# Rhodium-Catalyzed Cyclization of Acceptor-Substituted Biphenyl α-Diazoketones: A Study of Substitution Effect on Chemoselectivity

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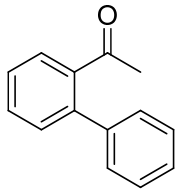
## *Supporting Information*

## 1) Preparation and NMR spectra of 1a-g

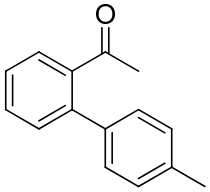
**1-([1,1'-Biphenyl]-2-yl)ethanone (1a)**[20]



**1a**

To a $N_2$–protected flask containing $MnCl_2$ (5.04 g, 97%, 38.8 mmol) and LiCl (3.32 g, 77.5 mmol), 78 mL of THF was added. The suspension was stirred at -10 °C for 30 min, followed by adding with PhMgCl (19.4 mL, 2 M in THF, 38.8 mmol) via a syringe over 20 min. The resulting mixture was then allowed to stir at rt for 1 min and added with a solution of 2'-chloroacetophenone (2.6 mL, 97%, 19.4 mmol) in THF (16 mL). After stirring for an additional 5 min, the reaction mixture was quenched by 50 mL of aqueous HCl solution (1 N) and extracted with ether (500 mL). The organic layer was separated and washed with saturated aqueous $NaHCO_3$ (100 mL x 1), water (100 mL x 1) and brine (100 mL x 1). After concentration, the crude residue was purified by flash chromatography (hexane-ethyl acetate 30:1, 15:1, 5:1) to give 1.6 g of **1a** (42%). $^1$H NMR (300 MHz, $CDCl_3$): δ 7.57-7.49 (m, 2 H), 7.47-7.34 (m, 7 H), 2.01 (s, 3 H) ppm; $^{13}$C NMR (75 MHz, $CDCl_3$): δ 204.9, 140.9, 140.8, 140.6, 130.7, 130.3, 128.9, 128.7, 127.9, 127.9, 127.5, 30.5 ppm.

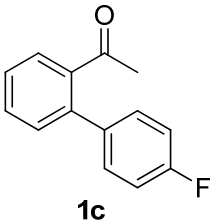
**1-(4'-Methyl-[1,1'-biphenyl]-2-yl)ethanone (1b)**



**1b**

In the typical procedure, the known compound **1b**[s1] was prepared by using *p*-tolylmagnesium bromide (0.5 M in ether) as the reagent. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.55-7.48 (m, 2 H), 7.39 (t, *J* = 7.6 Hz, 2 H), 7.24 (m, 4 H), 2.41 (s, 3 H), 2.02 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 205.1, 140.9, 140.5, 137.8, 137.8, 130.7, 130.2, 129.4, 128.8, 127.8, 127.2, 30.5, 21.2 ppm.

**1-(4'-Fluoro-[1,1'-biphenyl]-2-yl)ethanone (1c)**

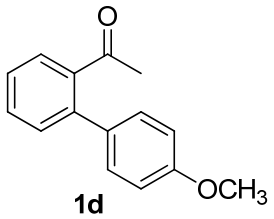


**1c**

In the typical procedure, the known compound **1c**[s2] was prepared by using

4-fluorophenylmagnesium bromide (0.8 M in THF) as the reagent. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* =7.6, 1.0 Hz, 1 H), 7.50 (ddd, *J* =7.6, 7.4, 1.0 Hz, 1 H), 7.42 (ddd, *J* =7.6, 7.4, 1.0 Hz, 1 H), 7.35 (d, *J* = 7.6 Hz, 1 H), 7.30 (dd, *J* = 8.6 Hz, $J_{H\text{-}F}$ = 5.4 Hz, 2 H), 7.12 (dd, *J* = 8.6 Hz, $J_{H\text{-}F}$ = 8.6 Hz, 2 H), 2.05 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 204.3, 162.7 (d, $J_{C\text{-}F}$ = 246.2 Hz), 140.8, 139.4, 136.8 (d, $J_{C\text{-}F}$ = 3.6 Hz), 130.8, 130.5 (d, $J_{C\text{-}F}$ = 8.1 Hz), 130.3, 127.9, 127.6, 115.7 (d, $J_{C\text{-}F}$ = 21.4 Hz), 30.4 ppm.

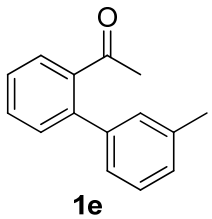
**1-(4'-Methoxy-[1,1'-biphenyl]-2-yl)ethanone (1d)**




**1d**

In the typical procedure, the known compound **1d**[s3] was prepared by using 4-methoxyphenylmagnesium bromide (1 M in THF) as the reagent. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.8 Hz, 1 H), 7.48 (d, *J* = 8.1 Hz, 1 H), 7.38 (m, 2 H), 7.27 (m, 2 H), 6.96 (d, *J* = 8.5 Hz, 2 H), 3.85 (s, 3 H), 2.02 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 205.3, 159.6, 140.9, 140.1, 133.0, 130.7, 130.2, 130.0, 127.8, 127.1, 114.2, 55.3, 30.4 ppm.

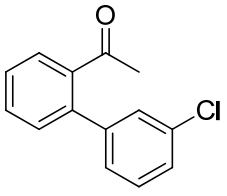
**1-(3'-Methyl-[1,1'-biphenyl]-2-yl)ethanone (1e)**




**1e**

In the typical procedure, the known compound **1e**[s4] was prepared by using *m*-tolylmagnesium bromide (1 M in THF) as the reagent. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.7 Hz, 1 H), 7.50 (dd, *J* = 7.5, 7.4 Hz, 1 H), 7.40-7.38 (m, 2 H), 7.31 (t, *J* = 7.6 Hz, 1 H), 7.21 (d, *J* = 7.6 Hz, 1 H), 7.18 (s, 1 H), 7.14 (d, *J* = 7.6 Hz, 1 H), 2.40 (s, 3 H), 2.01 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 205.0, 140.9, 140.7, 140.7, 138.4, 130.7, 130.2, 129.6, 128.7, 128.6, 127.9, 127.4, 126.1, 30.5, 21.4 ppm.

**1-(3'-Chloro-[1,1'-biphenyl]-2-yl)ethanone (1f)**

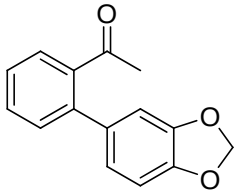



**1f**

In the typical procedure, compound **1f** was prepared by using

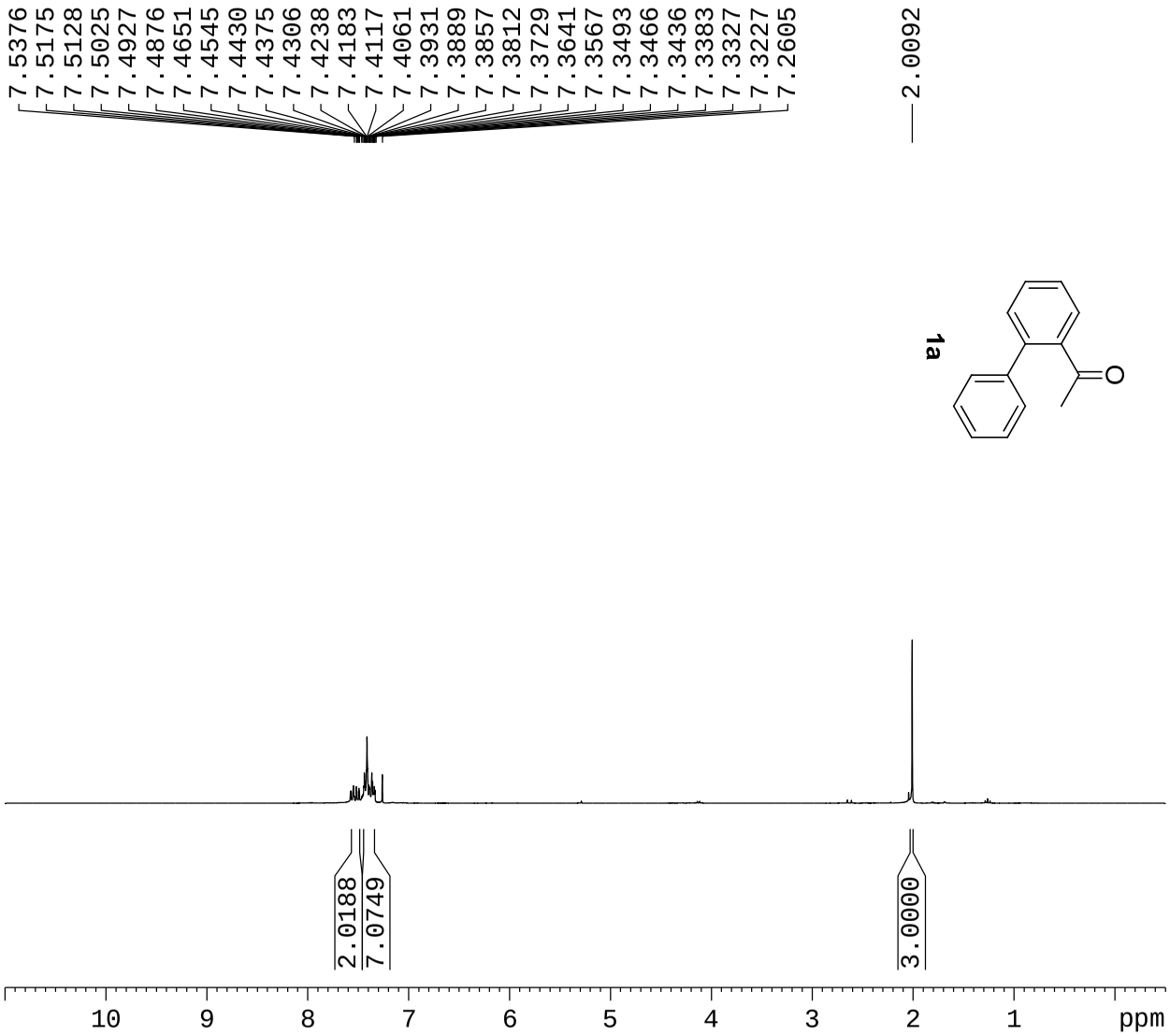
(3-chlorophenyl)magnesium bromide (0.5 M in THF) as the reagent. Flash chromatography (Hexane-ethyl acetate 60:1, 30:1, 15:1) gave **1f** as a pale yellow oil (76%). IR (neat) 3062, 1689, 1594, 1267, 791, 761 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 7.5, 0.8 Hz, 1 H), 7.52 (ddd, *J* = 7.5, 7.5, 1.2 Hz, 1 H), 7.44 (ddd, *J* = 7.5, 7.5, 0.8 Hz, 1 H), 7.40-7.32 (m, 3 H), 7.35 (s, 1 H), 7.19 (ddd, *J* = 7.0, 1.6, 1.6 Hz, 1 H), 2.10 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 204.1, 142.6, 140.5, 139.1, 134.6, 131.0, 130.3, 129.9, 128.7, 128.1, 128.0, 128.0, 127.2, 30.5 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{14}H_{11}ClO$: 230.0498; found: 230.0494.

**1-(2-(Benzo[*d*][1,3]dioxol-5-yl)phenyl)ethanone (1g)**



**1g**

In the typical procedure, compound **1g** was prepared by using 3,4-(methylenedioxy)phenylmagnesium bromide (0.8 M in THF/toluene v/v 50/50) as the reagent. Flash chromatography (Hexane-ethyl acetate 50:1, 30:1, 20:1) gave **1g** as a yellow oil (18%). IR (neat) 3064, 1683, 1591, 1472, 1221, 1037, 750 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.52-7.46 (m, 2 H), 7.40-7.34 (m, 2 H), 6.85 (d, *J* = 8.0 Hz, 1 H), 6.84 (s, 1 H), 6.76 (d, *J* = 8.0 Hz, 1 H), 6.02 (s, 2 H), 2.07 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 205.0, 148.0, 147.6, 140.9, 140.1, 134.6, 130.7, 130.1, 127.8, 127.3, 122.7, 109.2, 108.6, 101.3, 30.4 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{15}H_{12}O_3$: 240.0786; found:240.0783.

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FIDRES 0.137219 Hz
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MCWRK 0.01500000 sec

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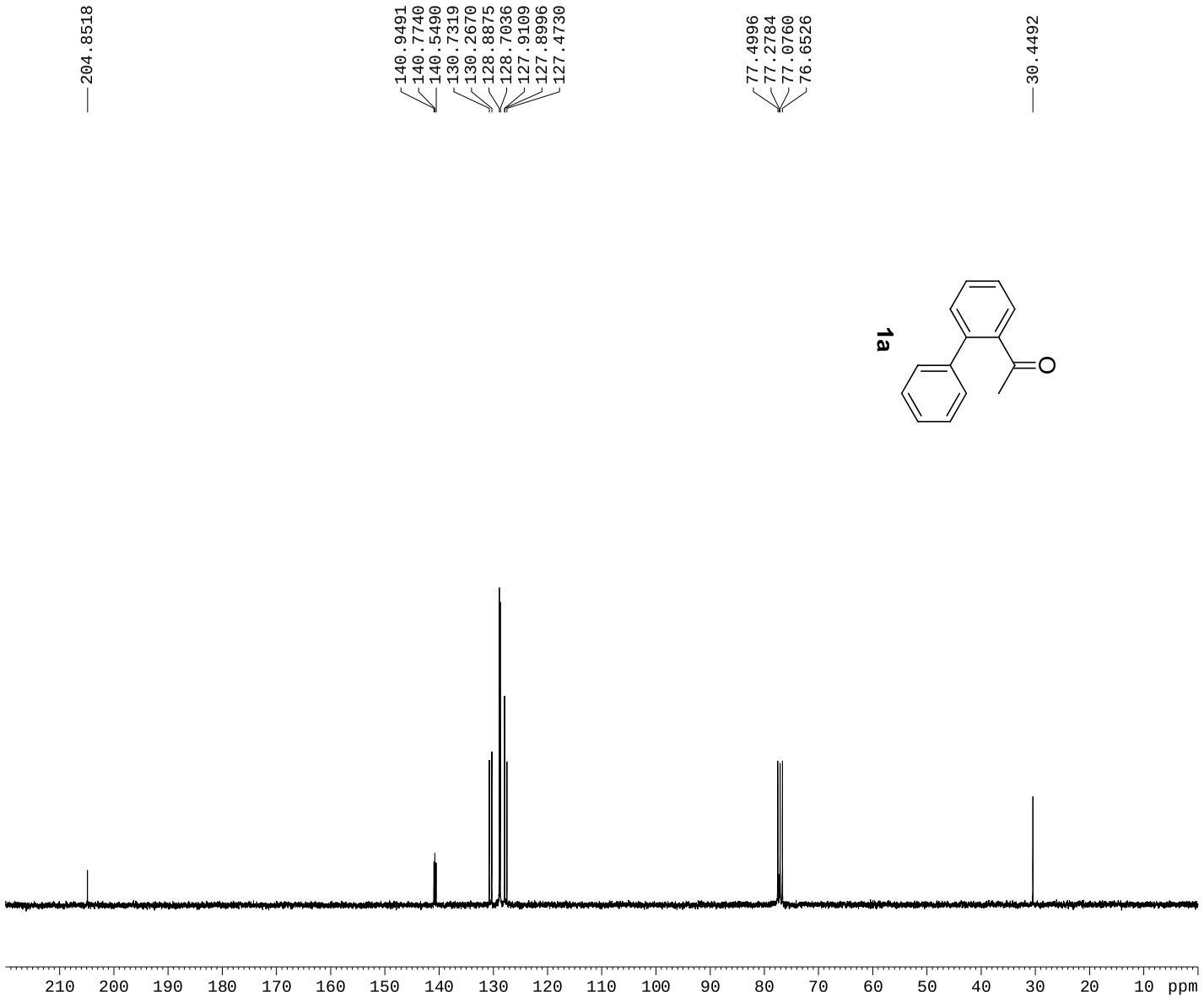


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HZCM           75.46775 Hz/cm
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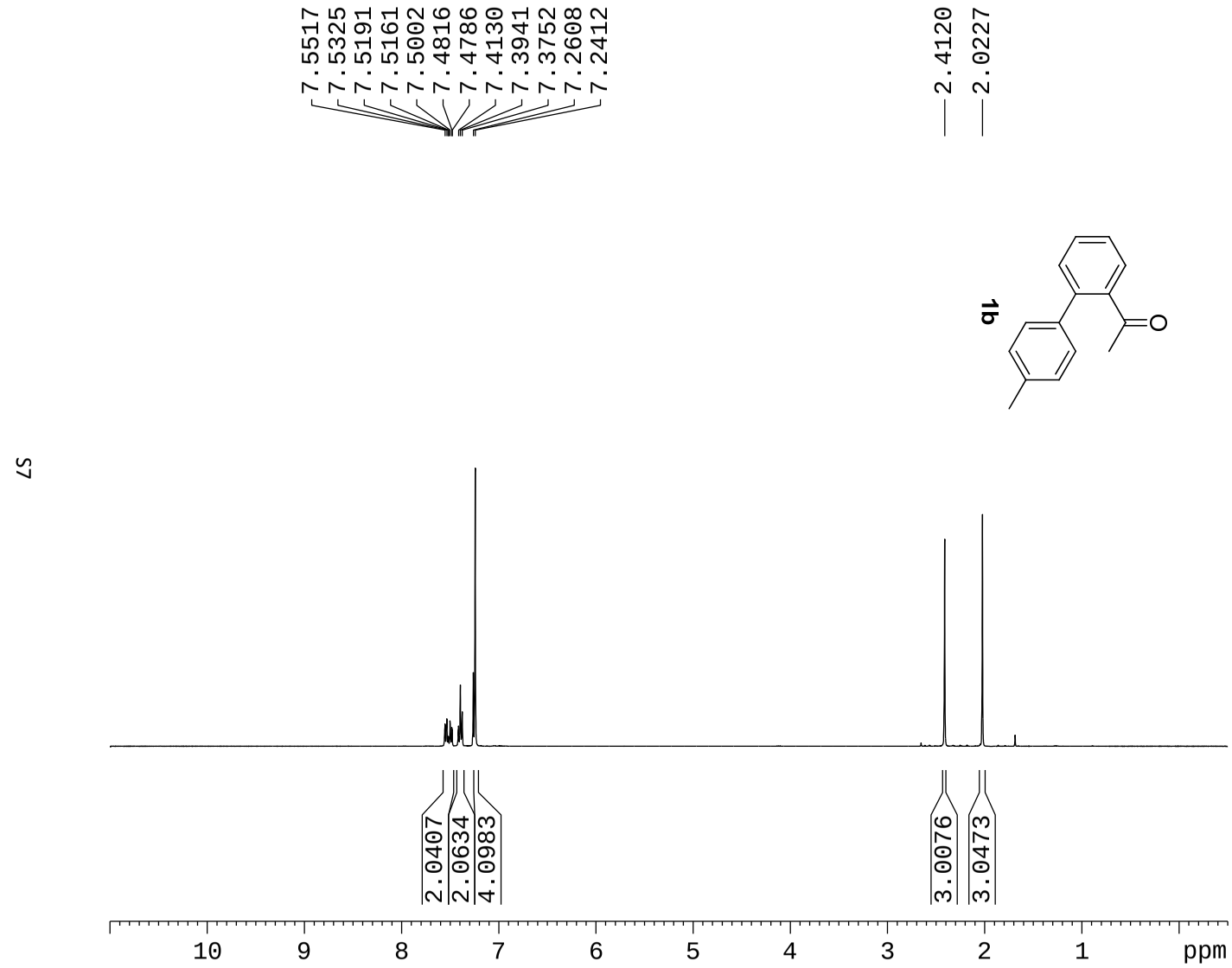


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TD                 32768
SOLVENT            CDCl3
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FIDRES          0.183399 Hz
AQ             2.7263477 sec
RG                   101
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DE                 6.50 usec
TE                299.8 K
D1           2.00000000 sec
TD0                    1

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PL1                0.90 dB
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F2 - Processing parameters
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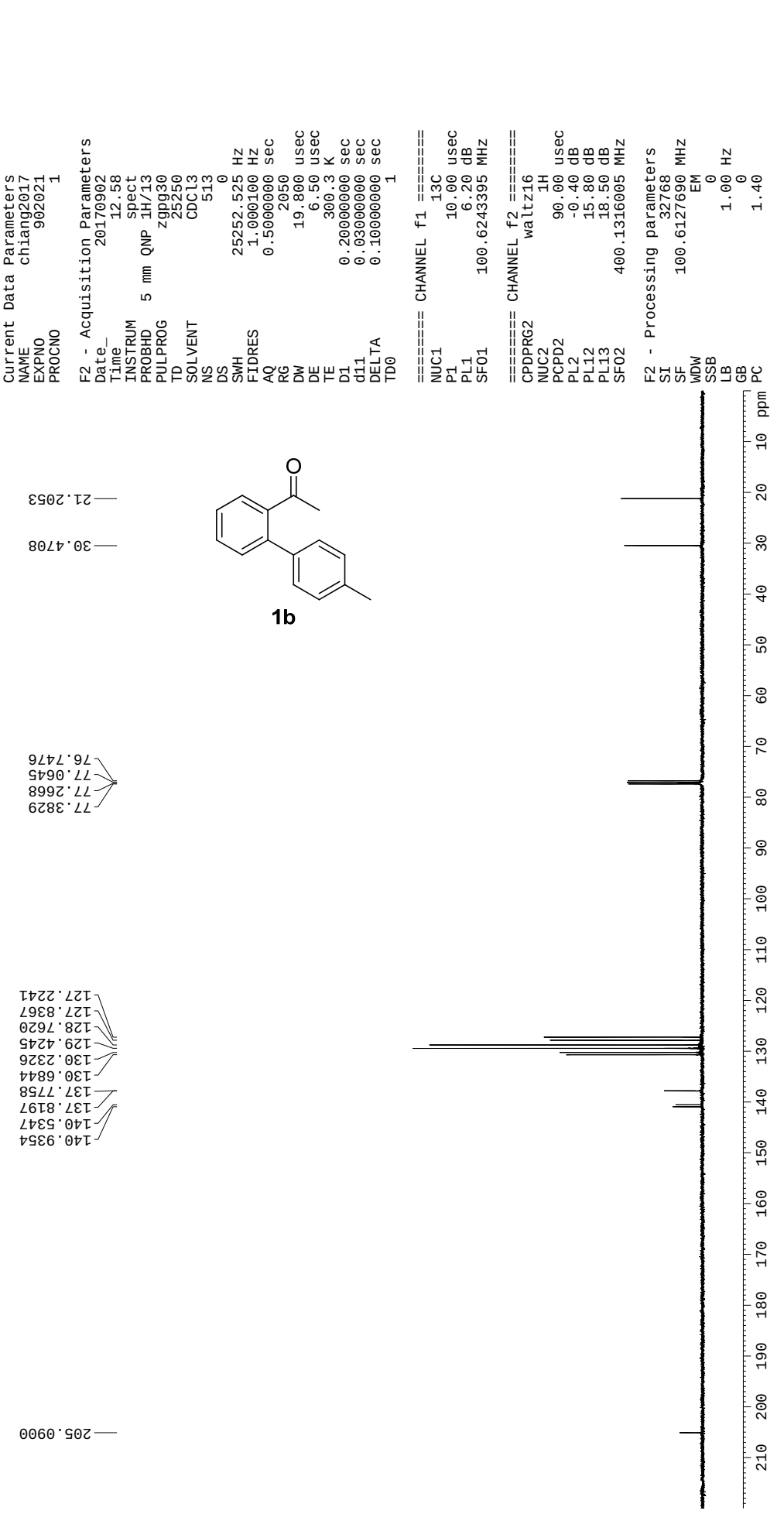
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TE 300.3 K
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NUC1 13C
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SFO1 100.6243395 MHz
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PCPD2 90.00 usec
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PL13 18.50 dB
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GB 0
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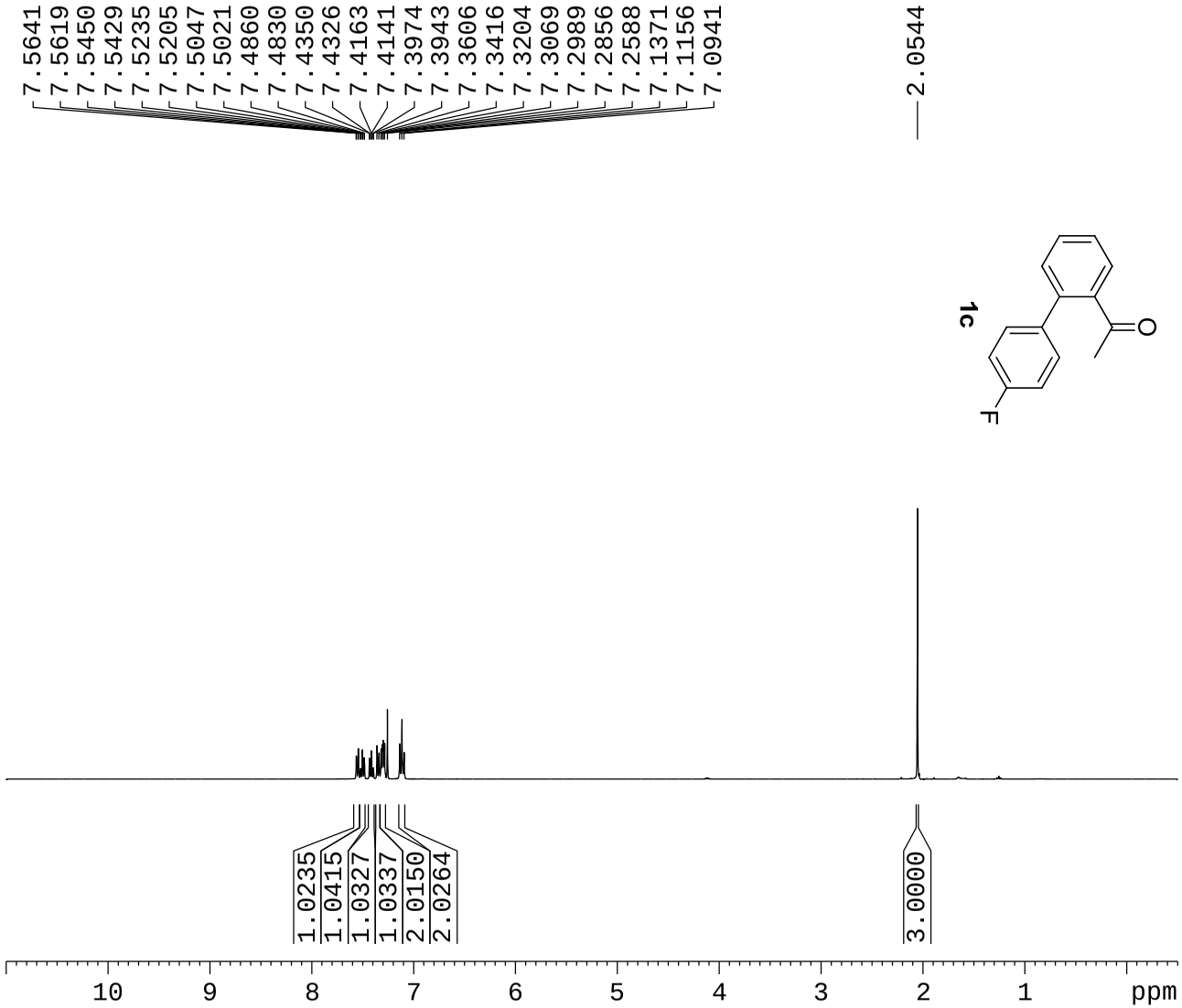
1b
ppm

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FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 203
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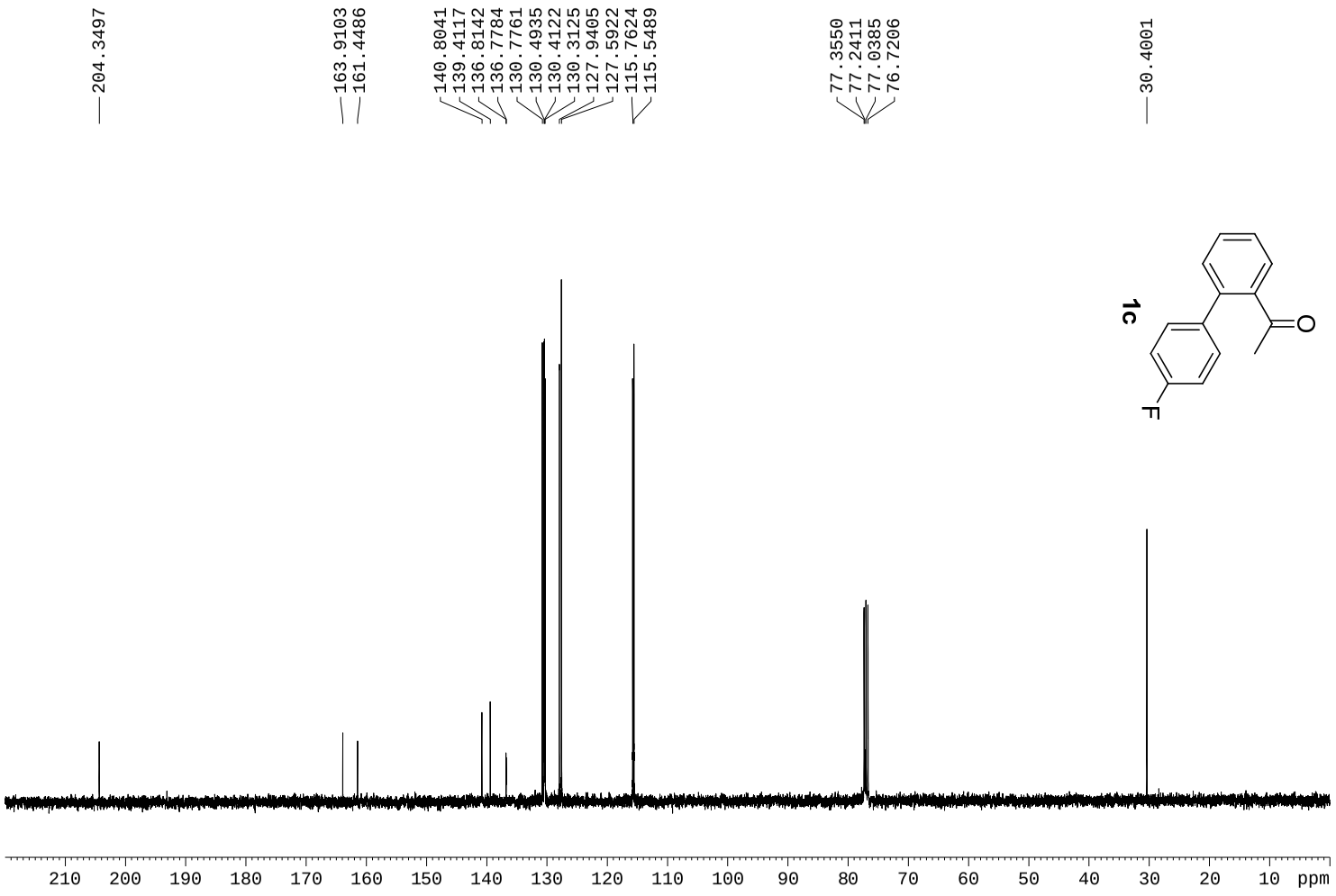


