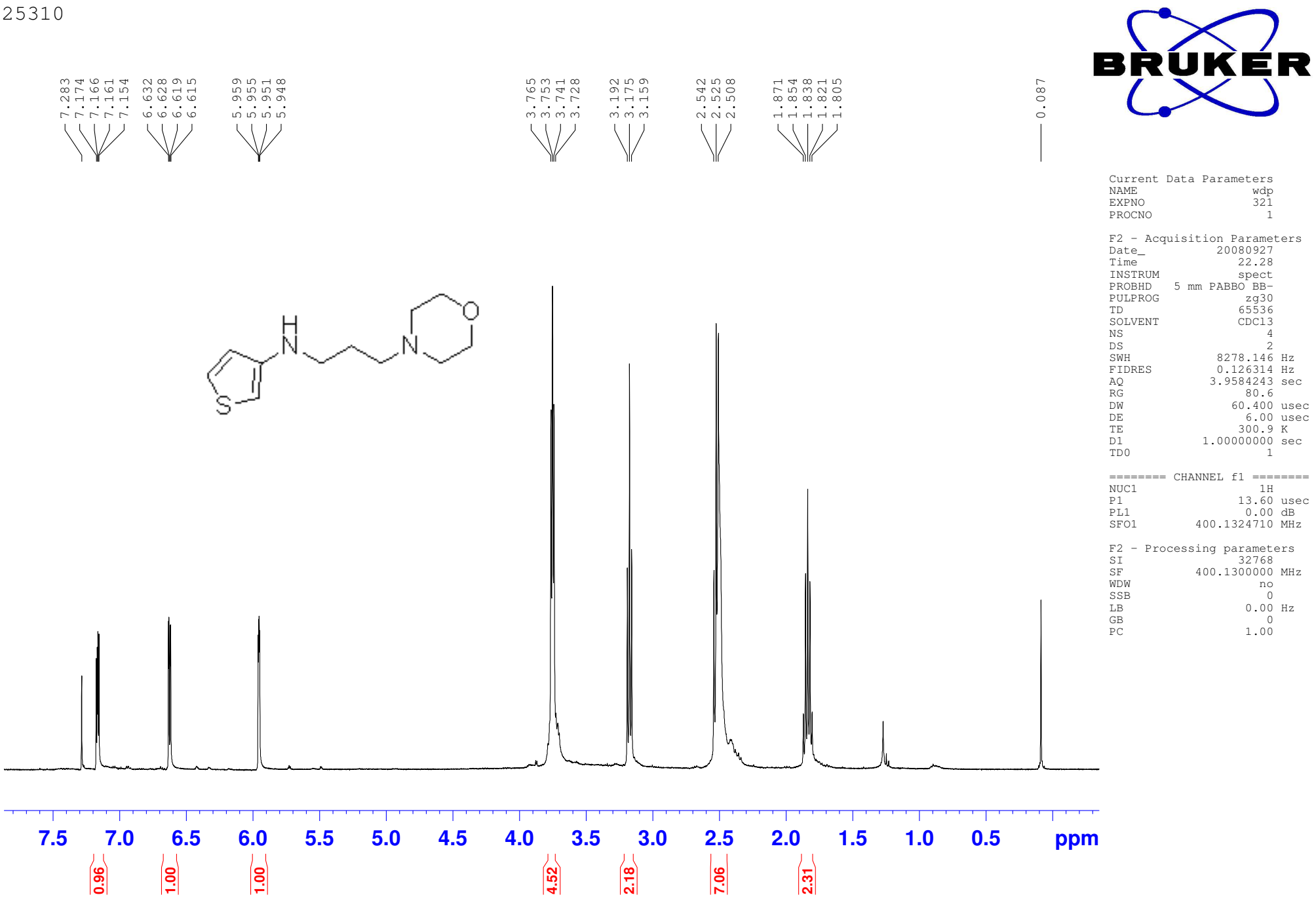
F2 - Acquisition Parameters
Date_           20080927
Time               22.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   38
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG               1149.4
DW               20.850 usec
DE                6.00 usec
TE               301.1 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.20 usec
PL1                1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.39 dB
PL13              15.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.40
```

125310



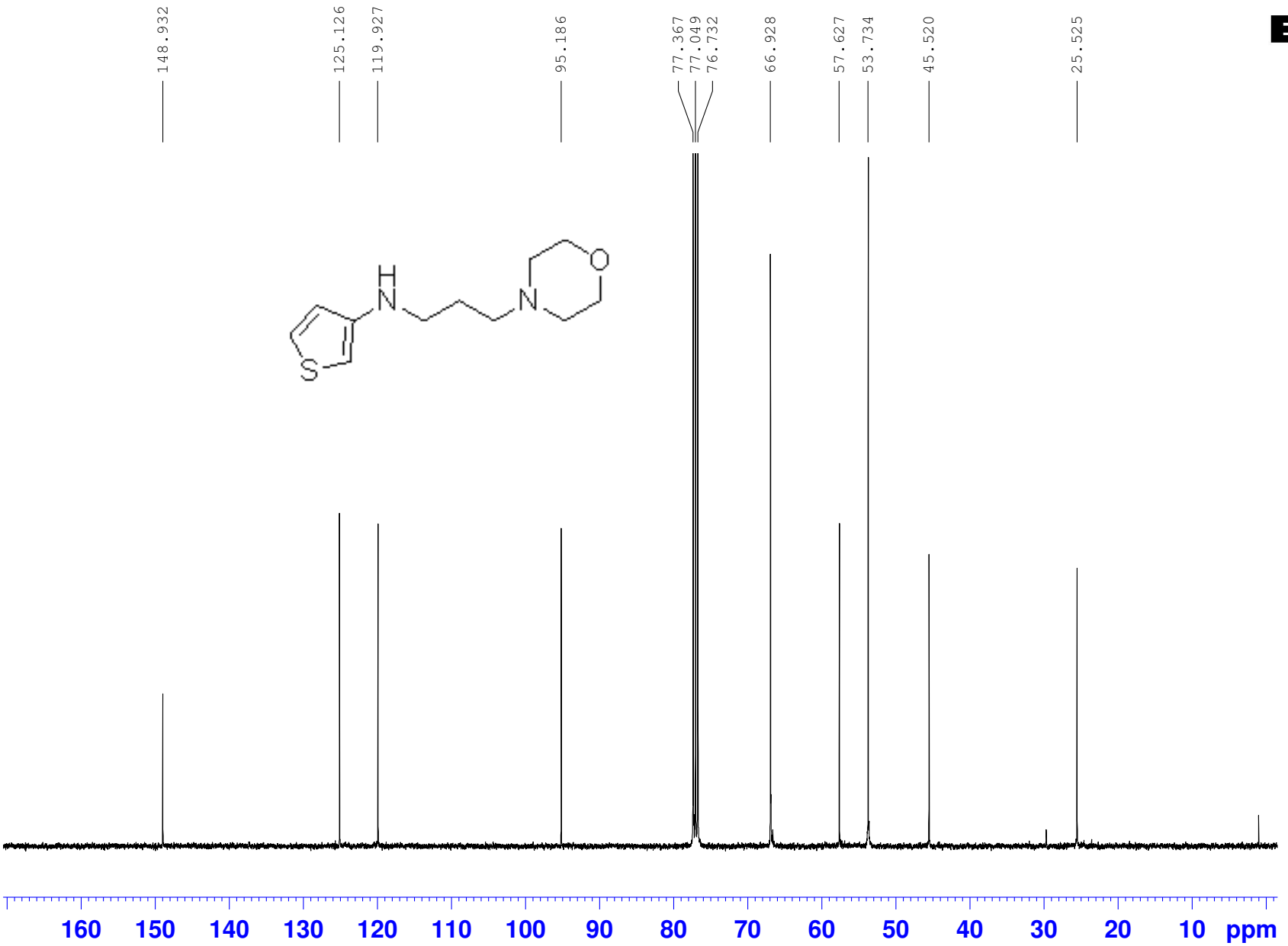
Current Data Parameters
NAME wdp
EXPNO 321
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080927
Time 22.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 80.6
DW 60.400 usec
DE 6.00 usec
TE 300.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

125310




```
Current Data Parameters
NAME                   wdp
EXPNO                  322
PROCNO                   1

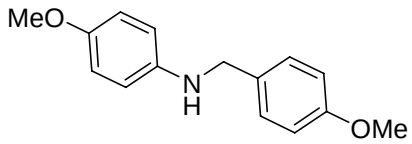
F2 - Acquisition Parameters
Date_           20080928
Time                 0.29
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                 724.1
DW                20.850 usec
DE                 6.00 usec
TE                301.9 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                 1.00 dB
SFO1         100.6228298 MHz

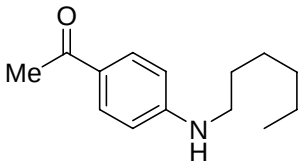
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.39 dB
PL13               15.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

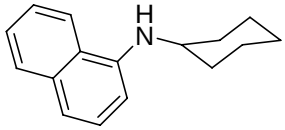
Amination of Aryl and Heteroaryl Bromides Table 4.



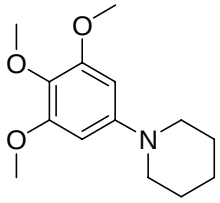
N-(4-methoxybenzyl)- 4-methoxy-benzenamine (9a). Following Procedure B, 1-bromo-4-methoxybenzene (187 mg, 1.0 mmol) was allowed to react with 4-methoxybenzylamine (206 mg, 1.5 mmol) for 14 h. The crude brown oil was purified by flash chromatography on silica gel eluent (15:1 petroleum ether: ethyl acetate) to provide 85 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.31 (d, *J*=8.4 Hz, 2 H; Phenyl), 6.89 (d, *J*=8.4 Hz, 2 H; Phenyl), 6.79 (d, *J*=9.2 Hz, 2 H; Phenyl), 6.65 (d, *J*=9.2 Hz, 2 H; Phenyl), 4.24 (s, 2 H; CH_2), 3.83 (s, 3 H; CH_3), 3.77 ppm (s, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=158.9, 152.5, 142.0, 131.4, 128.9, 114.9, 114.5, 114.0 (Phenyl), 55.8 (CH_2), 55.3 (CH_3), 48.9, ppm (CH_3); GC/MS: rt = 10.01 min, M/Z = 243.



