Direct Synthesis of Pyridines and Quinolines by Coupling of γ-Aminoalcohols with Secondary Alcohols Liberating H_2 Catalyzed by Ruthenium Pincer Complexes

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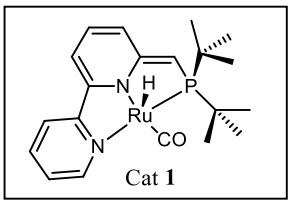
1. General Information

All experiments with metal complexes and phosphine ligands were carried out under an atmosphere of purified nitrogen in a Vacuum Atmospheres glovebox equipped with a MO 40-2 inert gas purifier or using standard Schlenk techniques. All solvents were reagent grade or better. All non-deuterated solvents were refluxed oversodium/benzophenoneketyl and distilled under argon atmosphere. Deuterated solvents were used as received. All solvents were degassed with argon and kept in the glove box over 4Å molecular sieves. Most of the chemicals used in the catalytic reactions were purified according to standard procedures (vaccum distillation).[1]All ^{1}H NMR (300 MHz), ^{13}C NMR (75 MHz) or^{31}P NMR spectra were recorded on a Bruker AMX-300 NMR spectrometer for a $CDCl_3$ solution and reported in ppm (δ). NMR spectroscopy abbreviations: br, broad; s,singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Mass spectra were recorded on MicromassPlatform LCZ 4000, using Electro Spray Ionization (ESI) mode. GC–MS was carried out on HP 6890 (flame ionization detector and thermal conductivity detector) and HP 5973 (MS detector) instruments equipped with a 30 m column (Restek 5MS, 0.32 mm internal diameter) with a 5% phenylmethylsiliconecoating (0.25 mm) and helium as carrier gas. GC analysis were carried out using a Carboxen 1000 column on a HP 690 series GC system or HP-5 cross linked 5% phenylmethylsilicone column (30m ×0.32mm × 0.25 µm film thickness, FID) on a HP 6890 series GC system using m-xylene (1 mmol) as an internal standard.

2. Experimental Procedures

Complexes: The ruthenium-pincer complexes**1** and **2**were prepared according to reported procedures from our group.[2]



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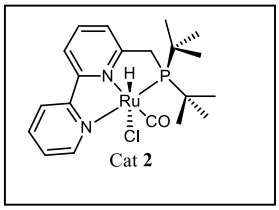
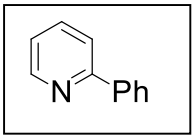


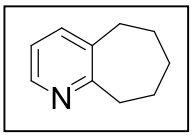
Cat **2**

General procedure for the synthesis of pyridines and quinolines:
Complex **2** (0.01 mmol), γ–aminoalcohol (2 mmol), secondary alcohol (4 mmol), KO^{t}Bu (2 mmol) and toluene+THF (2 mL+ 0.5 mL) were added to a Schlenk flask under an atmosphere of nitrogen in a Vacuum Atmospheres glovebox. The flask was equipped with a condenser and the solution was refluxed with stirring in an open system under argon at 135 °C oil bath temperature. The reaction products were analyzed by GC-MS. After cooling to room temperature, the reaction mixture was extracted with dichloromethane and filtered through celite. Evaporation of the solvent gave the crude product which was further purified by column chromatography using silica geland 5-20% ethyl acetate/ hexane as eluant.

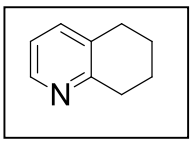
3. Spectroscopic characterization of the pyridines and quinolines:



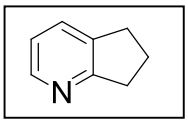
2-phenylpyridine:[31]H NMR ($CDCl_3$, 300 MHz) δ 8.7 (d, *J* =4.8 Hz, 1H), 8.03-7.99 (m, 2H), 7.70 (d,*J* =3.42 Hz, 2H), 7.42-7.32 (m, 3H), 7.51-7.39 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 157.44, 149.69, 139.41, 136.77, 128.99, 128.78, 126.93, 122.12, 120.57.



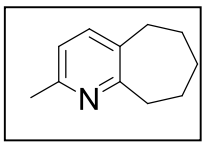
6,7,8,9-tetrahydro-5H-cyclohepta[b]pyridine:[41]H NMR ($CDCl_3$, 300 MHz) δ 8.24 (dd, *J*=4.56, 1.23 Hz, 1H), 7.32(d, *J*=6.75 Hz, 1H), 6.99-6.95 (m, 1H), 3.02-2.98 (m, 2H), 2.74-2.71(m, 2H),1.88-1.79(m, 2H), 1.69-1.58(m, 4H), ^{13}C NMR ($CDCl_3$, 75 MHz) δ 163.32, 146.18, 138.14, 136.43, 121.22, 39.53, 35.38, 32.58, 27.98, 26.51.



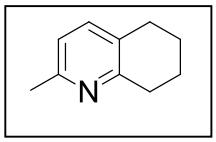
5,6,7,8-tetrahydroquinoline:[51]H NMR ($CDCl_3$, 300 MHz) δ 8.31 (d, *J* =3.9 Hz, 1H), 7.32 (d, *J*=7.5 Hz, 1H), 7.01-6.97 (m, 1H),2.90 (t, *J*=6.39 Hz, 2H), 2.74 (t, *J*=6.27 Hz, 2H), 1.9-1.78 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 157.46, 146.84, 136.84, 132.36, 120.95, 32.58, 28.85, 23.16, 22.77.



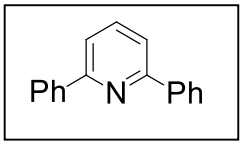
6,7-dihydro-5H-cyclopenta[b]pyridine:[61]H NMR ($CDCl_3$, 300 MHz) δ: 8.25 (d, *J* = 4.77 Hz, 1H), 7.39 (d, J = 7.56 Hz, 1H), 6.96-6.9 (m, 1H), 2.93 (t, *J* = 7.59 Hz, 2H), 2.85 (t, *J* = 7.29 Hz, 2H), 2.09-1.98(m, 2H) ^{13}C NMR ($CDCl_3$, 75 MHz) δ 165.51, 147.33, 136.79, 131.95, 120.86, 34.18, 30.66, 23.01.



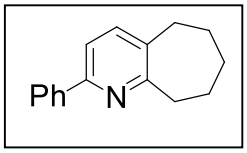
2-methyl-6,7,8,9-tetrahydro-5H-cyclohepta[b]pyridine:[71]H NMR ($CDCl_3$, 300 MHz) δ: 7.21(d, *J*=7.5 Hz, 1H), 6.82 (d, *J*=7.5 Hz, 1H), 2.98-2.95(m, 2H), 2.70-2.67(m, 2H), 2.45(s, 3H), 1.86-1.78 (m, 2H), 1.68-1.59(m, 4H) ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.53, 154.44, 136.92, 134.82, 120.5, 39.5, 34.9, 32.61, 28.17, 26.64, 24.05.



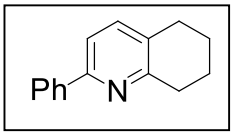
2-methyl-5,6,7,8-tetrahydroquinoline:[81]H NMR ($CDCl_3$, 300 MHz) δ:7.23 (d, *J* = 7.77 Hz, 1H), 6.87 (d, *J* = 7.74 Hz, 1H), 2.87(t, *J* = 6.27 Hz, 2H), 2.70(t, *J* = 6.33 Hz, 2H), 2.47(s, 3H), 1.9-1.73(m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.51, 155.12, 137.30, 129.08, 120.59, 32.63, 28.50, 24.22, 23.29, 22.91.



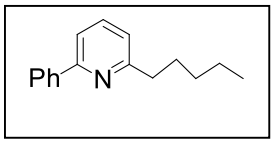
2,6-diphenylpyridine:[91]H NMR ($CDCl_3$, 300 MHz) δ:8.22 (d, J = 7.08 Hz, 4H), 7.84-7.79 (m, 1H), 7.72-7.02 (m, 2H), 7.58-7.46 (m, 6H);^{13}C NMR ($CDCl_3$, 75 MHz) δ156.86, 139.55, 137.57, 129.07, 128.78, 127.07, 118.71.



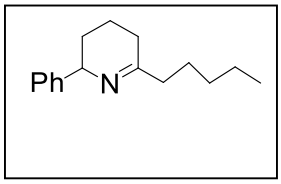
2-phenyl-6,7,8,9-tetrahydro-5H-cyclohepta[b]pyridine: ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.99(d, *J*= 7.2 Hz, 2H), 7.48-7.34 (m, 5H), 3.16-3.13(m, 2H), 2.83-2.79 (m, 2H), 1.91-1.87 (m, 2H), 1.9-1.70 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ163.21, 154.10, 139.90, 137.27, 136.62, 128.70, 128.37, 126.86, 117.95, 39.83, 35.10, 32.67, 28.20, 26.75.



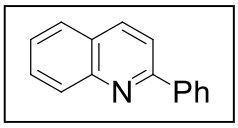
2-phenyl-5,6,7,8-tetrahydroquinoline:[10] ^{1}H NMR ($CDCl_3$, 300 MHz) δ:7.97–7.94 (m, 2H), 7.48–7.35 (m, 5H), 3.00 (t, *J* = 6.27 Hz, 2H), 2.80 (t, *J* = 6.36 Hz, 2H) 1.98–1.90 (m, 2H), 1.89–1.80 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 157.4, 154.9, 140.1, 137.6, 130.9, 128.8, 128.5, 127.0, 118.1, 33.1, 28.8, 23.4, 23.0.



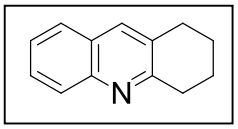
2-pentyl-6-phenylpyridine: ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 8.02-7.98(m, 2H), 7.65(t, *J*= 7.74 Hz, 1H), 7.53-7.39 (m, 4H), 7.08(d, *J*= 7.77 Hz, 1H), 2.86 (t, *J*= 7.65 Hz, 2H), 1.84-1.79 (m, 2H), 1.39-1.37(m, 4H), 0.92(t, *J*= 6.96 Hz, 3H)^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.58, 156.93, 140.04, 136.92, 128.78, 127.14, 121.11, 117.82, 38.66, 31.79, 29.63, 22.73, 14.21.HRMS (ESI) calculated for$C_{16}H_{19}N$ $[M+H]^+$ 226.1590 Found 226.1588.



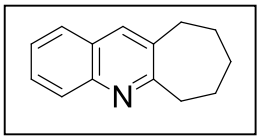
6-pentyl-2-phenyl-2,3,4,5-tetrahydropyridine: ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.81-7.78(m, 2H), 7.38-7.36 (m, 3H), 3.59-3,49 (m, 1H), 2.72-2.42(m, 2H), 1.94-1.47(m, 5H),1.38-1.19(m, 7H) 0.92(t, *J*= 6.78 Hz, 3H);^{13}C NMR ($CDCl_3$, 75 MHz) δ 164.31, 140.51, 129.43, 128.24, 126.17, 58.43, 37.88, 32.27, 27.32, 27.03, 26.20, 22.84, 19.14, 14.24.HRMS (ESI) calculated for$C_{16}H_{23}N$ $[M+H]^+$ 230.1892 Found 230.1891.



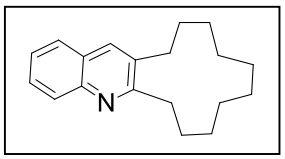
2-phenylquinoline:[11]^{1}H NMR ($CDCl_3$, 300 MHz) δ:8.23-8.15 (m, 4 H), 7.89-7.70 (m, 3H), 7.56-7.44 (m, 4 H);^{13}C NMR ($CDCl_3$, 75 MHz) δ157.50, 148.40, 139.80, 136.94, 129.86, 129.81, 129.47, 128.99, 127.72, 127.60, 127.32, 126.43, 119.16.



1,2,3,4-tetrahydroacridine:[12]^{1}H NMR ($CDCl_3$, 300 MHz) δ:7.97 (d, J = 8.49 Hz, 1H) 7.75 (s, 1H), 7.67-7.56 (m, 2H); 7.40 (t, J = 7.41, 1.5 Hz, 1H), 3.11 (t, J = 6.39 Hz, 2H), 2.93 (t, J = 6.45 Hz, 2H), 2.93-2.01 (m, 2H), 1.9-1.82 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.33, 146.67, 134.99, 130.98, 128.50,128.33, 127.25, 126.93, 125.55, 33.63, 29.29, 23.28, 22.96.