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PCPD2             90.00 usec
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PL12              15.80 dB
PL13              18.50 dB
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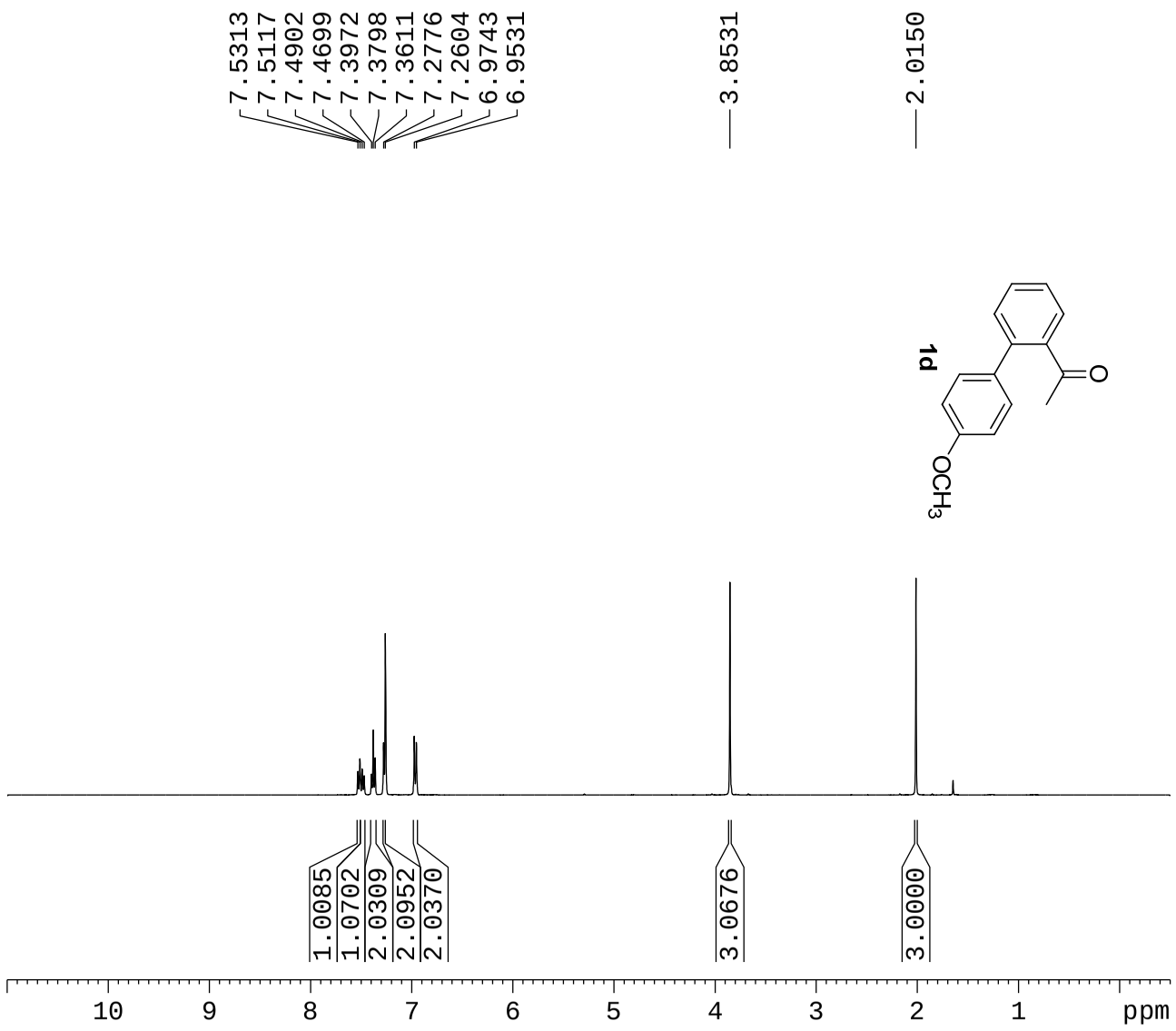


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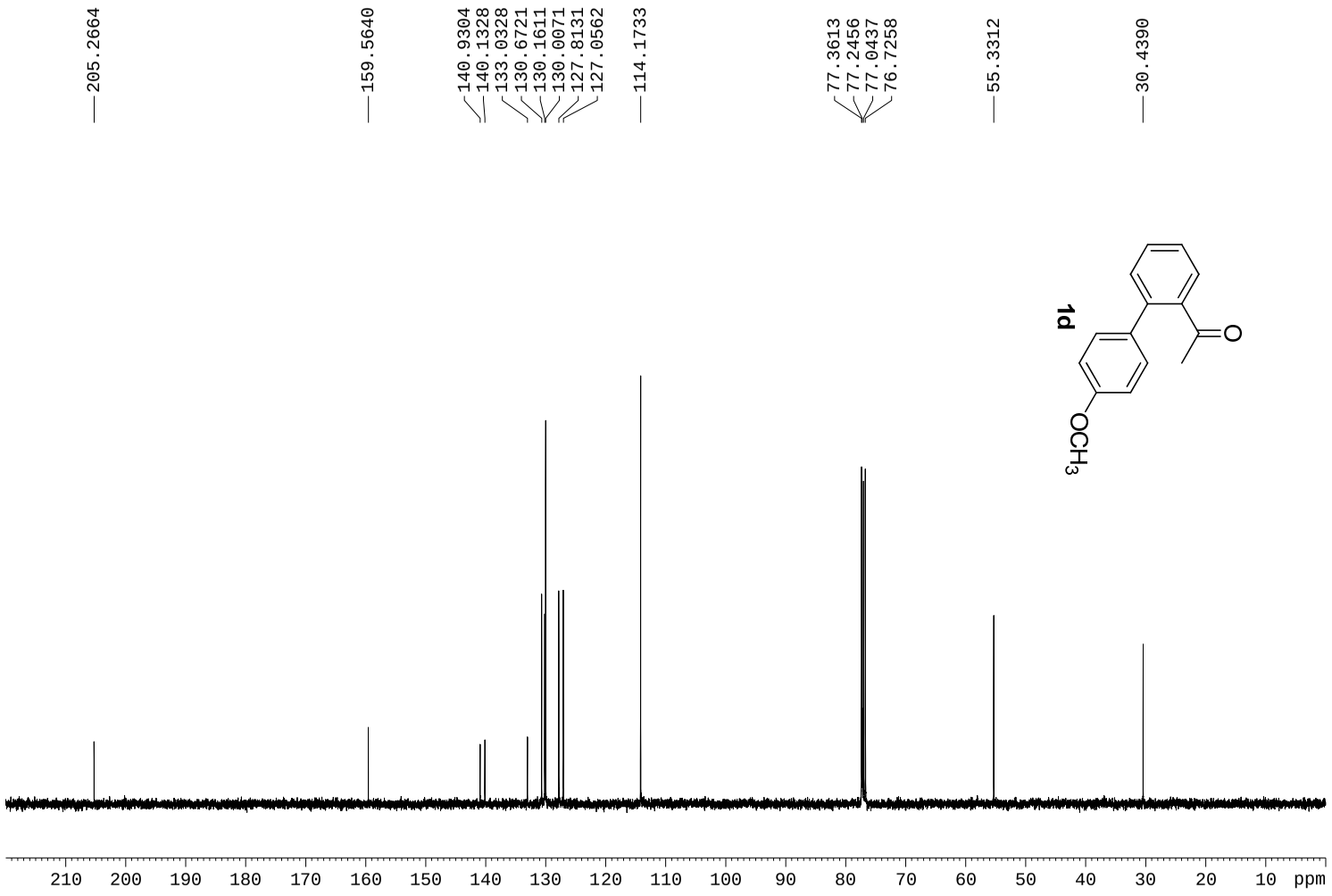


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TE               299.1 K
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TD0                   1

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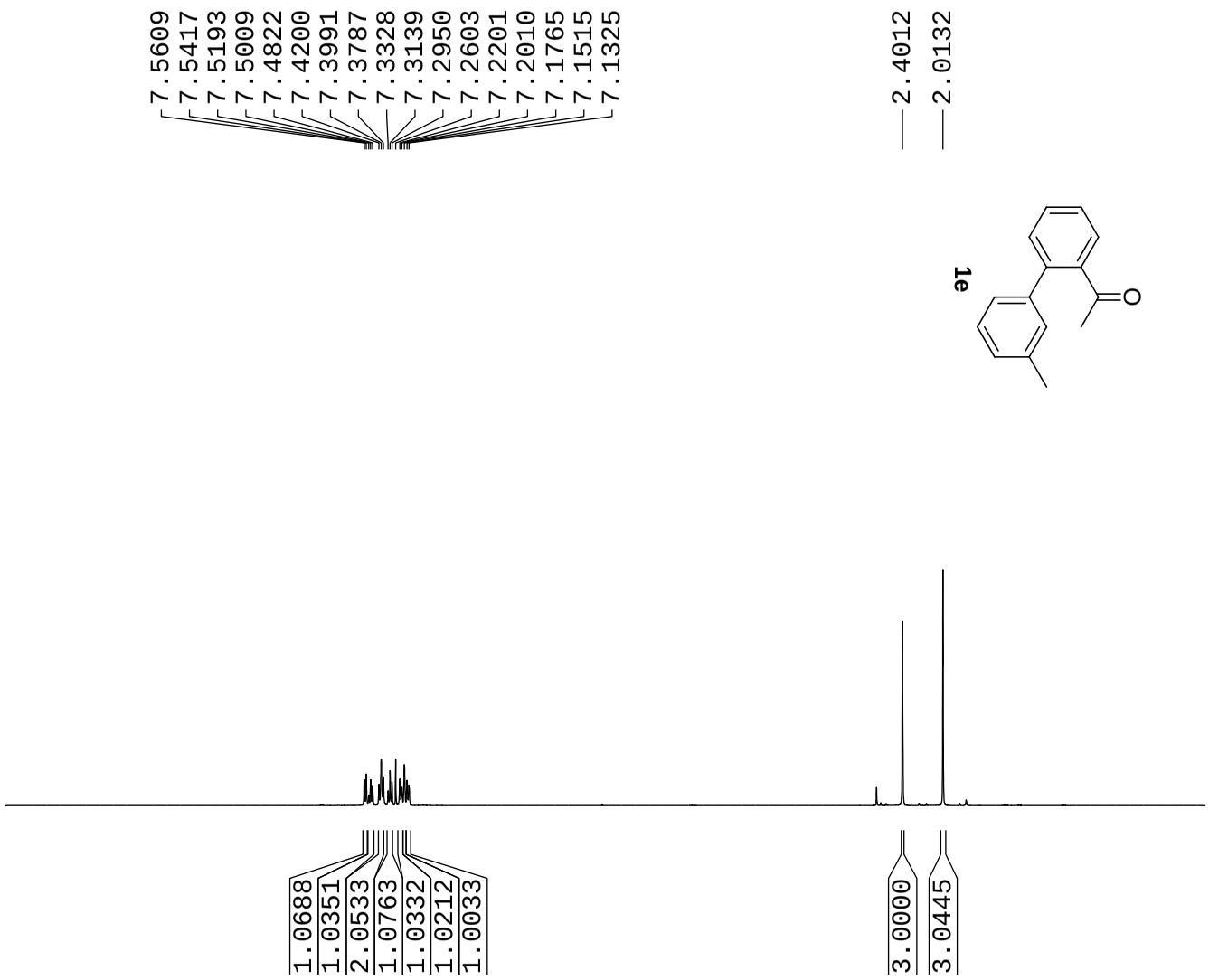


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DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
TD0 1

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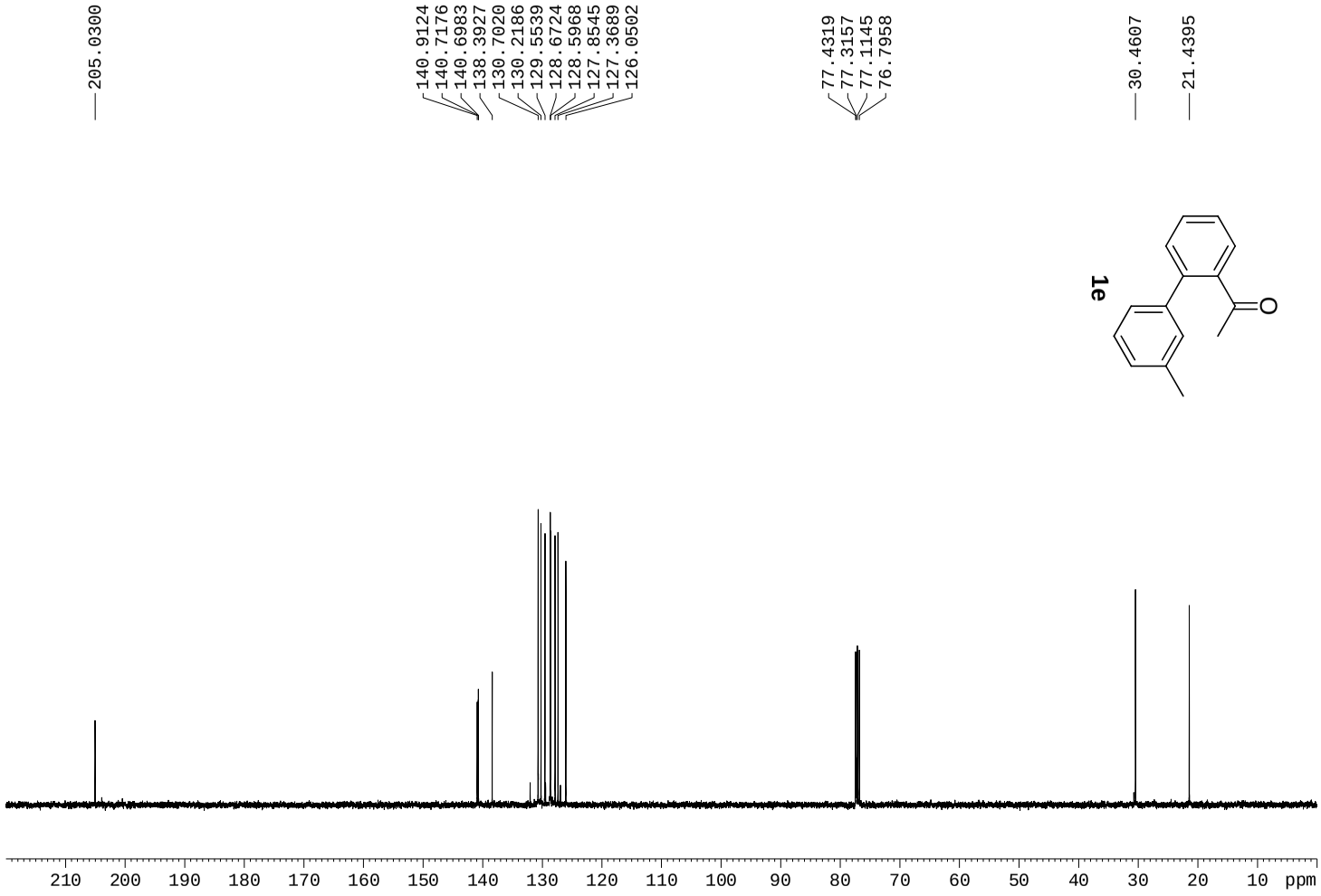


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RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                296.7 K
D1           0.50000000 sec
d11          0.03000000 sec
DELTA        0.40000001 sec
TD0                   1

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SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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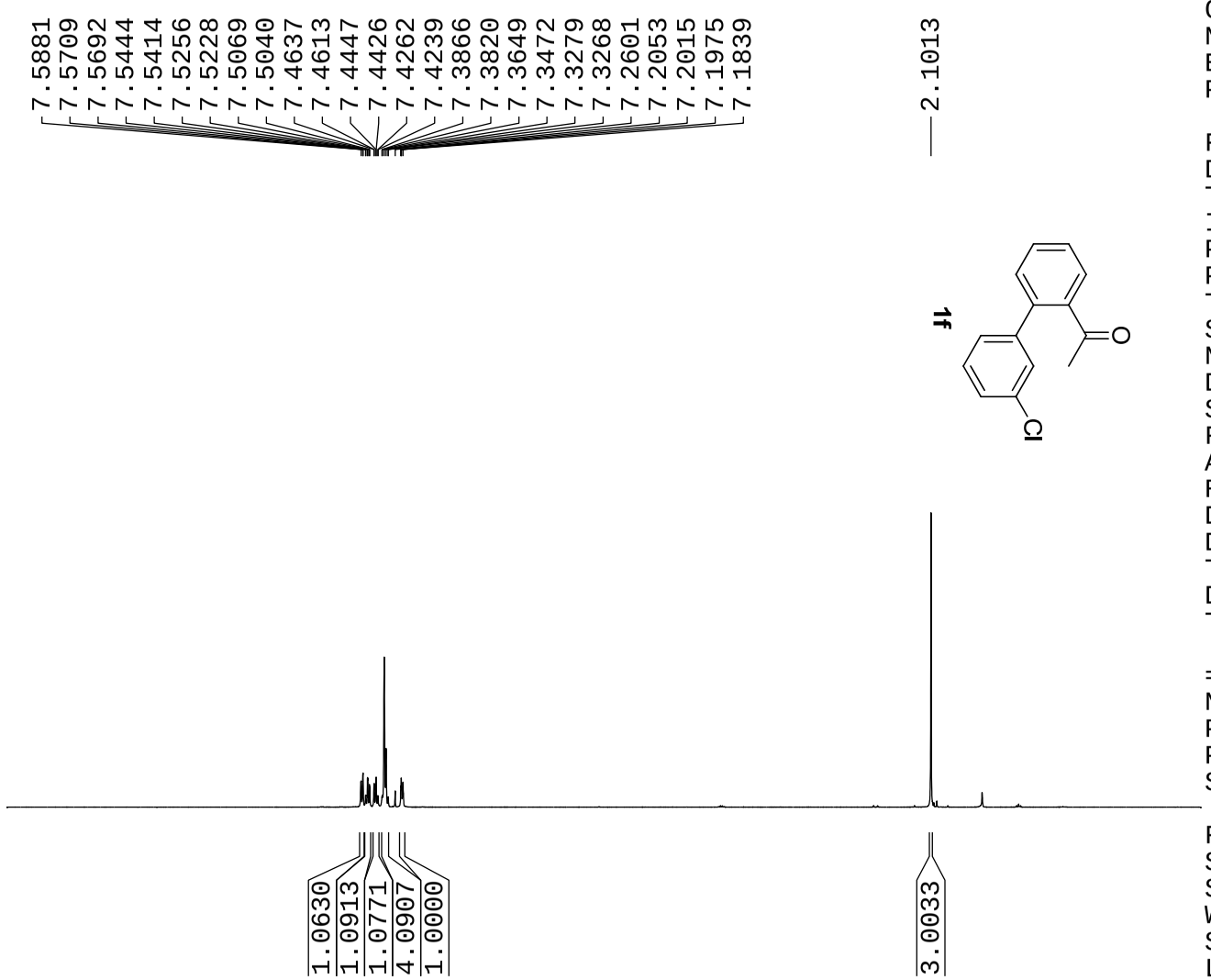


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NAME chen2018
EXPNO 208011
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
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SOLVENT CDCl3
NS 14
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SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 456
DW 83.200 usec
DE 6.50 usec
TE 292.2 K
D1 2.00000000 sec
TD0 1

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NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME chen2018
EXPNO 208012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180208
Time 16.47
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 292.3 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
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NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
Cl
1f
204.0637
142.6134
140.5413
139.0995
134.5890
130.9626
130.3109
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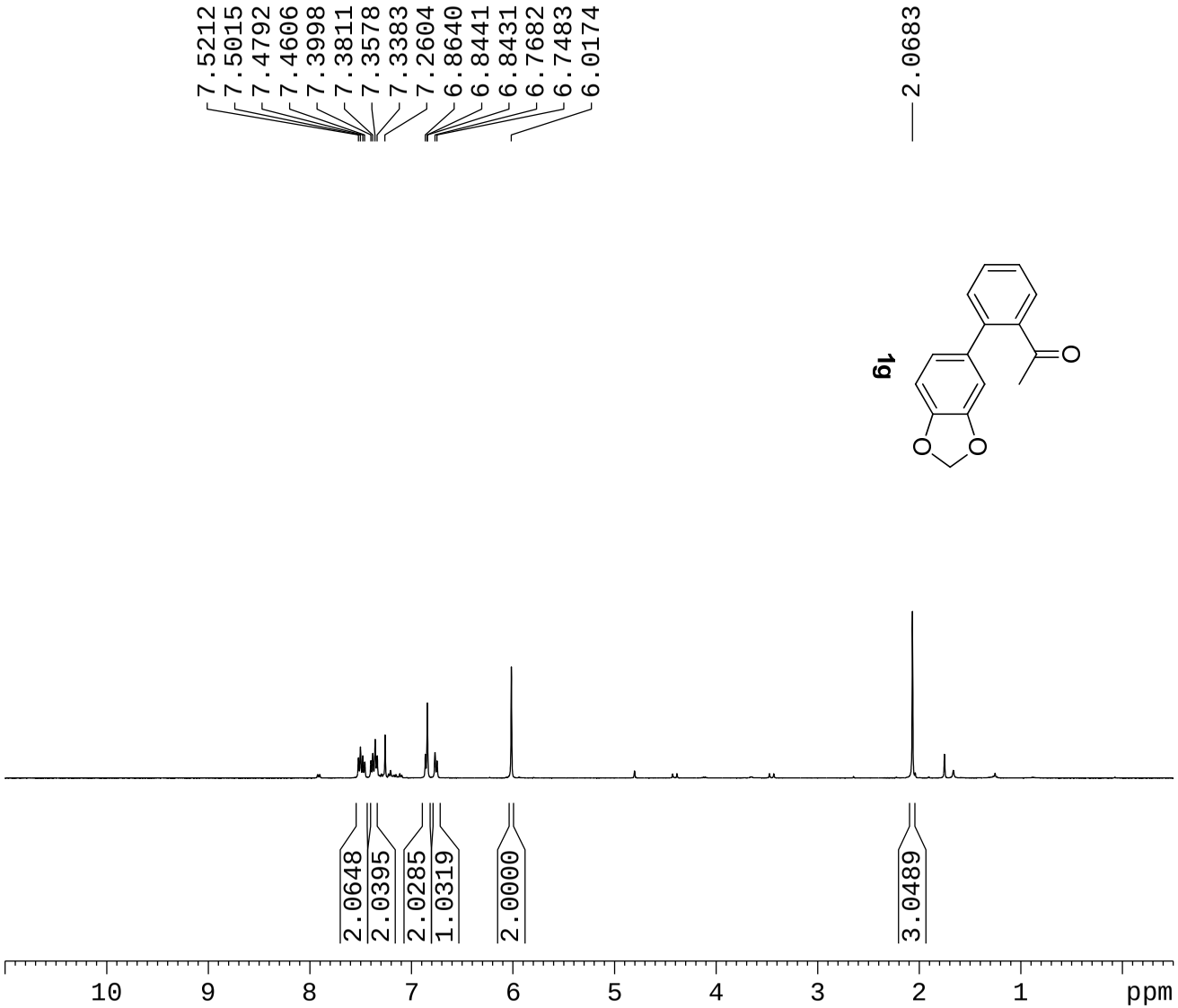
ppm

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EXPNO             320011
PROCNO                 1

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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    11
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   322
DW                83.200 usec
DE                 6.50 usec
TE                297.3 K
D1           2.00000000 sec
TD0                    1

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P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
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WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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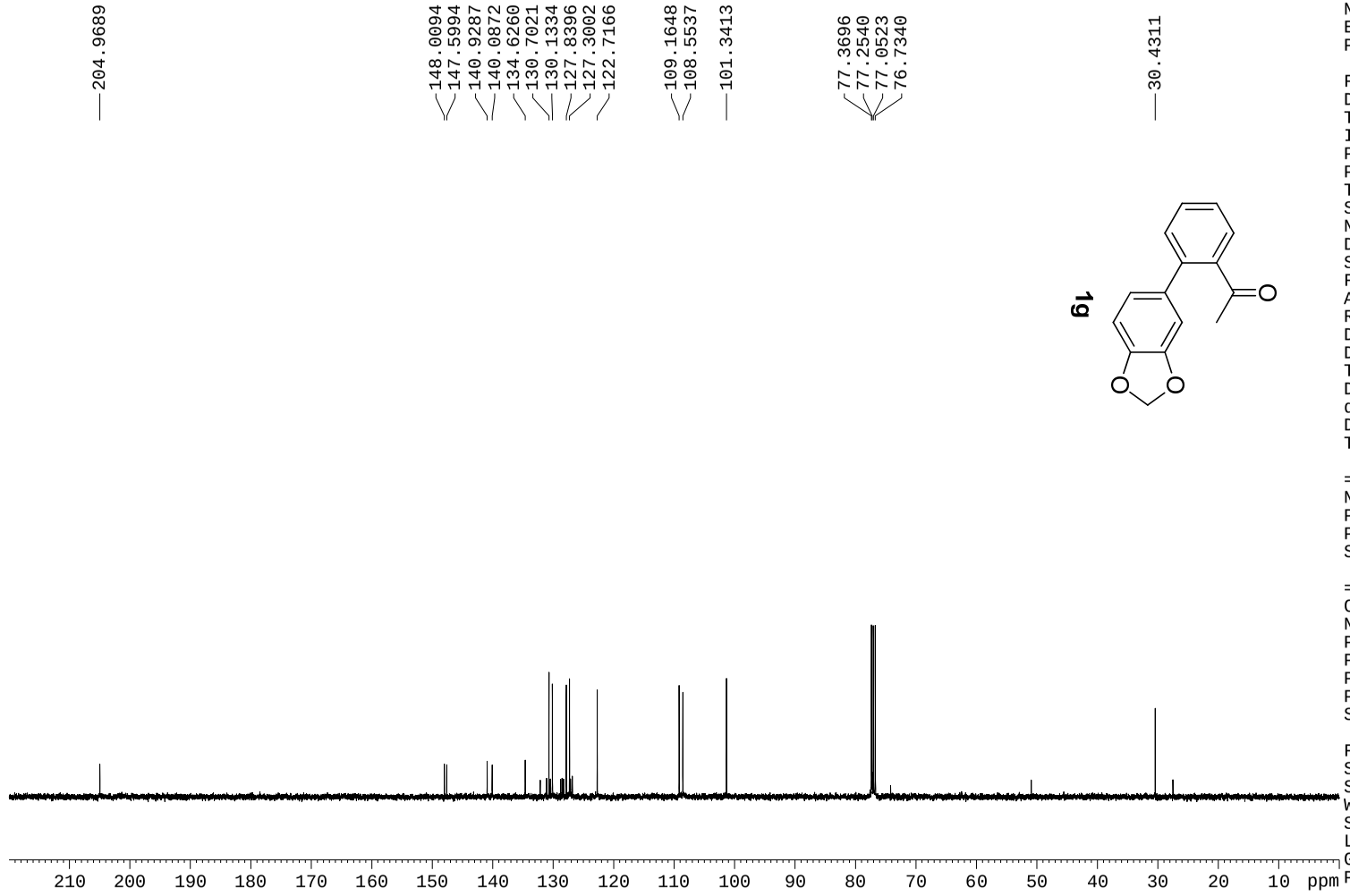


**1g**



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Current Data Parameters
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EXPNO             320021
PROCNO                 1

F2 - Acquisition Parameters
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Time                9.59
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  246
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               297.4 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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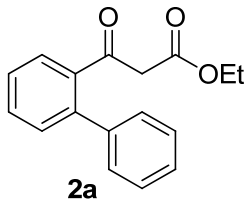
**2) Preparation of 2**

**i) Preparation and NMR spectra of 2a, 2d-f**

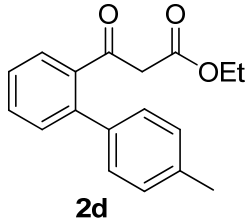
**Ethyl 3-([1,1'-biphenyl]-2-yl)-3-oxopropanoate (2a)**




**2a**

To a stirred suspension of NaH (250.1 mg, 60% dispersion in mineral oil, 6.25 mmol) in toluene (12 mL), diethyl carbonate (0.55 mL, 99%, 4.46 mmol) was added. The mixture was heated at reflux for 1 min followed by the slow addition of a solution of **1a** (438 mg, 2.23 mmol) in toluene (1.7 mL) over 5 min. With stirring, the reaction mixture was continued to reflux for another 15 min, cooled to rt, quenched by glacial acetic acid (6 mL) and dispensed in 40 mL of ice-cold water. The resulting aqueous solution was extracted with ethyl acetate (200 mL x 2). The organic layers were combined and washed with water (100 mL) and brine (100 mL). After concentration, the crude residue was subjected to chromatography (hexane-ethyl acetate 60:1, 30:1, 10:1) to give **2a** in the equilibrium with its enol form (339.4 mg, 57%, keto:enol = 83:17). IR (neat) 3060, 1740, 1693, 1624, 1241, 1192, 744, 701 $cm^{-1}$; $^1H$ NMR (300 MHz, $CDCl_3$) keto-form**:** δ 7.62-7.52 (m, 2 H), 7.46-7.33 (m, 7 H), 4.06 (q, *J* = 7.1 Hz, 2 H), 3.28 (s, 2 H), 1.16 (t, *J* = 7.1 Hz, 3 H) ppm; enol-form**:** δ 12.25 (s, 1 H), 7.62-7.52 (m, 2 H), 7.46-7.33 (m, 7 H), 5.06 (s, 1 H), 4.17 (q, *J* = 7.1 Hz, 2 H), 1.26 (t, *J* = 7.1 Hz, 3 H) ppm; $^{13}C$ NMR (75 MHz, $CDCl_3$) keto-form**:** δ 198.9, 167.0, 140.6, 140.1, 139.4, 131.3, 130.4, 129.0, 128.9, 128.7, 128.2, 127.5, 61.1, 48.8, 14.0 ppm; enol-form**:** δ 198.9, 167.0, 140.6, 140.1, 139.4, 130.9, 130.1, 129.0, 129.0, 128.9, 128.3, 127.2, 92.9, 60.2, 14.2 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{16}O_3$: 268.1099; found: 268.1097.

**Ethyl 3-(4'-methyl-[1,1'-biphenyl]-2-yl)-3-oxopropanoate (2d)**

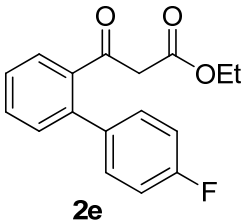



**2d**

In the typical procedure, the titled compound was synthesized from **1b**. Chromatographic purification (hexane-ethyl acetate 60:1, 40:1, 20:1) afforded **2d** in the equilibrium with its enol form (45%, keto:enol = 85:15). IR (neat) 3059, 3024, 2982, 1743, 1694, 1645, 1622, 1269, 1192, 822, 762 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) keto-form**:** δ 7.61 (d, *J* = 7.8 Hz, 1 H), 7.56-7.52 (m, 1 H) , 7.47-7.36 (m, 2

H), 7.27-7.26 (m, 4 H), 4.07 (q, $J$ =7.1 Hz, 2 H), 3.30 (s, 2 H), 2.43 (s, 3 H), 1.18 (t, $J$ = 7.1 Hz, 3 H) ppm; enol-form**:** δ 12.25 (s, 1 H), 7.61 (d, $J$ = 7.8 Hz, 1 H), 7.56-7.52 (m, 1 H), 7.47-7.36 (m, 2 H), 7.20 (d, $J$ = 8.4 Hz, 4 H), 5.11 (s, 1 H), 4.20 (q, $J$ =7.1 Hz, 2 H), 2.40 (s, 3 H), 1.29 (t, $J$ =7.1 Hz, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) keto-form**:** δ 199.0, 167.0, 140.6, 139.4, 138.1, 137.2, 131.2, 130.3, 129.6, 128.9, 128.6, 127.3, 61.0, 48.8, 21.2, 14.0; enol-form**:** 199.0, 167.0, 140.6, 139.4, 138.1, 137.2, 130.8, 130.1, 129.1, 129.0, 128.5, 127.0, 92.6, 60.1, 21.2, 14.2 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{18}H_{18}O_3$: 282.1256; found: 282.1251.

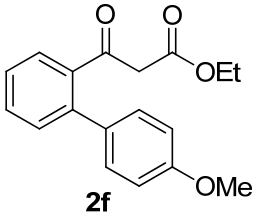
**Ethyl 3-(4'-fluoro-[1,1'-biphenyl]-2-yl)-3-oxopropanoate (2e)**




**2e**

In the typical procedure, the titled compound was synthesized from **1c**. Chromatographic purification (hexane-ethyl acetate 80:1, 40:1) afforded **2e** in the equilibrium with its enol form (48%, keto:enol = 77:23). IR (neat) 3264, 3062, 2985, 1739, 1696, 1513, 839, 819, 763 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) keto-form**:** δ 7.60 (d, $J$ = 7.8 Hz, 1 H), 7.57-7.52 (m, 1 H), 7.49-7.41 (m, 1 H), 7.39-7.31 (m, 3 H), 7.13 (dd, $J$ = 8.5 Hz, $J_{H\text{-}F}$ = 8.5 Hz, 2 H), 4.08 (q, $J$ = 7.1 Hz, 2 H), 3.34 (s, 2 H), 1.18 (t, $J$ = 7.1 Hz, 3 H) ppm; enol-form**:** δ 12.26 (s, 1 H), 7.60 (d, $J$ = 7.8 Hz, 1 H), 7.57-7.52 (m, 1 H), 7.49-7.41 (m, 1 H), 7.39-7.31 (m, 3 H), 7.07 (dd, $J$ = 8.6 Hz, $J_{H\text{-}F}$ = 8.6 Hz, 2 H), 5.05 (s, 1 H), 4.18 (q, $J$ = 7.2 Hz, 2 H), 1.27 (t, $J$ = 7.2 Hz, 3 H) ppm. $^{13}$C NMR (100 MHz, $CDCl_3$) keto-form**:** δ 201.1, 166.9, 162.8 (d, $J_{C\text{-}F}$ = 246.9 Hz), 139.6, 139.2, 136.1 (d, $J_{C\text{-}F}$ = 3.7 Hz), 131.4, 130.6, 130.5 (d, $J_{C\text{-}F}$ = 7.0 Hz), 128.6, 127.7, 115.9 (d, $J_{C\text{-}F}$ = 21.5 Hz), 61.2, 48.7, 14.0 ppm; enol-form**:** δ 198.5, 166.9, 162.8 (d, $J_{C\text{-}F}$ = 246.9 Hz), 139.6, 139.2, 136.1 (d, $J_{C\text{-}F}$ = 3.7 Hz), 131.4, 130.8, 130.2 (d, $J_{C\text{-}F}$ = 6.6 Hz), 129.1, 127.4, 115.2 (d, $J_{C\text{-}F}$ = 21.8 Hz), 92.8, 60.3, 14.2 ppm; $^{19}$F NMR (376 MHz, $CDCl_3$): keto-form: δ -113.9, enol-form: δ -115.4 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{15}FO_3$: 286.1005; found: 286.1012.