N-*n*-hexyl-4-acety-benzenamine (9b). Following Procedure B, 1-(4-bromophenyl)ethanone (199 mg, 1.0 mmol) was allowed to react with *n*-hexanamine (152 mg, 1.5 mmol) for 8 h. The crude brown oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 95 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.81 (d, *J*=8.8 Hz, 2 H; Phenyl), 6.55 (d, *J*=8.8 Hz, 2 H; Phenyl), 4.47 (br s, 1 H; NH), 3.15 (t, *J*=7.6 Hz, 2 H; -NH-C*H*$_2$), 2.49 (s, 3 H; CH_3), 1.67-1.59 (m, 2 H; CH_2), 1.42-1.27 (m, 6 H; CH_2), 0.91 ppm (m, 3 H; CH_3); ^{13}C NMR($CDCl_3$) : δ=196.3 (-C=O), 152.4, 130.8, 126.4, 111.3 (Phenyl), 43.4 (-NH-C*H*$_2$), 31.6 (CH_3), 29.2, 26.7, 25.9, 22.6 (CH_2), 14.0 ppm (CH_3); GC/MS: rt = 9.27 min, M/Z = 219.

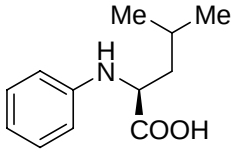


1-(N-cyclohexyl)-naphthalenamine (9c). Following Procedure B, 1-bromonaphthalene (207 mg, 1.0 mmol) was allowed to react with cyclohexanamin*e* (149 mg, 1.5 mmol) for 18 h. The crude brown oil was purified by flash chromatography on silica gel eluent (100:1 petroleum ether: ethyl acetate) to provide 83 % yield of the desired product as a colourless oil. ^{1}H NMR($CDCl_3$): δ=7.87-7.83 (m, 2 H; Phenyl), 7.52-7.45 (m, 2 H; Phenyl), 7.38 (t, *J*=8.0 Hz, 1 H; Phenyl), 7.25 (d, *J*=8.0 Hz, 1 H; Phenyl), 6.71 (d, *J*=7.6 Hz, 1 H; Phenyl), 4.38 (br s, 1 H; NH), 3.57-3.49 (m, 1 H; -NH-C*H*-), 2.22 (m, 2 H; CH_2), 2.19-1.85 (m, 2 H; CH_2), 1.78-1.73 (m, 1 H; CH_2), 1.56-1.41 ppm (m, 5 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=142.3, 134.6, 128.7, 126.7, 125.6, 124.5, 123.5, 119.9, 116.8, 104.9 (Phenyl), 51.9 (-NH-C*H*-), 33.3, 26.1, 25.1 ppm (CH_2); GC/MS: rt = 9.66 min, M/Z = 225.

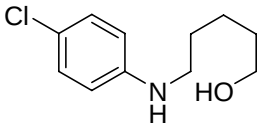


1-piperidine-3,4,5-trimethoxyphenyl (9d). Following Procedure B, 5-bromo-1,2,3-trimethoxybenzene (247 mg, 1.0 mmol) was allowed to react with piperidine (128 mg, 1.5 mmol) for 20 h. The crude brown oil was purified by flash

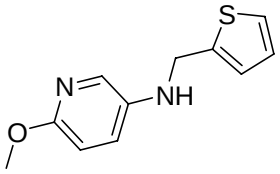
chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 80 % yield of the desired product as a pale yellow oil. ^{1}H NMR($CDCl_3$): δ=6.22 (s, 2 H; Phenyl), 3.86 (s, 6 H; CH_3), 3.80 (s, 3 H; CH_3), 3.09 (t, *J*=5.6 Hz, 4 H; -N-CH_2), 1.78-1.72 (m, 4 H; CH_2), 1.61-1.58 ppm (m, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=153.5, 149.6, 132.1, 95.3 (Phenyl), 60.9 (CH_3), 56.1 (CH_3), 51.8 (-N-CH_2-), 25.9, 24.2 ppm (CH_2); GC/MS: rt = 9.01 min, M/Z = 251.



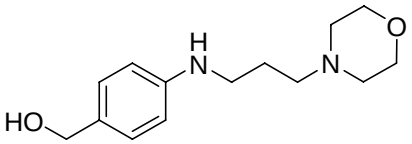
(S)-4-methyl-2-(phenylamino)pentanoic acid[6] (9e). Following Procedure B, bromobenzene (157 mg, 1.0 mmol) was allowed to react with (S)-2-amino-4-methylpentanoic acid (197 mg, 1.5 mmol) for 12 h. The crude brown oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 93 % yield of the desired product as a white solid. ^{1}H NMR(*d*-DMSO): δ=7.05 (t, *J*=8.0 Hz, 2 H; Phenyl), 6.53 (m, 3 H; Phenyl), 3.83 (m, 1 H; -N-CH-), 1.78 (m, 1 H; -C*H*-CH_3), 1.64-1.57 (m, 2 H; CH_2), 0.94 (d, *J*=6.8 Hz, 3 H; CH_3), 0.88 ppm (d, *J*=6.4 Hz, 3 H; CH_3); ^{13}C NMR(*d*-DMSO) : δ=176.3, 148.5, 129.3, 116.6, 112.8 (Phenyl), 54.7 (-N-CH-), 41.6 (-C*H*-CH_3), 24.9 (CH_2), 23.2 (CH_3), 22.2 ppm (CH_3); $[\alpha]_D^{18}$ = -48.0^{o} (c 0.80, acetone), literature: -45.0^{o}; MS (APCI): M/Z: 206 [M-].



5-(4-chlorophenylamino)-1-pentanol (9f). Following Procedure B, 1-bromo-4-chlorobenzene (191 mg, 1.0 mmol) was allowed to react with 5-amino-1-pentanol (155 mg, 1.5 mmol) for 17 h. The crude brown oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 87 % yield of the desired product as a pale yellow oil. ^{1}H NMR($CDCl_3$): δ=7.11 (m, 2 H; Phenyl), 6.54 (m, 2 H; Phenyl), 3.66 (t, *J*=6.4 Hz, 2 H; -O-CH_2-), 3.09 (t, *J*=7.2 Hz, 2 H; -N-CH_2-), 2.58 (br s, 2 H; NH and OH), 1.70-1.59 (m, 4 H; CH_2), 1.53-1.47 ppm (m, 2 H; CH_2); ^{13}C NMR($CDCl_3$) : δ=146.7, 129.1, 122.0, 114.0 (Phenyl), 62.7 (-O-CH_2-), 44.2 (-N-CH_2-), 32.4, 29.1, 23.3 ppm (CH_2); GC/MS: rt = 8.94 min, M/Z = 213.



3-[N-(2-methylthiophen)]-6-methoxy-pyridinamine (9g). Following Procedure B, 5-bromo-2-methoxypyridine (188 mg, 1.0 mmol) was allowed to react with thiophen-2-ylmethanamine (170 mg, 1.5 mmol) for 18 h. The crude brown oil was purified by flash chromatography on silica gel eluent (10:1 petroleum ether: ethyl acetate) to provide 92 % yield of the desired product as a white solid. ^{1}H NMR($CDCl_3$): δ=7.66 (s, 1 H; Phenyl), 7.22 (d, *J*=5.2 Hz, 1 H; Phenyl), 7.06-6.96 (m, 3 H; Phenyl), 6.63 (d, *J*=8.8 Hz, 1 H; Phenyl), 4.48 (s, 2 H; CH_2), 3.88 (s, 3 H; CH_3), 3.74 ppm (br s, 1 H; NH); ^{13}C NMR($CDCl_3$) : δ=157.8, 142.5, 138.4, 130.9, 126.9, 126.3, 125.2, 124.8, 110.8 (Phenyl), 53.4 (CH_2), 44.4 ppm (CH_3); GC/MS: rt = 8.84 min, M/Z = 220.