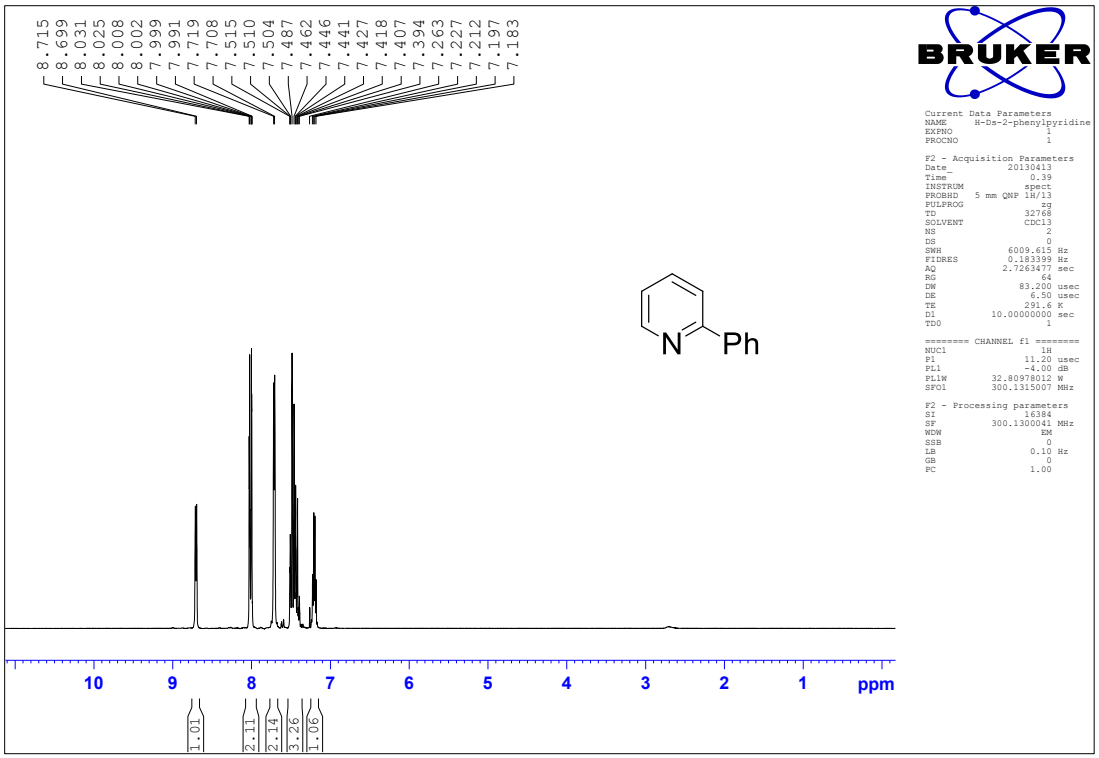


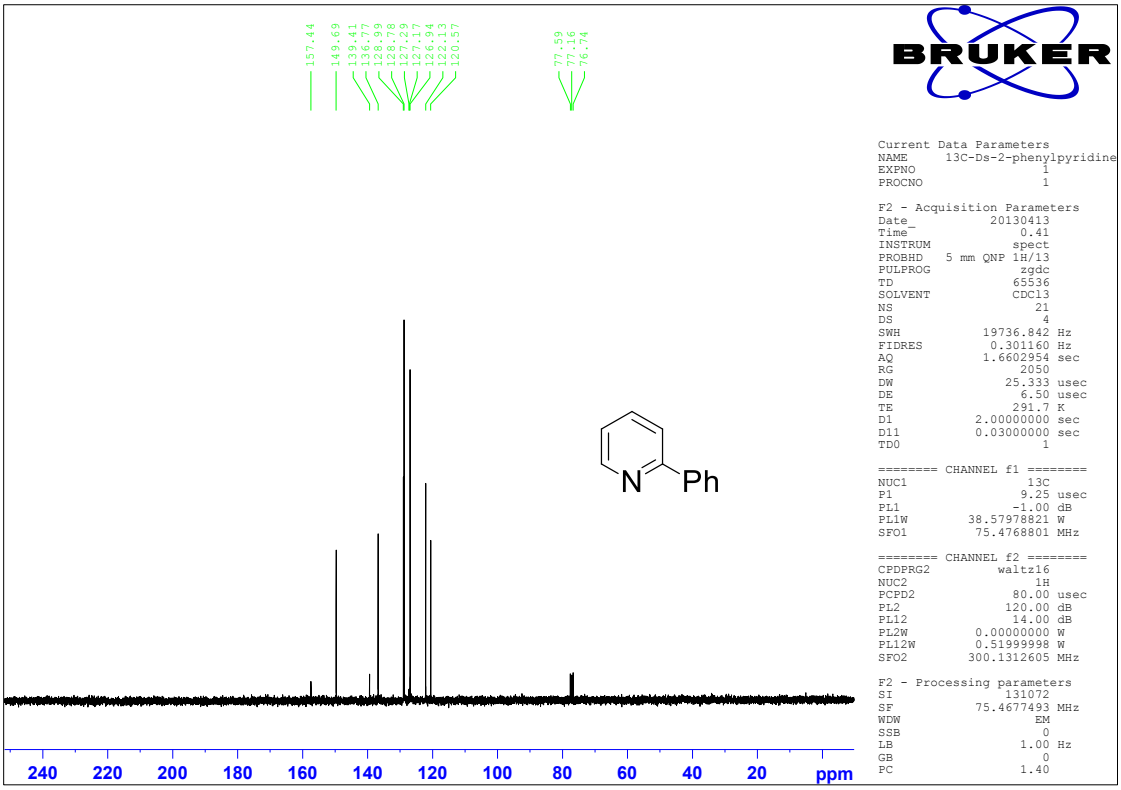
7,8,9,10-tetrahydro-6H-cyclohepta[b]quinoline:[13]^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.98 (d, J = 8.4 Hz, 1H) 7.74 (s, 1H), 7.67-7.56 (m, 2H); 7.43-7.28 (m, 1H), 3.20-3.16 (m, 2H), 2.90-2.86 (m, 2H), 1.89-1.68 (m, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 164.67, 146.29, 136.50, 134.56, 128.49, 128.45, 127.38, 126.83, 125.74, 40.1, 35.45, 32.25, 28.88, 27.04.

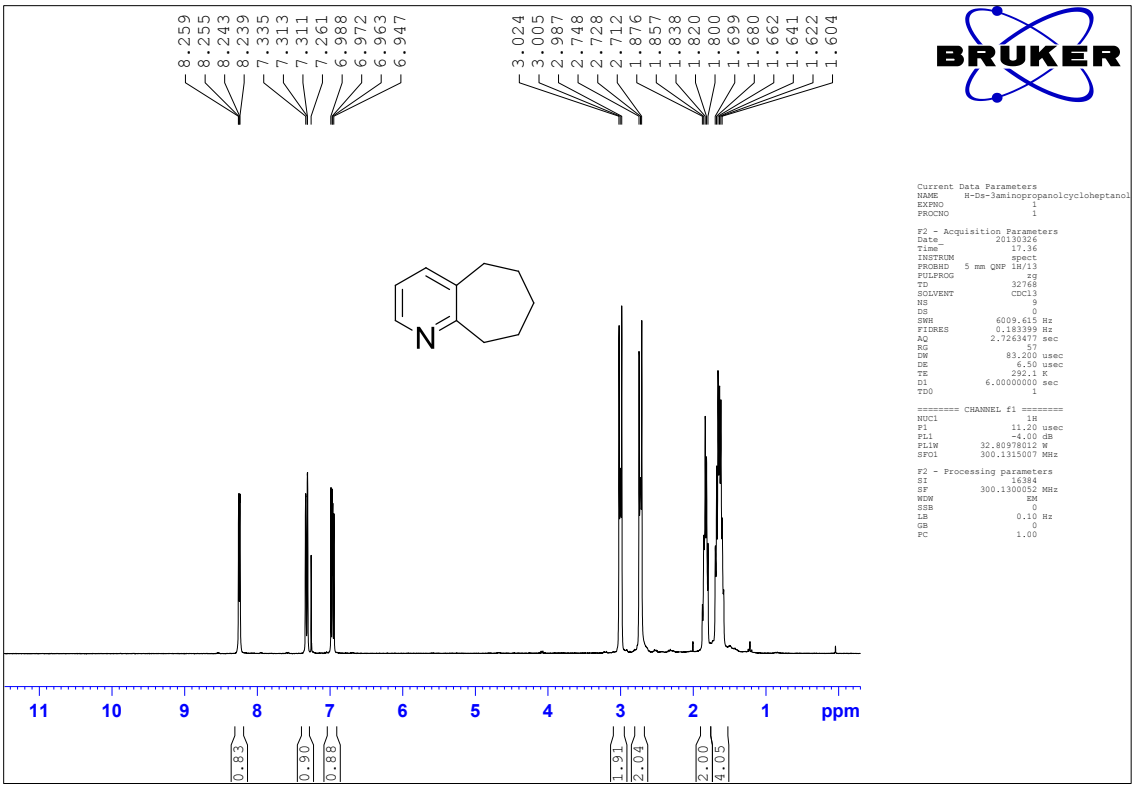


6,7,8,9,10,11,12,13,14,15-decacyclododeca[b]quinoline:[14]^{1}H NMR ($CDCl_3$, 300 MHz) δ: 8.00 (d, *J*= 8.46 Hz, 1H), 7.87 (s, 1H), 7.69 (d, *J*= 8.07 Hz, 1H), 7.60 (t,*J*= 7.65 Hz, 1H), 7.42 (t, *J*= 7.38 Hz, 1H), 3.00 (t, *J*= 7.68 Hz, 2H), 2.80 (t, *J*= 7.77 Hz, 2H), 2.00–1.91 (m, 2H), 1.83-1.74 (m, 2H), 1.56-1.41 (m, 12H),^{13}C NMR ($CDCl_3$, 75 MHz) δ162.76, 146.65, 135.86, 134.93, 128.49, 128.46, 127.24, 126.90, 125.62, 32.80, 29.83, 29.74, 28.59, 26.81, 26.57, 26.11, 25.54, 23.18, 23.09.