**Ethyl 3-(4'-methoxy-[1,1'-biphenyl]-2-yl)-3-oxopropanoate (2f)**




**2f**

In the typical procedure, the titled compound was synthesized from **1d**. Chromatographic purification (hexane-ethyl acetate 50, 40:1, 30:1, 10:1) afforded **2f**

in the equilibrium with its enol form (67%, keto:enol = 83:17). IR (neat) 3060, 2920, 1739, 1695, 1609, 1516, 1243, 1178, 833, 763 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) keto-form**:** δ 7.58 (d, *J* = 7.3 Hz, 1 H), 7.52 (dd, *J* = 7.5, 1 Hz, 1 H), 7.42-7.26 (m, 2 H), 7.29-7.26 (m, 2 H), 6.97 (d, *J* = 8.6 Hz, 2 H), 4.06 (q, *J* = 7.1 Hz, 2 H), 3.86 (s, 3 H), 3.28 (s, 2H), 1.17 (t, *J* = 7.1 Hz, 3 H) ppm; enol-form**:** δ 12.28 (s, 1 H), 7.54-7.47 (m, 1 H), 7.47-7.43 (m, 1 H), 7.38-7.28 (m, 4 H), 6.92 (d, *J* = 8.7 Hz, 2 H), 5.08 (s, 1 H), 4.18 (q, *J* = 7.1 Hz, 2 H), 3.84 (s, 3 H), 1.27 (t, *J* = 7.1 Hz, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) keto-form**:** δ 199.3, 167.1, 159.7, 140.2, 139.3, 132.3, 131.2, 130.2, 130.1, 128.6, 127.1, 114.1, 61.1, 55.3, 48.7, 14.0 ppm; enol-form**:** 174.4, 172.6, 158.9, 133.6, 133.3, 130.8, 130.1, 129.7 (x 2), 129.1, 126.8, 113.7, 92.6, 60.2, 55.3, 14.2 ppm.

Current Data Parameters
NAME chiang2017
EXPNO 50611
PROCNO 1
F2 - Acquisition Parameters
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Time 11.23
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 362
DW 111.200 use
DE 6.50 use
TE 303.2 K
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MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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PL1 0.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 16384
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
O
O
OEt
OH
O
OEt
2a
(keto/enol = 83/17)
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4.0948
4.1360
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4.1835
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5.0595
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7.3406
7.3457
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7.3722
7.3856
7.3914
7.4096
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4
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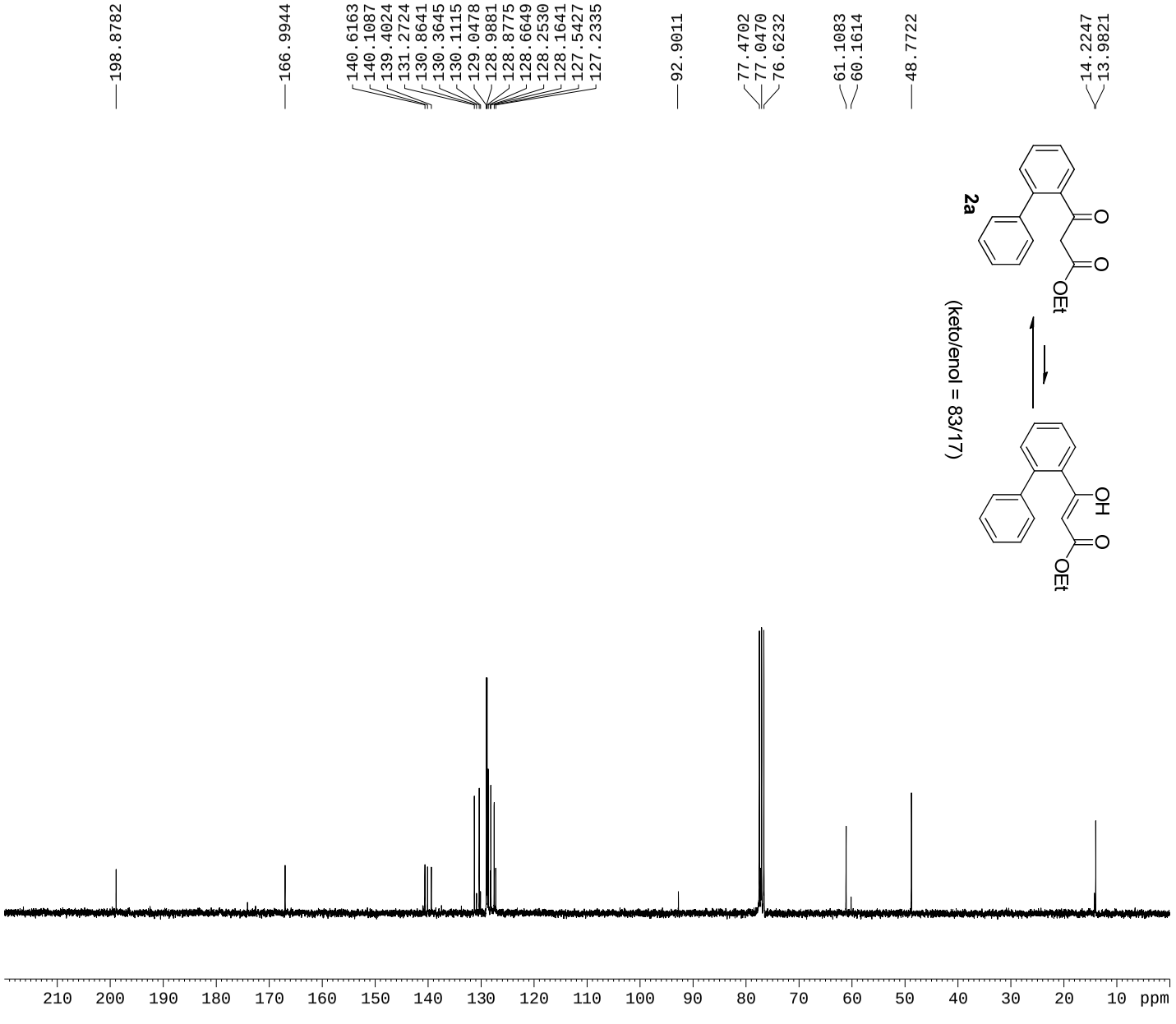
ppm

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PROCNO                 1

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TD                65536
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NS                  848
DS                    0
SWH           18832.393 Hz
FIDRES         0.287360 Hz
AQ             1.7400308 sec
RG                13004
DW               26.550 usec
DE                6.50 usec
TE                304.2 K
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d11           0.03000000 sec
DELTA         0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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NUC1                13C
P1                11.10 usec
PL1                0.00 dB
SFO1         75.4763978 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              18.80 dB
PL13              21.80 dB
SFO2        300.1313506 MHz

F2 - Processing parameters
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SF           75.4677477 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
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CY                 12.50 cm
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F2P              120.000 ppm
F2              9056.13 Hz
PPMCM            1.50000 ppm/cm
HZCM           113.20163 Hz/cm
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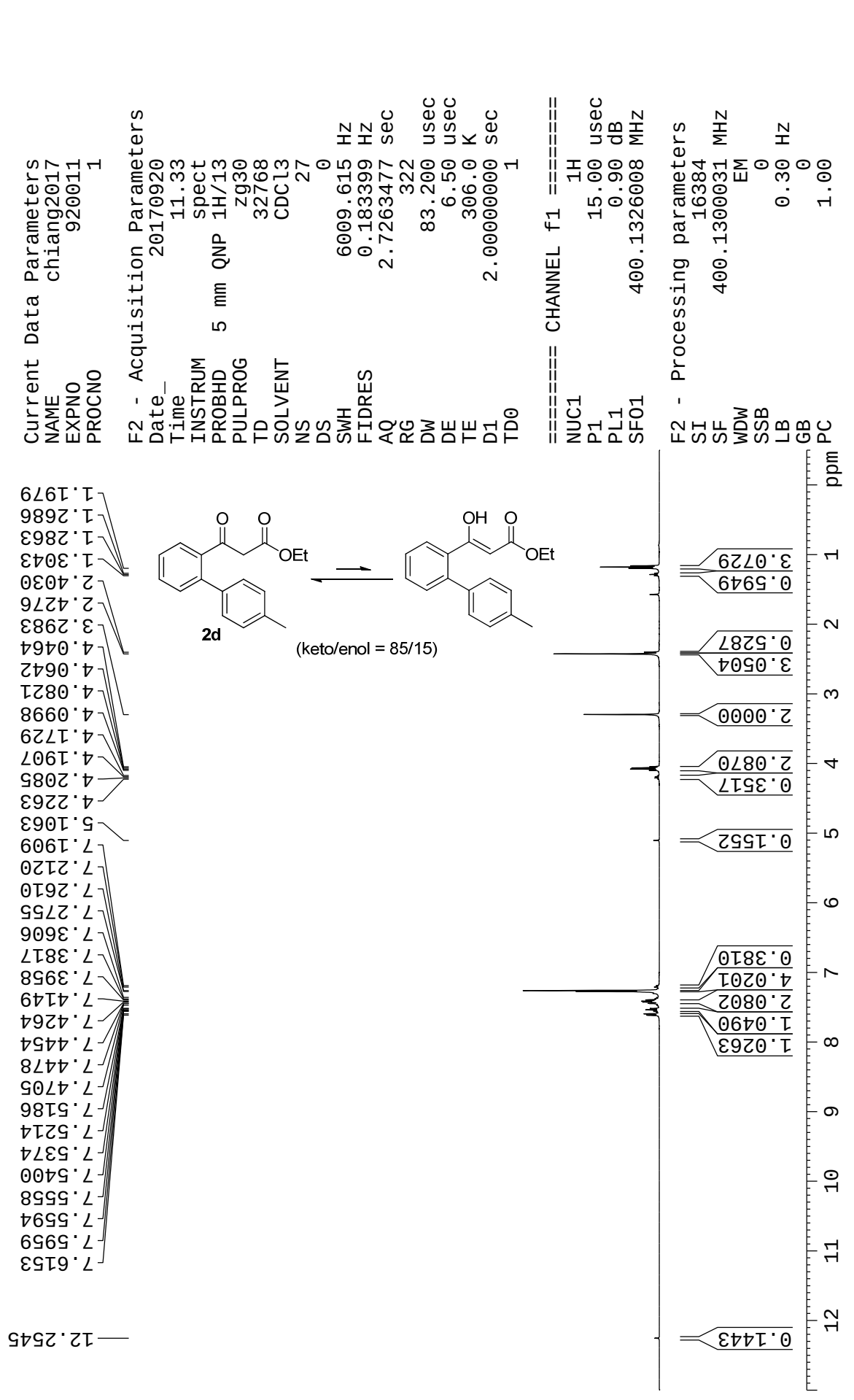
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PROCNO 1
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 27
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 322
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DE 6.50 usec
TE 306.0 K
D1 2.00000000 sec
TD0 1
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NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
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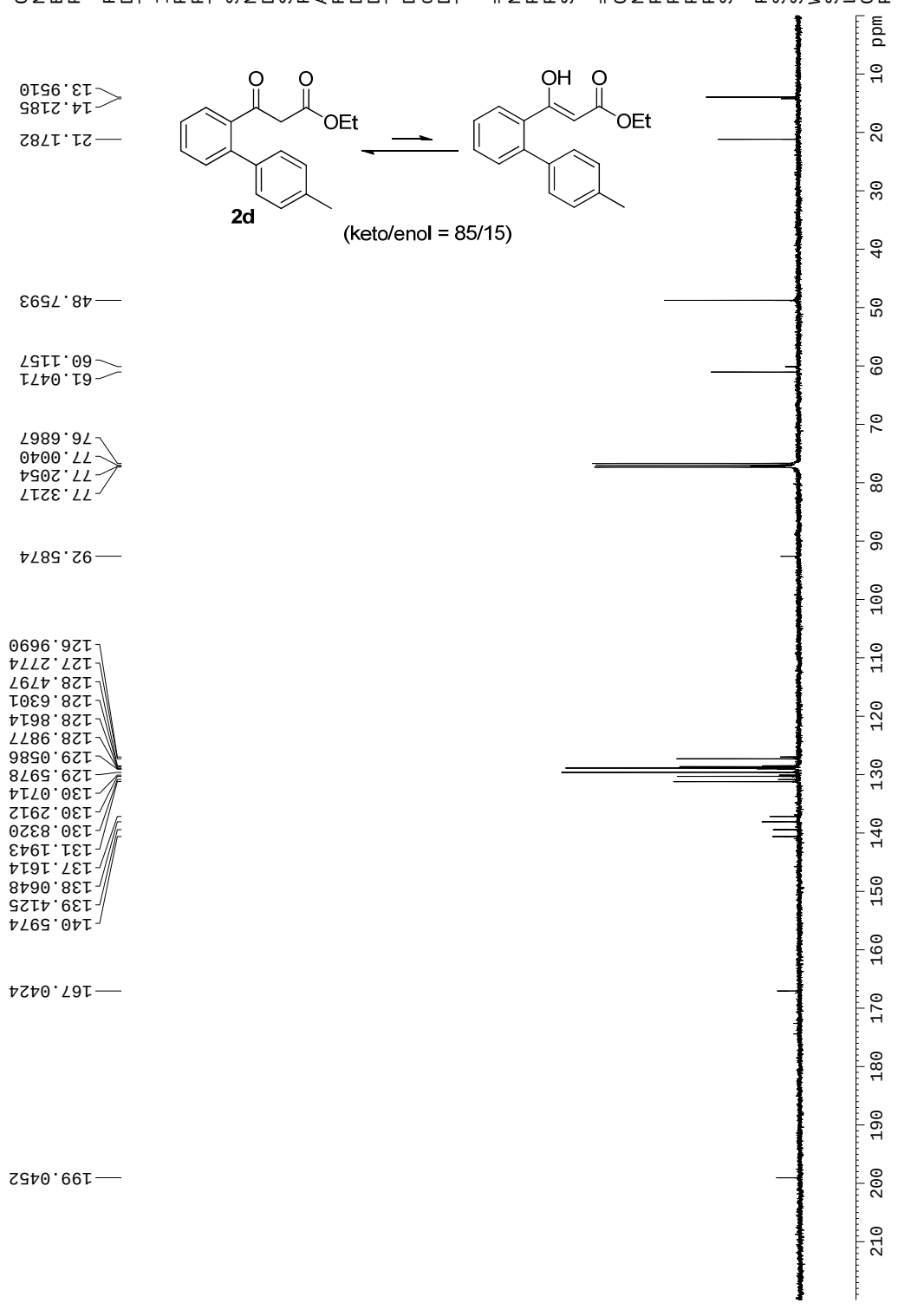
2d
(keto/enol = 85/15)
OEt
OH
ppm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EXPNO 920021
PROCNO 1
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Time 11.47
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PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 2068
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 306.5 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.9510
14.2185
21.1782
48.7593
60.1157
61.0471
76.6867
77.0040
77.2054
77.3217
92.5874
126.9690
127.2774
128.4797
128.6301
128.8614
128.9877
129.0586
129.5978
130.0714
130.2912
130.8320
131.1943
137.1614
138.0648
139.4125
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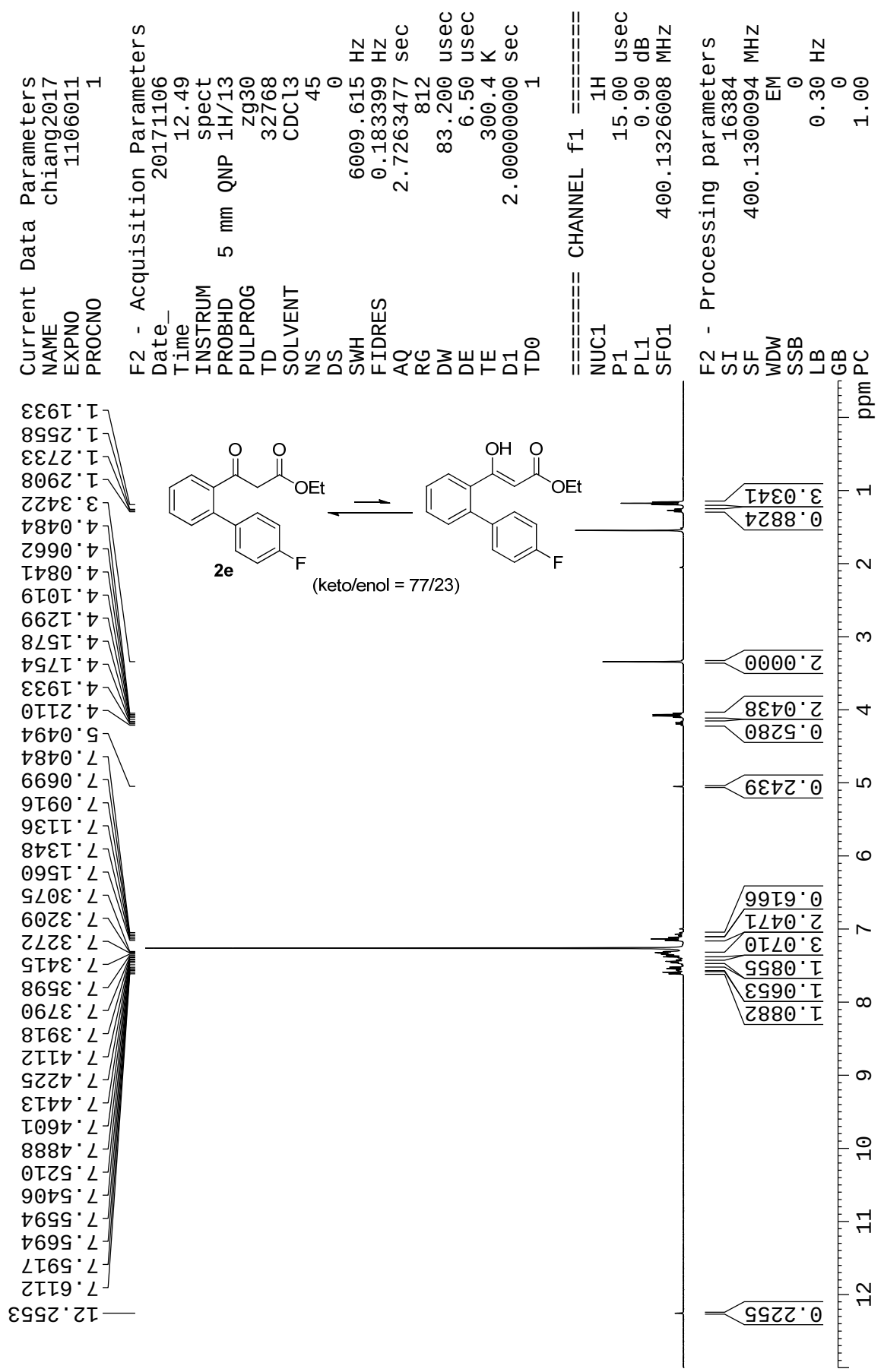
O
O
OEt
OH
O
OEt
2d
(keto/enol = 85/15)
ppm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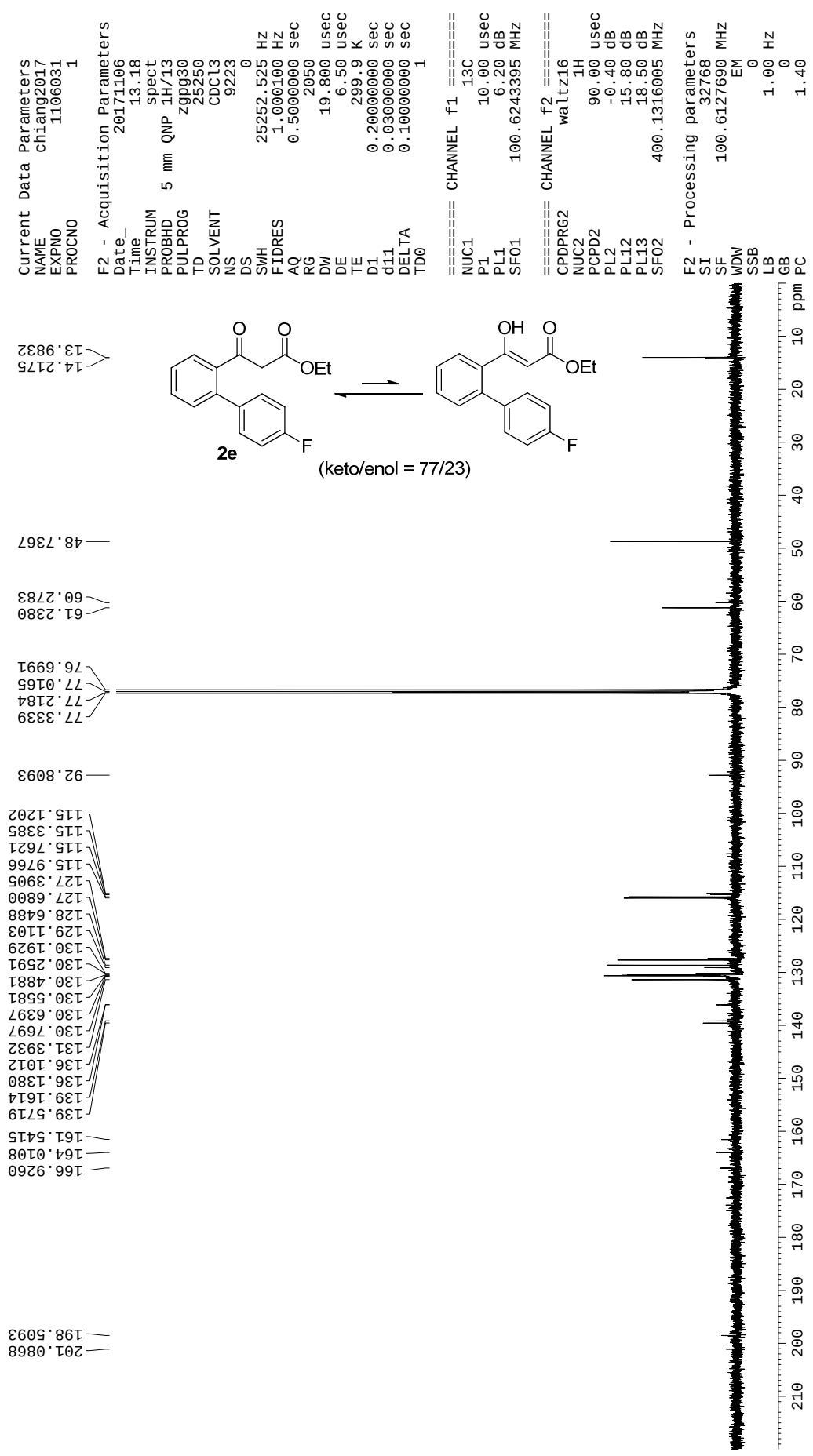
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 45
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 812
DW 83.200 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2e
(keto/enol = 77/23)
OEt
OH
F
ppm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PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 9223
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 299.9 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
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NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
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61.2380
76.6991
77.0165
77.2184
77.3339
92.8093
115.1202
115.3385
115.7621
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127.6800
128.6488
129.1103
130.1929
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130.4881
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O
OEt
F
2e
OH
O
OEt
F
(keto/enol = 77/23)
ppm

Current Data Parameters
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EXPNO 1106041
PROCNO 1
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OEt
F
2e
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(keto/enol = 77/23)
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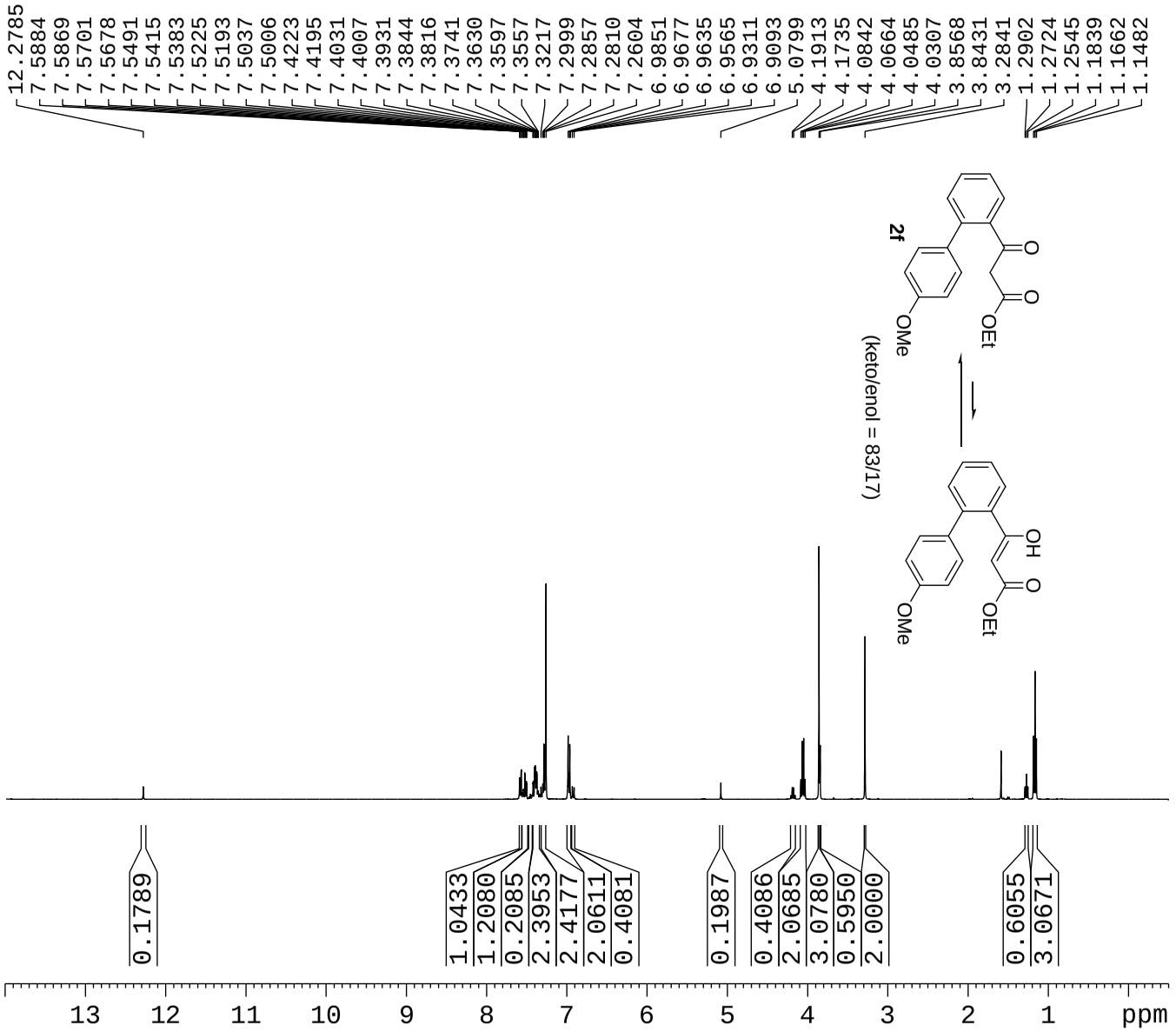


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| PROCNO | 1 |

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| D1 | 1.00000000 sec |
| TD0 | 1 |

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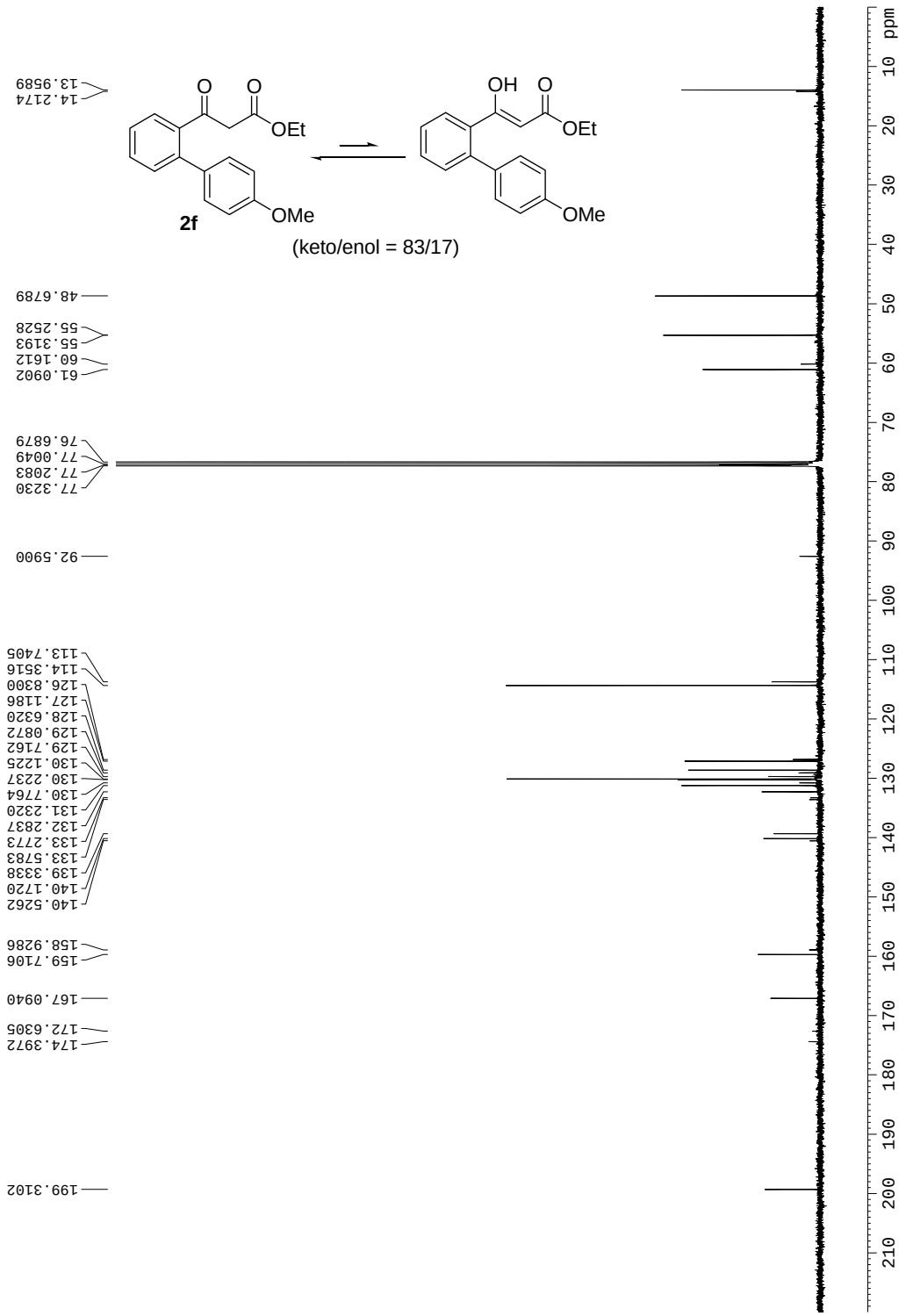
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DE 6.50 usec
TE 296.1 K
D1 0.50000000 sec
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PL13 18.50 dB
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F2 - Processing parameters
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130.2237
130.7764
131.2320
132.2837
133.2773
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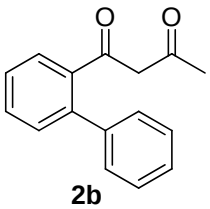
140.5262
158.9286
159.7106
167.0940
172.6305
174.3972
199.3102
OEt
OMe
2f
OH
(keto/enol = 83/17)
ppm

**ii) Preparation and NMR spectra of 2b, 2g-l**

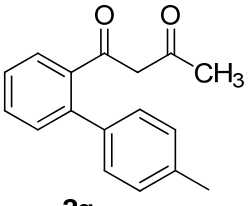
**1-([1,1'-biphenyl]-2-yl)butane-1,3-dione (2b)**



**2b**

To a stirred solution of **1a** (362 mg, 1.85 mmol) in toluene (15 mL) pre-cooled at 0 °C, LiHMDS (2.77 mL, 1 M in toluene, 2.77 mmol) was dropwise added over 5 min. After stirring at 0 °C for 30 min, acetyl chloride (0.27 mL, 98%, 3.69 mmol) was added in one portion. The reaction mixture was then allowed to stir at rt for an additional 3 min, quenched by glacial acetic acid (3 mL) and diluted with ethyl acetate (300 mL). The solution was washed with water (100 mL) and brine (100 mL), and concentrated under reduced pressure. The crude residue was subjected to chromatography (hexane-ethyl acetate 70:1, 50:1, 40:1) to afford **2b** in the equilibrium with its enol-form (235.4 mg, 54%, keto/enol = 37/63). IR (neat) 3060, 3027, 2925, 1602, 1277, 772, 746, 700 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) keto-form: δ 7.66 (d, *J* = 7.7 Hz, 1 H), 7.56-7.49 (m, 1 H), 7.44-7.35 (m, 7 H), 3.38 (s, 2 H), 2.01 (s, 3 H) ppm; enol-form: δ 15.59 (s, 1 H), 7.66 (d, *J* = 7.7 Hz, 1 H), 7.56-7.49 (m, 1 H), 7.44-7.35 (m, 7 H), 5.36 (s, 1 H), 1.92 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) keto-form: δ 190.9, 188.4, 141.0, 140.8, 136.1, 130.7, 130.6, 128.9, 128.8, 128.3, 127.4, 127.3, 57.4, 24.9 ppm; enol-form: δ 190.9, 188.4, 141.0, 140.8, 136.1, 130.7, 130.6, 128.9, 128.8, 128.3, 127.4, 127.3, 102.3, 24.9 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{14}O_2$: 238.0994; found: 238.0987.