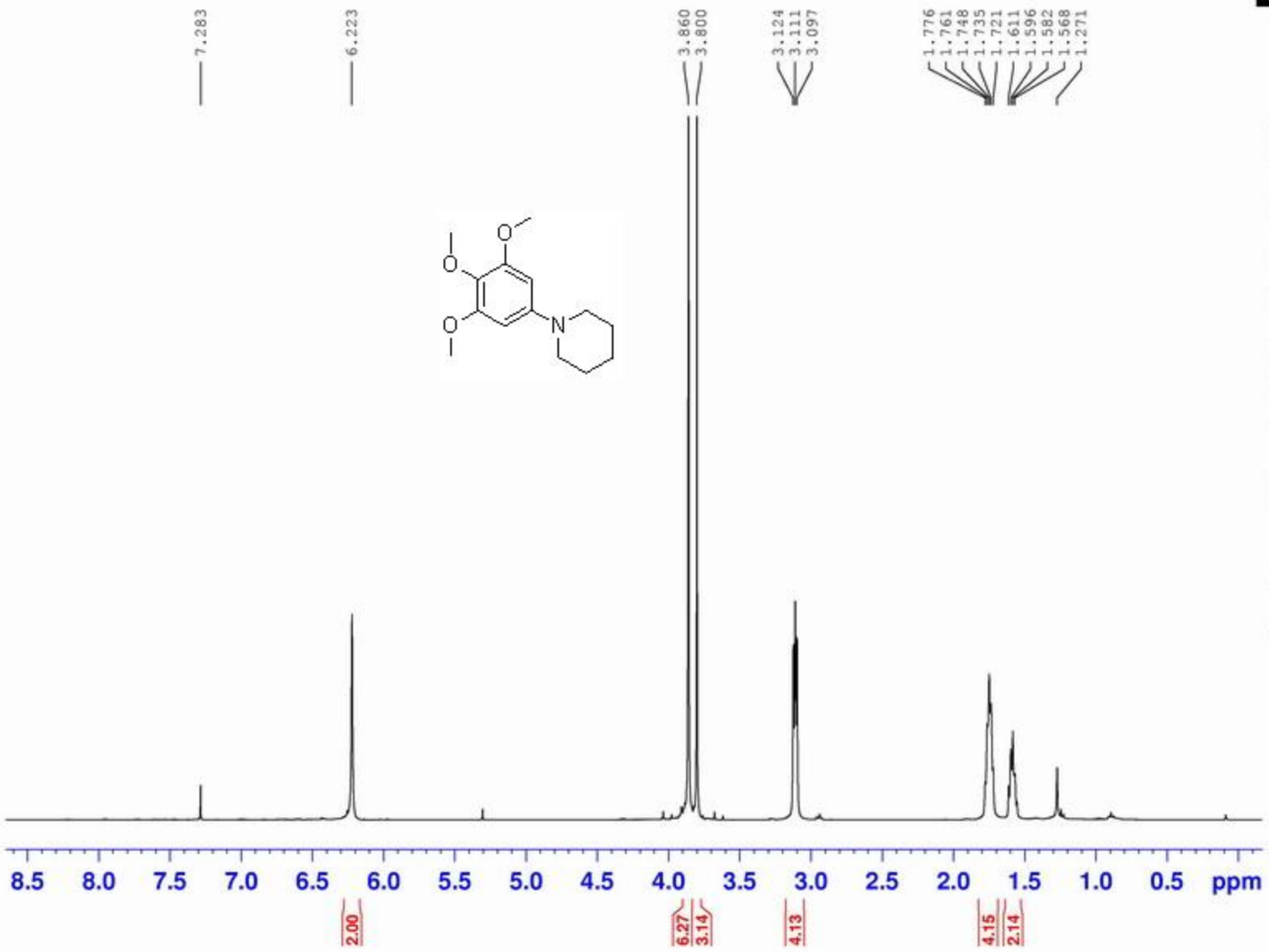


4-(3-morpholinopropylamino)-benzylnol (9h). Following Procedure B, 4-bromobenzylnol (187 mg, 1.0 mmol) was allowed to react with 1-(3-morpholino)propanamine (216 mg, 1.5 mmol) for 17 h. The crude brown oil was purified by flash chromatography on silica gel eluent (30:1 CH_2Cl_2: MeOH) to provide 90 % yield of the desired product as a white solid. ^{1}H NMR(*d*-DMSO): δ=7.00 (d, *J*=8.0 Hz, 2 H; Phenyl), 6.49 (d, *J*=8.4 Hz, 2 H; Phenyl), 5.5 (br s, 1 H; NH), 4.77 (t, *J*=5.2 Hz, 1 H; OH), 4.29 (d, *J*=5.2 Hz, 2 H; -C*H*$_2$-OH), 3.58 (s, 4 H; -O-CH_2-), 3.03 (br s, 2 H; -NH-CH_2-), 2.36 (s, 6 H; -N-CH_2-), 1.66 ppm (t, *J*=6.8 Hz, 2 H; CH_2); ^{13}C NMR(*d*-DMSO) : δ=148.5, 129.8, 128.4, 112.1 (Phenyl), 66.7 (-C*H*$_2$-OH), 63.6 (-O-CH_2-), 56.7 (-NH-CH_2-), 53.9 (-N-CH_2-), 41.8 (-N-CH_2-), 26.1 ppm (CH_2); MS (EI): m/z: 250 (35) [M+], 162 (25), 132 (45), 100 (99); HRMS (EI): calcd for $C_{14}H_{22}O_2N_2$: 250.1676 [M+]; found: 250.1674.

Refrence

1. K. Yoshikazu, M. Hiroshi, M. Denichi, EP277755.
2. M. Kawase, M. Teshima, S. Saito, S. Tani, *Heterocycles*, 1998, **48**, 2103.
3. H. Hamana, M. Yamazaki, *Chem. Pharm. Bull.* 1963, **11**, 415.
4. M. Andrei V., *Org. Lett.*, 2004, **6**, 2253.
5. (a) F. Savina, L. Fulvio, L. Antonio, M. Angelica, T. Vincenzo, A. Rosa, *Chirality*, 2000, **12**, 697; (b) Q. Jiang, D. Jiang, Y. Jiang, H. Fu, Y. Zhao, *Synlett*, 2007, 1836.
6. S. Röttger, P. J. R. Sjöberg and M. Larhed, *J. Comb. Chem.*, 2007, **9**, 204.

125307



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PROCNO	1

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PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	9
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	6.00 usec
TE	302.0 K
D1	1.00000000 sec
TD0	1

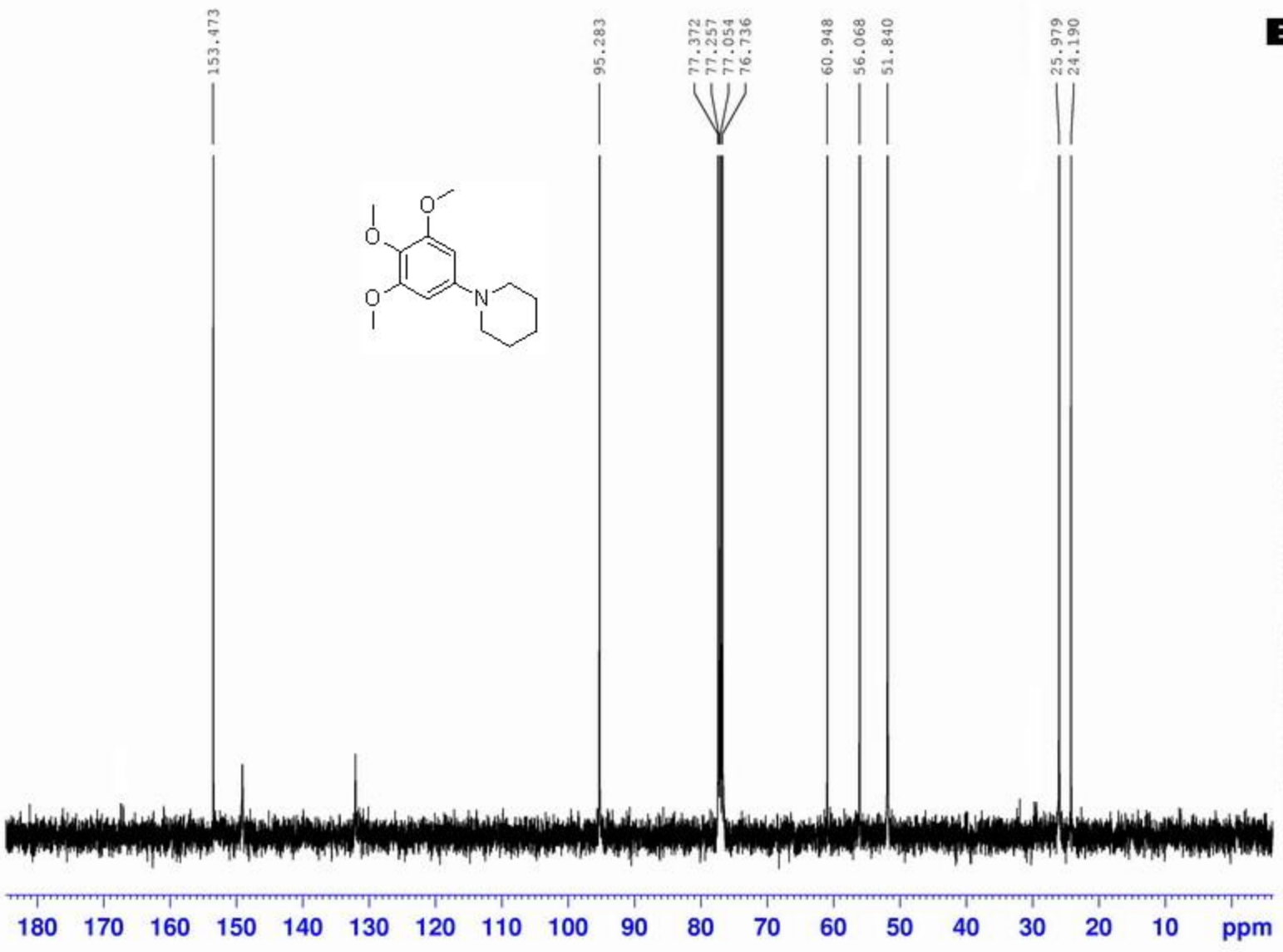
======== CHANNEL f1 ========

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

125307



BRUKER

Current Data Parameters
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EXPNO 316
PROCNO 1

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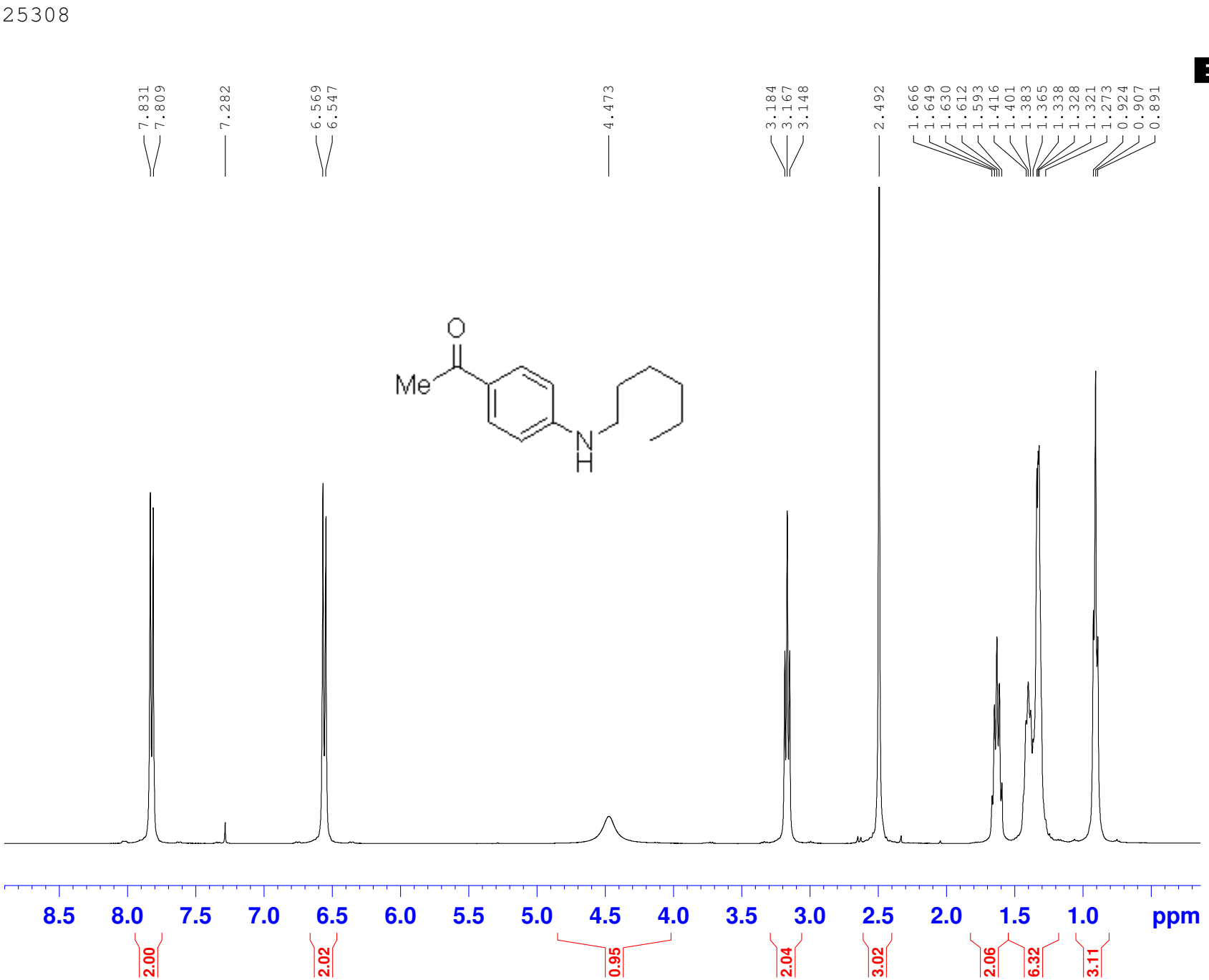
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 414
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 302.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125308



