Palladium-catalyzed C–N cross coupling of sulfinamides and aryl halides

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Supporting Information

CONTENTS

General Reagent Information

All reactions were carried out under an argon atmosphere. All glassware used was dried in electric oven at 120 ^{o}C. Racemic, (*R*)- and (*S*)-tert-butanesulfinamides are purchased from Jingzhou Winchem Pharm. & Chem. Co., Ltd. All other chemicals were purchased and used as received.

General Analytical Information

All compounds were characterized by ^{1}H NMR, ^{13}C NMR, ESI-MS and IR spectroscopy, and elemental analysis. Copies of the ^{1}H and ^{13}C spectra can be found at the end of the Supporting Information. Nuclear Magnetic Resonance spectra were recorded on a 300MHz instrument or a 400 MHz instrument. All ^{1}H NMR experiments are reported in δ units, parts per million (ppm), and were measured relative to the signals for residual chloroform (7.26 ppm) in the deuterated solvent, unless otherwise stated. All ^{13}C NMR spectra are reported in ppm relative to deuteron-chloroform (77.23 ppm), unless otherwise stated, and all were obtained with ^{1}H decoupling. All IR spectra were taken on an infrared spectrometer. All chiral HPLC analyses were performed on a liquid chromatography with a Chiralcel OD-H chiral column (4.6 mm × 250 mm × 5 μm). All rotation data are recorded on an auto rotation (Na D line, cell long 10cm, λ = 589 nm). Electron-spraying ionization mass spectra are recorded on an LC/MS instrument. Elemental analyses of these compounds are performed on an elemental analyzer.

X-ray structure determination

A single crystal of (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide was cultured from a solution of petroleum ether and ethyl acetate in a test tube. The single-crystal data were collected on an CCD diffractometer. X-ray generator was operated at 50 kV and 1 mA using an Enhance Mo*Kα* radiation. Data were collected with a ω scan width of 1°. The data reduction, empirical absorption correction, and space group determination were done using CrysAlisPro RED (Oxford Diffraction, 2009). The crystal structure was solved by direct methods and refined by full-matrix leasts quares method using SHELXL97, present in the program suite WinGx (Version 1.63.04a). The molecular diagrams were generated using ORTEP 3 and the packing diagrams were generated using CAMERON. Geometrical calculations were done using PARST95 and PLATON. The positions of all H atoms were fixed geometrically and refined isotropically using the riding atom model.

Crystal Data for (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide 3g. $C_{11}H_{18}NO_2S$, M =228.32, Orthorhombic, a = 7.4418(9) Å, b = 9.7027(12) Å, c = 16.862(2) Å, U = 1217.5(3) Å^3, T = 293.0, space group $P2_12_12_1$ (no. 19), Z = 4, μ(Mo Kα) = 0.248, 7010 reflections measured, 2481 unique (R_{int} = 0.0309) which were used in all calculations. The final $wR(F_2)$ was 0.0916 (all data).

Description about the single crystal (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide 3g.

All of the *N*-aryl sulfinamides are crystalline solid and readily to crystalize. In order to probe the internal reasons of these crystalline *N*-aryl sulfinamides, three-dimensional molecular structures and crystal-packing modes of *N*-aryl *tert*-butanesulfinamide, a crystal of (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide was cultured and analyzed.[1] The ORTEP style plot (Figure 1) shows the three dimension structure of (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide **3g**, and its stereo-structure has uniform configuration (Figure 2) and is consistent with (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide (Figure 1). The crystal stacking plot (Figure 2) clearly demonstrates the strong N–H···O=S hydrogen bonds in a head-to-tail fashion throughout the single crystal, which is

different from the racemic *N*-aryl tert-butanesulfinamides with a couple of hydrogen bonds between (*R*)- and (*S*)-isomers.[2] The intermolecular hydrogen bonds throughout the single crystal should be the main intermolecular force to keep *N*-aryl *tert*-butanesulfinamides in crystalline solid and make the melting points of our single enantiomer products commonly higher than that of the corresponding racemic ones.[2]

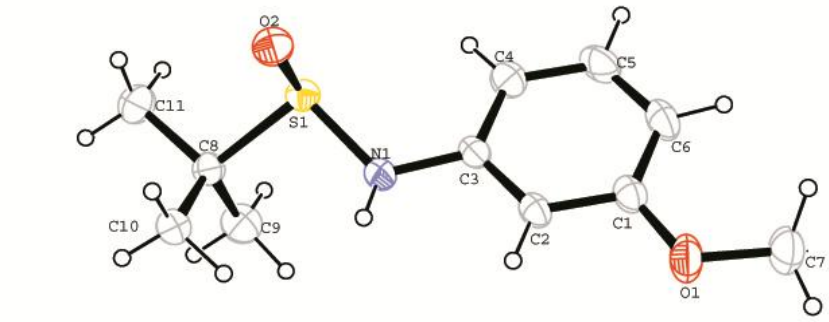


Figure 1. ORTEP style plot of (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide **3g** in the solid state. Thermal ellipsoids are drawn at the 50% probability level.

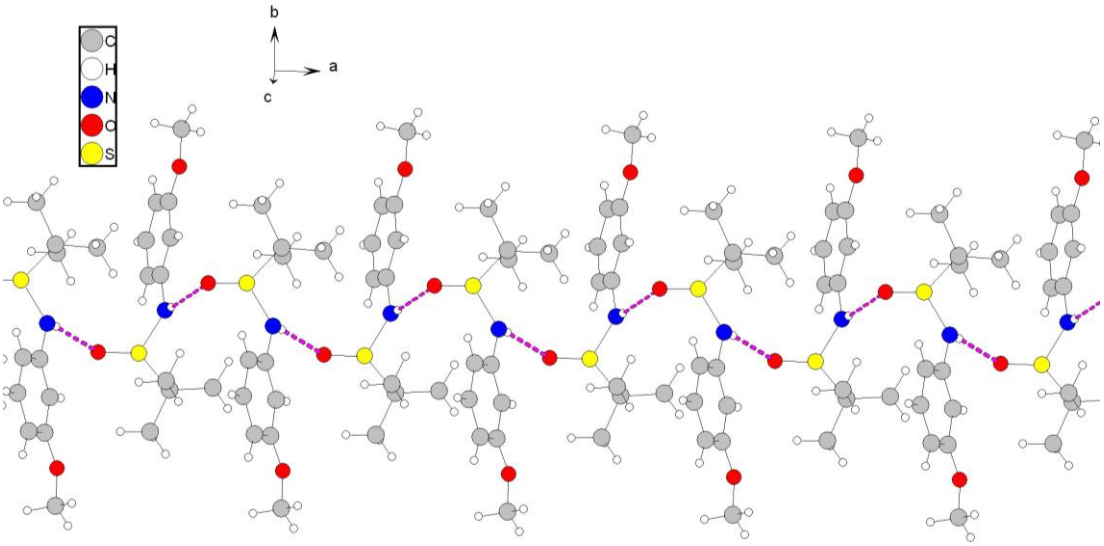


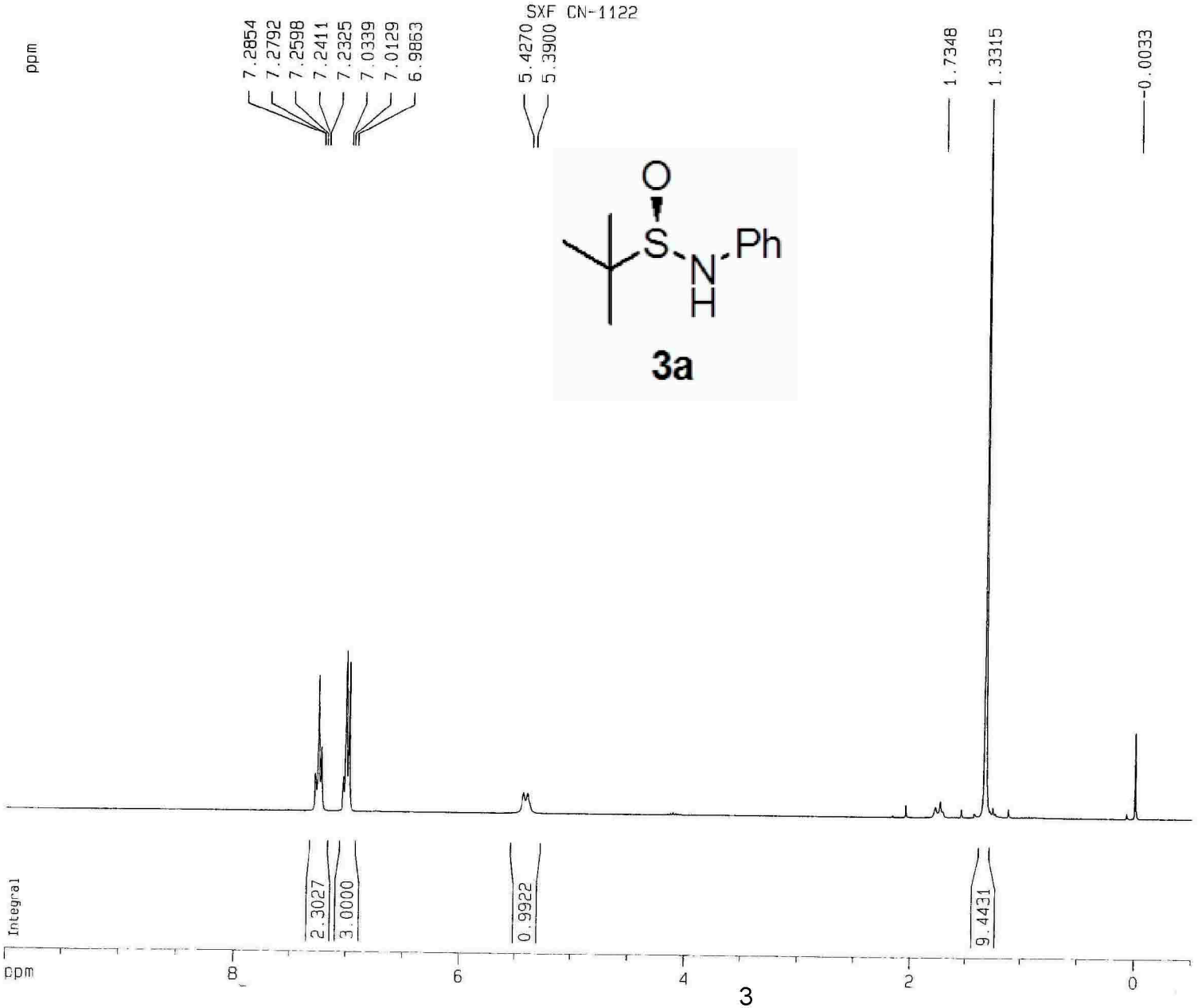
Figure 2. Crystal stacking plot of (*R*)-*N*-(3-methoxyphenyl) *tert*-butanesulfinamide **3g**.

References

(1) CCDC 862954 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambrige Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

(2) (a) Datta, M.; Buglass, A. J.; Elsegood, M. R. J. *Acta Cryst.* **2009**, *E65*, o2034. (b) Datta, M.; Buglass, A. J.; Elsegood, M. R. J. *Acta Cryst.* **2010**, *E66*, o109. (c) Datta, M.; Buglass, A. J.; Elsegood, M. R. J. *Acta Cryst.* **2009**, *E65*, o2823.

Table 2, entry 1



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FIDRES	0.182959 Hz
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RG	128
DW	83.400 usec
DE	6.00 usec
TE	294.1 K
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MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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PL1	0.10 dB
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F2 - Processing parameters

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SF	300.1300125 MHz
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SSB	0
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PC	1.00

1D NMR plot parameters

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CY	30.00 cm
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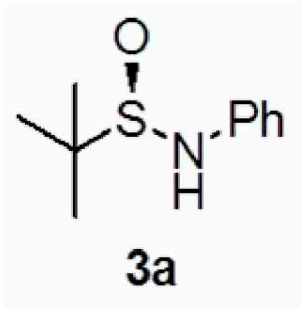
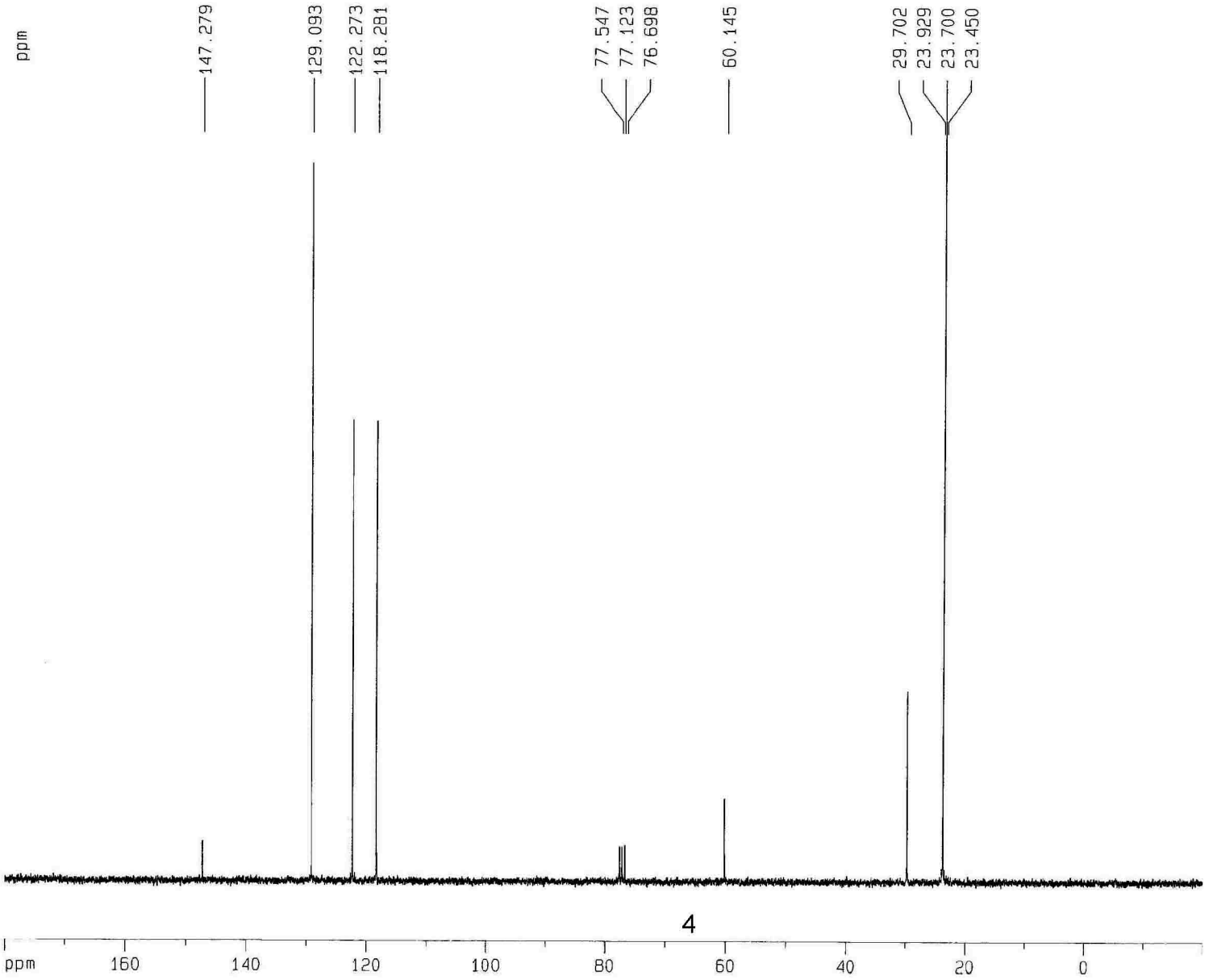
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.52500 ppm/cm
HZCM	157.56825 Hz/cm

Table 2, entry 1

QL-002



3a

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PL1               -0.81 dB
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PCPD2            80.00 usec
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PL12             17.74 dB
PL13             17.74 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
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SF            75.4677512 MHz
WDW                    EM
SSB                     0
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GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  30.00 cm
F1P                180.000 ppm
F1               13584.20 Hz
F2P                -20.000 ppm
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PPMCM             10.00000 ppm/cm
HZCM             754.67749 Hz/cm
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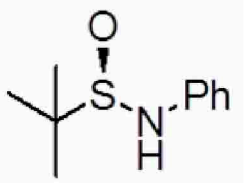
Table 2, entry 1

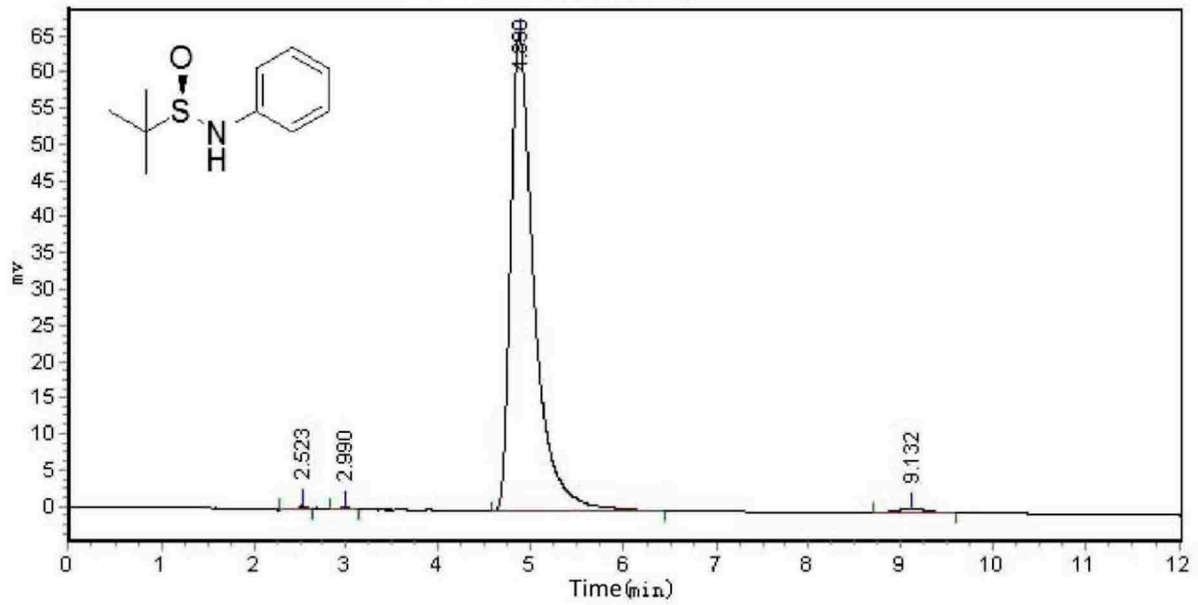
(R)-N-phenyl tert-butanesulfinamide



3a

Affiliation: Chengdu University of Technology
Operator: Xiaofei Sun
Date: 2011-01-15, 07:02:50 pm

