Supporting Information

N-Heterocyclic Carbene-Catalyzed [4+1] Annulation of Phthalaldehyde and Imines

Fang-Gang Sun and Song Ye *

Beijing National Laboratory for Molecular Sciences, CAS Key Laboratory of Molecular Recognition and Function, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China

songye@iccas.ac.cn

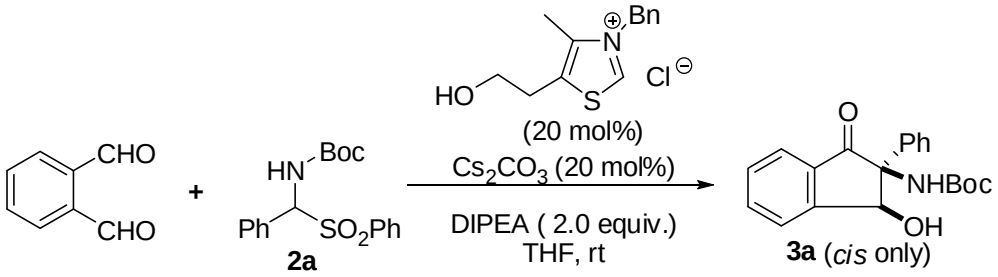
Table of Contents

Part I Experimental part

General Information

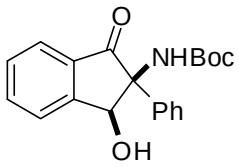
All reactions were carried out under an argon atmosphere in oven-dried glassware with magnetic stirring. CH_2Cl_2 and CH_3CN were distilled from CaH_2. Phthalaldehyde was used after recrystallization from petroleum ether. *tert*-Butyl aryl(phenylsulfonyl)methylcarbamate [1] were prepared according to the literatures. Column chromatograph was performed on silica gel 200 ~ 300 mesh. All ^1H NMR (300 MHz), ^{13}C NMR (75 MHz) spectra were recorded in $CDCl_3$, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ^1H NMR Spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were reported as wavenumber (cm^{-1}).

1.1 Synthesis of *cis*-indanones via a Cascade Stetter-Aldol Reaction Catalyzed by NHC 4a



Typical procedure. To an oven-dried 50 mL Schlenk tube equipped with a stir bar was charged with thiazolium salt (26.9 mg, 0.1 mmol) and Cs_2CO_3 (32.6 mg, 0.1 mmol). The tube was closed with a septum, evacuated, and back-filled with argon. To

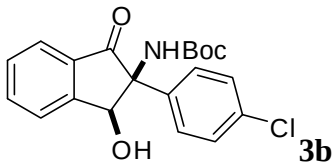
this mixture was added distilled solvent 5 mL, then stirred for 10 min at room temperature. DIPEA (173.9 μL, 1 mmol), *tert*-butyl phenyl(phenylsulfonyl) methylcarbamate (173.5 mg, 0.5 mmol), and phthalaldehyde (100.5 mg, 0.75 mmol) was added to the tube. The mixture was further stirred for overnight, then diluted with ethyl acetate and passed through a short silica pad. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (ethyl acetate/petroleum ether) to give the desired product.



3a

***tert*-butyl-1-hydroxy-3-oxo-2-phenyl-2,3-dihydro-1H-inden-2-ylcarbamate**

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (76%) of *cis*-**3a** as a white solid, R_f = 0.17 (petroleum ether/ethyl acetate = 3/1), mp: 125-127 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82-7.74 (m, 3H), 7.57-7.52 (m, 1H), 7.30-7.22 (m, 5H), 5.78 (b, 1H), 5.65 (d, *J* = 6.6 Hz, 1H), 3.94 (b, 1H), 1.45 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.3, 156.8, 152.2, 138.7, 136.1, 133.7, 130.0, 128.6, 127.7, 126.8, 125.5, 124.7, 80.8, 77.6, 70.3, 28.0; IR (KBr) *v* 1723, 1696, 1163, 699; EIMS *m/z*: 339 (10.0), 222 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{20}H_{21}NO_4$, 339.1471; found 339.1474.

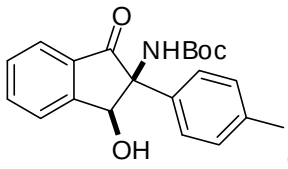


3b

***tert*-butyl-2-(4-chlorophenyl)-1-hydroxy-3-oxo-2,3-dihydro-1H-inden-2-ylcarbamate**

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (83%) of *cis*-**3a** as

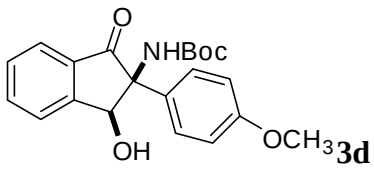
a waxy solid, R_f = 0.30 (petroleum ether/ethyl acetate = 3/1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80-7.75 (m, 3H), 7.57-7.52 (m, 1H), 7.26-7.22 (m, 2H), 7.15 (d, *J* = 8.7 Hz, 2H), 5.81 (b, 1H), 5.64 (d, *J* = 5.7 Hz, 1H), 3.85 (b, 1H), 1.44 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.0, 156.8, 152.3, 137.3, 136.6, 134.0, 133.7, 130.5, 129.0, 127.1, 126.0, 125.2, 81.3, 77.3, 70.2, 28.2; IR (KBr) *v* 1722, 1703, 1165; EIMS *m/z*: 373 (4.0), 256 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{20}H_{20}NO_4Cl$, 373.1081; found 373.1086.



3c

***tert*-butyl-1-hydroxy-3-oxo-2-p-tolyl-2,3-dihydro-1H-inden-2-ylcarbamate**

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (56%) of *cis*-**3a** as a waxy solid, R_f = 0.23 (petroleum ether/ethyl acetate = 3/1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79-7.71 (m, 3H), 7.53-7.48 (m, 1H), 7.13-7.04 (m, 4H), 5.79 (b, 1H), 5.63 (d, *J* = 6.9 Hz, 1H), 3.96 (b, 1H), 2.25 (s, 3H), 1.44 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.7, 157.2, 152.4, 137.9, 136.3, 135.9, 134.0, 130.3, 129.6, 127.1, 125.6, 124.9, 81.1, 78.0, 70.3, 28.2, 20.9; IR (KBr) *v* 1727, 1702, 704; EIMS *m/z*: 353 (8.0), 226 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{21}H_{23}NO_4$, 353.1627; found 353.1630.

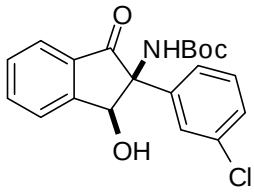


3d

***tert*-butyl-1-hydroxy-2-(4-methoxyphenyl)-3-oxo-2,3-dihydro-1H-inden-2-ylcarbamate**

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (40%) of *cis*-**3a** as

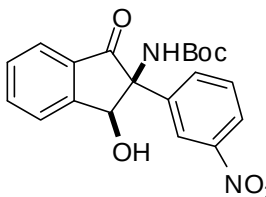
a waxy solid, R_f = 0.20 (petroleum ether/ethyl acetate = 3/1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81-7.72 (m, 3H), 7.55-7.50 (m, 1H), 7.16 (d, *J* = 8.7 Hz, 2H), 6.79 (d, *J* = 9.0 Hz, 2H), 5.74 (b, 1H), 5.63 (d, *J* = 6.3 Hz, 1H), 3.92 (b, 1H), 3.73 (s, 3H), 1.44 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.7, 159.3, 157.3, 152.3, 136.3, 134.0, 130.8, 130.3, 127.1, 127.0, 124.9, 114.3, 81.2, 78.0, 70.0, 55.3, 28.2; IR (KBr) *v* 1723, 1701, 1162; EIMS *m/z*: 369 (50.0), 251 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{21}H_{23}NO_5$, 369.1576; found 369.1581.



3e

***tert*-butyl-2-(3-chlorophenyl)-1-hydroxy-3-oxo-2,3-dihydro-1H-inden-2-ylcarbamate**

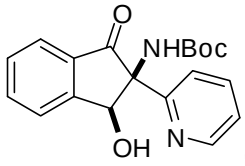
Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (87%) of cis-3a as a white solid, Rf = 0.24 (petroleum ether/ethyl acetate = 3/1), mp: 132-134 oC. 1H NMR (300 MHz, CDCl3) δ 7.80-7.76 (m, 3H), 7.57-7.53 (m, 1H), 7.26-7.15 (m, 3H), 7.05 (d, J = 6.6 Hz, 1H), 5.84 (b, 1H), 5.64 (d, J = 5.1 Hz, 1H), 3.91 (b, 1H), 1.45 (s, 9H); 13C NMR (75 MHz, CDCl3) δ 199.9, 156.9, 152.3, 140.8, 136.7, 135.0, 133.7, 130.6, 130.1, 128.3, 127.2, 125.9, 125.2, 123.8, 81.5, 77.3, 70.2, 28.2; IR (KBr) v 1720, 1694, 1161, 696; EIMS m/z: 373 (4.0), 256 (100); HRMS-(EI) (m/z): M+ calcd for $C_{20}H_{20}NO_4Cl$, 373.1081; found 373.1084.



3f

***tert*-butyl-1-hydroxy-2-(3-nitrophenyl)-3-oxo-2,3-dihydro-1H-inden-2-ylcarbamate**

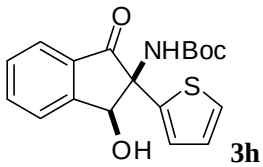
Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (91%) of *cis*-**3a** as a white solid, R_f = 0.17 (petroleum ether/ethyl acetate = 3/1), mp: 99-100 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 8.14-8.09 (m, 2H), 7.91-7.81 (m, 3H), 7.63-7.58 (m, 1H), 7.45 (d, *J* = 7.2 Hz, 2H), 5.93 (b, 1H), 5.76 (d, *J* = 5.4 Hz, 1H), 3.74 (b, 1H), 1.48 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.0, 156.4, 152.1, 148.5, 140.9, 136.9, 133.3, 131.4, 130.7, 129.7, 127.1, 125.4, 122.9, 120.7, 81.7, 77.4, 70.2, 28.2; IR (KBr) *v* 1719, 1703, 1159, 702; EIMS *m/z*: 384 (4.0), 267 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{20}H_{20}N_2O_6$, 384.1321; found 384.1326.