4.Figures reproducing ^{1}H and ^{13}C NMR spectra



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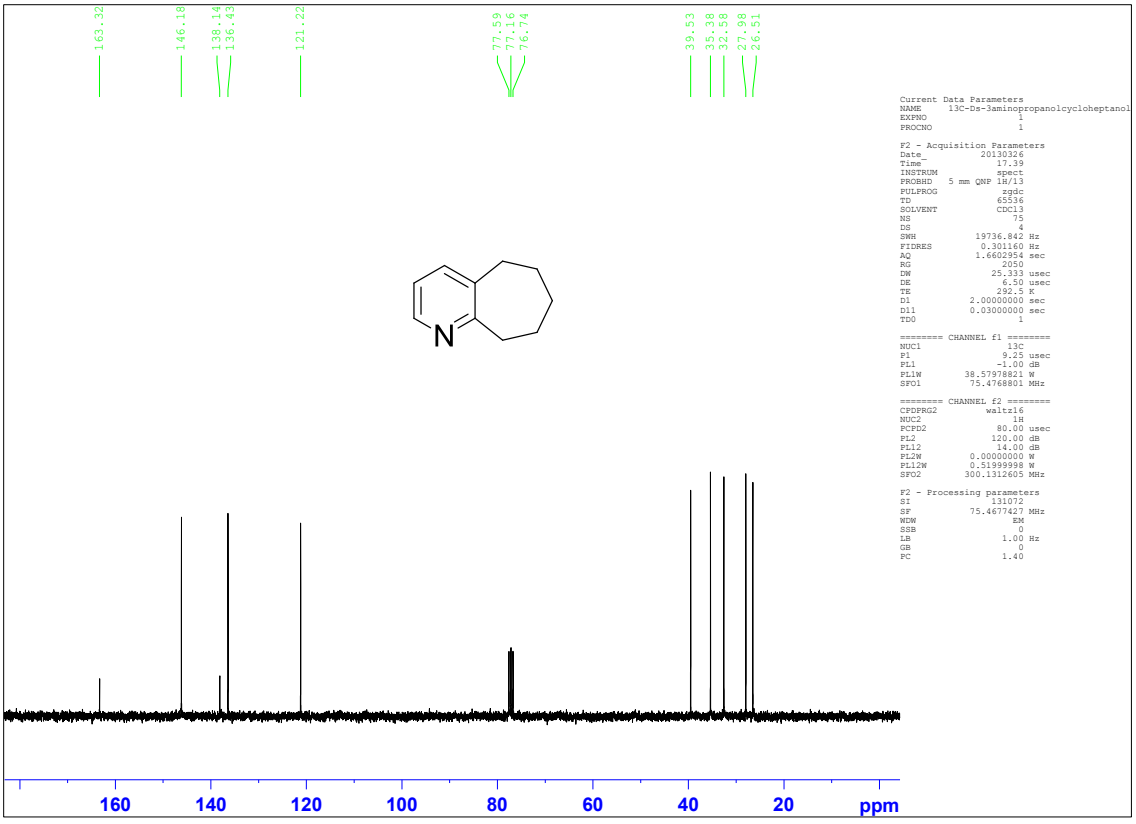
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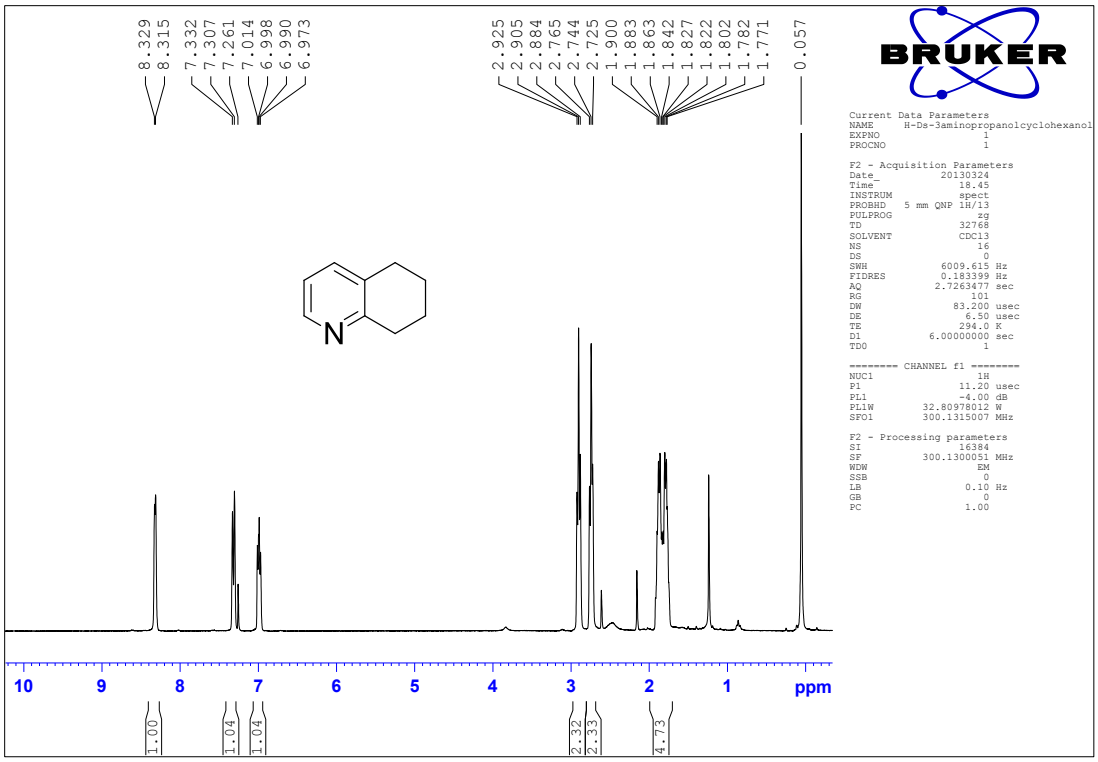
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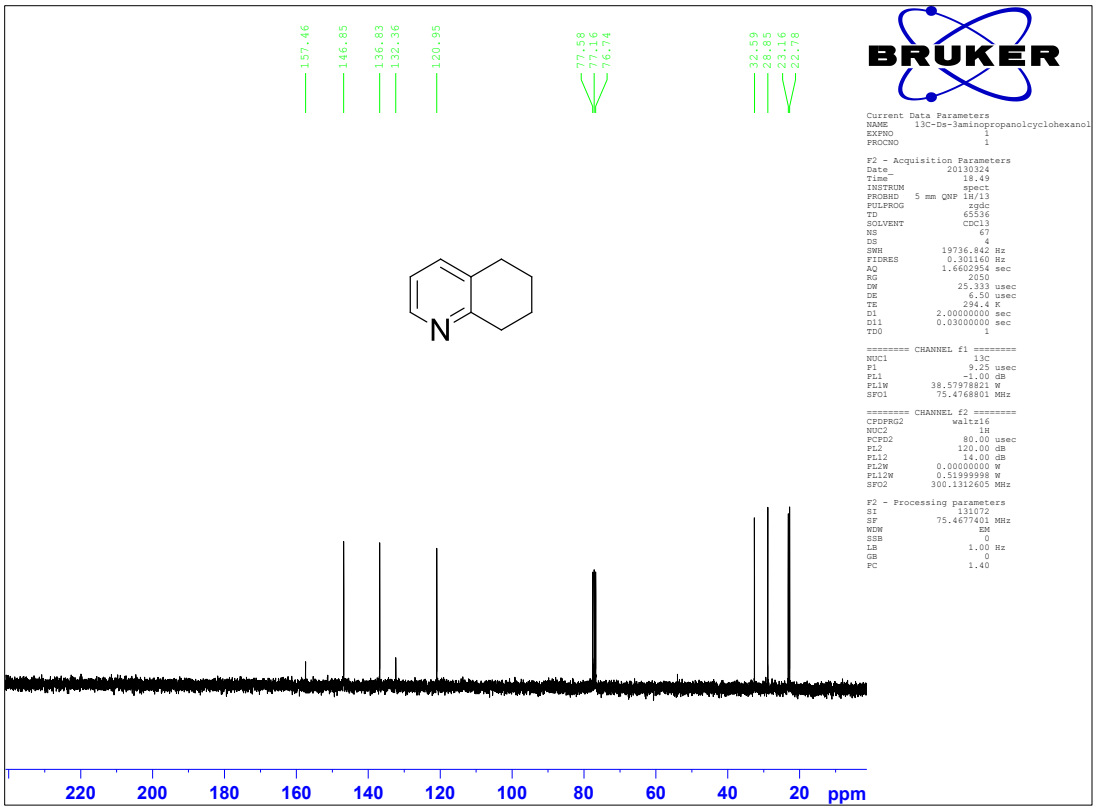
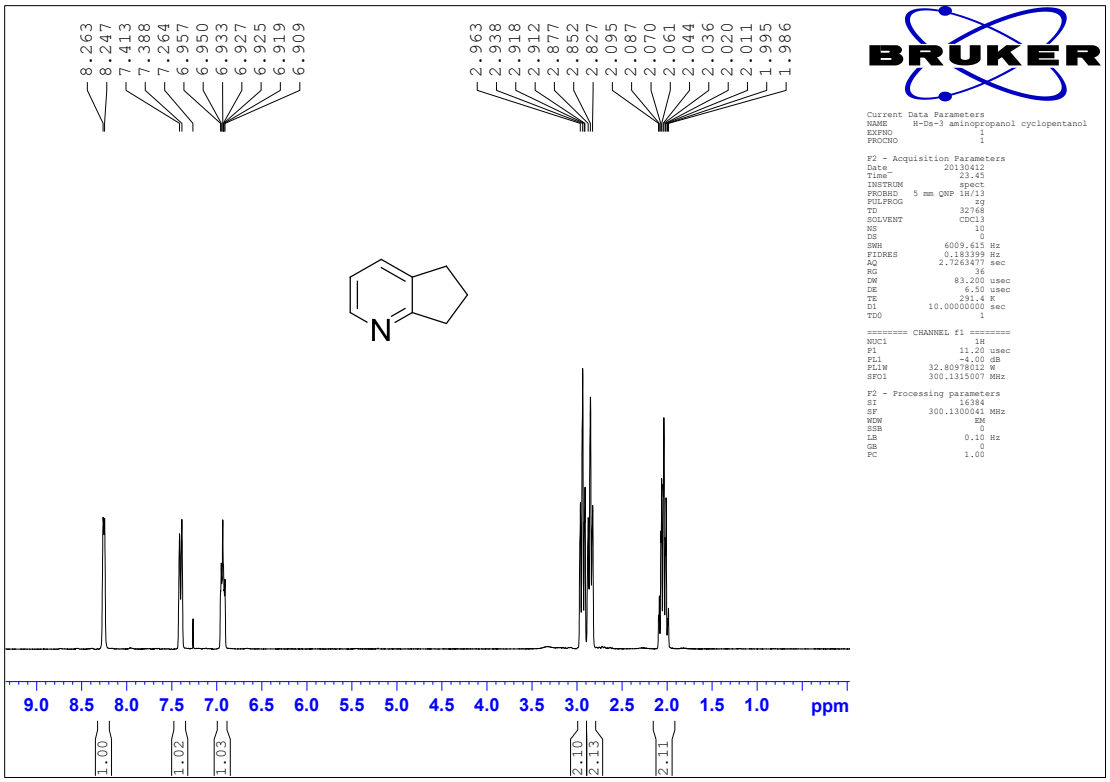
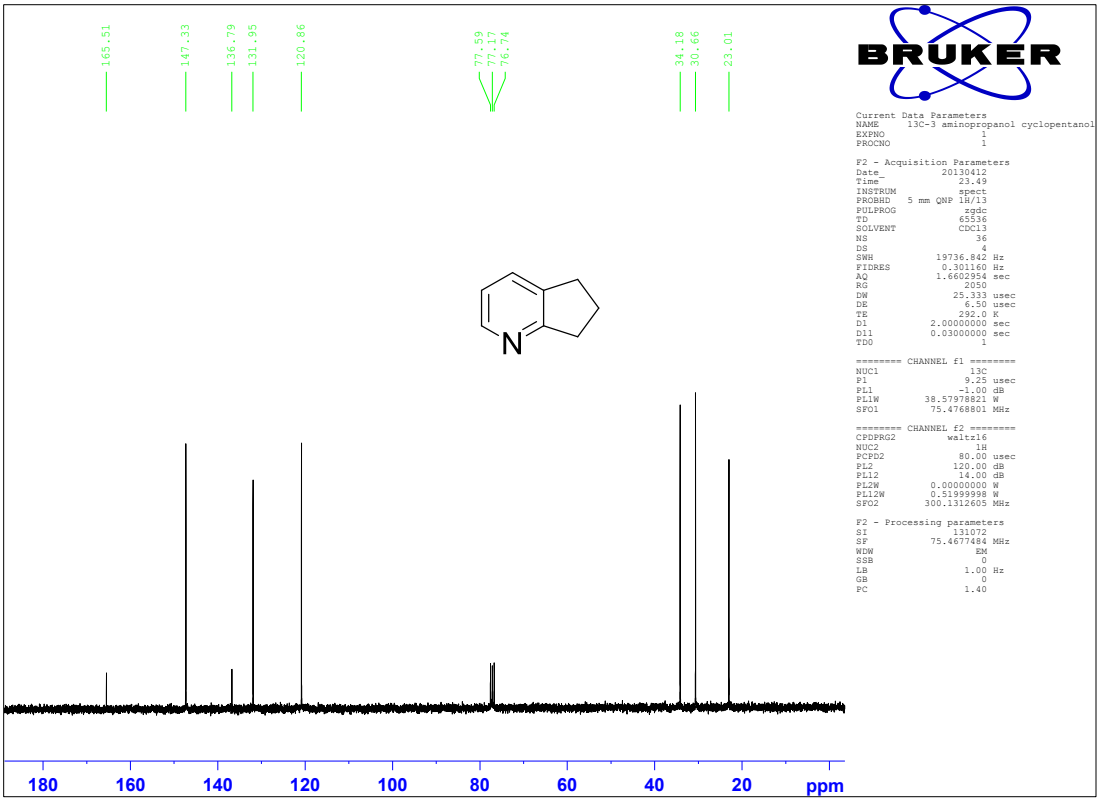
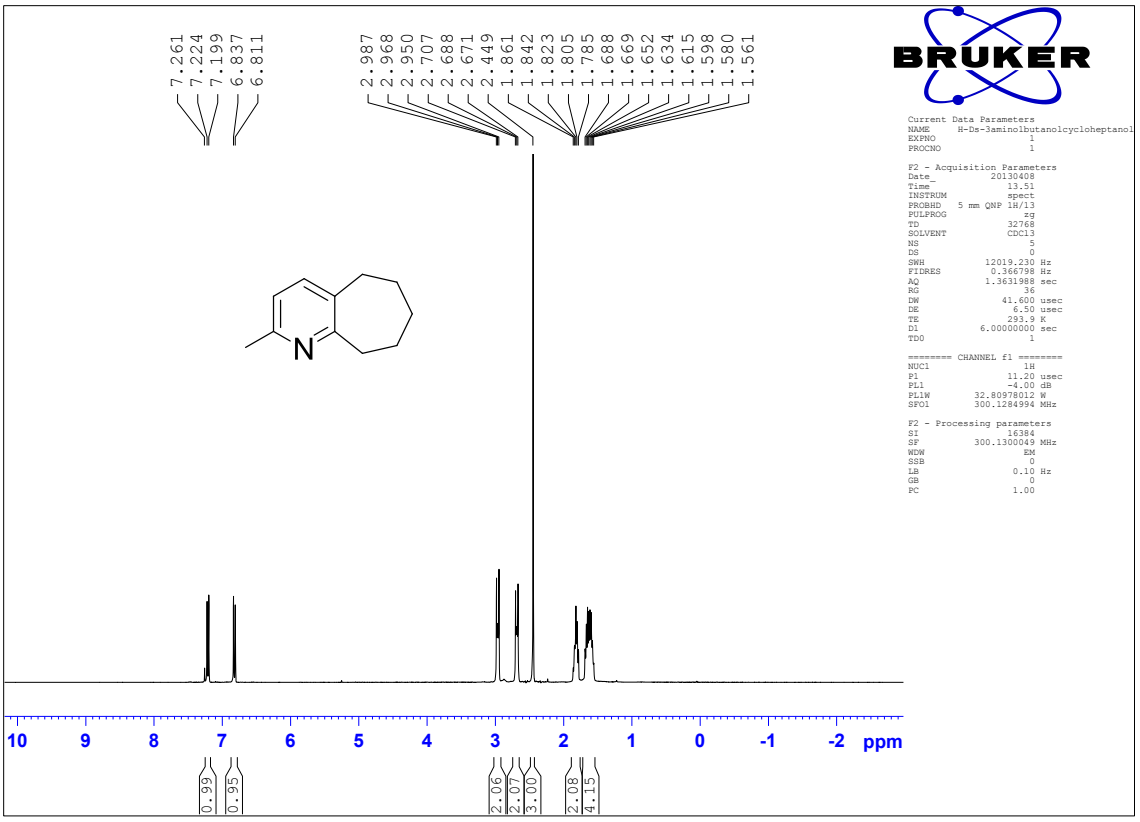
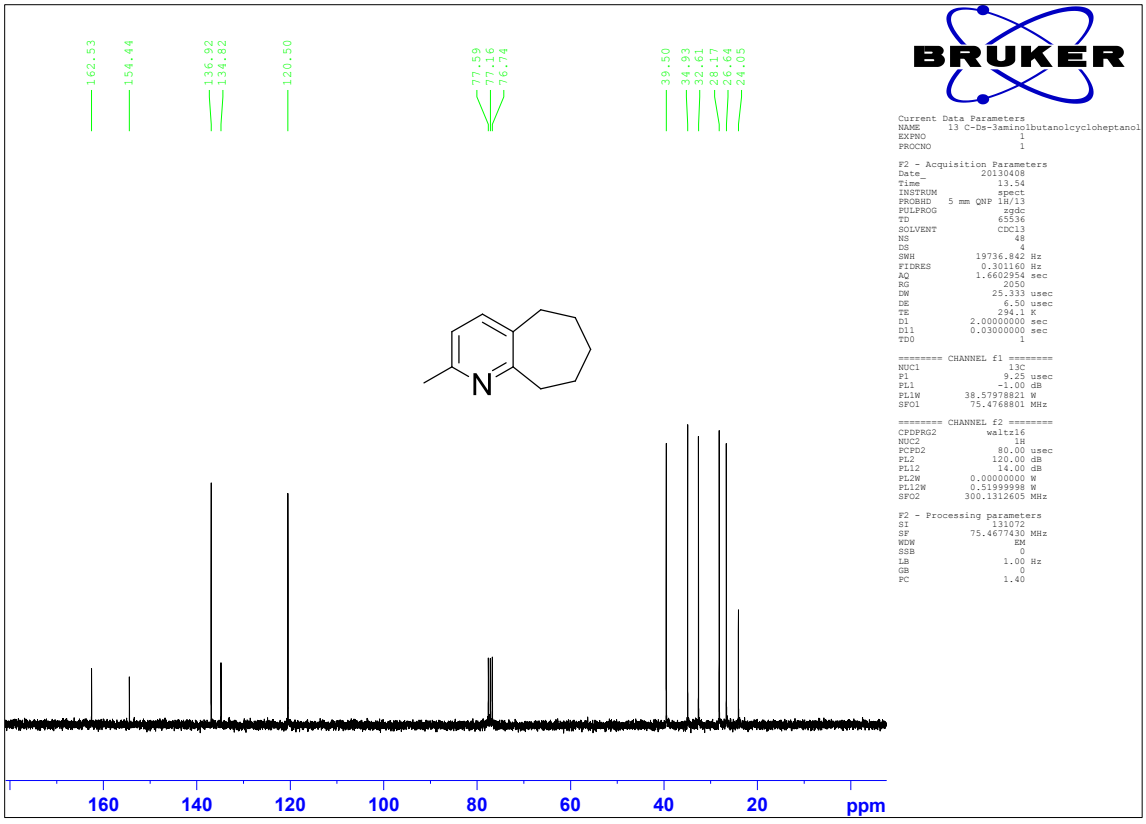
PC 1.00