**1-(4'-Methyl-[1,1'-biphenyl]-2-yl)butane-1,3-dione (2g)**

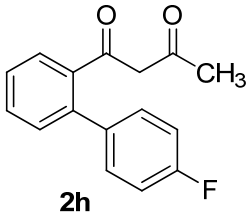


**2g**

Following the typical procedure, compound **2g** was prepared from **1b**. Chromatographic purification (hexane-ethyl acetate 90:1, 60:1, 30:1) gave **2g** as a pale yellow oil (31%; keto/enol = 15/85). IR (neat) 3059, 1762, 1602, 1516, 1274, 821, 759 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) keto-form: δ 7.64 (d, *J* = 7.3 Hz, 1 H), 7.53-7.47 (m, 1 H), 7.41-7.37 (m, 2 H), 7.25-7.18 (m, 4 H), 3.40 (s, 2 H), 2.42 (s, 3 H), 2.02 (s, 3 H) ppm; enol-form**:** δ 15.59 (s, 1 H), 7.64 (d, *J* = 7.3 Hz, 1 H), 7.53-7.47 (m, 1 H), 7.41-7.37 (m, 2 H), 7.25-7.18 (m, 4 H), 5.41 (s, 1 H), 2.39 (s, 3 H), 1.94 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) keto-form: δ 190.7, 188.8, 141.0, 137.9, 137.2,

136.1, 131.2, 130.6, 129.7, 128.6, 128.5, 127.4, 57.5, 24.9, 21.2 ppm; enol-form: δ 190.7, 188.8, 141.0, 137.9, 137.2, 136.1, 130.7, 130.6, 129.0, 128.8, 128.8, 127.1, 102.2, 24.9, 21.2 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{16}O_2$: 252.1150; found: 252.1141.

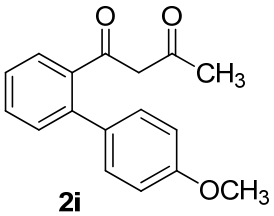
**1-(4'-Fluoro-[1,1'-biphenyl]-2-yl)butane-1,3-dione (2h)**




**2h**

Following the typical procedure, compound **2h** was prepared from **1c**. Chromatographic purification (hexane-ethyl acetate 80:1, 50:1, 20:1) gave **2h** as a white solid (30%; keto/enol = 8/92). IR (neat) 3061, 1607, 1515, 1480, 1223, 838, 817, 762 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) keto-form: δ 7.65 (d, *J* = 7.7 Hz, 1 H), 7.50 (dd, *J* = 7.7, 7.5 Hz, 1 H), 7.42 (dd, *J* = 7.6, 7.5 Hz, 1 H), 7.36 (d, *J* = 7.5 Hz, 1 H), 7.31 (dd, *J* = 8.6 Hz, $J_{H-F}$ = 5.6 Hz, 2 H), 7.08 (dd, *J* = 8.6 Hz, $J_{H-F}$ =8.6 Hz, 2 H), 3.46 (s, 2 H), 2.07 (s, 3 H) ppm; enol-form: δ 15.61 (s, 1 H), 7.65 (d, *J* = 7.7 Hz, 1 H), 7.50 (dd, *J* = 7.7, 7.5 Hz, 1 H), 7.42 (dd, *J* = 7.6, 7.5 Hz, 1 H), 7.36 (d, *J* = 7.5 Hz, 1 H), 7.31 (dd, *J* = 8.6 Hz, $J_{H-F}$ = 5.6 Hz, 2 H), 7.08 (dd, *J* = 8.6 Hz, $J_{H-F}$ = 8.6 Hz, 2 H), 5.40 (s, 1 H), 1.96 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) keto-form: δ 191.1, 188.4, 162.4 (d, $J_{C-F}$ = 245.3 Hz), 139.9, 136.8 (d, $J_{C-F}$ = 3.3 Hz), 136.1, 131.4, 130.6, 130.4 (d, $J_{C-F}$ = 9.1 Hz), 128.6, 127.8, 115.9 (d, $J_{C-F}$ = 20.9 Hz), 57.4, 20.5 ppm; enol-form: δ 191.1, 188.4, 162.4 (d, $J_{C-F}$ = 245.3 Hz), 139.9, 136.8 (d, $J_{C-F}$ = 3.3 Hz), 136.1, 130.7, 130.7, 130.5 (d, $J_{C-F}$ = 8.0 Hz), 128.9, 127.5, 115.3 (d, $J_{C-F}$ = 21.4 Hz), 102.2, 25.0 ppm; $^{19}F$ NMR (376 MHz, $CDCl_3$) keto-form: δ -113.6 ppm; enol-form: -115.0 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{13}F\,O_2$: 256.0900; found: 256.0905.

**1-(4'-Methoxy-[1,1'-biphenyl]-2-yl)butane-1,3-dione (2i)**

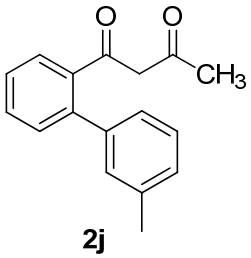



**2i**

Following the typical procedure, compound **2i** was prepared from **1d**. Chromatographic purification (hexane-ethyl acetate 40:1, 30:1, 20:1) gave **2i** as a yellow oil (18%; keto/enol = 6/94). IR (neat) 3061, 1760, 1610, 1517, 1295, 1249, 1179, 1036, 834, 763 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) keto-form: δ 7.63 (d, *J* = 7.6 Hz, 1 H), 7.52-7.46 (m, 1 H), 7.40-7.36 (m, 2 H), 7.31-7.28 (m, 2 H), 6.92 (d, *J* = 8.6 Hz, 2 H), 3.85 (s, 3 H), 3.39 (s, 2 H), 1.72 (s, 3 H) ppm; enol-form: δ 15.63 (s, 1 H),

7.63 (d, $J$ = 7.6 Hz, 1 H), 7.52-7.46 (m, 1 H), 7.40-7.36 (m, 2 H), 7.31-7.28 (m, 2 H), 6.92 (d, $J$ = 8.6 Hz, 2 H), 5.39 (s, 1 H), 3.85 (s, 3 H), 1.95 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) keto-form: δ 190.6, 189.1, 159.2, 140.6, 136.0, 133.1, 130.3, 129.9, 129.3, 128.9, 127.3, 113.6, 55.4, 55.3, 25.0 ppm; enol-form: δ 190.6, 189.1, 159.2, 140.6, 136.0, 133.1, 130.7, 130.6, 130.1, 128.8, 127.0, 113.8, 102.3, 55.3, 25.0 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{16}O_3$: 268.1099; found: 268.1092.

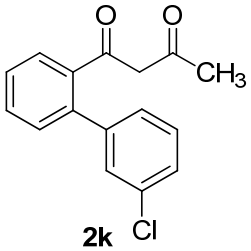
**1-(3'-Methyl-[1,1'-biphenyl]-2-yl)butane-1,3-dione (2j)**




**2j**

Following the typical procedure, compound **2j** was prepared from **1e**. Chromatographic purification (hexane-ethyl acetate 60:1, 30:1) gave **2j** as a yellow oil (20%; keto/enol = 11/89). IR (neat) IR (neat) 3061, 1764, 1728, 1689, 1604, 1290, 755, 700 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) keto-form: δ 7.58 (d, $J$ = 7.5 Hz, 1 H), 7.49 (dd, $J$ = 7.5, 7.4 Hz, 1 H), 7.28-7.25 (m, 3 H), 7.17 (s, 1 H), 7.15-7.12 (m, 2 H), 3.38 (s, 2 H), 2.41 (s, 3 H), 2.00 (s, 3 H) ppm; enol-form: δ 15.62 (s, 1 H), 7.65 (d, $J$ = 7.6 Hz, 1 H), 7.49 (dd, $J$ = 7.5, 7.4 Hz, 1 H), 7.43-7.34 (m, 3 H), 7.17 (s, 1 H), 7.15-7.12 (m, 2 H), 5.40 (s, 1 H), 2.38 (s, 3 H), 1.94 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) keto-form: δ 190.7, 188.7, 141.2, 140.7, 137.9, 136.0, 131.5, 131.3, 130.0, 128.9, 128.5, 127.6, 126.9, 126.0, 57.3, 25.5, 21.4 ppm; δ 190.7, 188.7, 141.2, 140.7, 137.9, 136.0, 130.8, 130.6, 129.6, 128.8, 128.2, 128.2, 127.3, 126.1, 102.2, 24.9, 21.5 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{16}O_2$: 252.1150; found: 252.1147.

**1-(3'-Chloro-[1,1'-biphenyl]-2-yl)butane-1,3-dione (2k)**

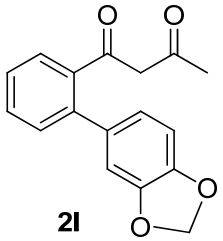



**2k**

Following the typical procedure, compound **2k** was prepared from **1f**. Chromatographic purification (hexane-ethyl acetate 80:1, 60:1, 30:1) gave **2k** as a pale yellow oil (45%; keto/enol = 6/94). IR (neat) 3062, 1760, 1595, 1567, 1286, 791, 757, 694 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) keto-form: δ 7.65 (d, $J$ = 7.7 Hz, 1 H), 7.53-7.49 (m, 1 H), 7.46-7.42 (m, 1 H), 7.37 (s, 1 H), 7.34-7.28 (m, 3 H), 7.22-7.20 (m, 1 H), 3.50 (s, 2 H), 2.09 (s, 3 H) ppm; enol-form: δ 15.60 (s, 1 H), 7.65 (d, $J$ = 7.7

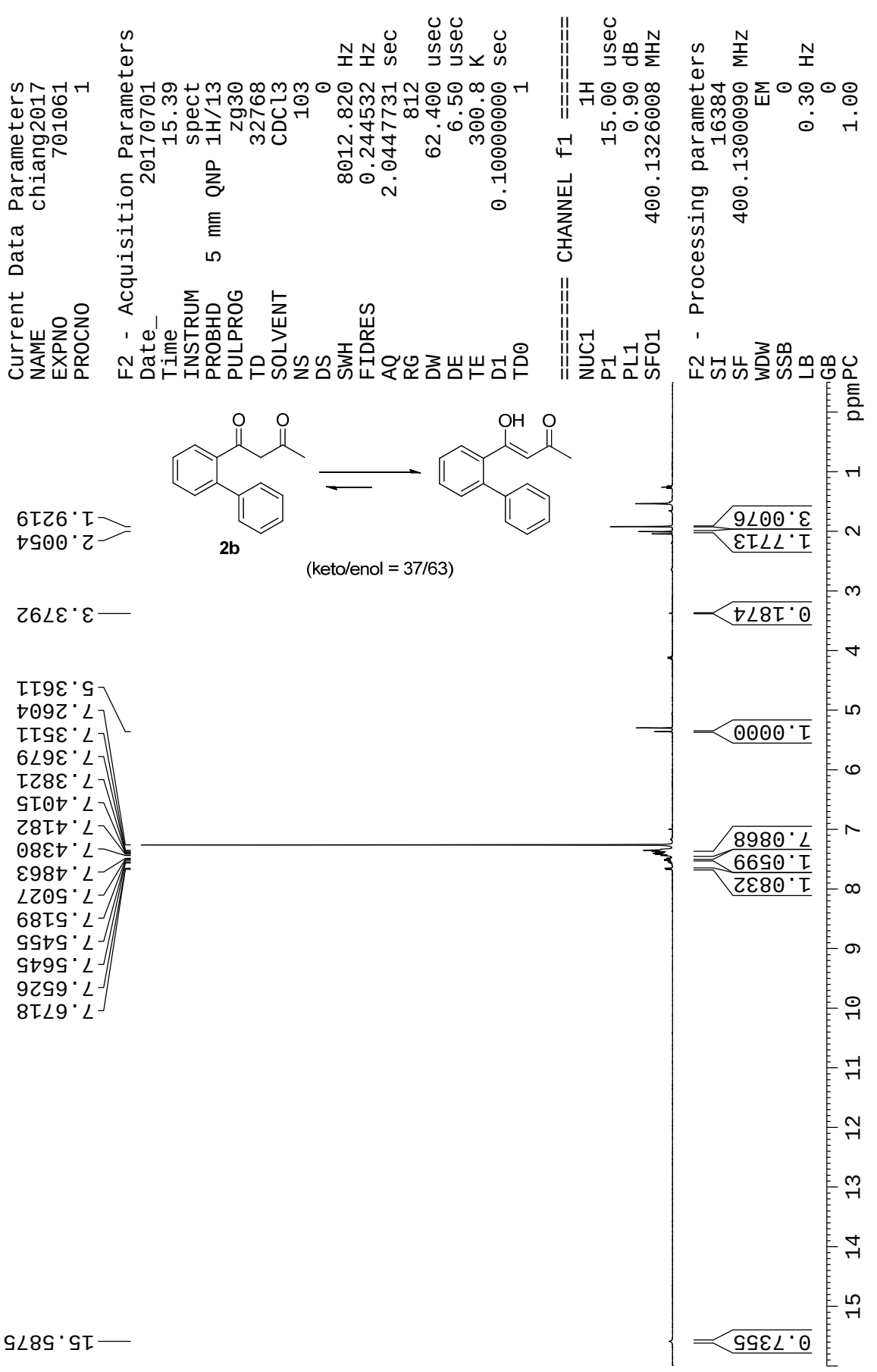
Hz, 1 H), 7.53-7.49 (m, 1 H), 7.46-7.42 (m, 1 H), 7.37 (s, 1 H), 7.34-7.28 (m, 3 H), 7.22-7.20 (m, 1 H), 5.46 (s, 1 H), 1.99 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) keto-form: δ 191.4, 187.9, 142.6, 139.5, 136.0, 134.2, 131.5, 130.5, 130.1, 129.4, 129.3, 129.0, 128.7, 128.2, 57.4, 20.5 ppm; enol-form: δ 191.4, 187.9, 142.6, 139.5, 136.0, 134.2, 130.8, 130.7, 129.5, 128.9, 128.8, 127.9, 127.5, 127.3, 102.1, 25.1 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{13}ClO_2$: 272.0604; found: 272.0609.

**1-(2-(Benzo[*d*][1,3]dioxol-5-yl)phenyl)butane-1,3-dione (2l)**



**2l**

Following the typical procedure, compound **2l** was prepared from **1g**. Chromatographic purification (hexane-ethyl acetate 60:1, 30:1, 20:1) gave **2l** as a yellow oil (31%; enol-form > 99%). IR (neat) 3061, 1687, 1605, 1221, 1038, 812, 761 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) enol-form: δ 15.63 (s, 1 H), 7.61 (dd, *J* = 7.8, 1.0 Hz, 1 H), 7.49-7.45 (m, 1 H), 7.32 (dd, *J* = 7.3, 1.4 Hz, 1 H), 7.37-7.34 (m, 1 H), 6.87-6.77 (m, 3 H), 6.00 (s, 2 H), 5.46 (s, 1 H), 1.99 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) enol-form: δ 190.6, 189.0, 147.6, 147.1, 140.5, 136.1, 134.7, 130.7, 130.6, 128.8, 127.2, 122.6, 109.4, 108.3, 102.1, 101.2, 25.0 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{14}O_4$: 282.0892; found: 282.0899.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(keto/enol = 37/63)
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7.5455
7.5189
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7.4380
7.4182
7.4015
7.3821
7.3679
7.3511
7.2604
5.3611
3.3792
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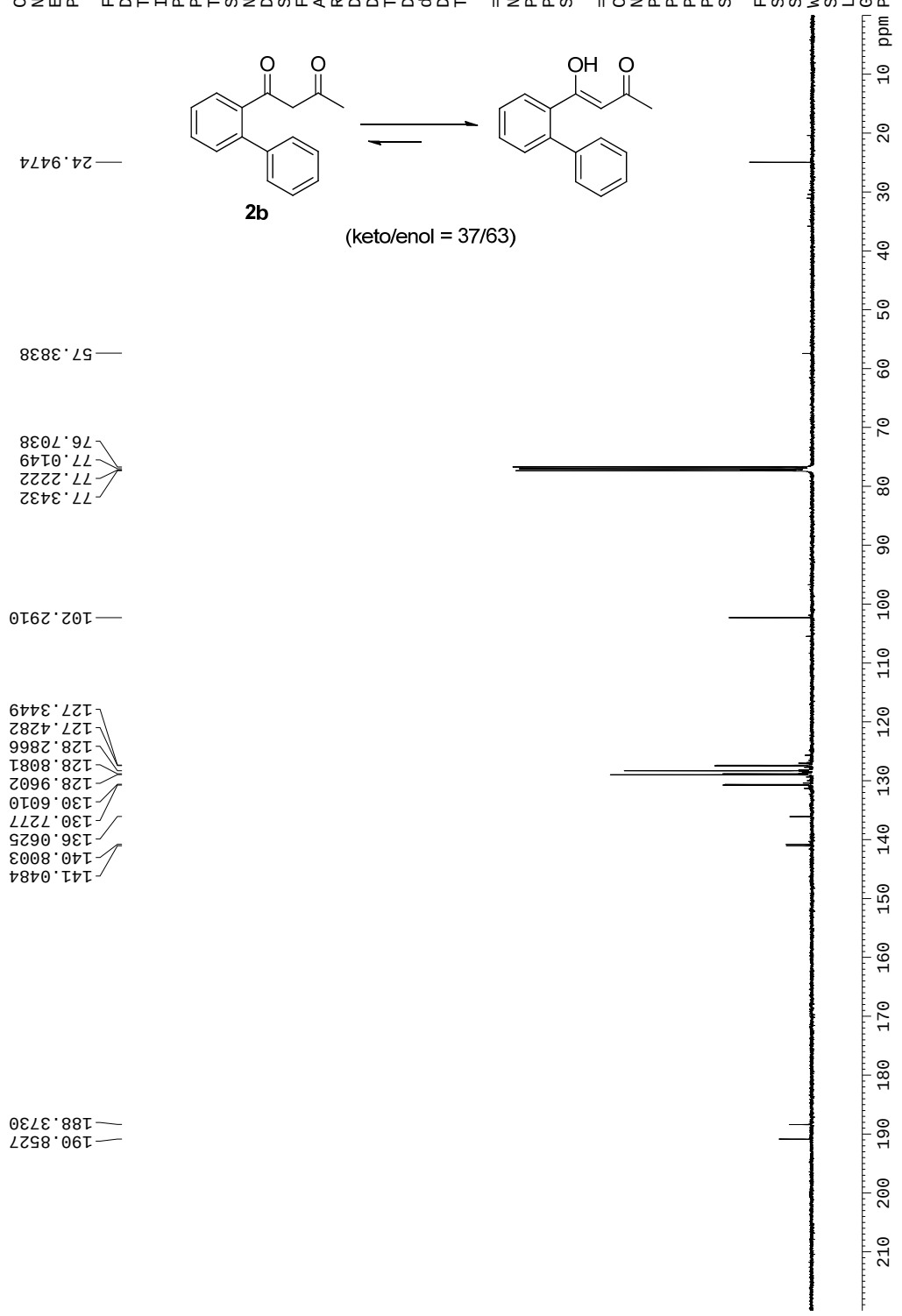
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d11 0.03000000 sec
DELTA 0.00000000 sec
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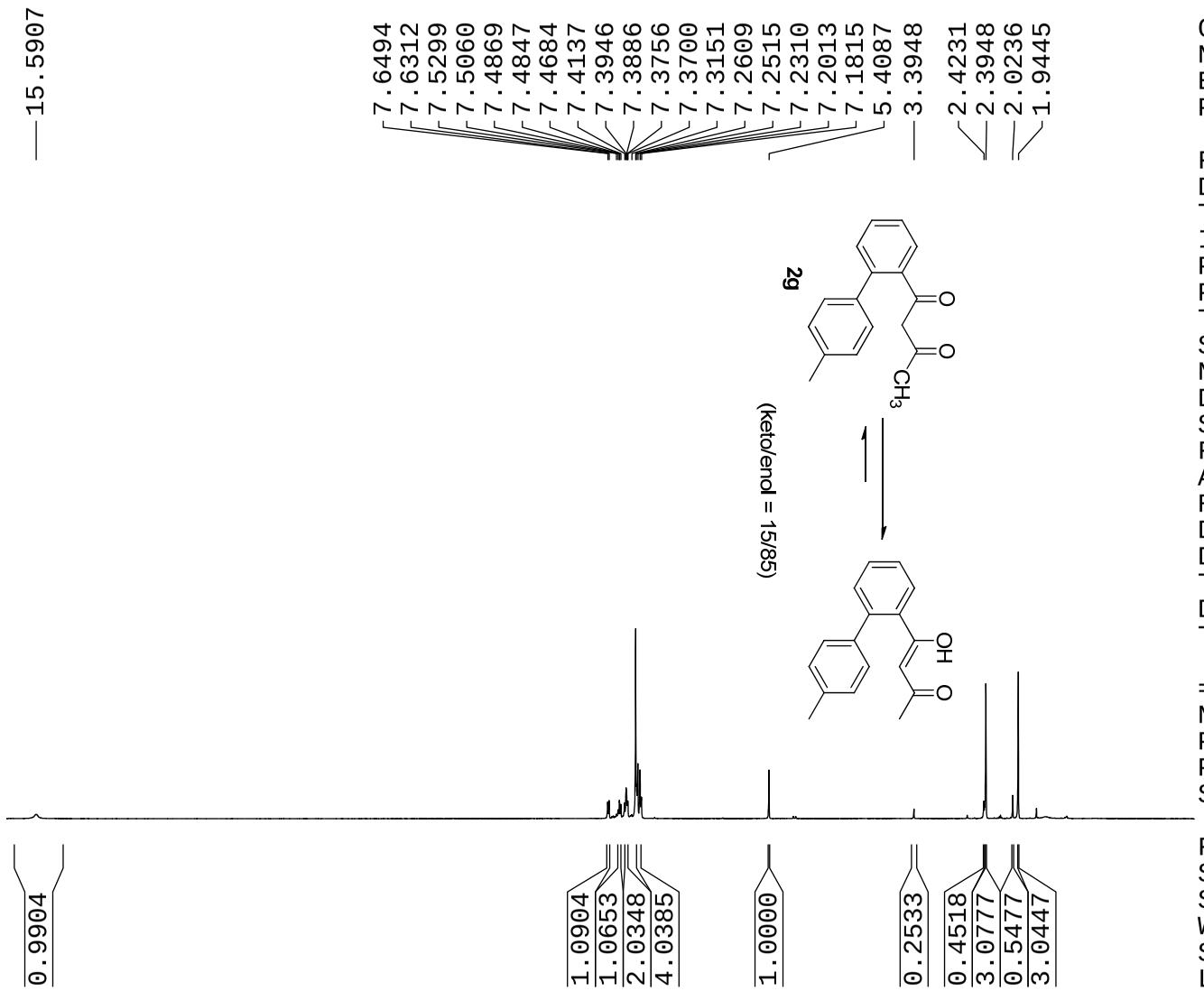
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.80 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
OH
O
2b
(keto/enol = 37/63)
24.9474
57.3838
76.7038
77.0149
77.2222
77.3432
102.2910
127.3449
127.4282
128.2866
128.8081
128.9602
130.6010
130.7277
136.0625
140.8003
141.0484
188.3730
190.8527
ppm

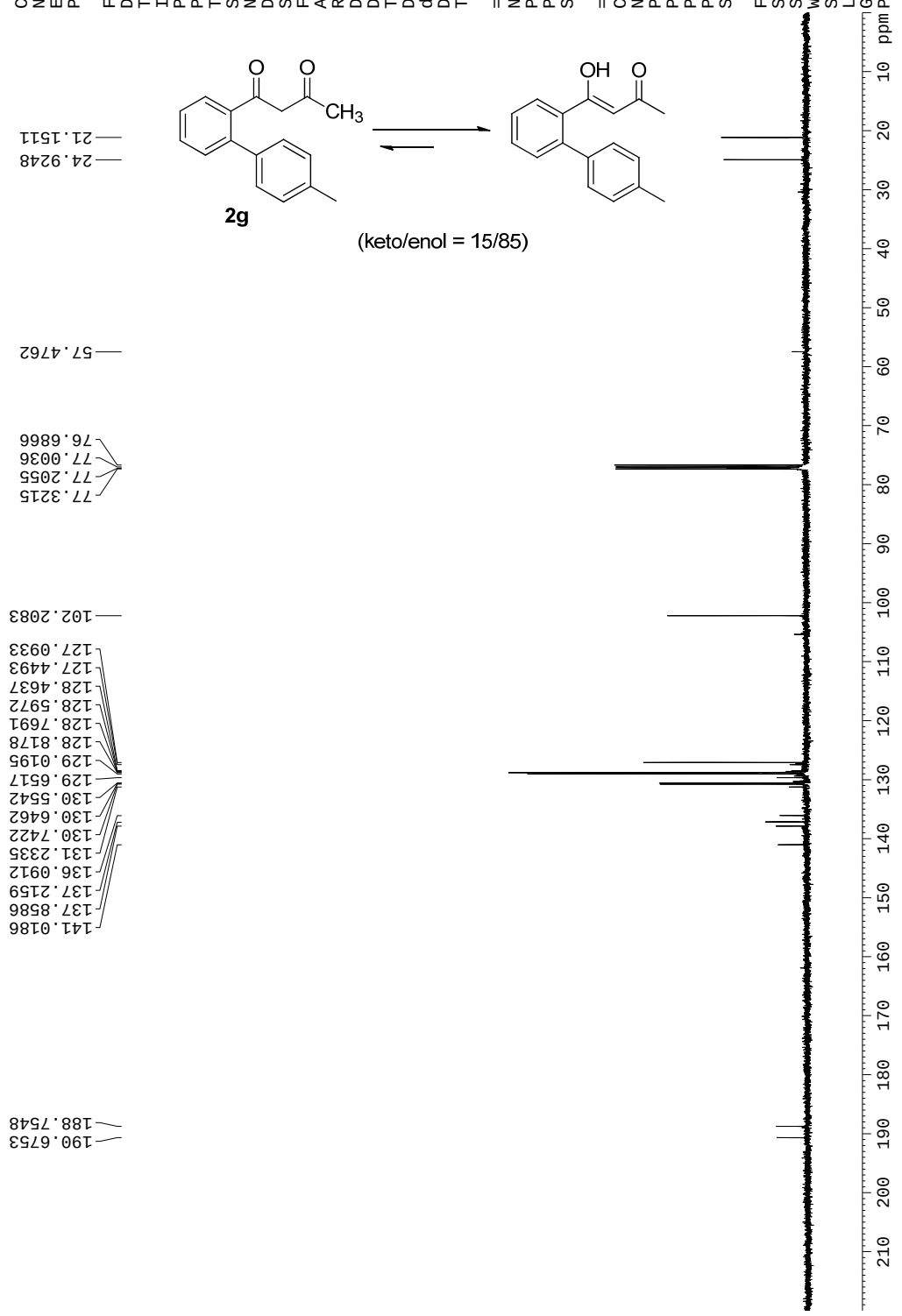


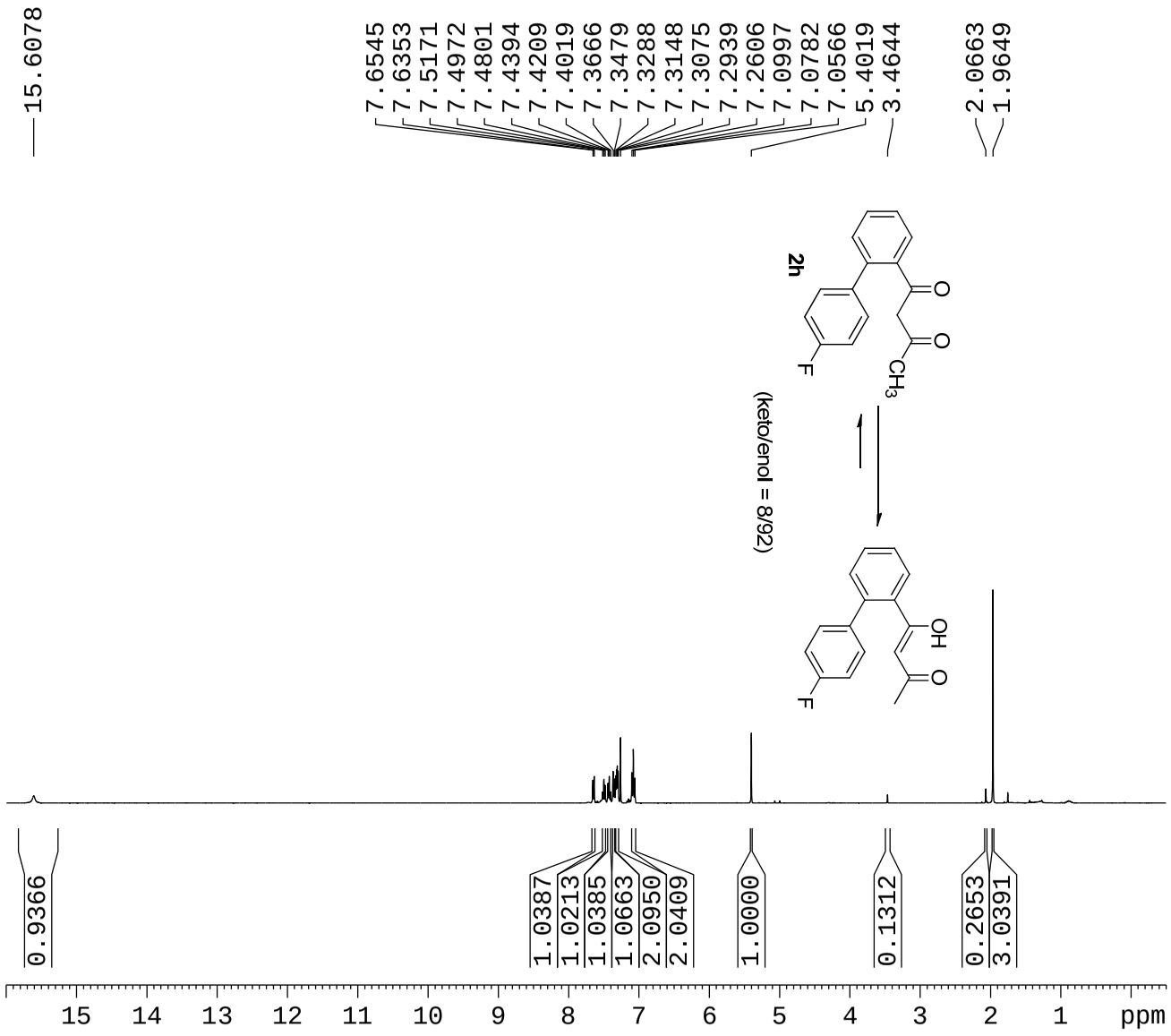

Current Data Parameters
NAME chiang2017
EXPNO 908031
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170908
Time 10.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 28
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 322
DW 62.400 usec
DE 6.50 usec
TE 306.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME chiang2017
EXPNO 908021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170908
Time 10.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 1701
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 306.9 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
CH3
OH
O
2g
(keto/enol = 15/85)
21.1511
24.9248
57.4762
76.6866
77.0036
77.2055
77.3215
102.2083
127.0933
127.4493
128.4637
128.5972
128.7691
128.8178
129.0195
129.6517
130.5542
130.6462
130.7422
131.2335
136.0912
137.2159
137.8586
141.0186
188.7548
190.6753
ppm