HPLC Spectrum(R01.dat)



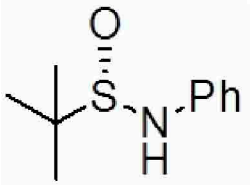
Analytical Result Table

Entry	Peak name	Retention time	Peak height	Peak area	Content
1		2.523	567.277	3837.419	0.0000
2		2.990	362.474	2489.500	0.0000
3	R	4.890	65930.711	1177633.625	0.0000
4	S	9.132	620.963	15766.500	0.0000
Total			67481.424	1199727.044	0.0000

2011-01-15

Table 2, entry 2

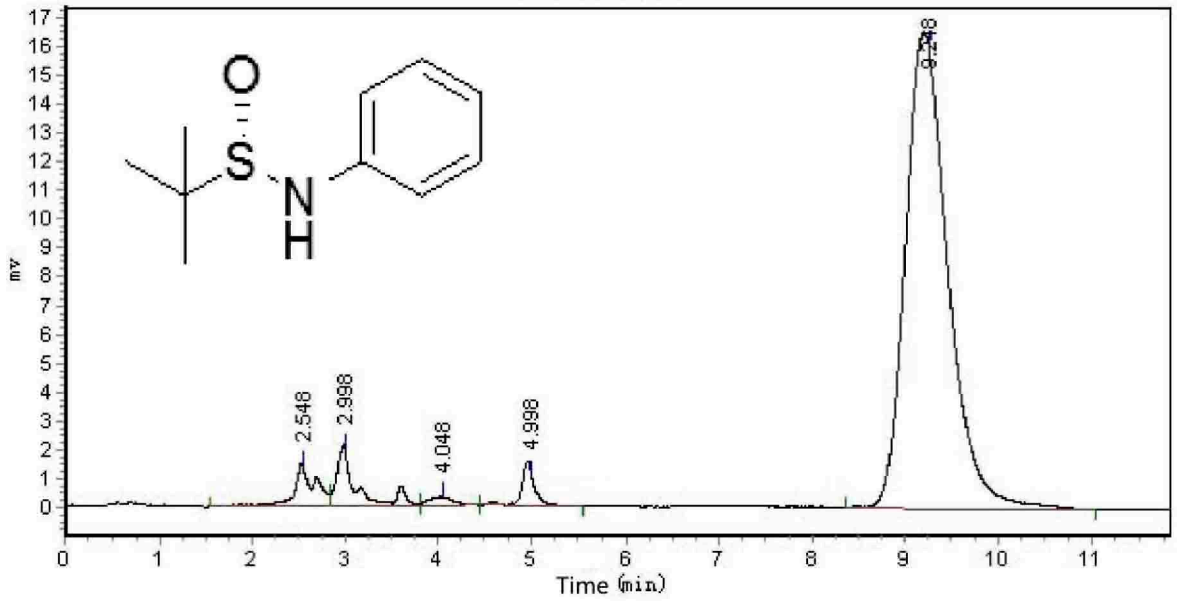
(S)-N-phenyl tert-butanesulfinamide



3b

Affiliation: Chengdu University of Technology
Operator: Xiaofei Sun
Date: 2011-01-15, 06:36:23 pm

HPLC Spectrum(S03.dat)



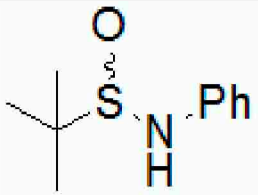
Analytical Result Table

Entry	Peak name	Retention time	Peak height	Peak area	Content
1		2.548	1363.931	23716.303	0.0000
2		2.998	2010.000	28350.172	0.0000
3		4.048	270.828	5021.024	0.0000
4		4.998	1385.000	14397.400	0.0000
5		9.248	16316.000	523086.000	0.0000
Total			21345.759	594570.899	0.0000

2011-01-15

Table 2, entry 3

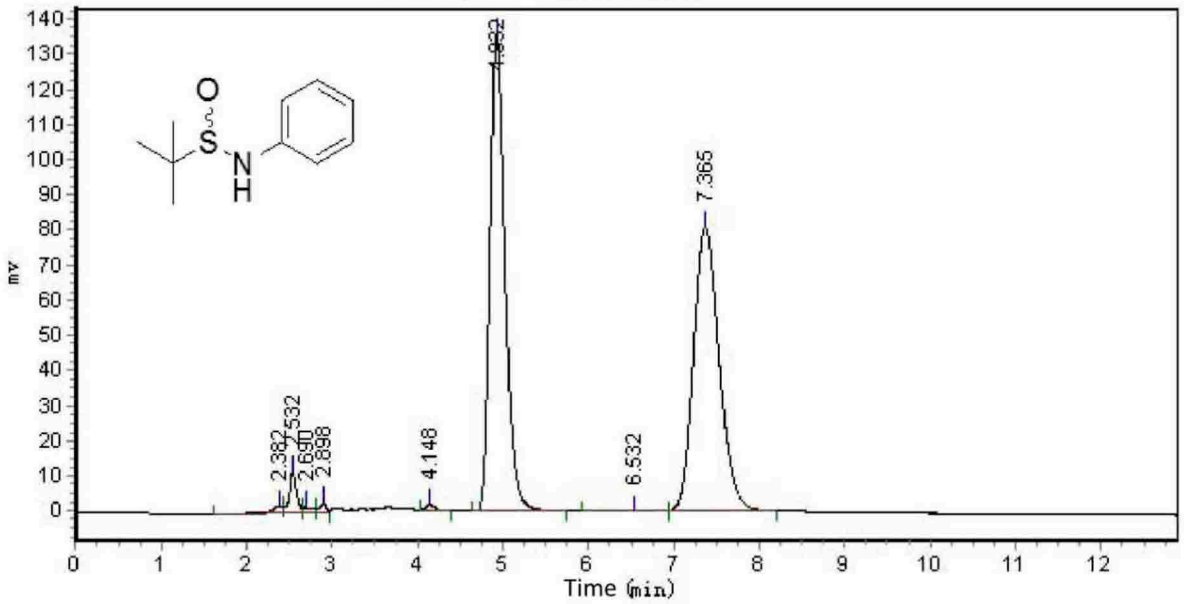
Racemic N-phenyl tert-butanesulfinamide



3c

Affiliation: Chengdu University of Technology
Operator: Xiaofei Sun
Date: 2011-01-15, 05:13:51 pm

HPLC Spectrum(rac01.dat)



Analytical Result Table

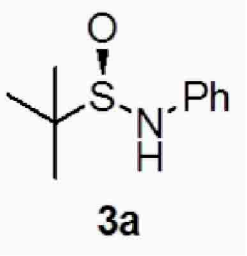
Entry	Peak name	Retention time	Peak height	Peak area	Content
1		2.382	1901.000	16946.096	0.0000
2		2.532	11262.392	63321.168	0.0000
3		2.690	1308.304	11017.030	0.0000
4		2.898	2293.348	11354.555	0.0000
5		4.148	1606.273	12283.800	0.0000
6	R	4.932	135888.641	1664425.125	0.0000
7		6.532	359.618	12779.982	0.0000
8	S	7.365	80747.813	1690092.375	0.0000
Total			235367.387	3482220.131	0.0000

2011-01-15

Table 2, entry 1

(*R*)-*N*-Phenyl *tert*-butanesulfinamide

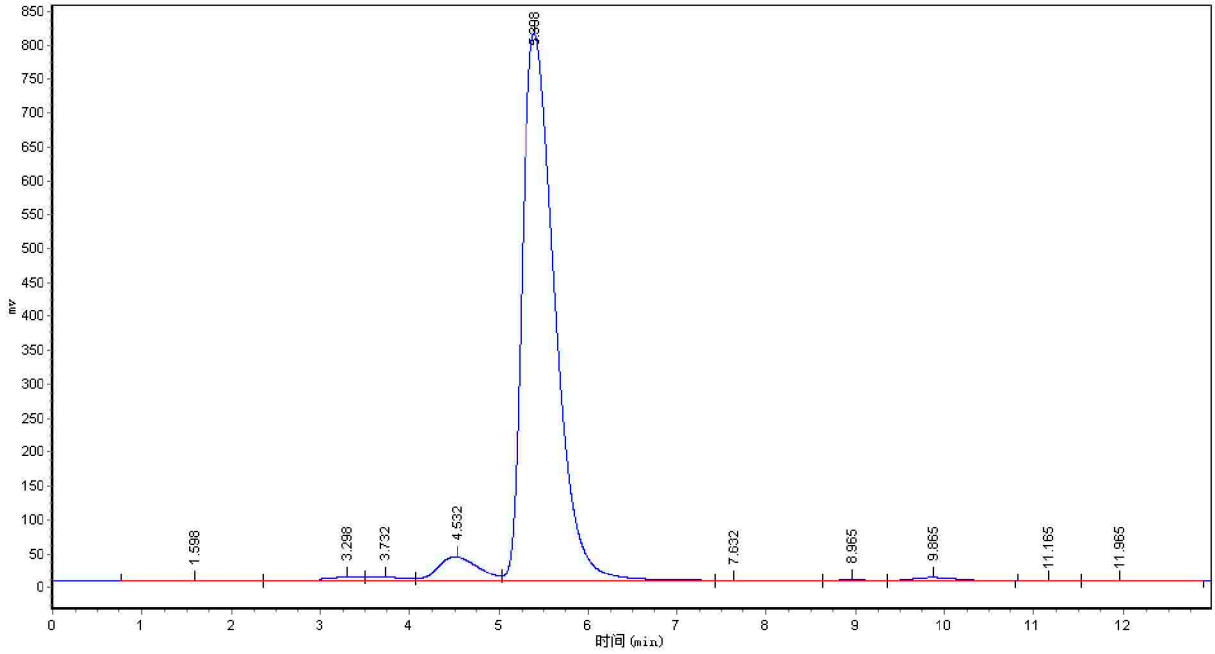
(Re-analyzed on March 3, 2012)



3a

Affiliation: Chengdu University of Technology
Date: 2012-03-03
Operator: Xingzhao Tu

HPLC, Diacel Chiralcel OD-H column, 90:10 hexanes/2-propanol, 1 mL/min, 254 nm.

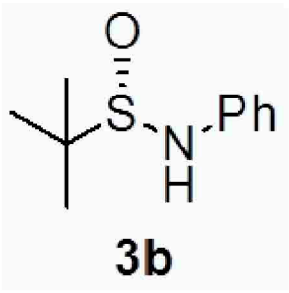


No.	Peak name	Retention time	Peak height	Peak area	%Area
1		1.598	180.896	8894.000	0.0400
2		3.298	6251.119	167816.031	0.7554
3		3.732	6260.245	182906.109	0.8233
4		4.532	36682.633	1171849.375	5.2751
5	R form	5.398	804724.875	20392360.000	91.7958
6		7.632	1120.383	58378.520	0.2628
7		8.965	2252.696	46997.949	0.2116
8	S form	9.865	5018.881	180851.250	0.8141
9		11.165	60.742	1450.914	0.0065
10		11.965	100.323	3412.370	0.0154

Table 2, entry 2

(*S*)-*N*-Phenyl *tert*-butanesulfinamide

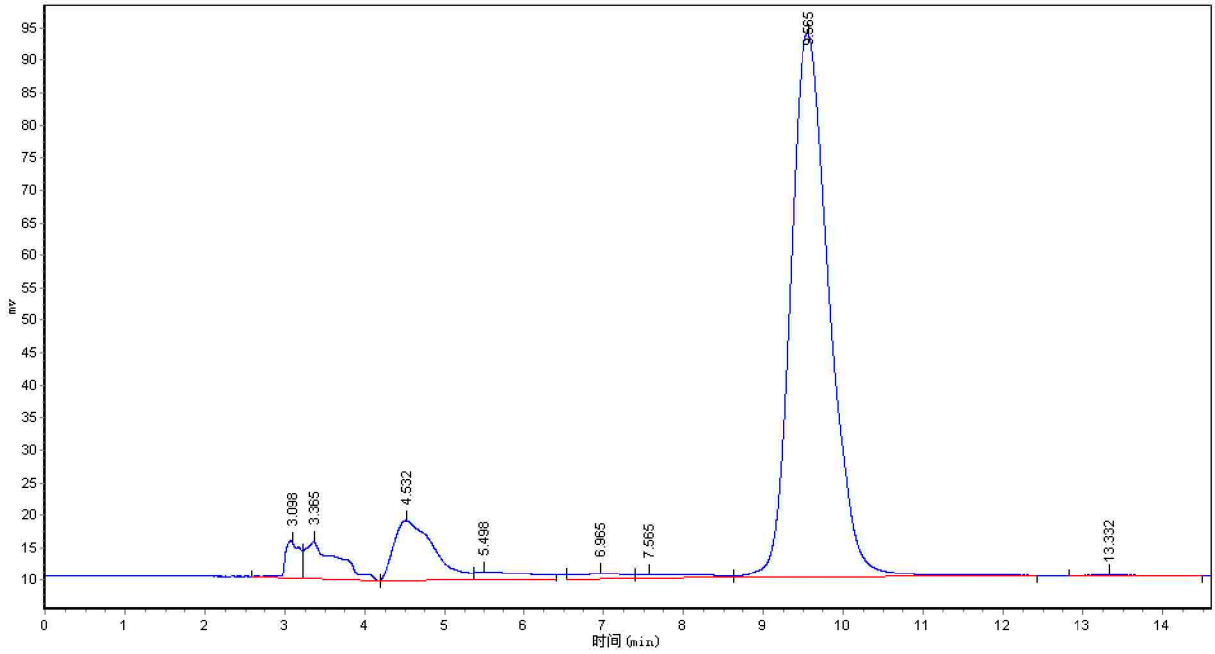
(Re-analyzed on March 2, 2012)



3b

Affiliation: Chengdu University of Technology
Date: 2012-03-02
Operator: Xingzhao Tu

HPLC, Diacel Chiralcel OD-H column, 90:10 hexanes/2-propanol, 1 mL/min, 254 nm.

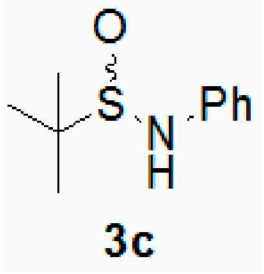


No.	Peak name	Retention time	Peak height	Peak area	content
1		3.098	5633.063	71939.641	2.0452
2		3.365	5659.563	162936.469	4.6321
3		4.532	9255.604	323006.406	9.1827
4	R form	5.498	1323.053	64346.371	1.8293
5		6.965	893.907	41569.352	1.1818
6		7.565	667.393	40414.406	1.1489
7	S form	9.565	83716.016	2811175.500	79.9181
8		13.332	45.400	2184.300	0.0621

Table 2, entry 3

Racemic *N*-Phenyl *tert*-butanesulfinamide

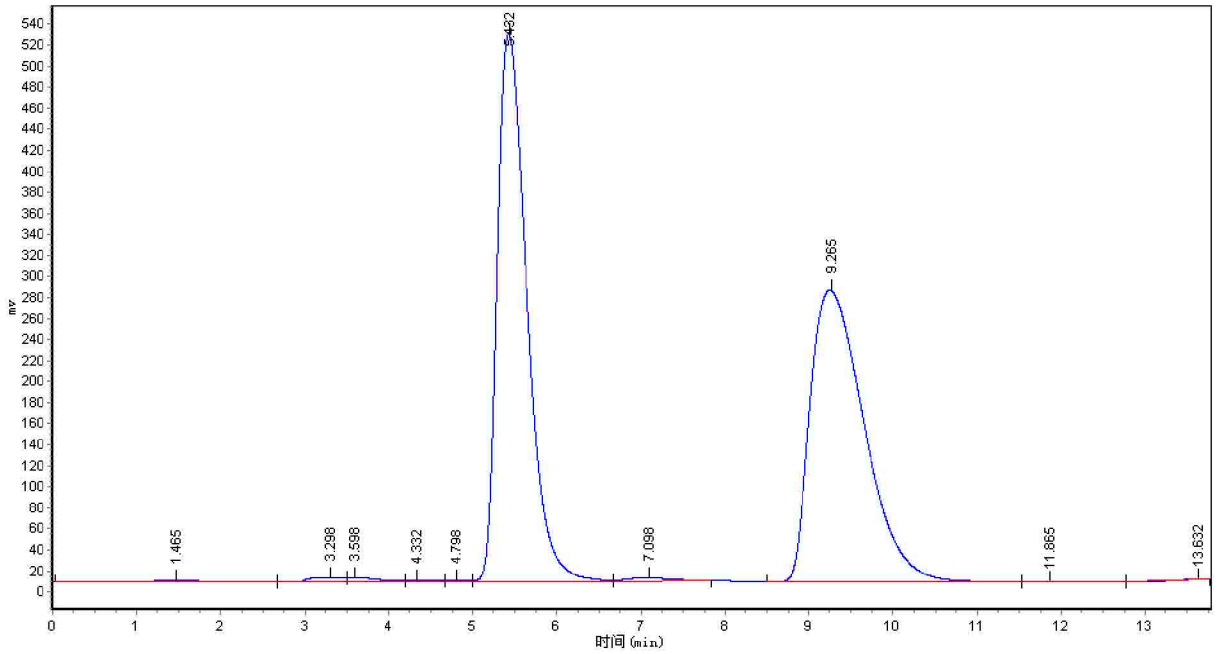
(Re-analyzed on March 3, 2012)



3c

Affiliation: Chengdu University of Technology
Date: 2012-03-03
Operator: Xingzhao Tu

HPLC, Diacel Chiralcel OD-H column, 90:10 hexanes/2-propanol, 1 mL/min, 254 nm.
(*R*)-N-phenyl *tert*-butanesulfinamide, r_t = 5.4 min; (*S*)-*N*-phenyl *tert*-butanesulfinamide, r_t = 9.2 min.