3g

***tert*-butyl-1-hydroxy-3-oxo-2-(pyridin-2-yl)-2,3-dihydro-1H-inden-2-ylcarbamate**

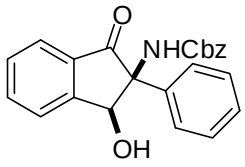
Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (87%) of *cis*-**3a** as a waxy solid, R_f = 0.17 (petroleum ether/ethyl acetate = 3/1). ^1H NMR (300 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.2 Hz, 1H), 7.87-7.77 (m, 3H), 7.57 (t, *J* = 7.5 Hz, 2H), 7.37 (b, 1H), 7.21-7.17 (m, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 5.41 (d, *J* = 10.2 Hz, 1H), 4.04 (d, *J* = 10.2 Hz, 1H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.7, 158.1, 155.7, 153.7, 148.3, 137.5, 136.4, 134.4, 130.2, 127.3, 124.7, 123.0, 119.8, 81.1, 78.3, 70.4, 28.2; IR (KBr) *v* 1725, 1697, 1165, 701; EIMS *m/z*: 340 (5.0), 223 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{19}H_{20}N_2O_4$, 340.1423; found 340.1425.



3h

***tert*-butyl-1-hydroxy-3-oxo-2-(thiophen-2-yl)-2,3-dihydro-1H-inden-2-ylcarbamate**

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (93%) of *cis*-**3a** as a white solid, R_f = 0.16 (petroleum ether/ethyl acetate = 3/1), mp: 135-136 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83-7.73 (m, 3H), 7.55 (t, *J* = 6.9 Hz, 1H), 7.18 (d, *J* = 4.5 Hz, 1H), 6.85-6.73 (m, 2H), 5.89 (b, 1H), 5.73 (d, *J* = 4.5 Hz, 1H), 3.96 (b, 1H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.5, 156.8, 151.7, 142.8, 136.4, 133.1, 130.5, 127.4, 127.2, 125.6, 125.3, 125.1, 81.4, 78.3, 68.4, 28.2; IR (KBr) *v* 1727, 1694, 1163, 703; EIMS *m/z*: 345 (4.0), 228 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{18}H_{19}NO_4S$, 345.1035; found 345.1039.

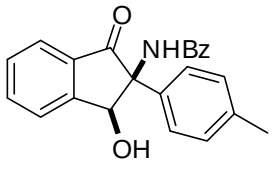


3i

benzyl-1-hydroxy-3-oxo-2-phenyl-2,3-dihydro-1H-inden-2-ylcarbamate

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (66%) of *cis*-**3a** as a waxy solid, R_f = 0.19 (petroleum ether/ethyl acetate = 3/1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82-7.74 (m, 3H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.35-7.23 (m, 11H), 6.07 (b, 1H), 5.72 (d, *J* = 5.7 Hz, 1H), 5,13 (s, 2H), 3.66 (d, *J* = 5.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.0, 157.4, 152.3, 138.4, 136.5, 135.8, 133.9, 130.5, 129.0, 128.6, 128.4, 128.2, 128.0, 127.1, 125.6, 125.1, 77.7, 70.6, 67.6; IR (KBr) *v* 1718, 1700,

1654, 697; EIMS *m/z*: 373 (4.0), 108 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{23}H_{19}NO_4$, 373.1314; found 373.1318.

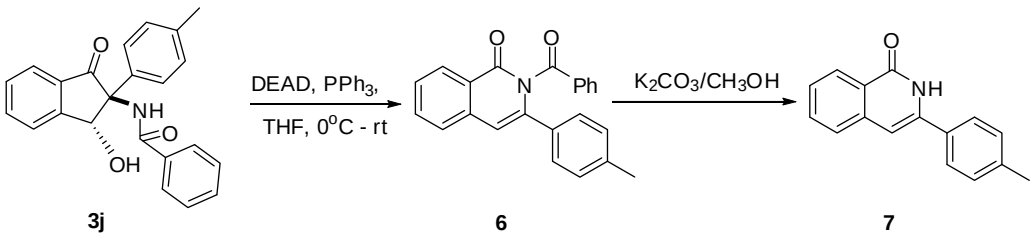


3j

N-(1-hydroxy-3-oxo-2-p-tolyl-2,3-dihydro-1H-inden-2-yl)benzamide

Purified with petroleum ether/ethyl acetate (3/1), yielding 78.5 mg (53%) of *cis*-**3a** as a white solid, R_f = 0.20 (petroleum ether/ethyl acetate = 3/1), mp: 107-108 ^oC. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92-7.76 (m, 5H), 7.57 (t, *J* = 7.2 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 2H), 7.30 (s, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 5.89 (d, *J* = 6.9 Hz, 1H), 3.90 (d, *J* = 6.9 Hz, 1H), 2.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.6, 169.1, 152.3, 138.2, 136.5, 135.3, 133.9, 133.1, 132.4, 130.4, 129.8, 128.8, 127.4, 127.2, 125.7, 125.0, 78.0, 71.3, 21.0; IR (KBr) *v* 1729, 1700, 1644, 1385, 696; EIMS *m/z*: 357 (4.0), 105 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{23}H_{19}NO_3$, 357.1365; found 357.1370.

1.2 Synthesis of isoquinolinone 6[2]



3j **6** **7**

2-benzoyl-3-*p*-tolylisoquinolin-1(2H)-one (6)

To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with the indanone (76 mg, 0.213 mmol) and PPh_3 (83.6 mg, 0.319 mmol). The tube was closed with a septum, evacuated, and back-filled with argon. To this mixture was added distilled solvent THF(2 mL), then the mixture was cooled to 0^{o}C. DEAD(55.6 mg, 0.319 mmol) was added to the tube. The mixture was further stirred for 1h at 0^{o}C, then 2h at room temperature. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (ethyl acetate/petroleum ether) to give the desired product.

Purified with petroleum ether/ethyl acetate (10/1), yielding 59.9 mg (83%) as a waxy solid, R_f = 0.35 (petroleum ether/ethyl acetate = 10/1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 (d, *J* = 7.2 Hz, 2H), 8.03-7.91 (m, 5H), 7.71 (t, *J* = 7.5 Hz, 1H), 7.61-7.51 (m, 3H), 7.28 (d, *J* = 8.1 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.0, 155.6, 149.6, 139.9, 138.8, 135.7, 134.0, 131.1, 130.6, 129.5, 129.1, 128.7, 127.5, 127.2, 126.9, 123.7, 120.6, 115.5, 21.3; IR (KBr) *v* 1654, 1364, 613; EIMS *m/z*: 339 (50.0), 235 (100); HRMS-(EI) (*m/z*): M^+ calcd for $C_{23}H_{17}NO_2$, 339.1259; found 339.1264.

3-p-tolylisoquinolin-1(2H)-one (7)

To an 10mL rounded-bottom flask equipped with a stir bar was added isoquinolinone (40mg, 0.118 mmol) and CH_3OH（1mL）.Then, K_2CO_3(32.6 mg, 0.236 mmol) was added to the mixture. The stirring was continuing for 3h. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel

(ethyl acetate/petroleum ether) to give the desired product.

Purified with petroleum ether/ethyl acetate (3/1-1/1), yielding 26.4 mg (95%) as a white solid, R_f = 0.23 (petroleum ether/ethyl acetate = 3/1), mp: 239-240°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.06 (s, 1H), 8.41 (d, *J* = 8.1 Hz, 1H), 7.70-7.57 (m, 4H), 7.50-7.45 (m, 1H), 7.32 (d, *J* = 8.1 Hz, 2H), 6.76 (s, 1H), 2.43 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.9, 139.8, 139.5, 138.4, 132.8, 131.5, 129.9, 127.5, 126.5, 126.0, 124.9, 103.8, 21.3; IR (KBr) *v* 1739, 1154, 810.

1.3 X-ray Crystal Structure

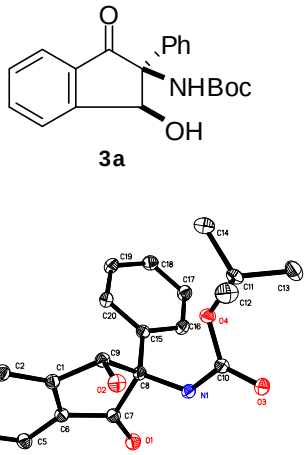


Figure S1. X-Ray crystal structure of *cis*-**3a**

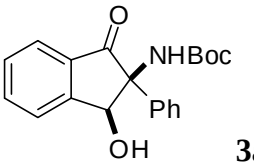
A colorless solution of **3a** in EtOAc was prepared. Crystals suitable for X-ray structural analysis were obtained by slow evaporation of the solvents at room temperature.

References

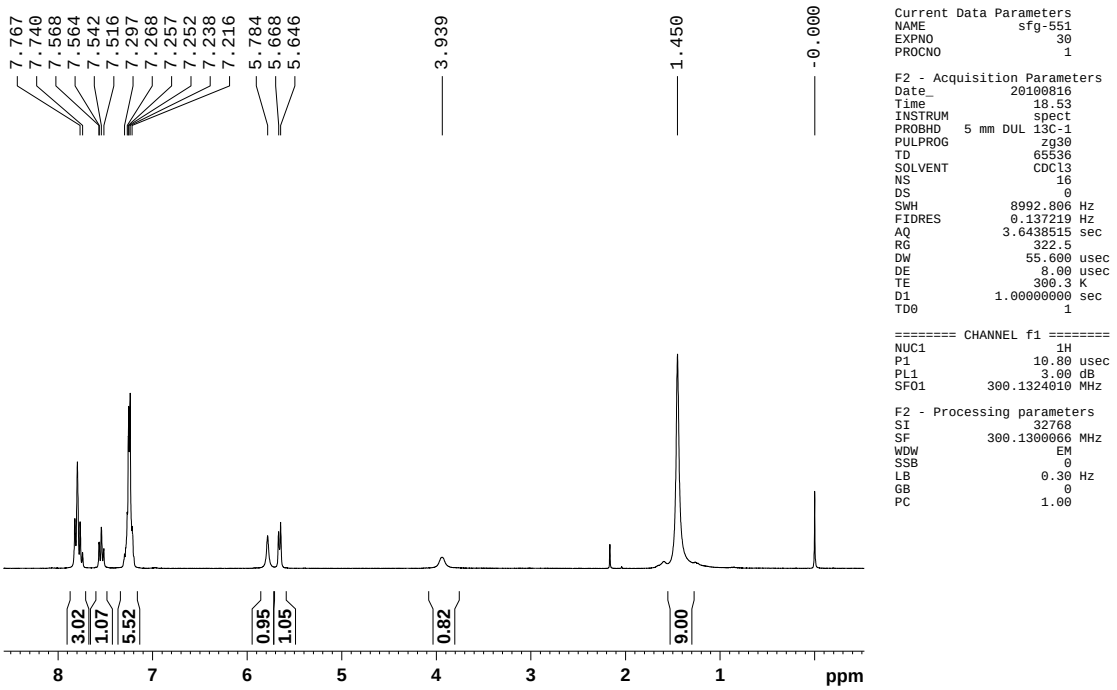
1. (a) C. Rampalakos and W. D. Wulff, *Adv. Synth. Catal.*, 2008, **350**, 1785. (b) M. D. Smith, C. R. Jones, P. G. Dan and A. J. Morrison, *Angew. Chem. Int. Ed.*, 2009, **48**, 7391. (c) T. Ollevier and Z. Li, *Adv. Synth. Catal.*, 2009, **351**, 3251.

