SUPPORTING INFORMATION for the article entitled

A mild method for the efficient, [3,3]-sigmatropic rearrangement of *N,O*-diacyl hydroxylamines

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Table of Contents:

General Methods

Melting points were determined using a hot stage melting point apparatus. Optical Rotations were obtained using an automatic polarimeter, using 1dm cell with chloroform as the solvent, at a wavelength of 589 nm (sodium D line), and are quoted as $[\alpha]_D$, concentration *c* (g/100 mL) and recorded at room temperature. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a 300 MHz or 400 MHz spectrometer. The ^{1}H spectra were run in deuterochloroform ($CDCl_3$) with δ 7.26 (residual $CHCl_3$) used as an internal reference. Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded at 75 MHz or at 100 MHz with a 300 MHz or 400 MHz spectrometer and were run in deuterochloroform ($CDCl_3$) solutions with δ 77.16 ($CDCl_3$ solvent resonance) used as an internal reference. Accurate mass determinations were made at high resolution on an accurate mass LC-TOF system; ESI Conditions: 6L/min N_2, 325 °C drying gas temp., capillary voltage: 3500v, Fragmentor voltage: 150v. Infrared (IR) spectra were recorded neat on a FTIR equipped with a diamond ATR (cm^{-1} scale) and MCT detector. Analytical thin layer chromatography (TLC) was performed on silica gel 60 F_{254}. Detection was by UV light or stained using potassium permanganate dip. Flash Chromatography was performed using silica gel 60 (230-400 mesh).

Dichloromethane was distilled over P_2O_5. Triethylamine (NEt_3) was distilled over sodium wire. Trimethylsilyl trifluoromethanesulfonate (TMSOTf) and other commercially available reagents were purchased and used as received. All reactions were carried out under an argon atmosphere.

N,*O*-diacyl hydroxylamine starting materials were prepared according to the literature.[1]

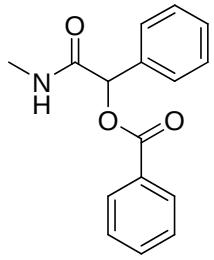
General [3,3] sigmatropic rearrangement procedure using a 1:1 ratio of TMSOTf to NEt_3: Preparation of 2-(methylamino)-2-oxo-1-phenylethyl benzoate (**6a**)

N-(benzoyloxy)-*N*-methyl-2-phenylacetamide (**5a**) (200 mg, 0.743 mmol) was dissolved in 3 mL dry DCM and the temperature lowered to -78 °C. To this, TMSOTf (135 μL, 0.743 mmol) was added, followed 5 min later by NEt_3 (104 μL, 0.743mmol). After stirring at this temperature for 30 min, the reaction was removed from the cooling bath, allowed to warm to room temperature, sealed and left to stir overnight. The reaction was quenched by pouring directly onto a silica gel plug and eluted with EtOAc. Solvent removal *in vacuo* left a white solid which upon spectroscopic analysis, proved to be pure **6a** (150 mg, 75%).

In all other examples where a mixture of products **6** and **7** were present, a crude ^{1}H NMR after the plug was used to determine product ratios. Then further column chromatography to separate the two products was undertaken.

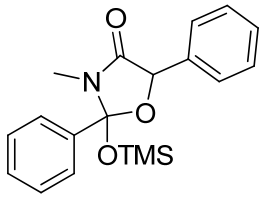
General [3,3] sigmatropic rearrangement procedure using a 2:2 ratio of TMSOTf to NEt_3: Preparation of 3-methyl-2,5-diphenyl-2-(trimethylsilyloxy)oxazolidin-4-one **(7a)**

N-(benzoyloxy)-*N*-methyl-2-phenylacetamide (**5a**) (100 mg, 0.371 mmol) was dissolved in 1.5 mL dry DCM and the temperature lowered to -78 °C. To this, TMSOTf (135 μL, 0.743 mmol) was added, followed 5 min later by NEt_3 (104 μL, 0.743 mmol). After stirring at this temperature for 30 min, the reaction was removed from the cooling bath, allowed to warm to room temperature, sealed and left to stir overnight. The reaction was quenched by pouring directly onto a silica gel plug and eluted with EtOAc. Solvent removal *in vacuo* left a white solid which upon spectroscopic analysis, showed a 10:1 mixture of **7a:6a** in a combined 90% yield. Column chromatography (20% EtOAc/Hexane) afforded **7a** as a white solid (102 mg, 80%).



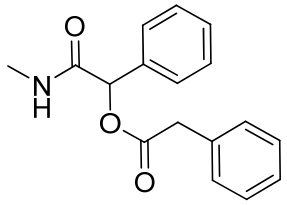
2-(methylamino)-2-oxo-1-phenylethyl benzoate (6a)

mp 142-143 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10-8.14 (2H, m), 7.28-7.65 (8H, m), 6.37 (1H, s), 6.20 (1H, brs), 2.90 (3H, d, *J*=4.8); ^{13}C NMR (75 MHz, $CDCl_3$) 169.1, 165.1, 135.8, 133.8, 130.0, 129.5, 128.9, 128.8, 128.8, 127.5, 76.1, 26.4; FT-IR ν_{max}/cm^{-1} 3311s, 3092w, 3065w, 3036w, 2949w, 1725vs, 1661vs, 1561s, 1496m, 1449m, 1315s, 1245vs, 1113s, 1025m, 985m, 708m, 683m; ESI-HRMS *m*/*z* 270.1124, 292.0947 ($C_{16}H_{15}NO_3 + H^+$ requires 270.1130, $C_{16}H_{15}NO_3 + Na^+$ requires 292.0950).



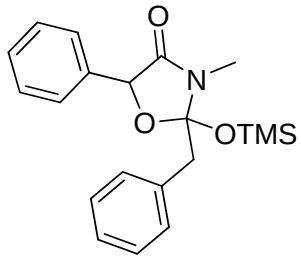
3-methyl-2,5-diphenyl-2-(trimethylsilyloxy)oxazolidin-4-one (7a)

mp 62-63 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.62 (10H, m), 5.52 (1H, s), 2.70 (3H, s), 0.22 (9H, s); ^{13}C NMR (100 MHz, $CDCl_3$) 169.5, 139.5, 135.5, 129.5, 128.9, 128.7, 128.7, 128.4, 127.0, 126.9, 126.8, 110.7, 78.6, 25.7, 1.2; FT-IR ν_{max}/cm^{-1} 3035w, 2957w, 1716s, 1424m, 1231m, 1090m, 1062m, 1035m, 869m, 839m, 760m, 698m; ESI-HRMS *m*/*z* 342.1519 ($C_{19}H_{23}NO_3Si + H^+$ requires 342.1525).



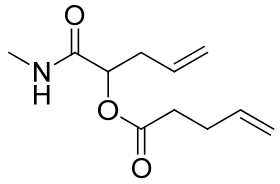
2-(methylamino)-2-oxo-1-phenylethyl 2-phenylacetate (6b)

mp 91-93 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.40 (10H, m), 6.10 (1H, s), 5.73 (1H, brs), 3.76 (2H, s), 2.7 (3H, d, *J*=4.8); ^{13}C NMR (100 MHz, $CDCl_3$) 169.5, 168.9, 135.5, 133.6, 129.4, 129.0, 129.0, 128.8, 127.6, 127.4, 75.7, 41.6, 26.1; FT-IR ν_{max}/cm^{-1} 3302s, 3092w, 3064w, 3032w, 2947w, 2917w, 1732s, 1663vs, 1566m, 1498m, 1453m, 1410m, 1355m, 1191m, 1155s, 866m; ESI-HRMS *m*/*z* 284.1281 ($C_{17}H_{17}NO_3$ + H^+ requires 284.1287).



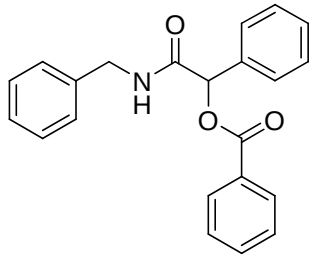
2-benzyl-3-methyl-5-phenyl-2-(trimethylsilyloxy)oxazolidin-4-one (7b)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16-7.26 (8H, m), 6.98-7.00 (2H, m), 5.25 (1H, s), 3.21 (2H, s), 2.97 (3H, s), 0.15 (9H, s); ^{13}C NMR (100 MHz, $CDCl_3$) 170.3, 135.4, 134.6, 131.0, 130.5, 128.7, 128.6, 128.5, 128.4, 127.5, 127.2, 127.1, 111.6, 79.2, 45.7, 26.2, 1.1; FT-IR $\nu_{max}/cm^{-}1$ 3089w, 3064m, 3032m, 2957m, 2930m, 2899m, 1719vs, 1453m, 1432s, 1395s, 1252s, 1162m, 1087s, 1070s, 1017m, 840s, 697m, 638m; ESI-HRMS *m*/*z* 356.1674 ($C_{20}H_{25}NO_3Si$ + H^+ requires 356.1682).



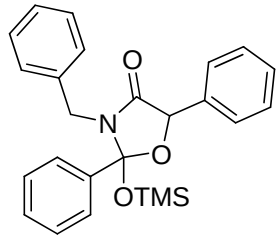
1-(methylamino)-1-oxopent-4-en-2-yl hex-5-enoate (6c)

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.08 (1H, br), 5.84 (1H, dddd, *J*=6.4, 10.2, 12.5, 16.7), 5.71 (1H, dddd, *J*=7.1, 10.2, 14.2, 17.1), 5.27 (1H, dd, *J*=4.7, 7), 5.06-5.13 (4H, m), 2.82 (3H, d, *J*=3.8), 2.65-2.71 (1H, m), 2.53-2.60 (1H, m), 2.48-2.52 (2H, m), 2.40-2.44 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) 171.6, 169.8, 136.7, 132.4, 118.9, 116.0, 73.2, 36.4, 33.6, 28.9, 26.0; FT-IR ν_{max}/cm^{-1} 3306br, 3080m, 2980m, 2942m, 1742vs, 1660vs, 1543s, 1370m, 1243m, 1160m, 918m, 843m; ESI-HRMS *m*/*z* 212.1282 ($C_{11}H_{17}NO_3 + H^+$ requires 212.1287).



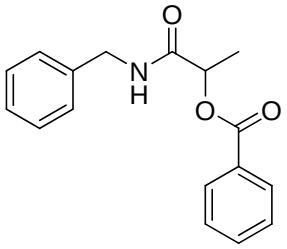
2-(benzylamino)-2-oxo-1-phenylethyl benzoate (6d)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21–8.19 (15H, m), 6.46 (1H, br), 6.39 (1H, s), 4.56 (1H, dd, *J*=5.8, 15.2), 4.48 (1H, dd, *J*=5.9, 15.2).



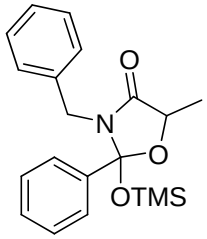
3-benzyl-2,5-diphenyl-2-(trimethylsilyloxy)oxazolidin-4-one (7d)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00–7.61 (15H, m) 5.60 (1H, s), 4.43 (1H, d, *J*=15.2 Hz), 4.21 (1H, d, *J*=15.2 Hz), 0.88 (s, 9H).



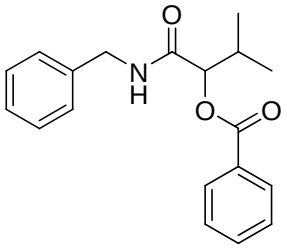
1-(benzylamino)-1-oxopropan-2-yl benzoate (6e)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24–8.05 (10H, m), 6.46 (1H, br), 5.54 (1H, q, *J*=6.9), 4.51 (2H, dd, *J*=2.0, 5.9), 1.65 (3H, d, *J*=6.6).



3-benzyl-5-methyl-2-phenyl-2-(trimethylsilyloxy)oxazolidin-4-one (7e)[2]

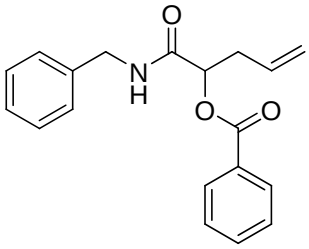
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99–7.48 (10H, m), 4.71 (1H, q, *J*=6.7), 4.39 (1H, d, *J*=15.2), 4.10 (1H, d, *J*=15.2), 1.63 (3H, d, *J*=6.8), 0.83 (9H, s).



1-(benzylamino)-3-methyl-1-oxobutan-2-yl benzoate (6f)

mp 112-113 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10-8.13 (1H, m), 8.06-8.08 (2H, m), 7.58-7.63 (1H, m), 7.45-7.49 (3H, m), 7.24-7.33 (3H, m), 5.38 (1H, d, *J*=4), 4.56 (1H, dd, *J*=6, 14.8), 4.44 (1H, dd, *J*=6, 14.8), 2.49-2.53 (1H, m), 1.07 (6H, dd, *J*=7.2, 10.4); ^{13}C NMR (100 MHz, $CDCl_3$) 169.5, 165.7, 138.0, 133.8, 130.3, 129.9, 129.5, 128.9, 128.8, 128.6, 127.7, 127.7, 78.8, 43.4, 31.0, 19.1, 17.2; FT-IR ν_{max}/cm^{-1} 3257m, 3090m,

2974m, 2936m, 1719s, 1655s, 1562m, 1430m, 1293m, 1248s, 1116m, 992m, 741m, 708m, 677m; ESI-HRMS *m*/*z* 312.1597 ($C_{19}H_{21}NO_3 + H^+$ requires 312.1600).