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EXPNO 317
PROCNO 1

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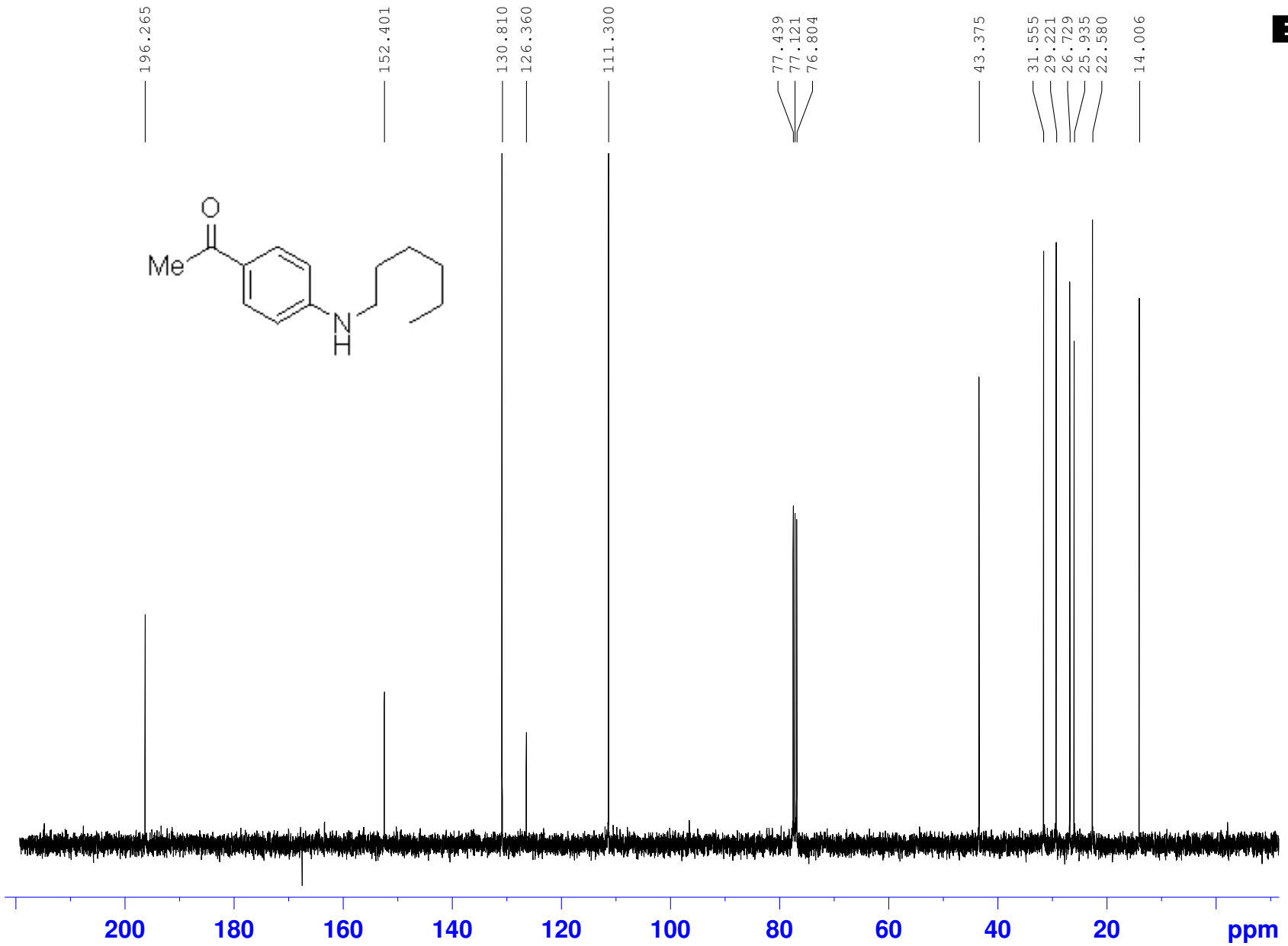
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.00 usec
TE 301.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125308



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NAME wdp
EXPNO 318
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 41
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.00 usec
TE 301.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
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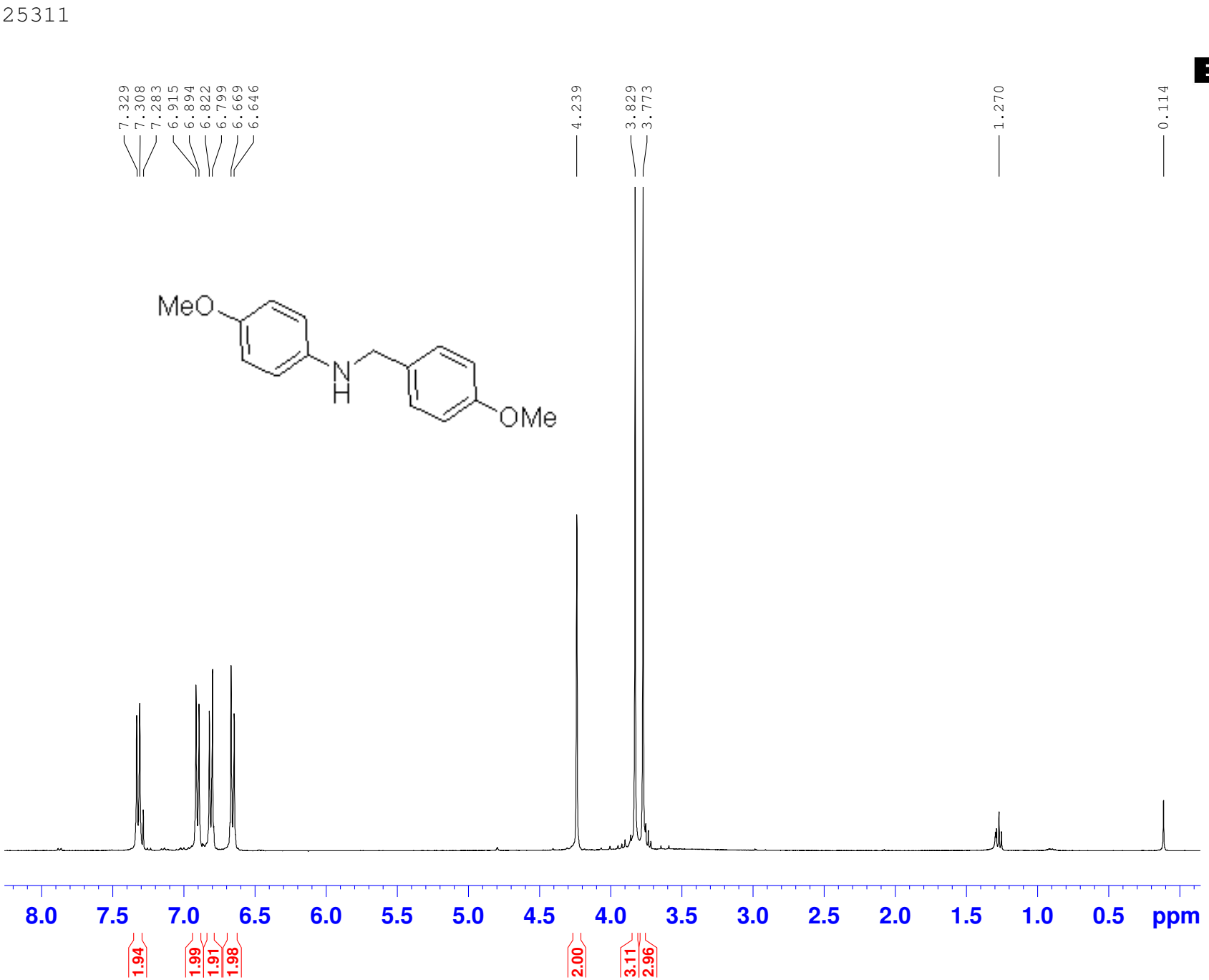
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125311

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EXPNO                 325
PROCNO                  1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   128
DW                60.400 usec
DE                 6.00 usec
TE                300.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.60 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
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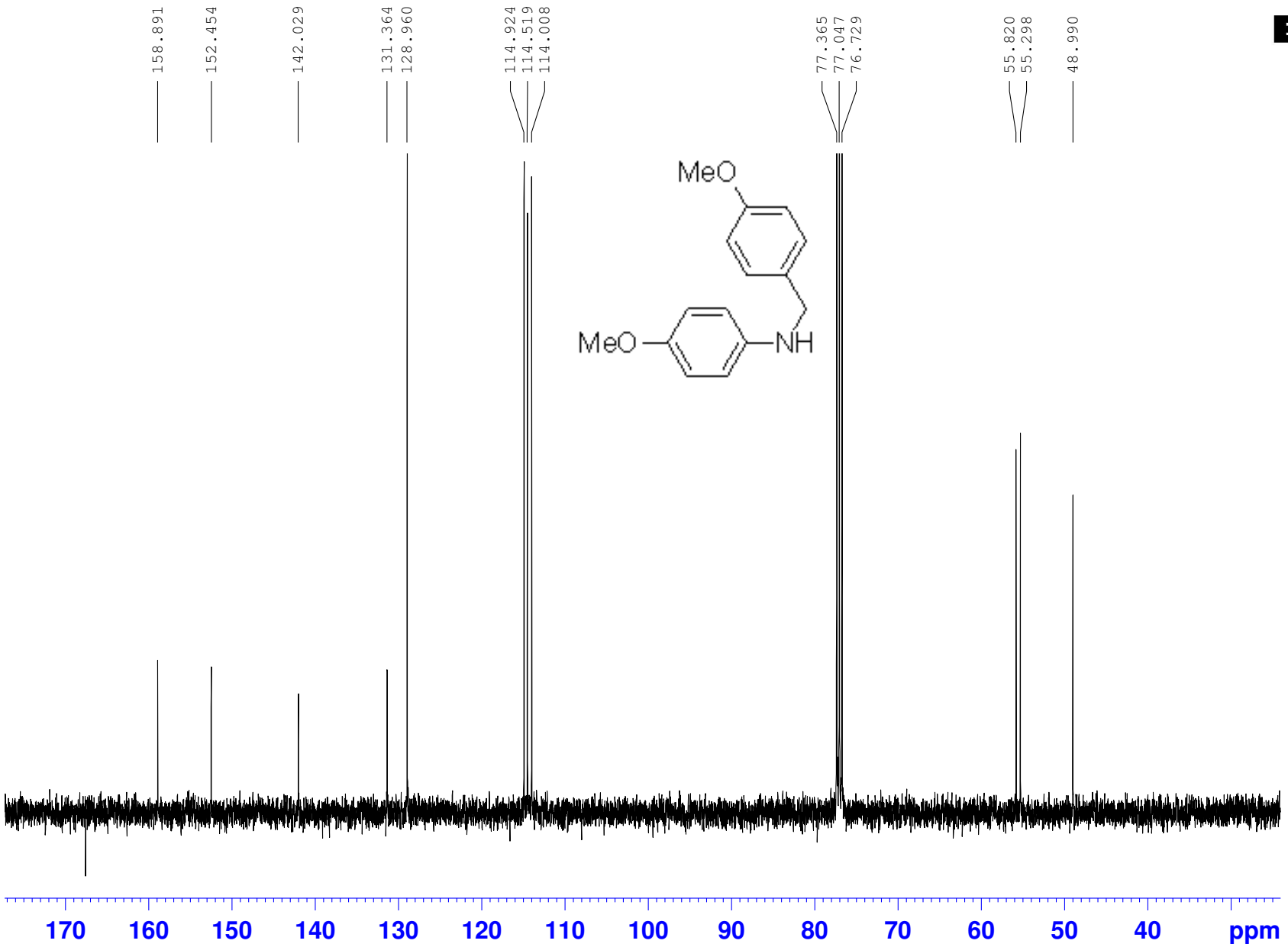
125311



