# Supporting Information

# Complications from Dual Roles of Sodium Hydride as a Base and as a Reducing Agent

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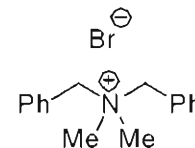
**Contents**

DHL-3_7

expl  s2pul

```
           SANPLE              DEC. & VT
date    Nov  9 2007    dfrq      499.864
solvent      CDC13     dn            H1
file           exp     dpwr          30
     ACQUISITION       uof            0
sfrq         499.864   dm           nnn
tn              H1     dmm            c
at            5.016    dmf          200
np            65536    dseq
sw           6533.3    dres         1.0
fb            4000     homo           n
bs               4           DEC2
tpwr            61     dfrq2          0
pw            13.5     dn2
d1           0.100     dpwr2          1
tof          269.9     dof2           0
nt               8     dm2            n
ct               8     dmm2           c
alock            n     dmf2         200
gain            30     dseq2
      FLAGS            dres2        1.0
il               n     homo2          n
in               n           OEC3
dp               y     dfrq3          0
hs              nn     dn3
    DISPLAY            dpwr3          1
sp           1120.6    dof3           0
wp           4695.8    dm3            n
vs              33     dmm3           c
sc               0     dmf3         200
wc             250     dseq3
hzmm          18.78    dres3        1.0
is          100.00     homo3          n
rfl          510.6        PROCESSING
rfp              0     wtfile
th               7     proc          ft
ins          2.000     fn          65536
al       ph           math           f

                      werr
                      wexp    process p1H
                      wbs
                      wnt          wft
```

3



2.03   3.06          2.00          3.03

DHL-3_7

exp2  s2pul

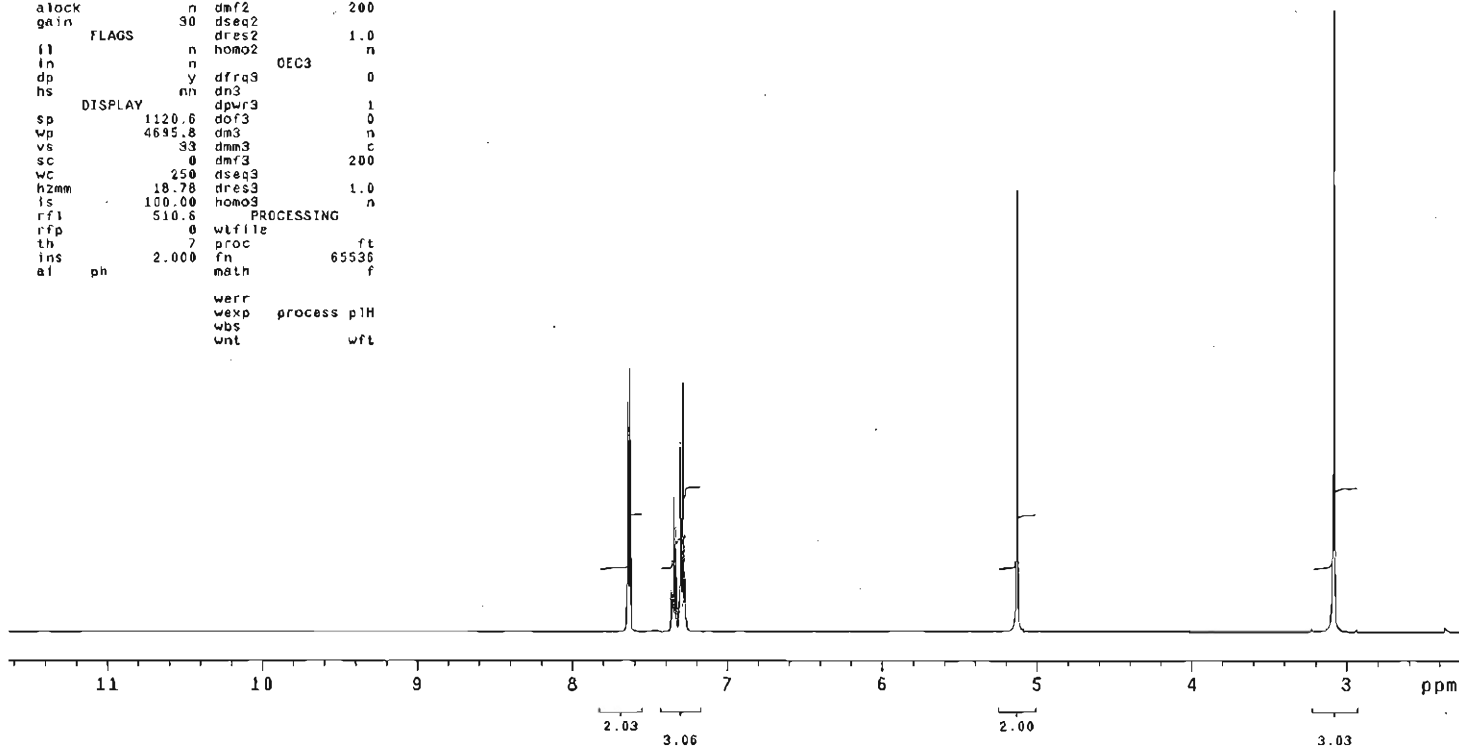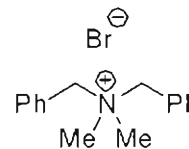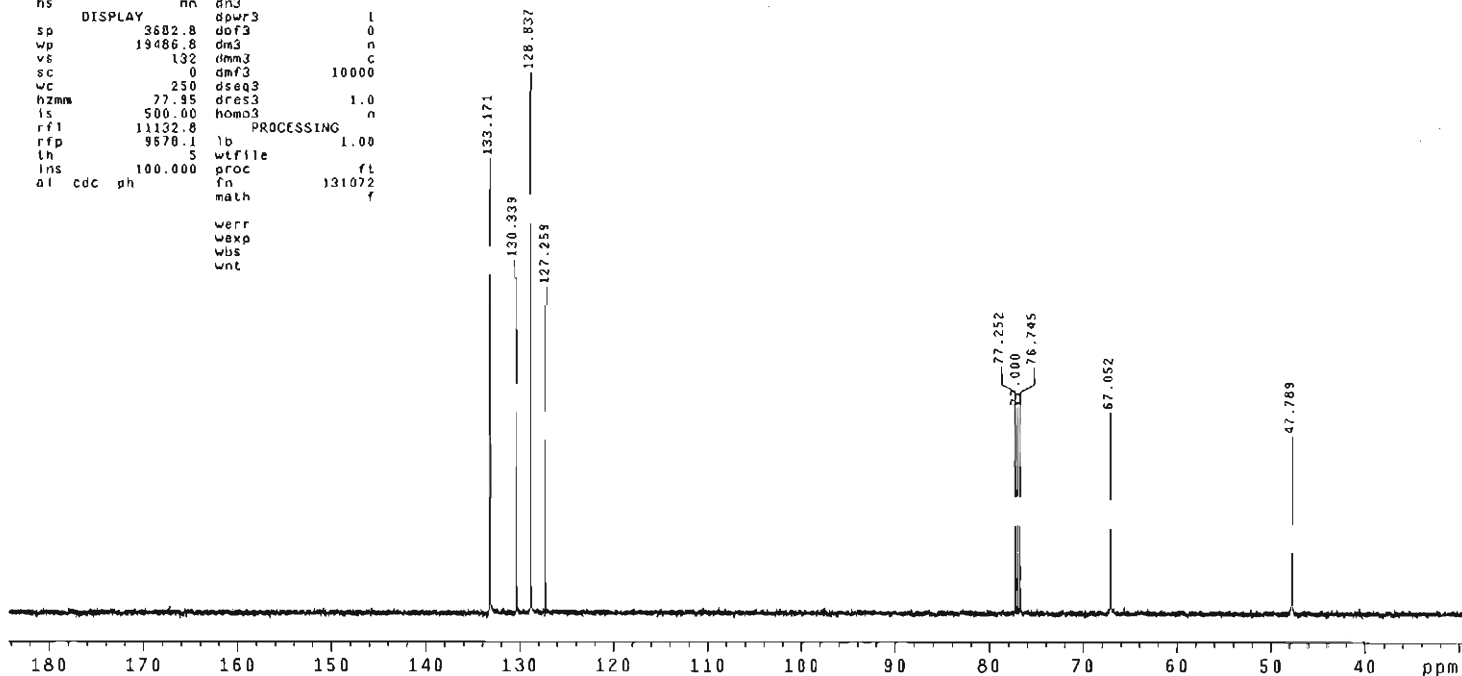         SAMPLE              DEC. & VT
date    Nov  9 2007     dfrq        499.864
solvent     CDC13       dn              H1
file         exp        dpwr            40
      ACQUISITION       dof              0
sfrq    125.702         dm             yyy
tn         C13          dmm              u
at        1.215         dmf         8787.35
np        65536         dseq
sw      26963.3         dres           1.0
fb        15000         homo             n
bs            4             DEC2
tpwr         52         dfrq2            0
pw         10.2         dn2
d1        1.800         dpwr2            1
tof       144.5         dof2             0
nt          640         dm2              n
ct           82         dmm2             c
alock         n         dmf2         10000
gain    not used        dseq2
      FLAGS             dres2          1.0
il            n         homo2            n
in            n             DEC3
dp            y         dfrq3            0
hs           nn         dn3
    DISPLAY             dpwr3            1
sp       3682.8         dof3             0
wp      19486.8         dm3              n
vs          132         dmm3             c
sc            0         dmf3         10000
wc          250         dseq3
hzmm      77.95         dres3          1.0
is       500.00         homo3            n
rfl     11132.8            PROCESSING
rfp      9578.1         lb             1.00
th            5         wtfile
ins     100.000         proc            ft
al  cdc  ph             fn          131072
                        math             f