No.	Peak name	Retention time	Peak height	Peak area	%Area
1		1.465	2041.278	71299.898	0.2789
2		3.298	4163.574	111637.867	0.4367
3		3.598	3542.793	93777.305	0.3669
4		4.332	1097.774	24640.658	0.0964
5		4.798	936.671	15603.623	0.0610
6	R form	5.432	520036.250	12750765.000	49.8833
7		7.098	3339.019	109928.320	0.4301
8	S form	9.265	277306.156	12364625.000	48.3727
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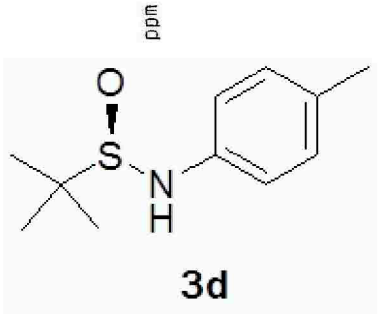
Table 2, entry 4



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FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 291.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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PL1 0.10 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
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WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

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CY 30.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
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F2 -150.06 Hz
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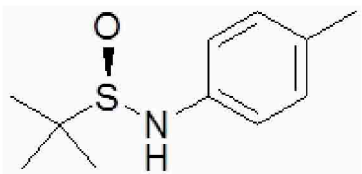
C-CN0725-1



3d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PROCNO                   1

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NS                     71
DS                      4
SWH            22675.736 Hz
FIDRES         0.346004 Hz
AQ             1.4451183 sec
RG                   8192
DW               22.050 usec
DE                6.00 usec
TE                299.1 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
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P1                10.50 usec
PL1               -0.81 dB
SFO1          75.4760505 MHz

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PL2                0.10 dB
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SFO2         300.1312005 MHz

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

1D NMR plot parameters
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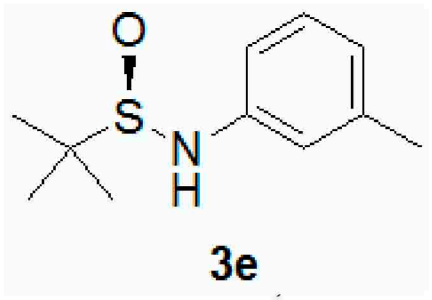

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PROCNO	1

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TD	32768
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NS	8
DS	0
SWH	5995.204 Hz
FIDRES	0.182959 Hz
AQ	2.7329011 sec
RG	128
DW	83.400 usec
DE	6.00 usec
TE	292.7 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	10.50 usec
PL1	0.10 dB
SF01	300.1321009 MHz

F2 - Processing parameters

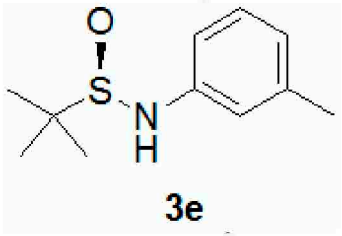
SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.52500 ppm/cm
HZCM	157.56825 Hz/cm

Table 2, entry 5

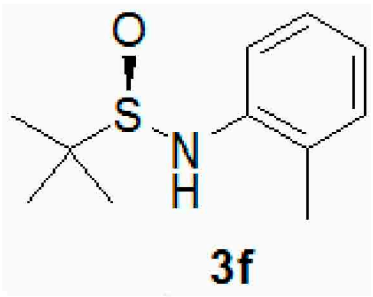
C-CN0308-4



3e


```
Current Data Parameters
NAME          zql-2011-52
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_          20110524
Time              16.20
INSTRUM           av300
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG                 8192
DW               22.050 usec
DE                6.00 usec
TE               299.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PL1               -0.81 dB
SFO1         75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            80.00 usec
PL2               0.10 dB
PL12             17.74 dB
PL13             17.74 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                 65536
SF           75.4677580 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
CY               15.00 cm
F1P             200.500 ppm
F1            15131.29 Hz
F2P              -5.500 ppm
F2             -415.07 Hz
PPMCM         10.30000 ppm/cm
HZCM         777.31793 Hz/cm
```

Table 2, entry 6



3f

Current Data Parameters
NAME zql-2011-13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110328
Time 17.35
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 292.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

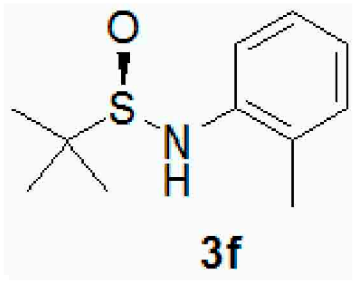
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

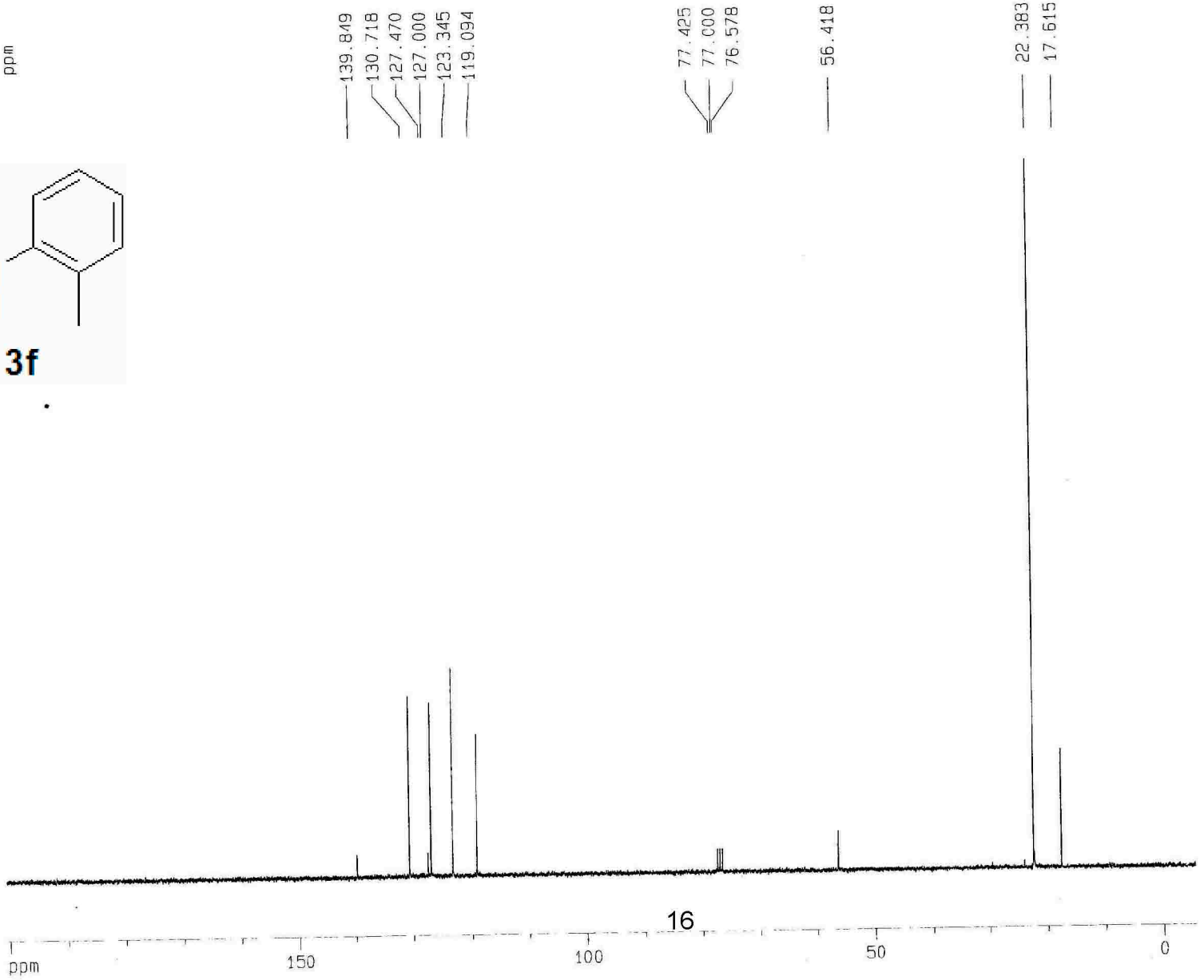
1D NMR plot parameters
CX 20.00 cm
CY 35.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Table 2, entry 6

C-CN0308-3



3f




```
Current Data Parameters
NAME                  zql-2011-13
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20110328
Time                        17.40
INSTRUM                    av300
PROBHD     5 mm QNP 1H/13
PULPROG                     zgpg
TD                         65536
SOLVENT                    CDCl3
NS                             64
DS                              4
SWH                   22675.736 Hz
FIDRES                 0.346004 Hz
AQ                    1.4451188 sec
RG                           8192
DW                      22.050 usec
DE                       6.00 usec
TE                        293.2 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                          13C
P1                         10.50 usec
PL1                        -0.81 dB
SFO1                  75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         0.10 dB
PL12                       17.74 dB
PL13                       17.74 dB
SFO2                 300.1312005 MHz

F2 - Processing parameters
SI                            65536
SF                    75.4677603 MHz
WDW                              EM
SSB                               0
LB                             1.00 Hz
GB                                0
PC                             1.40

1D NMR plot parameters
CX                            20.00 cm
CY                            12.00 cm
F1P                         200.500 ppm
F1                        15131.29 Hz
F2P                          -5.500 ppm
F2                         -415.07 Hz
PPMCM                     10.30000 ppm/cm
HZCM                    777.31793 Hz/cm
```

Table 2, entry 7

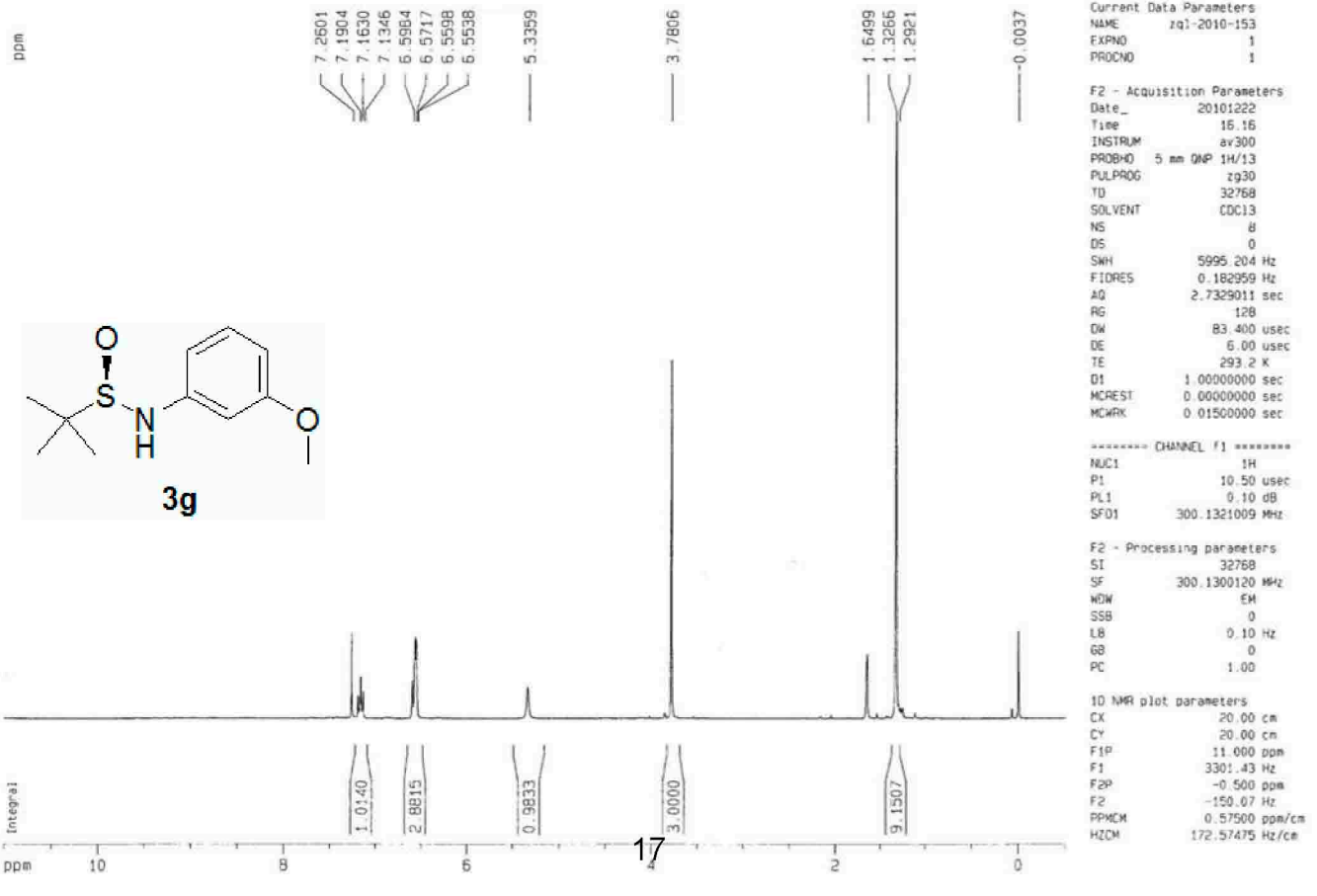
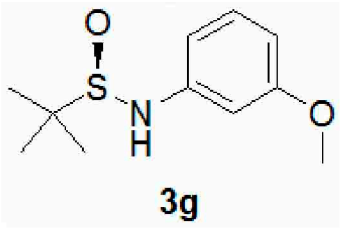
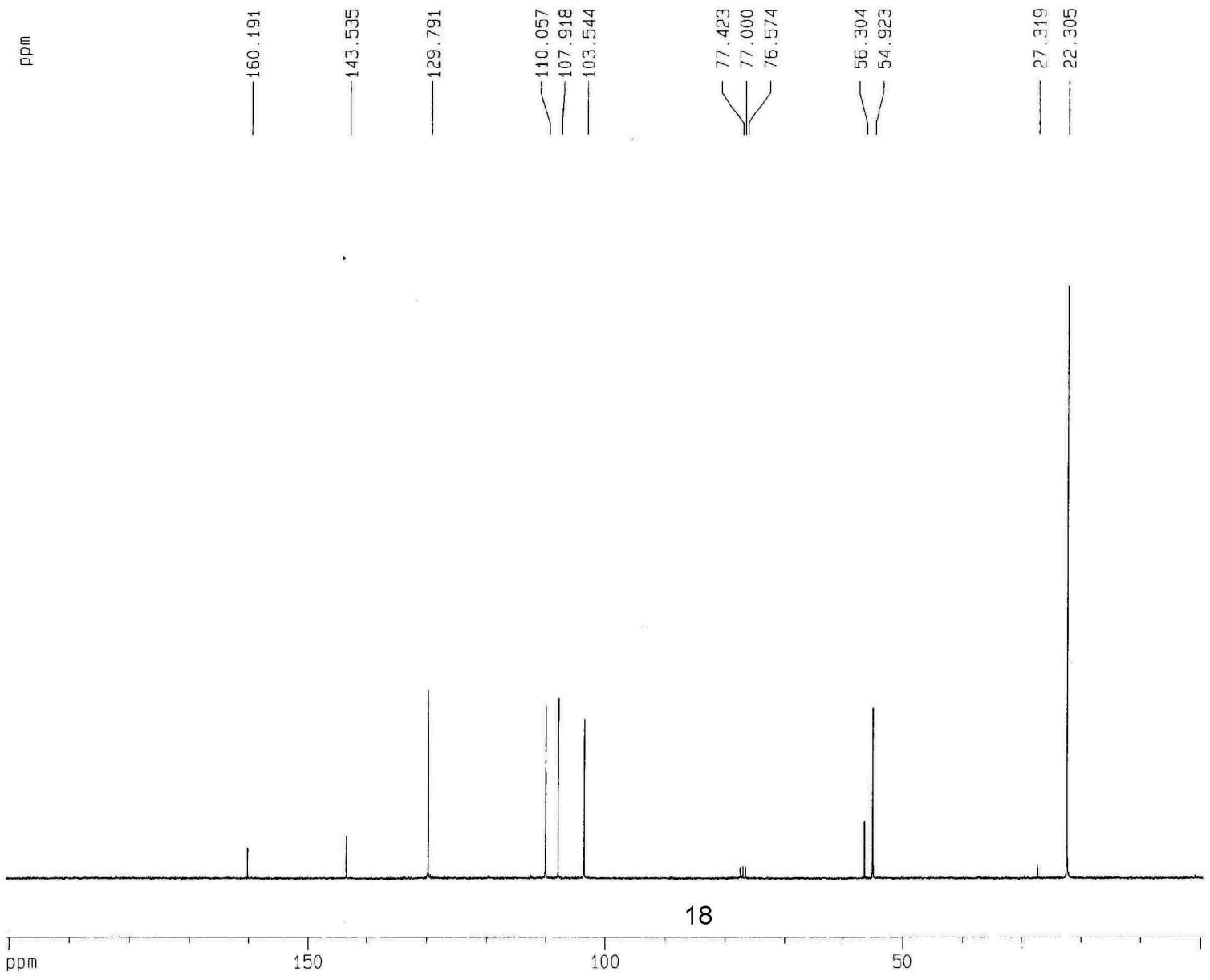


Table 2, entry 7



3g

sxf
C-CN1216a



Current Data Parameters
NAME zq1-2010-160
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101228
Time 11 33
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT MeOD
NS 128
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 293.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