Current Data Parameters
NAME wdp
EXPNO 324
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080928
Time 9.04
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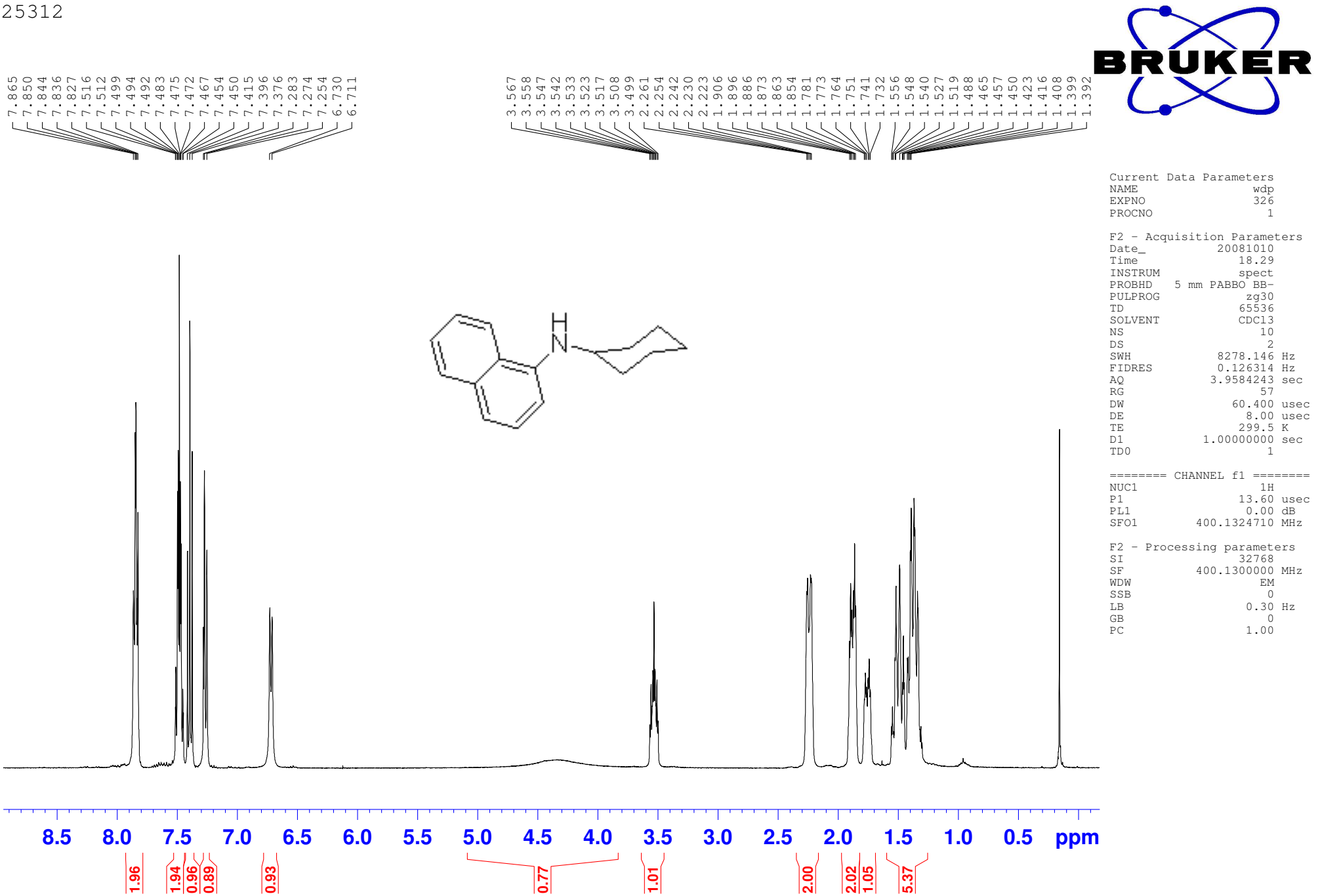
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 103
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 912.3
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.39 dB
PL13 15.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125312

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Current Data Parameters
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EXPNO                 326
PROCNO                  1

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Time               18.29
INSTRUM            spect
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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    10
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                    57
DW                60.400 usec
DE                 8.00 usec
TE                299.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.60 usec
PL1                 0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF            400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
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PC                  1.00
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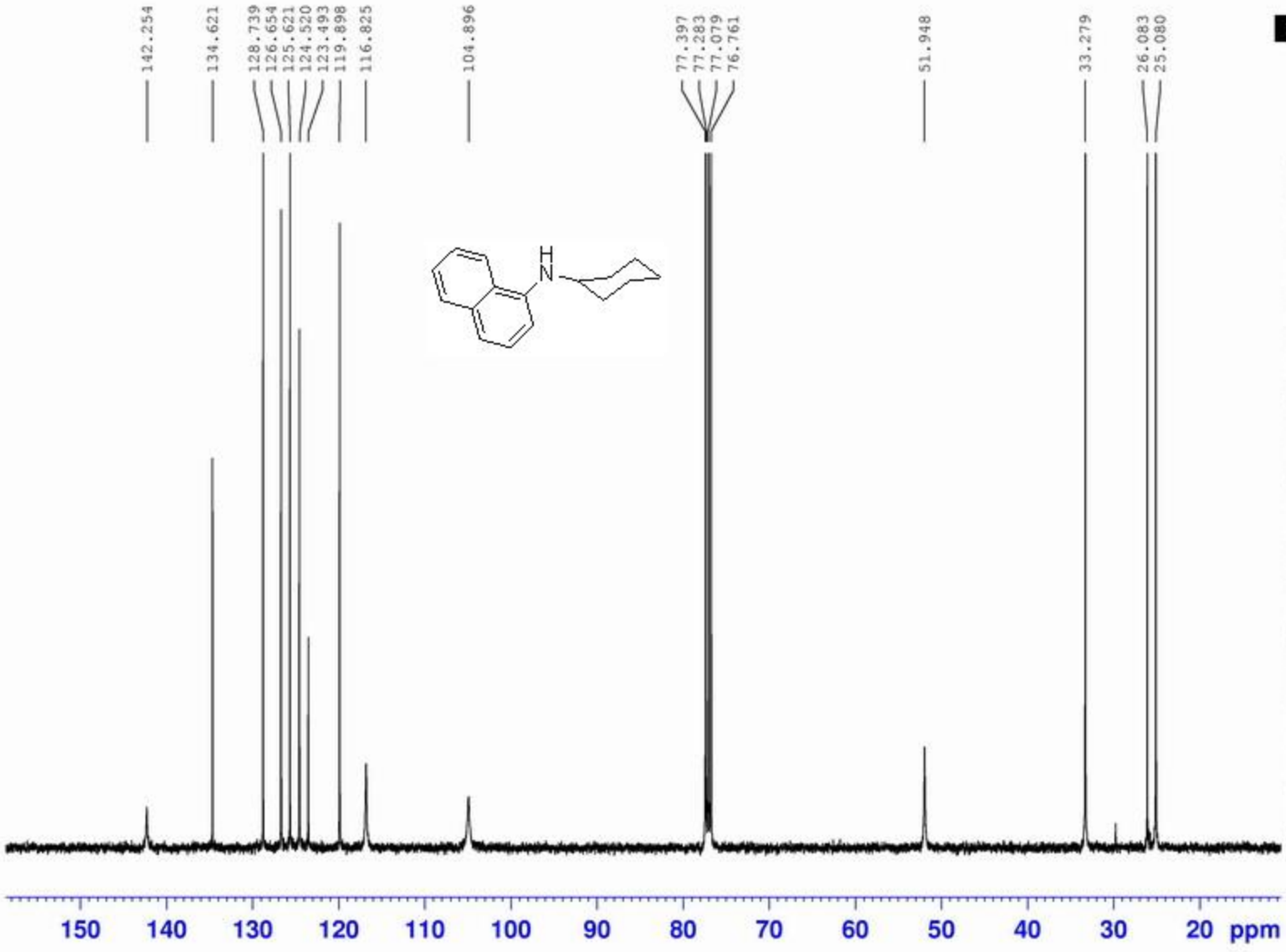
125312



BRUKER

Current Data Parameters

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EXPNO	327
PROCNO	1

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FIDRES	0.365918 Hz
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RG	32768
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DE	8.00 usec
TE	300.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