                        werr
                        wexp
                        wbs
                        wnt

133.171  130.339  128.837  127.259  77.252  77.000  76.745  67.052  47.789

180  170  160  150  140  130  120  110  100  90  80  70  60  50  40  ppm

DHL-3_7-BnBr-DMF-NaH·

exp7 s2pul

```
            SAMPLE                 DEC. & VT
date     Sep 24 2008    dfrq          499.866
solvent         dmf     dn                 H1
file            exp     dpwr               30
     ACQUISITION        dof                 0
sfrq        499.866     dm                nnn
tn               H1     dmm                 c
at            5.016     dmf               200
np            65536     dseq
sw           6533.3     dres              1.0
fb             4000     homo                n
bs                4          DEC2
tpwr             61     dfrq2               0
pw             13.5     dn2
d1            0.100     dpwr2               1
tof           269.9     dof2                0
nt               16     dm2                 n
ct               16     dmm2                c
alock             n     dmf2              200
gain       not used     dseq2
       FLAGS            dres2             1.0
il                n     homo2               n
in                n          DEC3
dp                y     dfrq3               0
hs               nn     dn3
     DISPLAY            dpwr3               1
sp              9.2     dof3                0
wp           5073.6     dm3                 n
vs               27     dmm3                c
sc                0     dmf3              200
wc              250     dseq3
hzmm          20.29     dres3             1.0
is           105.52     homo3               n
rfl          4537.1        PROCESSING
rfp          4013.9     wtfile
th               61     proc               ft
ins           4.000     fn              65536
ai       ph            math                 f

                        werr
                        wexp    process p1H
                        wbs
                        int               wft
```
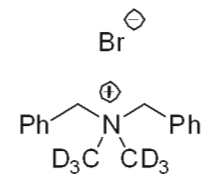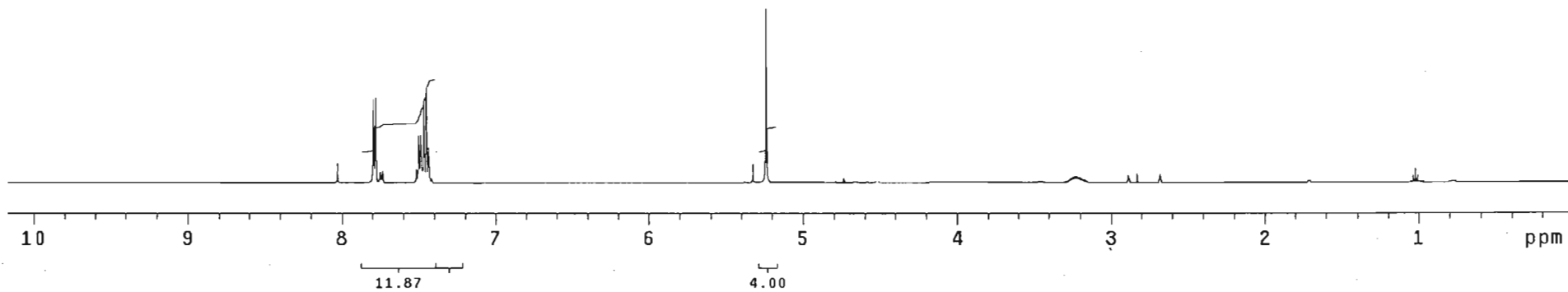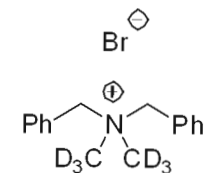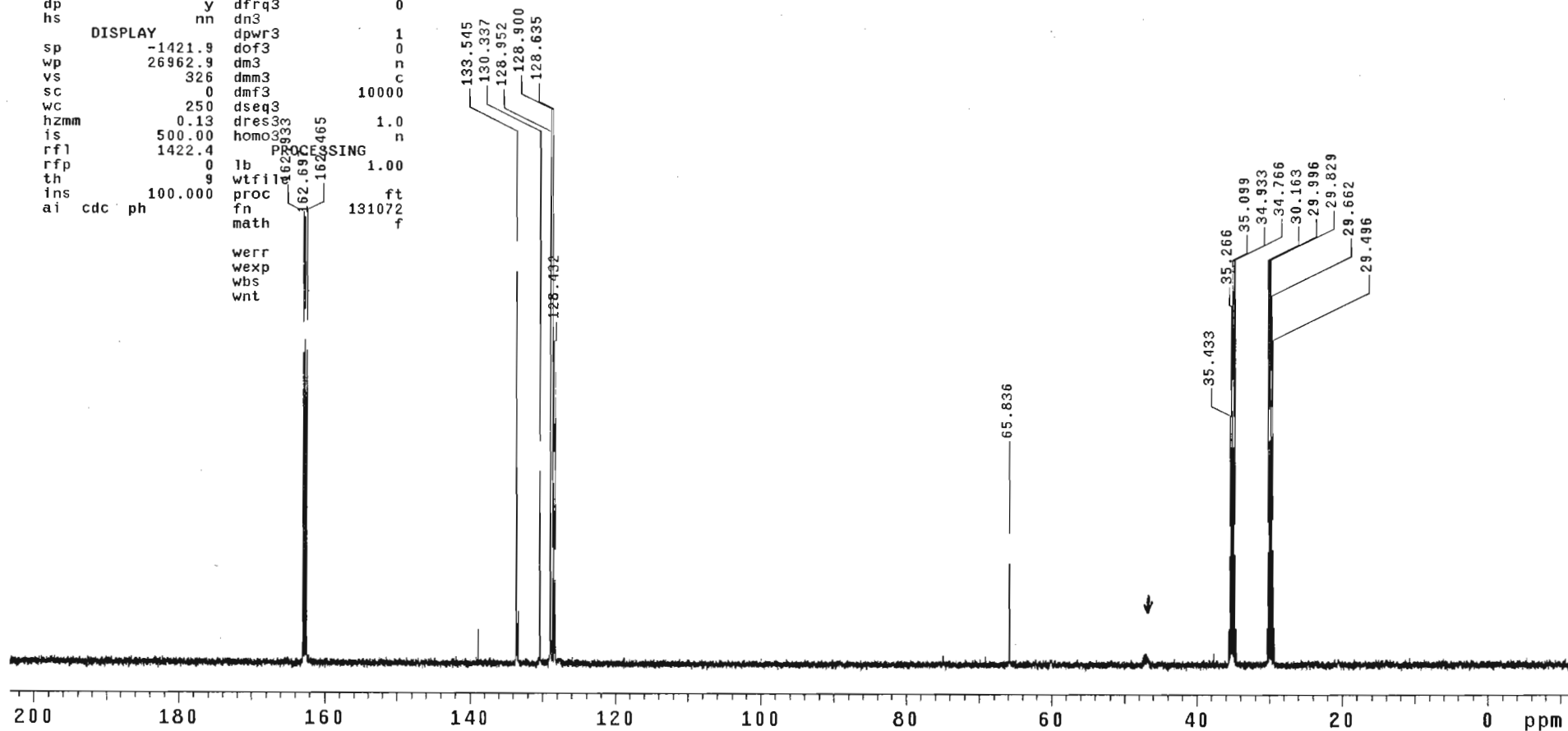