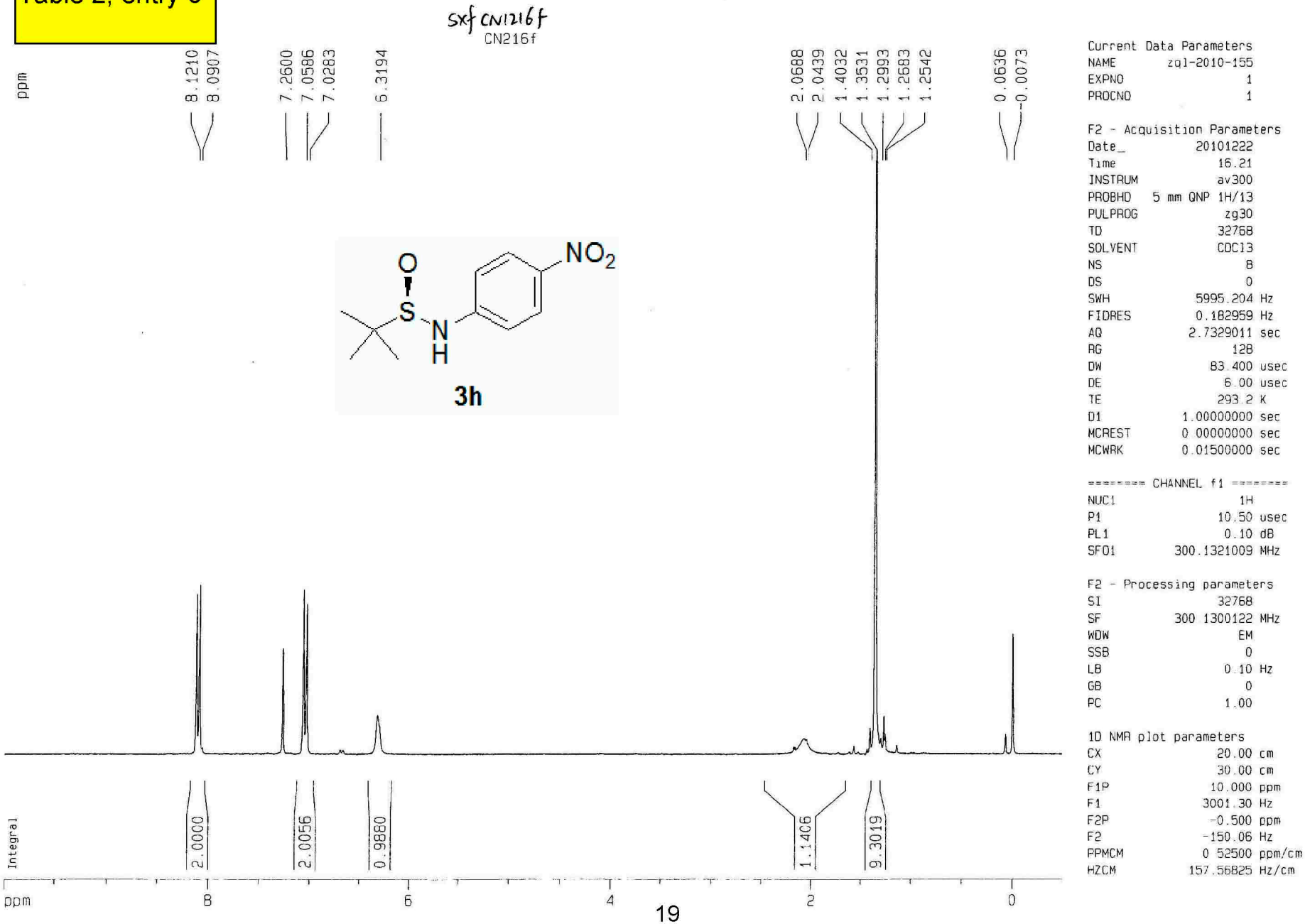
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677648 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.05000 ppm/cm
HZCM 758.45105 Hz/cm

Table 2, entry 8



Current Data Parameters
NAME zql-2010-155
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101222
Time 16.21
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

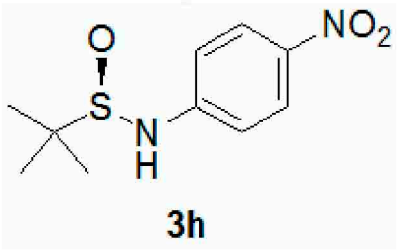
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

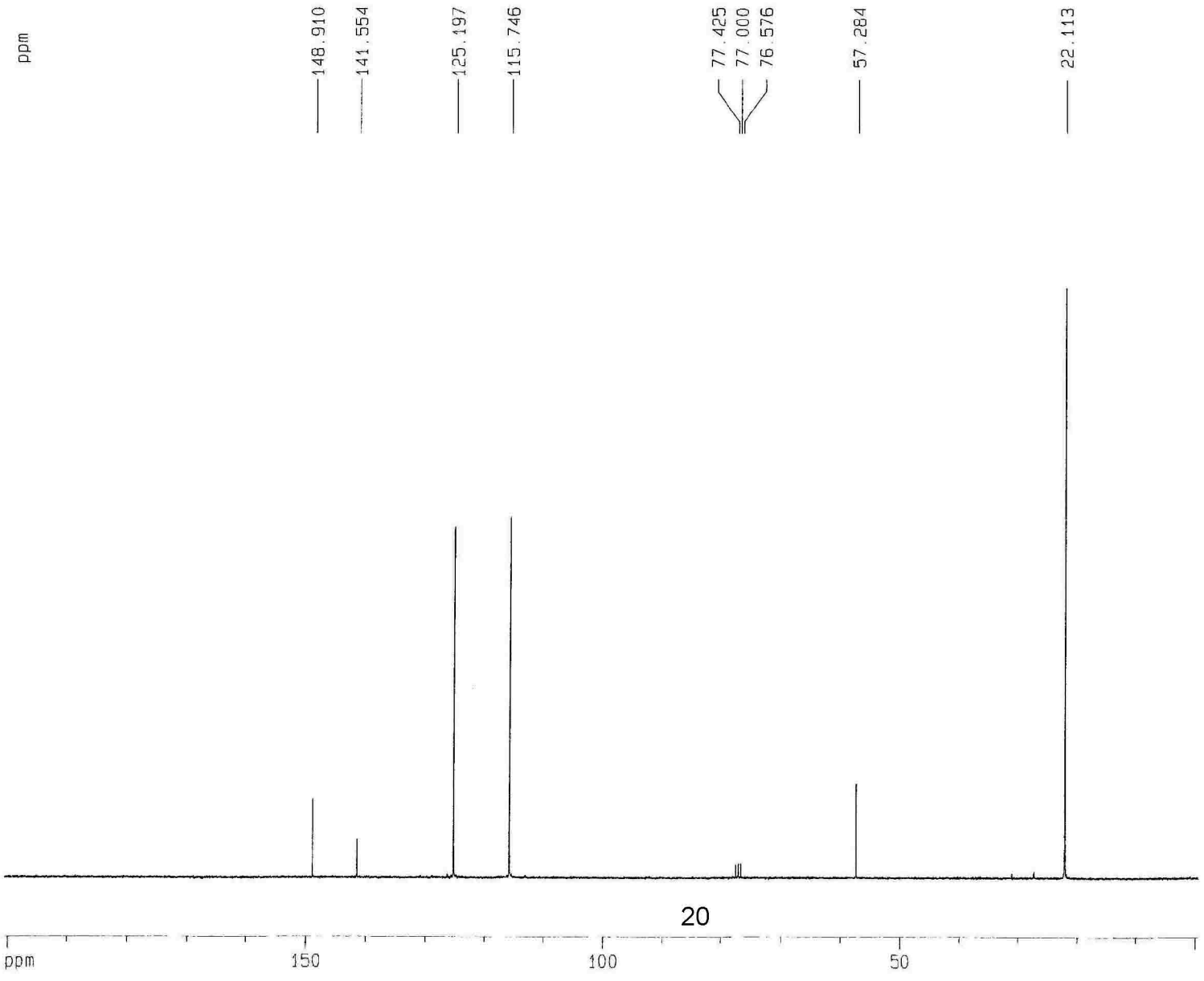
1D NMR plot parameters
CX 20.00 cm
CY 30.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Table 2, entry 8

sxf
C-CN 1216f

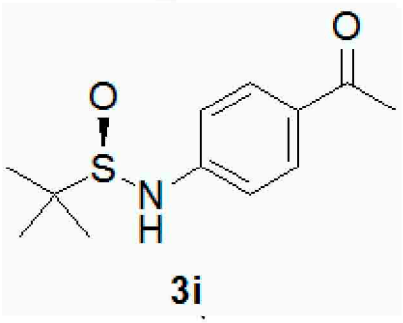


3h




```
Current Data Parameters
NAME          zql-2010-162
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101228
Time                 15.29
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg
TD                   65536
SOLVENT               MeOD
NS                      76
DS                       4
SWH           22675.736 Hz
FIDRES         0.346004 Hz
AQ             1.4451188 sec
RG                    8192
DW               22.050 usec
DE                6.00 usec
TE                293.5 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                10.50 usec
PL1                -0.81 dB
SFO1          75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             80.00 usec
PL2                0.10 dB
PL12              17.74 dB
PL13              17.74 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                    65536
SF           75.4677650 MHz
WDW                      EM
SSB                       0
LB                 1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              200.500 ppm
F1             15131.29 Hz
F2P               -0.500 ppm
F2              -37.73 Hz
PPMCM          10.05000 ppm/cm
HZCM          758.45105 Hz/cm
```

Table 2, entry 9

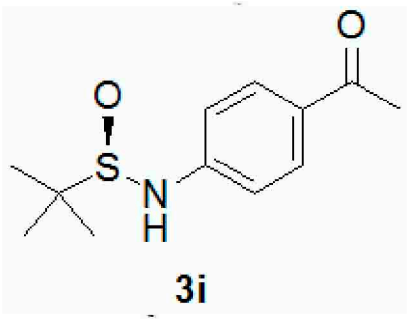


3i

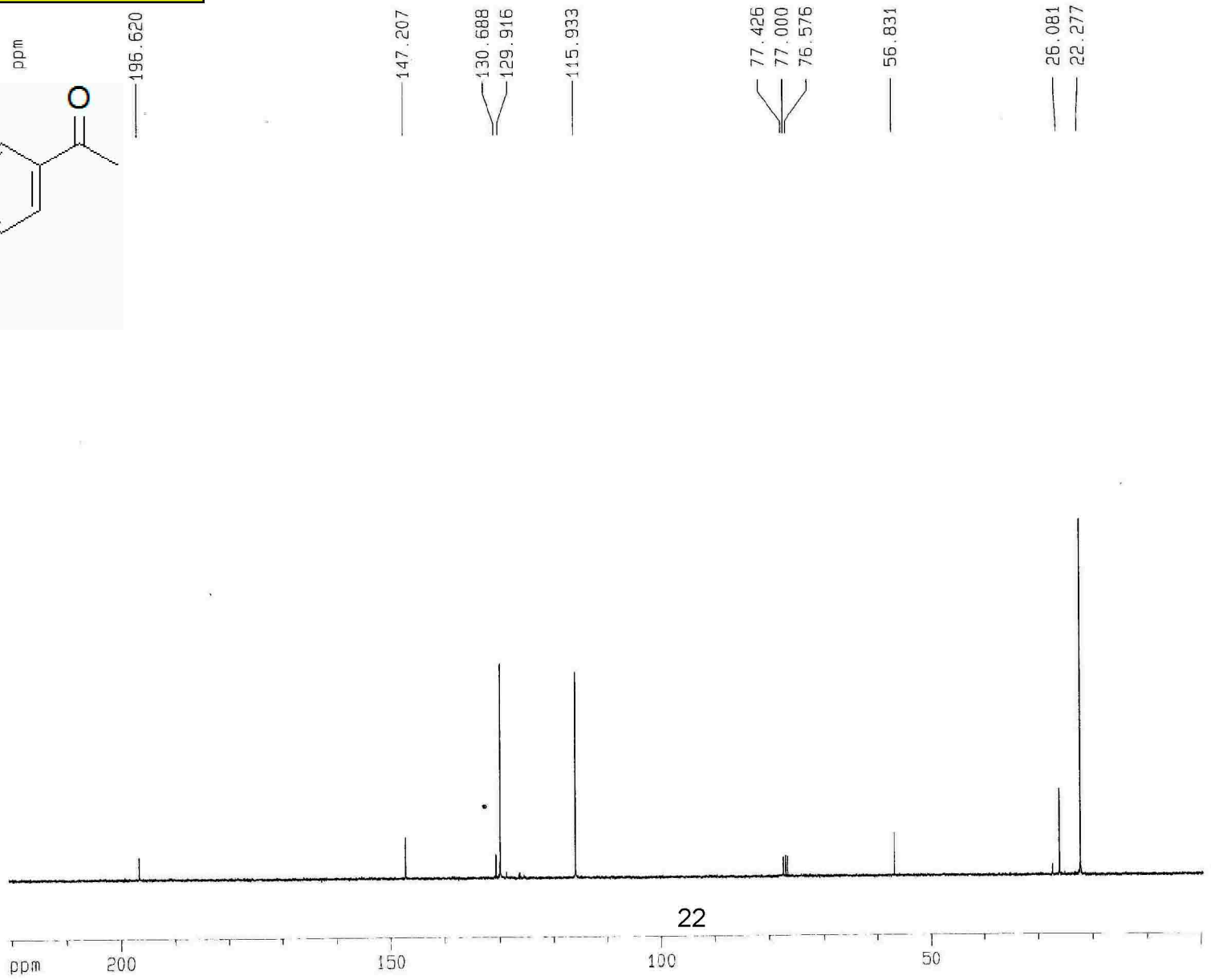
CM12130


```
Current Data Parameters
NAME                  zql-2011-3
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20110110
Time                       16.00
INSTRUM                    av300
PROBHD     5 mm QNP 1H/13
PULPROG                     zg30
TD                         32768
SOLVENT                    CDCl3
NS                             8
DS                             0
SWH                     5995.204 Hz
FIDRES                  0.182959 Hz
AQ                      2.7329011 sec
RG                           128
DW                         83.400 usec
DE                          6.00 usec
TE                         290.9 K
D1                      1.00000000 sec
MCREST                  0.00000000 sec
MCWRK                   0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                         10.50 usec
PL1                         0.10 dB
SFO1                300.1321009 MHz

F2 - Processing parameters
SI                         32768
SF                  300.1300120 MHz
WDW                           EM
SSB                            0
LB                          0.10 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
CY                         10.00 cm
F1P                        10.000 ppm
F1                       3001.30 Hz
F2P                        -0.500 ppm
F2                      -150.06 Hz
PPMCM                   0.52500 ppm/cm
HZCM                  157.56825 Hz/cm
```

Table 2, entry 9

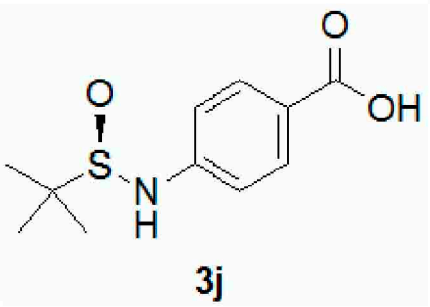


3i




```
Current Data Parameters
NAME                  zql-2011-3
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20110110
Time                       16 02
INSTRUM                    av300
PROBHD   5 mm QNP 1H/13
PULPROG                     zgpg
TD                         65536
SOLVENT                    CDCl3
NS                            64
DS                             4
SWH                  22675.736 Hz
FIDRES                0.346004 Hz
AQ                   1.4451188 sec
RG                          8192
DW                     22.050 usec
DE                      6.00 usec
TE                      290.9 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                         13C
P1                      10.50 usec
PL1                      -0.81 dB
SFO1                75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                   80.00 usec
PL2                      0.10 dB
PL12                    17.74 dB
PL13                    17.74 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                         65536
SF                  75.4677621 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                      20.00 cm
CY                       6.00 cm
F1P                   220.500 ppm
F1                  16640.64 Hz
F2P                    -0.500 ppm
F2                    -37.73 Hz
PPMCM                11.05000 ppm/cm
HZCM                833.91882 Hz/cm
```

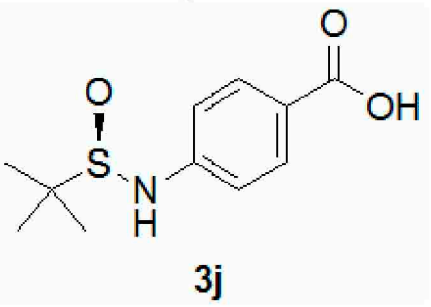
Table 2, entry 10



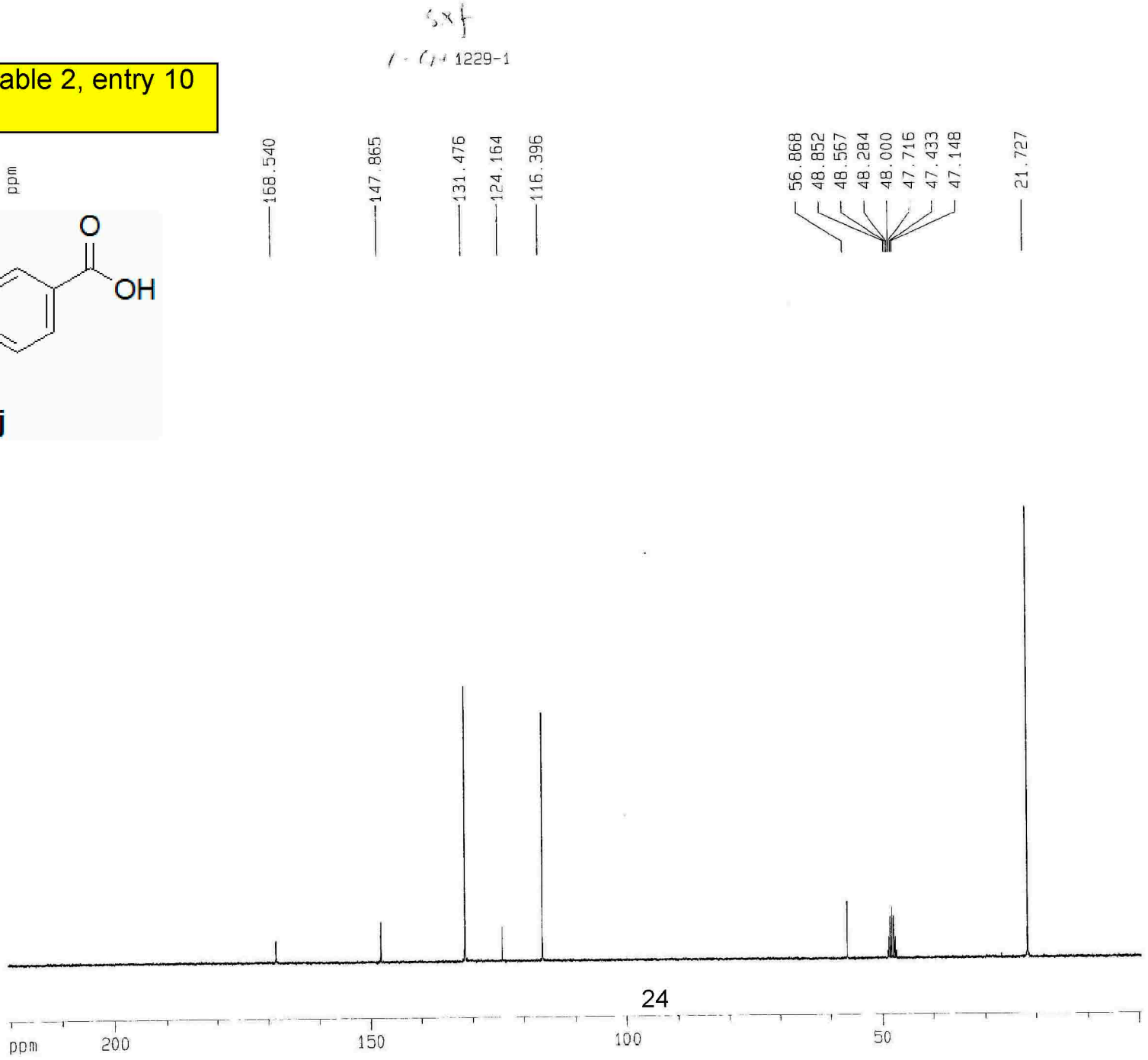
3j


```
Current Data Parameters
NAME                zql-2011-5
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20110110
Time                   16.11
INSTRUM                av300
PROBHD   5 mm QNP 1H/13
PULPROG                 zg30
TD                     32768
SOLVENT                MeOD
NS                         8
DS                         0
SWH                5995.204 Hz
FIDRES            0.182959 Hz
AQ                2.7329011 sec
RG                       128
DW                  83.400 usec
DE                   6.00 usec
TE                   290.9 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                   10.50 usec
PL1                   0.10 dB
SFO1          300.1321009 MHz

F2 - Processing parameters
SI                       32768
SF            300.1300077 MHz
WDW                       EM
SSB                        0
LB                    0.10 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    30.00 cm
F1P                  10.000 ppm
F1                 3001.30 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM              0.52500 ppm/cm
HZCM             157.56825 Hz/cm
```