NUC1	13C
P1	12.20 usec
PL1	1.00 dB
SFO1	100.6228298 MHz

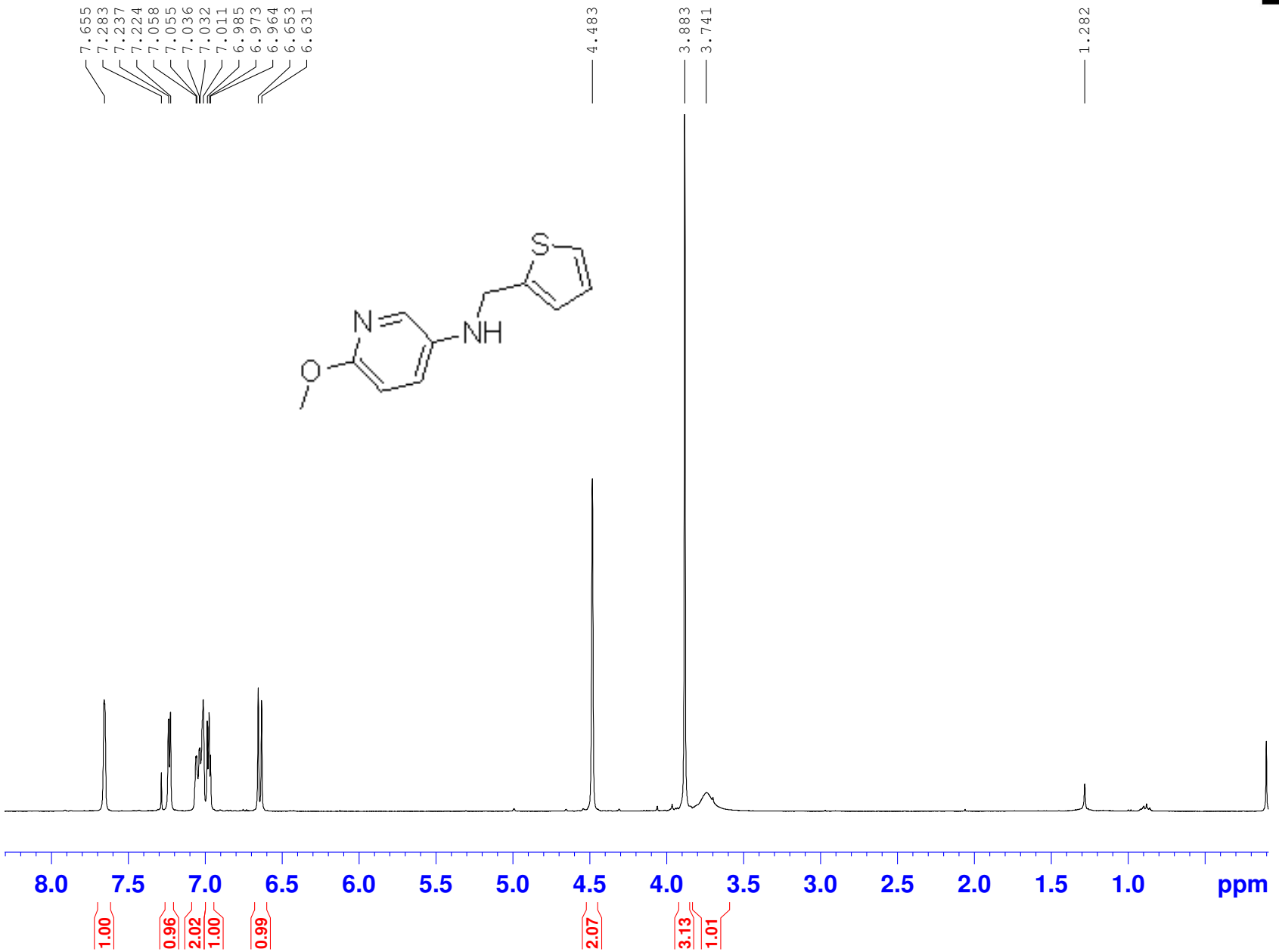
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL12	15.39 dB
PL13	15.50 dB
PL2	0.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

125313



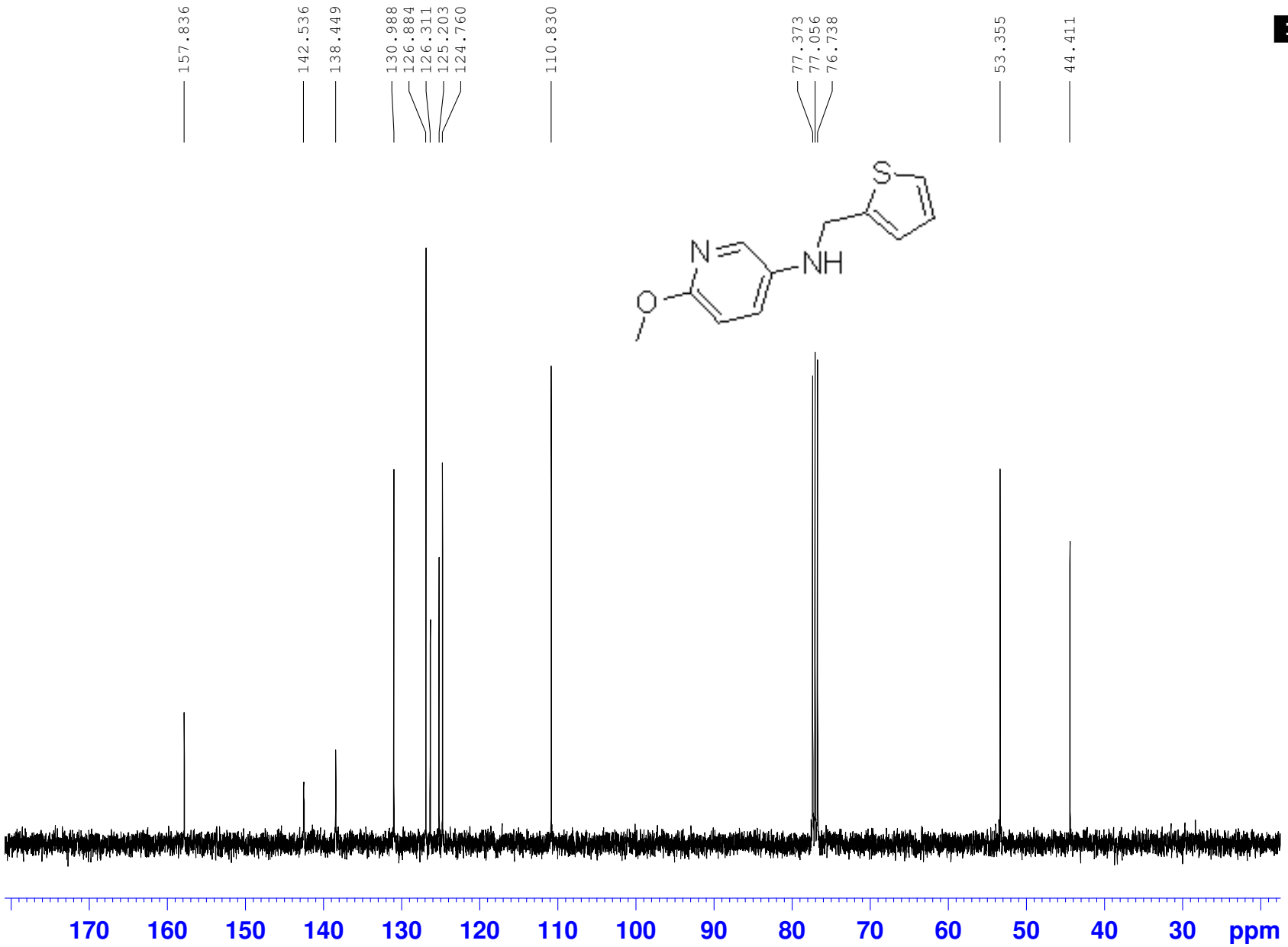
Current Data Parameters
NAME wdp
EXPNO 331
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081010
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 8.00 usec
TE 299.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125313



Current Data Parameters
NAME wdp
EXPNO 332
PROCNO 1

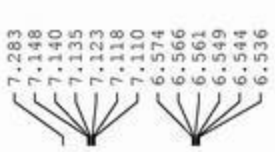
F2 - Acquisition Parameters
Date_ 20081010
Time 20.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 108
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 8.00 usec
TE 300.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

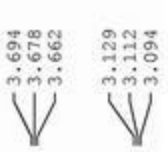
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

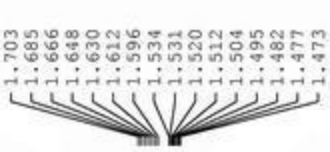
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 15.39 dB
PL13 15.50 dB
PL2 0.00 dB
SFO2 400.1316005 MHz

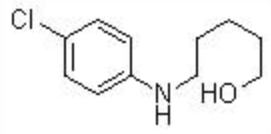
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