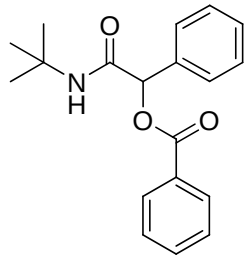


1-(benzylamino)-1-oxopent-4-en-2-yl benzoate (6g)

mp 103-104 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (1H, d, *J*=7.12), 8.03 (1H, d, *J*=7.8), 7.25-7.63 (8H, m), 6.40 (1H, br), 5.83 (1H, dddd, *J*=7.0, 10.2, 14.2, 17.1), 5.56 (1H, dd, *J*=4.9, 6.8), 5.18 (1H, dm, *J*=17.1), 5.12 (1H, dm, *J*=10.2), 4.56 (1H, dd, *J*=6, 15), 4.46 (1H, dd, *J*=6, 15), 2.74-2.88 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2, 165.3, 137.8, 133.7, 132.1, 130.2, 129.8, 129.3, 128.8, 128.5, 127.6, 119.2, 73.67, 43.3, 36.4; FT-IR ν_{max}/cm^{-1} 3306bdm, 3066m, 3033m, 2930m, 1722vs, 1662s, 1602w, 1539m, 1452m, 1316w, 1267s, 1176w, 1110m, 1070m, 1027w, 922w, 711m; ESI-HRMS *m*/*z* 310.1440 ($C_{19}H_{19}NO_3 + H^+$ requires 310.1443).

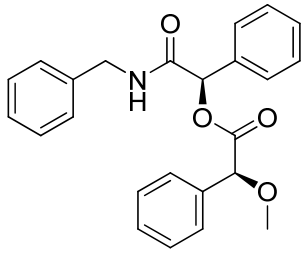
Preparation of 2-(*tert*-butylamino)-2-oxo-1-phenylethyl benzoate (6h):

N-(benzoyloxy)-*N*-*tert*-butyl-2-phenylacetamide (40 mg, 0.128 mmol) was dissolved in 1 mL dry DCM and the temperature lowered to -78 °C. To this, TMSOTf (47 μL, 0.257 mmol) was added, followed 5 min later by NEt_3 (36 μL, 0.257 mmol). After stirring at this temperature for 30 min, the reaction was warmed to room temperature, then heated to reflux and left overnight. The reaction was quenched by pouring directly onto a silica gel plug and eluted with EtOAc. Solvent removal *in vacuo* left an oil which upon spectroscopic analysis, showed approximately 25% conversion to **7h**. A pure sample of **7h** could not be obtained, even after extensive chromatography. Thus spectroscopic data reported here are for an impure sample with only selected resonances included (see scanned spectra for full details).



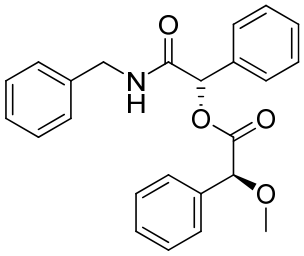
2-(*tert*-butylamino)-2-oxo-1-phenylethyl benzoate (6h)

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27-8.10 (10H, m), 6.22 (1H, s), 5.97 (1H, brs), 1.37 (9H, s); ^{13}C NMR ($CDCl_3$, 100 MHz) 167.3, 164.9, 76.1, 51.6, 28.7; ESI-HRMS *m*/*z* 312.1594 ($C_{19}H_{22}NO_3 + H^+$ requires 312.1600).



(S)-((R)-2-(benzylamino)-2-oxo-1-phenylethyl) 2-methoxy-2-phenylacetate (9)

mp 77-79 ºC; $[\alpha]_D^{25}$= -40.0° (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.32 (13H, m), 7.14-7.16 (2H, m), 6.20 (1H, br), 6.13 (1H, s), 4.89 (1H, s), 4.37 (2H, d, *J*=5.8), 3.40 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) 169.0, 168.0, 137.7, 135.7, 135.1, 129.1, 129.1, 128.9, 128.9, 128.8, 127.8, 127.2, 127.0, 82.83, 76.0, 57.8, 43.5; FT-IR ν_{max}/cm^{-1} 3297br, 3063m, 3031m, 2928s, 2829w, 1751vs, 1661vs, 1530m, 1495m, 1453m, 1358w, 1259m, 1166s, 1103s, 1028m, 731m, 695s; ESI-HRMS *m*/*z* 390.1699 ($C_{24}H_{23}NO_4 + H^+$ requires 390.1705).



(S)-((S)-2-(benzylamino)-2-oxo-1-phenylethyl) 2-methoxy-2-phenylacetate (10)

$[\alpha]_D^{25}$ = 58.9° (*c* 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.28-7.41 (12H, m), 7.20-7.23 (3H, m), 6.23 (1H, s), 5.80 (1H, br), 4.83 (1H, s), 4.24 (1H, dd, *J*=6, 14.8), 4.15 (1H, dd, *J*=6, 14.8), 3.38 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) 168.6, 168.0, 137.6, 135.9, 135.2, 129.2, 128.9, 128.8, 127.8, 127.7, 127.5, 127.2, 82.4, 75.6, 57.6, 43.3; FT-IR ν_{max}/cm^{-1} 3414s, 3308br, 3064m, 3032m, 2930w, 2830s, 1754vs, 1668vs, 1603s, 1527s, 1496s, 1454s, 1359s, 1243s, 1167s, 1109s, 1029w, 1002w, 734m, 697m; ESI-HRMS *m*/*z* 390.1702 ($C_{24}H_{23}NO_4 + H^+$ requires 390.1705).

Procedure for diastereomeric product determination

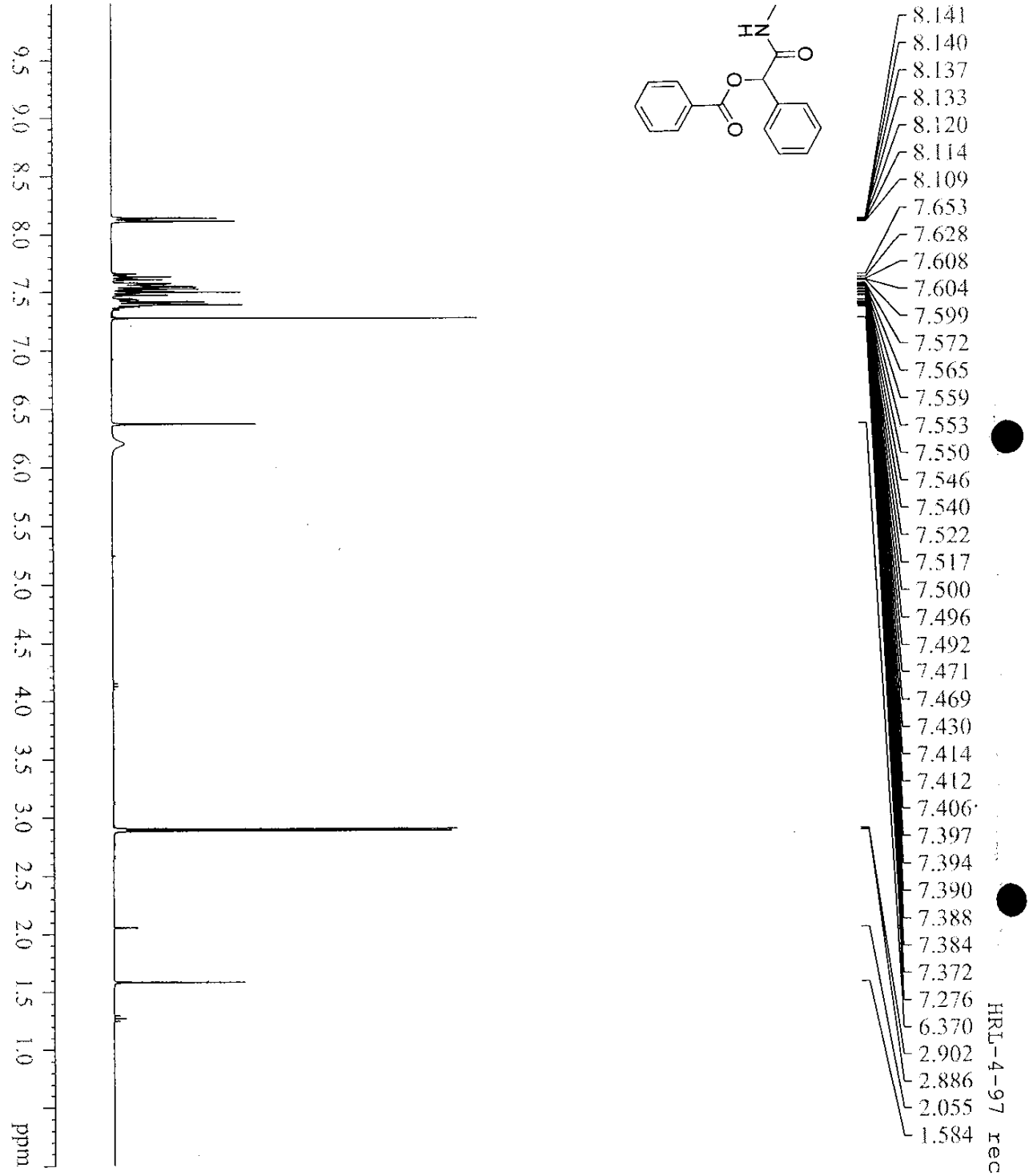
N-benzyl-(*S*)-mandelamide (100 mg, 0.414 mmol), (*S*)-(*O*-methyl)mandelic acid (69 mg, 0.414 mmol), EDCI.HCl (240 mg, 1.243 mmol) and DMAP (152 mg, 1.243 mmol) were all combined and dissolved in 3 mL DCM. The reaction was left stirring overnight at room temperature, then quenched with water. The layers were separated, and the organics washed with 1M HCl, then brine. The solvent was removed *in vacuo* and the crude material was purified using column chromatography (40% EtOAc/Hexane) to give **10** in 85% yield. Spectral data was identical with that of the rearranged material.

References

1. Clark, A. J.; Al-Faiyaz, Y. S. S.; Broadhurst, M. J. Patel, D.; Peacock, J. L. *J. Chem. Soc.*, *Perkin Trans. 1.* **2000**, 1117-1127.

2. Kamimura, A.; Omata, Y.; Kakehi, A.; Shirai, M. *Tetrahedron*. **2002**, *58*, 8763-8770.

^{1}H NMR (300 MHz, $CDCl_3$) **6a**

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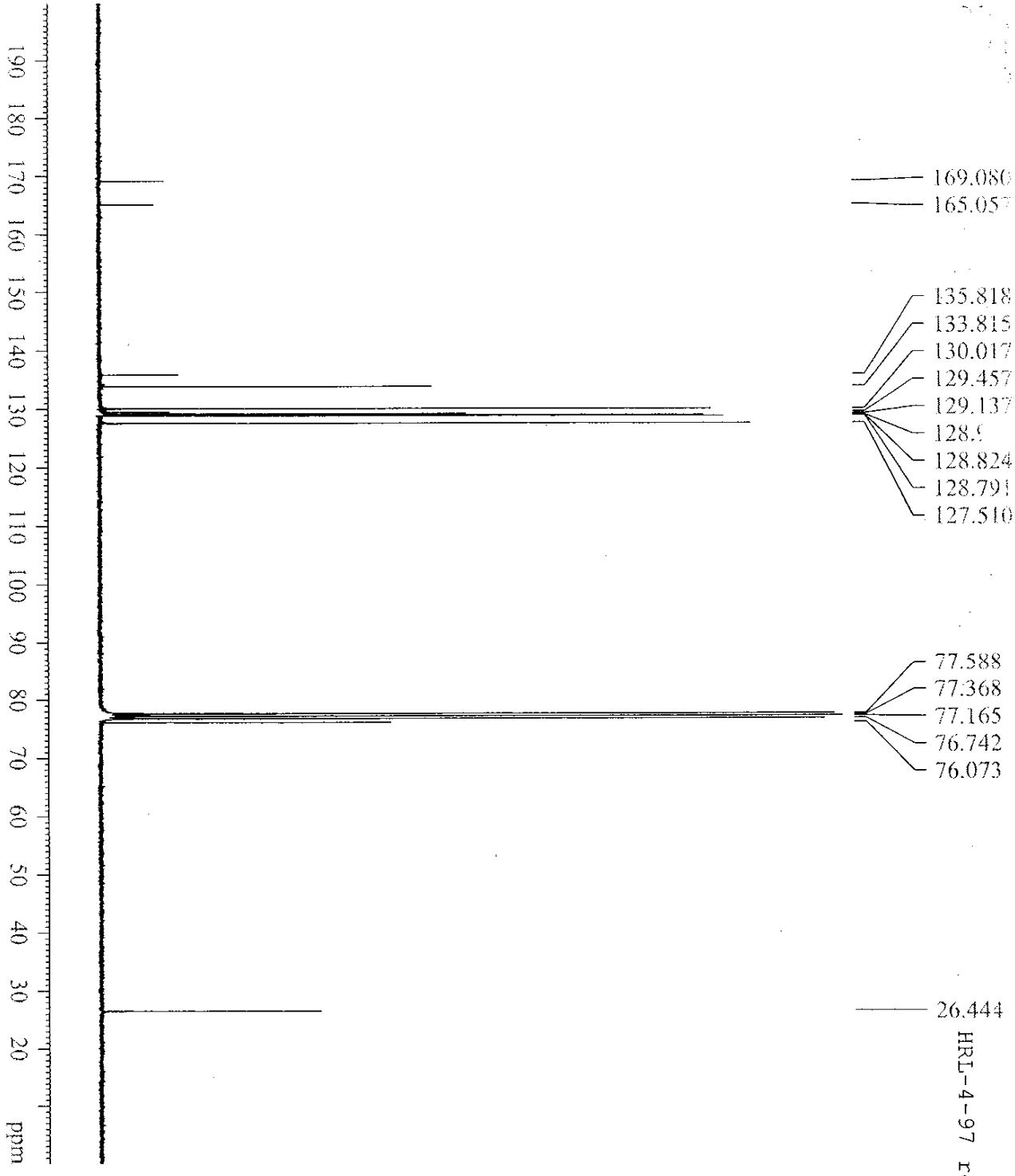
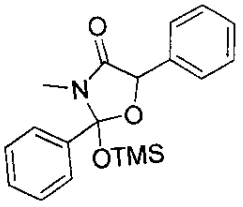
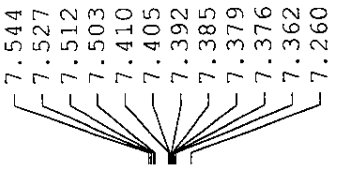
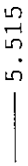
^{13}C NMR (75 MHz, $CDCl_3$) **6a**