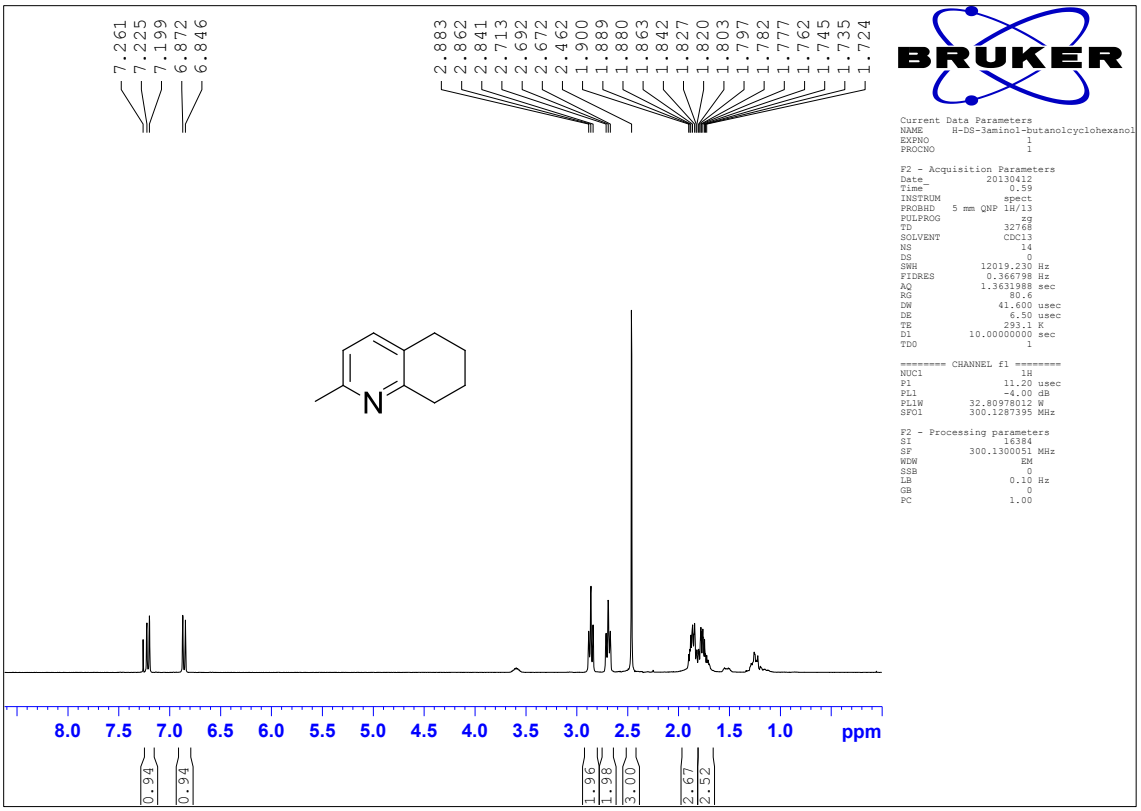

157.46
146.85
136.83
132.36
120.95
77.58
77.16
76.74
32.59
28.85
23.16
22.78
N
220
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME 13C-Ds-3aminopropanolcyclohexanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130324
Time 18.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDC13
NS 67
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PL1W 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677401 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

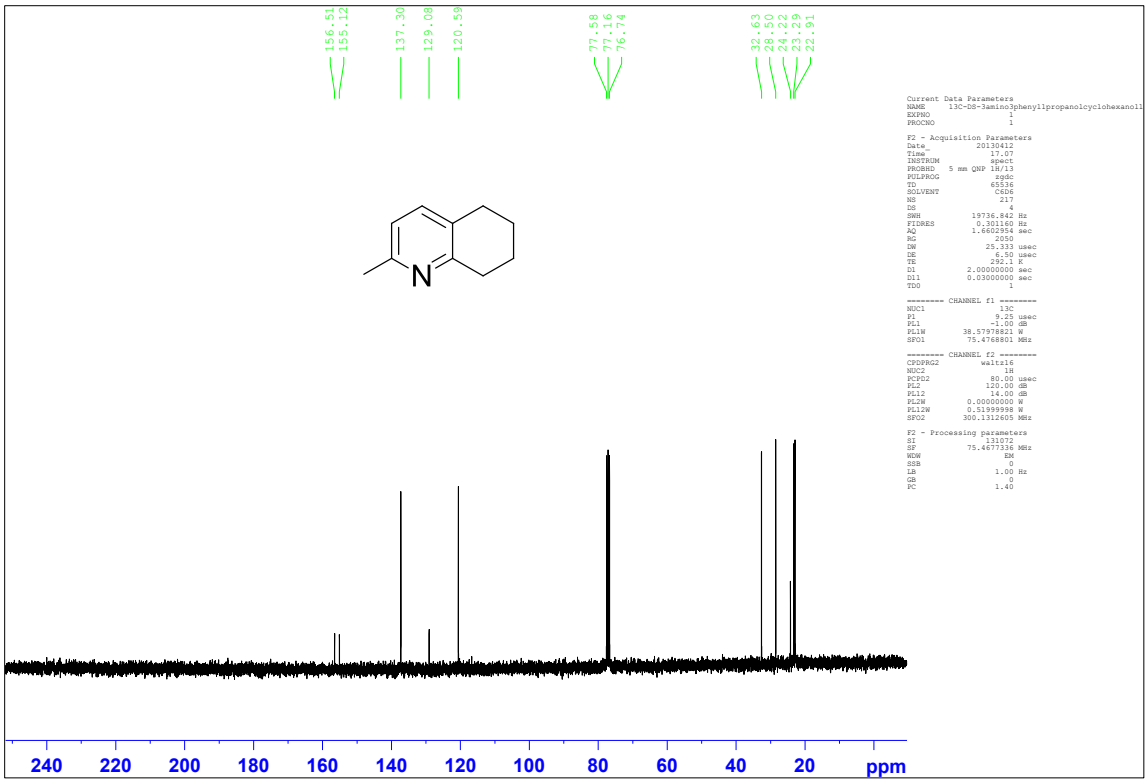

8.263
8.247
7.413
7.388
7.264
6.957
6.950
6.933
6.927
6.925
6.919
6.909
2.963
2.938
2.918
2.912
2.877
2.852
2.827
2.095
2.087
2.070
2.061
2.044
2.036
2.020
2.011
1.995
1.986
N
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
1.02
1.03
2.10
2.13
2.11
BRUKER
Current Data Parameters
NAME H-Ds-3 aminopropanol cyclopentanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130412
Time 23.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 10
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 36
DW 83.200 usec
DE 6.50 usec
TE 291.4 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PL1W 32.80978012 W
SFO1 300.1315007 MHz
F2 - Processing parameters
SI 16384
SF 300.1300041 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

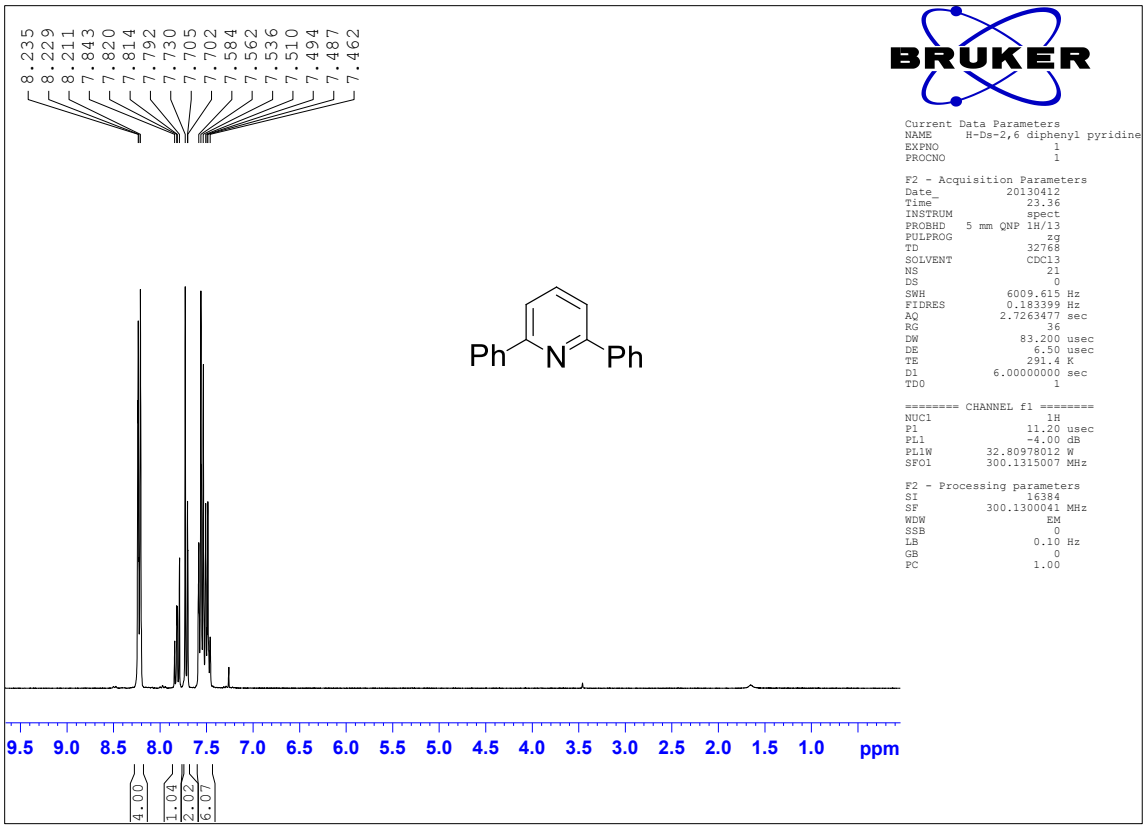

165.51
147.33
136.79
131.95
120.86
77.59
77.17
76.74
34.18
30.66
23.01
N
180 160 140 120 100 80 60 40 20 ppm
BRUKER
Current Data Parameters
NAME 13C-3 aminopropanol cyclopentanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130412
Time 23.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 36
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 292.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PL1W 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677484 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

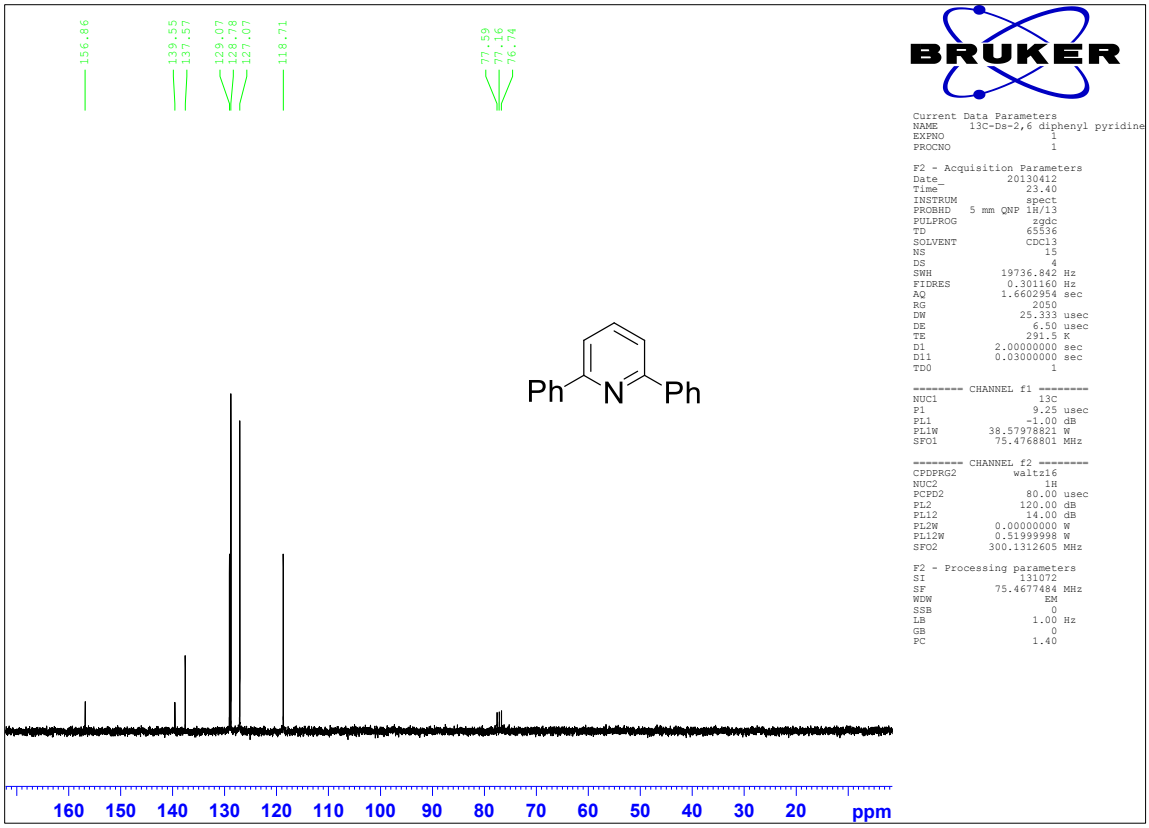

7.261
7.224
7.199
6.837
6.811
2.987
2.968
2.950
2.707
2.688
2.671
2.449
1.861
1.842
1.823
1.805
1.785
1.688
1.669
1.652
1.634
1.615
1.598
1.580
1.561
N
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
0.99
0.95
2.06
2.07
3.00
2.08
4.15
BRUKER
Current Data Parameters
NAME H-Ds-3aminolbutanolcycloheptanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130408
Time 13.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 36
DW 41.600 usec
DE 6.50 usec
TE 293.9 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PL1W 32.80978012 W
SFO1 300.1284994 MHz
F2 - Processing parameters
SI 16384
SF 300.1300049 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