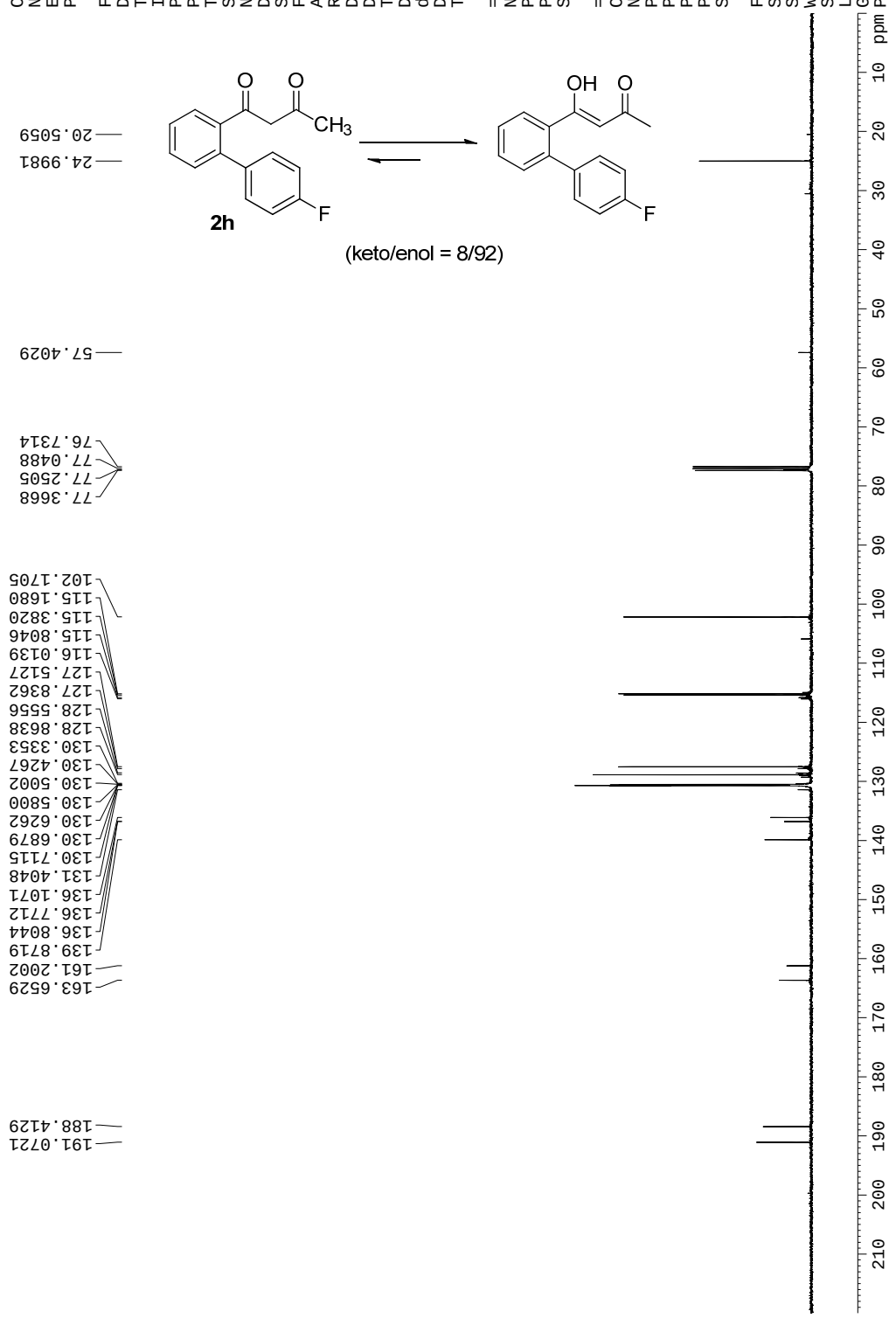


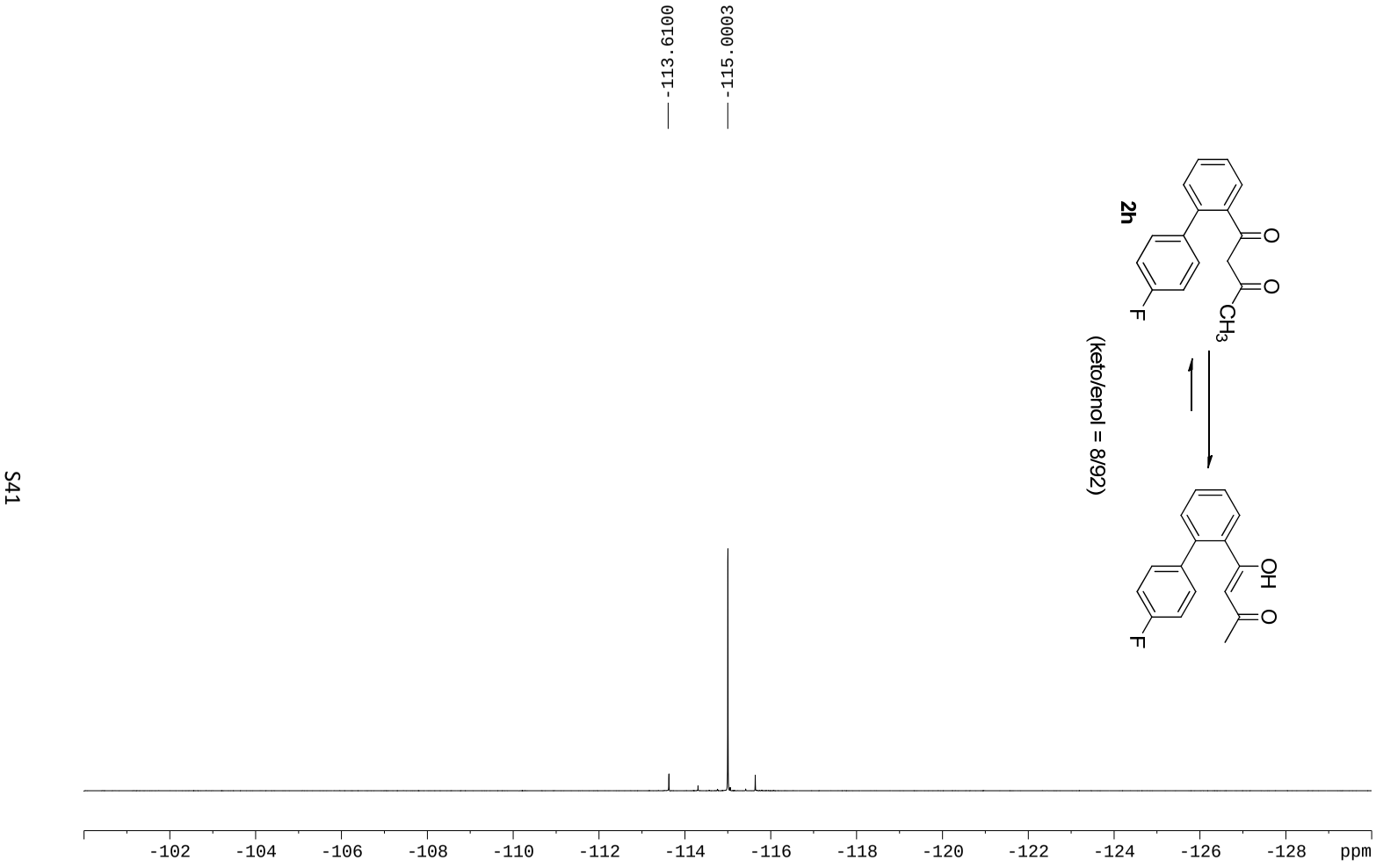

Current Data Parameters
NAME chiang2017
EXPNO 1021021
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171021
Time 10.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 21
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 287
DW 62.400 usec
DE 6.50 usec
TE 301.6 K
D1 2.00000000 sec
TD0 1

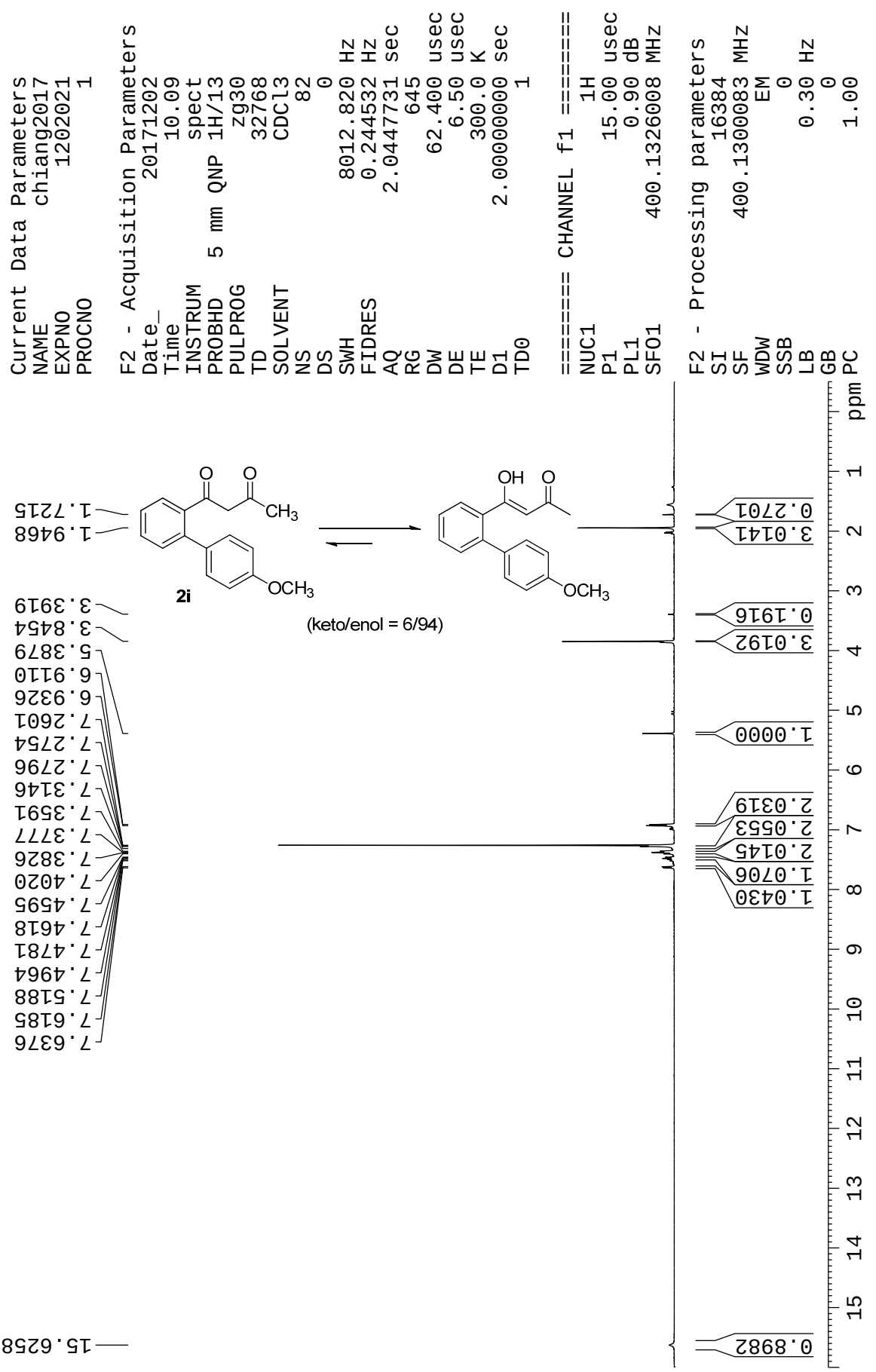
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

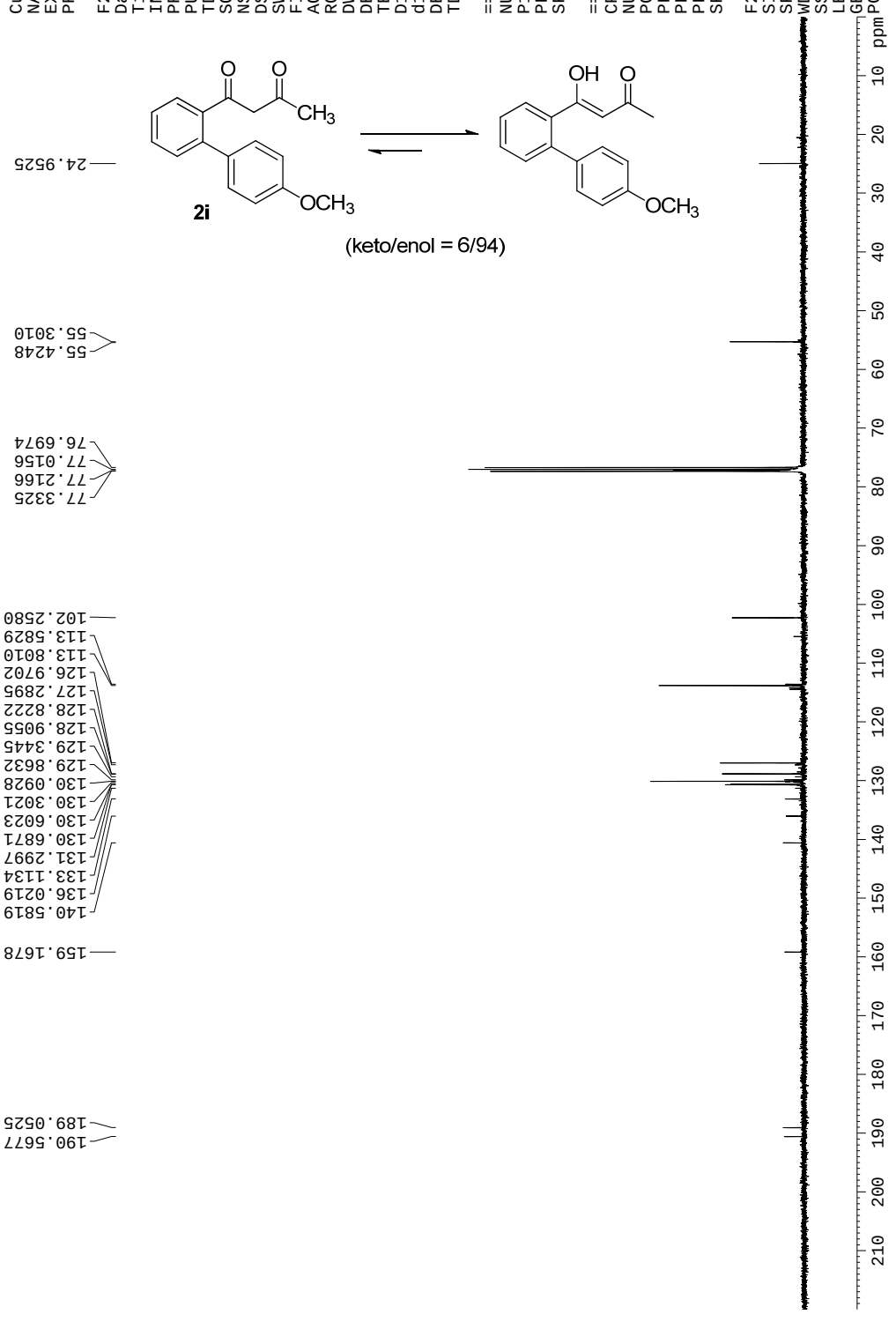
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

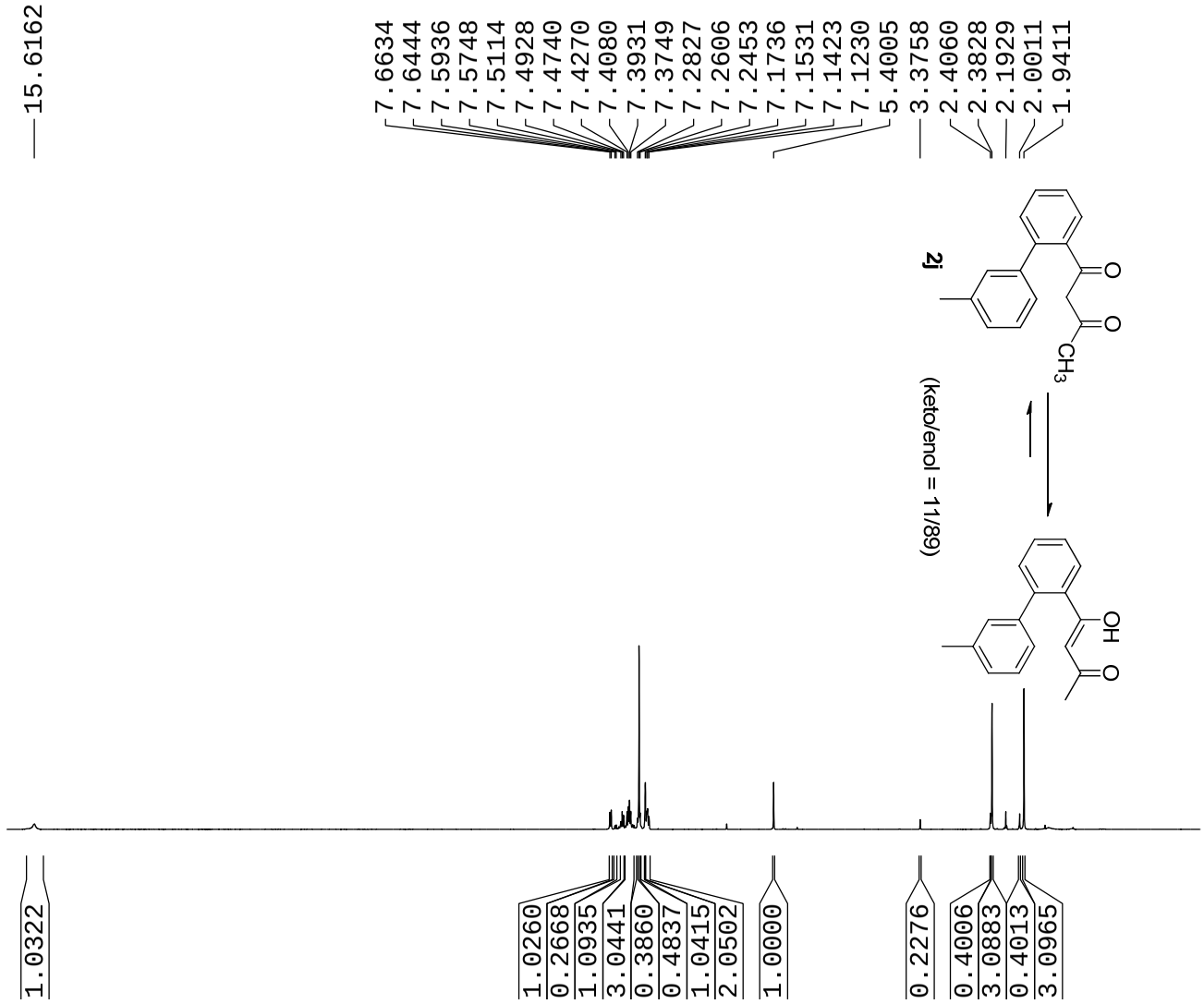

Current Data Parameters
NAME chiang2017
EXPNO 1021031
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171021
Time_ 10.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 1907
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 301.6 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.5059
24.9981
57.4029
76.7314
77.0488
77.2505
77.3668
102.1705
115.1680
115.3820
115.8046
116.0139
127.5127
127.8362
128.5556
128.8638
130.3353
130.4267
130.5002
130.5800
130.6262
130.6879
130.7115
131.4048
136.1071
136.7712
136.8044
139.8719
161.2002
163.6529
191.0721
188.4129
O
O
CH3
F
2h
OH
O
F
(keto/enol = 8/92)
ppm



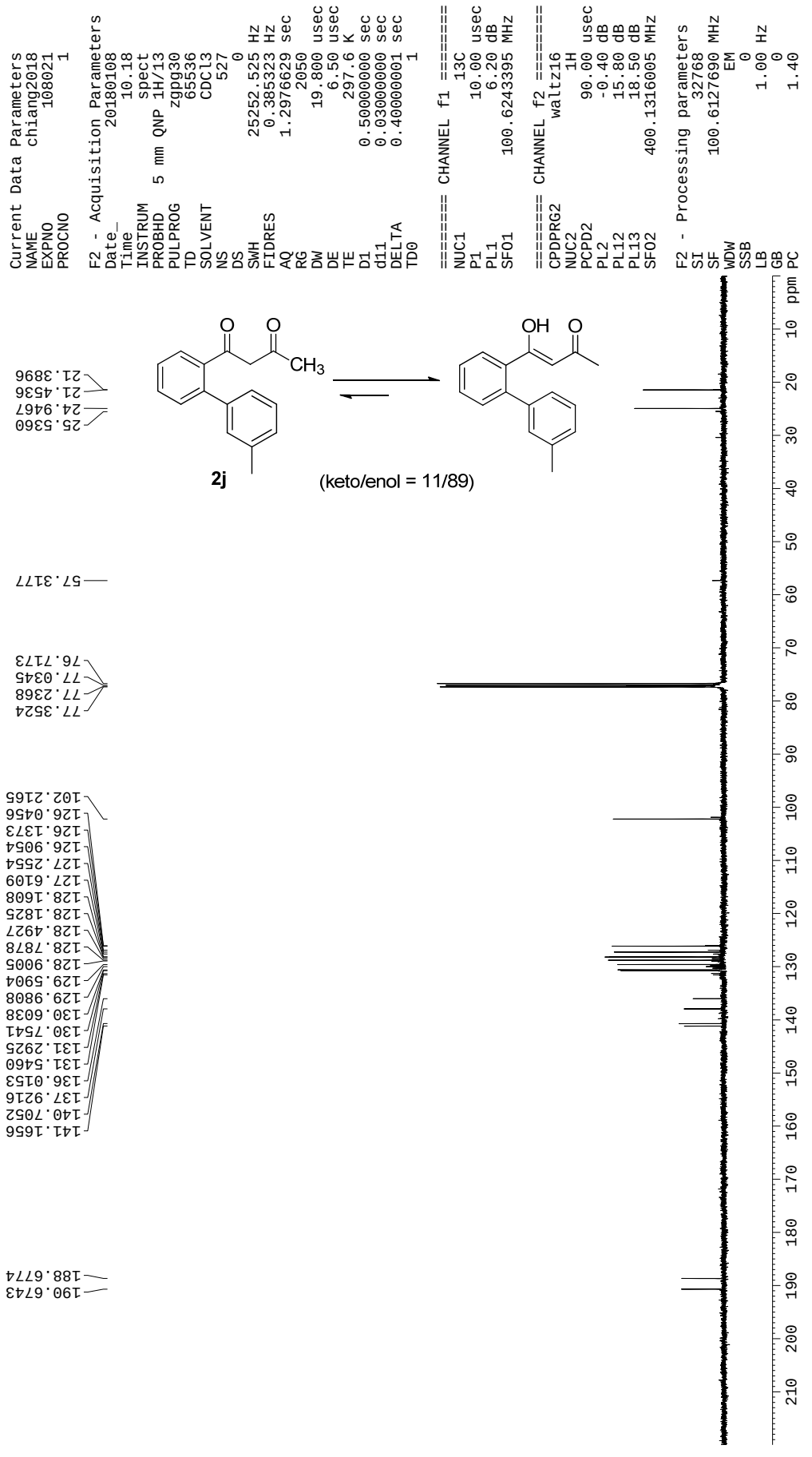

```
Current Data Parameters
NAME         chiang2017
EXPNO           1021041
PROCNO                1

F2 - Acquisition Parameters
Date_          20171021
Time              10.44
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG        zgfhigqn
TD              131072
SOLVENT            CDCl3
NS                    14
DS                     0
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ             0.7340532 sec
RG                  2050
DW                 5.600 usec
DE                 6.50 usec
TE                300.7 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 19F
P1                 20.00 usec
PL1                 2.50 dB
SFO1         376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF           376.4983660 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

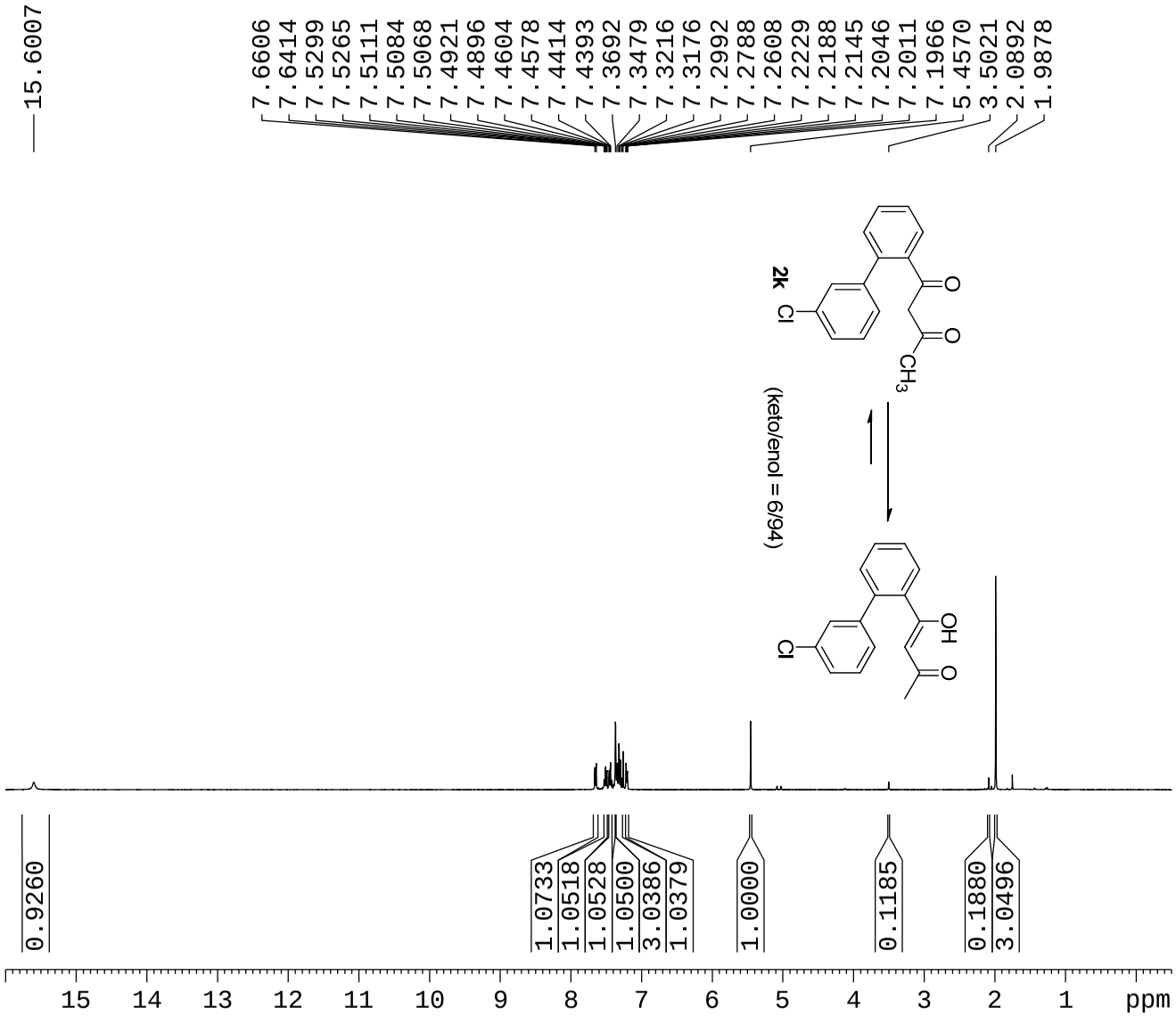

Current Data Parameters
NAME chiang2017
EXPNO 1202021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171202
Time 10.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 82
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 645
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2i
(keto/enol = 6/94)
1.7215
1.9468
3.3919
3.8454
5.3879
6.9110
6.9326
7.2601
7.2754
7.2796
7.3146
7.3591
7.3777
7.3826
7.4020
7.4595
7.4618
7.4781
7.4964
7.5188
7.6185
7.6376
15.6258
0.2701
3.0141
0.1916
3.0192
1.0000
2.0319
2.0553
2.0145
1.0706
1.0430
0.8982
ppm


Current Data Parameters
NAME chiang2017
EXPNO 1202031
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171202
Time 10.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 3053
DS 0
SWH 25252.525 Hz
FIDRES 1.0001100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 299.8 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
CH3
OCH3
2i
OH
O
OCH3
(keto/enol = 6/94)
24.9525
55.3010
55.4248
76.6974
77.0156
77.2166
77.3325
102.2580
113.5829
113.8010
126.9702
127.2895
128.8222
128.9055
129.3445
129.8632
130.0928
130.3021
130.6023
130.6871
131.2997
133.1134
136.0219
140.5819
159.1678
189.0525
190.5677
ppm

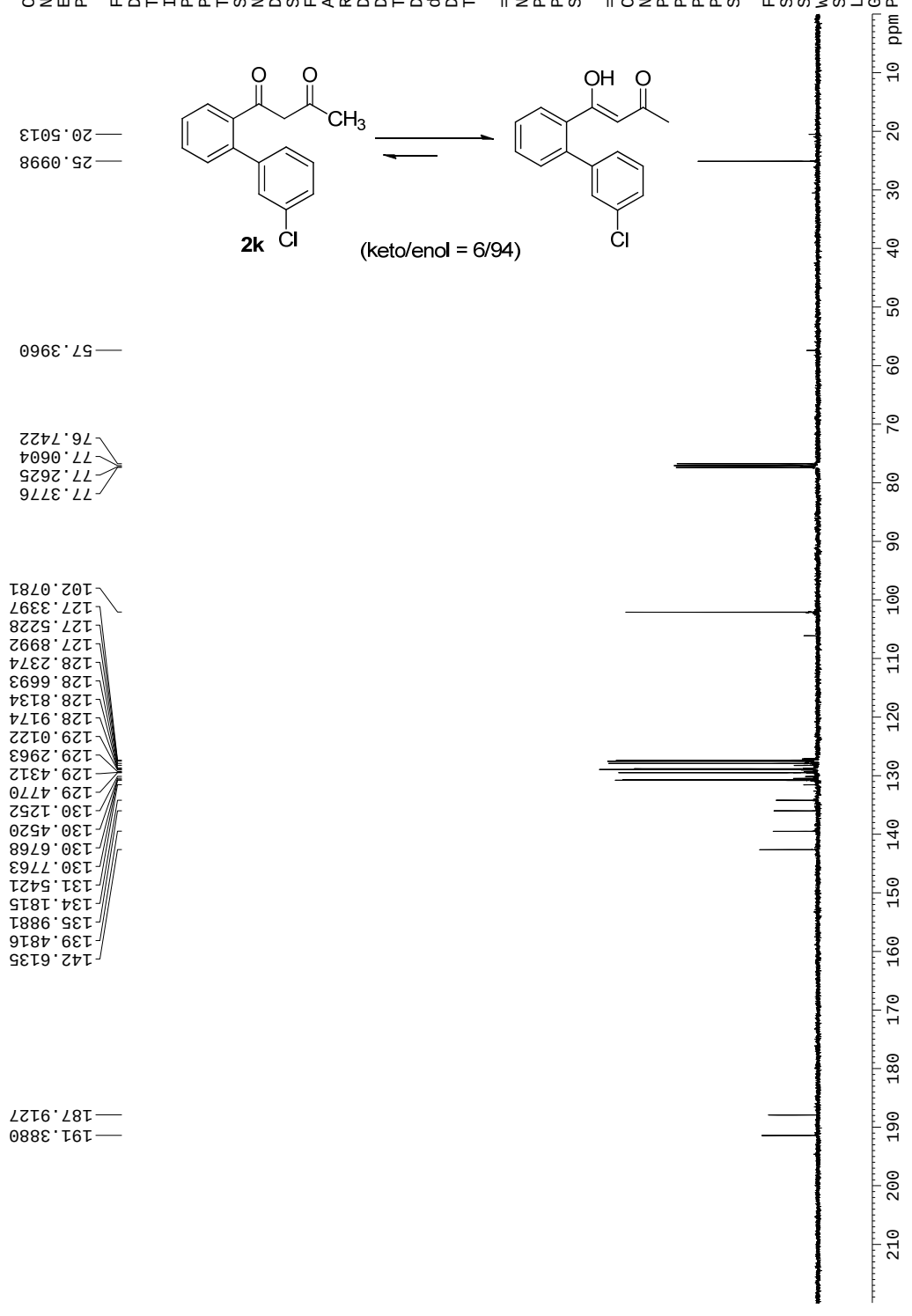


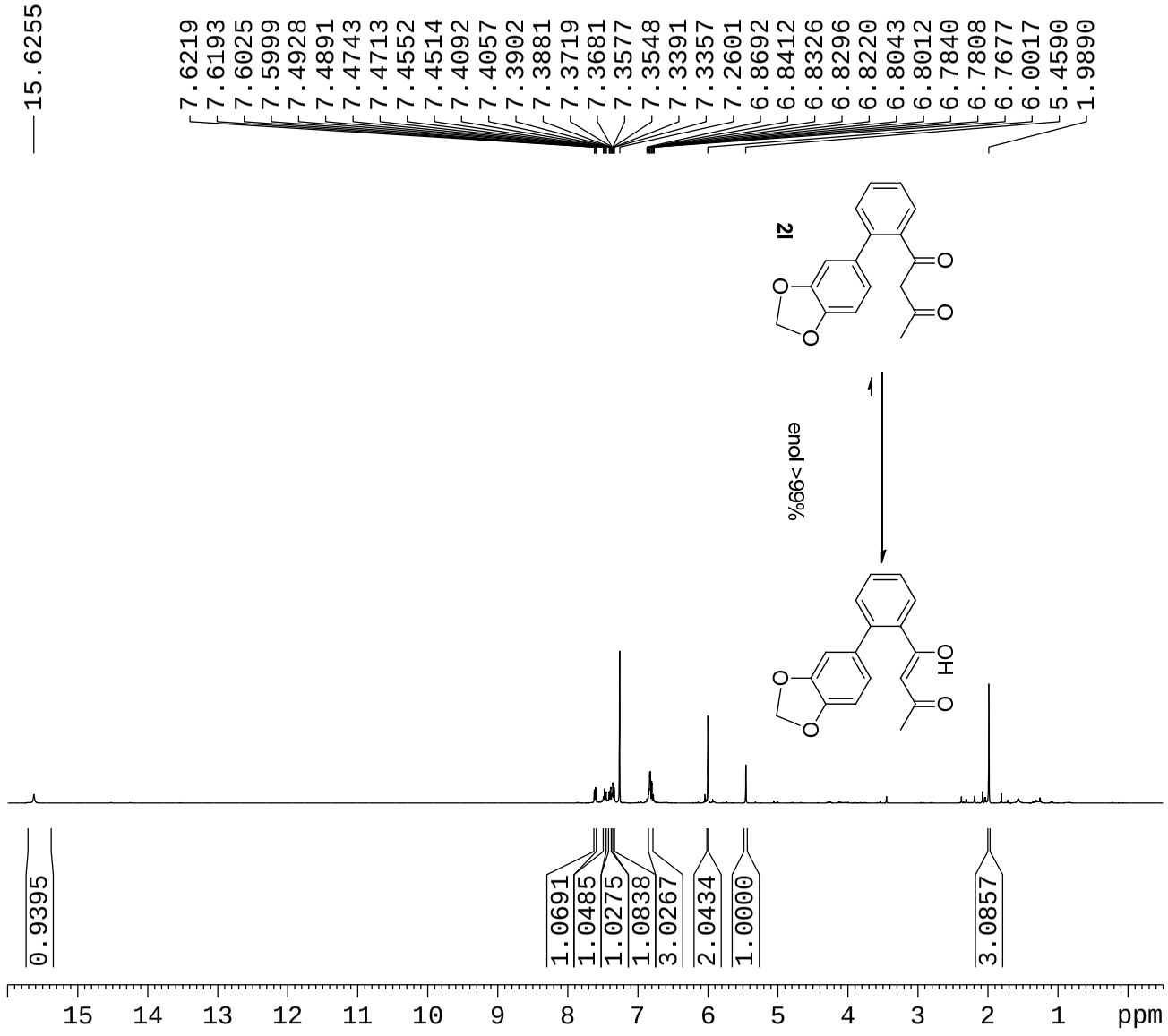