2. W.-J. Cho, M.-J. Park, B.-H. Chung and C.-O. Lee, *Bioorg. Med. Chem. Lett.*, 1998, **8**, 41-46.

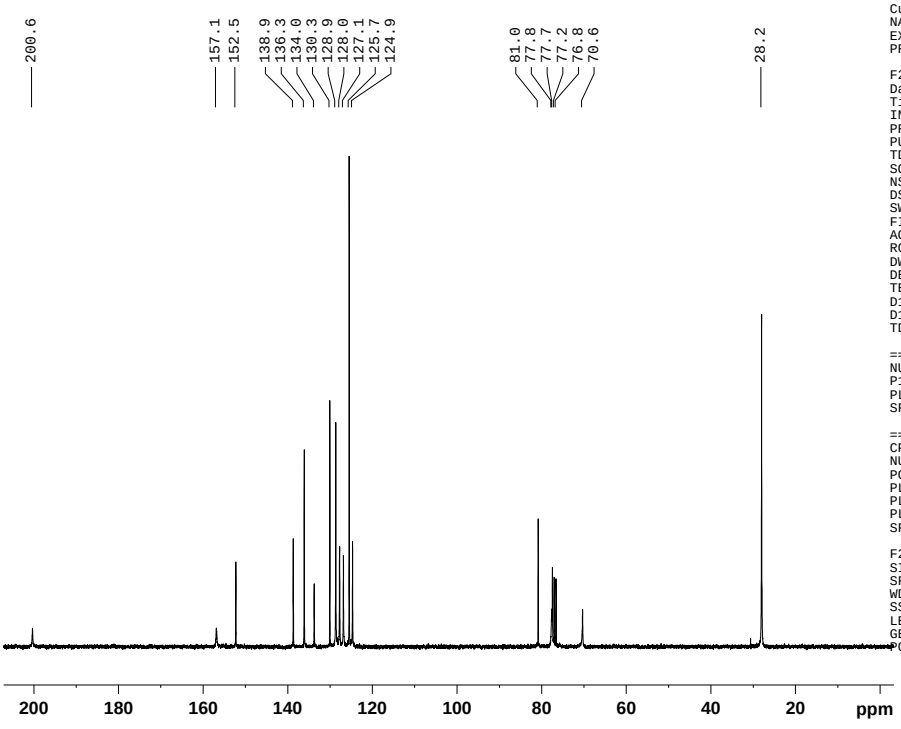
Part II Copy of NMR Spectra



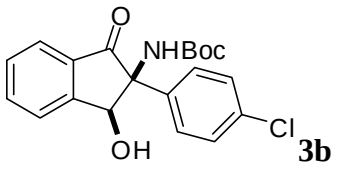
3a



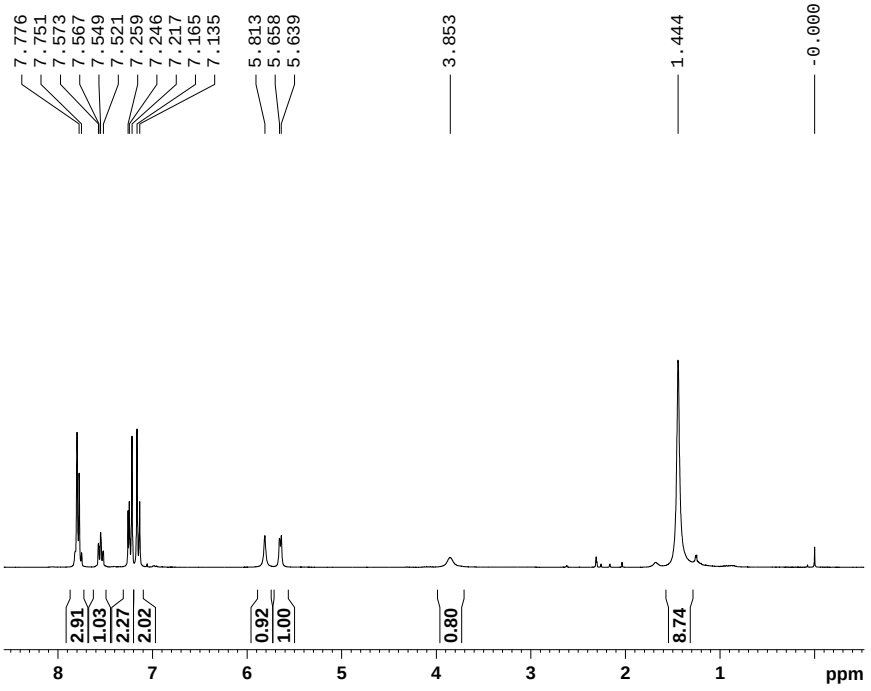

```
Current Data Parameters
NAME                  sfg-551
EXPNO                      30
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100816
Time                   18.53
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                3.6438515 sec
RG                     322.5
DW                    55.600 usec
DE                     8.00 usec
TE                     300.3 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.80 usec
PL1                     3.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF               300.1300066 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



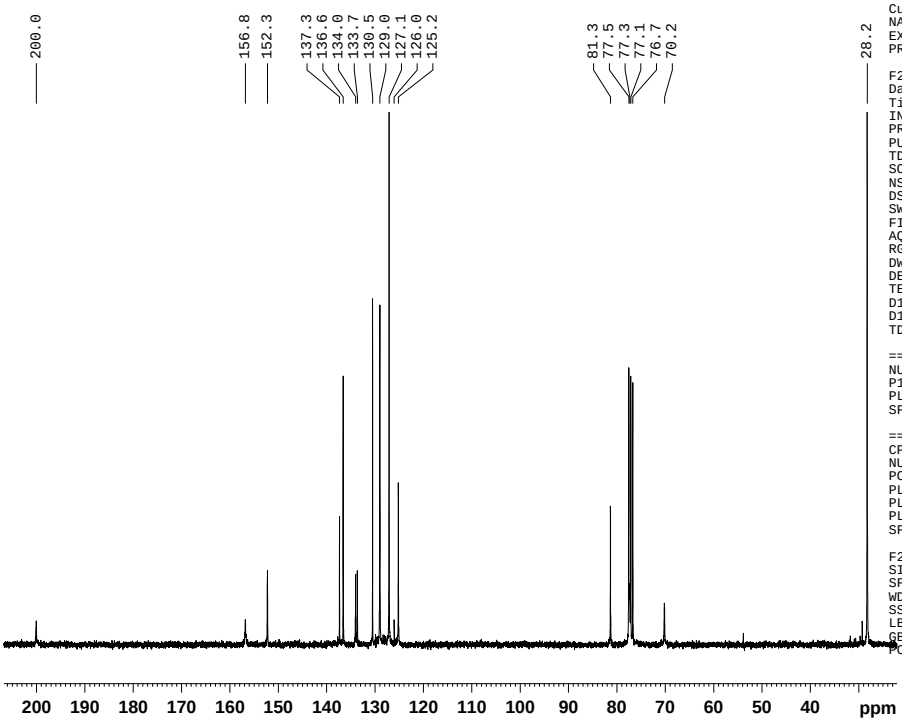

```
Current Data Parameters
NAME                  sfg-551
EXPNO                      40
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100817
Time                    8.43
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       218
DS                         4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                1.8219508 sec
RG                    5160.6
DW                    27.800 usec
DE                     8.00 usec
TE                     301.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.50 usec
PL1                     2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     3.00 dB
PL12                   22.33 dB
PL13                   23.00 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                     32768
SF               75.4677672 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