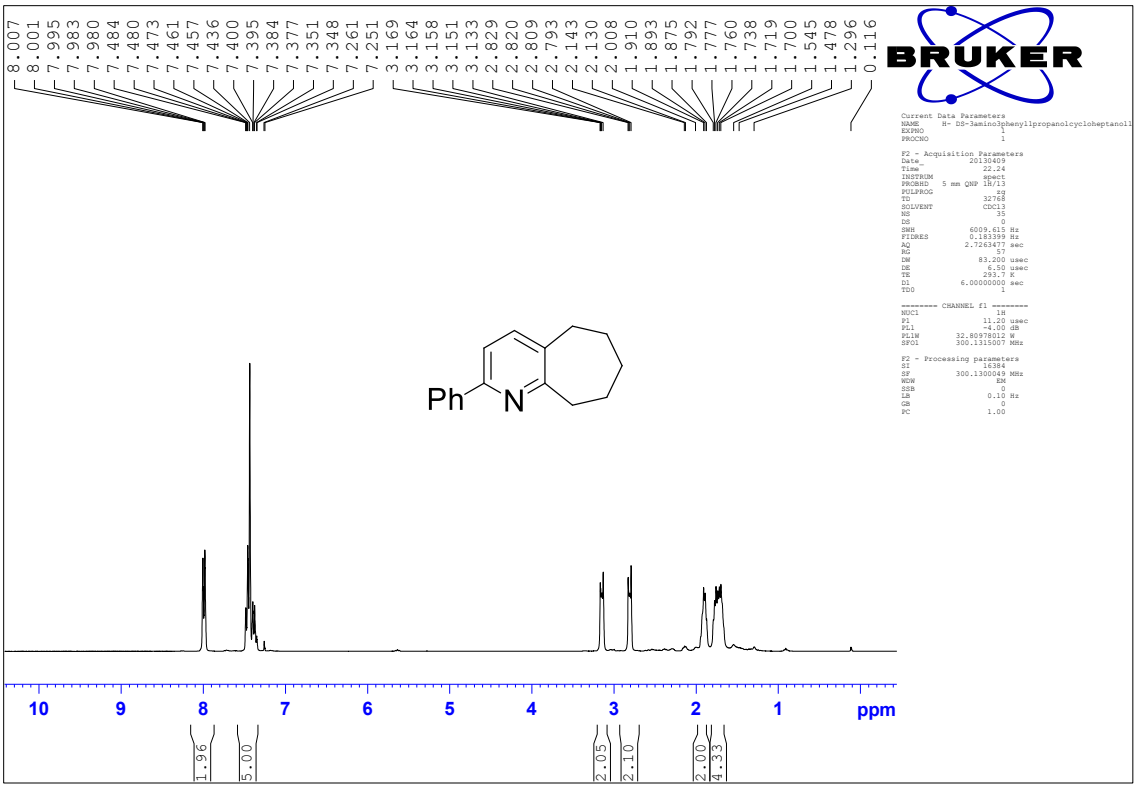

162.53
154.44
136.92
134.82
120.50
77.59
77.16
76.74
39.50
34.93
32.61
28.17
26.64
24.05
N
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME 13 C-Ds-3aminolbutanolcycloheptanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130408
Time 13.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 48
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PL1W 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677430 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

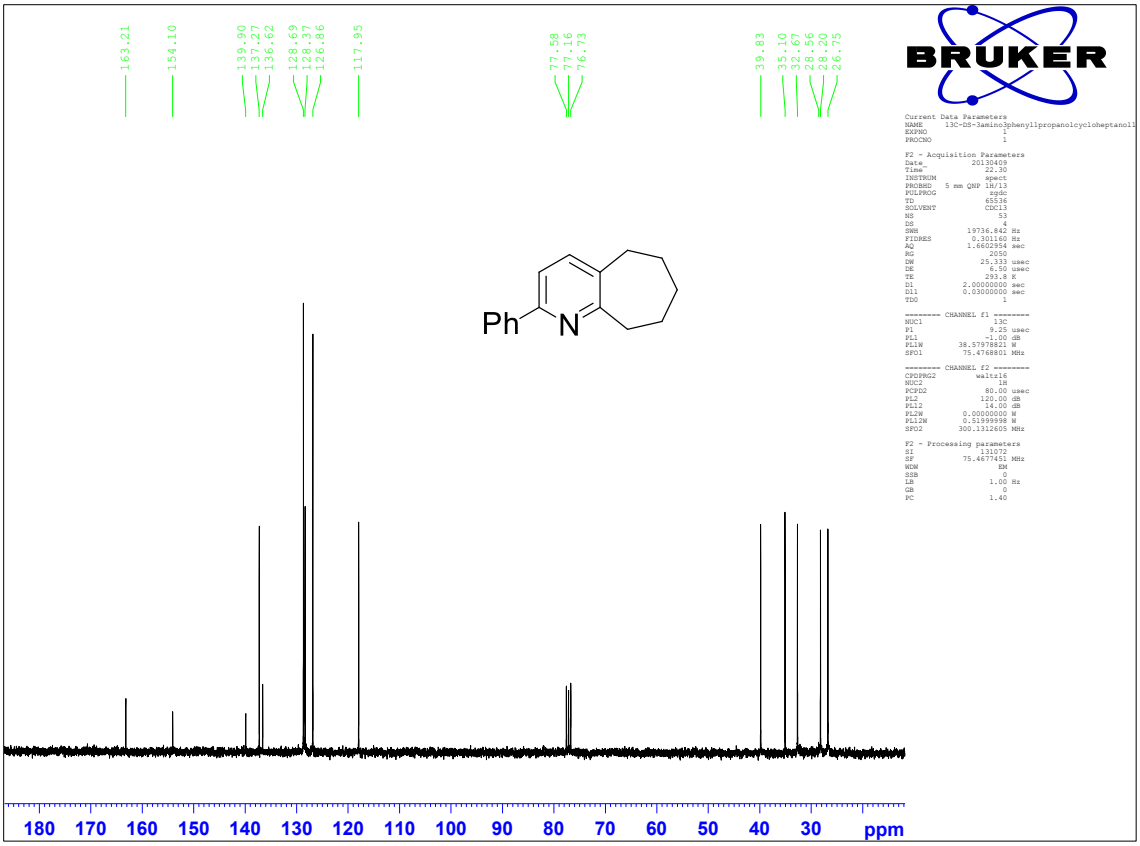

7.261
7.225
7.199
6.872
6.846
2.883
2.862
2.841
2.713
2.692
2.672
2.462
2.900
1.889
1.880
1.863
1.842
1.827
1.820
1.803
1.797
1.782
1.777
1.762
1.745
1.735
1.724
BRUKER
Current Data Parameters
NAME H-DS-3aminol-butanolcyclohexanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130412
Time 0.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 14
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 80.6
DW 41.600 usec
DE 6.50 usec
TE 293.1 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PL1W 32.80978012 W
SFO1 300.1287395 MHz
F2 - Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
N
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.94
0.94
1.96
1.98
3.00
2.67
2.52

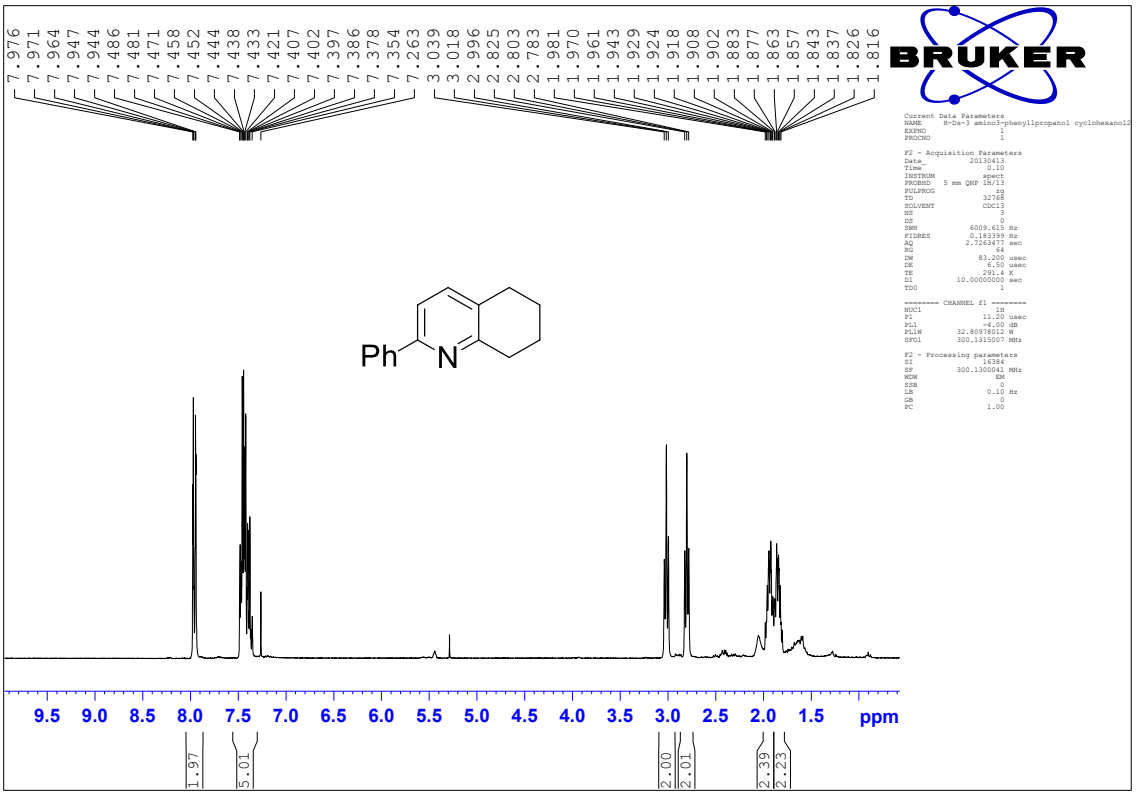

156.51
155.12
137.30
129.08
120.59
77.58
77.16
76.74
32.63
28.50
24.22
23.29
22.91
Current Data Parameters
NAME 13C-DS-3amino3phenyllpropanolcyclohexanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130412
Time 17.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT C6D6
NS 217
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 292.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PL1W 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677336 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
240 220 200 180 160 140 120 100 80 60 40 20 ppm

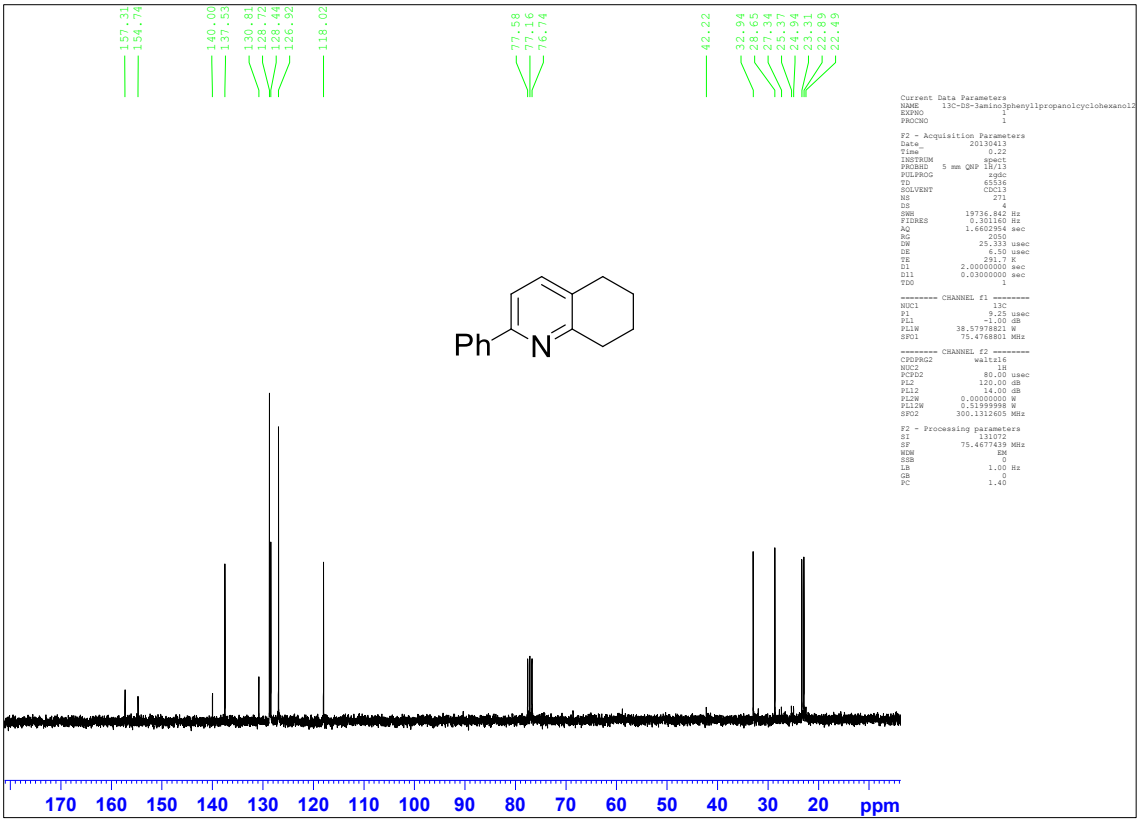

8.235
8.229
8.211
7.843
7.820
7.814
7.792
7.730
7.705
7.702
7.584
7.562
7.536
7.510
7.494
7.487
7.462
Ph
N
Ph
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
4.00
1.04
2.02
6.07
BRUKER
Current Data Parameters
NAME H-Ds-2,6 diphenyl pyridine
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130412
Time 23.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 21
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 36
DW 83.200 usec
DE 6.50 usec
TE 291.4 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PL1W 32.80978012 W
SFO1 300.1315007 MHz
F2 - Processing parameters
SI 16384
SF 300.1300041 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