```
Current Data Parameters
NAME          chiang2018
EXPNO             108011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180108
Time               10.05
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    24
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              2.0447731 sec
RG                   362
DW                62.400 usec
DE                 6.50 usec
TE                297.4 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300081 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2018
EXPNO 108021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180108
Time 10.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 527
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.6 K
D1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000001 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
CH3
OH
O
2j
(keto/enol = 11/89)
21.3896
21.4536
24.9467
25.5360
57.3177
76.7173
77.0345
77.2368
77.3524
102.2165
126.0456
126.1373
126.9054
127.2554
127.6109
128.1608
128.1825
128.4927
128.7878
128.9005
129.5904
129.9808
130.6038
130.7541
131.2925
131.5460
136.0153
137.9216
140.7052
141.1656
188.6774
190.6743
ppm




```
Current Data Parameters
NAME          chiang2018
EXPNO             228011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180228
Time               10.22
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             2.0447731 sec
RG                   322
DW                62.400 usec
DE                 6.50 usec
TE                297.5 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300091 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```


Current Data Parameters
NAME chiang2018
EXPNO 228021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180228
Time 10.33
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 788
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.9 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.5013
25.0998
57.3960
76.7422
77.0604
77.2625
77.3776
102.0781
127.3397
127.5228
127.8992
128.2374
128.6693
128.8134
128.9174
129.0122
129.2963
129.4312
129.4770
130.1252
130.4520
130.6768
130.7763
131.5421
134.1815
135.9881
139.4816
142.6135
187.9127
191.3880
2k
(keto/enol = 6/94)
ppm




Current Data Parameters
NAME chiang2018
EXPNO 322011
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180322
Time 14.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 50
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 406
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
TD0 1

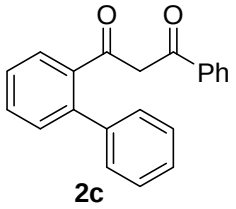
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME chiang2018
EXPNO 322021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180322
Time 14.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 1591
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.1 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
24.9844
76.7069
77.0244
77.2262
77.3419
101.1592
102.0965
108.2939
109.4172
122.6335
127.2003
128.8252
130.6083
130.6990
134.7180
136.1265
140.5245
147.1343
147.6029
188.9865
190.6142
2l
enol >99%
OH
O
ppm

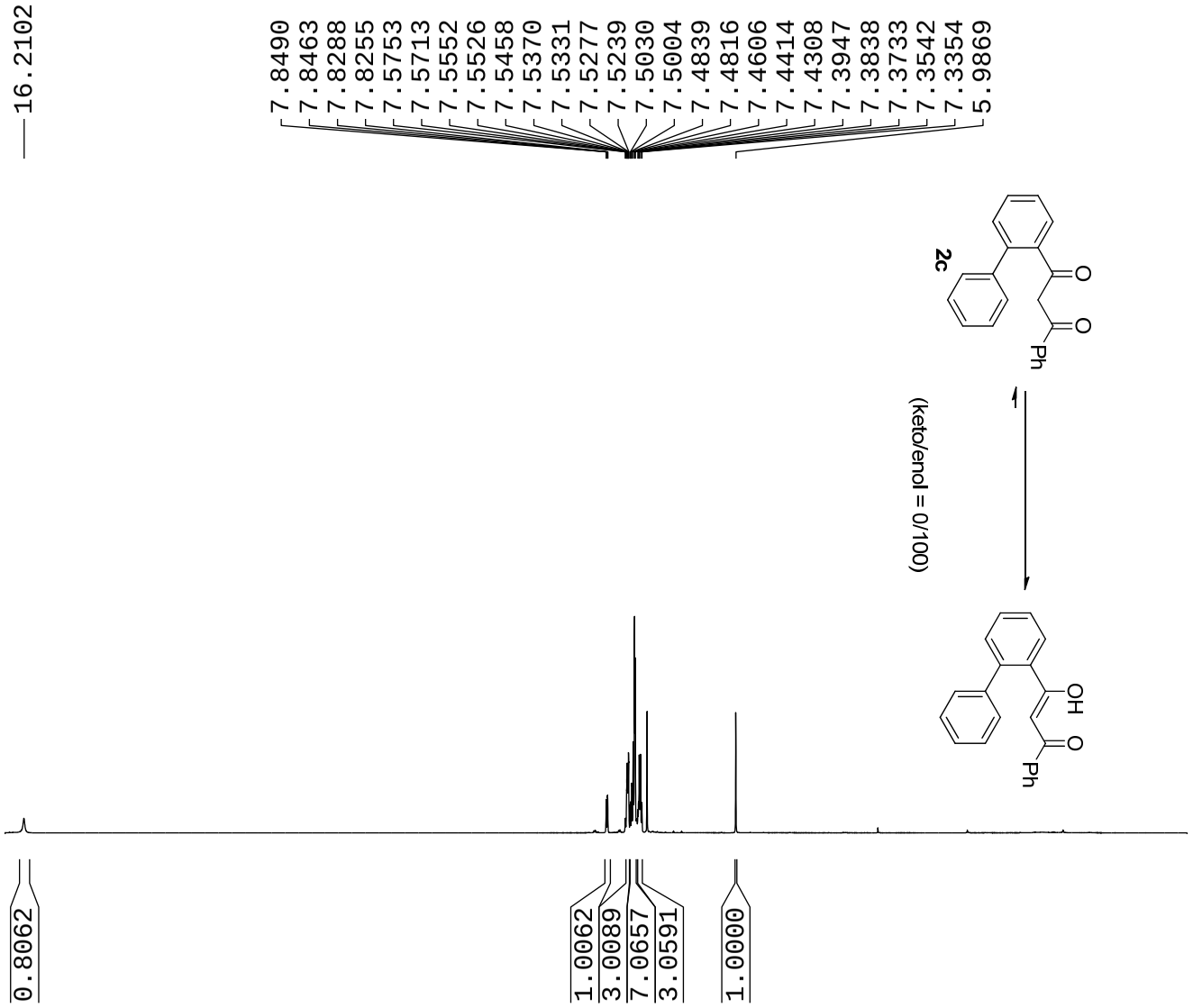
### iii) Preparation and NMR spectra of 2c

**1-([1,1′-Biphenyl]-2-yl)-3-phenylpropane-1,3-dione (2c)**




**2c**

To a stirred solution of **1a** (746.7 mg, 3.805 mmol) in toluene (30 mL) pre-cooled at 0 °C, LiHMDS (5.71 mL, 1 M in toluene, 5.71 mmol) was dropwise added over 10 min. After stirring at 0 °C for 10 min, benzoyl chloride (0.89 mL, 99%, 7.61 mmol) was added in one portion. The reaction mixture was then allowed to stir at rt for an additional 2 min, quenched by glacial acetic acid (3 mL) and diluted with ethyl acetate (500 mL). The solution was washed with water (150 mL) and brine (150 mL), and concentrated under reduced pressure. The crude residue was subjected to chromatography (hexane-ethyl acetate 80:1, 60:1, 30:1) to afford **2c** existing in its enol-form (792 mg, 69%, keto/enol = 0/100). IR (neat) 3060, 3027, 1601, 1571, 1218, 765, 746, 700, 609 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) enol-form: δ 16.21 (s, 1 H), 7.84 (dd, *J* = 8.2, 1.3 Hz, 1 H), 7.58-7.52(m, 3 H), 7.50-7.43 (m, 7 H), 7.39-7.34 (m, 3 H), 5.99 (s, 1 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) enol-form: δ 190.3, 182.6, 141.3, 141.1, 136.6, 135.0, 132.1, 130.9, 130.8, 129.2, 129.0, 128.5, 128.4, 127.6, 127.5, 127.0, 99.4 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{21}H_{16}O_2$: 300.1150; found: 300.1160.




```
Current Data Parameters
NAME          chiang2017
EXPNO             717031
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170717
Time               11.09
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    20
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             2.0447731 sec
RG                   181
DW                62.400 usec
DE                 6.50 usec
TE                303.6 K
D1           0.10000000 sec
TD0                    1

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PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
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SF          400.1300087 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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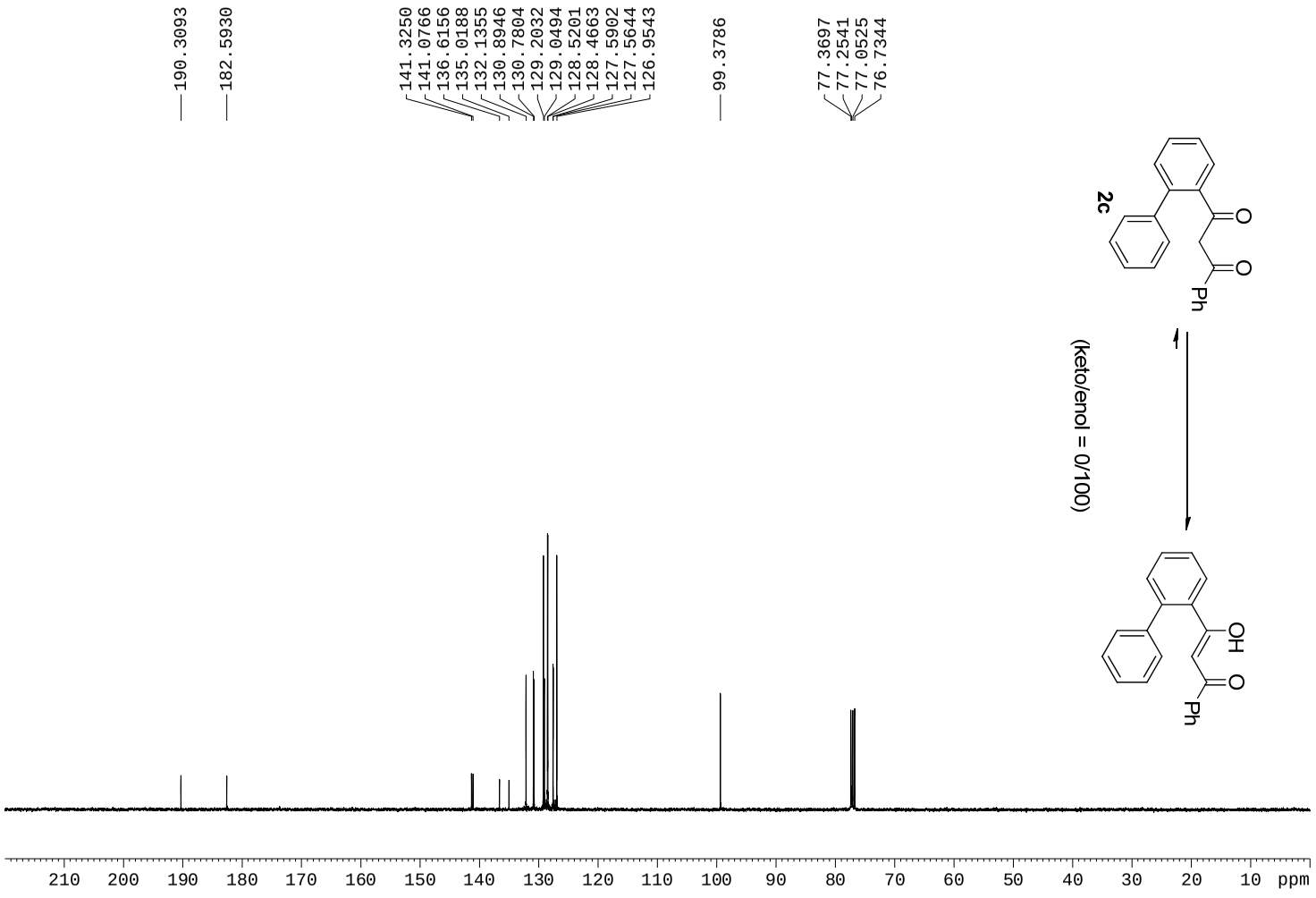


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EXPNO             717041
PROCNO                 1

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FIDRES         1.000100 Hz
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DELTA         0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
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NUC2                  1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF          100.6127690 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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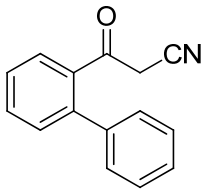
## iv) Preparation and NMR spectra of 2m-s

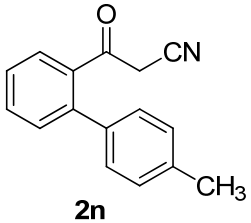
**3-([1,1'-Biphenyl]-2-yl)-3-oxopropanenitrile (2m)**[S5]




**2m**

Under $N_2$ protection, *n*-BuLi (4 mL, 2.0 M in cyclohexane, 7.98 mmol) was dropwise added to a stirred solution of diisopropylamine (0.94 mL, 6.65 mmol) in THF (14 mL) at 0 ºC over 5 min. After the mixture was stirred at 0 ºC for 3 min and then chilled to -80 ºC, a solution of **1a** (856 mg, 4.36 mmol) in THF (14 mL) was slowly added via a syringe over 10 minutes. Stirring was continued at -80 ºC for 25 min before the quick addition of *p*-TsCN (95%, 1.02 g, 5.32 mmol) dissolved in THF (7 mL). The reaction mixture was then allowed to stir at room temperature for 23 hrs, quenched by $H_2O$ (20 mL) and extracted with ethyl acetate (300 mL). The organic layer was separated and successively washed with saturated aqueous $NH_4Cl$ solution (50 mL x 2), $H_2O$ (50 mL) and brine (50 mL). After concentration, the crude reside was purified by flash chromatography (hexane-EtOAc 16:1, 10:1, 8:1) to afford 569.4 mg of **2m** (58%). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.64-7.57 (m, 2 H), 7.52-7.43 (m, 5 H), 7.37-7.33 (m, 2 H), 3.21 (s, 2 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 193.6, 140.9, 139.6, 137.4, 132.3, 130.5, 129.4, 129.4, 128.9, 128.9, 128.0, 113.7, 32.1 ppm.

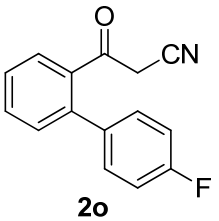
**3-(4'-Methyl-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (2n)**




**2n**

The titled compound was synthesized from **1b** by following the typical procedure. Flash chromatography (hexane-EtOAc 16:1, 8:1) gave **2n** as a pale yellow oil (62%). IR (neat) 3025, 2921, 2261, 1702, 1595, 824, 762 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.6 Hz, 1 H), 7.59 (t, *J* = 7.6 Hz, 1 H), 7.46 (dd, *J* = 7.6, 7.6 Hz, 1 H), 7.43 (d, *J* = 7.6 Hz, 1 H), 7.30 (d, *J* = 7.9 Hz, 2 H), 7.24 (d, *J* = 7.9 Hz, 2 H), 3.22 (s, 2 H), 2.44 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 193.7, 140.9, 139.0, 137.4, 136.6, 132.2, 130.4, 130.1, 128.8, 128.8, 127.7, 113.8, 32.1, 21.2 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{13}NO$: 235.0997; found: 235.0989.

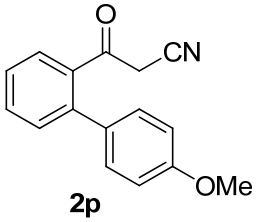
**3-(4'-Fluoro-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (2o)**




**2o**

The titled compound was synthesized from **1c** by following the typical procedure. Flash chromatography (hexane-EtOAc 15:1, 10:1, 5:1) gave **2o** as a white solid (86%). IR (neat) 3066, 2959, 2262, 1701, 1606, 1225, 841, 764, 720 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.64-7.57 (m, 2 H), 7.49 (dd, $J$ = 7.6, 7.6 Hz, 1 H), 7.41 (d, $J$ = 8.1 Hz, 1 H), 7.36-7.30 (m, 2 H), 7.19 (dd, $J_{H\text{-}F}$ = 8.5, $J$ = 8.5 Hz, 2 H), 3.28 (s, 2 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 193.3, 163.1 (d, $J_{C\text{-}F}$ = 248.2 Hz), 139.7, 137.2, 135.6 (d, $J_{C\text{-}F}$ = 3.4 Hz), 132.4, 130.6, 130.6 (d, $J_{C\text{-}F}$ = 8.1 Hz), 128.9, 128.1, 116.5 (d, $J_{C\text{-}F}$ = 21.5 Hz), 113.5, 32.2 ppm; $^{19}F$ NMR (376 MHz, $CDCl_3$) δ -112.3 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{15}H_{10}FNO$: 239.0746; found: 239.0742.

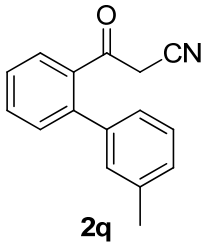
**3-(4'-Methoxy-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (2p)**




**2p**

The titled compound was synthesized from **1d** by following the typical procedure. Flash chromatography (hexane-EtOAc 10:1, 5:1, 3:1) gave **2p** as a yellow oil (49%). IR (neat) 3060, 2960, 2261, 1698, 1609, 1247, 837, 765 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 2 H), 7.47-7.40 (m, 2 H), 7.27 (d, $J$ = 8.6 Hz, 2 H), 7.02 (d, $J$ = 8.6 Hz, 2 H), 3.87 (s, 3 H), 3.23 (s, 2 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 193.9, 160.2, 140.5, 137.3, 132.3, 131.6, 130.4, 130.1, 128.8, 127.5, 114.9, 113.8, 55.4, 32.1 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{13}NO_2$: 251.0946; found: 251.0948.

**3-(3'-Methyl-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (2q)**

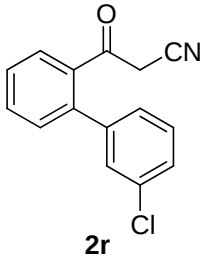



**2q**

The titled compound was synthesized from **1e** by following the typical procedure. Flash chromatography (hexane-EtOAc 16:1, 8:1) gave **2q** as a yellow oil (67%). IR (neat) 3060, 2958, 2261, 1699, 1595, 760, 706 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.61 (d, $J$ = 7.6 Hz, 1 H), 7.60 (dd, $J$ = 7.6, 7.6 Hz, 1 H), 7.46 (dd, $J$ = 7.6, 7.6 Hz, 1

H), 7.44 (d, *J* = 7.6 Hz, 1 H), 7.38 (dd, *J* = 7.6, 7.6 Hz, 1 H), 7.29 (d, *J* = 7.6 Hz, 1 H), 7.17 (s, 1 H), 7.15 (d, *J* = 7.6 Hz, 1 H), 3.20 (s, 2 H), 2.43 (s, 3 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 193.8, 141.0, 139.5, 139.4, 137.4, 132.3, 130.4, 129.7, 129.5, 129.3, 128.9, 127.9, 125.9, 113.8, 32.1, 21.4 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{13}NO$: 235.0997; found: 235.0990.

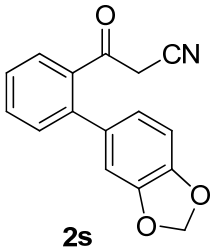
**3-(3'-Chloro-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (2r)**




**2r**

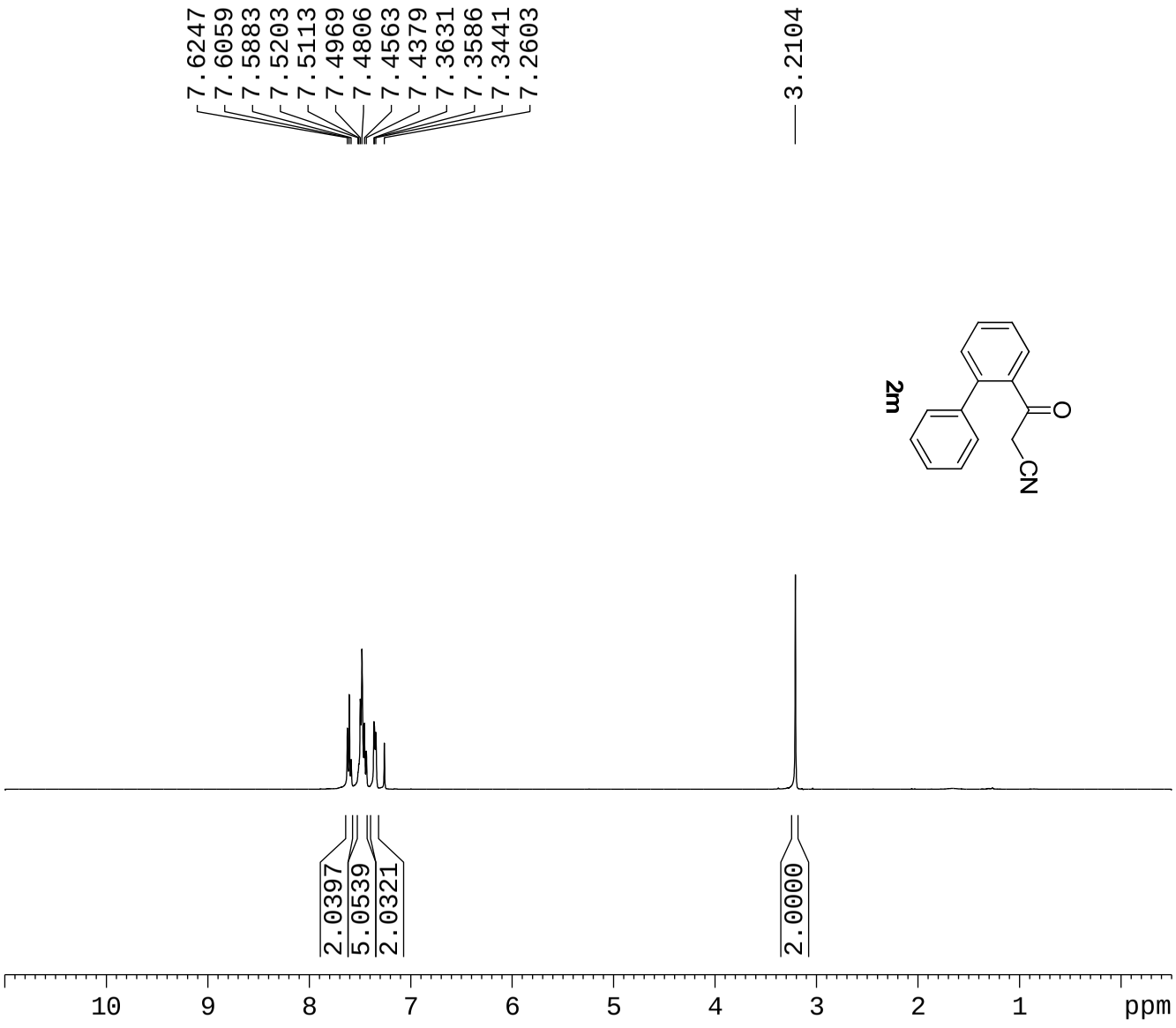
The titled compound was synthesized from **1f** by following the typical procedure. Flash chromatography (hexane-EtOAc 10:1, 5:1, 3:1) gave **2r** as a colorless oil (56%). IR (neat) 3063, 2957, 2262, 1702, 1593, 792, 761, 698 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.65-7.59 (m, 2 H), 7.50 (dd, *J* = 7.7, 7.7 Hz, 1 H), 7.47-7.37 (m, 4 H), 7.20 (d, *J* = 6.9 Hz, 1 H), 3.34 (s, 2 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 192.8, 141.4, 139.4, 137.0, 135.3, 132.5, 130.6, 130.5, 129.0, 128.9, 128.7, 128.5, 127.2, 113.5, 32.2 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{15}H_{10}ClNO$: 255.0451; found: 255.0456.

**3-(2-(Benzo[*d*][1,3]dioxol-5-yl)phenyl)-3-oxopropanenitrile (2s)**




**2s**

The titled compound was synthesized from **1g** by following the typical procedure. Flash chromatography (hexane-EtOAc 15:1, 8:1, 5:1) gave **2s** as a pale yellow oil (10%). IR (neat) 3064, 2909, 2261, 1699, 1595, 1224, 1038, 817, 764 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.60-7.55 (m, 2 H), 7.45 (ddd, *J* = 8.0, 7.1, 0.9 Hz, 1 H), 7.41 (d, *J* = 8.2 Hz, 1 H), 6.91 (d, *J* = 7.9 Hz, 1 H), 6.87 (d, *J* = 1.7 Hz, 1 H), 6.75 (dd, *J* = 7.9, 1.7 Hz, 1 H), 6.07 (s, 2 H), 3.31 (s, 2 H) ppm; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 193.6, 148.6, 148.4, 140.4, 137.4, 133.3, 132.3, 130.4, 128.9, 127.8, 122.9, 113.8, 109.1, 109.0, 101.7, 32.1 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{11}NO_3$: 265.0739; found: 265.0742.

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| SOLVENT | CDCl3 |
| NS | 12 |
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| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 101 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 303.5 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

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| WDW | EM |
| SSB | 0 |
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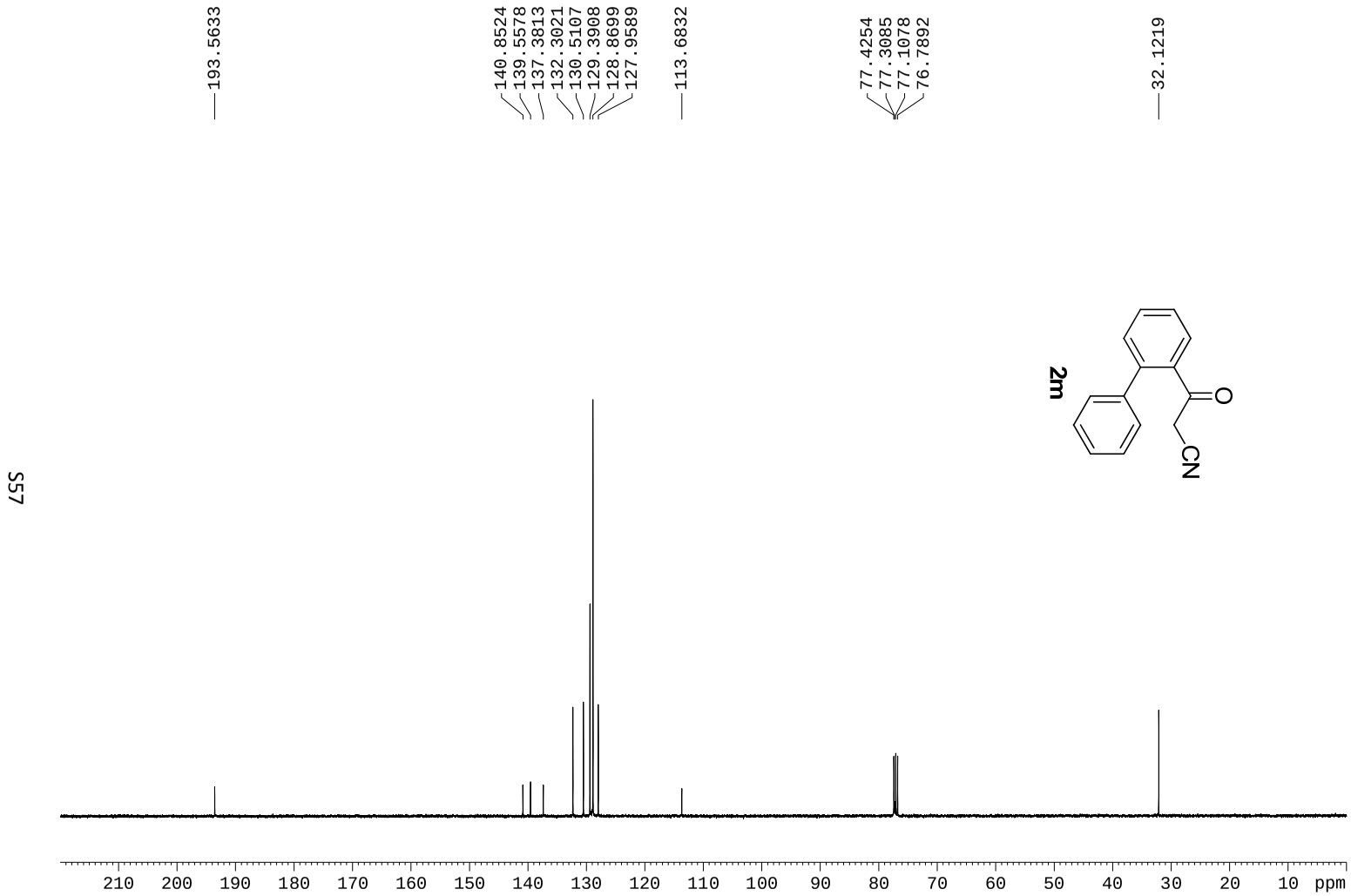


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SOLVENT           CDCl3
NS                  223
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                303.7 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                    1

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NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

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NUC2                 1H
PCPD2             90.00 usec
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PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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WDW                  EM
SSB                   0
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GB                    0
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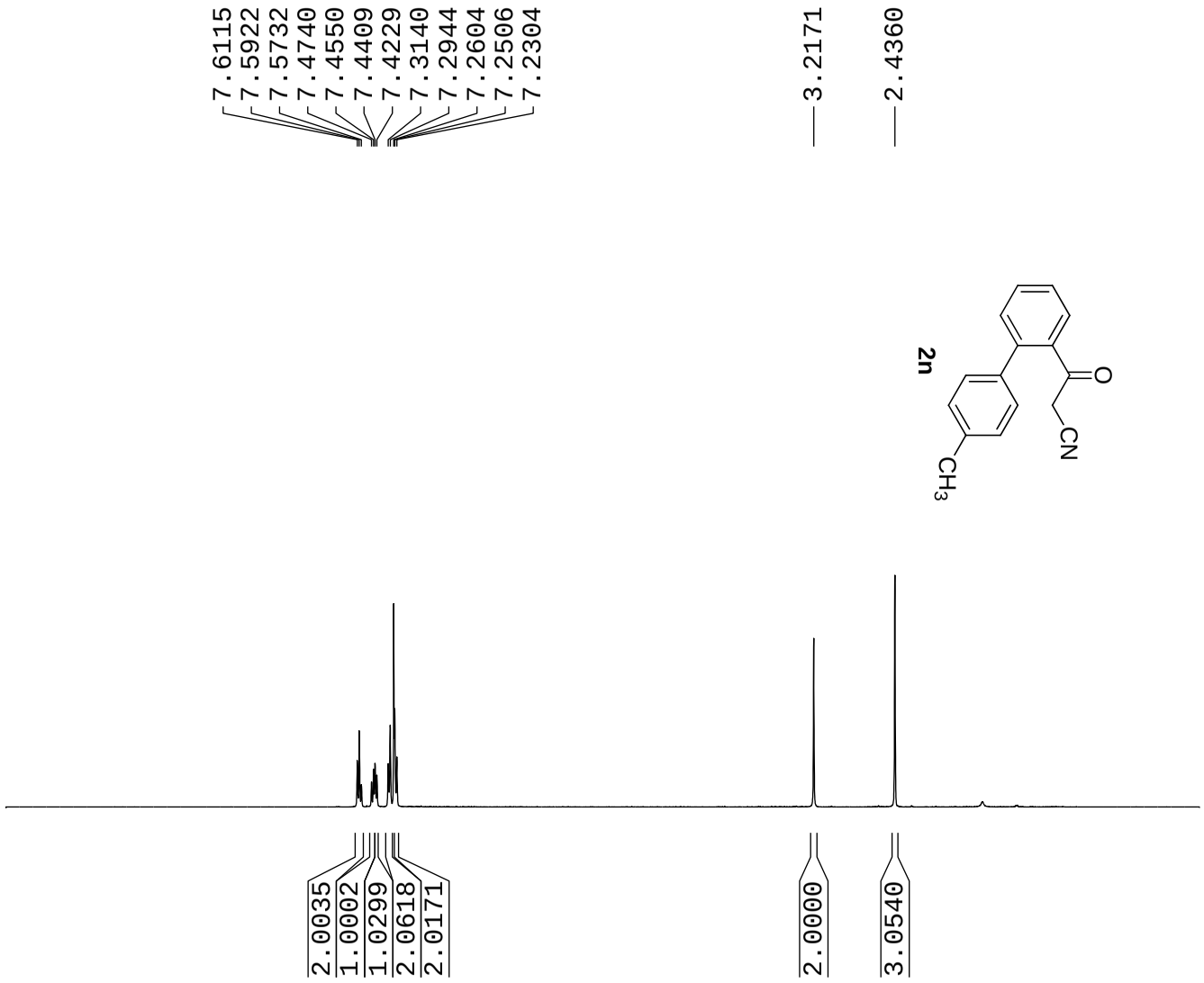