Table 2, entry 10

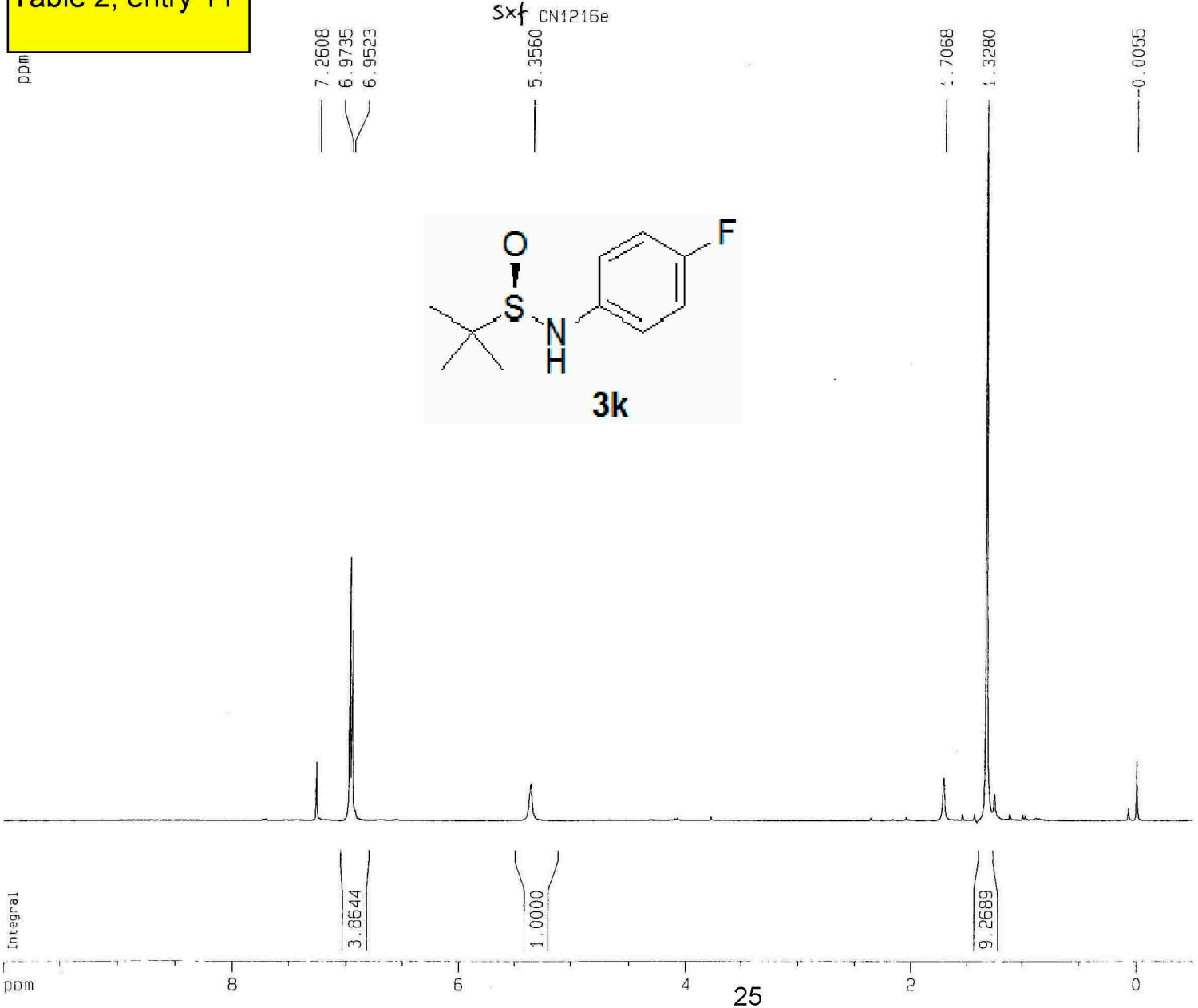


3j




```
Current Data Parameters
NAME                  zql-2011-5
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20110110
Time                      16.15
INSTRUM                   av300
PROBHD   5 mm QNP 1H/13
PULPROG                    zgpg
TD                        65536
SOLVENT                    MeOD
NS                           64
DS                            4
SWH                22675.736 Hz
FIDRES             0.346004 Hz
AQ                 1.4451188 sec
RG                       8192
DW                   22.050 usec
DE                    6.00 usec
TE                    291.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.50 usec
PL1                   -0.81 dB
SFO1              75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    0.10 dB
PL12                  17.74 dB
PL13                  17.74 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                        65536
SF                75.4677275 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     8.00 cm
F1P                  220.500 ppm
F1                 16640.63 Hz
F2P                   -0.500 ppm
F2                   -37.73 Hz
PPMCM                11.05000 ppm/cm
HZCM               833.91840 Hz/cm
```

Table 2, entry 11



Current Data Parameters
NAME zql-2010-154
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101222
Time 16.19
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

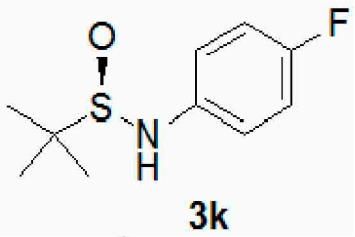
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300120 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

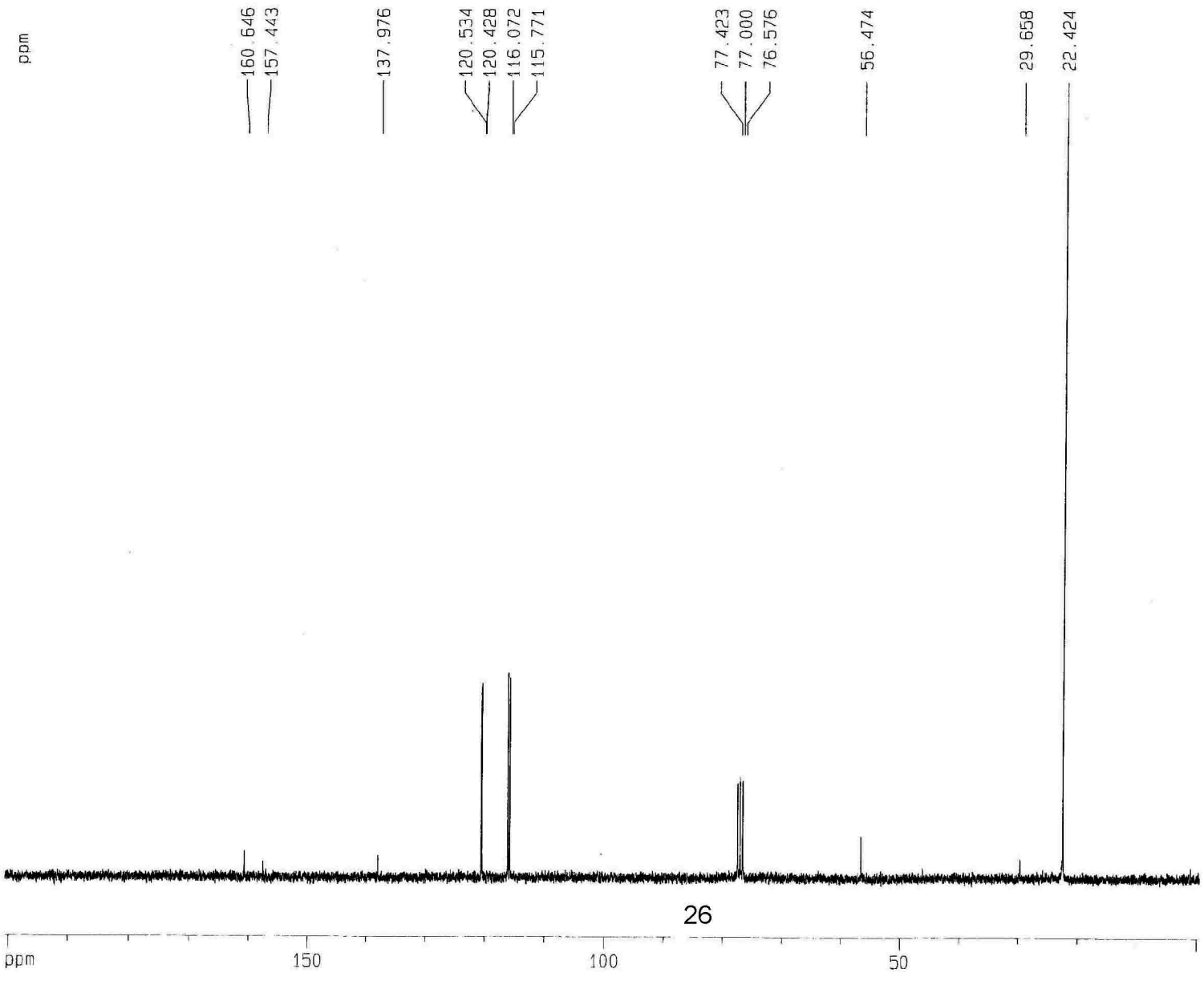
1D NMR plot parameters
CX 20.00 cm
CY 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.55825 Hz/cm

Table 2, entry 11

C-CN sxf 1216e

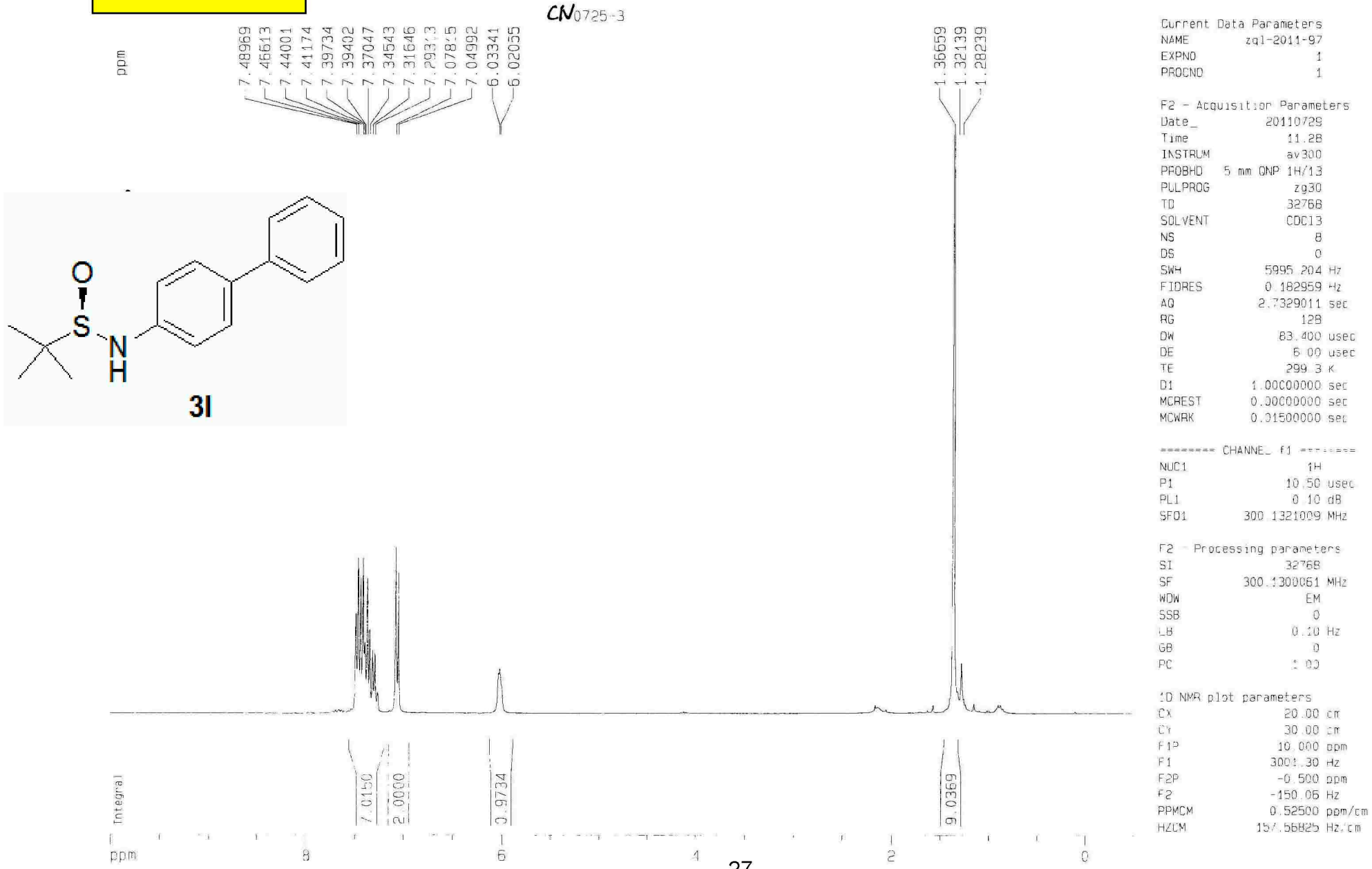


3k




```
Current Data Parameters
NAME                  zq1-2010-161
EXPNO                            2
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20101228
Time                         11.47
INSTRUM                      av300
PROBHD     5 mm QNP 1H/13
PULPROG                       zgpg
TD                           65536
SOLVENT                       MeOD
NS                             256
DS                               4
SWH                    22675.736 Hz
FIDRES                  0.346004 Hz
AQ                     1.4451188 sec
RG                            8192
DW                       22.050 usec
DE                        6.00 usec
TE                         293.1 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                          13C
P1                       10.50 usec
PL1                       -0.81 dB
SFO1                 75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                    80.00 usec
PL2                       0.10 dB
PL12                     17.74 dB
PL13                     17.74 dB
SFO2                300.1312005 MHz

F2 - Processing parameters
SI                           65536
SF                    75.4677544 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40

1D NMR plot parameters
CX                           20.00 cm
CY                           14.00 cm
F1P                         200.500 ppm
F1                        15131.29 Hz
F2P                          -0.500 ppm
F2                          -37.73 Hz
PPMCM                     10.05000 ppm/cm
HZCM                     758.45099 Hz/cm
```

Table 2, entry 12



Current Data Parameters
NAME zql-2011-97
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110729
Time 11.28
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 299.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

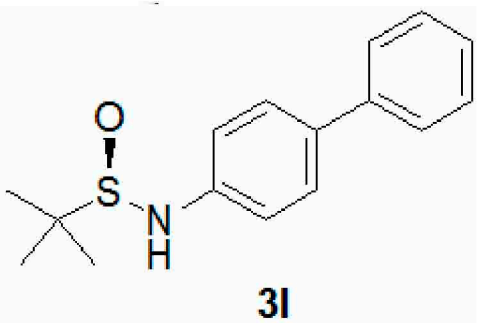
1D NMR plot parameters
CX 20.00 cm
CY 30.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

Table 2, entry 12

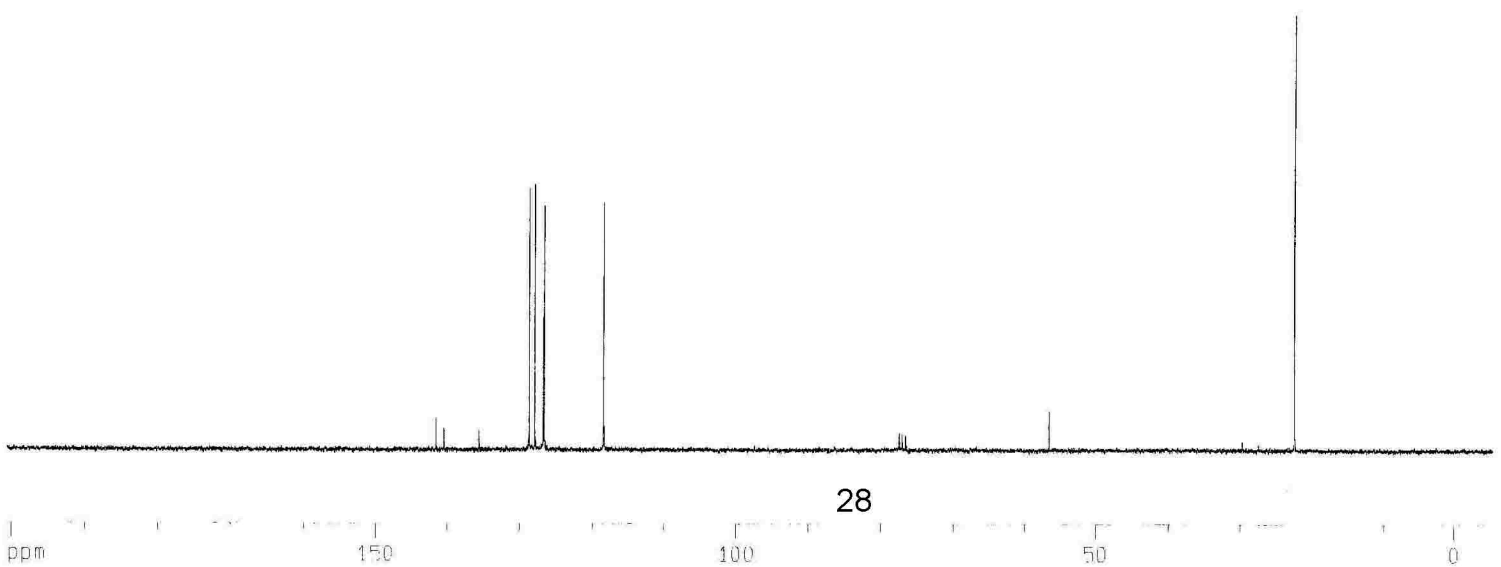
C-CN0725-3

ppm

141.553
140.426
135.594
128.625
127.851
126.752
126.609
118.370
77.423
77.000
76.577
36.492
22.448