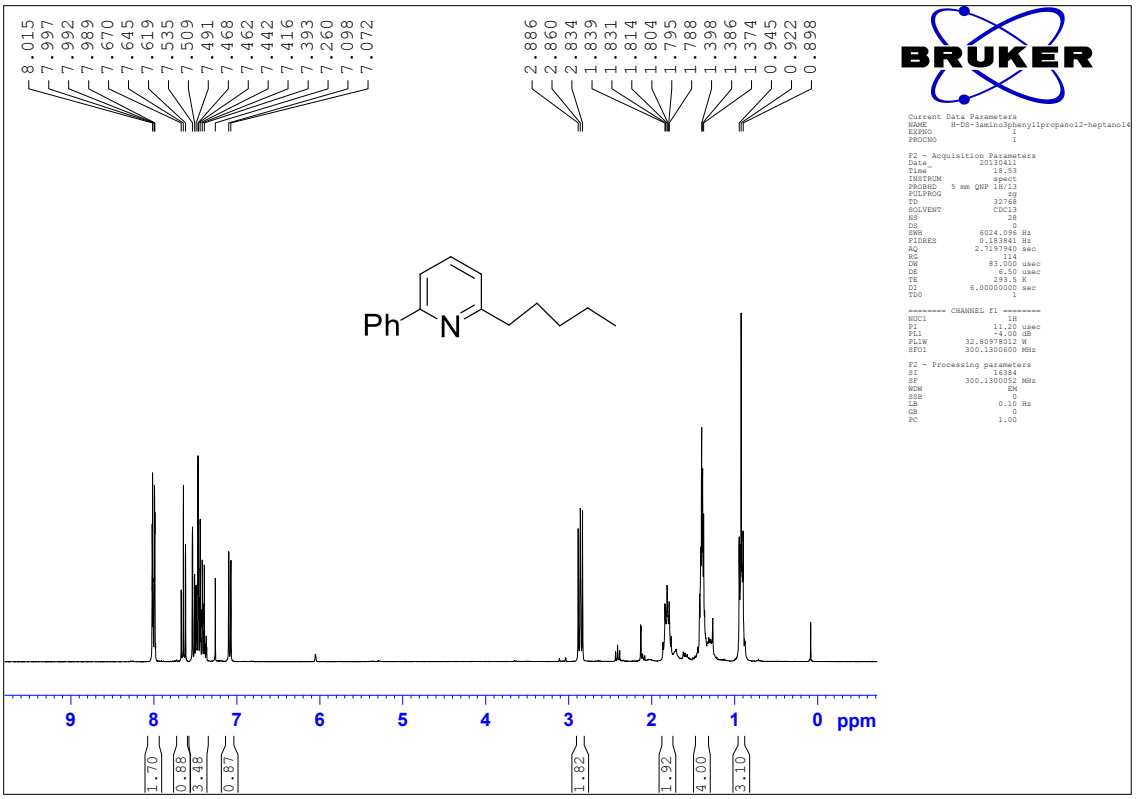

156.86
139.55
137.57
129.07
128.78
127.07
118.71
77.59
77.16
76.74
Ph
N
Ph
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
BRUKER
Current Data Parameters
NAME 13C-Ds-2,6 diphenyl pyridine
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130412
Time 23.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 15
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 291.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PL1W 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677484 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

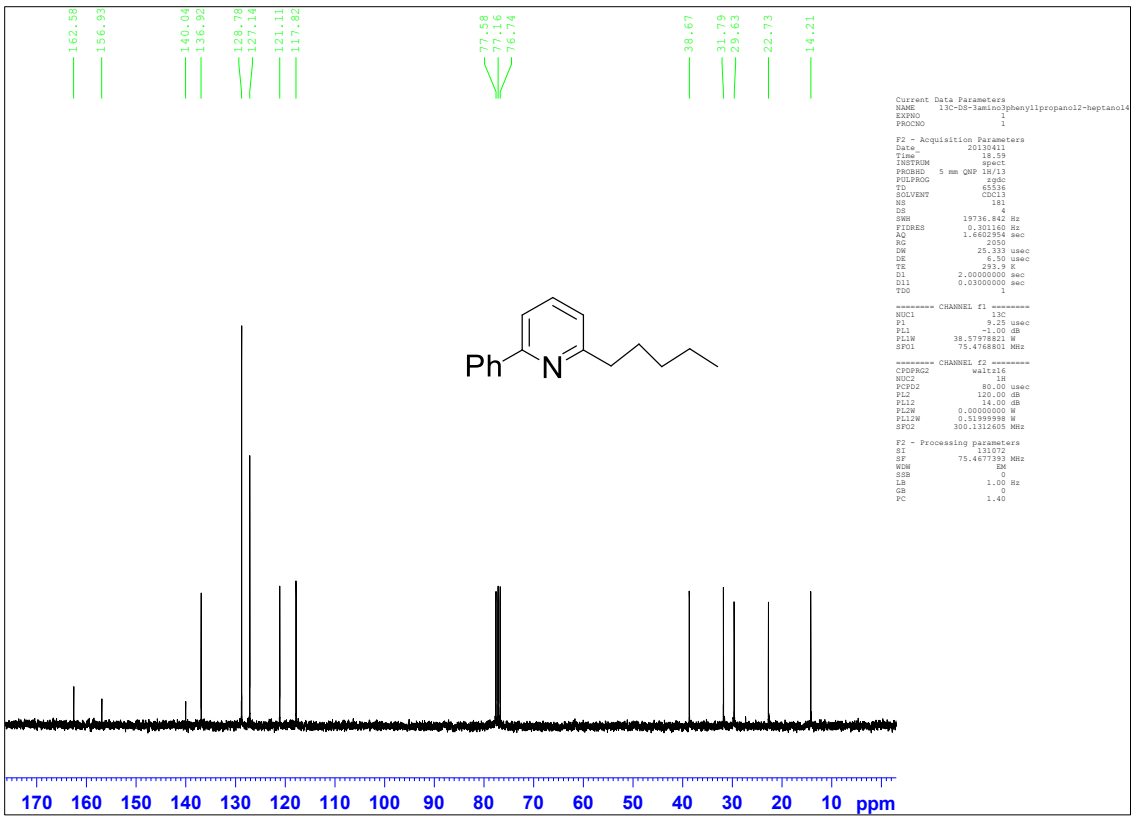

8.007
8.001
7.995
7.983
7.980
7.484
7.480
7.473
7.461
7.457
7.436
7.400
7.395
7.384
7.377
7.351
7.348
7.261
7.251
3.169
3.164
3.158
3.151
3.133
2.829
2.820
2.809
2.793
2.143
2.130
2.008
1.910
1.893
1.875
1.792
1.777
1.760
1.738
1.719
1.700
1.545
1.478
1.296
0.116
BRUKER
Ph
N
10
9
8
7
6
5
4
3
2
1
ppm
1.96
5.00
2.05
2.10
2.00
4.33

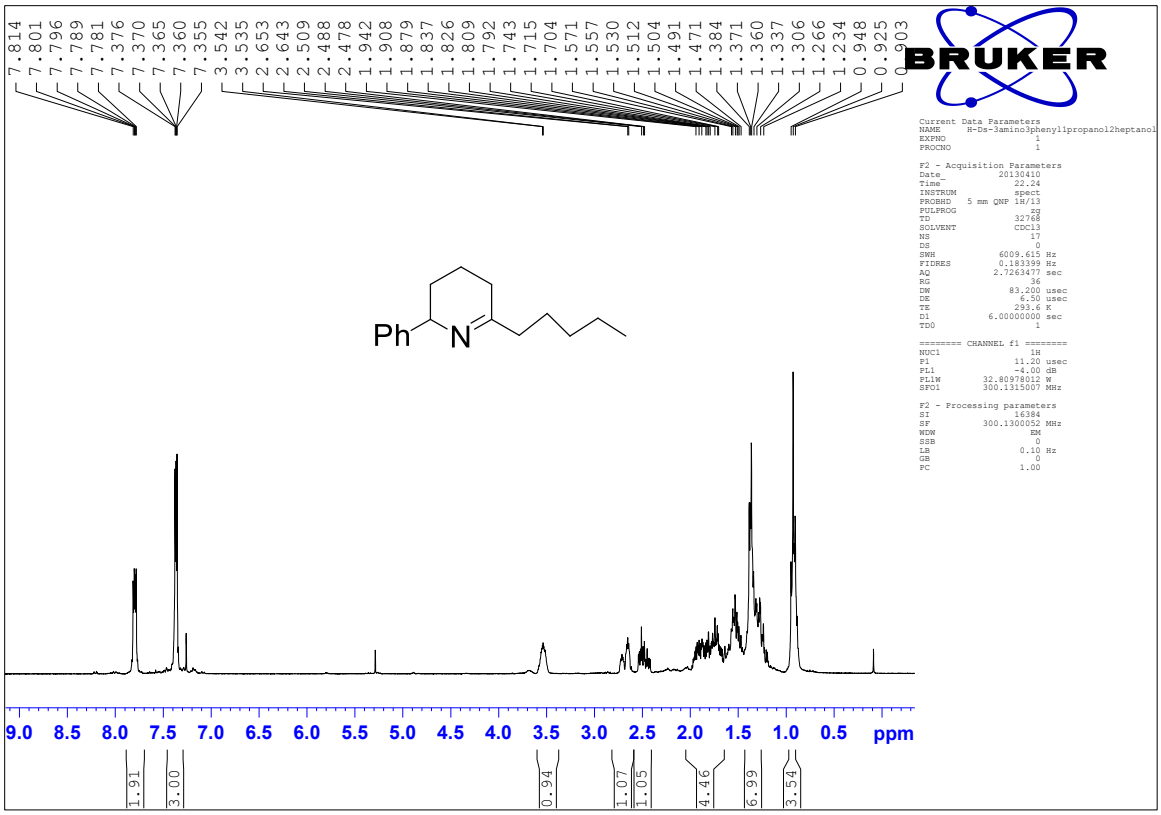

163.21
154.10
139.90
137.27
136.62
128.69
128.37
126.86
117.95
77.58
77.16
76.73
39.83
35.10
32.67
28.56
28.20
26.75
BRUKER
Ph
N
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

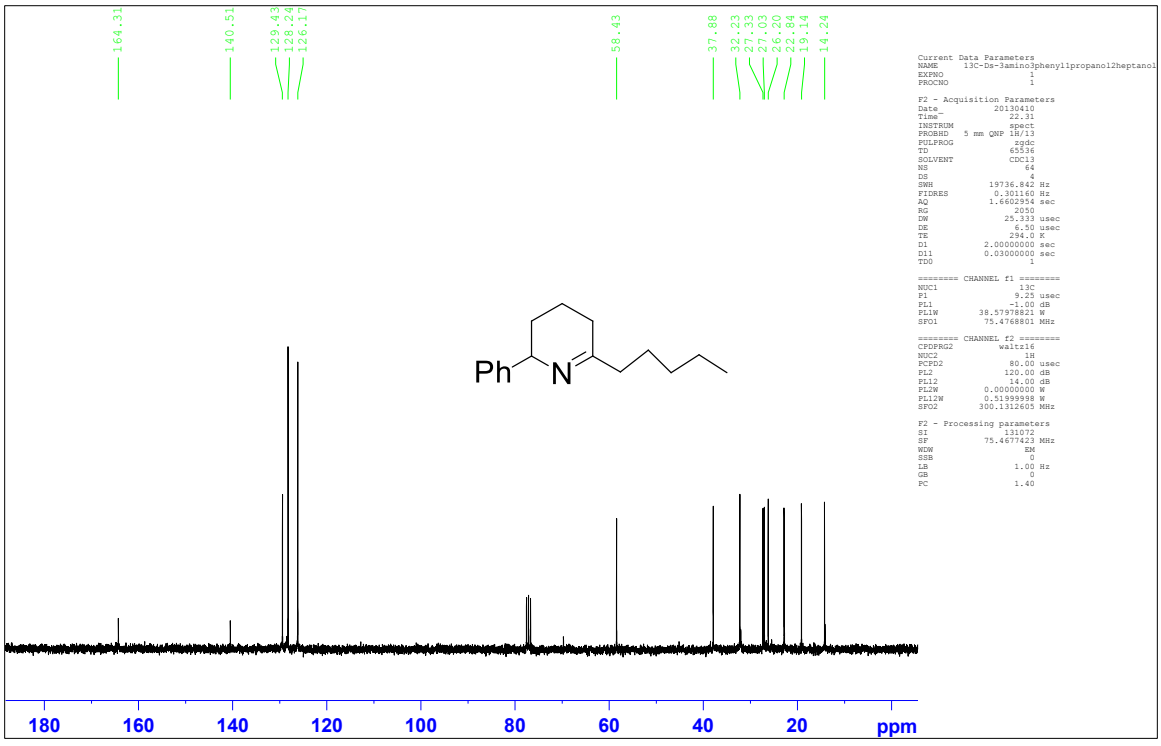

7.976
7.971
7.964
7.947
7.944
7.486
7.481
7.471
7.458
7.452
7.444
7.438
7.433
7.421
7.407
7.402
7.397
7.386
7.378
7.354
7.263
3.039
3.018
2.996
2.825
2.803
2.783
1.981
1.970
1.961
1.943
1.929
1.924
1.918
1.908
1.902
1.883
1.877
1.863
1.857
1.843
1.837
1.826
1.816
BRUKER
Ph
N
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
1.97
5.01
2.00
2.01
2.39
2.23