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EXPNO 1116011
PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 15
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 406
DW 83.200 usec
DE 6.50 usec
TE 301.2 K
D1 2.00000000 sec
TD0 1

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NUC1 1H
P1 15.00 usec
PL1 0.90 dB
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F2 - Processing parameters
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SF 400.1300082 MHz
WDW EM
SSB 0
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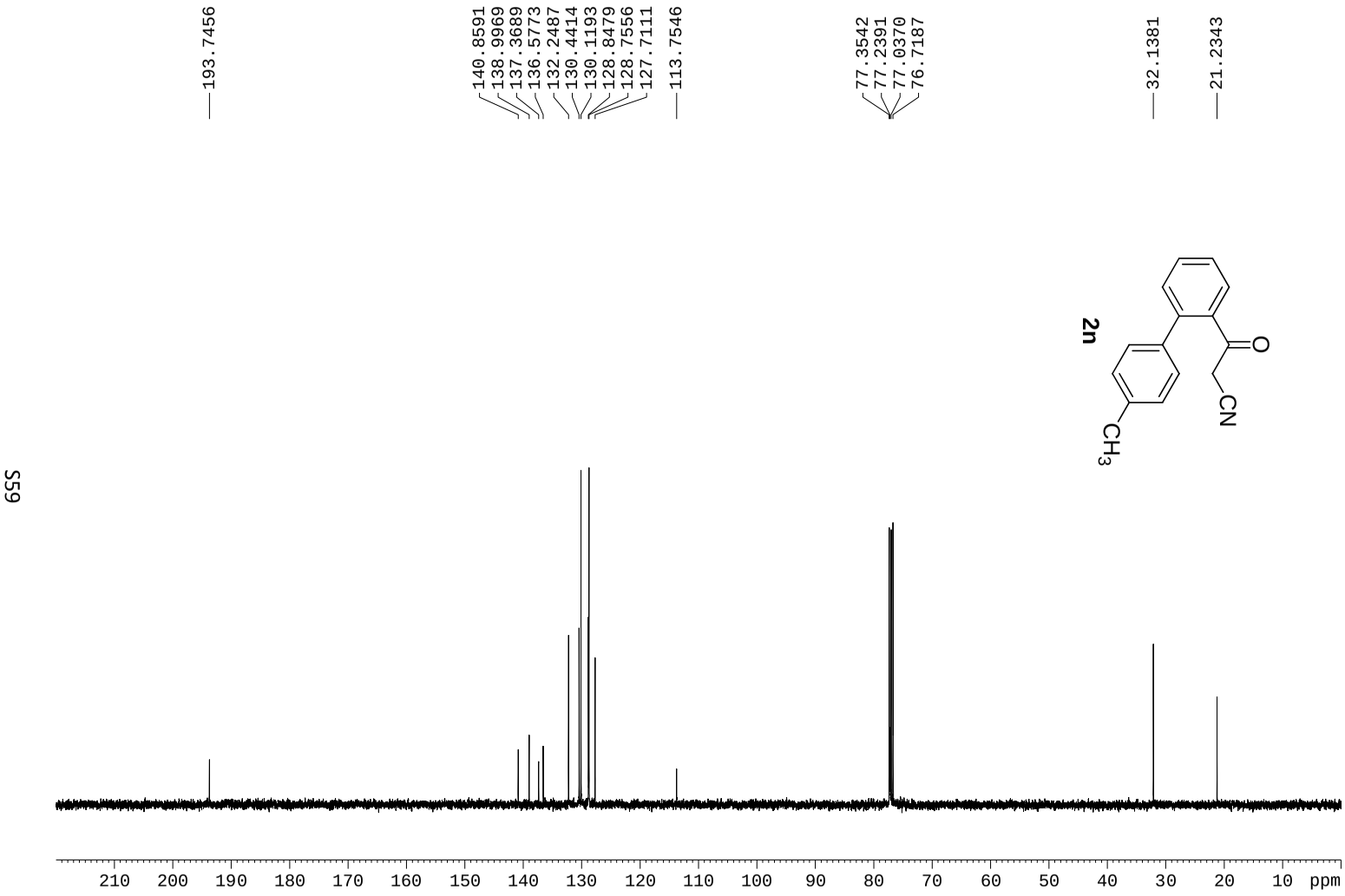
PC 1.00

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EXPNO            1116012
PROCNO                 1

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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  251
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                301.4 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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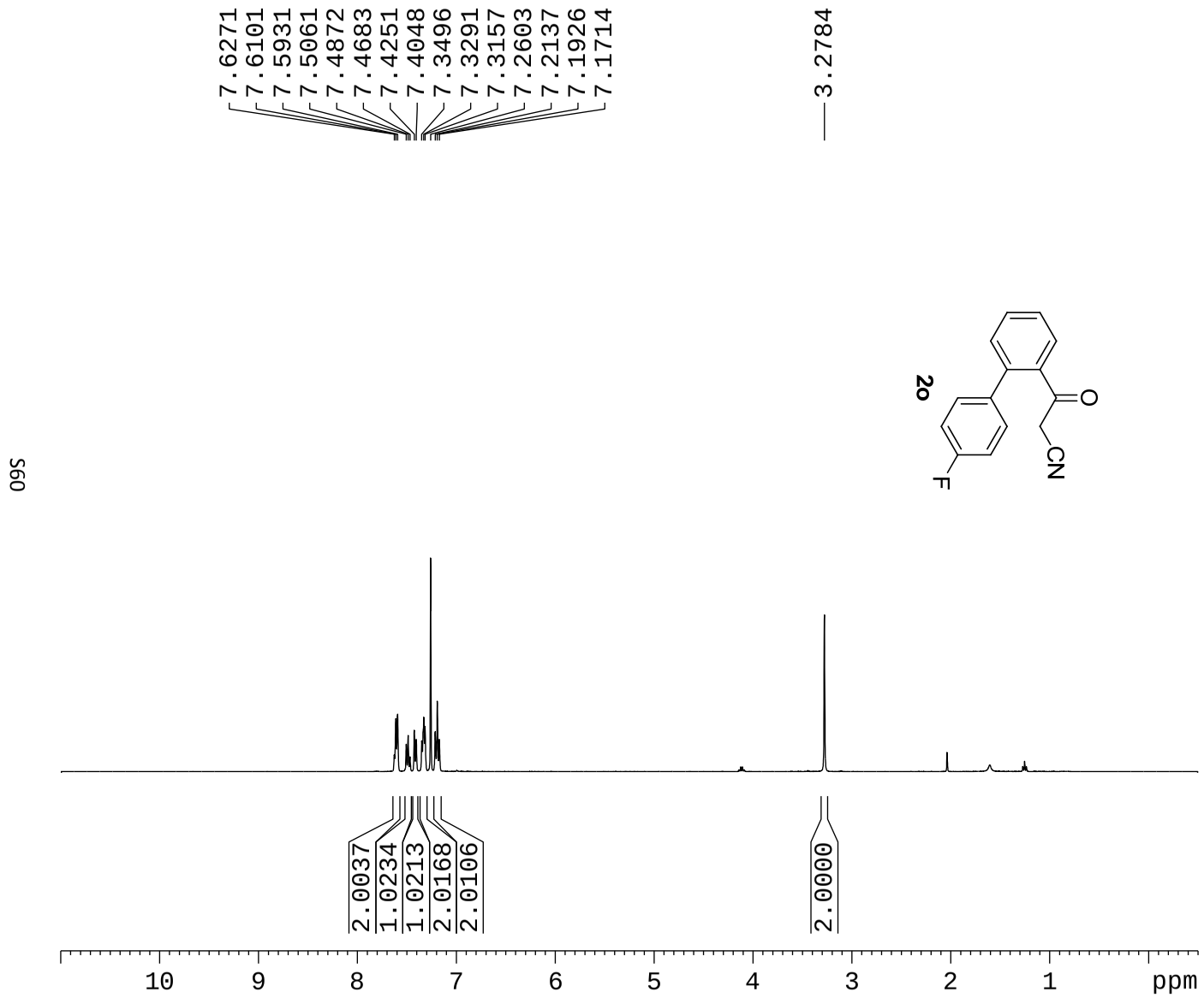


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EXPNO            1207011
PROCNO                 1

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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    17
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                297.7 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300082 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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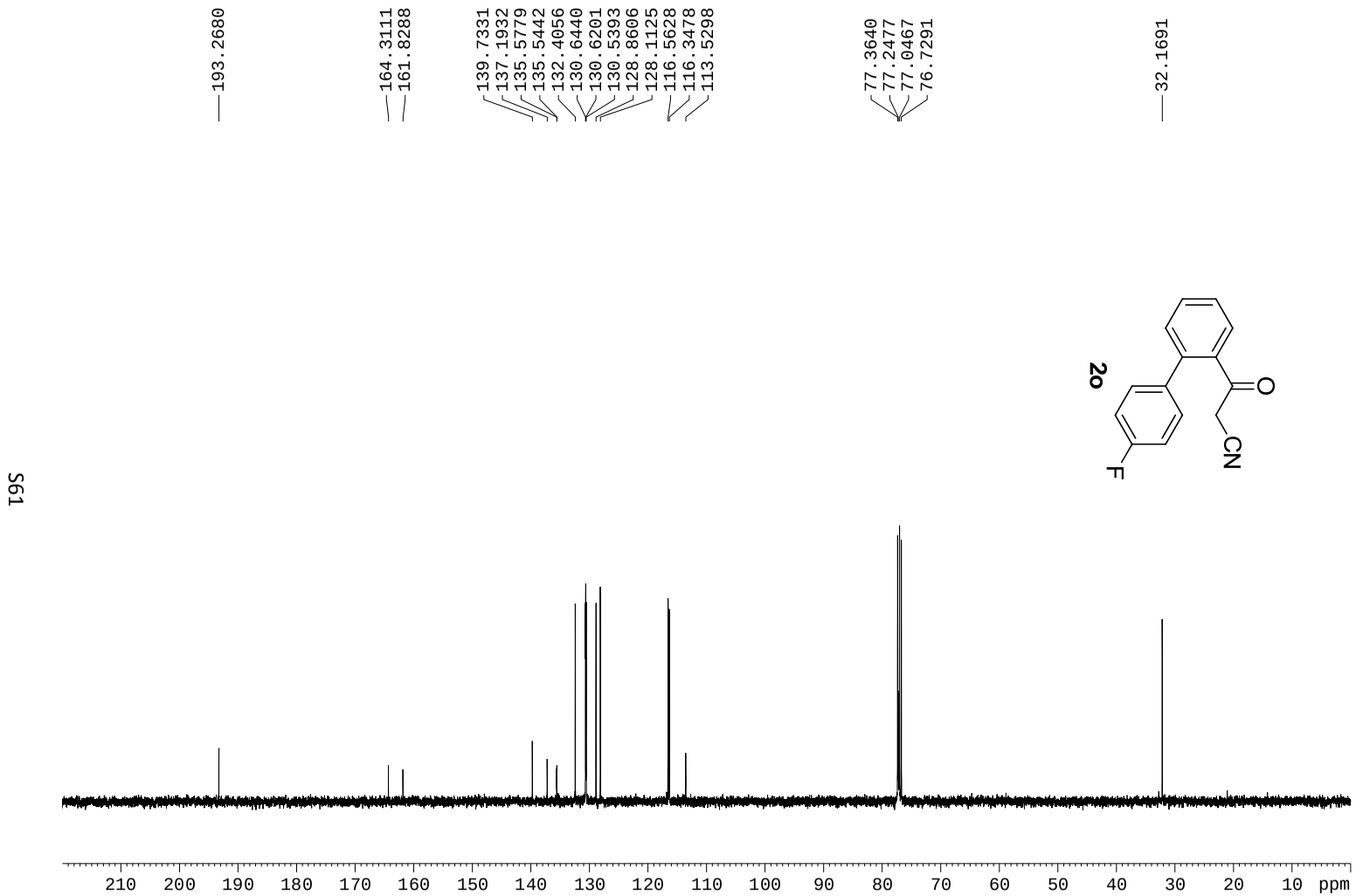




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EXPNO            1207012
PROCNO                 1

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Date_           20171207
Time               15.42
INSTRUM            spect
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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  257
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                297.9 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
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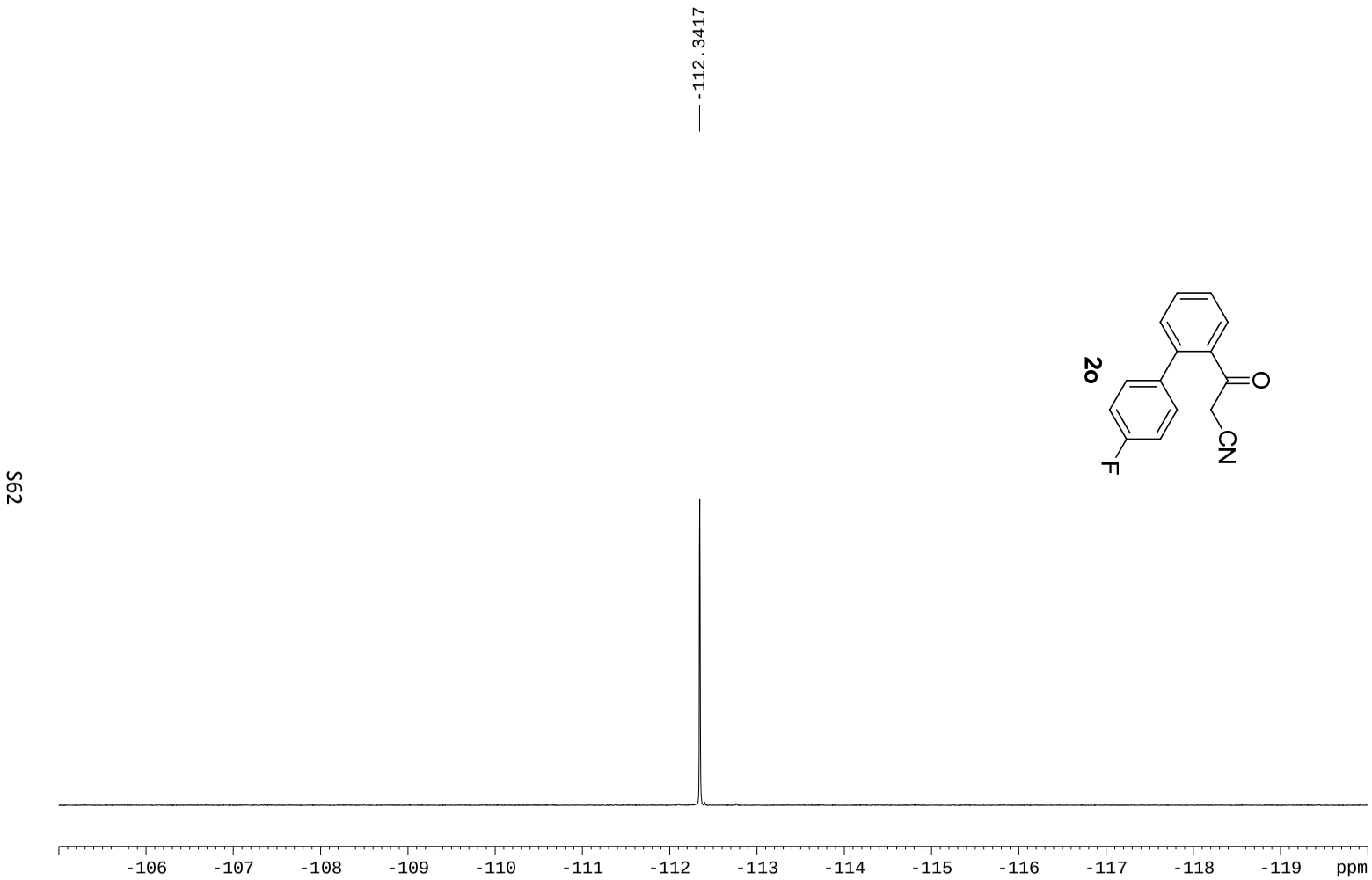





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EXPNO            1207013
PROCNO                 1

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PULPROG         zgfhigqn
TD               131072
SOLVENT             CDCl3
NS                      7
DS                      0
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340532 sec
RG                   2050
DW                  5.600 usec
DE                  6.50 usec
TE                 297.9 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   19F
P1                   20.00 usec
PL1                   2.50 dB
SFO1         376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -0.40 dB
PL12                15.80 dB
SFO2         400.1316005 MHz

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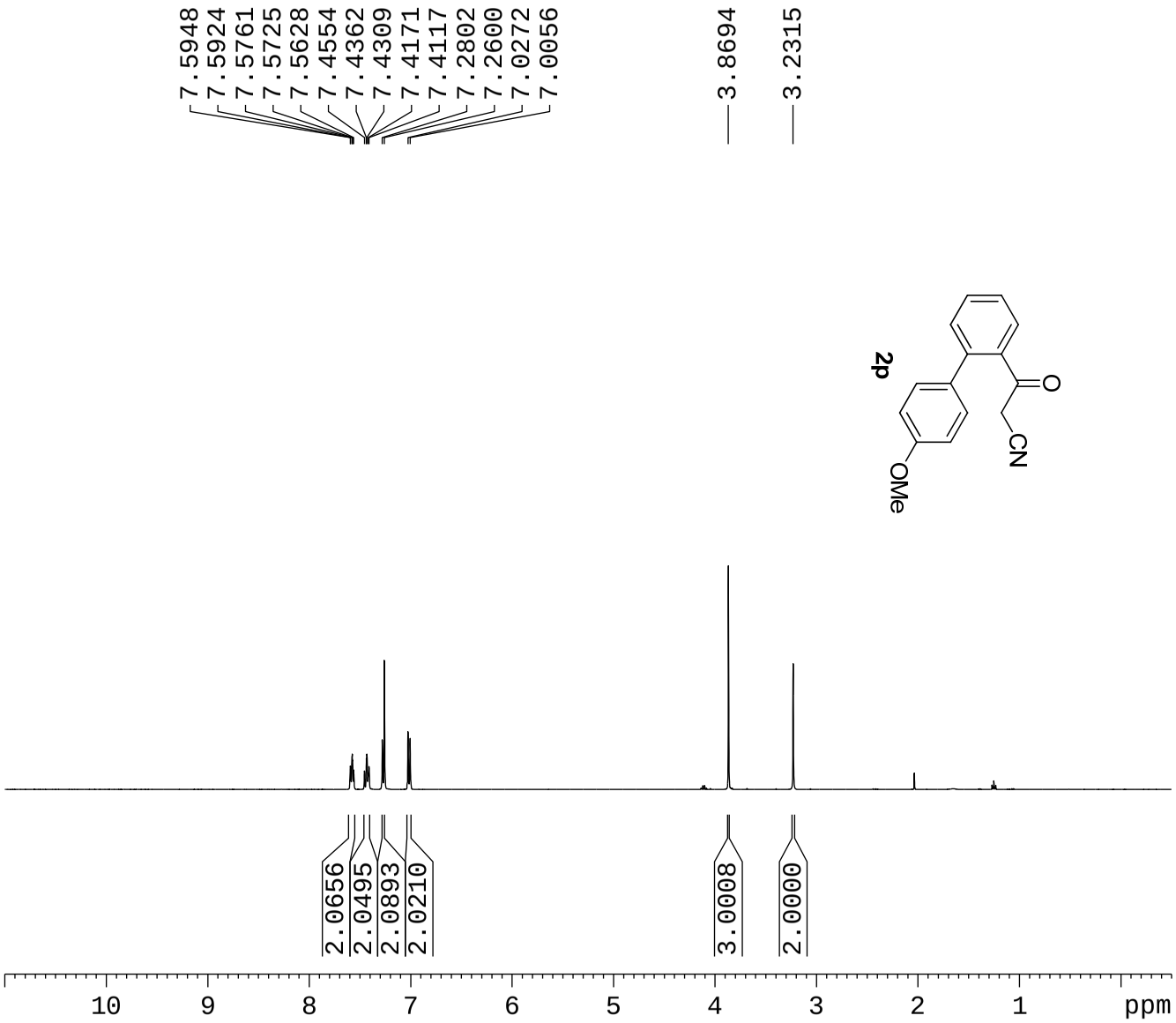


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EXPNO            1228011
PROCNO                 1

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INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    22
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   287
DW                83.200 usec
DE                 6.50 usec
TE                296.3 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300082 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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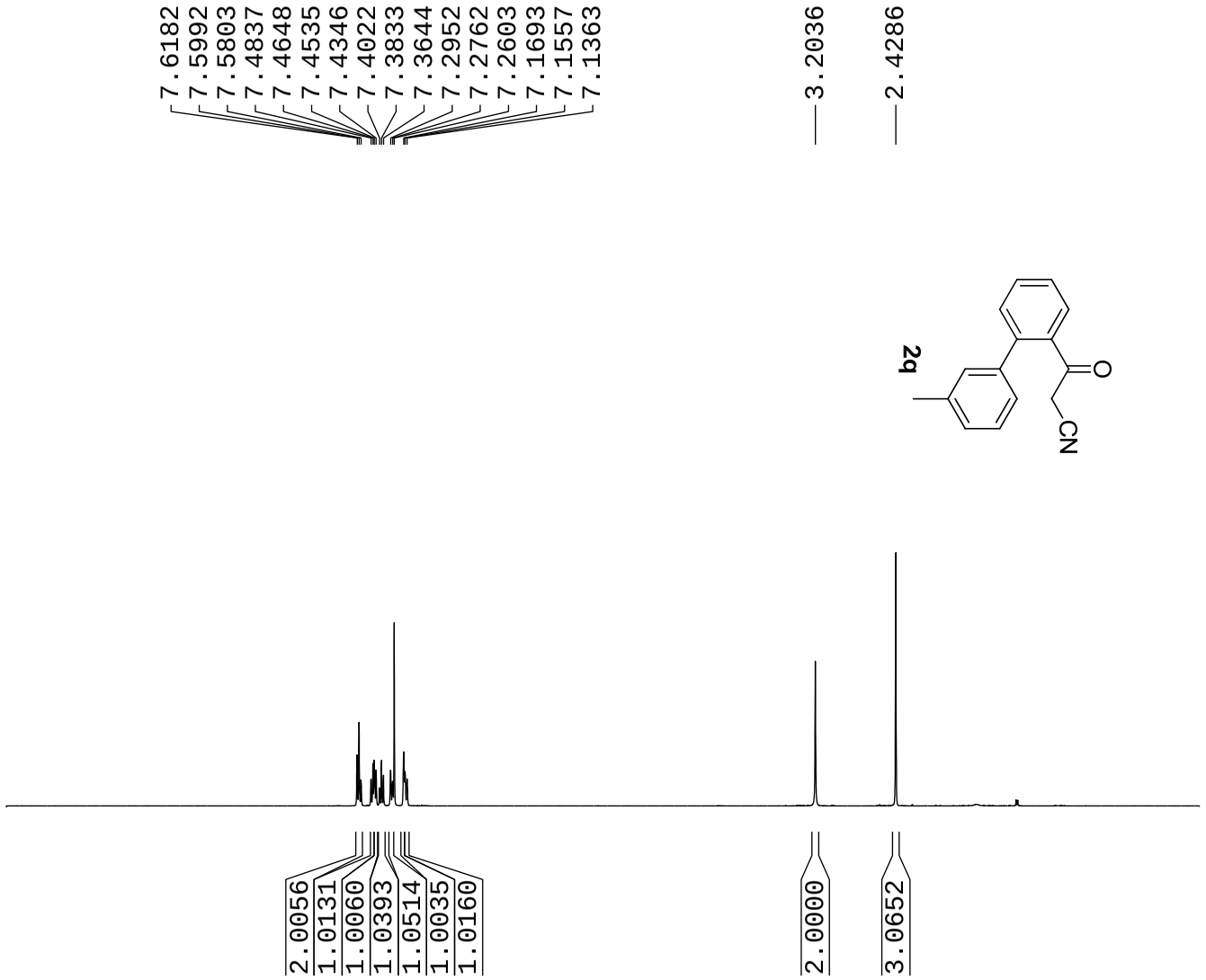
Current Data Parameters
NAME chen2017
EXPNO 1228012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171228
Time 10.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 297
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 296.4 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
CN
OMe
2p
32.1161
55.4380
76.7563
77.0744
77.2759
77.3920
113.8364
114.8715
127.5311
128.8496
130.0828
130.4187
131.6292
132.2658
137.3267
140.4985
160.2137
193.9175
ppm

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Current Data Parameters
NAME            chen2018
EXPNO             123011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180123
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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7263477 sec
RG                   287
DW                83.200 usec
DE                 6.50 usec
TE                296.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
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SF          400.1300082 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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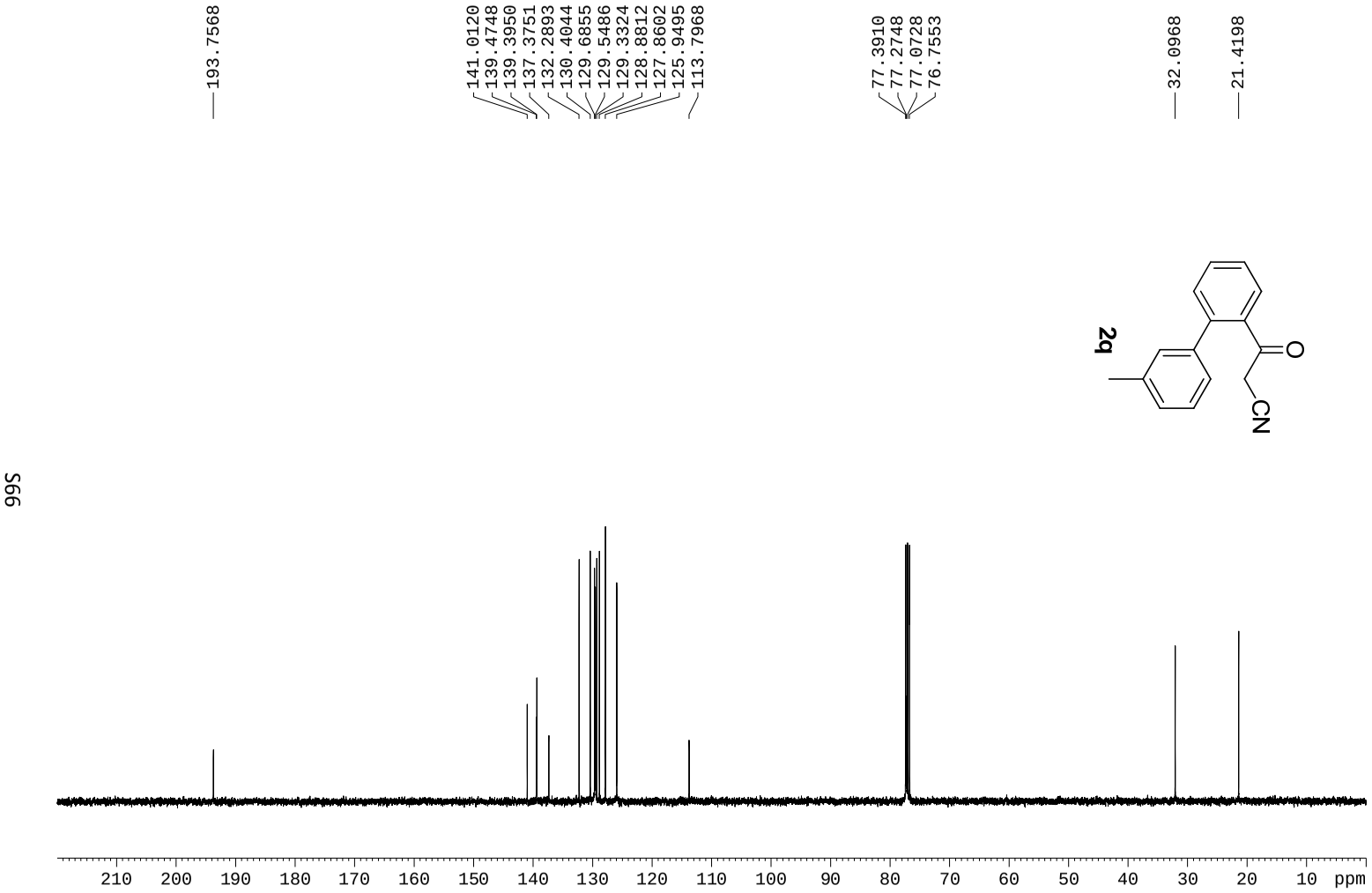


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EXPNO             123012
PROCNO                 1

F2 - Acquisition Parameters
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Time               10.03
INSTRUM            spect
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PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   329
DS                     0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ             1.2976629 sec
RG                  2050
DW                19.800 usec
DE                 6.50 usec
TE                296.4 K
D1             0.50000000 sec
d11            0.03000000 sec
DELTA          0.40000001 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
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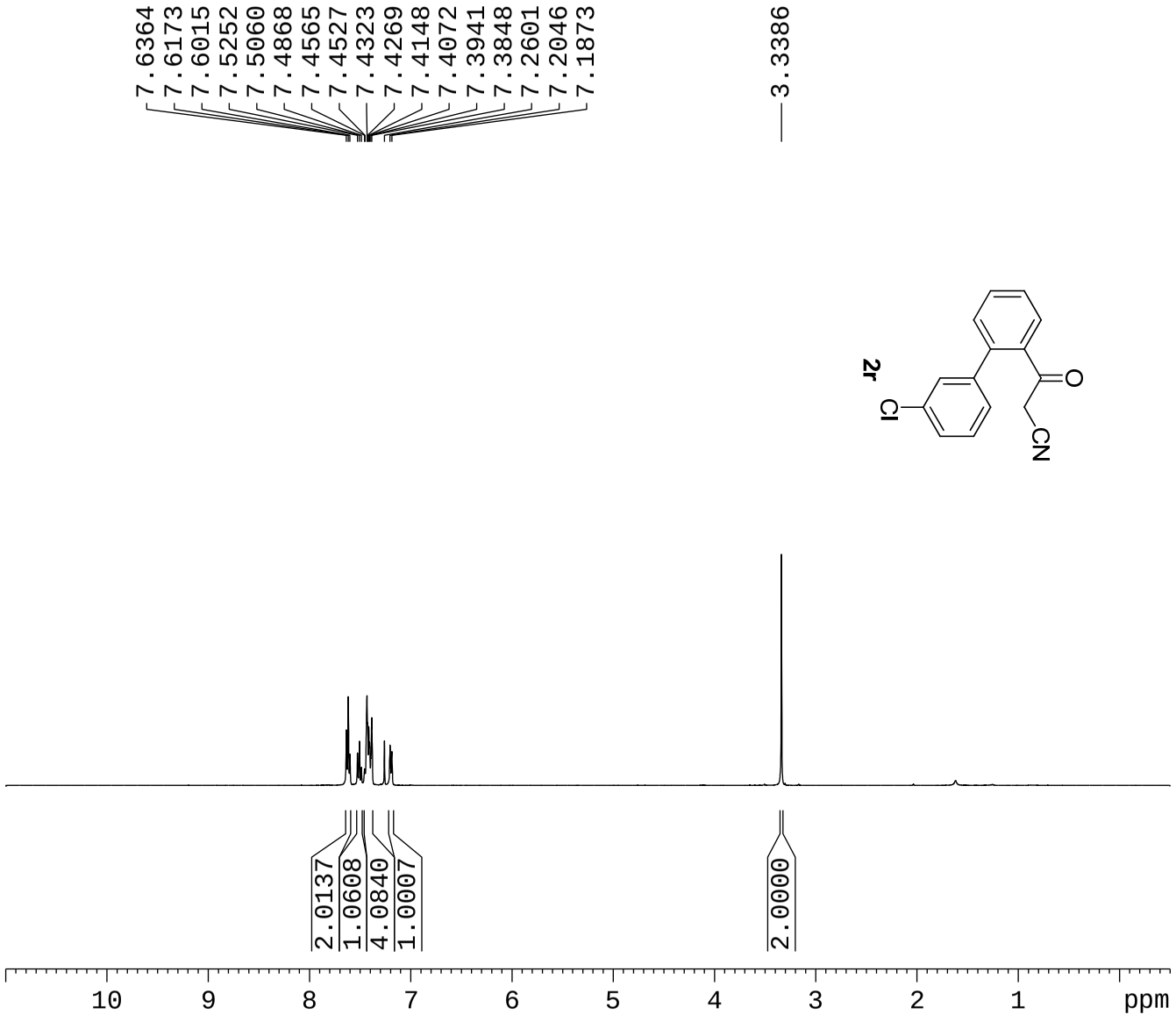