3l



Current Data Parameters
NAME zql-2011-97
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110729
Time 11.30
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 299.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

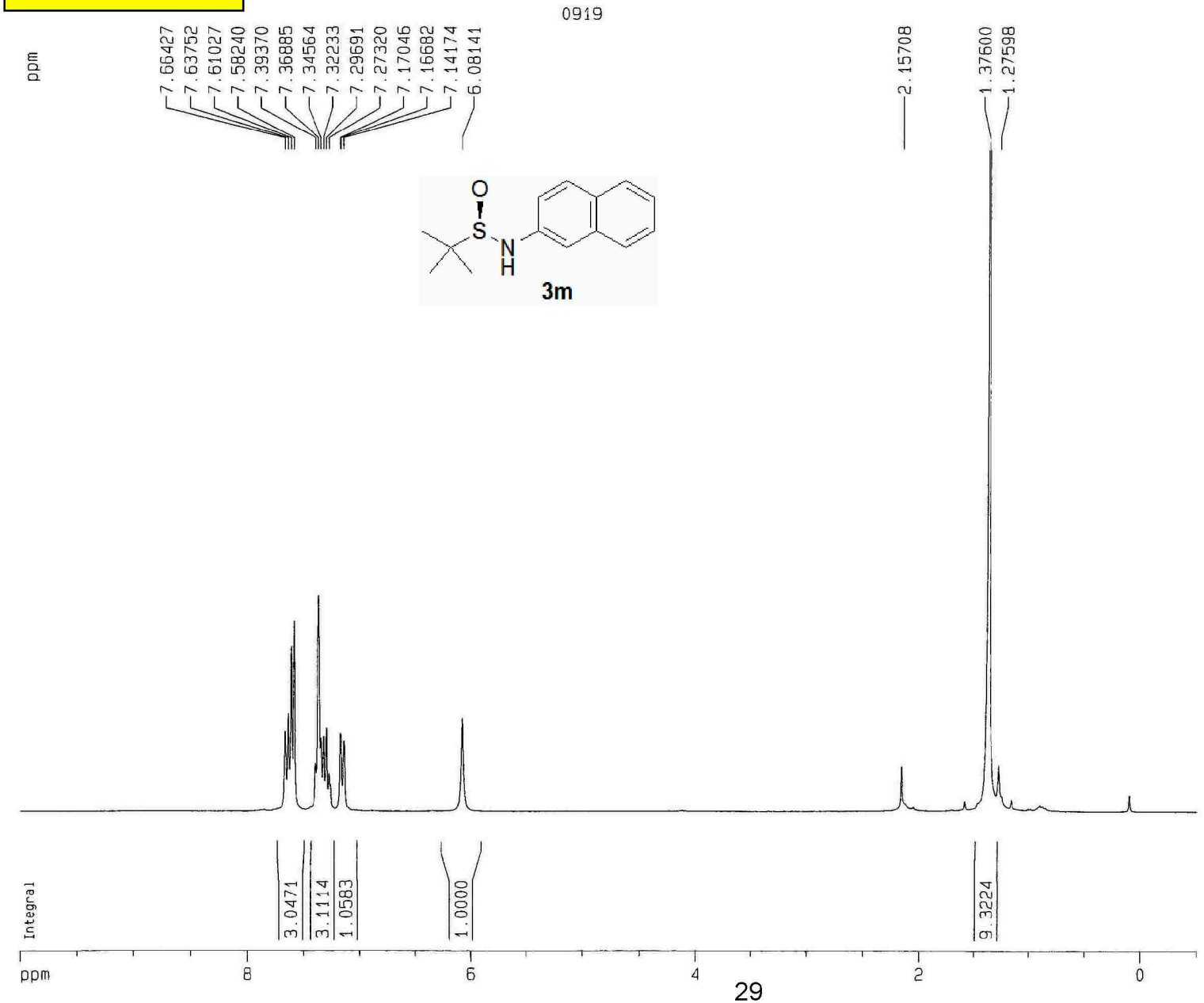
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677569 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.500 ppm
F1 15137.29 Hz
F2P -5.503 ppm
F2 -415.07 Hz
PPMCM 18.30003 ppm/cm
HZCM 777.31793 Hz/cm

Table 2, entry 13



Current Data Parameters

NAME	zql-2011-145
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20111012
Time	15.25
INSTRUM	av300
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.182959 Hz
AQ	2.7329011 sec
RG	128
DW	83.400 usec
DE	6.00 usec
TE	297.3 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	10.50 usec
PL1	0.10 dB
SF01	300.1321009 MHz

F2 - Processing parameters

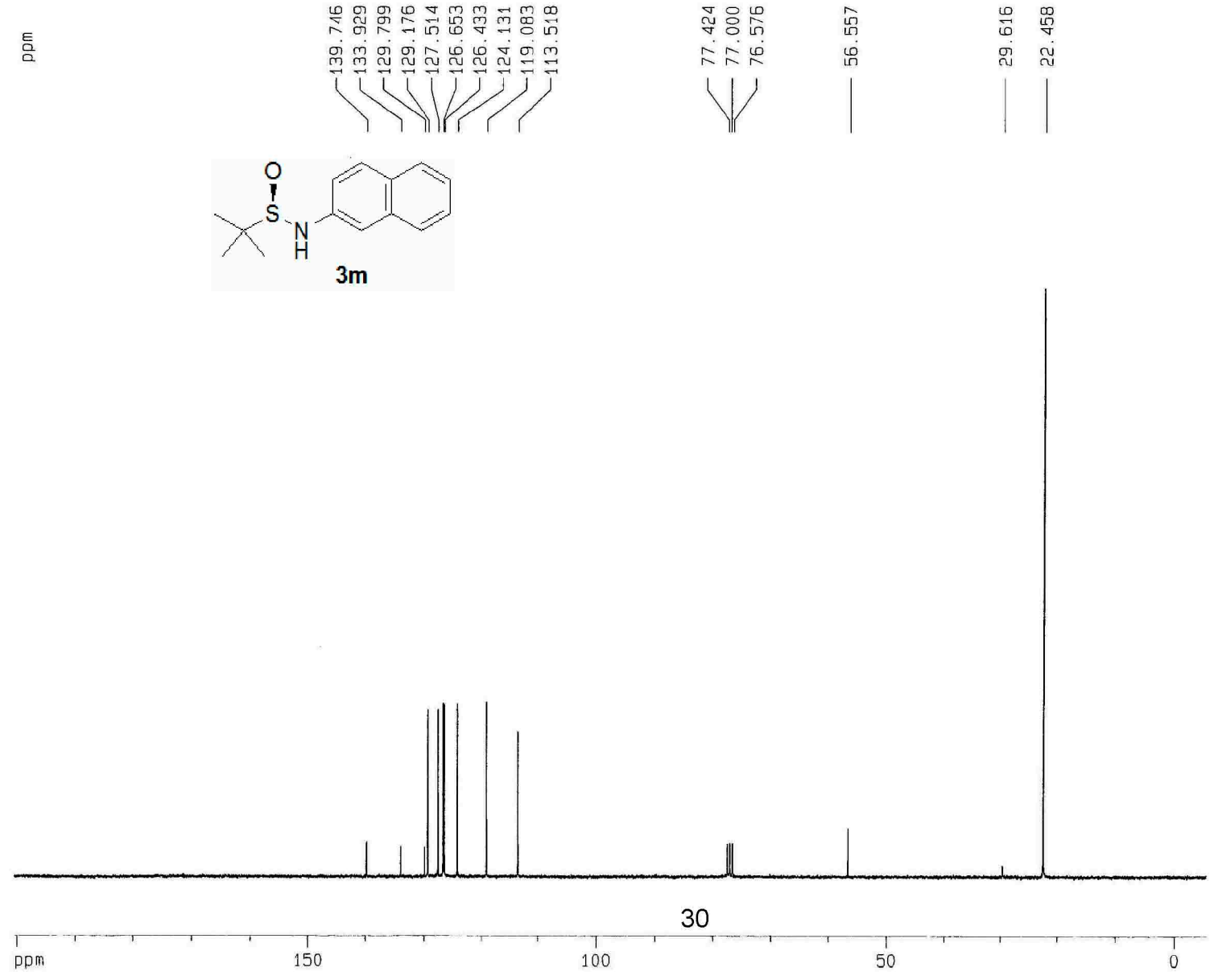
SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.52500 ppm/cm
HZCM	157.56825 Hz/cm

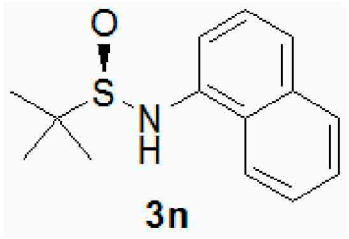
Table 2, entry 13

0919




```
Current Data Parameters
NAME        zq1-2011-145
EXPNO               2
PROCNO              1

F2 - Acquisition Parameters
Date_          20111012
Time              15.45
INSTRUM           av300
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg
TD               65536
SOLVENT           CDCl3
NS                 256
DS                   4
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG                8192
DW              22.050 usec
DE               6.00 usec
TE              298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               10.50 usec
PL1              -0.81 dB
SFO1         75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PL2               0.10 dB
PL12             17.74 dB
PL13             17.74 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                65536
SF           75.4677575 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40

1D NMR plot parameters
CX               20.00 cm
CY               10.00 cm
F1P             200.500 ppm
F1            15131.29 Hz
F2P              -5.500 ppm
F2             -415.07 Hz
PPMCM          10.30000 ppm/cm
HZCM          777.31793 Hz/cm
```

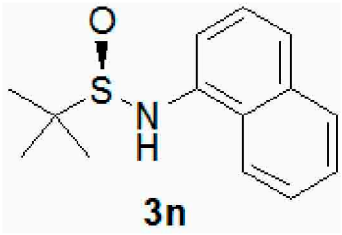
Table 2, entry 14



3n


```
Current Data Parameters
NAME                  zql-2011-15
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20110328
Time                        18.12
INSTRUM                     av300
PROBHD   5 mm QNP 1H/13
PULPROG                      zg30
TD                          32768
SOLVENT                     CDCl3
NS                             16
DS                              0
SWH                   5995.204 Hz
FIDRES                0.182959 Hz
AQ                    2.7329011 sec
RG                            128
DW                     83.400 usec
DE                      6.00 usec
TE                      292.8 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                     10.50 usec
PL1                     0.10 dB
SFO1                300.1321009 MHz

F2 - Processing parameters
SI                          32768
SF                 300.1300051 MHz
WDW                            EM
SSB                             0
LB                      0.10 Hz
GB                              0
PC                           1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     35.00 cm
F1P                   10.000 ppm
F1                   3001.30 Hz
F2P                    0.500 ppm
F2                  -150.06 Hz
PPMCM                0.52500 ppm/cm
HZCM                157.56825 Hz/cm
```

Table 2, entry 14

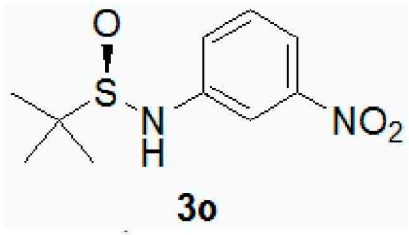


3n

CGCN-0308-5


```
Current Data Parameters
NAME          zgl-2011-15
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              20110328
Time                 18.15
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg
TD                  65536
SOLVENT              CDCl3
NS                      64
DS                       4
SWH            22675.736 Hz
FIDRES         0.346004 Hz
AQ             1.4451188 sec
RG                    8192
DW                22.050 usec
DE                 6.00 usec
TE                293.1 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                 10.50 usec
PL1                -0.81 dB
SFO1          75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 0.10 dB
PL12               17.74 dB
PL13               17.74 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                    65536
SF            75.4677585 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.00 cm
F1P                200.500 ppm
F1               15131.29 Hz
F2P                 -5.500 ppm
F2                -415.07 Hz
PPMCM             10.30000 ppm/cm
HZCM             777.31793 Hz/cm
```

Table 2, entry 18

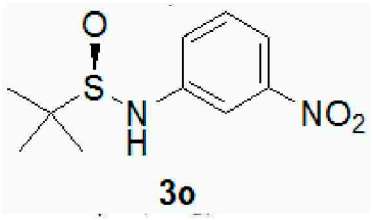


3o


```
Current Data Parameters
NAME          zq1-2011-135
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110922
Time                 16.36
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              5995.204 Hz
FIDRES           0.182959 Hz
AQ               2.7329011 sec
RG                    128
DW                 83.400 usec
DE                  6.00 usec
TE                 296.7 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.50 usec
PL1                 0.10 dB
SFO1         300.1321009 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300061 MHz
WDW                    EM
SSB                     0
LB                  0.10 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               10.000 ppm
F1               3001.30 Hz
F2P               -1.000 ppm
F2               -300.13 Hz
PPMCM             0.55000 ppm/cm
HZCM            165.07150 Hz/cm
```

Table 2, entry 18

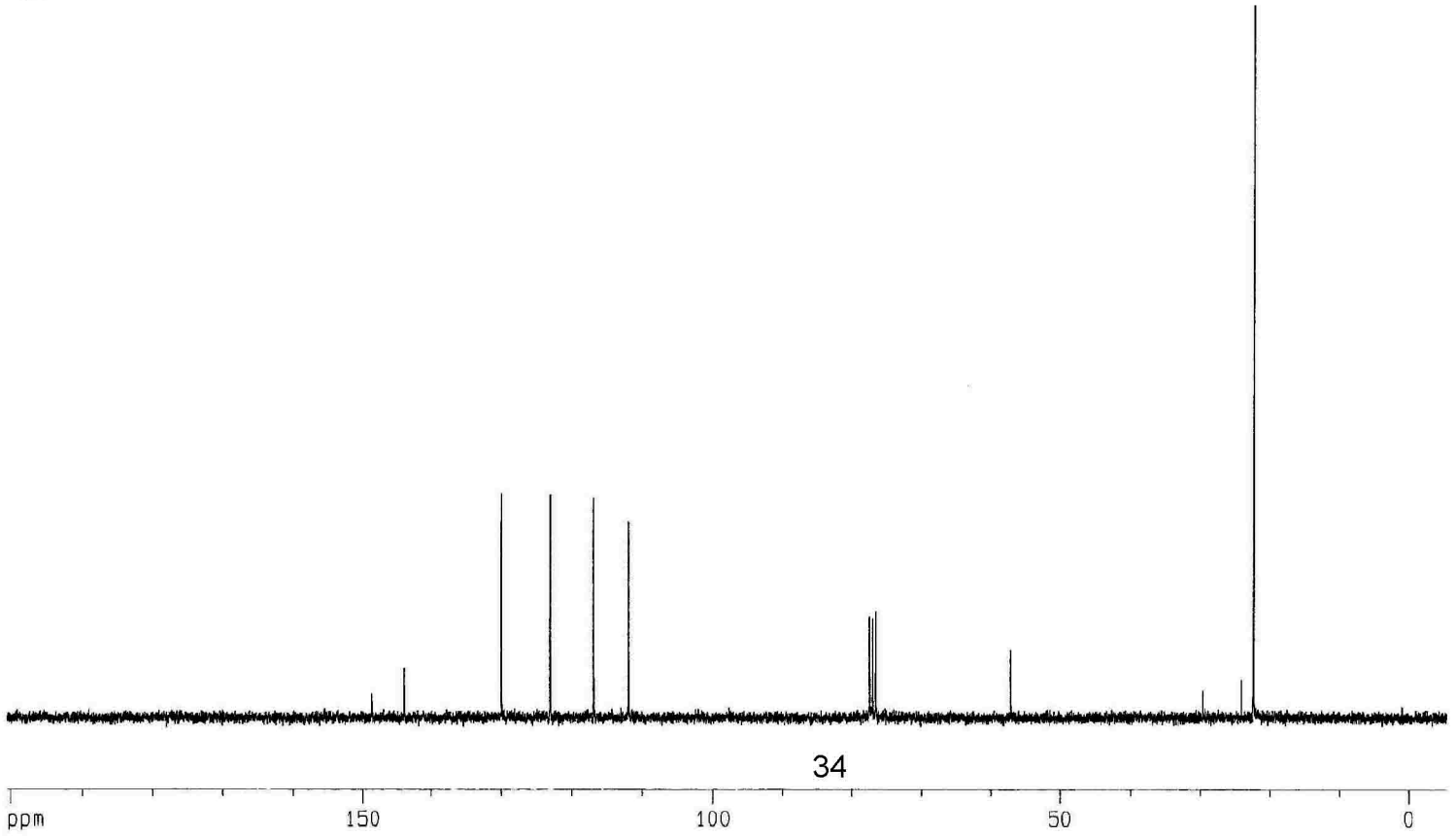
C-CN0811-2



3o

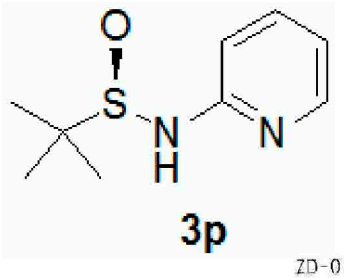
ppm

148.717
143.874
129.923
123.019
116.890
111.974
77.424
77.000
76.576
57.103
29.640
24.119
22.400