3-D



11.87    4.00

DHL-3_7-BnBr-DMF-NaH

exp8  s2pul

SAMPLE
date      Sep 24 2008
solvent         dmf
file            exp
ACQUISITION
sfrq       125.703
tn             C13
at           1.215
np           65536
sw         26963.3
fb           15000
bs               4
tpwr            52
pw            10.2
d1           1.800
tof          144.5
nt            1200
ct             256
alock            n
gain     not used
FLAGS
il               n
in               n
dp               y
hs              nn
DISPLAY
sp         -1421.9
wp         26962.9
vs             326
sc               0
wc             250
hzmm          0.13
is          500.00
rfl         1422.4
rfp              0
th               9
ins        100.000
ai   cdc  ph

DEC. & VT
dfrq       499.866
dn              H1
dpwr            40
dof              0
dm             yyy
dmm              w
dmf        8787.35
dseq
dres           1.0
homo             n
DEC2
dfrq2            0
dn2
dpwr2            1
dof2             0
dm2              n
dmm2             c
dmf2         10000
dseq2
dres2          1.0
homo2            n
DEC3
dfrq3            0
dn3
dpwr3            1
dof3             0
dm3              n
dmm3             c
dmf3         10000
dseq3
dres3          1.0
homo3            n
PROCESSING
lb            1.00
wtfile
proc            ft
fn          131072
math             f

werr
wexp
wbs
wnt

3-D

DHL-56

exp1 s2pul

```
        SAMPLE              DEC. & VT
date    Mar 26 2008    dfrq        499.864
solvent      CDC13     dn                H1
file          exp      dpwr              30
    ACQUISITION        dof                0
sfrq      499.864      dm               nnn
tn             H1      dmm                c
at          5.016      dmf              200
np          65536      dseq
sw         6533.3      dres             1.0
fb           4000      homo               n
bs              4              DEC2
tpwr           61      dfrq2              0
pw           13.5      dn2
d1          0.100      dpwr2              1
tof         269.9      dof2               0
nt             16      dm2                n
ct             16      dmm2               c
alock           n      dmf2             200
gain     not used      dseq2
    FLAGS              dres2            1.0
il              n      homo2              n
in              n              DEC3
dp              y      dfrq3              0
hs             nn      dn3
    DISPLAY            dpwr3              1
sp          399.6      dof3               0
wp         4601.5      dm3                n
vs             53      dmm3               c
sc              0      dmf3             200
wc            250      dseq3
hzmm        18.41      dres3            1.0
is          50.61      homo3              n
rfl         510.6          PROCESSING
rfp             0      wtfile
th              7      proc              ft
ins        10.000      fn             65536
ai      ph            math               f

                       werr
                       wexp    process p1H
                       wbs
                       wnt             wft
```
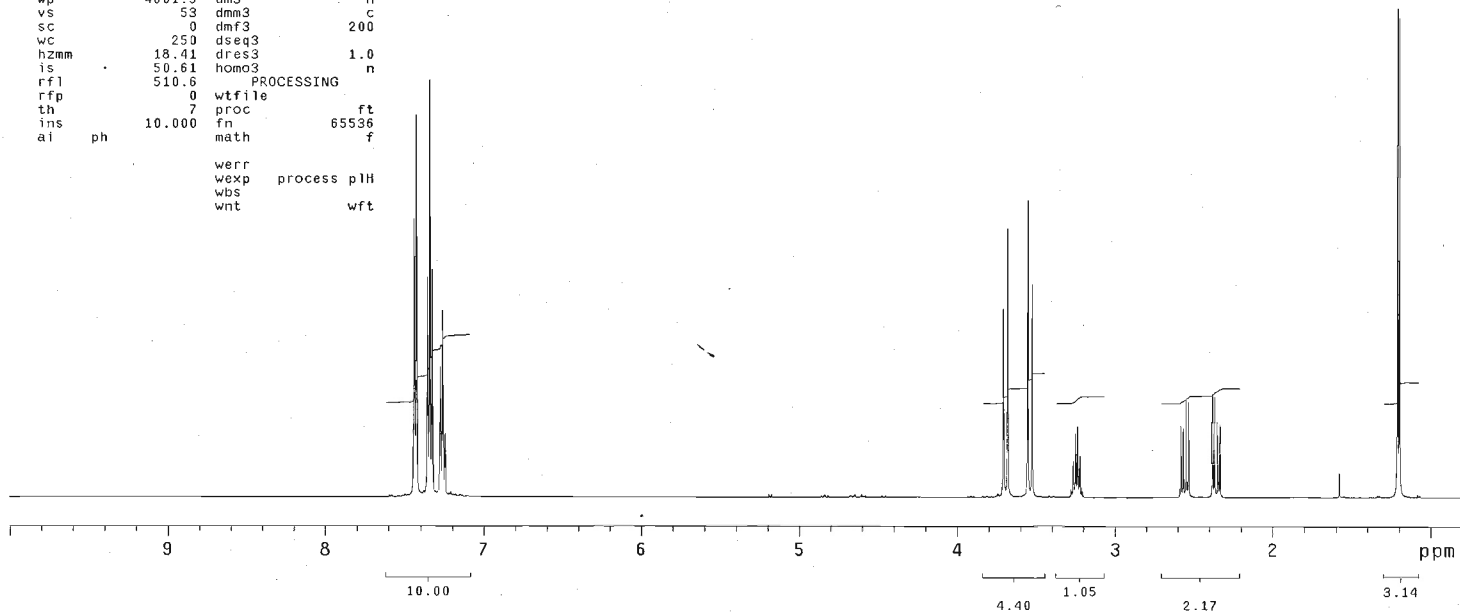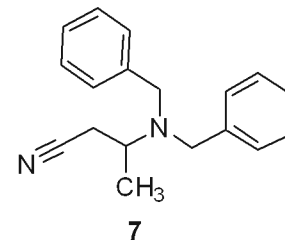
7