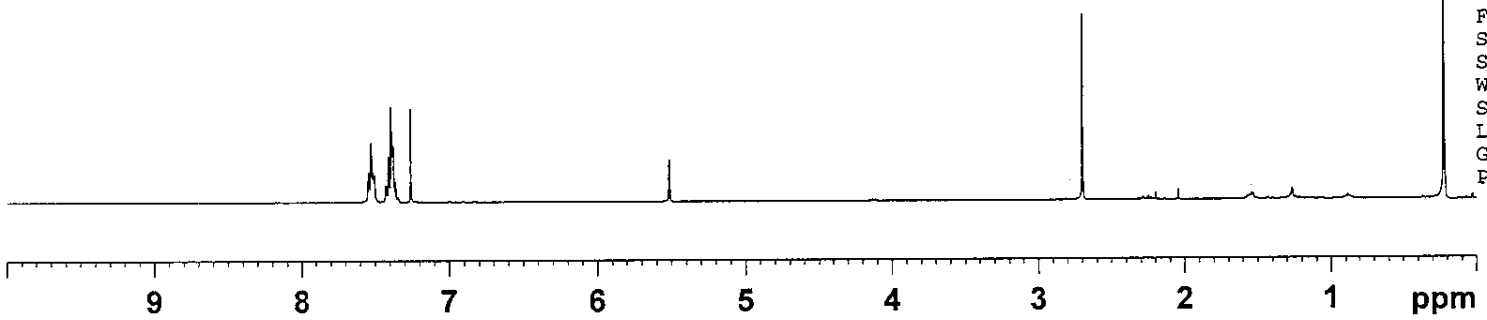






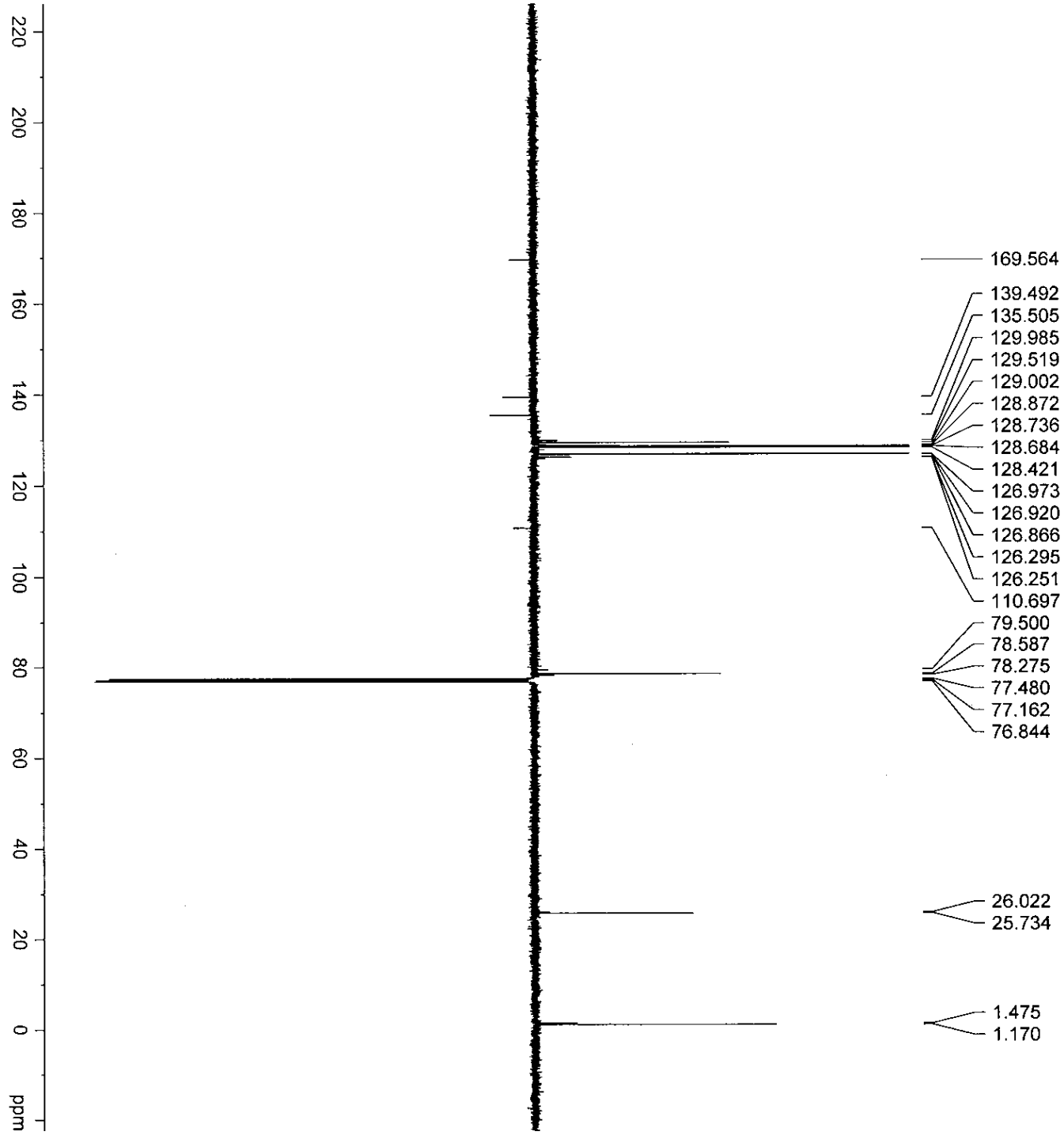


^{1}H NMR (400 MHz, $CDCl_3$) **7a**




```
Current Data Parameters
NAME                  hrl-4-98
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090330
Time                     16.59
INSTRUM                  spect
PROBHD    5 mm 5mm 1H/2H
PULPROG                    zg
TD                      48076
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 4807.692 Hz
FIDRES              0.100002 Hz
AQ                  4.9999542 sec
RG                        114
DW                    104.000 usec
DE                     14.56 usec
TE                     303.2 K
D1                 5.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      8.00 usec
PL1                     1.95 dB
PL1W                9.58740711 W
SFO1               400.1322007 MHz

F2 - Processing parameters
SI                      32768
SF                 400.1300099 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```

^{13}C NMR (100 MHz, $CDCl_3$) **7a**



HRL-4-98 2eq.

Current Data Parameters
NAME hrl-4-98
EXPNO 2
PROCNO 1

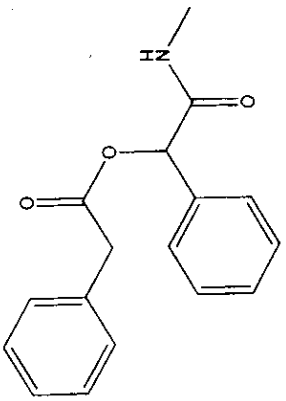
F2 - Acquisition Parameters
Date_ 20090330
Time_ 17.05
INSTRUM spect
PROBHD 5 mm 5mm 1H/2H
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 7268
DS 4
SWH 25000.000 Hz
FIDRES 0.381470 Hz
AQ 1.3107700 sec
RG 2050
DW 20.000 usec
DE 15.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 6.00000000 sec
D20 0.00689655 sec
TD0 1

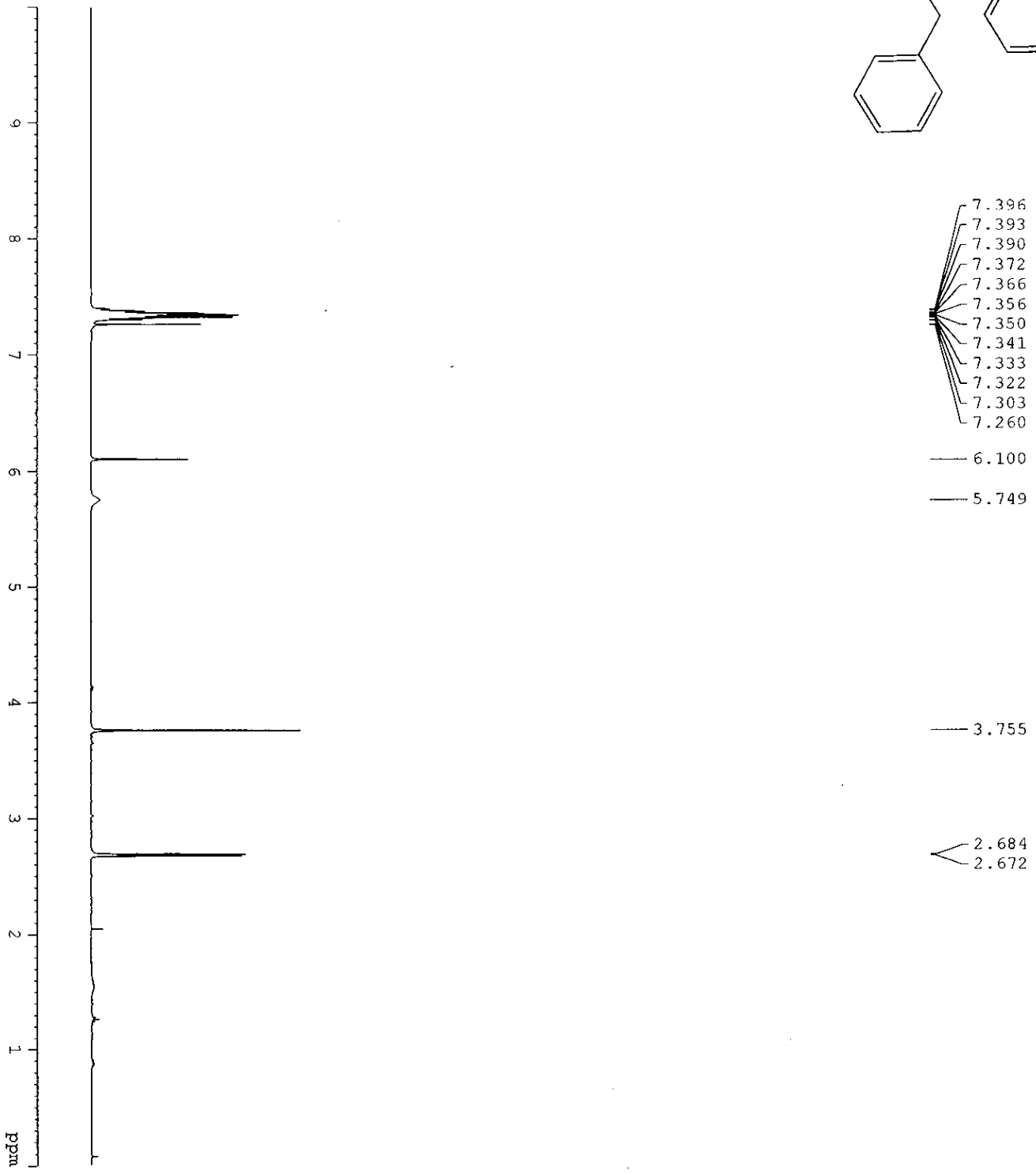
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
P2 24.40 usec
PL1 0.00 dB
PL1W 95.45168304 W
SFO1 100.6230043 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 2.00 dB
PL12 23.16 dB
PL2W 9.47766113 W
PL12W 0.07256065 W
SFO2 400.1320000 MHz

F2 - Processing parameters
SI 65536
SF 100.6127525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

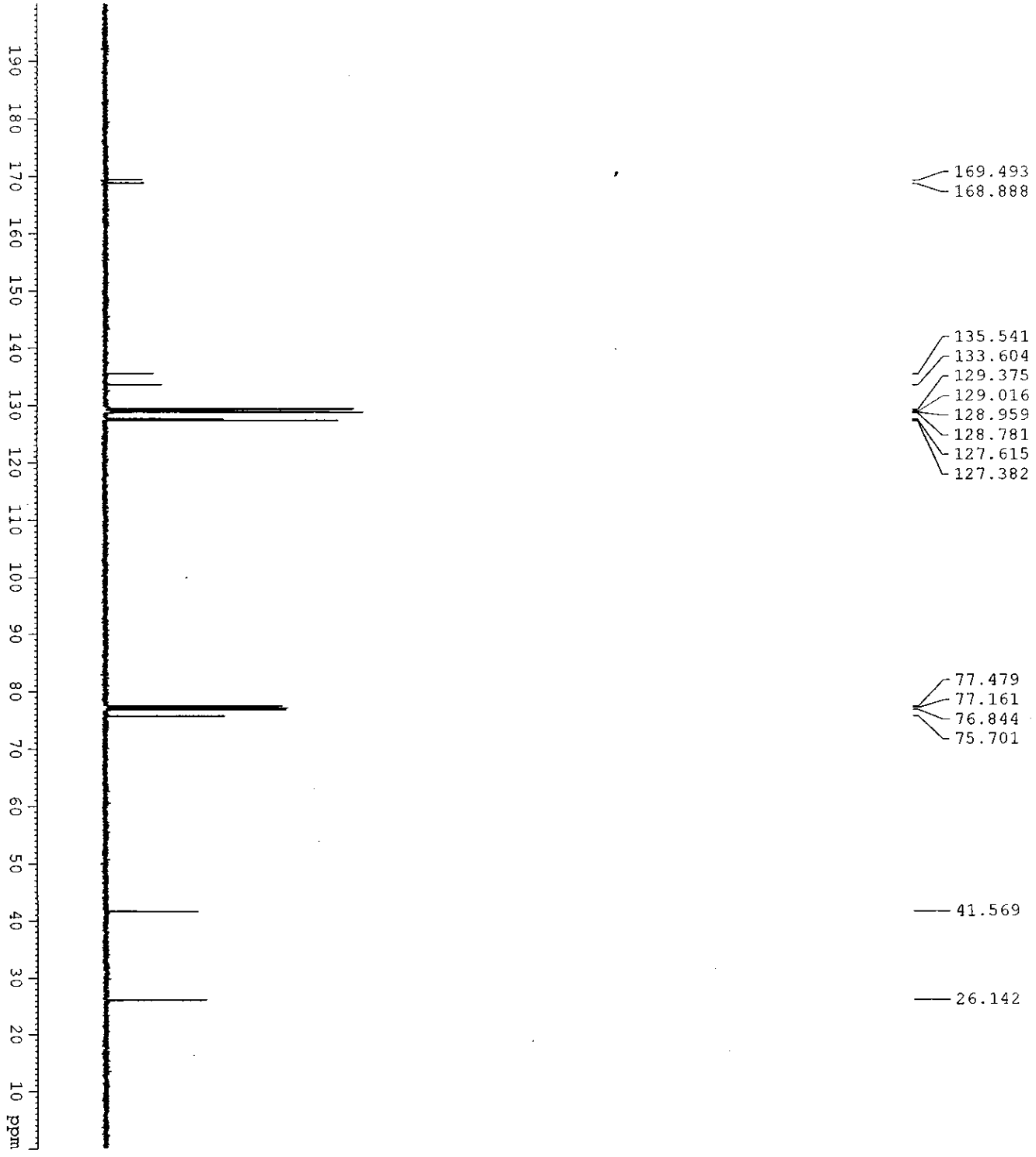
^{1}H NMR (400 MHz, $CDCl_3$) **6b**



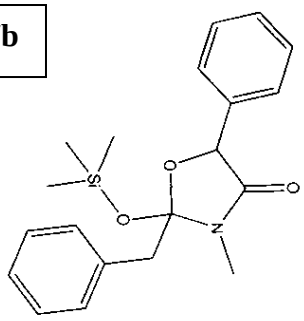