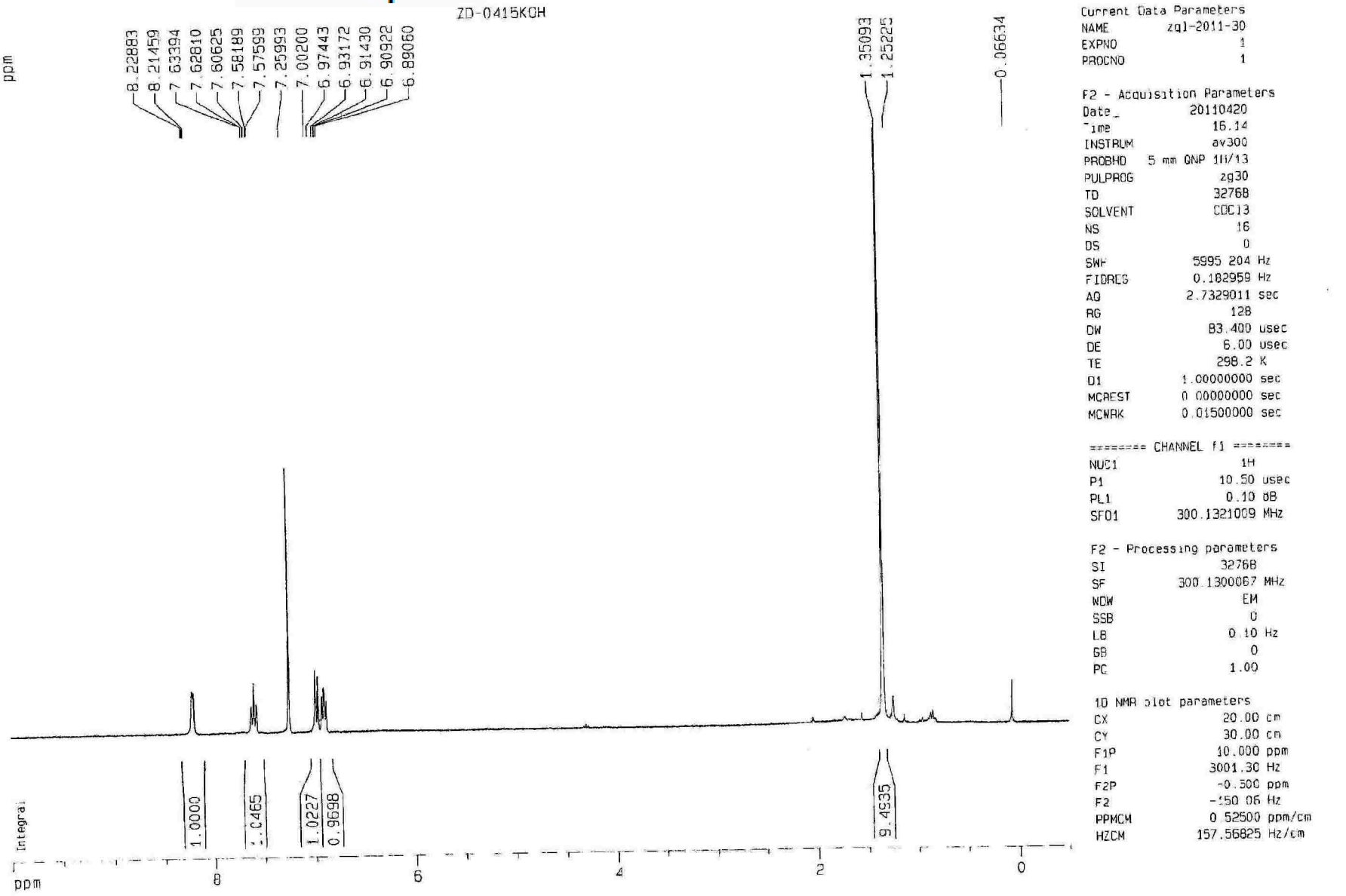



```
Current Data Parameters
NAME        zgl-2011-135
EXPNO               2
PROCNO              1

F2 - Acquisition Parameters
Date_          20110922
Time             17.15
INSTRUM          av300
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg
TD              65536
SOLVENT          CDCl3
NS                130
DS                  4
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG               8192
DW             22.050 usec
DE              6.00 usec
TE             297.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1              10.50 usec
PL1             -0.81 dB
SFO1        75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           80.00 usec
PL2              0.10 dB
PL12            17.74 dB
PL13            17.74 dB
SFO2       300.1312005 MHz

F2 - Processing parameters
SI              65536
SF          75.4677526 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40

1D NMR plot parameters
CX             20.00 cm
CY             10.00 cm
F1P           200.500 ppm
F1          15131.28 Hz
F2P            -5.500 ppm
F2           -415.07 Hz
PPMCM        10.30000 ppm/cm
HZCM        777.31787 Hz/cm
```

Table 2, entry 19



3p



Current Data Parameters

NAME	zql-2011-30
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20110420
Time	16.14
INSTRUM	av300
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.182959 Hz
AQ	2.7329011 sec
RG	128
DW	83.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	10.50 usec
PL1	0.10 dB
SFO1	300.1321009 MHz

F2 - Processing parameters

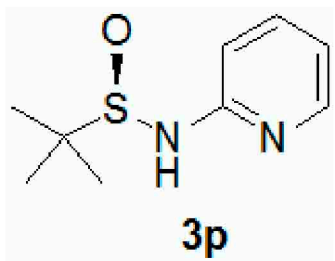
SI	32768
SF	300.1300067 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.52500 ppm/cm
HZCM	157.56825 Hz/cm

Table 2, entry 19

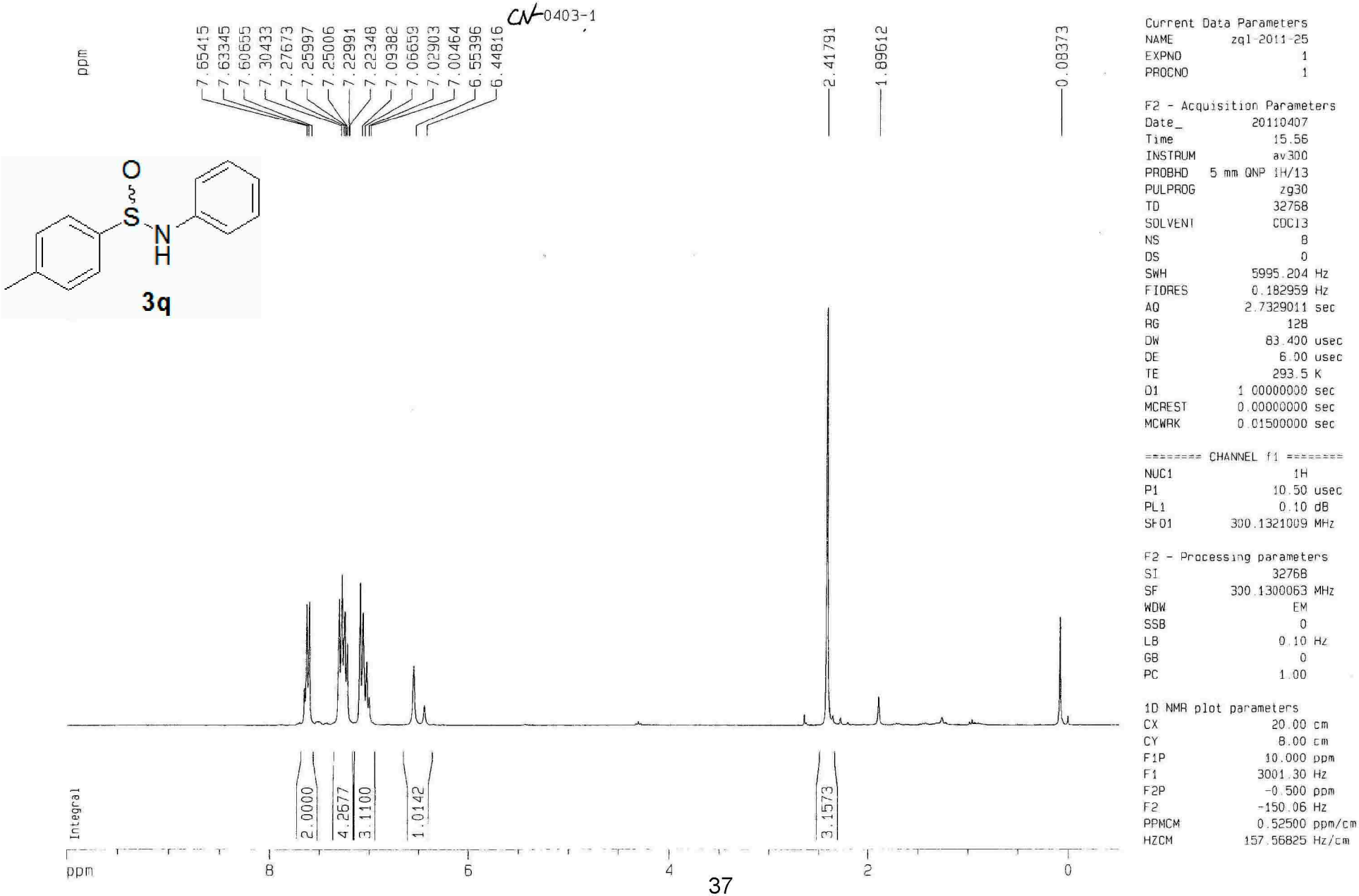
C-CN0608-4



3p


```
Current Data Parameters
NAME          zgl-2011-105
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110803
Time                 16.15
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg
TD                  65536
SOLVENT              CDCl3
NS                      64
DS                       4
SWH              22675.736 Hz
FIDRES           0.346004 Hz
AQ               1.4451188 sec
RG                    8192
DW                22.050 usec
DE                 6.00 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                  10.50 usec
PL1                 -0.81 dB
SFO1           75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               60.00 usec
PL2                  0.10 dB
PL12                17.74 dB
PL13                17.74 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                      65536
SF             75.4677545 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  12.00 cm
F1P               200.500 ppm
F1              15131.29 Hz
F2P                -5.500 ppm
F2               -415.07 Hz
PPMCM             10.30000 ppm/cm
HZCM             777.31793 Hz/cm
```

Table 2, entry 20



Current Data Parameters
NAME zql-2011-25
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110407
Time 15.56
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

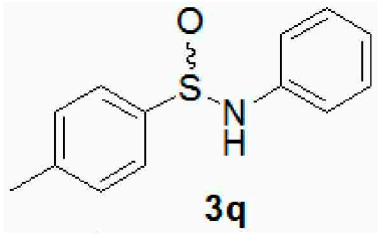
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.10 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

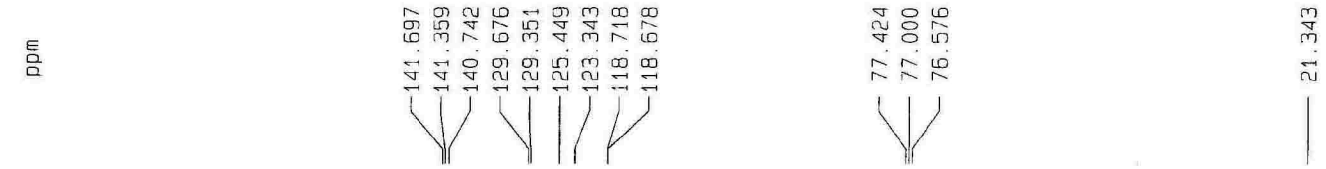
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

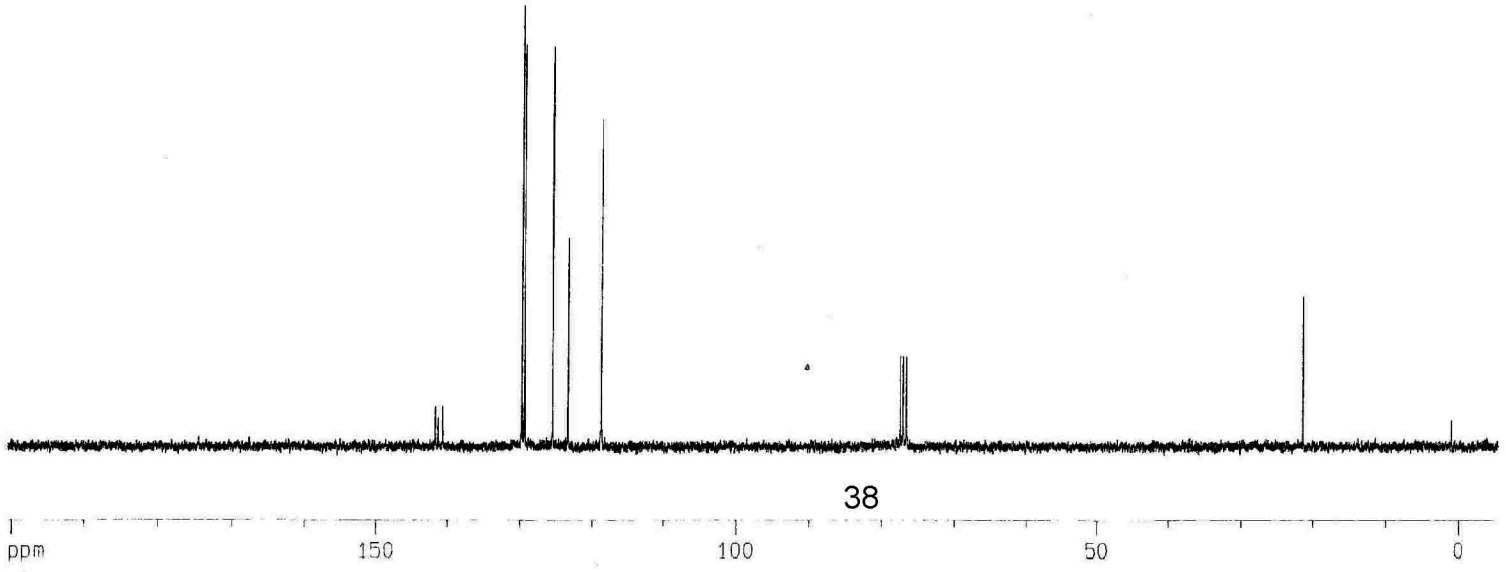
Table 2, entry 20

C-CN0403-1



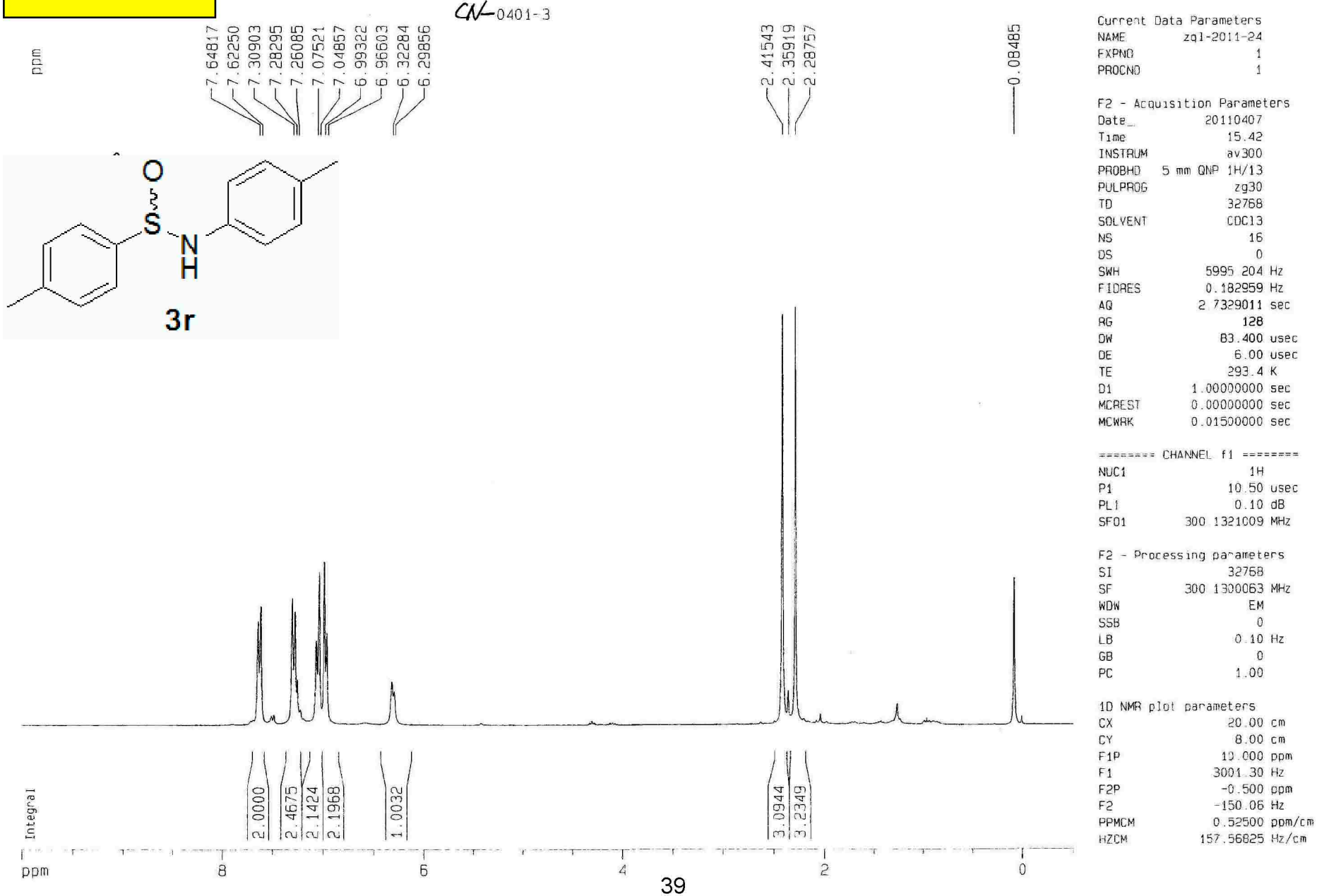
3q






```
Current Data Parameters
NAME                  zq1-2011-25
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20110407
Time                        16.01
INSTRUM                     av300
PROBHD   5 mm QNP 1H/13
PULPROG                     zgpg
TD                         65536
SOLVENT                    CDCl3
NS                           128
DS                             4
SWH                    22675.736 Hz
FIDRES                  0.346004 Hz
AQ                     1.4451188 sec
RG                          8192
DW                        22.050 usec
DE                         6.00 usec
TE                        293.7 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                         13C
P1                         10.50 usec
PL1                        -0.81 dB
SFO1                75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         0.10 dB
PL12                       17.74 dB
PL13                       17.74 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                         65536
SF                  75.4677562 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
CY                          6.00 cm
F1P                       200.500 ppm
F1                      15131.29 Hz
F2P                        -5.500 ppm
F2                       -415.07 Hz
PPMCM                   10.30000 ppm/cm
HZCM                   777.31793 Hz/cm
```

Table 2, entry 21



Current Data Parameters

NAME	zq1-2011-24
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20110407
Time	15.42
INSTRUM	av300
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.182959 Hz
AQ	2.7329011 sec
RG	128
DW	83.400 usec
DE	6.00 usec
TE	293.4 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	10.50 usec
PL1	0.10 dB
SFO1	300.1321009 MHz

F2 - Processing parameters

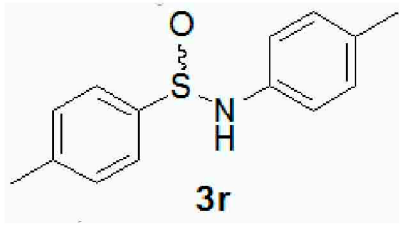
SI	32768
SF	300.1300063 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	8.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.52500 ppm/cm
HZCM	157.56825 Hz/cm