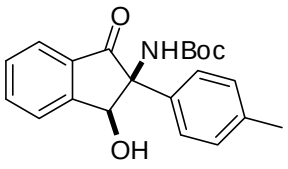
3b



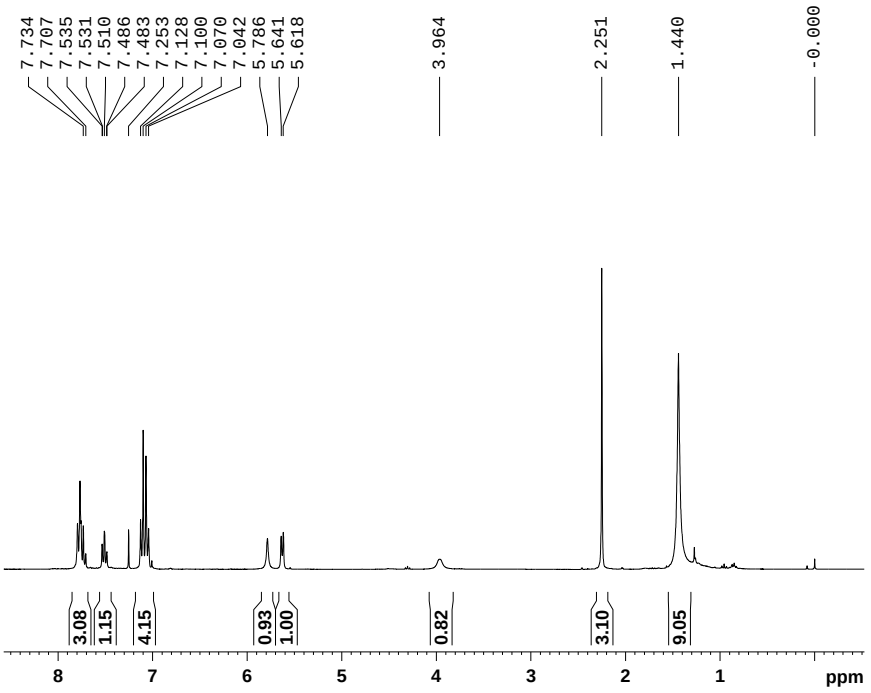

```
Current Data Parameters
NAME                  sfg-593
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100906
Time                    8.28
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                  3.6438515 sec
RG                       128
DW                    55.600 usec
DE                     8.00 usec
TE                     301.1 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.80 usec
PL1                     3.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF                300.1300061 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



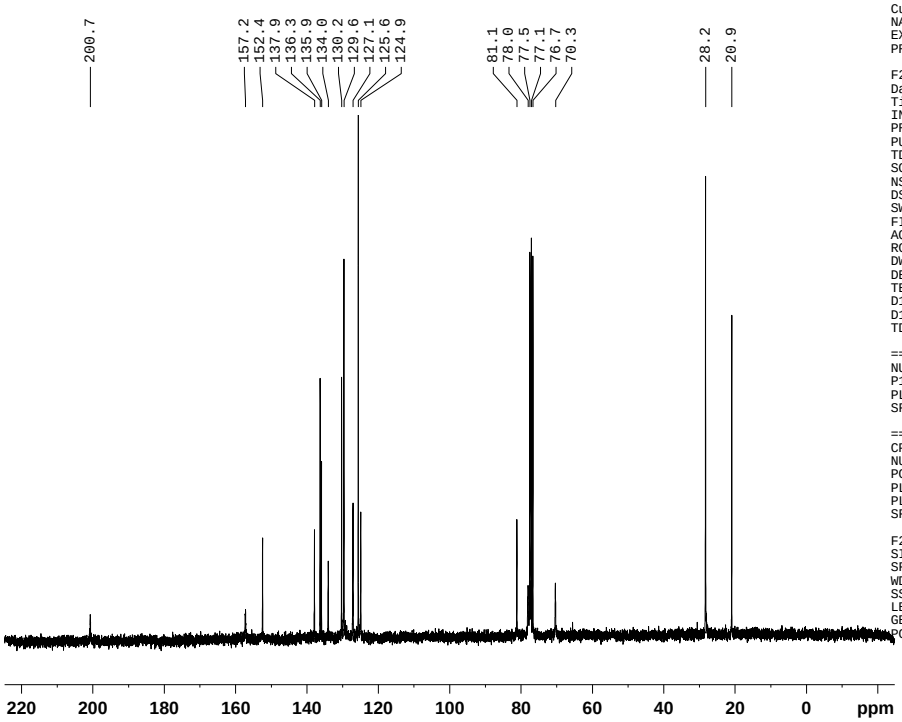

```
Current Data Parameters
NAME                  sfg-593
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_               20101027
Time                   23.38
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       420
DS                         4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                  1.8219508 sec
RG                      5792.6
DW                    27.800 usec
DE                     8.00 usec
TE                     297.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     12.50 usec
PL1                     2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     3.00 dB
PL12                   22.33 dB
PL13                   23.00 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                     32768
SF                75.4677490 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



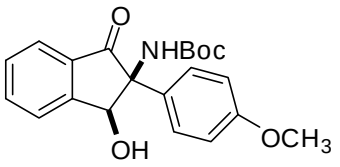
3c



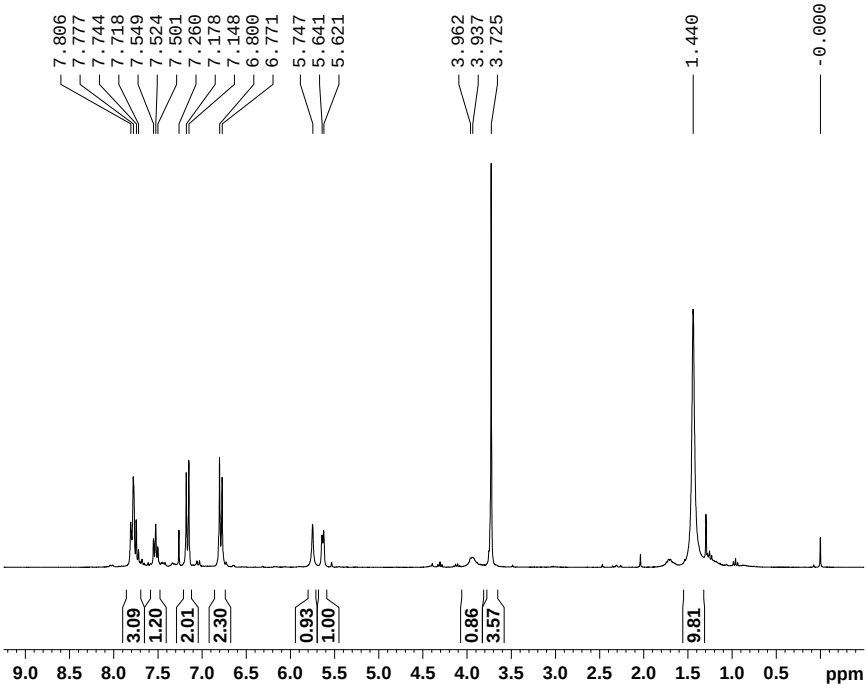

```
Current Data Parameters
NAME                 sfg-582
EXPNO                     20
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100828
Time                  15.54
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       15
DS                        0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                      71.8
DW                    55.600 usec
DE                     8.00 usec
TE                    300.8 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.80 usec
PL1                     3.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                        32768
SF                300.1300079 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



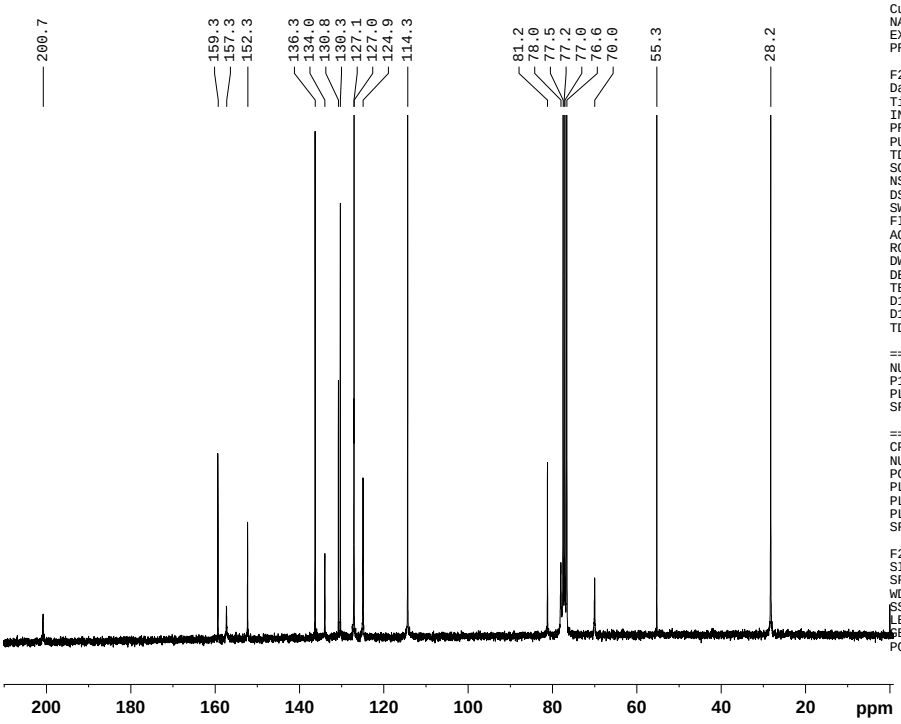

```
Current Data Parameters
NAME                 sfg-582
EXPNO                     40
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100828
Time                  16.43
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgig30
TD                    32768
SOLVENT               CDCl3
NS                     1080
DS                        0
SWH               18832.393 Hz
FIDRES             0.574719 Hz
AQ                 0.8700404 sec
RG                    5160.6
DW                    26.550 usec
DE                     8.00 usec
TE                    301.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.50 usec
PL1                     2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     3.00 dB
PL12                   22.33 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                75.4677490 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```