10.00    4.40    1.05    2.17    3.14

DHL-56

Pulse Sequence: relayh

Solvent: CDCl3
Ambient temperature
INOVA-500  "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.147 sec
Width     3482.8 Hz
2D Width 3482.8 Hz
4 repetitions
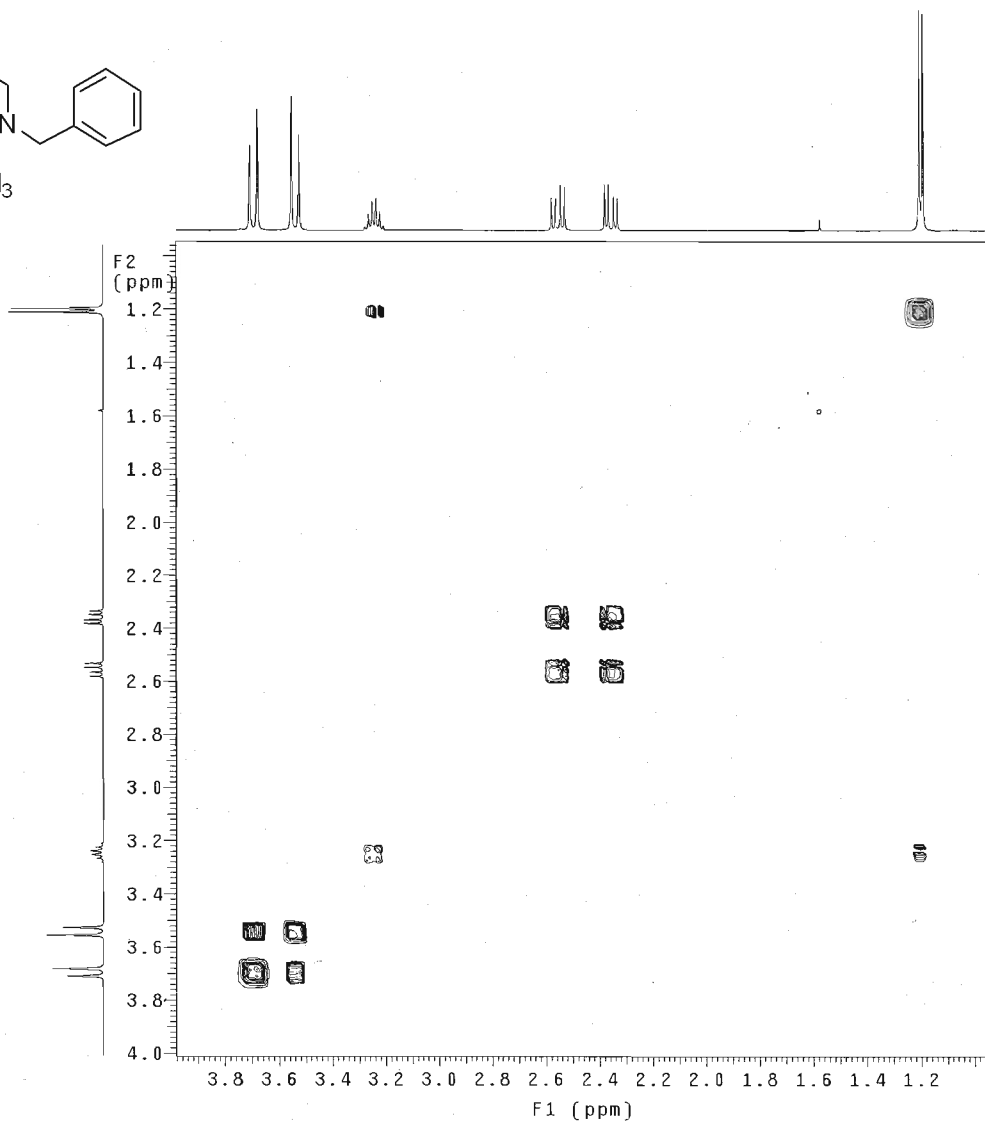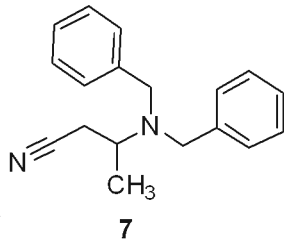256 increments
OBSERVE   H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.074 sec
F1 DATA PROCESSING
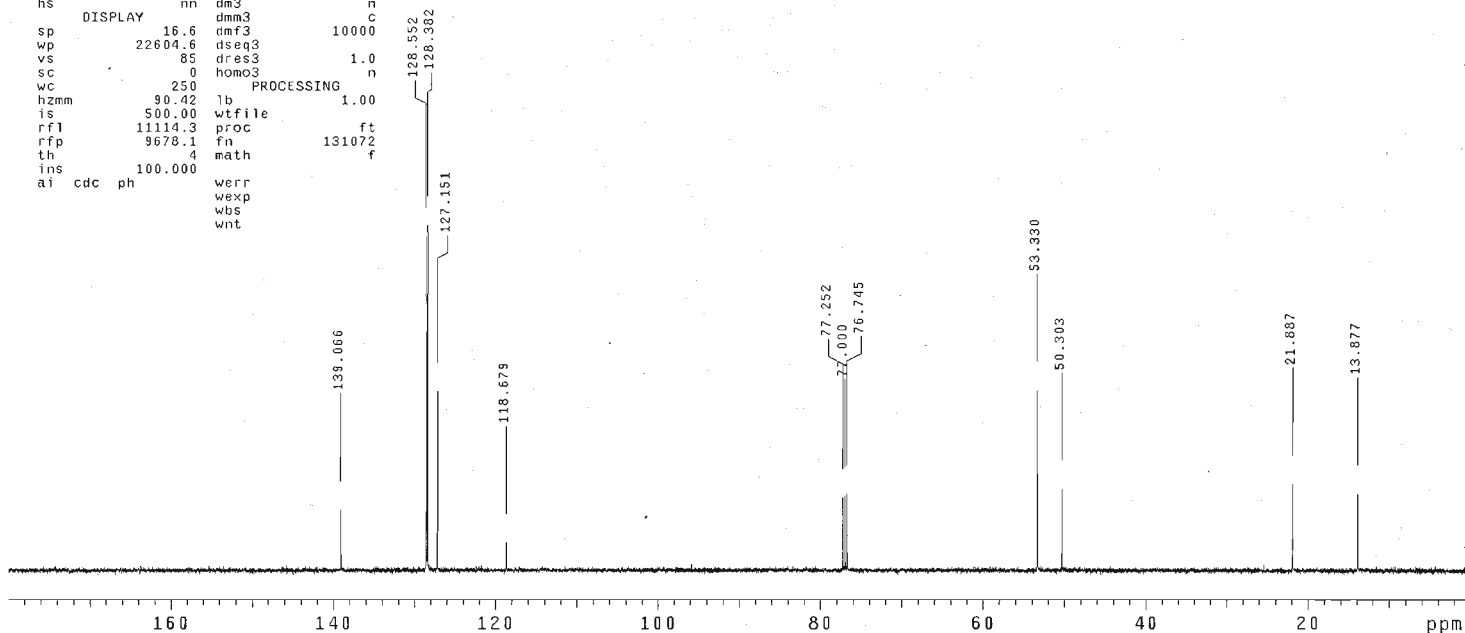Sine bell 0.037 sec
FT size 1024 x 1024
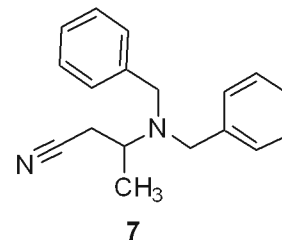Total time 25 min, 41 sec

**7**

DHL-56

exp6  s2pul

|   | SAMPLE | | DEC. & VT | |
| date | Mar 26 2008 | dfrq | | 499.864 |
| solvent | CDCl3 | dn | | H1 |
| file | /afs/nd.edu/u~ | dpwr | | 40 |
| ser26/dhesek/Priva~ | | dof | | 0 |
| te/DHL/DHL-56-C.fi~ | | dm | | yyy |
| d | | dmm | | w |
| ACQUISITION | | dmf | | 8787.35 |
| sfrq | 125.702 | dseq | | |
| tn | C13 | dres | | 1.0 |
| at | 1.215 | homo | | n |
| np | 65536 | | DEC2 | |
| sw | 26963.3 | dfrq2 | | 0 |
| fb | 15000 | dn2 | | |
| bs | 4 | dpwr2 | | 1 |
| tpwr | 52 | dof2 | | 0 |
| pw | 10.2 | dm2 | | n |
| d1 | 1.800 | dmm2 | | c |
| tof | 144.5 | dmf2 | | 10000 |
| nt | 640 | dseq2 | | |
| ct | 47 | dres2 | | 1.0 |
| alock | n | homo2 | | n |
| gain | not used | | DEC3 | |
| FLAGS | | dfrq3 | | 0 |
| il | n | dn3 | | |
| in | n | dpwr3 | | 1 |
| dp | y | dof3 | | 0 |
| hs | nn | dm3 | | n |
| | | dmm3 | | c |
| DISPLAY | | dmf3 | | 10000 |
| sp | 16.6 | dseq3 | | |
| wp | 22604.6 | dres3 | | 1.0 |
| vs | 85 | homo3 | | n |
| sc | 0 | PROCESSING | | |
| wc | 250 | | | |
| hzmm | 90.42 | lb | | 1.00 |
| is | 500.00 | wtfile | | |
| rfl | 11114.3 | proc | | ft |
| rfp | 9678.1 | fn | | 131072 |
| th | 4 | math | | f |
| ins | 100.000 | | | |
| ai  cdc  ph | | werr | | |
| | | wexp | | |
| | | wbs | | |
| | | wnt | | |

7

128.552
128.382
127.151
139.066
118.679
77.252
77.000
76.745
53.330
50.303
21.887
13.877

160   140   120   100   80   60   40   20   ppm

- S 8 -

DHL-56

Pulse Sequence: dept
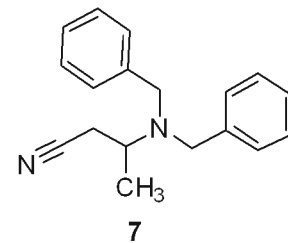
CH3 carbons

CH2 carbons

CH carbons

all protonated carbons

150    140    130    120    110    100    90    80    70    60    50    40    30    20    10    ppm



7

DHL-56

7



F1
(ppm)
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
3.2
3.4
3.6
3.8
4.0

55   50   45   40   35   30   25   20   15

F2 (ppm)