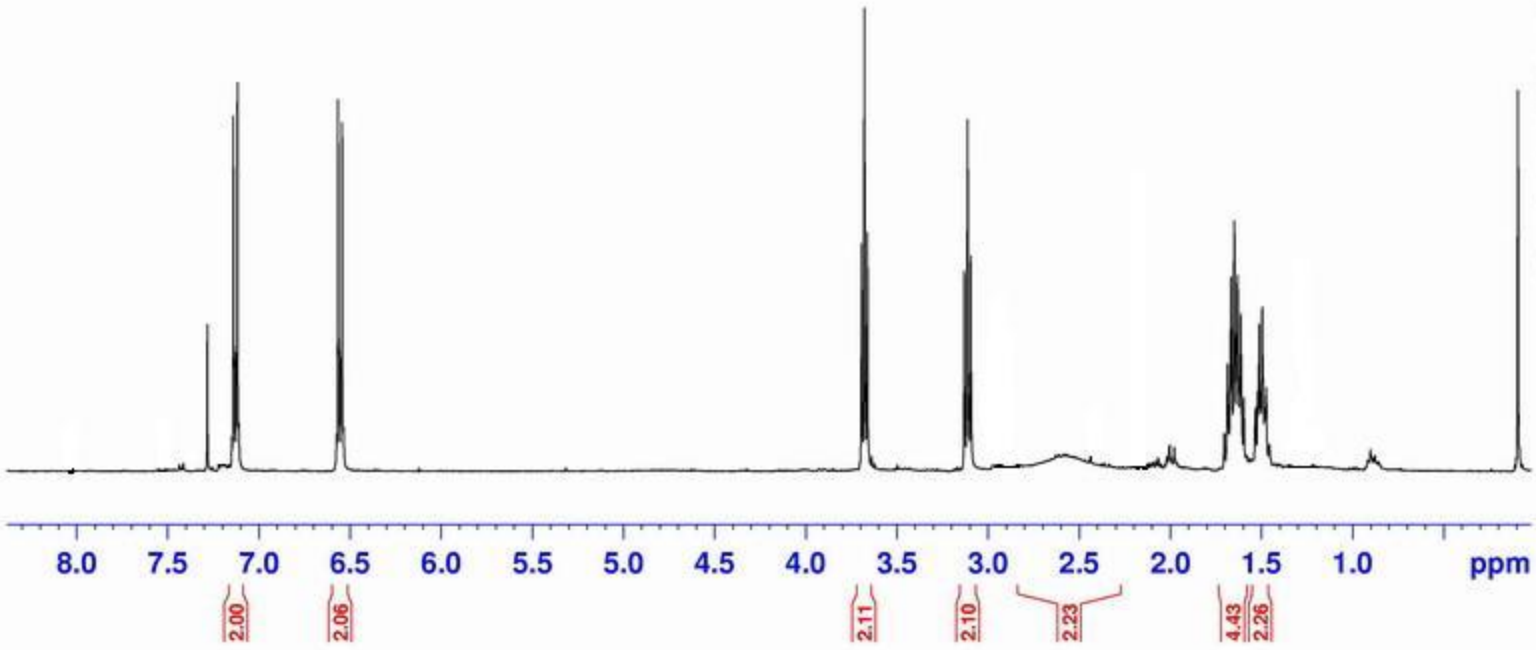
12531











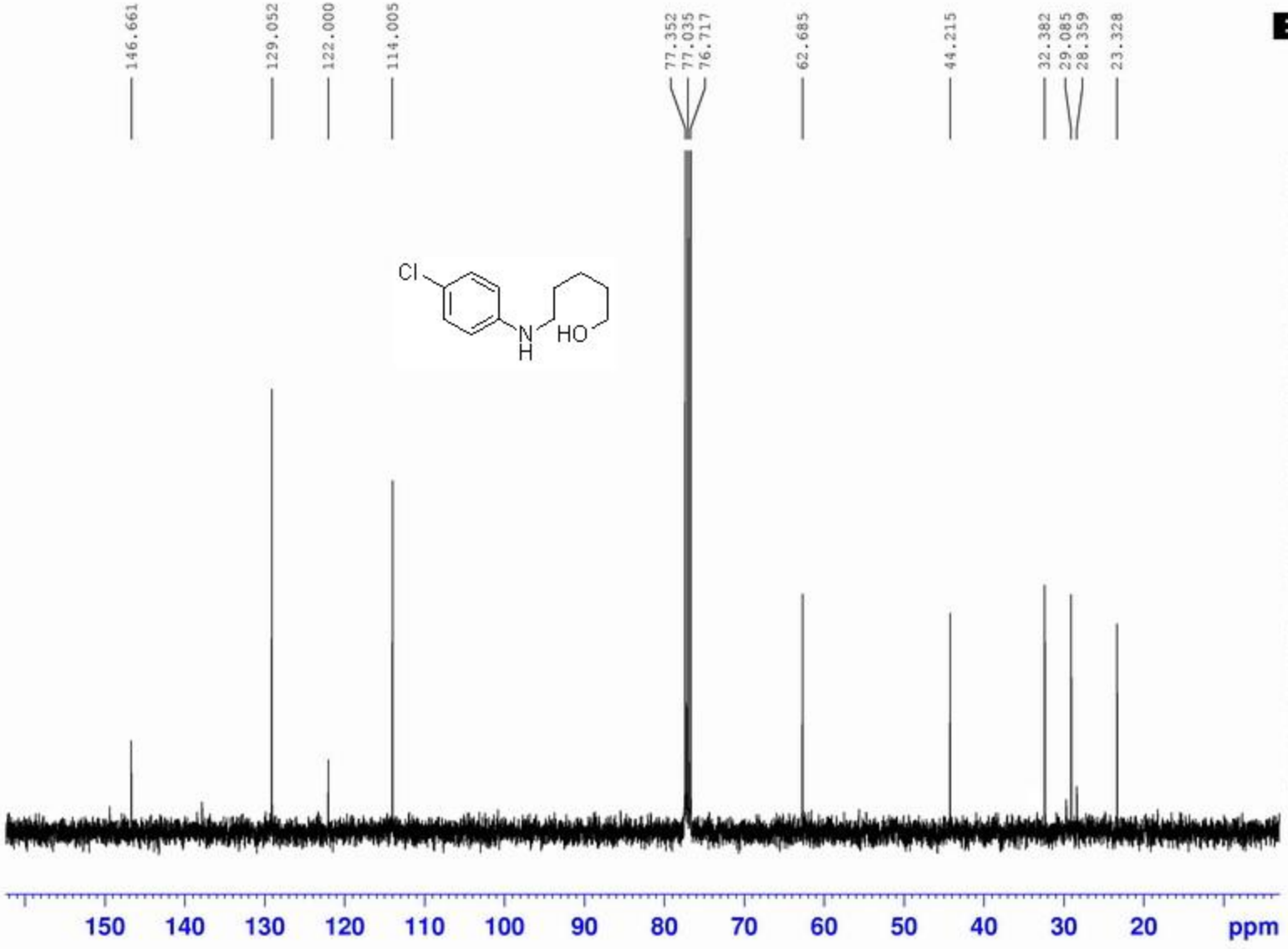
Current Data Parameters
NAME wdp
EXPNO 328
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081010
Time 19.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 8.00 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125315



BRUKER

Current Data Parameters
NAME wdp
EXPNO 329
PROCNO 1

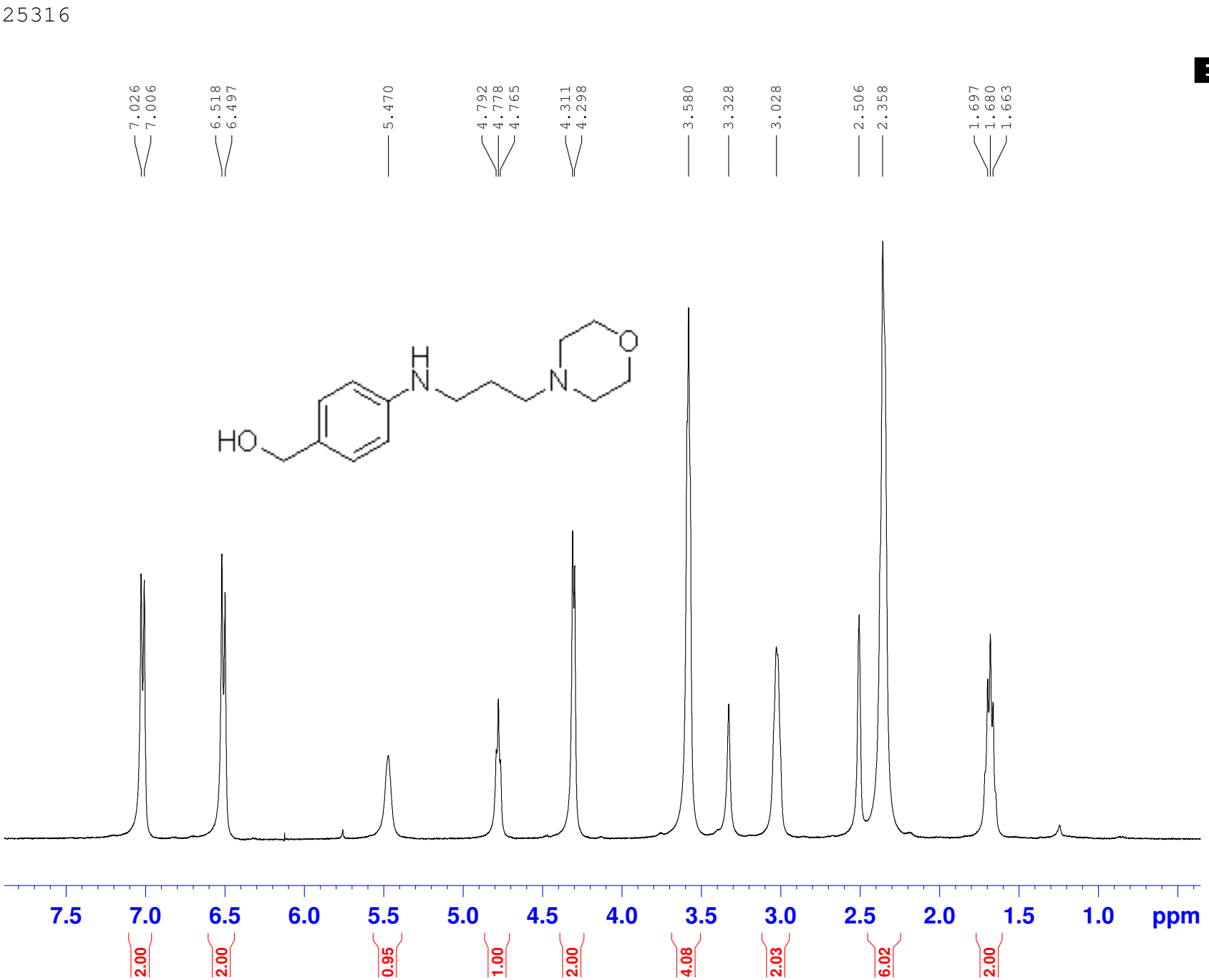
F2 - Acquisition Parameters
Date_ 20081010
Time 20.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 68
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 8.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 15.39 dB
PL13 15.50 dB
PL2 0.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