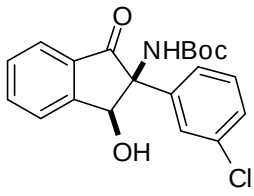
3d



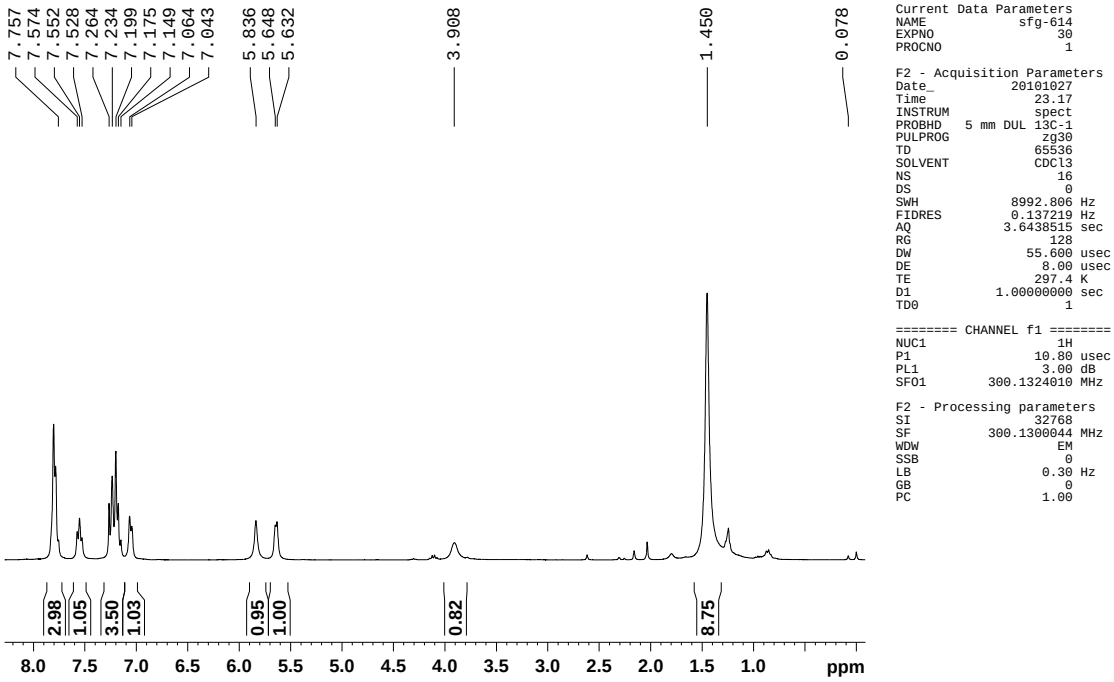

```
Current Data Parameters
NAME                  sfg-583
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100903
Time                   16.42
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                3.6438515 sec
RG                       128
DW                    55.600 usec
DE                     8.00 usec
TE                     301.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.80 usec
PL1                      3.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                         32768
SF                300.1300058 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



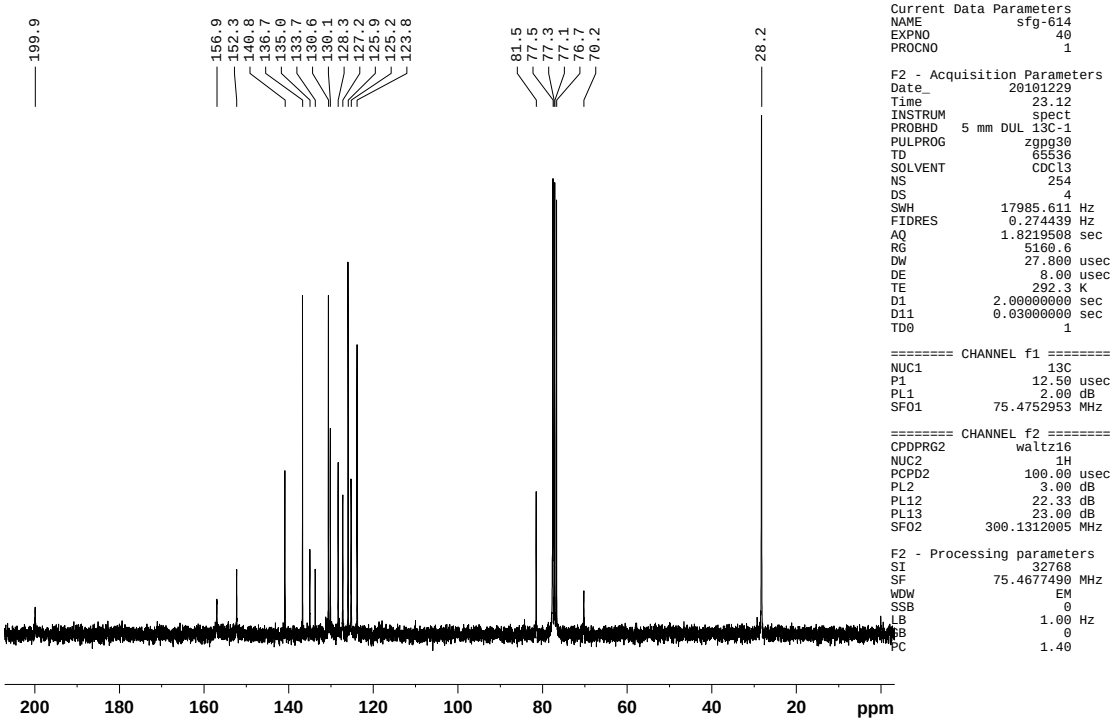

```
Current Data Parameters
NAME                  sfg-583
EXPNO                      20
PROCNO                      1

F2 - Acquisition Parameters
Date_               20101028
Time                    0.13
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       6925
DS                          4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                1.8219508 sec
RG                     5792.6
DW                    27.800 usec
DE                     8.00 usec
TE                     298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                      12.50 usec
PL1                      2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                      3.00 dB
PL12                    22.33 dB
PL13                    23.00 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                75.4677490 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



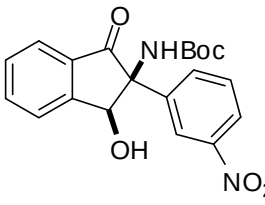
3e



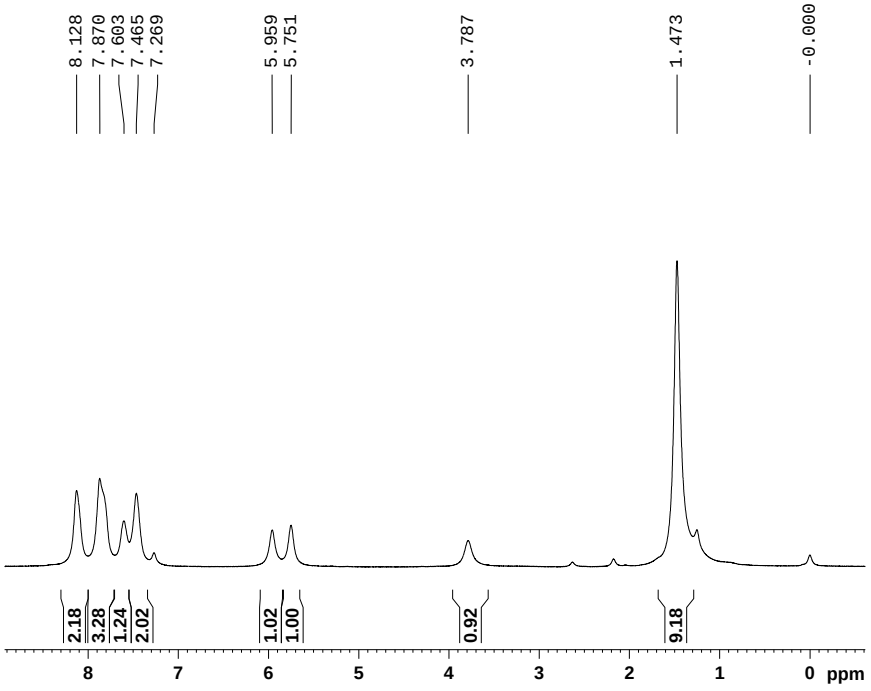

```
Current Data Parameters
NAME                  sfg-614
EXPNO                      30
PROCNO                      1

F2 - Acquisition Parameters
Date_                20101027
Time                    23.17
INSTRUM                 spect
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                3.6438515 sec
RG                       128
DW                    55.600 usec
DE                     8.00 usec
TE                    297.4 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.80 usec
PL1                      3.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                         32768
SF                300.1300044 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



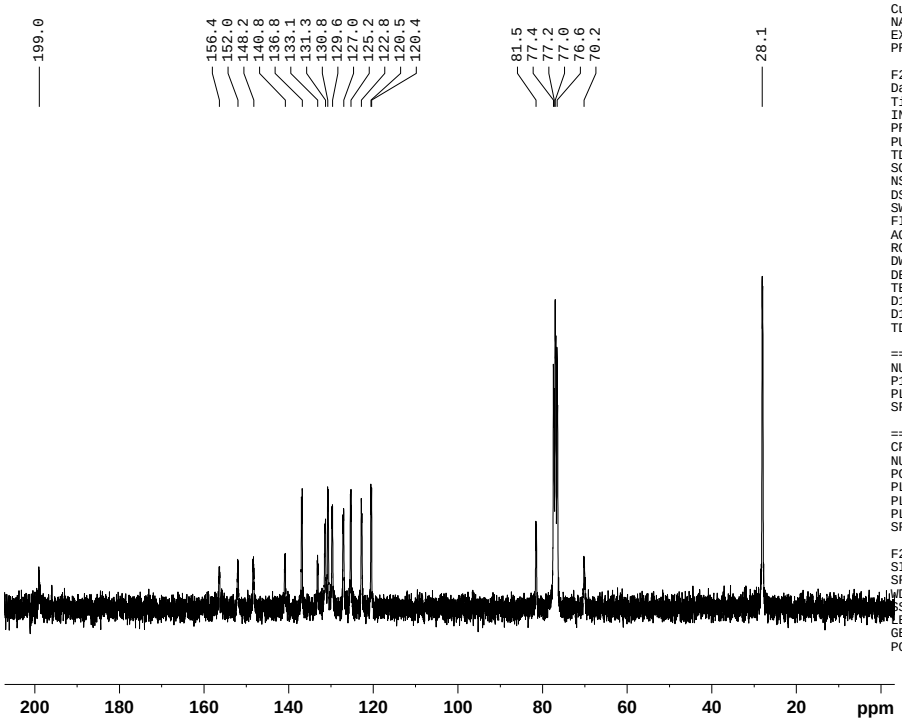

```
Current Data Parameters
NAME                  sfg-614
EXPNO                      40
PROCNO                      1

F2 - Acquisition Parameters
Date_                20101229
Time                    23.12
INSTRUM                 spect
PROBHD   5 mm DUL 13C-1
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       254
DS                         4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                1.8219508 sec
RG                     5160.6
DW                    27.800 usec
DE                     8.00 usec
TE                    292.3 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      12.50 usec
PL1                      2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                      3.00 dB
PL12                    22.33 dB
PL13                    23.00 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                75.4677490 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