DHL-56b

exp3  s2pul

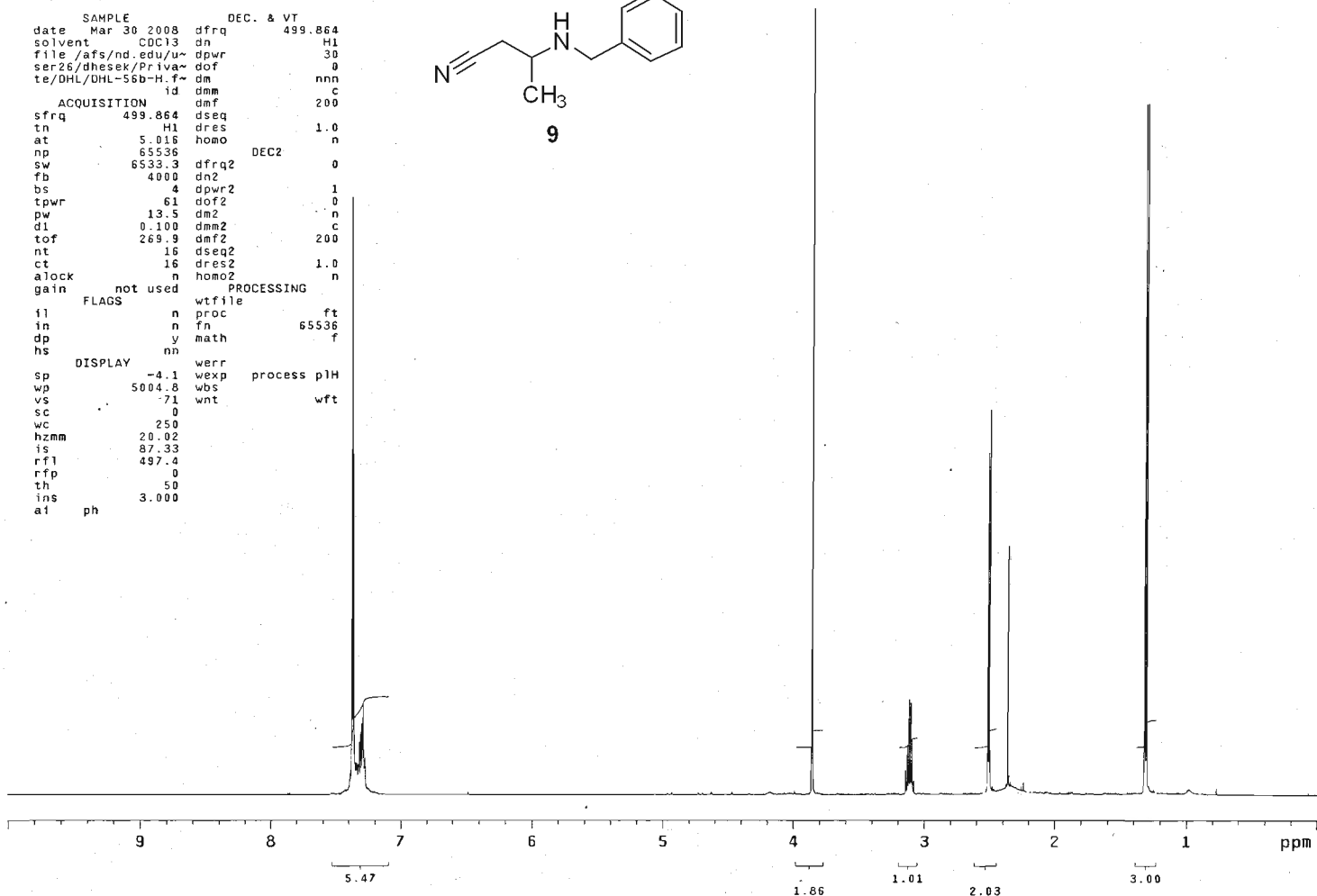|                | SAMPLE          |       | DEC. & VT |
|----------------|-----------------|-------|-----------|
| date           | Mar 30 2008     | dfrq  | 499.864   |
| solvent        | CDCl3           | dn    | H1        |
| file /afs/nd.edu/u~ |            | dpwr  | 30        |
| ser26/dhesek/Priva~ |            | dof   | 0         |
| te/DHL/DHL-56b-H.f~ |            | dm    | nnn       |
|                | id              | dmm   | c         |
| ACQUISITION    |                 | dmf   | 200       |
| sfrq           | 499.864         | dseq  |           |
| tn             | H1              | dres  | 1.0       |
| at             | 5.016           | homo  | n         |
| np             | 65536           |       | DEC2      |
| sw             | 6533.3          | dfrq2 | 0         |
| fb             | 4000            | dn2   | 1         |
| bs             | 4               | dpwr2 | 1         |
| tpwr           | 61              | dof2  | 0         |
| pw             | 13.5            | dm2   | n         |
| d1             | 0.100           | dmm2  | c         |
| tof            | 269.9           | dmf2  | 200       |
| nt             | 16              | dseq2 |           |
| ct             | 16              | dres2 | 1.0       |
| alock          | not used        | homo2 | n         |
| gain           | not used        | PROCESSING |      |
| FLAGS          |                 | wtfile |          |
| il             | n               | proc  | ft        |
| in             | n               | fn    | 65536     |
| dp             | y               | math  | f         |
| hs             | nn              |       |           |
| DISPLAY        |                 | werr  |           |
| sp             | -4.1            | wexp  | process plH |
| wp             | 5004.8          | wbs   |           |
| vs             | 71              | wnt   | wft       |
| sc             | 0               |       |           |
| wc             | 250             |       |           |
| hzmm           | 20.02           |       |           |
| is             | 87.33           |       |           |
| rfl            | 497.4           |       |           |
| rfp            | 0               |       |           |
| th             | 50              |       |           |
| ins            | 3.000           |       |           |
| ai    ph       |                 |       |           |

**9**

5.47    1.86    1.01    2.03    3.00

DHL-56b

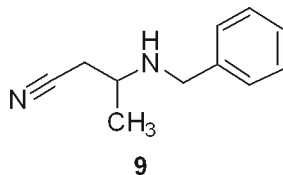exp2   s2pul

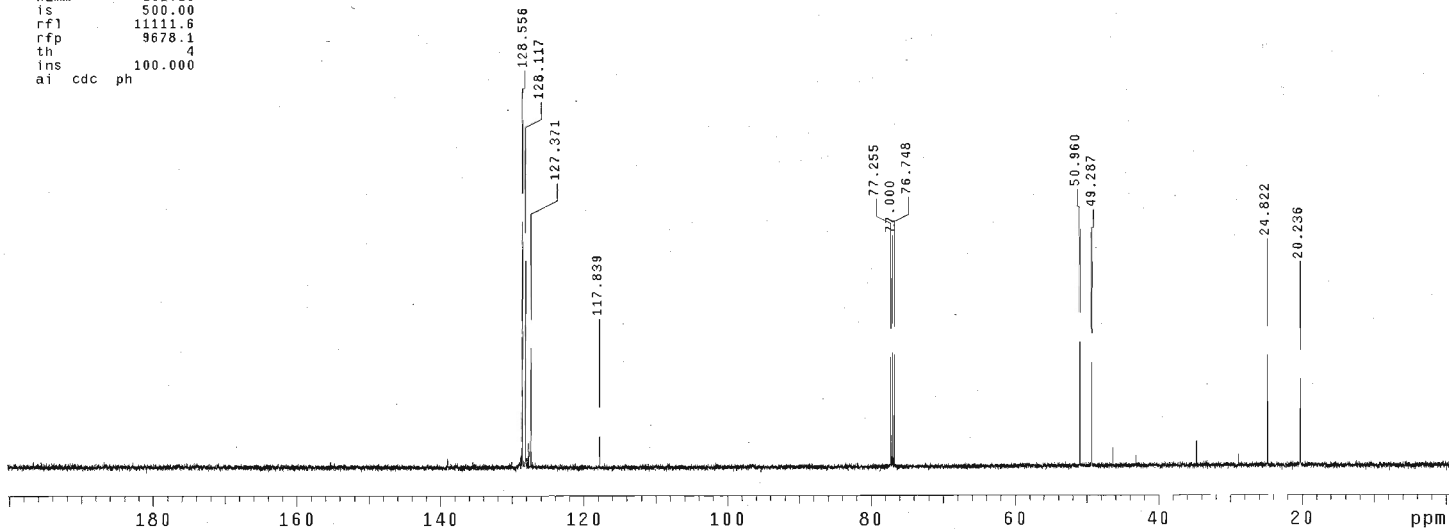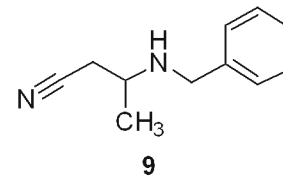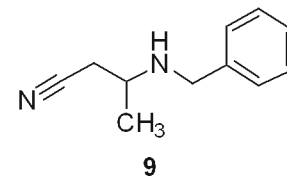SAMPLE                    DEC. & VT
date    Mar 30 2008   dfrq      499.864
solvent      CDC13    dn             H1
file /afs/nd.edu/u~   dpwr           40
ser26/dhesek/Priva~   dof             0
te/DHL/DHL-56b-C.f~   dm            yyy
            id        dmm             w
   ACQUISITION        dmf       8787.35
sfrq     125.702      dseq
tn           C13      dres          1.0
at         1.215      homo            n
np         65536        PROCESSING
sw       26963.3      lb           1.00
fb         15000      wtfile
bs             4      proc           ft
tpwr          52      fn         131072
pw          10.2      math           ·f
d1         1.800
tof        144.5      werr
nt          1200      wexp
ct           107      wbs
alock          n      wnt
gain    not used
      FLAGS
il             n
in             n
dp             y
hs            nn
    DISPLAY
sp        -122.6
wp       25297.8
vs           146
sc             0
wc           250
hzmm      101.19
is        500.00
rfl      11111.6
rfp       9678.1
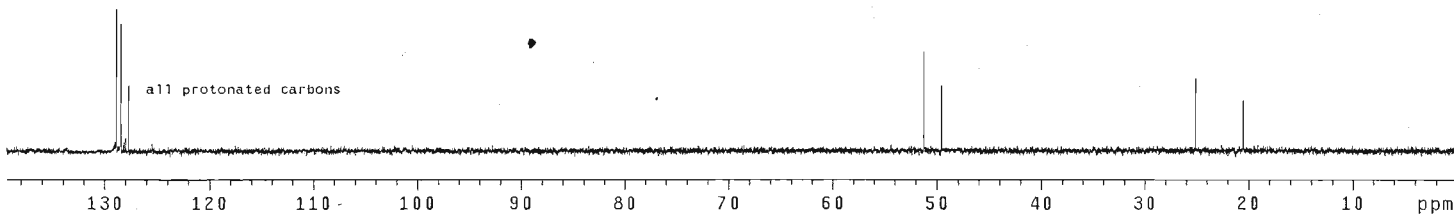th             4
ins      100.000
ai   cdc  ph