```
Avance III -1H Spectrum

NAME                  hrl-5-35
EXPNO                        1
PROCNO                       1
Date_              20090723
Time_                    13.50
INSTRUM                  spect
PROBHD     5 mm 5mm 1H/2H
PULPROG                   zg
TD                       48076
SOLVENT                  CDCl3
NS                          24
DS                           2
SWH                 4807.692 Hz
FIDRES              0.100002 Hz
AQ                  4.9999542 sec
RG                         144
DW                  104.000 usec
DE                   14.56 usec
TE                       303.2 K
D1                  5.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                   8.00 usec
PL1                  1.95 dB
PL1W                 9.58740711 W
SFO1                400.1322007 MHz
SI                       32768
SF                  400.1300099 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00
```

^{13}C NMR (100 MHz, $CDCl_3$) **6b**



^{1}H NMR (400 MHz, $CDCl_3$) **7b**



7.278
7.265
7.260
7.254
7.250
7.242
7.238
7.236
7.229
7.224
7.222
7.215
7.214
7.211
7.182
7.177
7.173
7.165
7.163
7.158
7.013
7.011
7.010
6.995
6.994
6.991
6.989
5.247

3.210
3.208
3.151
2.962
2.954

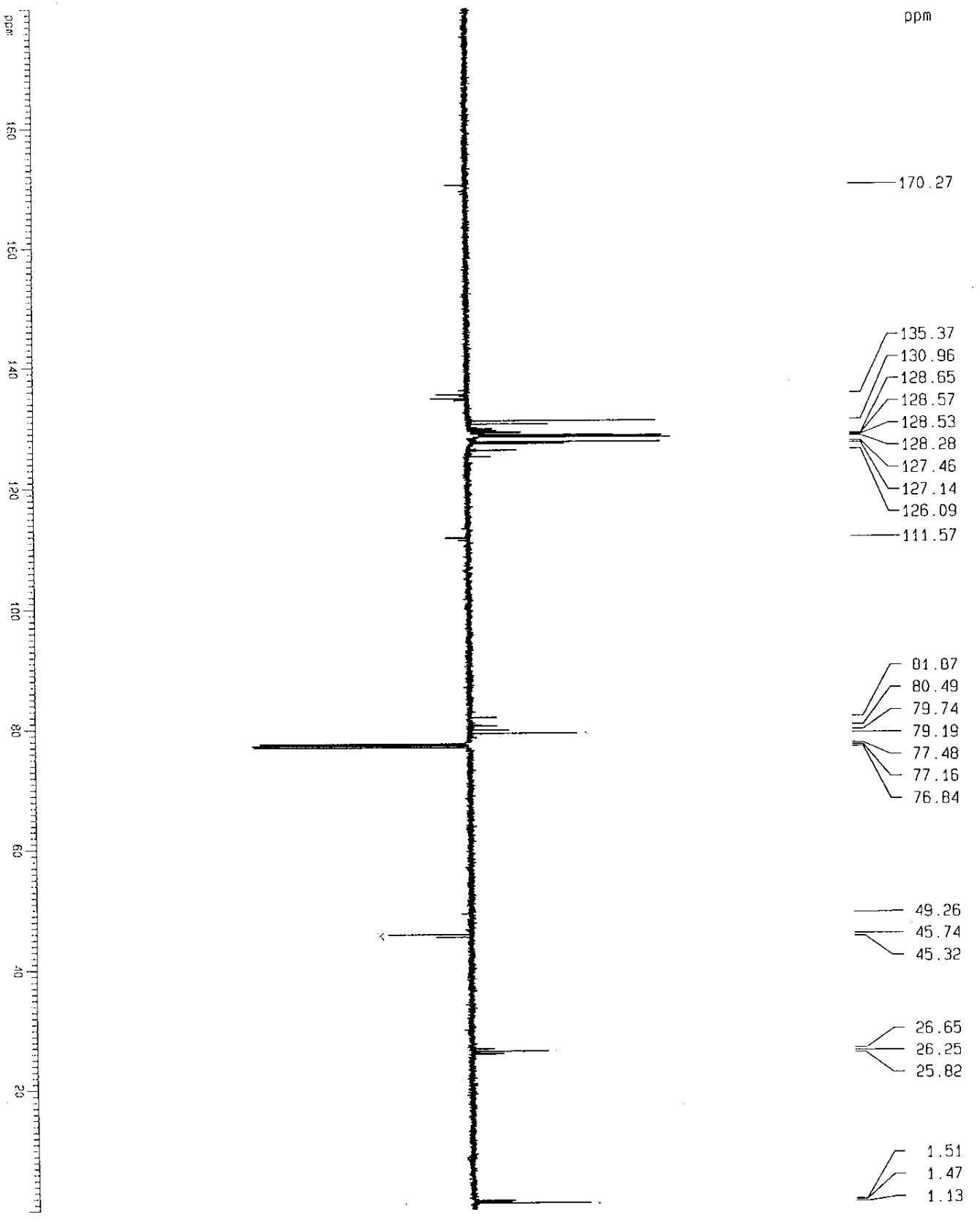
1.537

0.163
0.158

Avance III -1H Spectrum