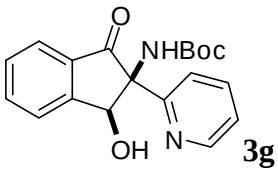
3f



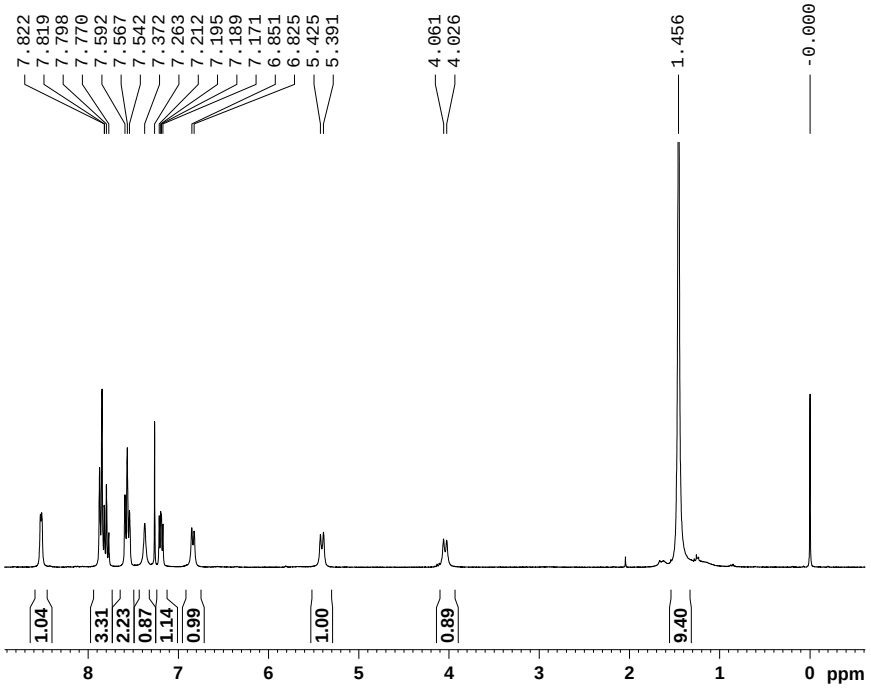

```
Current Data Parameters
NAME                  sfg-594
EXPNO                      50
PROCNO                      1

F2 - Acquisition Parameters
Date_               20110104
Time                   18.32
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        15
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                3.6438515 sec
RG                       181
DW                  55.600 usec
DE                   8.00 usec
TE                   293.4 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.80 usec
PL1                    3.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                        32768
SF                300.1300014 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



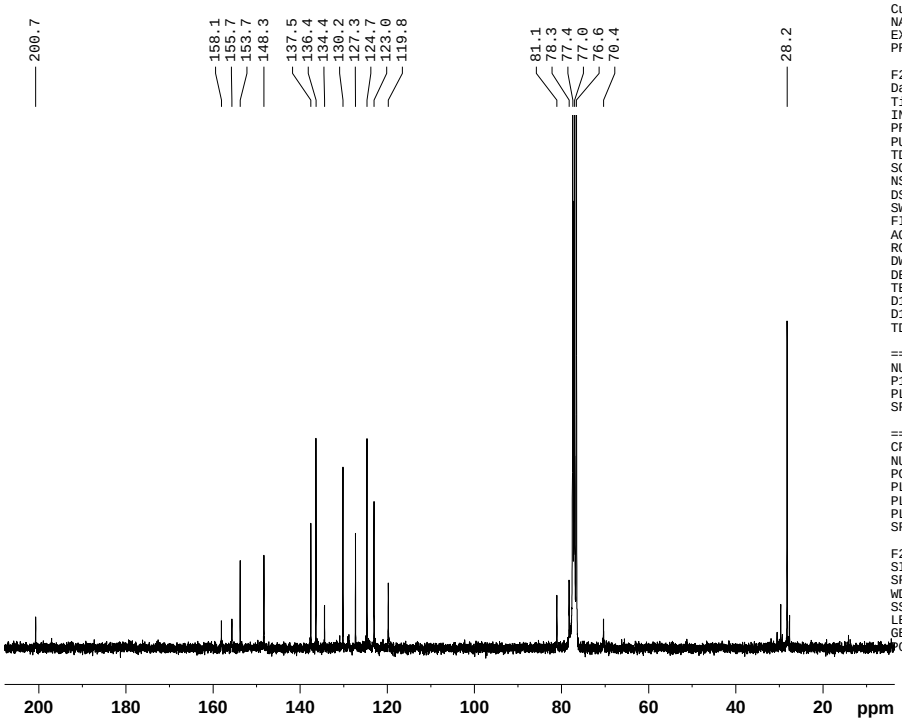

```
Current Data Parameters
NAME                  sfg-594
EXPNO                      60
PROCNO                      1

F2 - Acquisition Parameters
Date_               20110104
Time                   18.58
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       258
DS                         4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                1.8219508 sec
RG                   14596.5
DW                  27.800 usec
DE                   8.00 usec
TE                   293.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    3.00 dB
PL12                  22.33 dB
PL13                  23.00 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                75.4677588 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



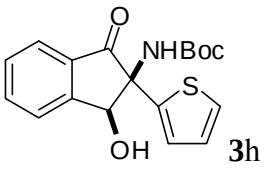
3g



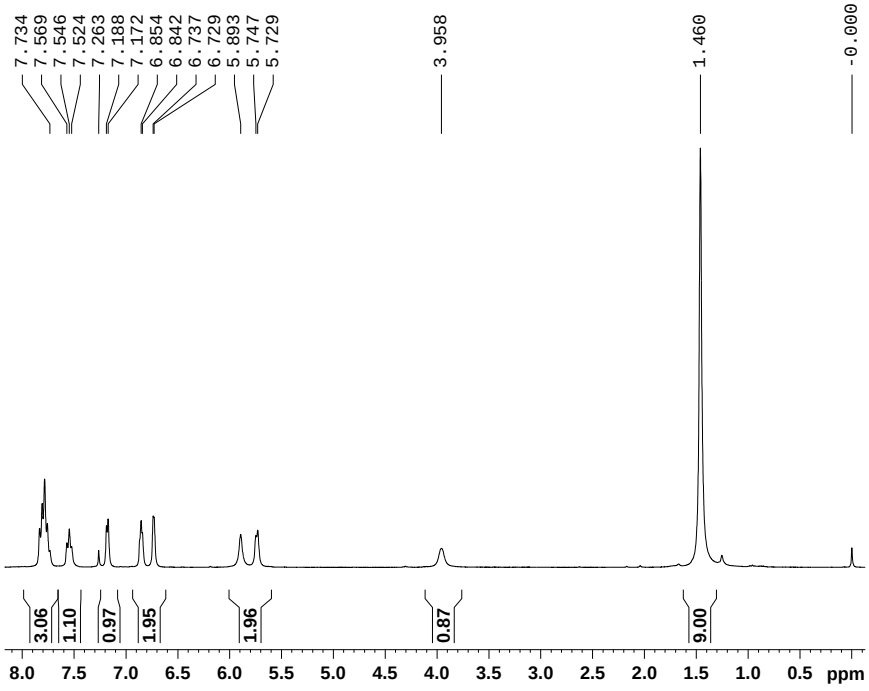

```
Current Data Parameters
NAME                  sfg-585
EXPNO                      40
PROCNO                      1

F2 - Acquisition Parameters
Date_              20101103
Time                    8.44
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                3.6438515 sec
RG                     322.5
DW                    55.600 usec
DE                     8.00 usec
TE                     295.7 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.80 usec
PL1                      3.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                         32768
SF                300.1300051 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



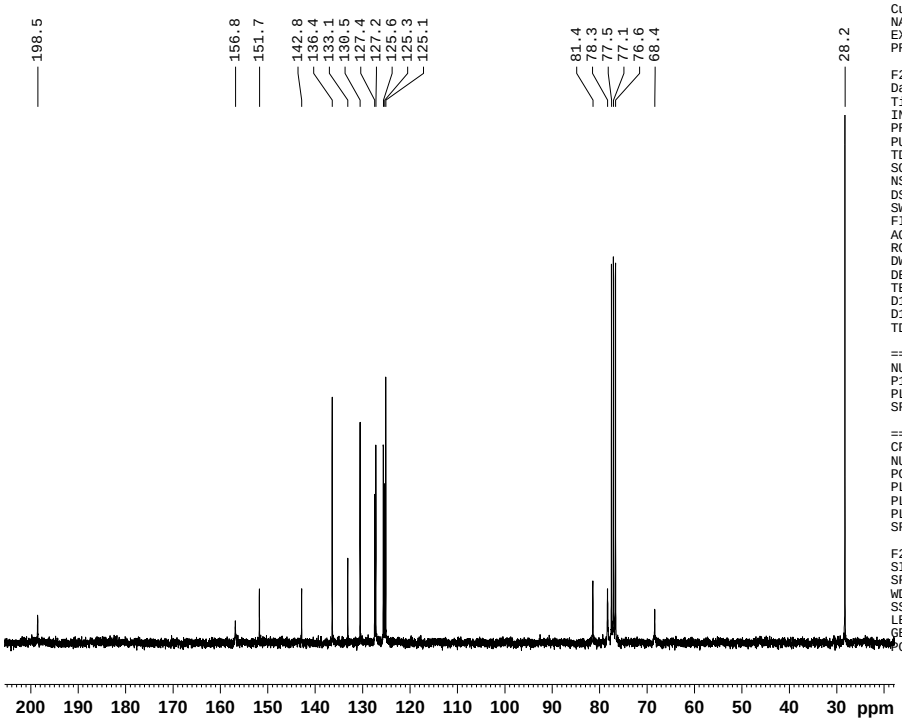

```
Current Data Parameters
NAME                  sfg-585
EXPNO                     100
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110106
Time                    0.40
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       6890
DS                          4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                1.8219508 sec
RG                     4597.6
DW                    27.800 usec
DE                     8.00 usec
TE                     292.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                      12.50 usec
PL1                      2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                      3.00 dB
PL12                    22.33 dB
PL13                    23.00 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                75.4587616 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