9

128.556
128.117
127.371
117.839
77.255
77.000
76.748
50.960
49.287
24.822
20.236

180     160     140     120     100      80      60      40      20     ppm

DHL-56b

Pulse Sequence: dept



CH3 carbons

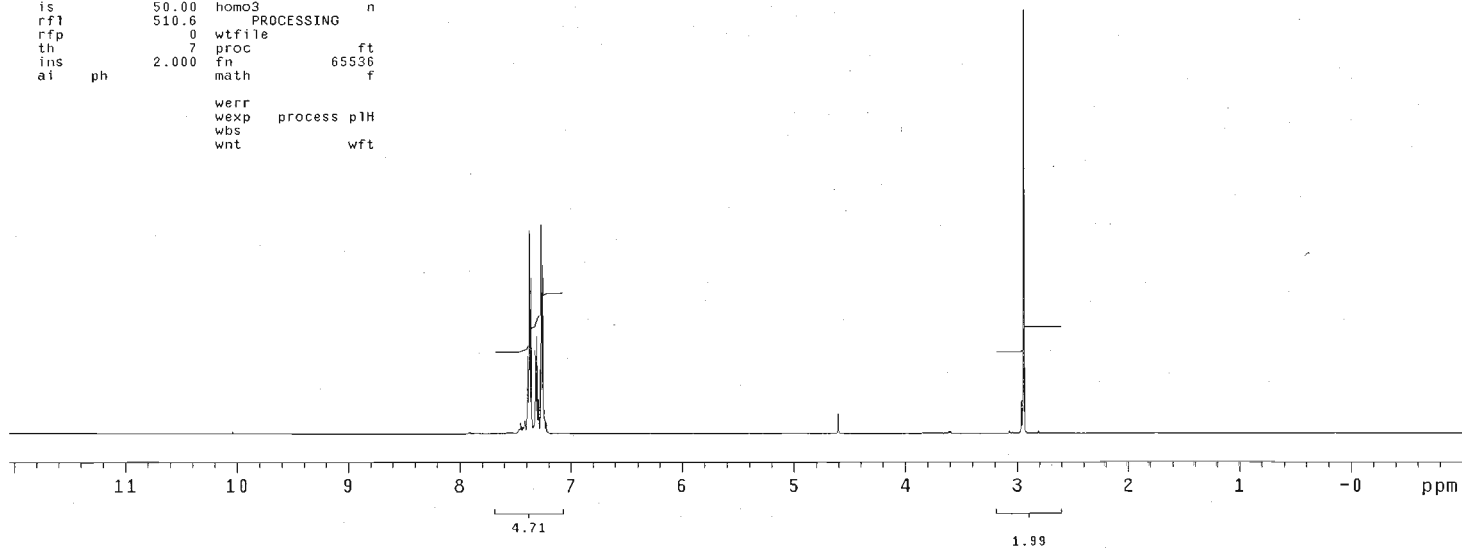CH2 carbons

CH carbons
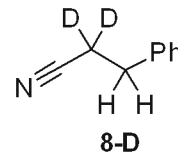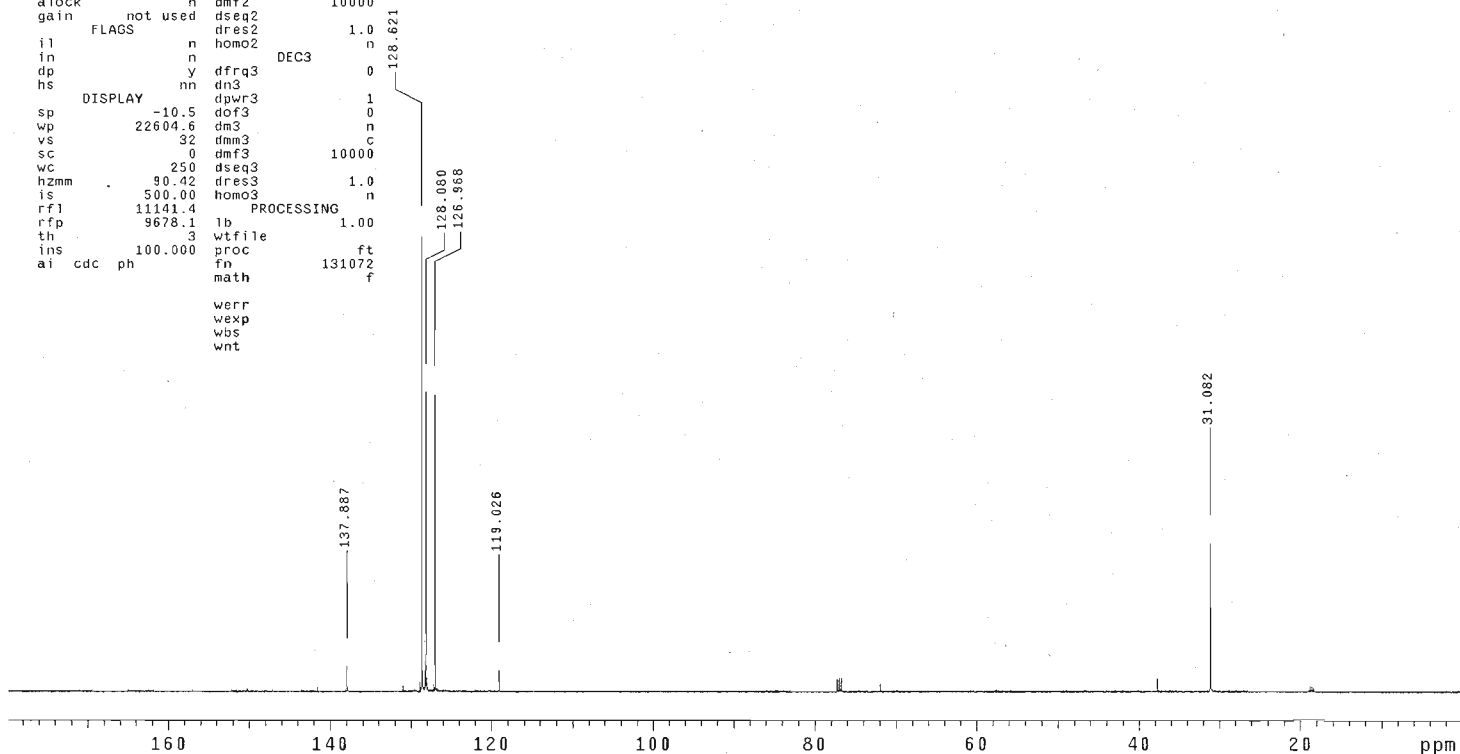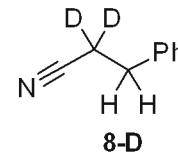
all protonated carbons