```
NAME                 hrl-5-39c
EXPNO                        4
PROCNO                       1
Date_                20090728
Time_                   20.36
INSTRUM                  spect
PROBHD     5 mm Smm 1H/2H
PULPROG                   zg
TD                       48076
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                  4807.692 Hz
FIDRES               0.100002 Hz
AQ                   4.9999542 sec
RG                         161
DW                   104.000 usec
DE                    14.56 usec
TE                    303.2 K
D1                   5.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                    8.00 usec
PL1                   1.95 dB
PL1W             9.58740711 W
SFO1             400.1322007 MHz
SI                       32768
SF               400.1300098 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.00
```

^{13}C NMR (100 MHz, $CDCl_3$) **7b**



Current Data Parameters
NAME hrl-3-99-4hNO-plug
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080522
Time 7.59
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG imod
TD 65536
SOLVENT
NS 3230
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 9195.2
DW 19.900 usec
DE 15.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 6.00000000 sec
d20 0.00689655 sec
DELTA 0.00001477 sec
NCREST 0.00000000 sec
MCWRK 0.01500000 sec

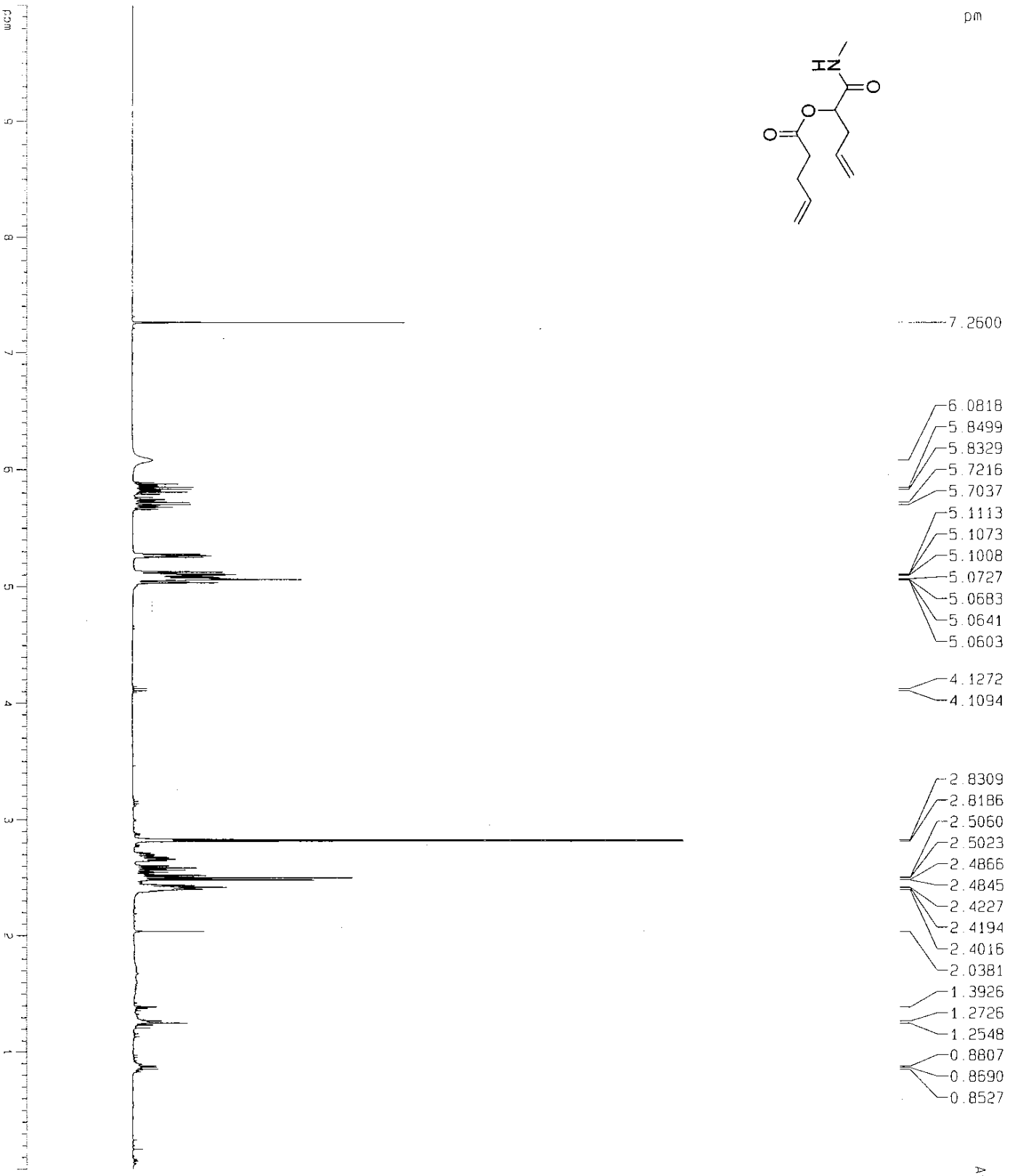
======== CHANNEL f1 ========
NUC1 13C
P1 11.60 usec
p2 23.20 usec
PL1 0.00 dB
SF01 100.6229643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 28.00 dB
SF02 400.1320000 MHz

F2 - Processing parameters
SI 65536
SF 100.6127535 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 4.04 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 9.09091 ppm/c