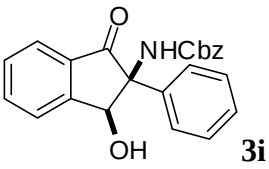
3h



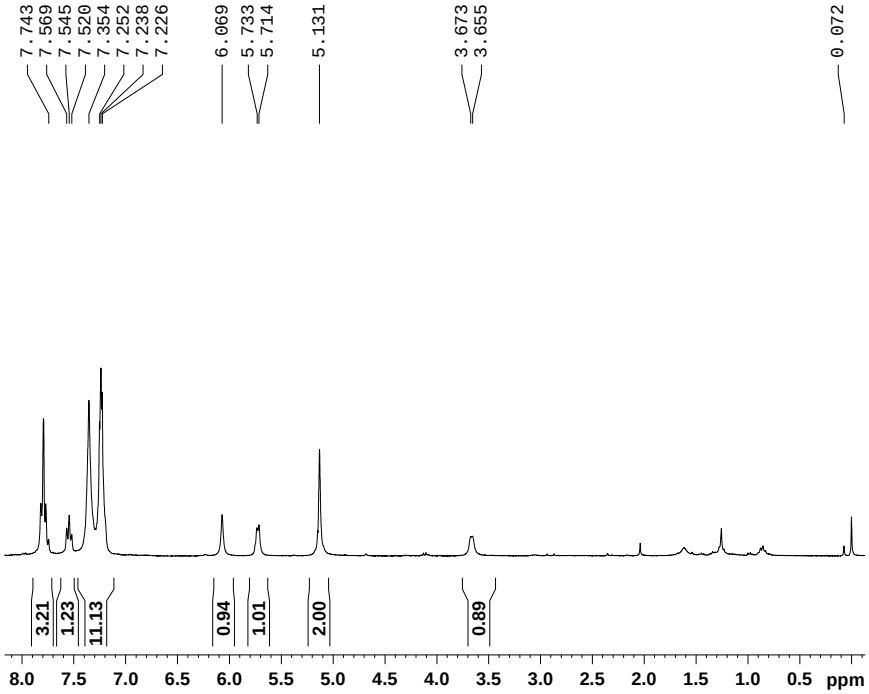

```
Current Data Parameters
NAME                  sfg-646
EXPNO                      30
PROCNO                      1

F2 - Acquisition Parameters
Date_               20101230
Time                   16.42
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        13
DS                         0
SWH                 8992.806 Hz
FIDRES              0.137219 Hz
AQ                3.6438515 sec
RG                       128
DW                    55.600 usec
DE                     8.00 usec
TE                    292.4 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.80 usec
PL1                     3.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                        32768
SF               300.1300049 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



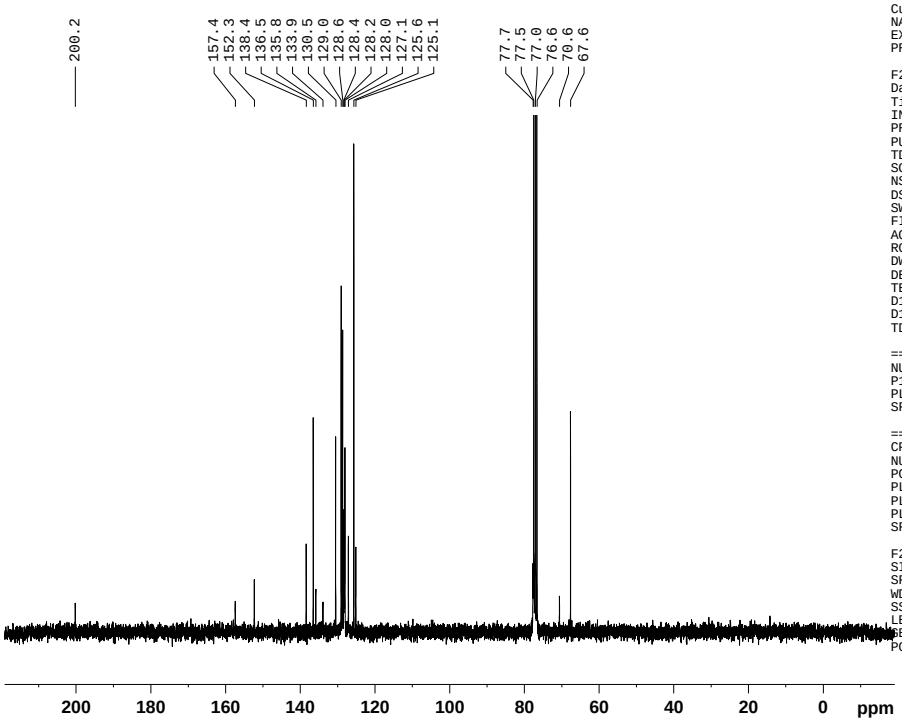

```
Current Data Parameters
NAME                  sfg-646
EXPNO                      31
PROCNO                      1

F2 - Acquisition Parameters
Date_               20101230
Time                   16.45
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       409
DS                         4
SWH                17985.611 Hz
FIDRES              0.274439 Hz
AQ                1.8219508 sec
RG                      8192
DW                    27.800 usec
DE                     8.00 usec
TE                    292.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.50 usec
PL1                     2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     3.00 dB
PL12                   22.33 dB
PL13                   23.00 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF               75.4677490 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```



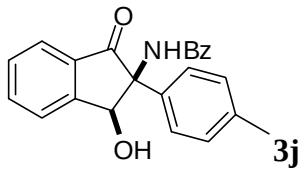
3i



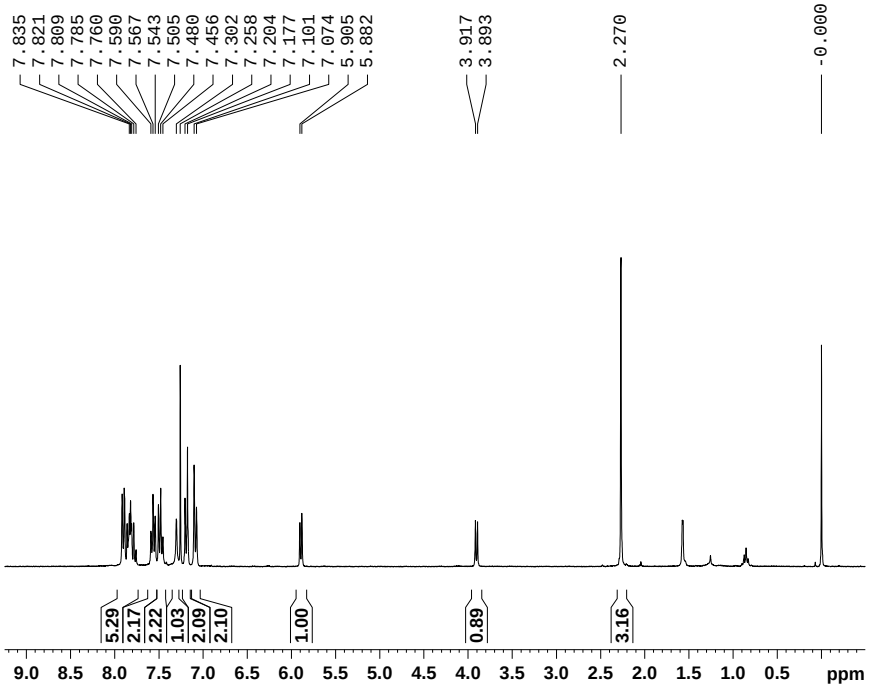

```
Current Data Parameters
NAME              sfg-615
EXPNO                  20
PROCNO                  1

F2 - Acquisition Parameters
Date_           20101028
Time               16.58
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   256
DW                55.600 usec
DE                 8.00 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.80 usec
PL1                 3.00 dB
SFO1         300.1324010 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300079 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



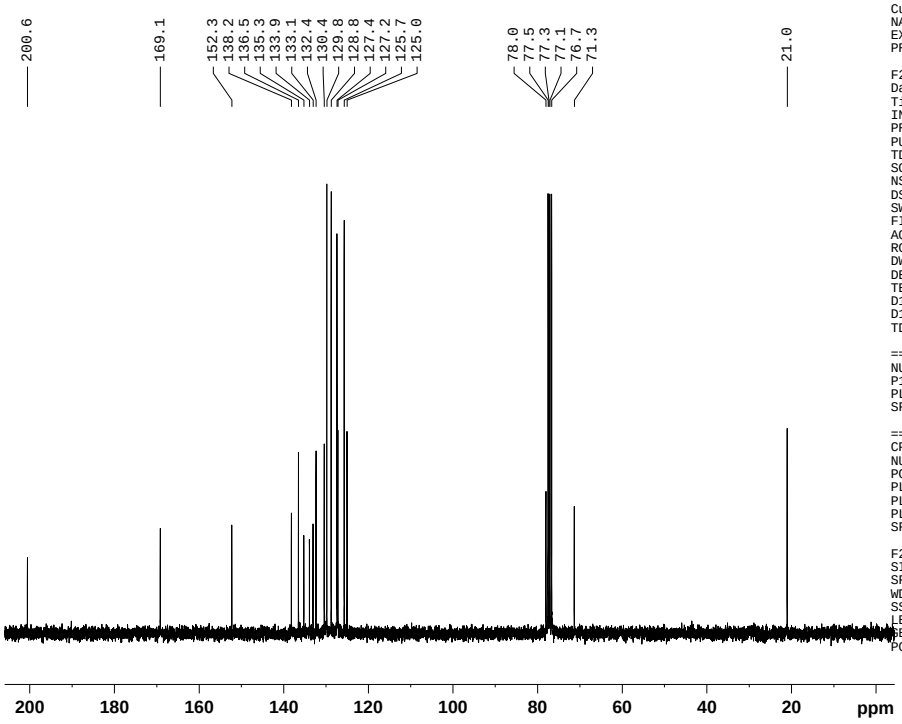

```
Current Data Parameters
NAME              sfg-615
EXPNO                  50
PROCNO                  1

F2 - Acquisition Parameters
Date_           20110106
Time               16.35
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   575
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                  8192
DW                27.800 usec
DE                 8.00 usec
TE                295.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.33 dB
PL13               23.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