DHL-56D-2C

exp4  s2pul

```
          SAMPLE                DEC. & VT
date     Apr 20 2008   dfrq         499.864
solvent       CDCl3    dn               H1
file            exp    dpwr             30
      ACQUISITION      dof               0
sfrq      499.864      dm              nnn
tn             H1      dmm               c
at          5.016      dmf             200
np          65536      dseq
sw         6533.3      dres            1.0
fb           4000      homo              n
bs              4              DEC2
tpwr           61      dfrq2             0
pw           13.5      dn2
d1          0.100      dpwr2             1
tof         269.9      dof2              0
nt             16      dm2               n
ct             16      dmm2              c
alock           n      dmf2            200
gain     not used      dseq2
      FLAGS            dres2           1.0
il              n      homo2             n
in              n              DEC3
dp              y      dfrq3             0
hs             nn      dn3
      DISPLAY          dpwr3             1
sp         -510.4      dof3              0
wp         6533.1      dm3               n
vs             39      dmm3              c
sc              0      dmf3            200
wc            250      dseq3
hzmm        26.13      dres3           1.0
is          50.00      homo3             n
rfl         510.6         PROCESSING
rfp             0      wtfile
th              7      proc             ft
ins         2.000      fn            65536
ai       ph           math              f

                      werr
                      wexp    process p1H
                      wbs
                      wnt             wft
```

8-D

4.71

1.99

11  10  9  8  7  6  5  4  3  2  1  -0  ppm

DHL-56D-2C

exp2  s2pul

          SAMPLE                    DEC. & VT
date    Apr 20 2008   dfrq        499.864
solvent       CDCl3   dn              H1
file            exp   dpwr            40
    ACQUISITION       dof              0
sfrq      125.702     dm             yyy
tn            C13      dmm             w
at          1.215     dmf         8787.35
np          65536     dseq
sw         26963.3    dres           1.0
fb          15000     homo             n
bs              4             DEC2
tpwr           52     dfrq2            0
pw           10.2     dn2
d1          1.800     dpwr2            1
tof         144.5     dof2             0
nt            640     dm2              n
ct             64     dmm2             c
alock           n     dmf2         10000
gain     not used     dseq2
     FLAGS            dres2          1.0
il              n     homo2            n
in              n             DEC3
dp              y     dfrq3            0
hs             nn     dn3
    DISPLAY           dpwr3            1
sp          -10.5     dof3             0
wp        22604.6     dm3              n
vs             32     dmm3             c
sc              0     dmf3         10000
wc            250     dseq3
hzmm        90.42     dres3          1.0
is         500.00     homo3            n
rfl       11141.4        PROCESSING
rfp        9678.1     lb            1.00
th              3     wtfile
ins      100.000      proc            ft
ai   cdc  ph          fn          131072
                      math             f

                      werr
                      wexp
                      wbs
                      wnt

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128.621
128.080
126.968
137.887
119.026
31.082

160   140   120   100   80   60   40   20   ppm

DHL-56D-2C

exp2  s2pul

            SAMPLE                    DEC. & VT
date    Apr 20 2008      dfrq          499.864
solvent         CDC13    dn                 H1
file            exp      dpwr              40
     ACQUISITION         dof                0
sfrq        125.702      dm                yyy
tn              C13      dmm                 w
at            1.215      dmf           8787.35
np            65536      dseq
sw          26963.3      dres             1.0
fb            15000      homo               n
bs                4              DEC2
tpwr             52      dfrq2              0
pw             10.2      dn2
d1            1.800      dpwr2              1
tof           144.5      dof2               0
nt              640      dm2                n
ct               68      dmm2               c
alock             n      dmf2           10000
gain     not used       dseq2
      FLAGS              dres2            1.0
il                n      homo2              n
in                n              DEC3
dp                y      dfrq3              0
hs               nn      dn3
      DISPLAY            dpwr3              1
sp           2115.3      dof3               0
wp           2550.8      dm3                n
vs              433      dmm3               c
sc                0      dmf3           10000
wc              250      dseq3
hzmm          10.20      dres3            1.0
is           500.00      homo3              n
rfl         11141.4          PROCESSING
rfp          9678.1      lb               1.00
th                6      wtfile
ins         100.000      proc              ft
ai   cdc  ph             fn            131072
                         math               f

                         werr
                         wexp
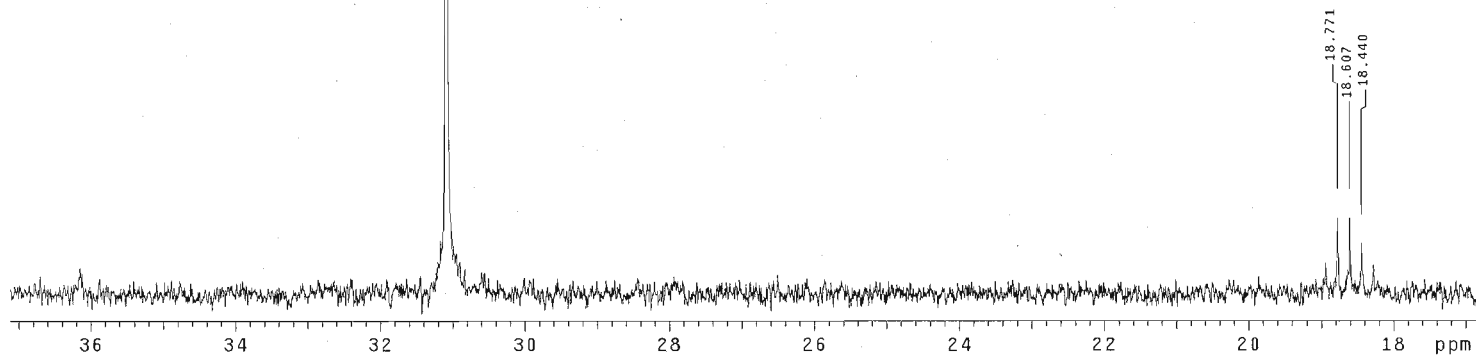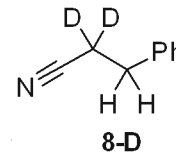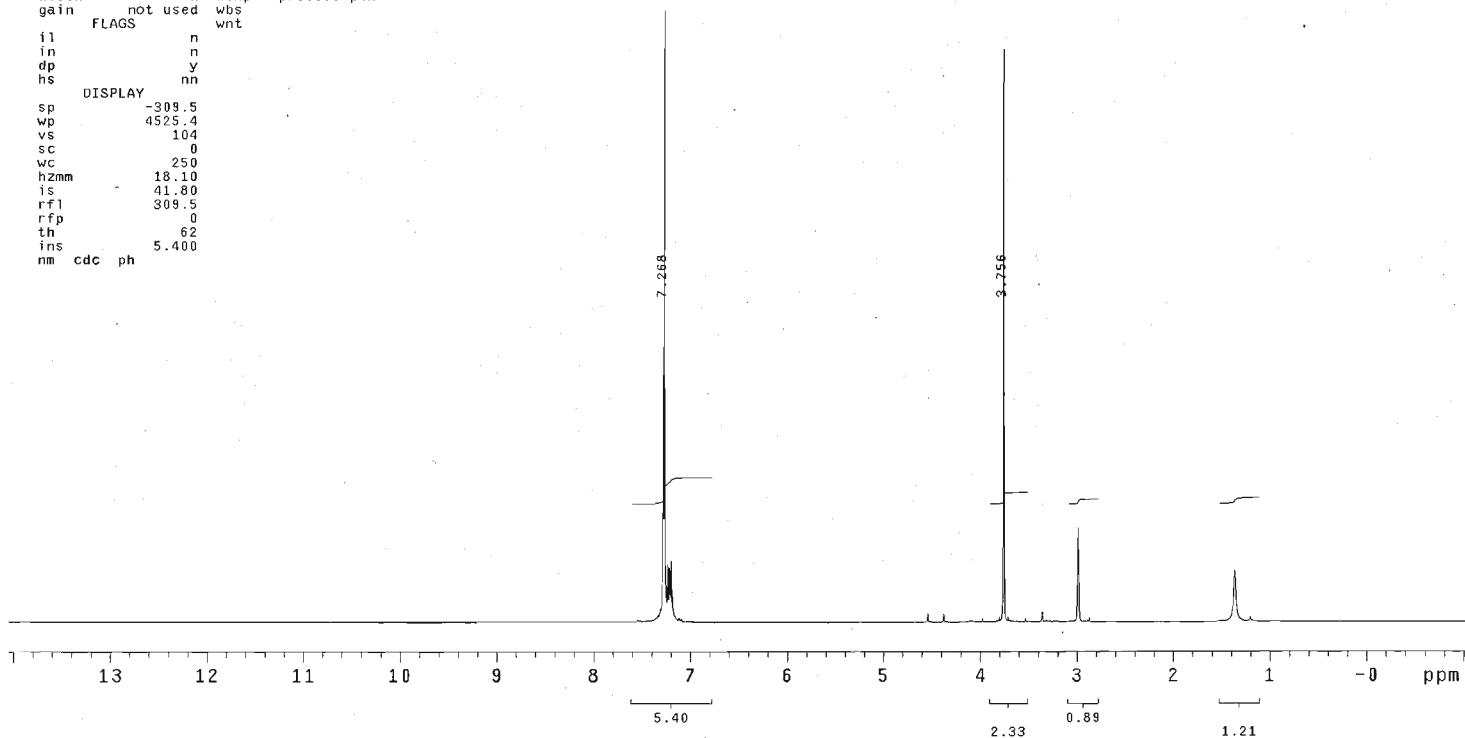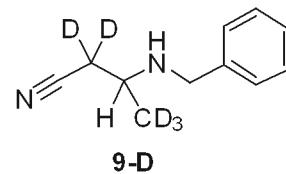                         wbs
                         wnt