HZCM 914.66144 Hz/cm

^{1}H NMR (400 MHz, $CDCl_3$) **6c**



Avance DRX400 NMR Spectrometer

Current Data Parameters
NAME HRL-3-91mix-9-12
EXPNO 1
PROCNO 1

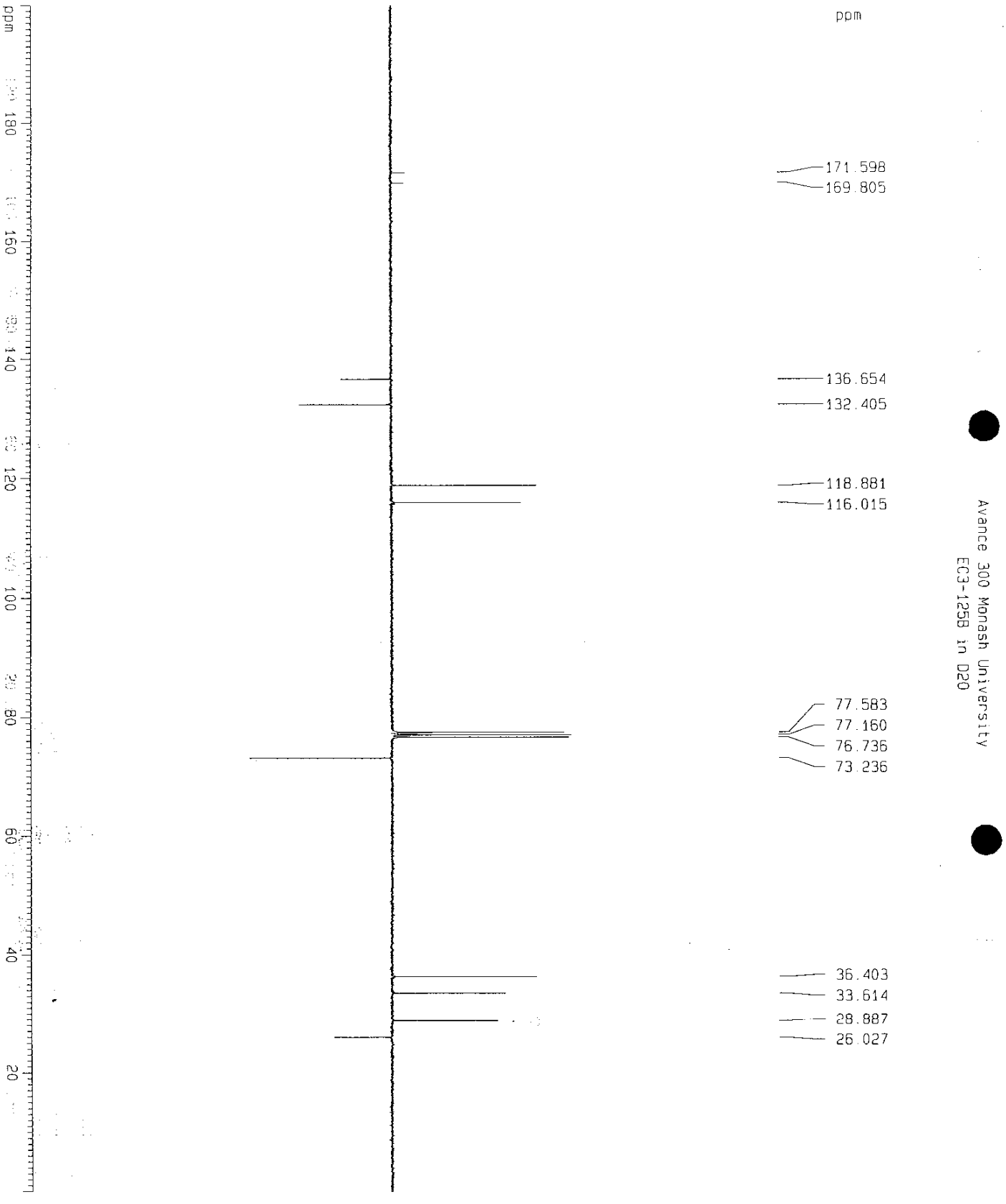
F2 - Acquisition Parameters
Date_ 20080514
Time 20.58
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 322.5
DW 104.400 usec
DE 15.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 4.00 dB
SFO1 400.1320006 MHz

F2 - Processing parameters
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.50 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.45455 ppm/cm
HZCM 181.87727 Hz/cm

^{13}C NMR (100 MHz, $CDCl_3$) **6c**



Current Data Parameters
NAME hrl-3-91mix-9-12
EXPNO 3
PROCNO 1

F2 - Acquisition Parameter
Date_ 20080514
Time 21.23
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG jmod
TD 32768
SOLVENT DMSO
NS 10936
DS 4
SWH 18382.354 Hz
FIDRES 0.560985 Hz
AQ 0.8913396 sec
RG 13004
DW 27.200 usec
DE 20.00 usec
TE 304.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
d20 0.00689655 sec
DELTA 0.00001082 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

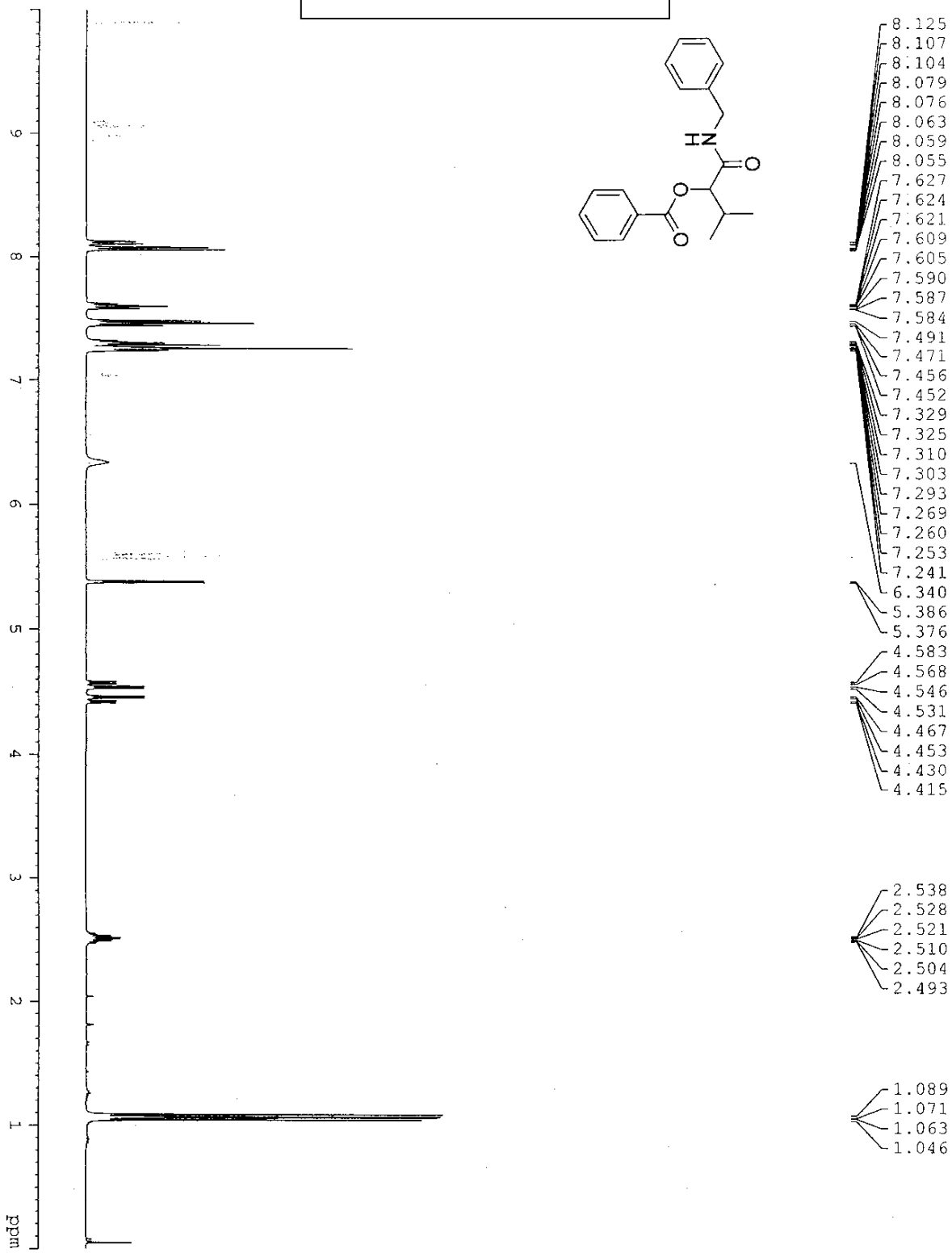
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
p2 17.00 usec
PL1 0.00 dB
SFO1 75.4760145 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.00 dB
SFO2 300.1314256 MHz

F2 - Processing parameters
SI 32768
SF 75.4677366 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 3.50 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.69565 ppm/cm
HZCM 656.24115 Hz

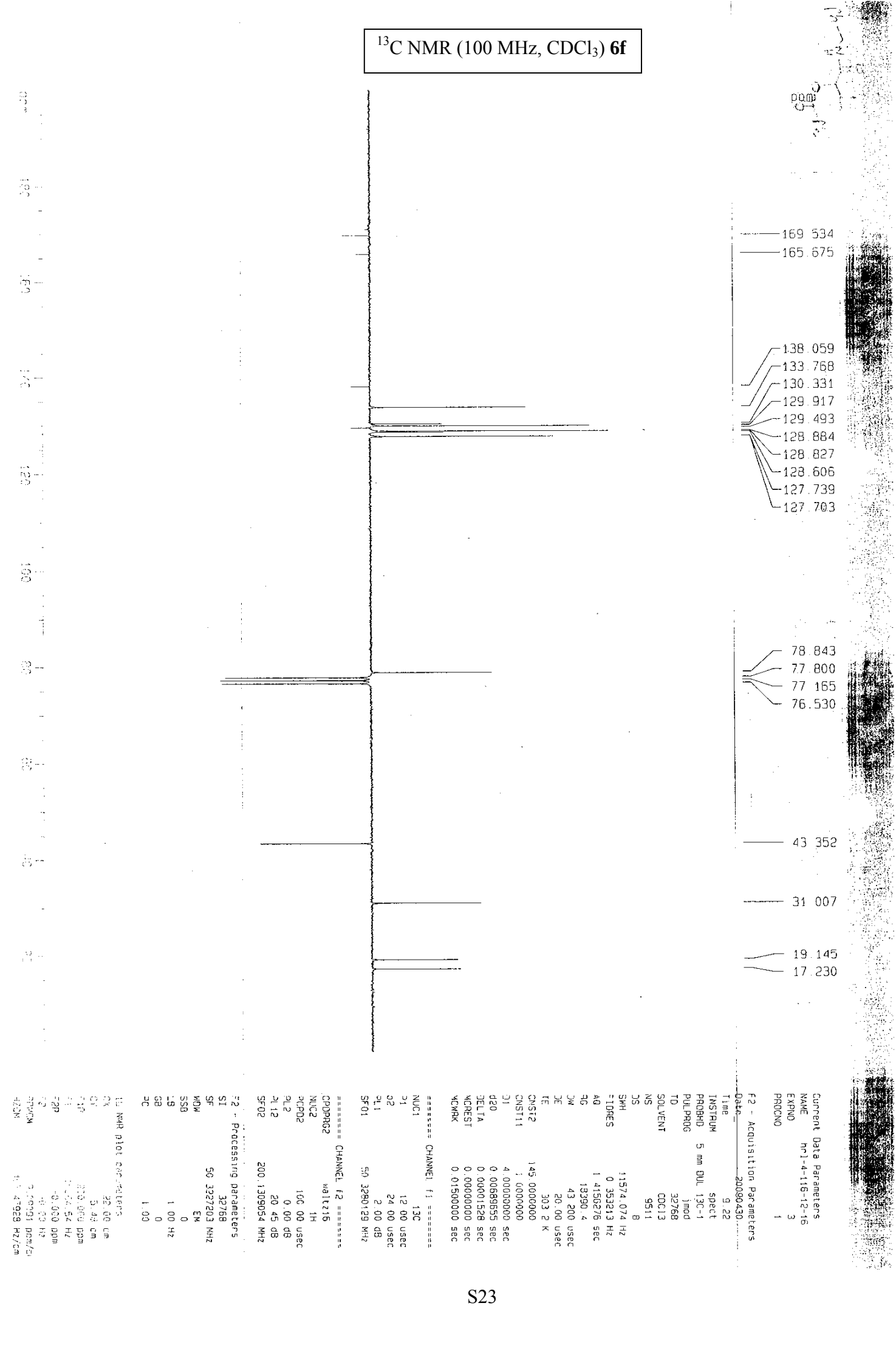
^{1}H NMR (400 MHz, $CDCl_3$) **6f**



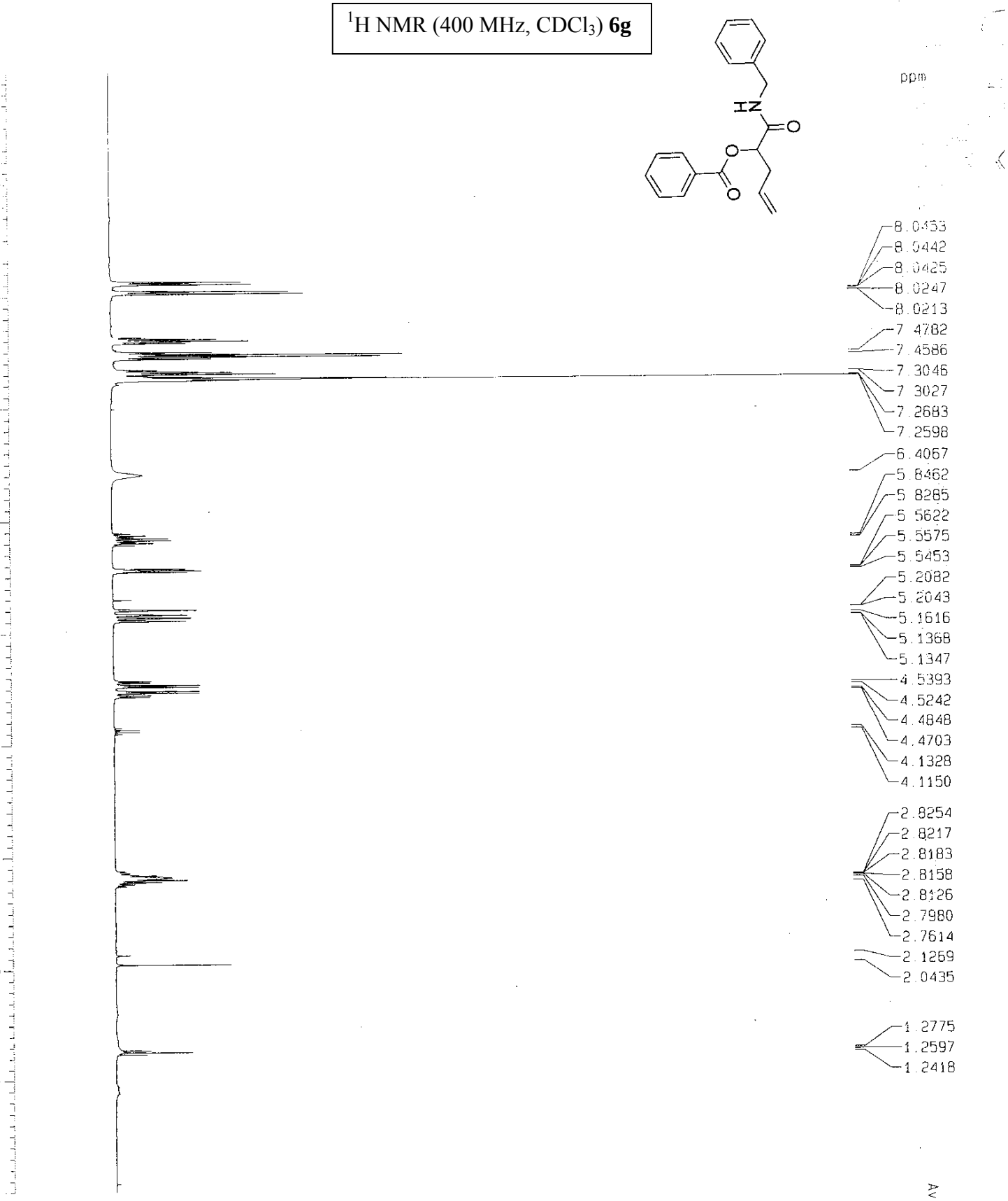