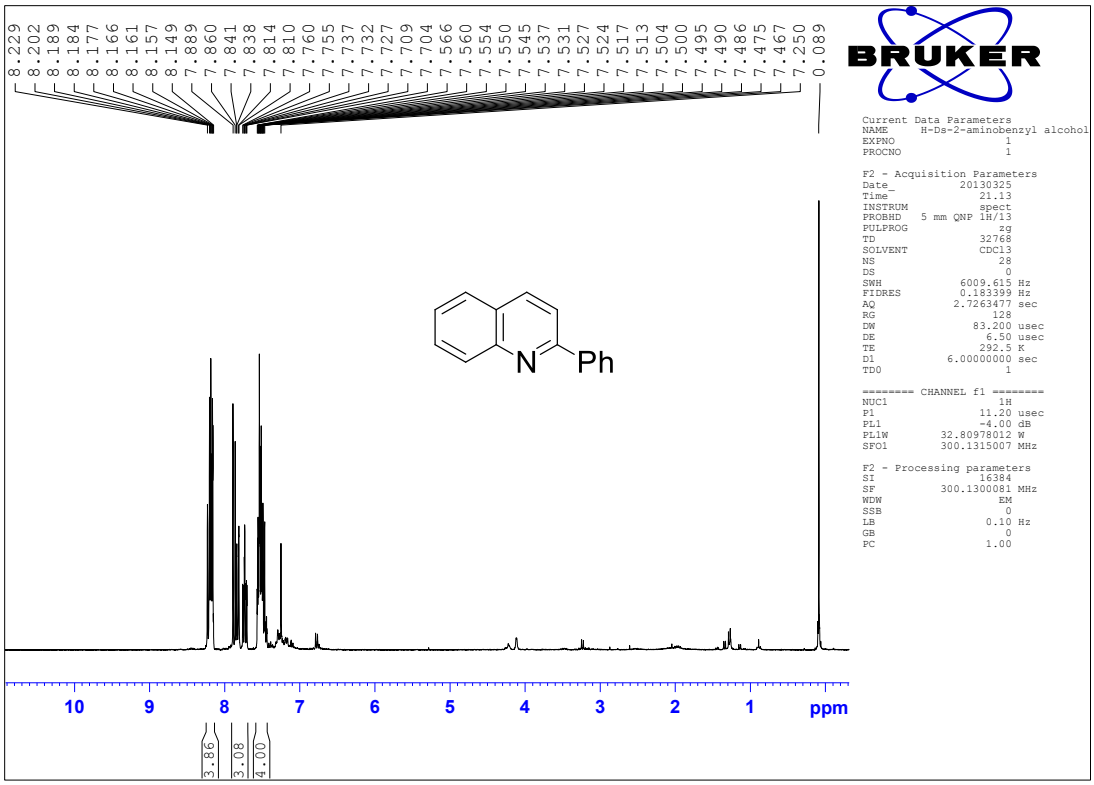

157.31
154.74
140.00
137.53
130.81
128.72
128.44
126.92
118.02
77.58
77.16
76.74
42.22
32.94
28.65
27.34
25.37
24.94
23.31
22.89
22.49
Ph
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

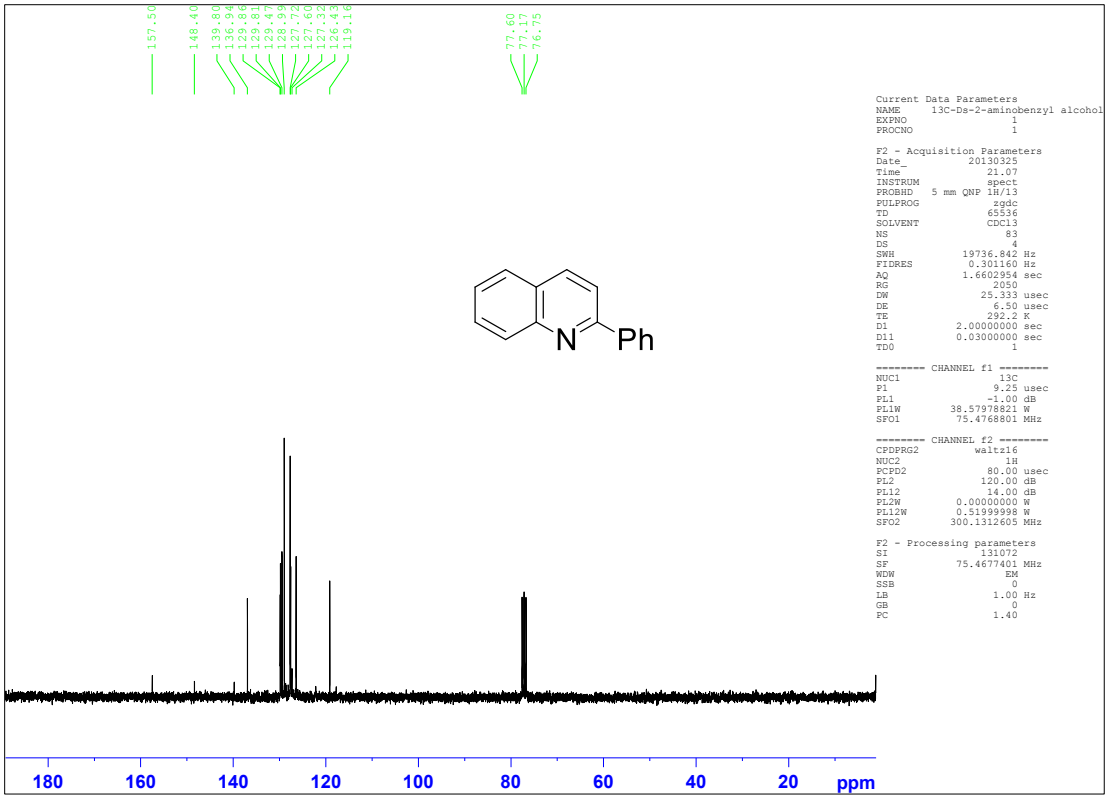

8.015
7.997
7.992
7.989
7.670
7.645
7.619
7.535
7.509
7.491
7.468
7.462
7.442
7.416
7.393
7.260
7.098
7.072
2.886
2.860
2.834
1.839
1.831
1.814
1.804
1.795
1.788
1.398
1.386
1.374
0.945
0.922
0.898
Ph
N
9
8
7
6
5
4
3
2
1
0
ppm
1.70
0.88
3.48
0.87
1.82
1.92
4.00
3.10
BRUKER
Current Data Parameters
NAME H-DS-3amino3phenyllpropanol2-heptanol4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130411
Time_ 18.53
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 28
DS 0
SWH 6024.096 Hz
FIDRES 0.183841 Hz
AQ 2.7197940 sec
RG 114
DW 83.000 usec
DE 6.50 usec
TE 293.5 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PLW 32.80978012 W
SFO1 300.1300600 MHz
F2 - Processing parameters
SI 16384
SF 300.1300052 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

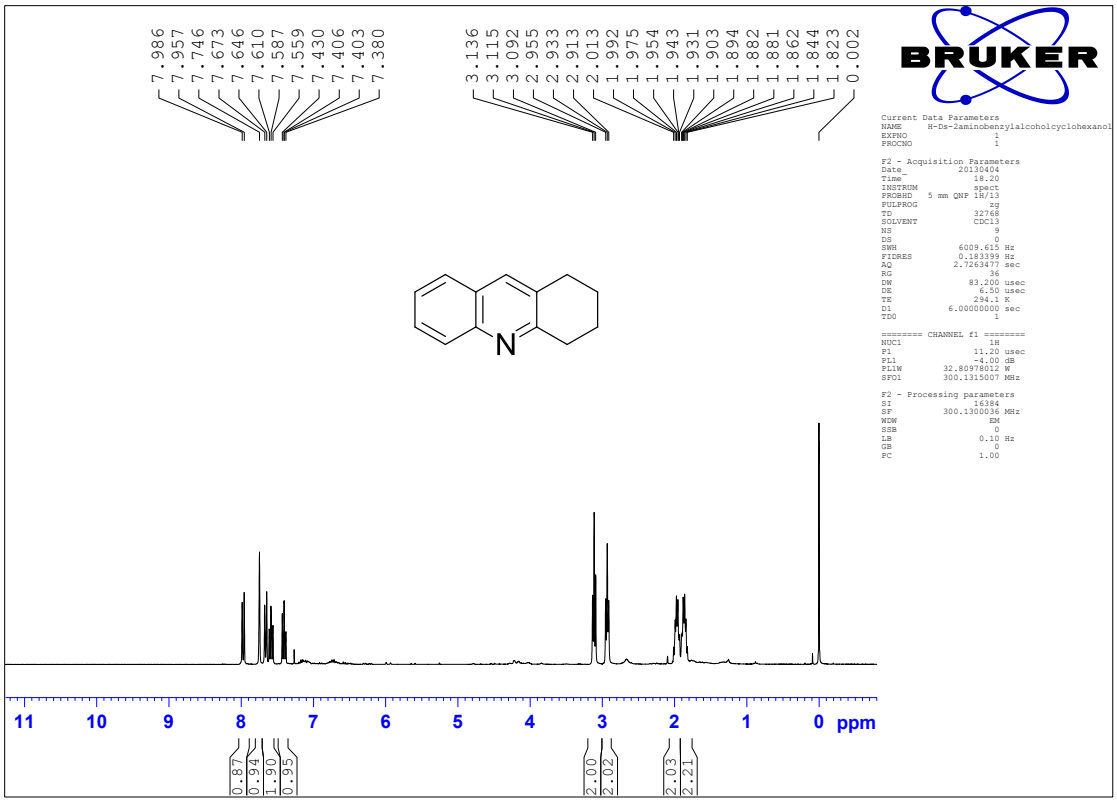

162.58
156.93
140.04
136.22
128.78
127.14
121.11
117.82
77.58
77.16
76.74
38.67
31.79
29.63
22.73
14.21
Ph
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
Current Data Parameters
NAME 13C-DS-3amino3phenyllpropanol2-heptanol4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130411
Time_ 18.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 181
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PLW 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677393 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

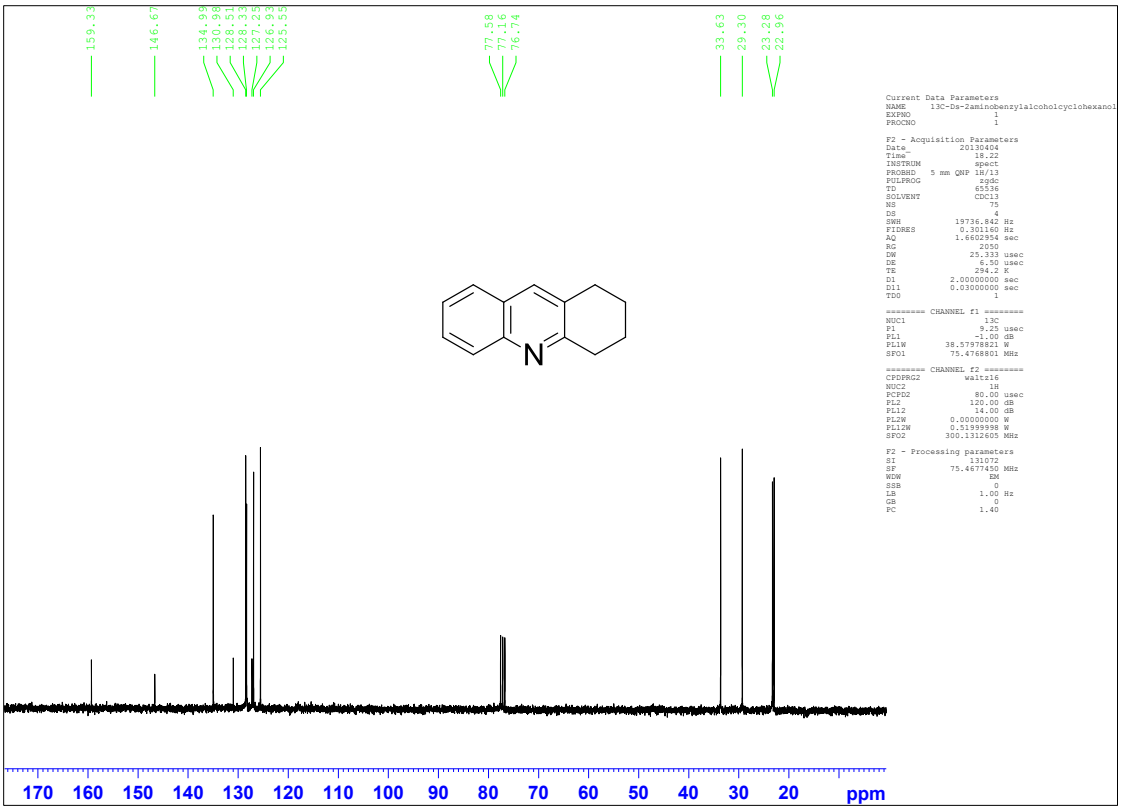

7.814 7.801 7.796 7.789 7.781 7.376 7.370 7.365 7.360 7.355 3.542 3.535 2.653 2.643 2.509 2.488 2.478 1.942 1.908 1.879 1.837 1.826 1.809 1.792 1.743 1.715 1.704 1.571 1.557 1.530 1.512 1.504 1.491 1.471 1.384 1.371 1.360 1.337 1.306 1.266 1.234 0.948 0.925 0.903
BRUKER
Ph
N
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.91 3.00 0.94 1.07 1.05 4.46 6.99 3.54

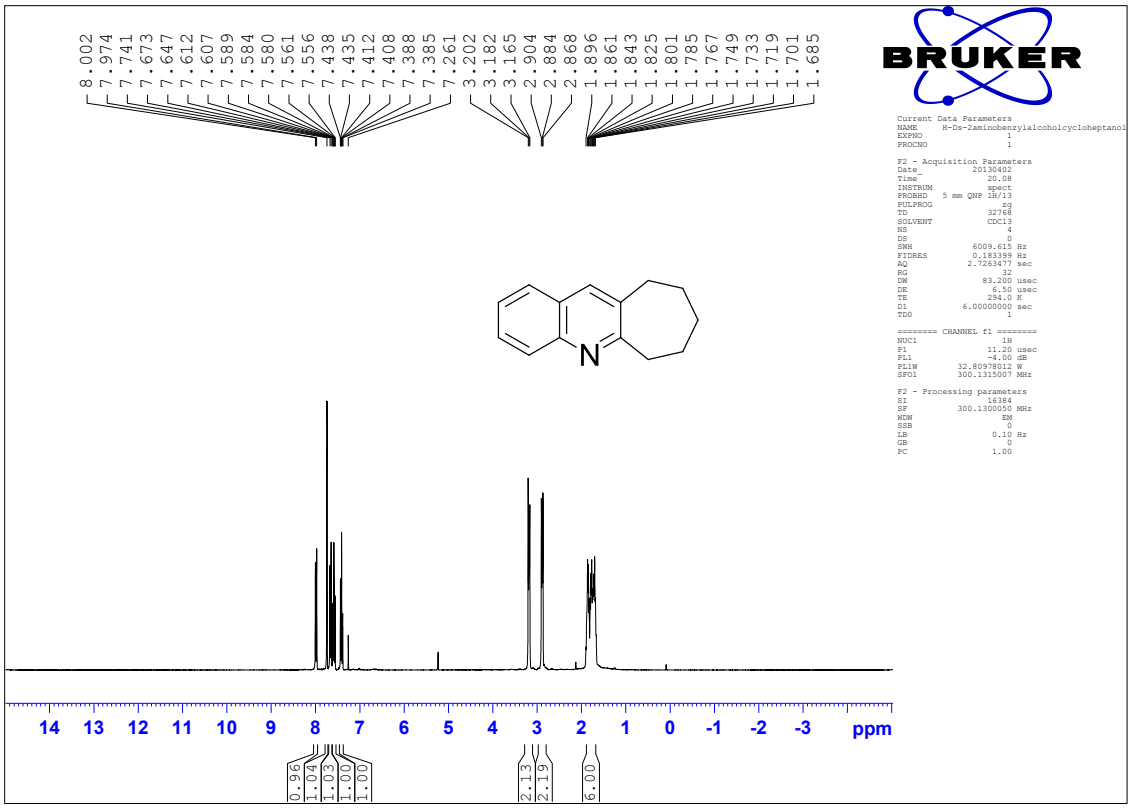

164.31 140.51 129.43 128.24 126.17 58.43 37.88 32.23 27.33 27.03 26.20 22.84 19.14 14.24
Ph
N
180 160 140 120 100 80 60 40 20 ppm