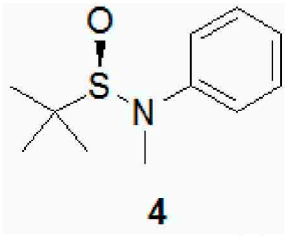
Table 2, entry 21

C-CN0401-3



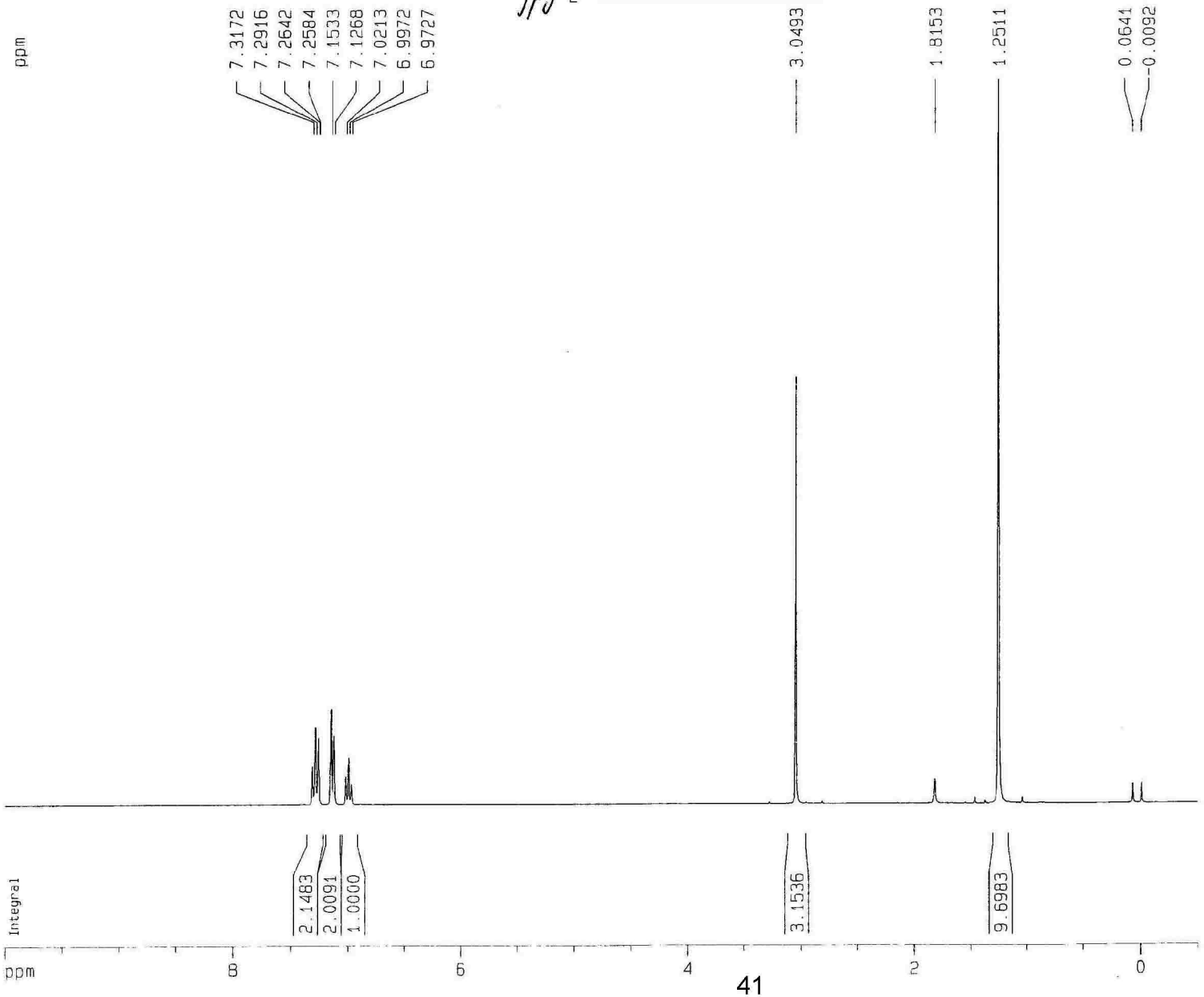
3r


```
Current Data Parameters
NAME          zql-2011-24
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              20110407
Time                 15.46
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      4
SWH           22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG                   8192
DW               22.050 usec
DE                6.00 usec
TE                293.7 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                10.50 usec
PL1               -0.81 dB
SFO1         75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                0.10 dB
PL12              17.74 dB
PL13              17.74 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                  65536
SF           75.4677569 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  6.00 cm
F1P              200.500 ppm
F1             15131.29 Hz
F2P               -5.500 ppm
F2              -415.07 Hz
PPMCM           10.30000 ppm/cm
HZCM           777.31793 Hz/cm
```



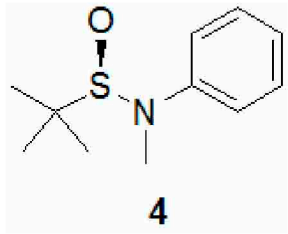
4

gpj 2

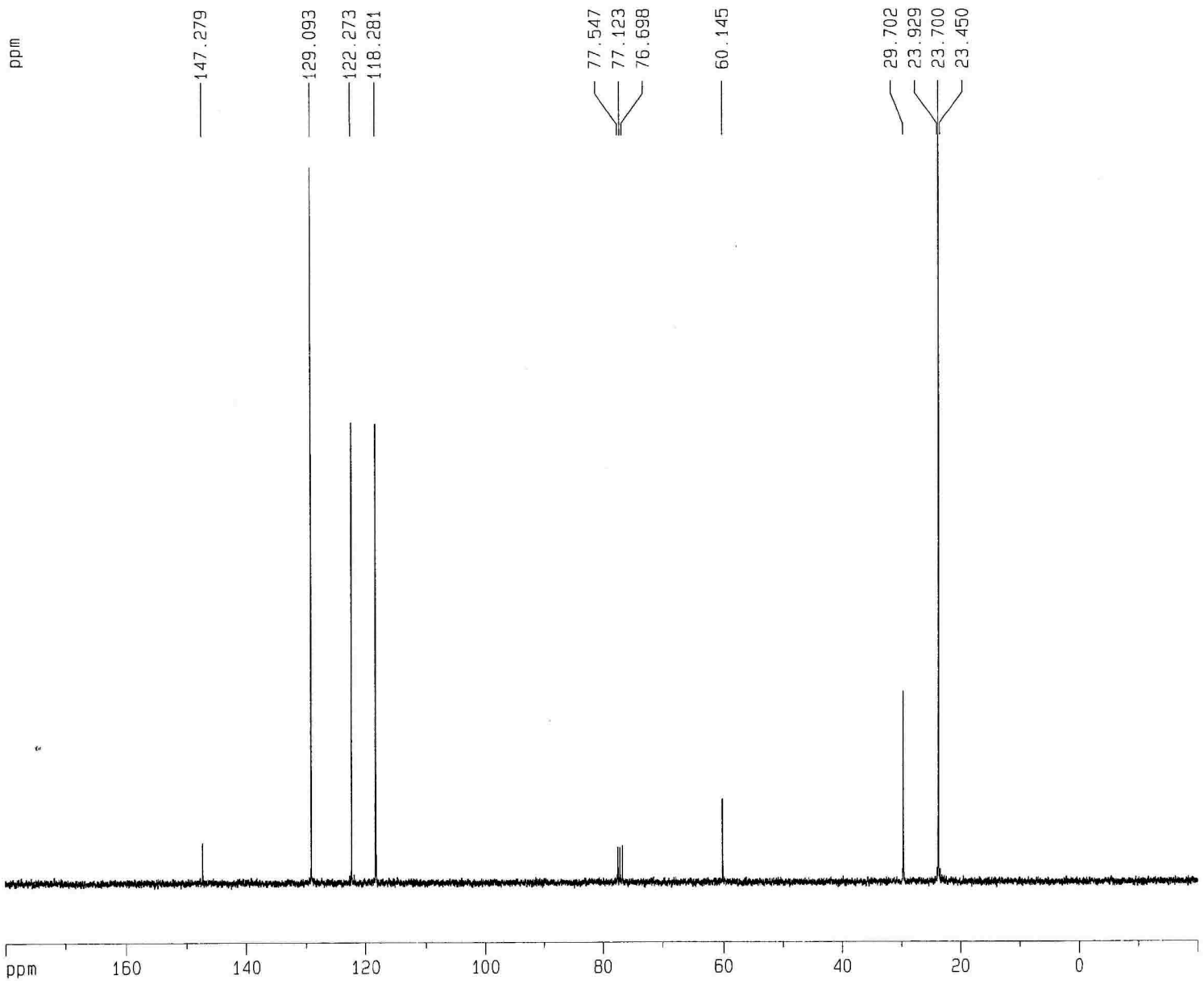



```
Current Data Parameters
NAME          zql-2010-152
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20101222
Time                 16.14
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH               5995.204 Hz
FIDRES            0.182959 Hz
AQ               2.7329011 sec
RG                    128
DW                 83.400 usec
DE                  6.00 usec
TE                 293.1 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.50 usec
PL1                 0.10 dB
SFO1         300.1321009 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300127 MHz
WDW                    EM
SSB                     0
LB                  0.10 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  20.00 cm
F1P                10.000 ppm
F1                3001.30 Hz
F2P                -0.500 ppm
F2                -150.06 Hz
PPMCM             0.52500 ppm/cm
HZCM            157.56825 Hz/cm
```

QL-002



4



Current Data Parameters

NAME	zql-2011-123
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20110905
Time	16.15
INSTRUM	av300
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg
TD	65536
SOLVENT	CDCl3
NS	128
DS	4
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	8192
DW	22.050 usec
DE	6.00 usec
TE	300.3 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	10.50 usec
PL1	-0.81 dB
SFO1	75.4760505 MHz

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

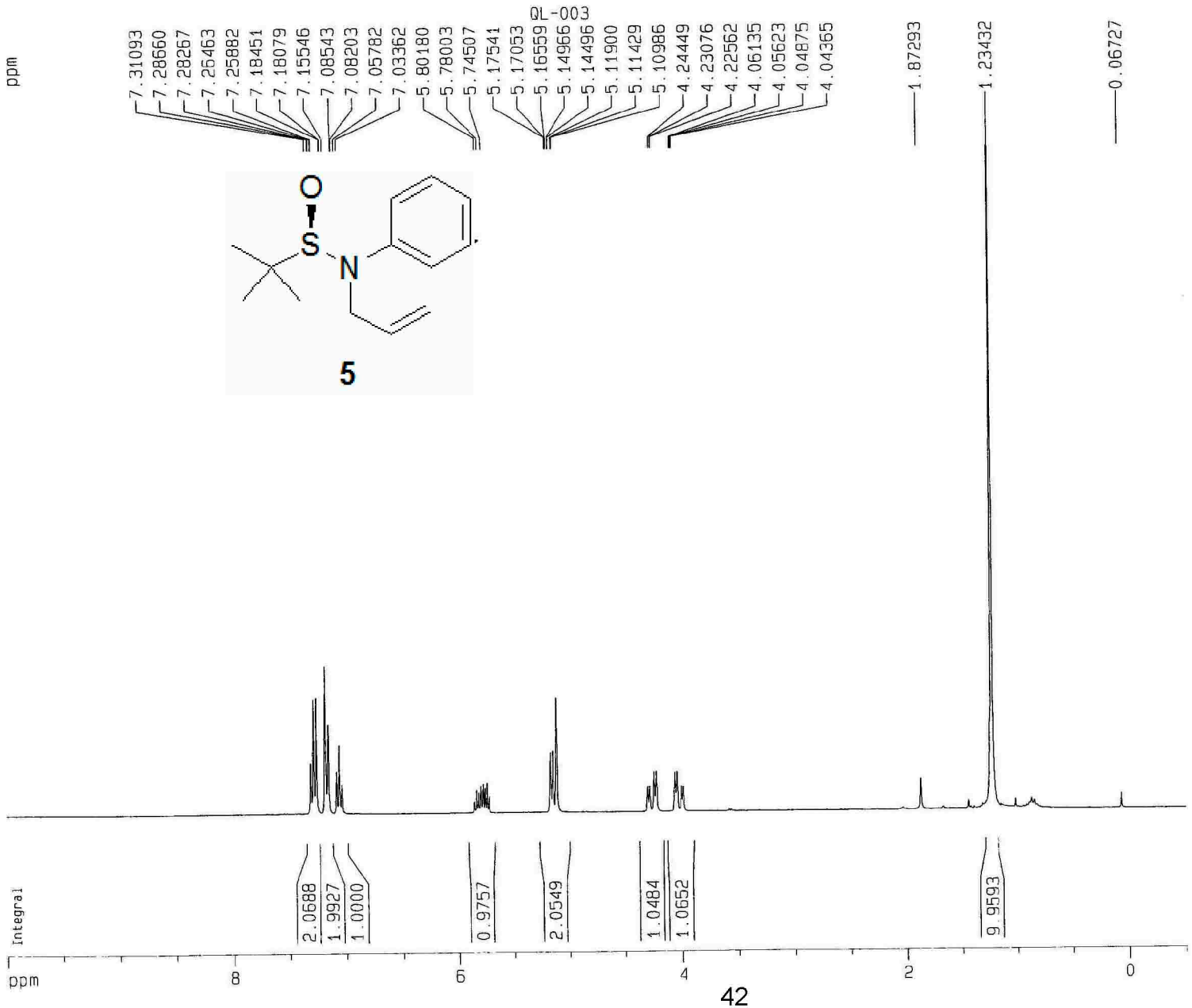
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.10 dB
PL12	17.74 dB
PL13	17.74 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	65536
SF	75.4677512 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	180.000 ppm
F1	13584.20 Hz
F2P	-20.000 ppm
F2	-1509.35 Hz
PPMCM	10.00000 ppm/cm
HZCM	754.67749 Hz/cm



Current Data Parameters

NAME	zql-2011-127
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20110920
Time	15.00
INSTRUM	av300
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	5995.204 Hz
FIDRES	0.182959 Hz
AQ	2.7329011 sec
RG	32
DW	83.400 usec
DE	6.00 usec
TE	296.7 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	10.50 usec
PL1	0.10 dB
SF01	300.1321009 MHz

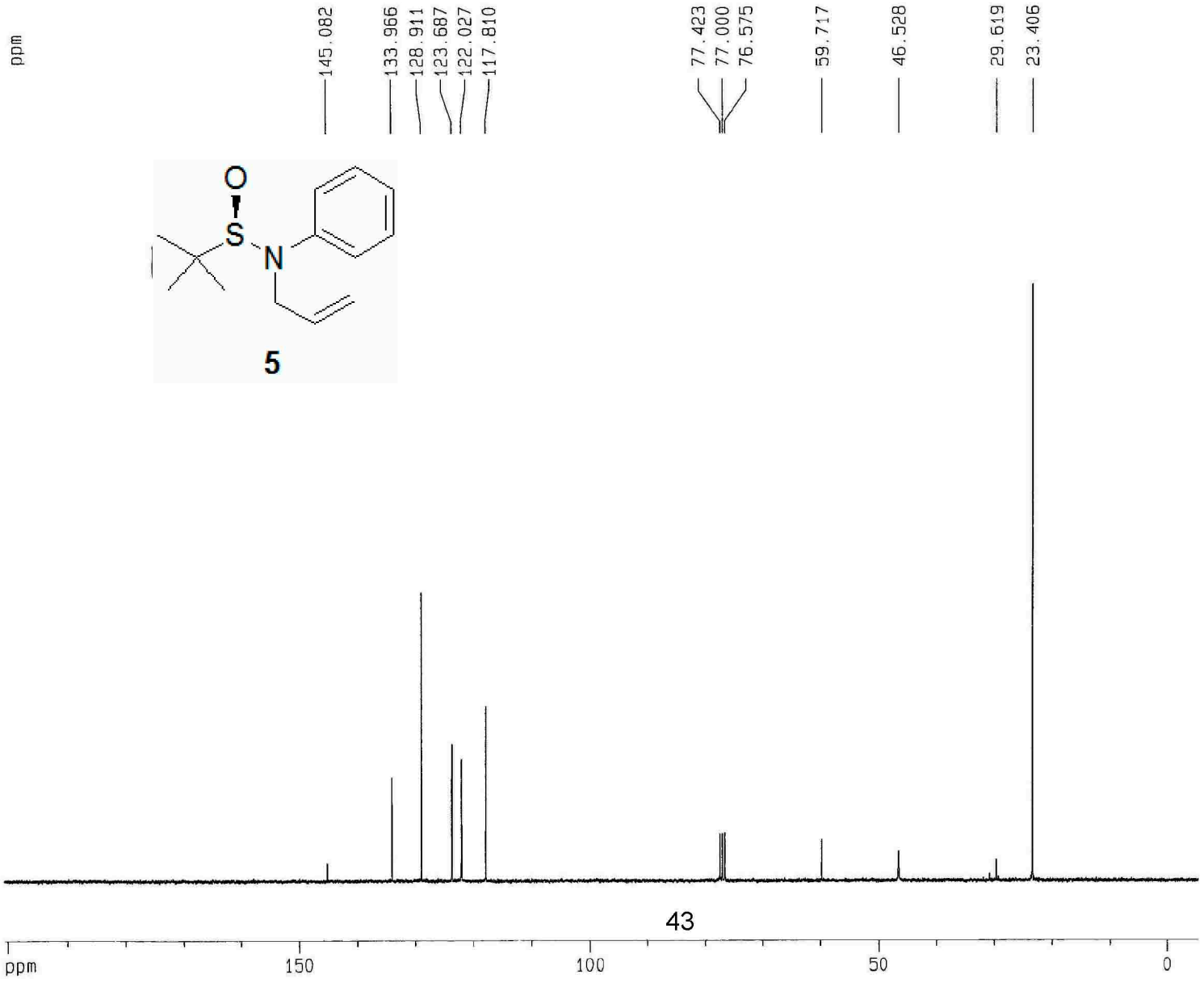
F2 - Processing parameters

SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.52500 ppm/cm
HZCM	157.56825 Hz/cm

QL-003



Current Data Parameters
NAME zql-2011-137
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110922
Time 17.08
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -0.81 dB
SFO1 75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.10 dB
PL12 17.74 dB
PL13 17.74 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31793 Hz/cm