```
NAME                 hrl-4-116-12-16
EXPNO                             1
PROCNO                            1
Date_                      20090429
Time_                         18.49
INSTRUM                       spect
PROBHD     5 mm 5mm 1H/2H
PULPROG                          zg
TD                            48076
SOLVENT                      CDCl3
NS                               16
DS                                2
SWH                        4807.692 Hz
FIDRES                        0.100002 Hz
AQ                         4.9999542 sec
RG                               45.2
DW                            104.000 usec
DE                             14.56 usec
TE                             303.2 K
D1                         5.00000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                             1H
P1                             8.00 usec
PL1                            1.95 dB
PL1W                     9.58740711 W
SFO1                400.1322007 MHz
SI                            32768
SF                  400.1300097 MHz
WDW                              EM
SSB                               0
LB                             0.30 Hz
GB                                0
PC                             1.00
```

hrl-4-116 f12-16

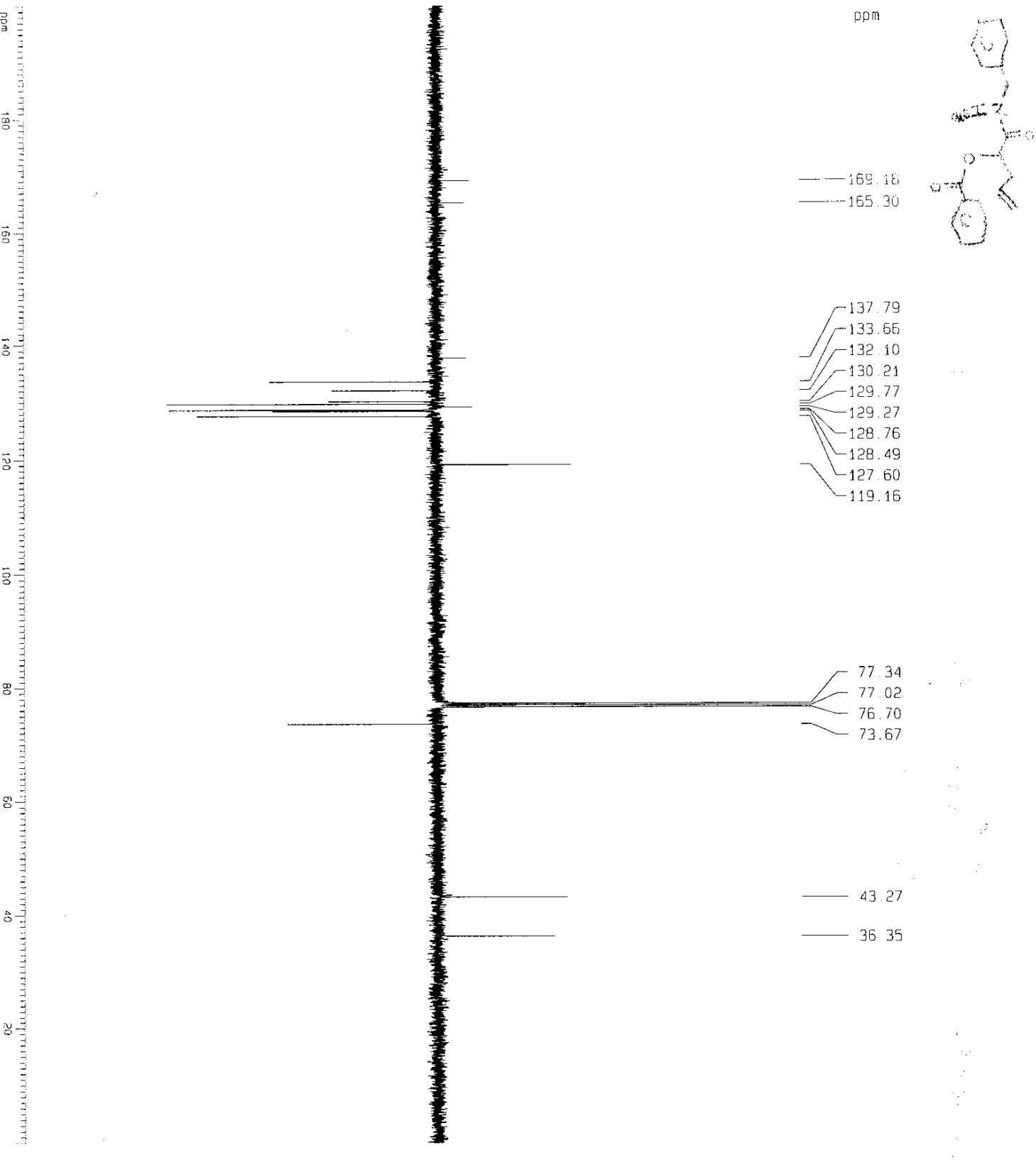
^{13}C NMR (100 MHz, $CDCl_3$) **6f**



^{1}H NMR (400 MHz, $CDCl_3$) **6g**



^{13}C NMR (100 MHz, $CDCl_3$) **6g**



Current Data Parameters
NAME hrl-4-44-2
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081212
Time 7.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 65536
SOLVENT
NS 4103
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 9195.2
DW 19.900 usec
DE 15.50 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 6.00000000 sec
d20 0.00689655 sec
DELTA 0.00001477 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

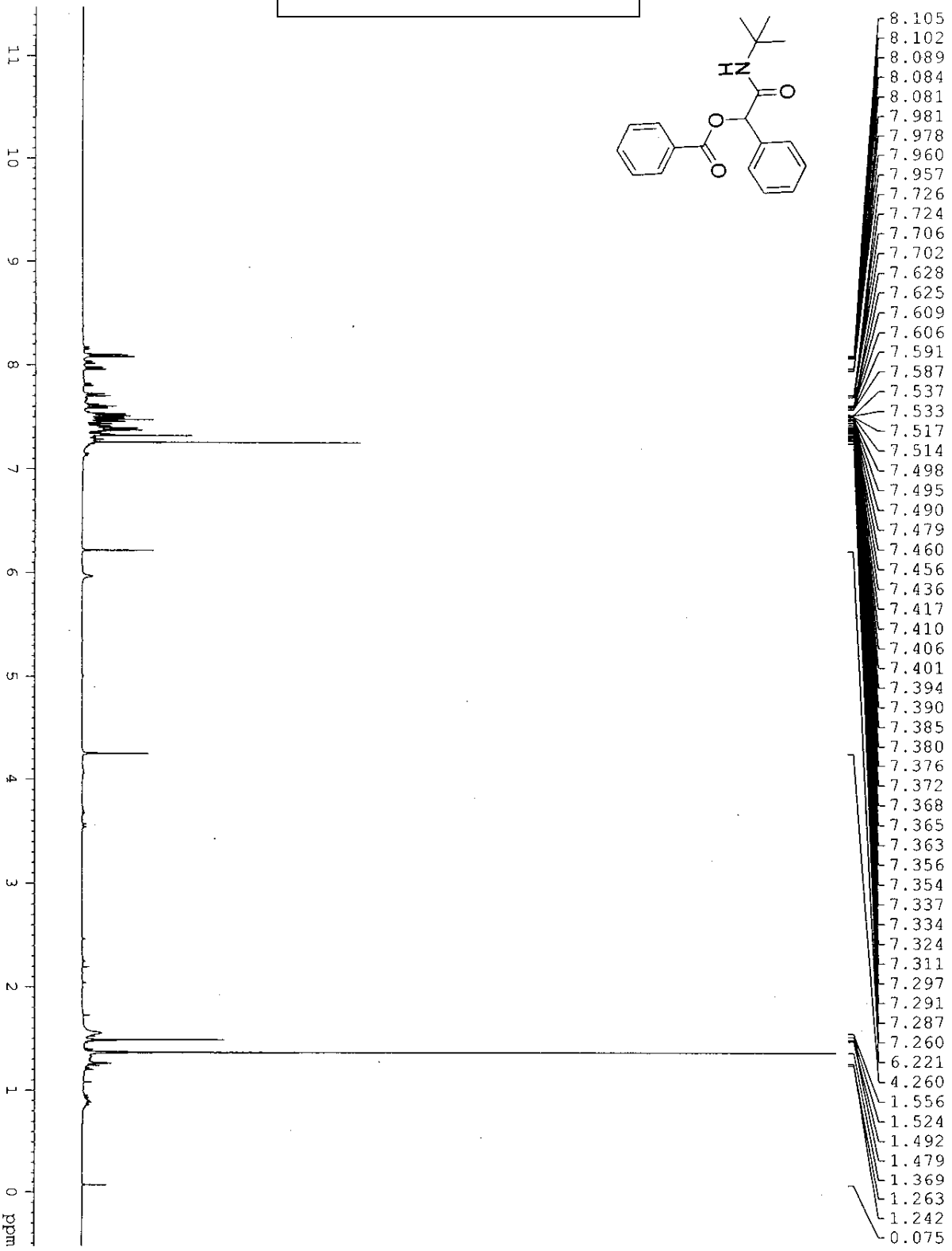
======== CHANNEL f1 ========
NUC1 13C
P1 11.60 usec
d2 23.20 usec
PL1 0.00 dB
SFO1 100.6229643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 28.00 dB
SFO2 400.1320000 MHz

F2 - Processing parameters
SI 65536
SF 100.6127670 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 12.34 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 9.09091 ppm/cm
HZCM 914.66156 Hz/cm

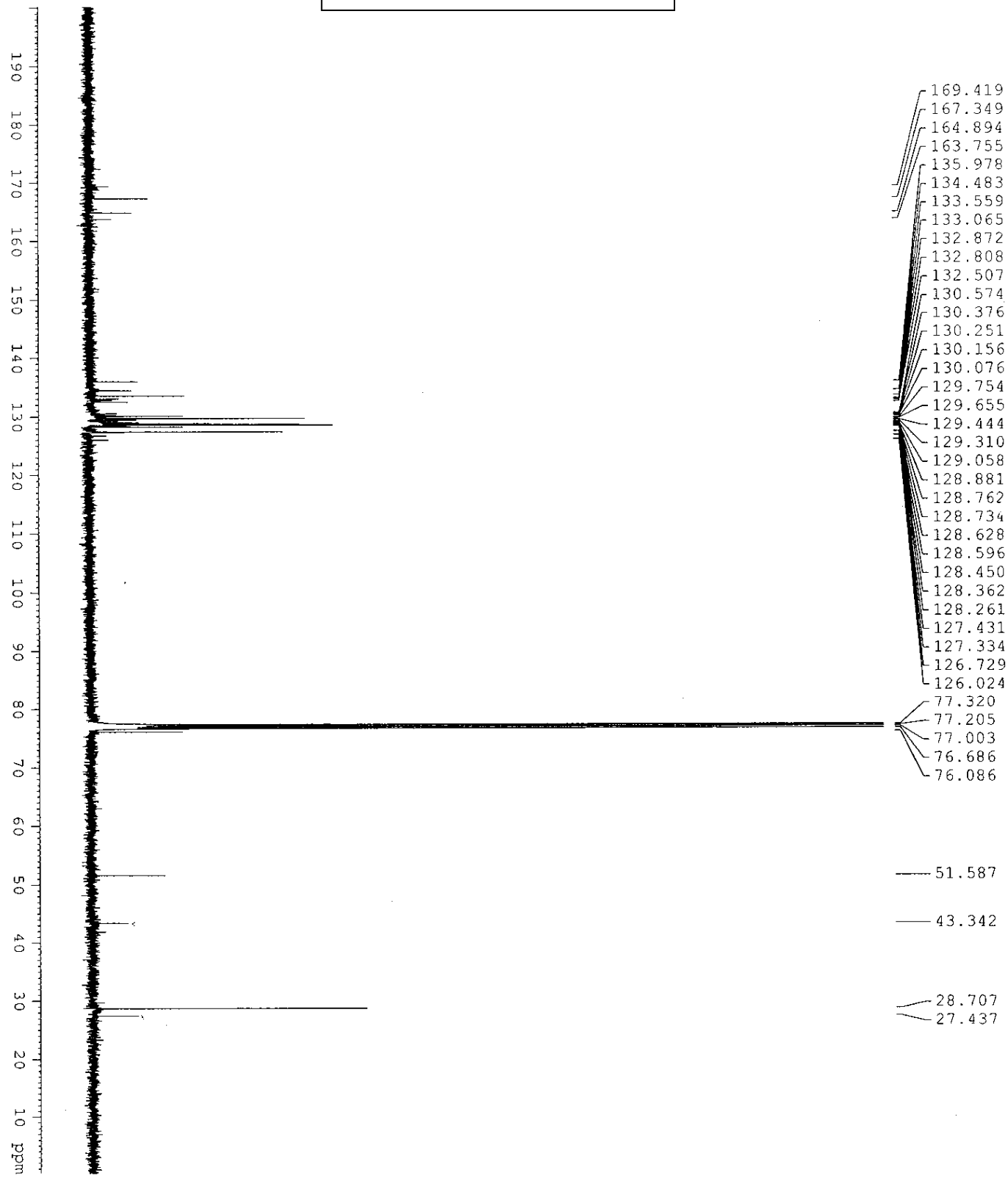
^{1}H NMR (400 MHz, $CDCl_3$) **6h**



hrl-4-100-7-8 t-bu 2eq

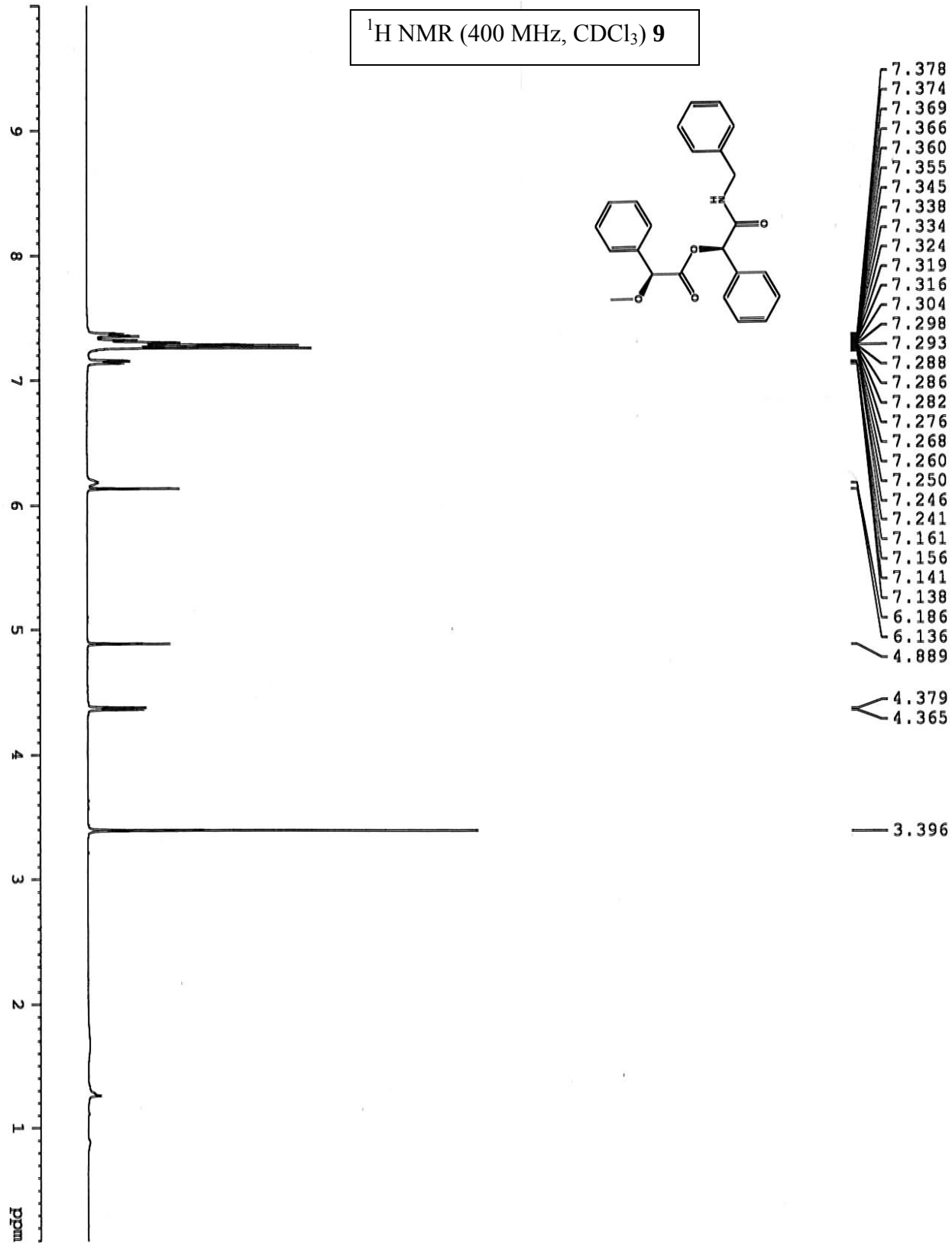
NAME	hrl-4-100-7-8
EXPNO	1
PROCNO	1
Date_	20090403
Time_	18.07
INSTRUM	spect
PROBHD	5 mm 5mm 1H/2H
PULPROG	zg
TD	48076
SOLVENT	CDCl3
NS	32
DS	2
SWH	4807.692 Hz
FIDRES	0.100002 Hz
AQ	4.9999542 sec
RG	181
DW	104.000 usec
DE	14.56 usec
TE	303.2 K
D1	5.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	8.00 usec
PL1	1.95 dB
PL1W	9.58740711 W
SFO1	400.1322007 MHz
SI	32768
SF	400.1300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

^{13}C NMR (100 MHz, $CDCl_3$) **6h**



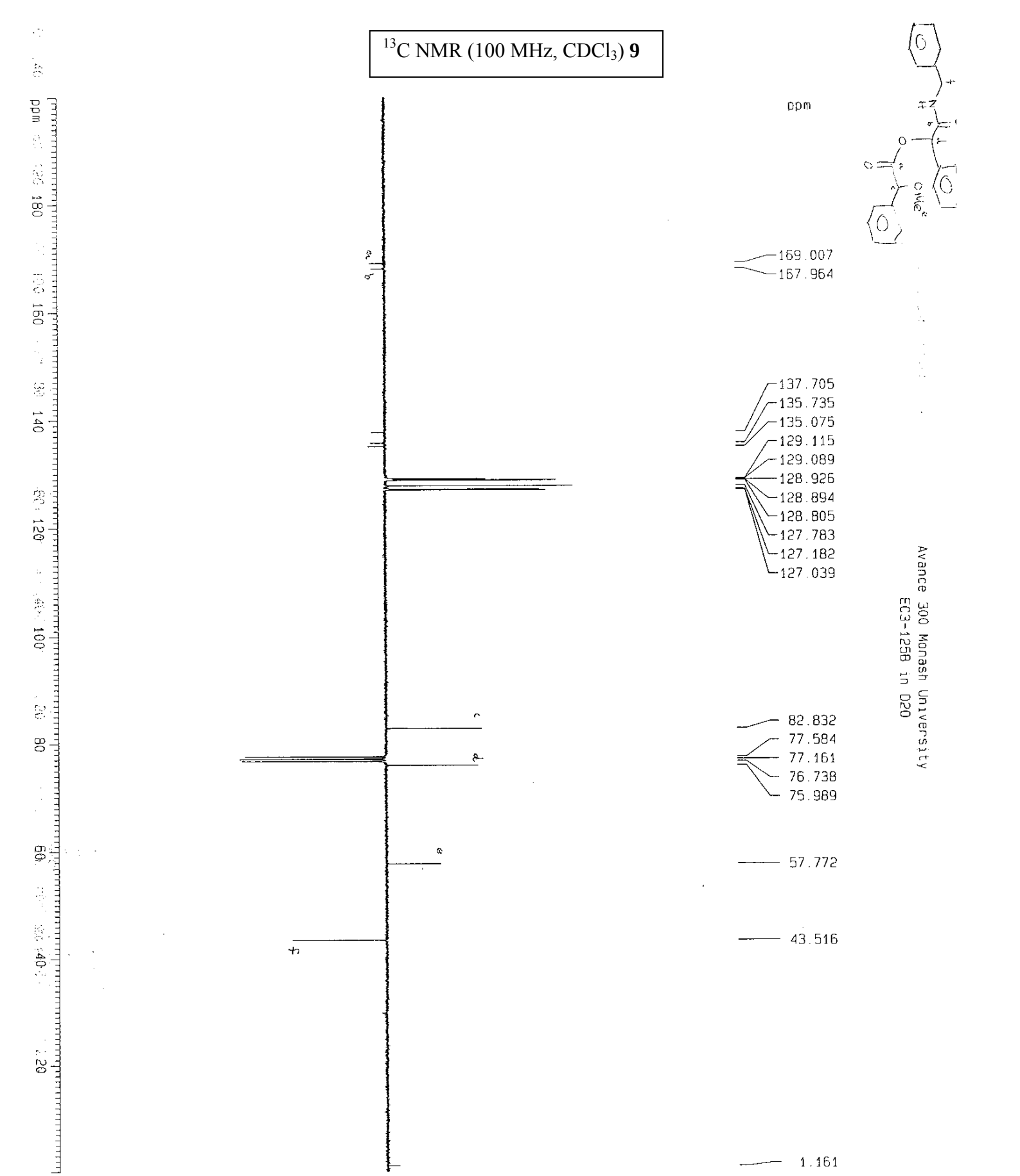