125316



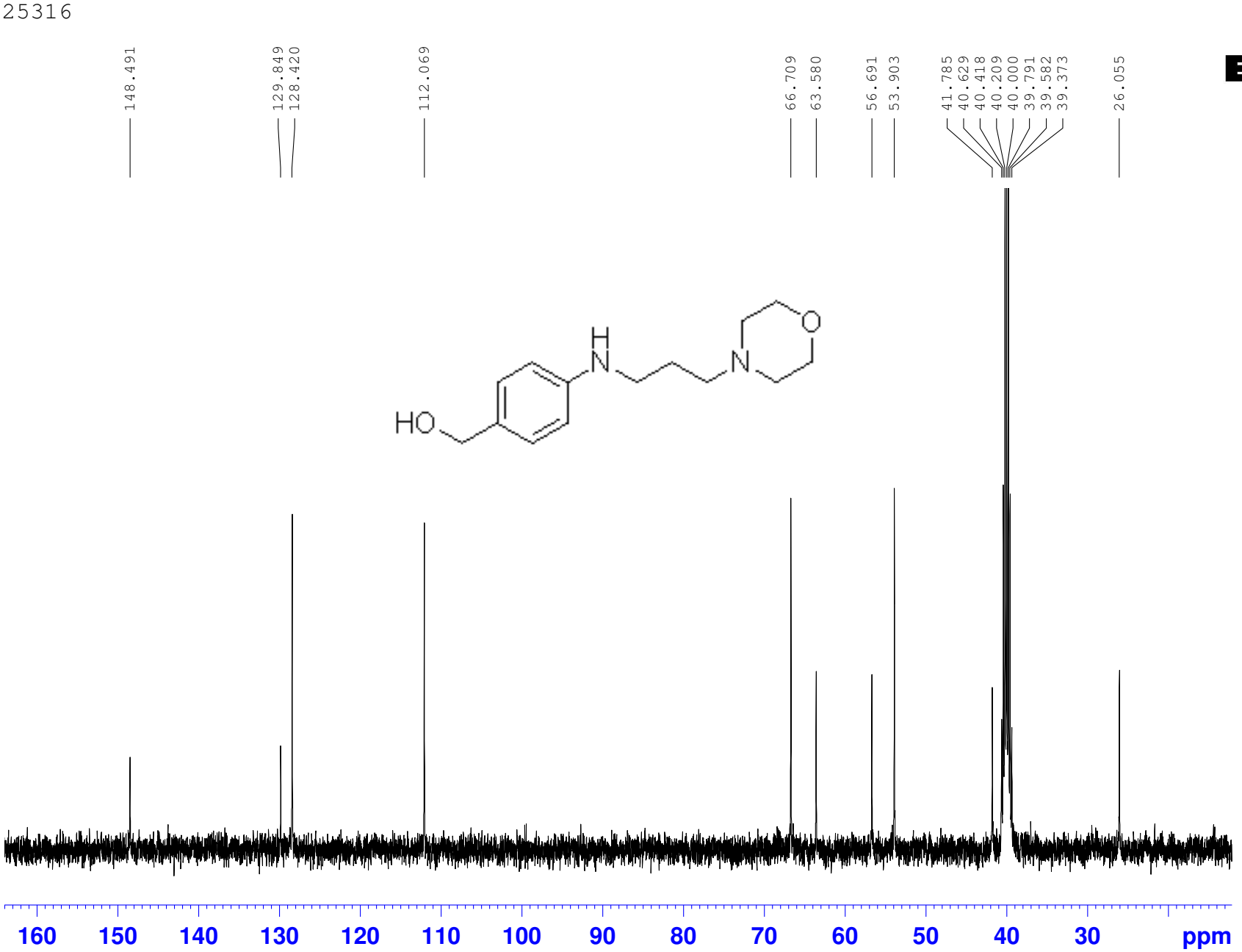
Current Data Parameters
NAME wdp
EXPNO 335
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081010
Time 21.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 8.00 usec
TE 299.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

125316




```
Current Data Parameters
NAME                  wdp
EXPNO                 334
PROCNO                  1

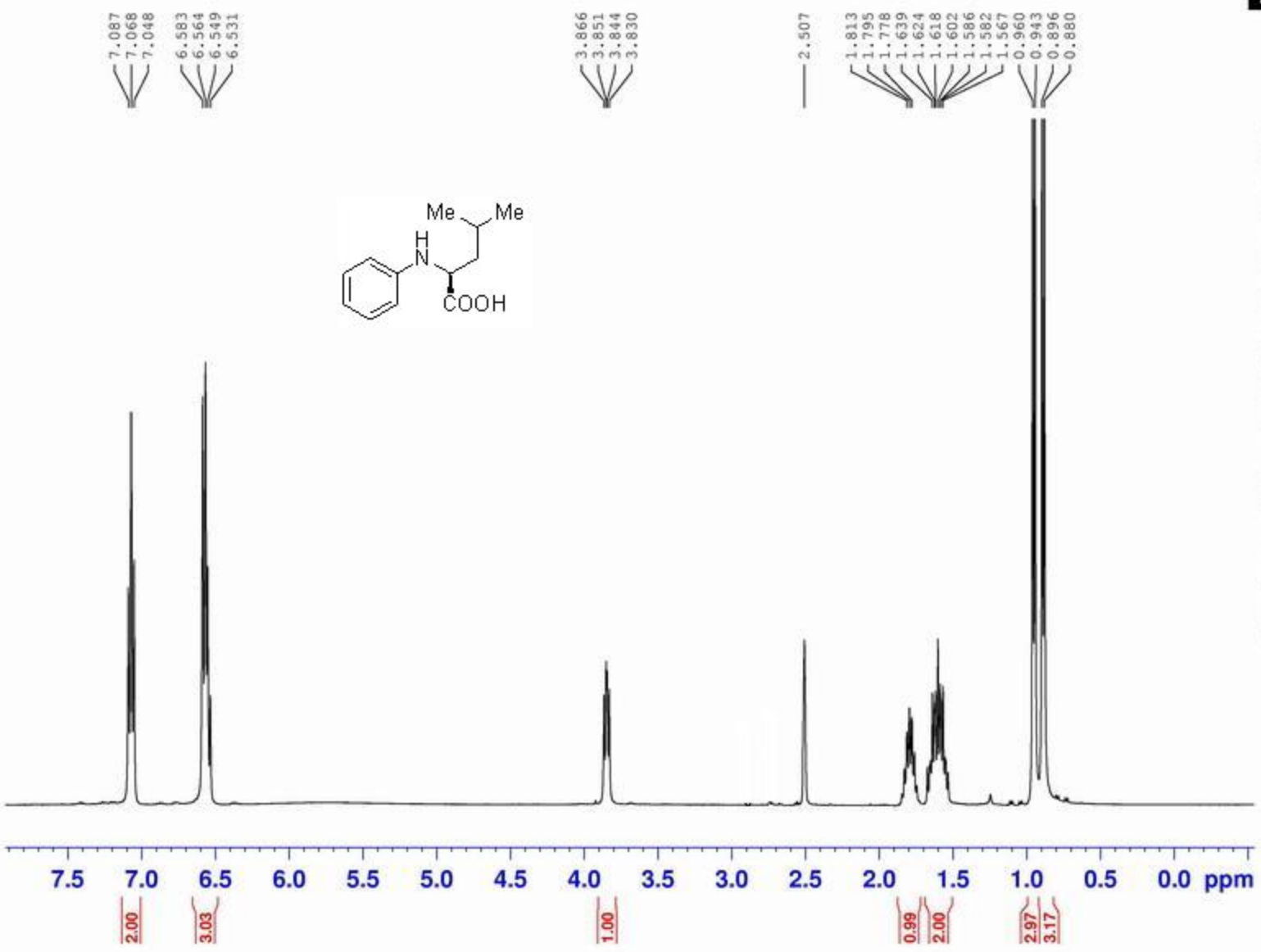
F2 - Acquisition Parameters
Date_           20081010
Time               21.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                    40
DS                     4
SWH            23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                 32768
DW                20.850 usec
DE                 8.00 usec
TE                 300.1 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                1.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL12               15.39 dB
PL13               15.50 dB
PL2                 0.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

125323



Current Data Parameters

NAME	wdp
EXPNO	340
PROCNO	1

F2 - Acquisition Parameters

Date_	20081012
Time	12.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	8.00 usec
TE	300.8 K
D1	1.00000000 sec
TD0	1

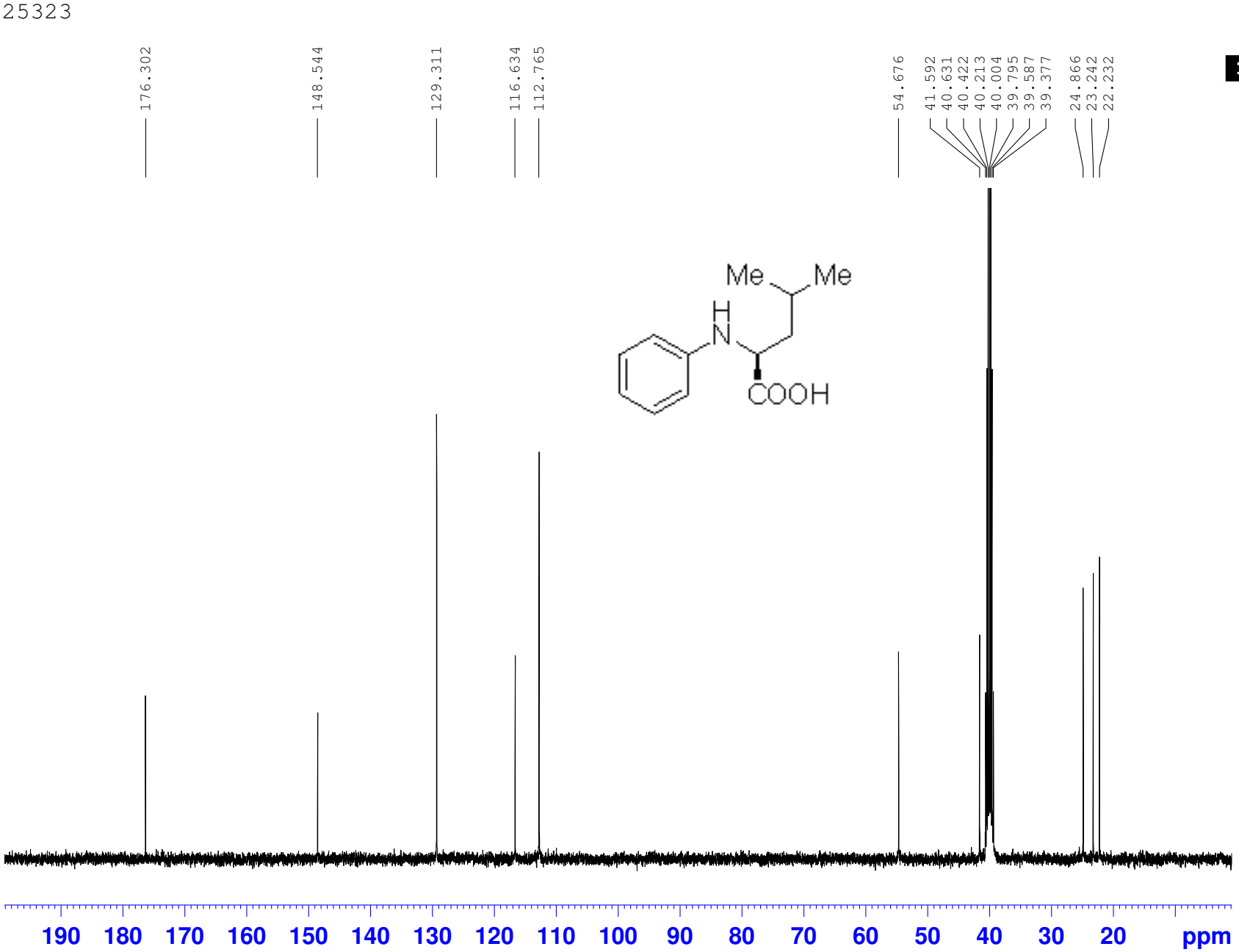
======== CHANNEL f1 ========

NUC1	1H
P1	13.60 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

125323




```
Current Data Parameters
NAME                  wdp
EXPNO                 341
PROCNO                  1

F2 - Acquisition Parameters
Date_           20081012
Time                13.08
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                     234
DS                       4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                   26008
DW                20.850 usec
DE                 8.00 usec
TE                 301.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.20 usec
PL1                 1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL12               15.39 dB
PL13               15.50 dB
PL2                 0.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```