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31.082

18.771
18.607
18.440

exp3   s2pul

```
          SAMPLE                  DEC. & VT
date     Apr 18 2008    dfrq        299.948
solvent        cdcl3    dn               H1
file             exp    dpwr             30
      ACQUISITION       dof               0
sfrq        299.949     dm              nnn
tn               H1     dmm               c
at            3.620     dmf             200
np            32768     dseq
sw           4525.4     dres            1.0
fb             2600     homo              n
bs                4        PROCESSING
tpwr             59     wtfile
pw             14.0     proc             ft
d1            0.380     fn            65536
tof           481.6     math              f
nt               16
ct               16     werr
alock             n     wexp     process p1H
gain       not used     wbs
       FLAGS            wnt
il                n
in                n
dp                y
hs               nn
       DISPLAY
sp           -309.5
wp           4525.4
vs              104
sc                0
wc              250
hzmm          18.10
is            41.80
rfl           309.5
rfp               0
th               62
ins           5.400
nm   cdc  ph
```
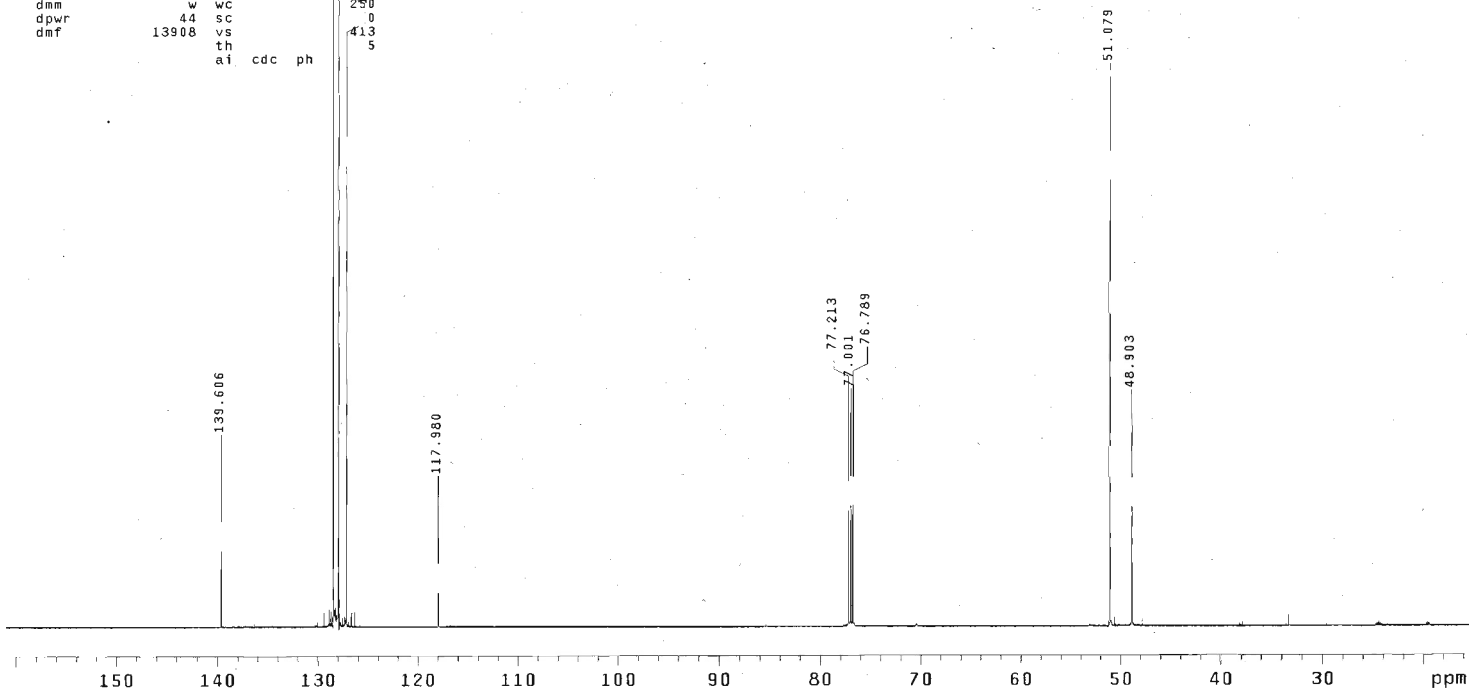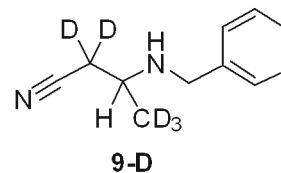
9-D



7.268

3.756

5.40

2.33   0.89   1.21

13  12  11  10  9  8  7  6  5  4  3  2  1  -0  ppm

DHL-56D-3C

exp2  Carbon

SAMPLE
date    Apr 25 2008
solvent       cdcl3
file            exp
ACQUISITION
sw        26041.7
at          1.783
np          92842
fb          17000
bs             16
d1          1.220
nt           1200
ct           1200
TRANSMITTER
tn            C13
sfrq      150.849
tof        -2982.6
tpwr           58
pw          7.500
DECOUPLER
dn             H1
dof             0
dm            yyy
dmm             w
dpwr           44
dmf         13908

SPECIAL
temp          22.0
gain      not used
spin      not used
hst          0.008
pw90         7.500
alfa        10.000
FLAGS
il              n
in              n
dp              y
hs             nn
PROCESSING
lb           0.50
fn         262144
DISPLAY
sp         2268.5
wp        22015.8
rfl       13333.2
rfp       11514.5
rp        -123.0
lp          11.6
PLOT
wc            250
sc              0
vs            413
th              5
ai    cdc  ph

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128.490
128.464
127.951

139.606

117.980

77.213
77.001
76.789

51.079

48.903

150  140  130  120  110  100  90  80  70  60  50  40  30  ppm

| SAMPLE | | SPECIAL | |
|---|---|---|---|
| date | Apr 25 2008 | temp | 22.0 |
| solvent | cdcl3 | gain | not used |
| file | exp | spin | not used |
| ACQUISITION | | hst | 0.008 |
| sw | 26041.7 | pw90 | 7.500 |
| at | 1.783 | alfa | 10.000 |
| np | 92842 | FLAGS | |
| fb | 17000 | il | n |
| bs | 16 | in | n |
| d1 | 1.220 | dp | y |
| nt | 1200 | hs | nn |
| ct | 1200 | PROCESSING | |
| TRANSMITTER | | lb | 0.50 |
| tn | C13 | fn | 262144 |
| sfrq | 150.849 | DISPLAY | |
| tof | -2982.6 | sp | 2124.7 |
| tpwr | 58 | wp | 2763.1 |
| pw | 7.500 | rfl | 13333.2 |
| DECOUPLER | | rfp | 11614.5 |
| dn | H1 | rp | -123.0 |
| dof | 0 | lp | 11.6 |
| dm | yyy | PLOT | |
| dmm | w | wc | 250 |
| dpwr | 44 | sc | 0 |
| dmf | 13908 | vs | 11217 |
| | | th | 12 |
| | | ai  cdc  ph | |



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Peaks: 24.581, 24.442, 24.302, 19.723, 19.596, 19.463