```
Avance III - 13C with 1H Powergated Decoupling

NAME                hrl-4-100-7-8
EXPNO                          4
PROCNO                         1
Date_               20090403
Time_                      22.17
INSTRUM                    spect
PROBHD     5 mm 5mm 1H/2H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        5640
DS                           4
SWH                  22058.824 Hz
FIDRES                0.336591 Hz
AQ                    1.4855326 sec
RG                        2050
DW                    22.667 usec
DE                    12.00 usec
TE                    303.2 K
D1                    10.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                       14.00 usec
PL1                       0.00 dB
PL1W                95.45168304 W
SFO1                100.6223263 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  80.00 usec
PL2                     1.95 dB
PL12                   22.20 dB
PL13                   28.00 dB
PL2W                9.58740711 W
PL12W               0.09051095 W
PL13W               0.02380681 W
SFO2                400.1316005 MHz
SI                       32768
SF                  100.6127690 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40
```

^{1}H NMR (400 MHz, $CDCl_3$) **9**




```
NAME                    hrl-3-114-3c
EXPNO                              1
PROCNO                             1
Date_                       20090825
Time                           12.42
INSTRUM                       spect
PROBHD     5 mm 5mm 1H/2H
PULPROG                         zg
TD                              48076
SOLVENT                     CDCl3
NS                                32
DS                                 2
SWH                          4807.692 Hz
FIDRES                       0.100002 Hz
AQ                           4.9999542 sec
RG                               161
DW                           104.000 usec
DE                           14.56 usec
TE                           303.2 K
D1                           5.00000000 sec
TD0                                1

======== CHANNEL f1 ========
NUC1                             1H
P1                            8.00 usec
PL1                           1.95 dB
PL1W                     9.58740711 W
SFO1                     400.1322007 MHz
SI                              32768
SF                       400.1300098 MHz
WDW                             EM
SSB                              0
LB                            0.30 Hz
GB                               0
PC                            1.00
```

^{13}C NMR (100 MHz, $CDCl_3$) **9**



Current Data Parameters
NAME mrl-3-114-3
EXPNO 3
PROCNO 1

F2 - Acquisition Parameter
Date_ 20080618
Time 23.16
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG jmod
TD 32768
SOLVENT DMSO
NS 7339
DS 2
SWH 18382.354 Hz
FIDRES 0.560985 Hz
AQ 0.8913396 sec
RG 13004
DW 27.200 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 3.00000000 sec
d20 0.00689655 sec
DELTA 0.00001082 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

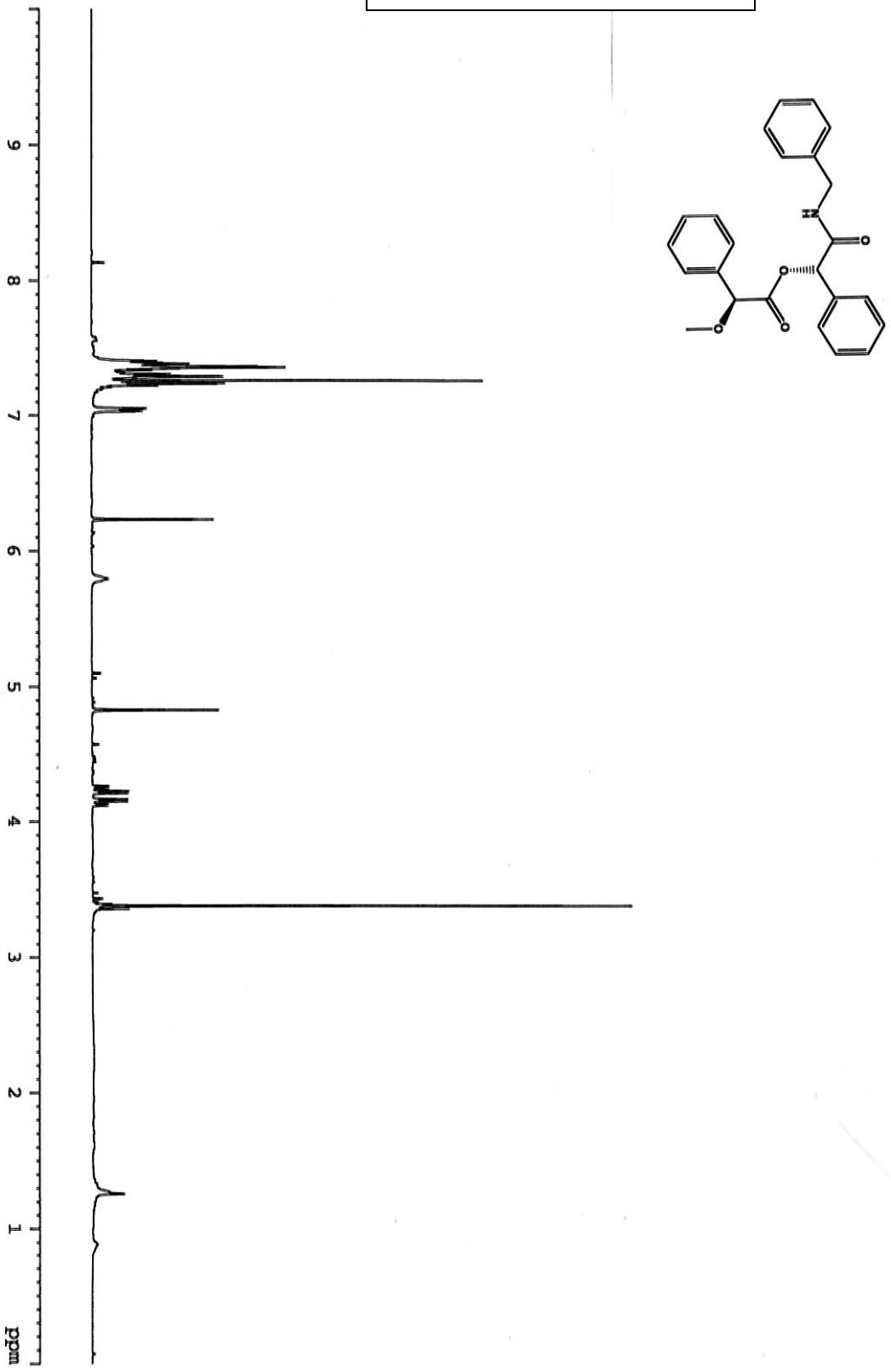
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
p2 17.00 usec
PL1 0.00 dB
SFO1 75.4760145 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.00 dB
SF02 300.1314256 MHz

F2 - Processing parameters
SI 32768
SF 75.4677366 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 4.10 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.69565 ppm/cm
HZCM 656.24115 Hz/cm

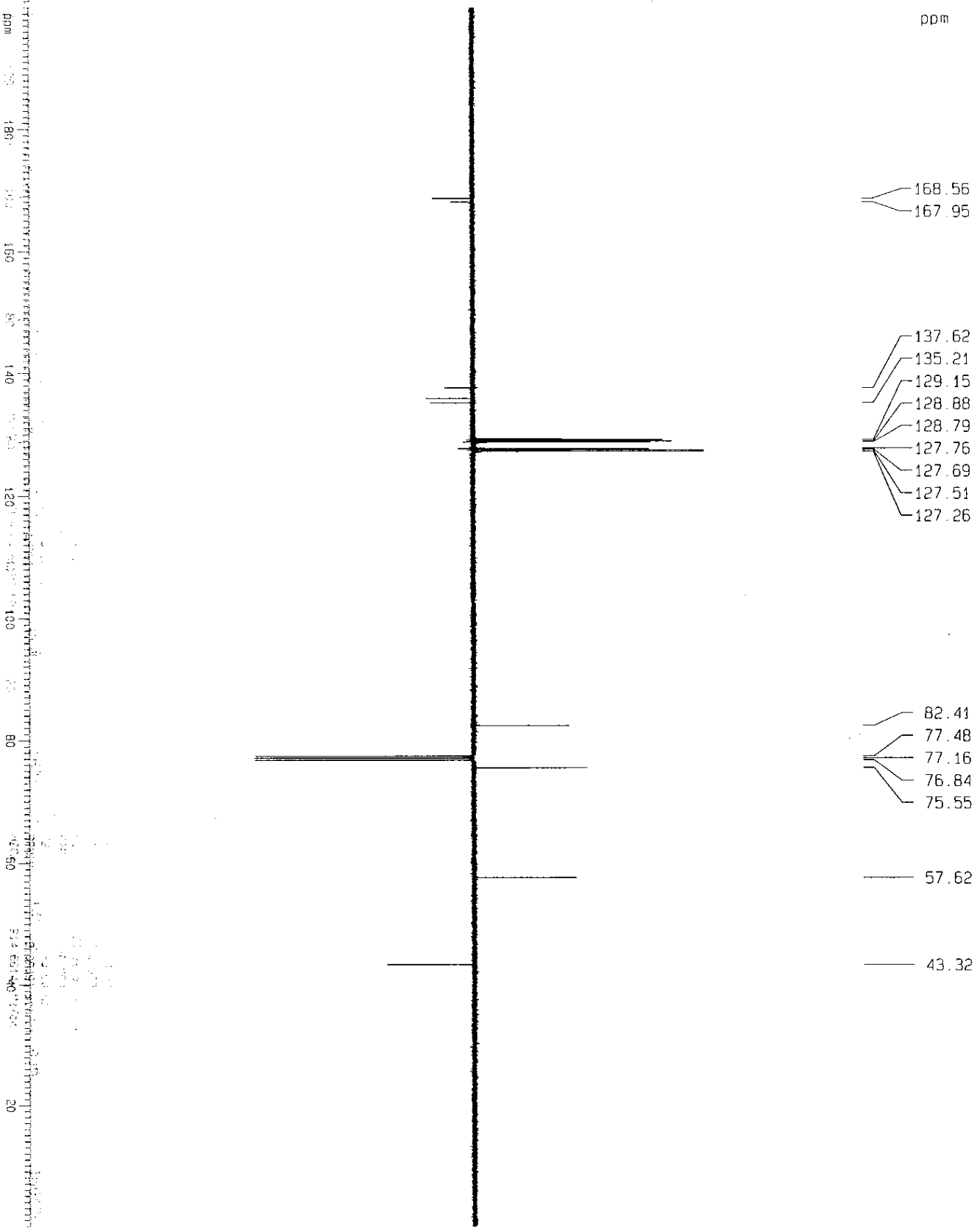
^{1}H NMR (400 MHz, $CDCl_3$) **10**



7.392
7.386
7.382
7.374
7.365
7.358
7.350
7.340
7.331
7.327
7.322
7.317
7.309
7.305
7.298
7.291
7.280
7.276
7.260
7.250
7.242
7.238
7.231
7.229
7.224
7.216
7.213
7.056
7.050
7.043
7.036
7.032
6.234
5.797
4.832
4.270
4.255
4.233
4.218
4.172
4.158
4.135
4.121
3.396
3.383
3.361

NAME	hrl-3-114-2c
EXPNO	1
PROCNO	1
Date_	20090825
Time_	12.55
INSTRUM	spect
PROBHD	5 mm 5mm 1H/2H
PULPROG	zg
TD	48076
SOLVENT	CDCl3
NS	32
DS	2
SWH	4807.692 Hz
FIDRES	0.100002 Hz
AQ	4.9999542 sec
RG	161
DW	104.000 usec
DE	14.56 usec
TE	303.2 K
D1	5.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	8.00 usec
PL1	1.95 dB
PL1W	9.58740711 W
SFO1	400.1322007 MHz
SI	32768
SF	400.1300098 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

^{13}C NMR (100 MHz, $CDCl_3$) **10**



Current Data Parameters
NAME hr1-3-114-2
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080620
Time 19.15
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 65536
SOLVENT
NS 9027
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 9195.2
DW 19.900 usec
DE 15.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 6.00000000 sec
d20 0.00689655 sec
DELTA 0.00001477 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 11.60 usec
p2 23.20 usec
PL1 0.00 dB
SF01 100.6229643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 28.00 dB
SF02 400.1320000 MHz

F2 - Processing parameters
SI 65536
SF 100.6127531 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 4.22 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 9.09091 ppm/c
HZCM 914.66144 Hz/cm

DRX 400