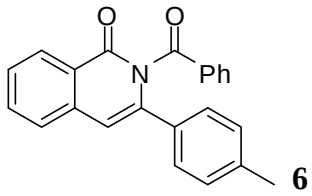
3j



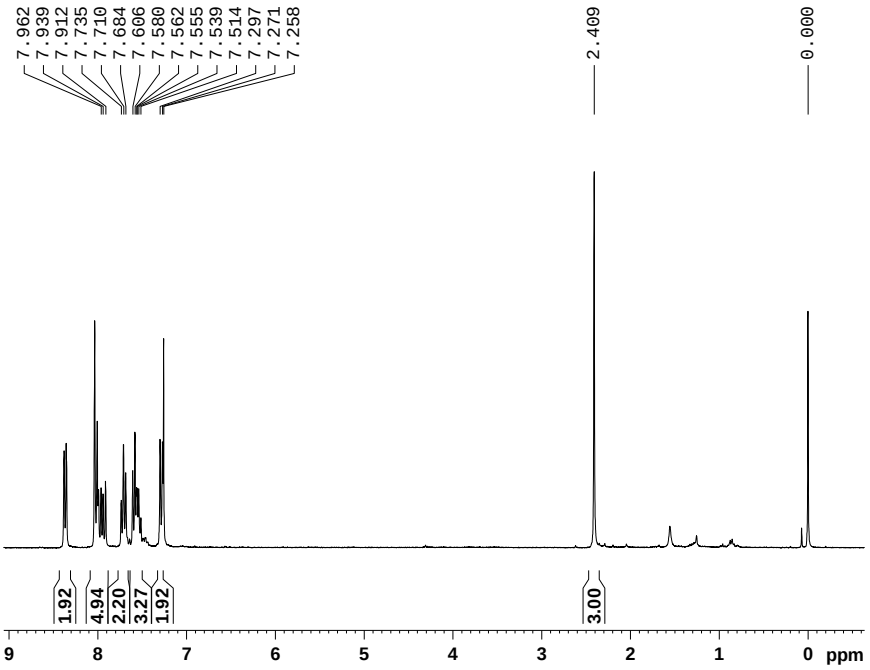

```
Current Data Parameters
NAME                  sfg-620
EXPNO                      20
PROCNO                      1

F2 - Acquisition Parameters
Date_              20101028
Time                  17.02
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8992.806 Hz
FIDRES            0.137219 Hz
AQ                3.6438515 sec
RG                      512
DW                 55.600 usec
DE                  8.00 usec
TE                  297.9 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                  10.80 usec
PL1                  3.00 dB
SFO1          300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF               300.1300066 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    1.00
```



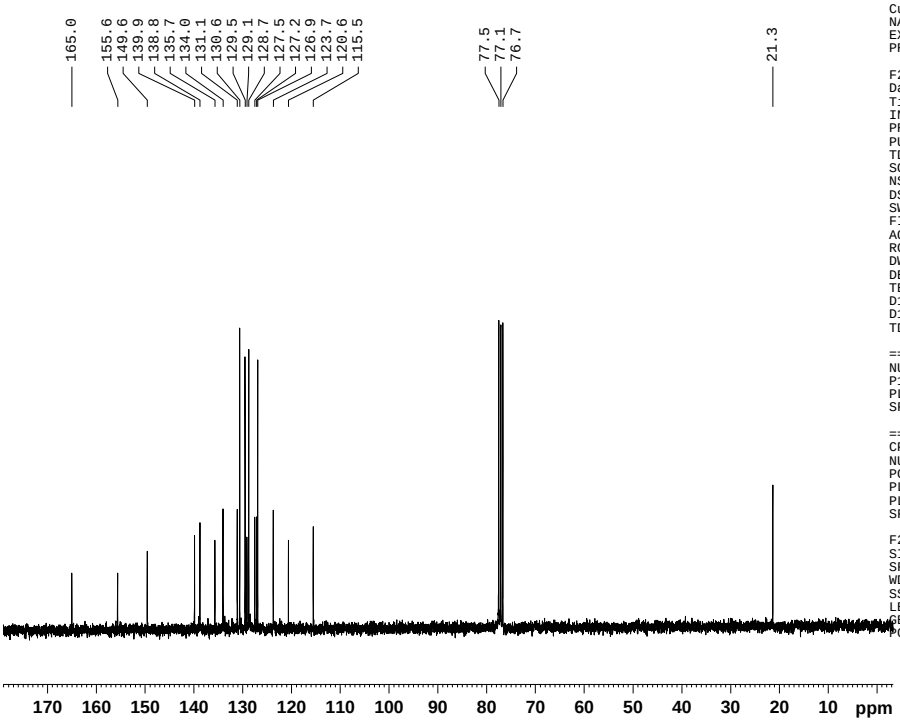

```
Current Data Parameters
NAME                  sfg-620
EXPNO                      31
PROCNO                      1

F2 - Acquisition Parameters
Date_              20101230
Time                  16.01
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      171
DS                        4
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                1.8219508 sec
RG                  11585.2
DW                 27.800 usec
DE                  8.00 usec
TE                  292.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                   13C
P1                  12.50 usec
PL1                  2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                  3.00 dB
PL12                22.33 dB
PL13                23.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                     32768
SF               75.4677490 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```



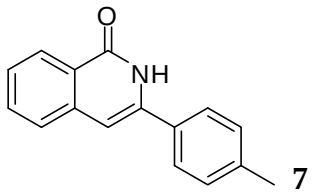
6



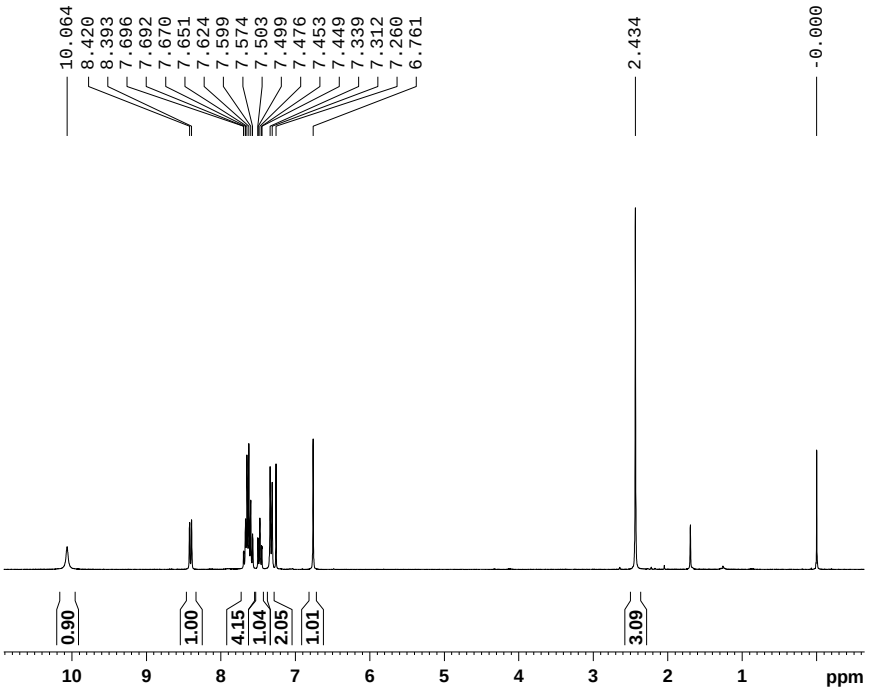

```
Current Data Parameters
NAME                  sfg-746
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110109
Time                   12.35
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8992.806 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                      512
DW                   55.600 usec
DE                    8.00 usec
TE                   295.3 K
D1                2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.80 usec
PL1                      3.00 dB
SFO1            300.1318008 MHz

F2 - Processing parameters
SI                         32768
SF                 300.1300067 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



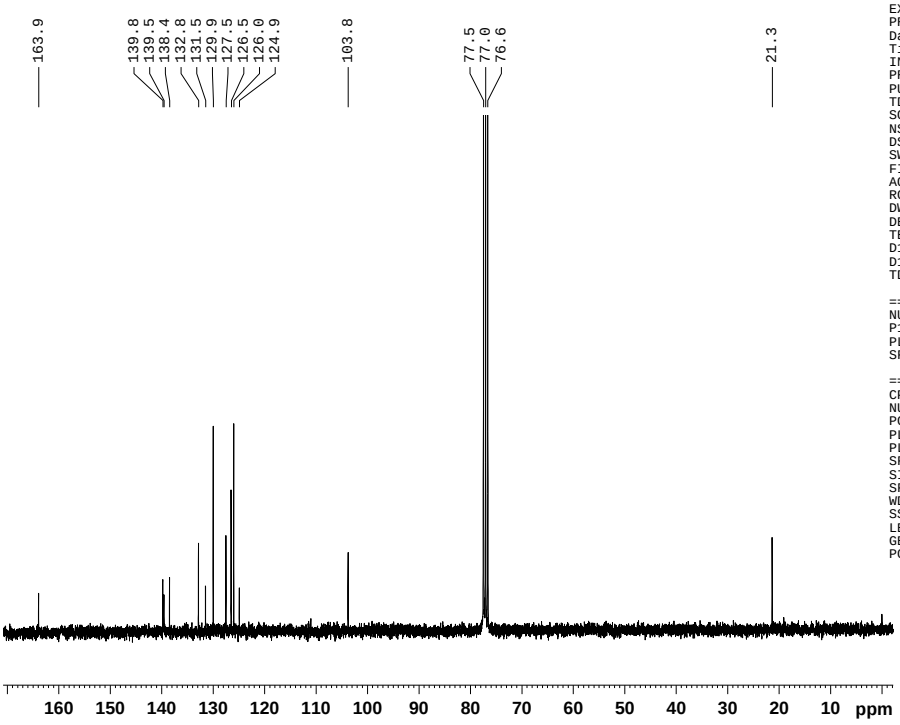

```
Current Data Parameters
NAME                  sfg-746
EXPNO                     111
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110109
Time                   17.40
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG              zgig30
TD                    32768
SOLVENT               CDCl3
NS                      221
DS                        0
SWH               18832.393 Hz
FIDRES             0.574719 Hz
AQ                 0.8700404 sec
RG                    4597.6
DW                   26.550 usec
DE                    8.00 usec
TE                   295.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      12.50 usec
PL1                      2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                      3.00 dB
PL12                    22.33 dB
SFO2            300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                 75.4677490 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



7




```
NAME            sfg-752
EXPNO                21
PROCNO                1
Date_          20110123
Time               9.31
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8992.806 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  362
DW               55.600 usec
DE                8.00 usec
TE               293.9 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.80 usec
PL1                3.00 dB
SFO1         300.1318008 MHz
SI                32768
SF           300.1300061 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```




```
NAME            sfg-752
EXPNO                11
PROCNO                1
Date_          20110123
Time               9.45
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgig30
TD                32768
SOLVENT           CDCl3
NS                  680
DS                    0
SWH           18832.393 Hz
FIDRES         0.574719 Hz
AQ             0.8700404 sec
RG                 4096
DW               26.550 usec
DE                8.00 usec
TE               294.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
SFO2         300.1312005 MHz
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```