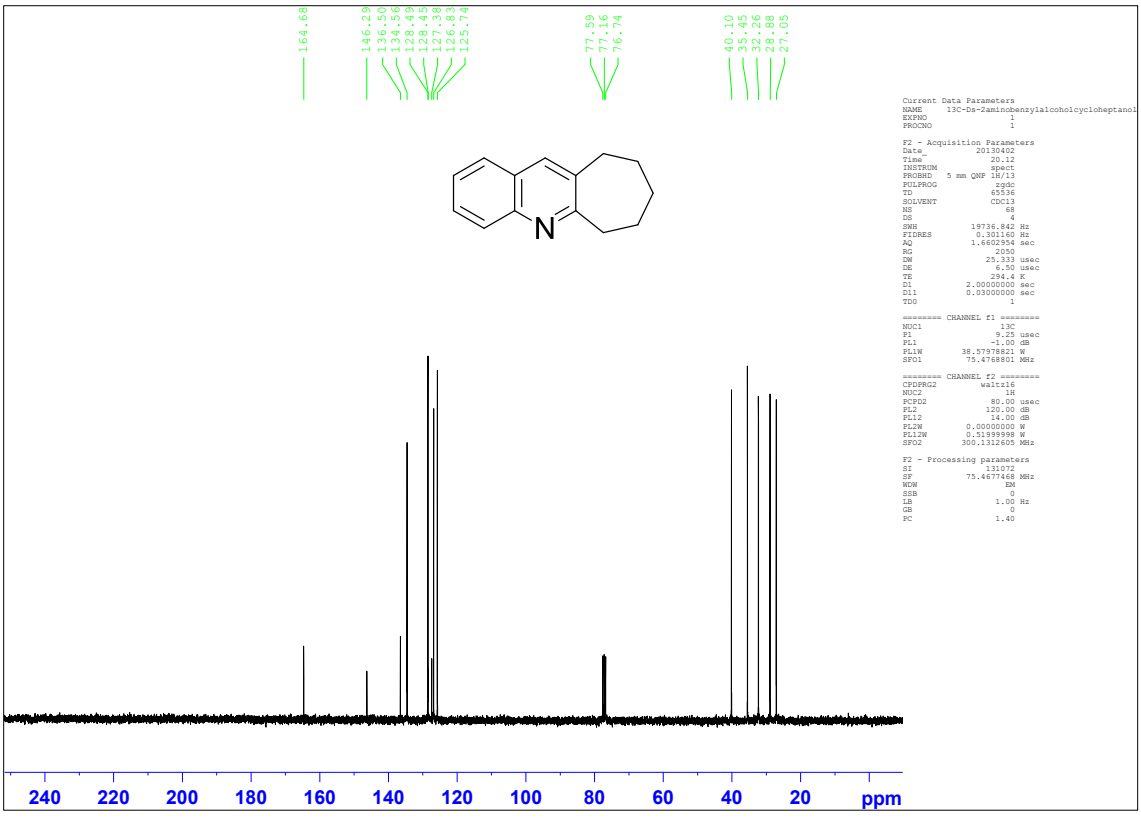

8.229
8.202
8.189
8.184
8.177
8.166
8.161
8.157
8.149
7.889
7.860
7.841
7.838
7.814
7.810
7.760
7.755
7.737
7.732
7.727
7.709
7.704
7.566
7.560
7.554
7.550
7.545
7.537
7.531
7.527
7.524
7.517
7.513
7.504
7.500
7.495
7.490
7.486
7.475
7.467
7.250
0.089
N
Ph
10
9
8
7
6
5
4
3
2
1
ppm
3.86
3.08
4.00
BRUKER
Current Data Parameters
NAME H-Ds-2-aminobenzyl alcohol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130325
Time 21.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 28
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 128
DW 83.200 usec
DE 6.50 usec
TE 292.5 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PL1W 32.80978012 W
SFO1 300.1315007 MHz
F2 - Processing parameters
SI 16384
SF 300.1300081 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

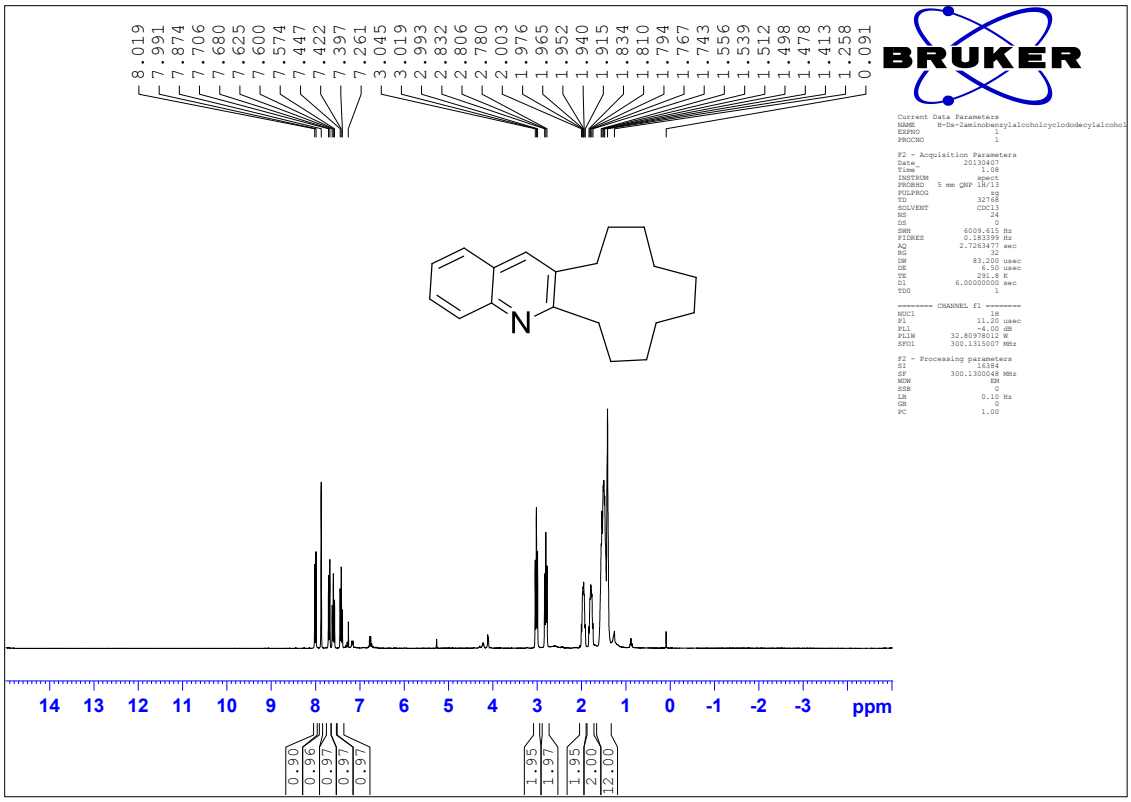

157.50
148.40
139.80
136.94
129.86
129.81
129.47
128.99
127.72
127.60
127.32
126.43
119.16
77.60
77.17
76.75
N
Ph
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME 13C-Ds-2-aminobenzyl alcohol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130325
Time 21.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 83
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 292.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PL1W 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL2W 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677401 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

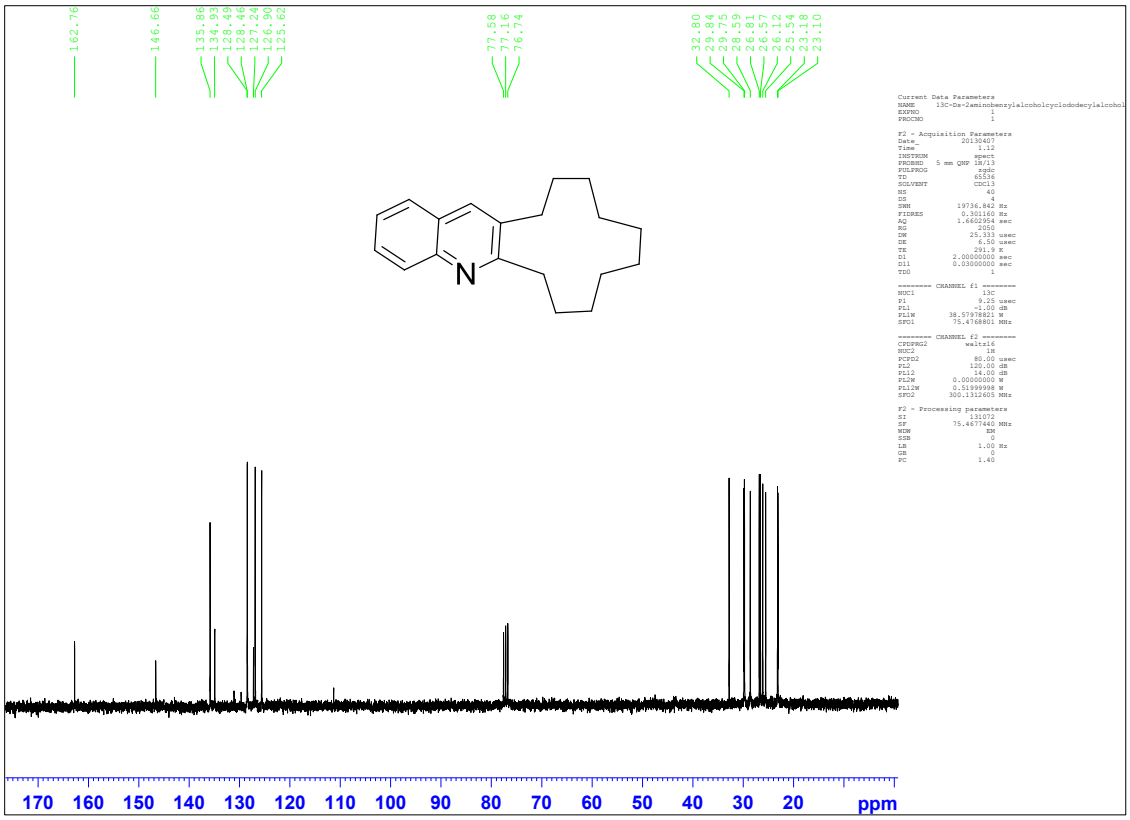

7.986
7.957
7.746
7.673
7.646
7.610
7.587
7.559
7.430
7.406
7.403
7.380
3.136
3.115
3.092
2.955
2.933
2.913
2.013
1.992
1.975
1.954
1.943
1.931
1.903
1.894
1.882
1.881
1.862
1.844
1.823
0.002
BRUKER
Current Data Parameters
NAME H-Ds-2aminobenzylalcoholcyclohexanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 20130404
Time 18.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 9
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 36
DW 83.200 usec
DE 6.50 usec
TE 294.1 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.20 usec
PL1 -4.00 dB
PLW 32.80978012 W
SFO1 300.1315007 MHz
F2 - Processing parameters
SI 16384
SF 300.1300036 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.87 0.94 1.90 0.95
2.00 2.02
2.03 2.21


159.33
146.67
134.99
130.98
128.51
128.33
127.25
126.93
125.55
77.58
77.16
76.74
33.63
29.30
23.28
22.96
Current Data Parameters
NAME 13C-Ds-2aminobenzylalcoholcyclohexanol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 20130404
Time 18.22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 75
DS 4
SWH 19736.842 Hz
FIDRES 0.301160 Hz
AQ 1.6602954 sec
RG 2050
DW 25.333 usec
DE 6.50 usec
TE 294.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -1.00 dB
PLW 38.57978821 W
SFO1 75.4768801 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 120.00 dB
PL12 14.00 dB
PLW 0.00000000 W
PL12W 0.51999998 W
SFO2 300.1312605 MHz
F2 - Processing parameters
SI 131072
SF 75.4677450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.002
7.974
7.741
7.673
7.647
7.612
7.607
7.589
7.584
7.580
7.561
7.556
7.438
7.435
7.412
7.408
7.388
7.385
7.261
3.202
3.182
3.165
2.904
2.884
2.868
1.896
1.861
1.843
1.825
1.801
1.785
1.767
1.749
1.733
1.719
1.701
1.685
BRUKER
N
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm
0.96
1.04
1.03
1.00
1.00
2.13
2.19
6.00


164.68
146.29
136.50
134.56
128.49
128.45
127.38
126.83
125.74
77.59
77.16
76.74
40.10
35.45
32.26
28.88
27.05
N
240 220 200 180 160 140 120 100 80 60 40 20 ppm


8.019
7.991
7.874
7.706
7.680
7.625
7.600
7.574
7.447
7.422
7.397
7.261
3.045
3.019
2.993
2.832
2.806
2.780
2.003
1.976
1.965
1.952
1.940
1.915
1.834
1.810
1.794
1.767
1.743
1.556
1.539
1.512
1.498
1.478
1.413
1.258
0.091
BRUKER
N
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm
0.90
0.96
0.97
0.97
0.97
1.95
1.97
1.95
2.00
12.00


162.76
146.66
135.86
134.93
128.49
128.46
127.24
126.90
125.62
77.58
77.16
76.74
32.80
29.84
29.75
28.59
26.81
26.57
26.12
25.54
23.18
23.10
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

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