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Current Data Parameters
NAME                  chen2018
EXPNO                   301011
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180302
Time                     10.15
INSTRUM                  spect
PROBHD   5 mm QNP 1H/13
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          13
DS                           0
SWH                   6009.615 Hz
FIDRES                0.183399 Hz
AQ                    2.7263477 sec
RG                         362
DW                      83.200 usec
DE                       6.50 usec
TE                      299.2 K
D1                    2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       15.00 usec
PL1                       0.90 dB
SFO1                400.1326008 MHz

F2 - Processing parameters
SI                       16384
SF                  400.1300093 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
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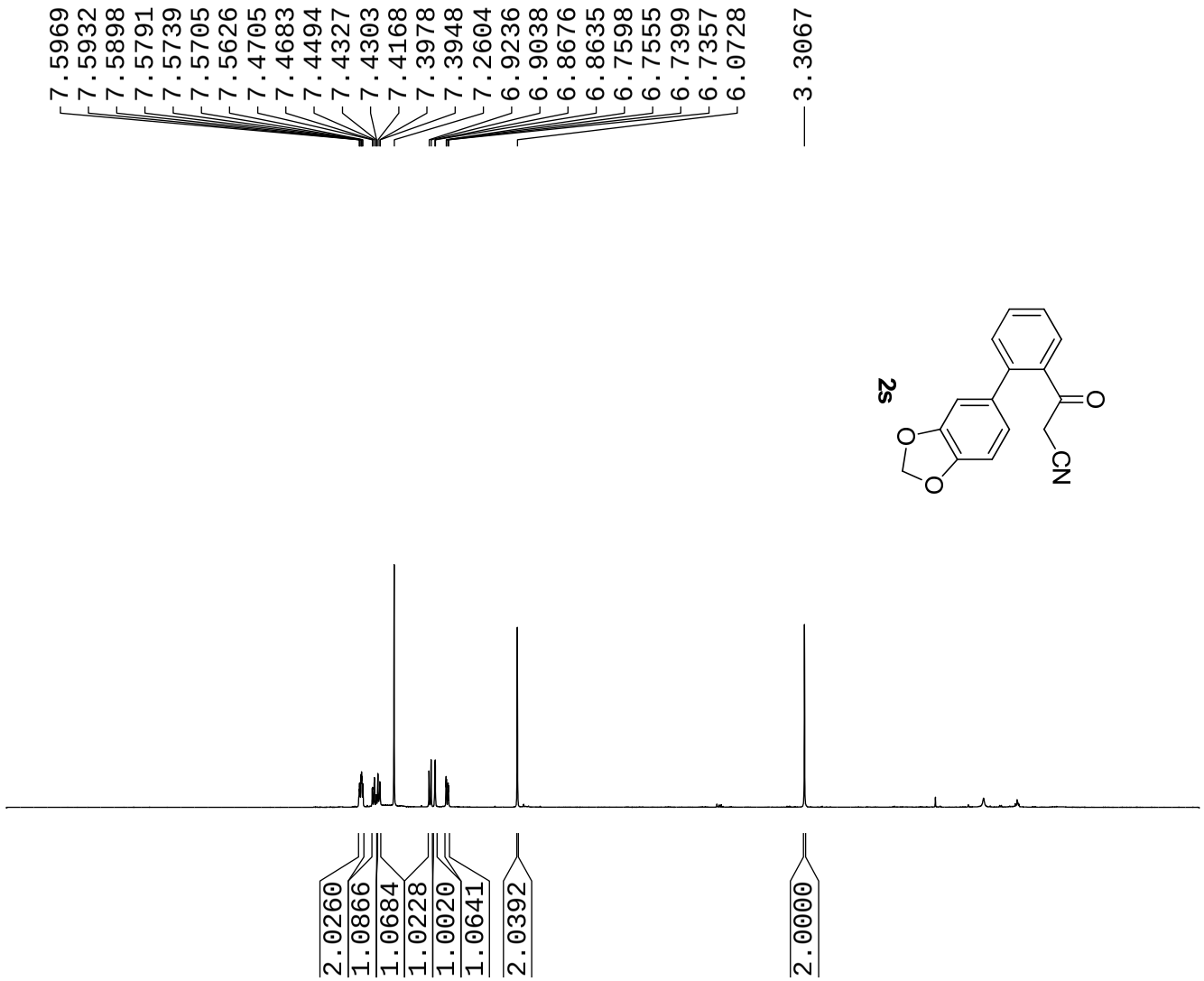
Current Data Parameters
NAME chen2018
EXPNO 301012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180302
Time 10.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 244
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 299.4 K
D1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000001 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
CN
Cl
2r
32.1895
77.3976
77.2796
77.0792
76.7619
113.4839
127.1794
128.4964
128.7172
128.8833
128.9631
130.5141
130.6386
132.5175
135.2928
136.9836
139.4413
141.3943
192.7586
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm




S69

Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 327021 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20180327 |
| Time | 12.38 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 33 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 406 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 293.5 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |

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

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
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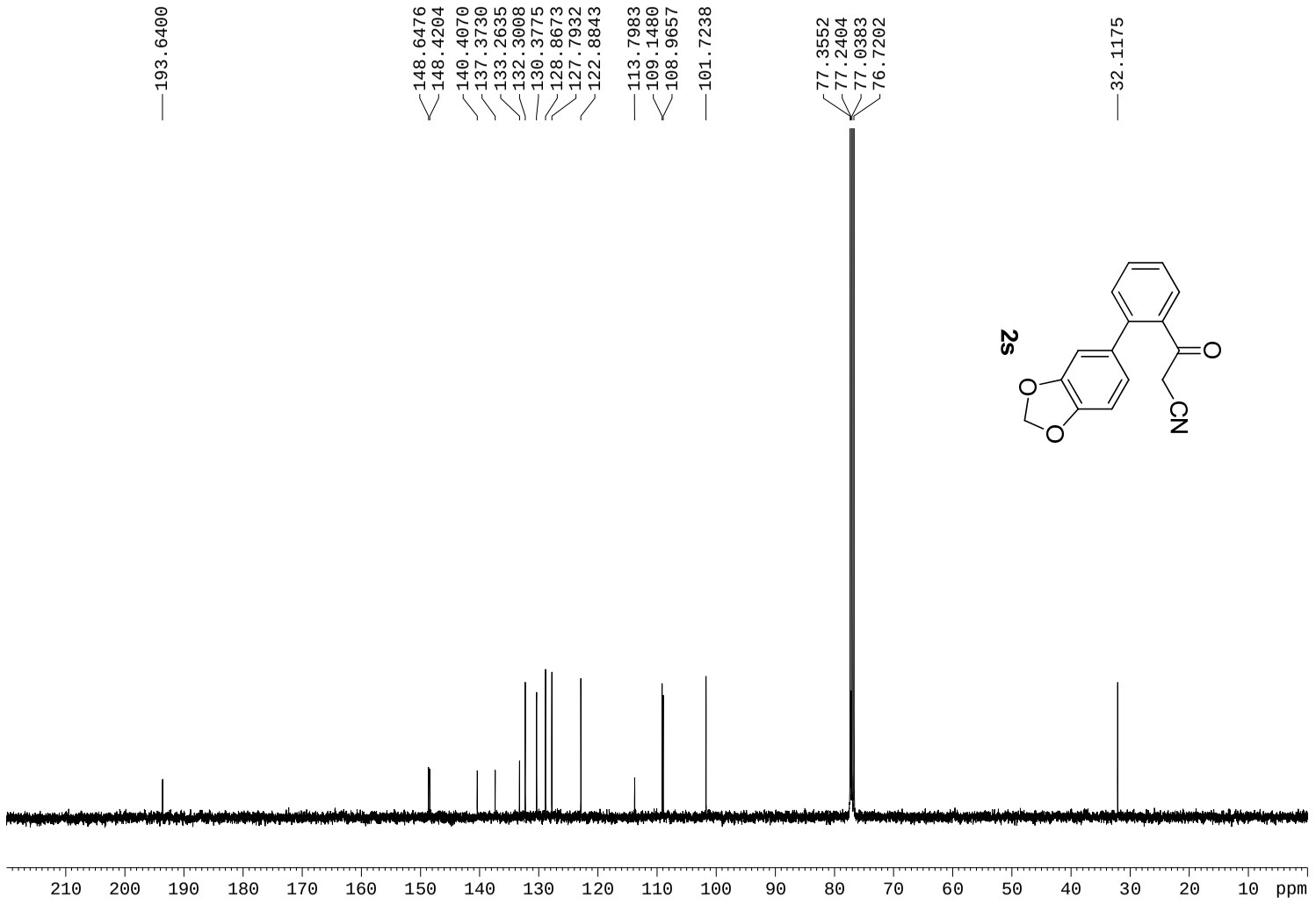
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

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EXPNO             327022
PROCNO                 1

F2 - Acquisition Parameters
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Time               12.45
INSTRUM            spect
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PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   949
DS                     0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                   406
DW                19.800 usec
DE                 6.50 usec
TE                293.9 K
D1            0.50000000 sec
d11           0.03000000 sec
DELTA         0.40000001 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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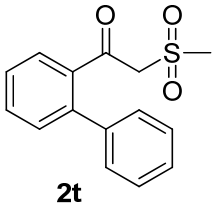
## v) Preparation and NMR spectra of 2t/u

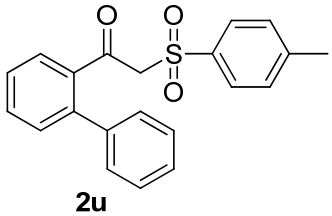
### 1-([1,1'-Biphenyl]-2-yl)-2-(methylsulfonyl)ethanone (2t)



**2t**

Under $N_2$ protection, *n*-BuLi (2.76 mL, 2.0 M in cyclohexane, 5.52 mmol) was dropwise added to a stirred solution of dimethyl sulfone (314.8 mg, 99%, 3.31 mmol) in THF (16 mL) at 0 °C. The resulting white cloudy solution was continued to stir at 0 °C for 40 min, followed by slowly adding with a solution of ethyl biphenyl-2-carboxylate (624.3 mg, 2.76 mmol) in THF (11.5 mL) over 5 min. The reaction mixture was then allowed to stir at rt for 31 hours, quenched by $H_2O$ (15 mL), and diluted with ethyl acetate (350 mL). The organic layer was separated and washed with saturated aqueous $NH_4Cl$ solution (50 mL x 2), water (50 mL) and brine (50 mL). After concentration, the crude mixture was subjected to chromatography (hexane-ethyl acetate 6:1, 4:1) to provide **2t** as a pale yellow solid (230.5 mg, 31%). IR (neat) 3017, 1686, 1313, 1152, 1125, 760, 746, 704 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.7 Hz, 1 H), 7.60 (ddd, *J* = 7.7, 7.7, 1.1 Hz, 1 H), 7.51-7.43 (m, 5 H), 7.35-7.31 (m, 2 H), 3.79 (s, 2 H), 2.98 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 195.3, 141.0, 139.7, 138.9, 132.3, 130.4, 129.3, 129.3, 129.0, 128.8, 127.9, 64.4, 41.9 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{15}H_{14}O_3S$: 274.0664; found: 274.0656.

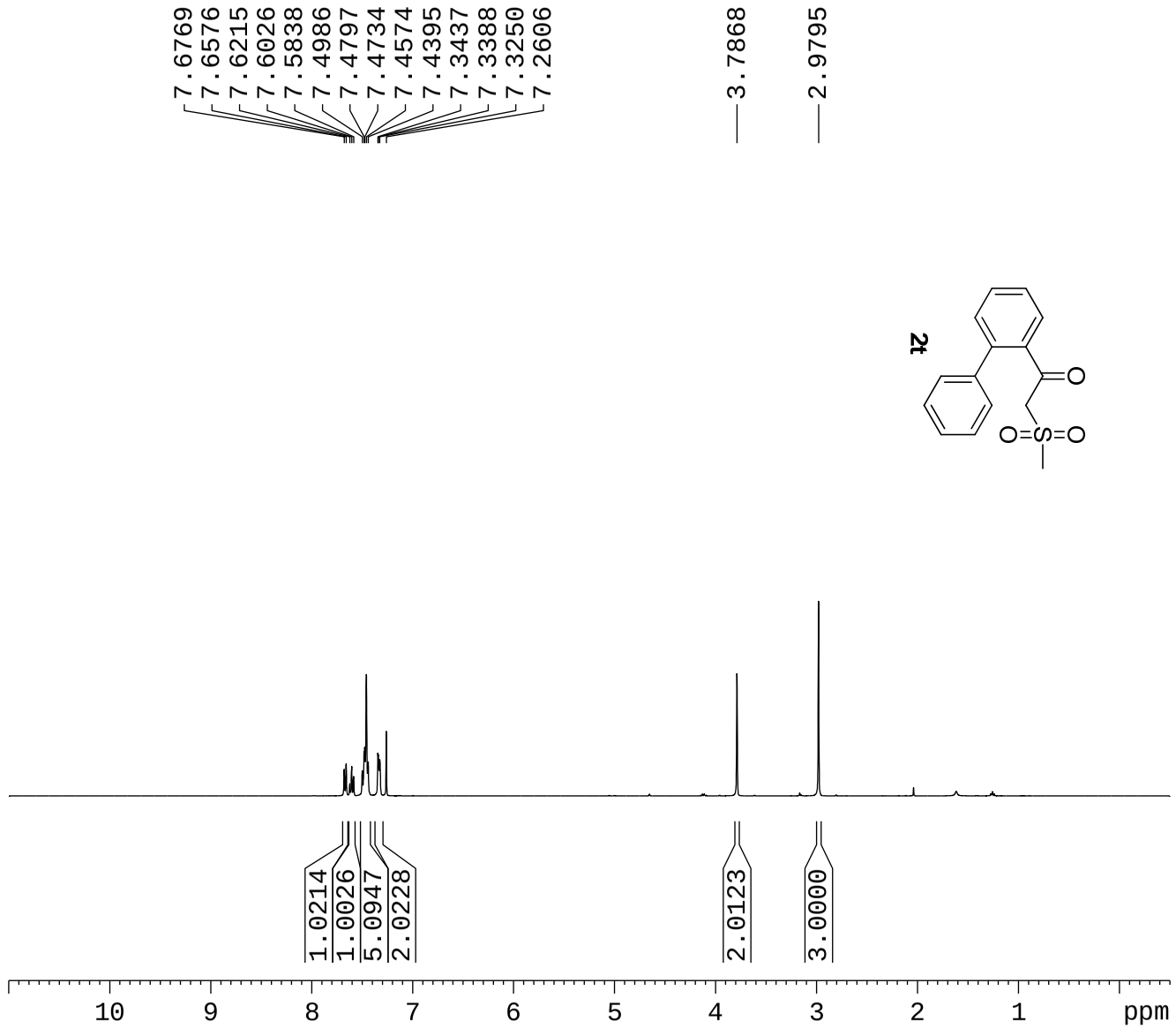
### 1-([1,1'-Biphenyl]-2-yl)-2-tosylethanone (2u)



**2u**

$Et_3N$ (0.2 mL, 99.5%, 1.25 mmol) was added to a stirred solution of **1a** (204.2 mg, 1.04 mmol) in MeOH (5.2 mL). After stirring at rt for an additional 30 min, sodium *p*-toluenesulfinate tetrahydrate (318.8 mg, 98%, 1.25 mmol) and iodine (265.4 mg, 99.5%, 1.04 mmol) were successively introduced to the mixture. The resulting yellow solution was then stirred in dark for 20 hours, diluted by ethyl acetate (200 mL), and washed with aqueous saturated $Na_2S_2O_3$ solution (30 mL x 2), water (30 mL x 2) and brine (30 mL). After concentration, the crude residue was subjected to chromatography (hexane-ethyl acetate 12:1, 10:1, 4:1) to provide **2u** as a yellow oil (154.4 mg, 42%). IR (neat) 3059, 1686, 1595, 1323, 1153, 757, 704 $cm^{-1}$; $^1H$ NMR

(400 MHz, $CDCl_3$) δ 7.56-7.48 (m, 4 H), 7.44-7.38 (m, 4 H), 7.28 (d, *J* = 7.6 Hz, 1 H), 7.26-7.18 (m, 4 H), 3.91 (s, 2 H), 2.42 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 194.3, 145.0, 140.7, 139.6, 138.9, 136.0, 131.9, 130.1, 129.6, 129.4, 129.2, 129.0, 128.6, 128.4, 127.7, 66.5, 21.7 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{21}H_{18}O_3S$: 350.0977; found:350.0968.

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EXPNO 628011
PROCNO 1

F2 - Acquisition Parameters
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Time 10.11
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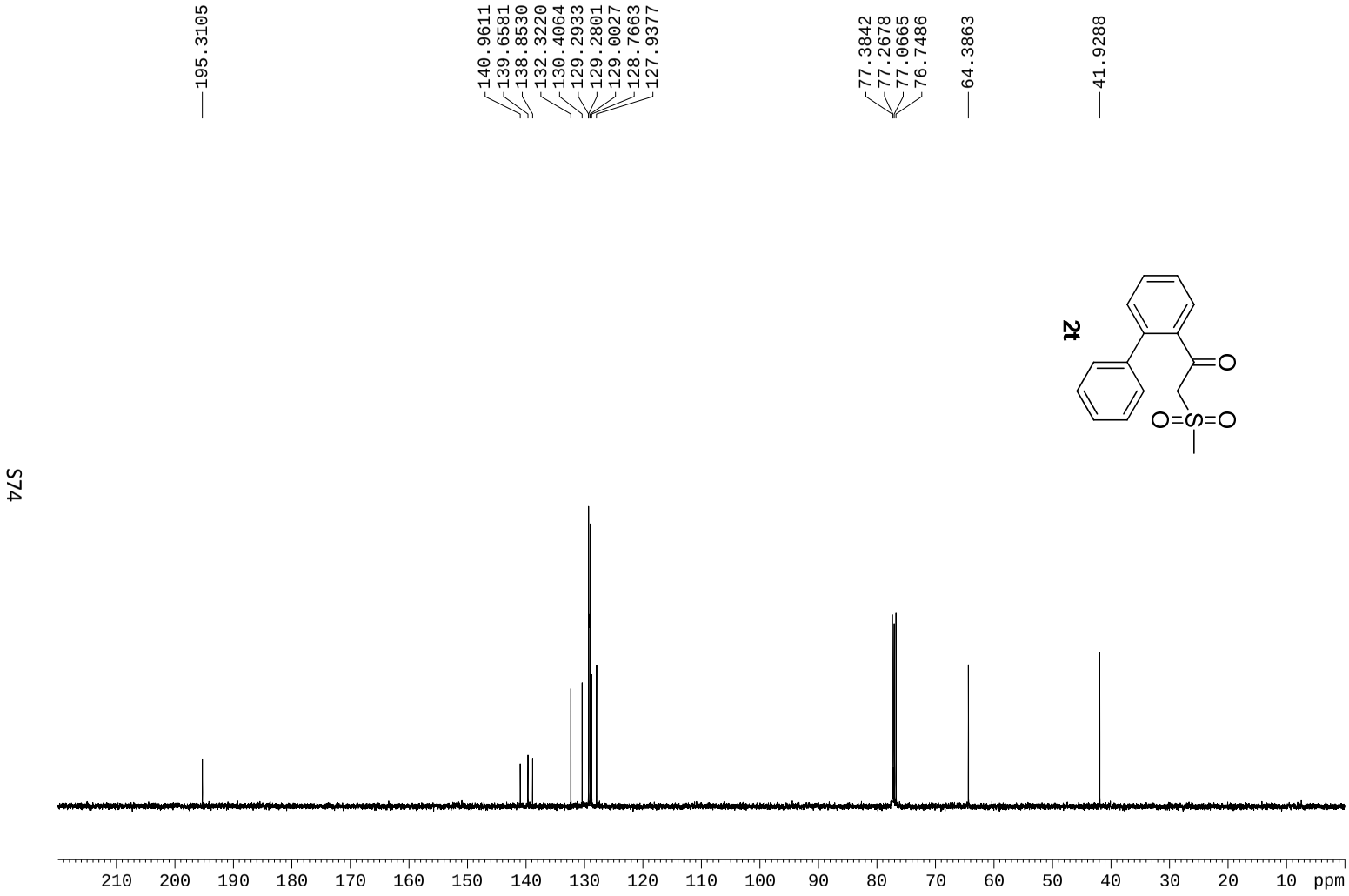
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 40
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 181
DW 83.200 usec
DE 6.50 usec
TE 303.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




Current Data Parameters

| | |
|---|---|
| NAME | chen2017 |
| EXPNO | 628012 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
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| Time | 10.17 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 176 |
| DS | 0 |
| SWH | 25252.525 Hz |
| FIDRES | 0.385323 Hz |
| AQ | 1.2976629 sec |
| RG | 2050 |
| DW | 19.800 usec |
| DE | 6.50 usec |
| TE | 303.2 K |
| D1 | 0.20000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 0.10000000 sec |
| TD0 | 1 |

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

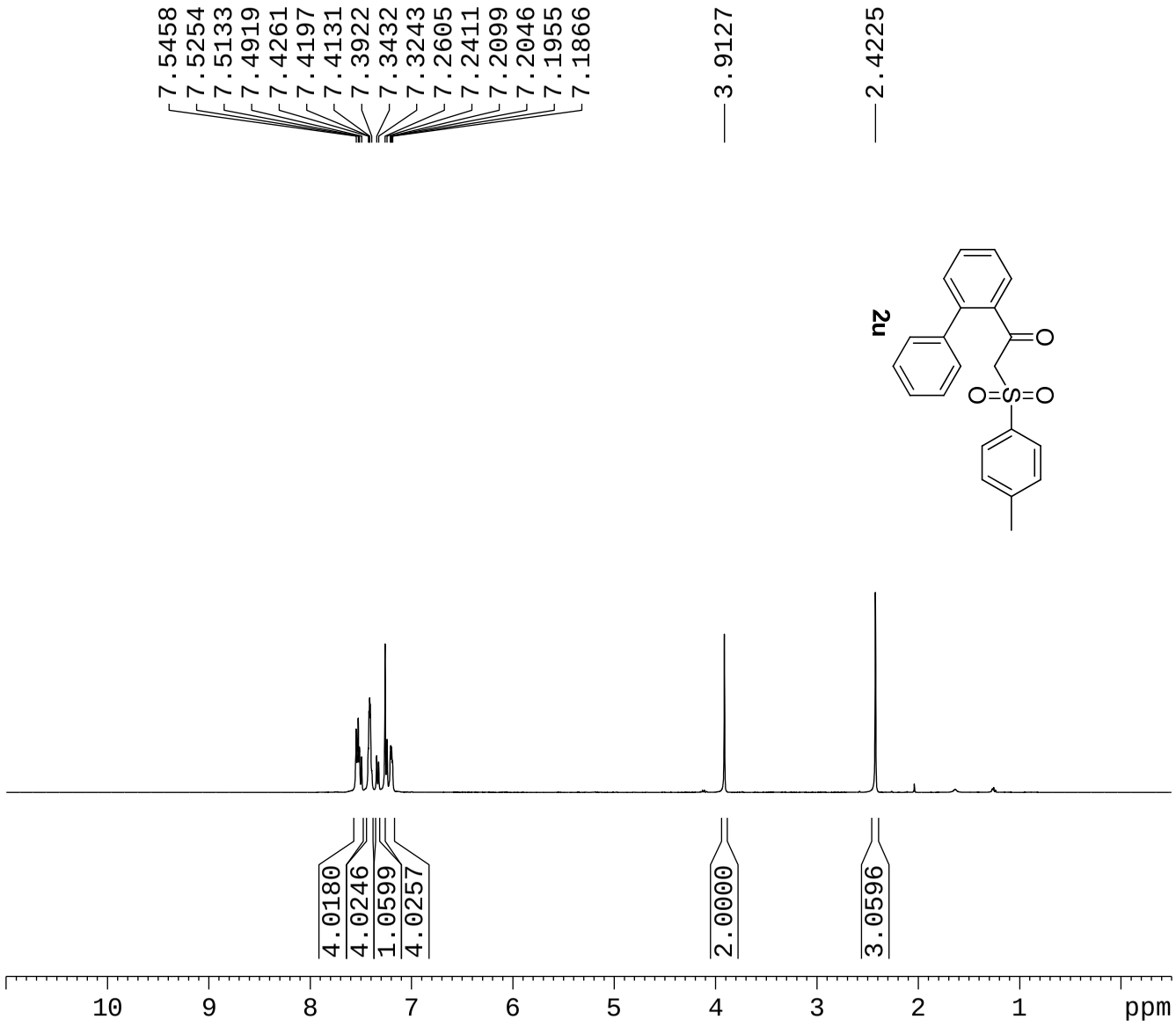
| | |
|---|---|
| NUC1 | 13C |
| P1 | 10.00 usec |
| PL1 | 6.20 dB |
| SFO1 | 100.6243395 MHz |

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

| | |
|---|---|
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 90.00 usec |
| PL2 | -0.40 dB |
| PL12 | 15.80 dB |
| PL13 | 18.80 dB |
| SFO2 | 400.1316005 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 32768 |
| SF | 100.6127690 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

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EXPNO             828011
PROCNO                 1

F2 - Acquisition Parameters
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Time               13.17
INSTRUM            spect
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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ               2.7263477 sec
RG                   128
DW                83.200 usec
DE                 6.50 usec
TE                301.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300093 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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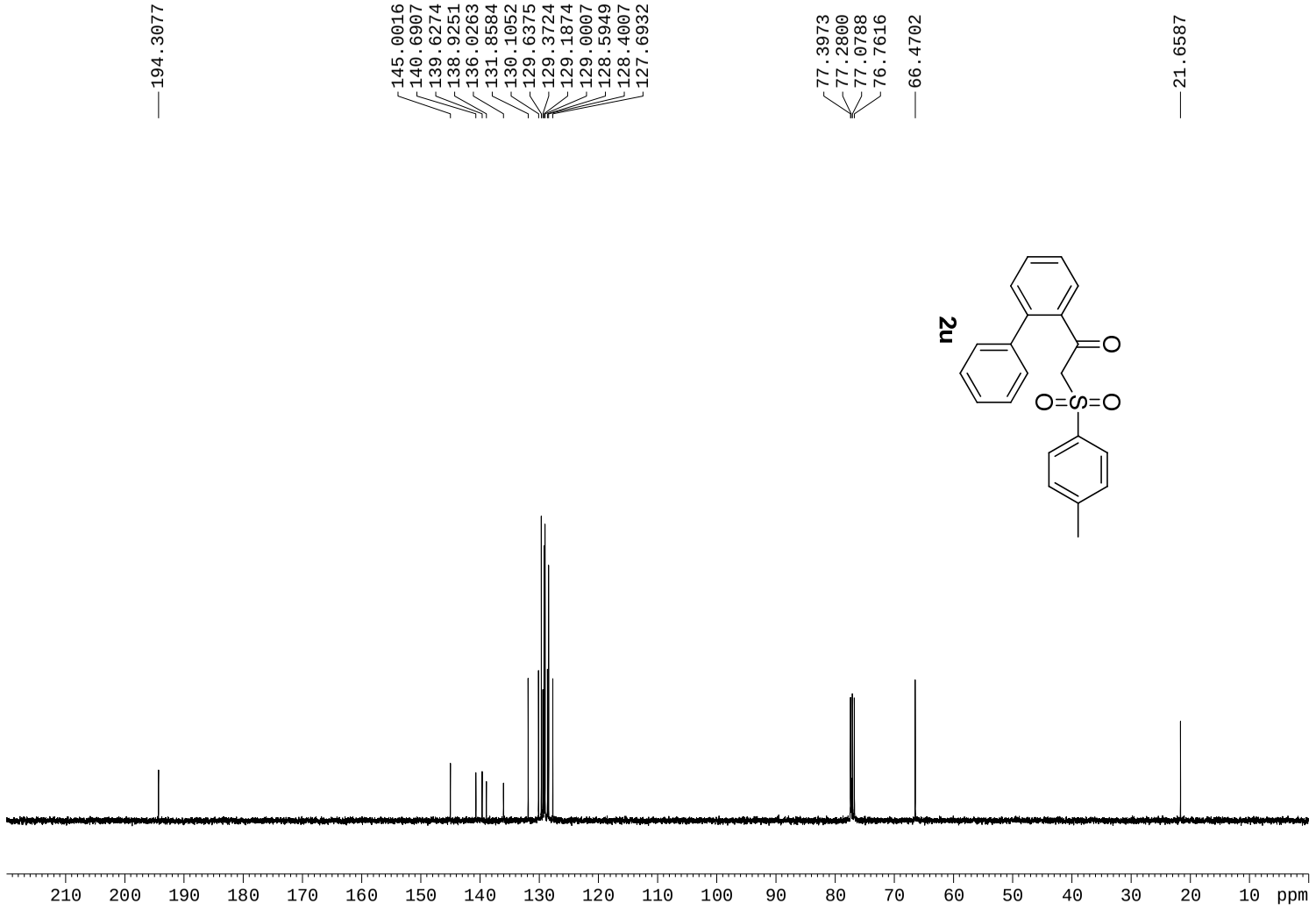


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Current Data Parameters
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EXPNO             828012
PROCNO                 1

F2 - Acquisition Parameters
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Time               13.26
INSTRUM            spect
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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  229
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW                19.800 usec
DE                 6.50 usec
TE                301.4 K
D1            0.20000000 sec
d11           0.03000000 sec
DELTA         0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```
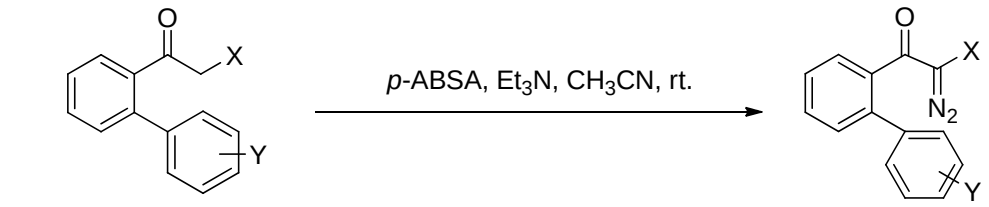
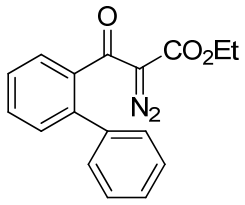

**3) Preparation of 3**

**i) Preparation and NMR spectra of 3a-l**




X = $CO_2Et$, $COCH_3$, COPh

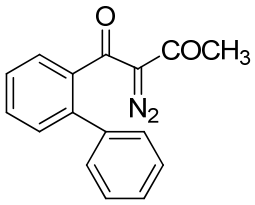
**Ethyl 3-([1,1'-biphenyl]-2-yl)-2-diazo-3-oxopropanoate (3a)**




**3a**

To a solution of **2a** (187 mg, 0.697 mmol) in acetonitrile (5 mL) under $N_2$ protection, $Et_3N$ (0.39 mL, 99.5%, 2.79 mmol) and *p*-acetamidobenzenesulfonyl azide (683 mg, 98%, 2.79 mmol) were successively added. The reaction mixture was stirred in dark for 9 hours, then diluted by $CH_2Cl_2$ (200 mL), and washed with water (50 mL x 2) and brine (100 mL). After concentration, the crude reside was purified by chromatography (hexane-ethyl acetate 15:1, 8:1) to afford **3a** as a colorless oil (188.3 mg, 92%). IR (neat) 3059, 2139, 1724, 1632, 1304, 776, 746 $cm^{-1}$; $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.56-7.50 (m, 1 H), 7.45-7.41 (m, 3 H), 7.38-7.33 (m, 5 H), 4.01 (q, *J* = 7.1 Hz, 2 H), 1.07 (t, *J* = 7.1 Hz, 3 H) ppm; $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 189.2, 160.2, 140.2, 139.7, 137.8, 130.6, 129.5, 129.1, 128.2, 127.7, 127.3, 127.1, 61.3, 60.4 (C=$N_2$), 14.0 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{14}N_2O_3$: 294.1004; found: 294.1006.

**1-([1,1'-Biphenyl]-2-yl)-2-diazobutane-1,3-dione (3b)**

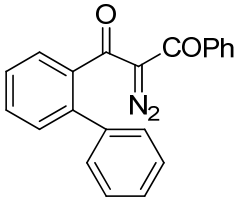



**3b**

The titled compound was synthesized from **2b** by following the typical procedure. The reaction proceeded for 14 hours, and the chromatographic purification (hexane-ethyl acetate 30:1, 10:1, 5:1) gave **3b** as a pale yellow oil (67%). IR (neat) 3062, 2120, 1658, 1652, 1313, 1231, 745, 700 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 7.4, 7.1 Hz, 1 H), 7.51-7.44(m, 3 H), 7.42-7.38 (m, 5 H), 2.39 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 189.8, 187.3, 139.6, 138.7, 137.4, 131.3, 130.1,

128.7, 128.7, 128.5, 128.1, 127.7, 87.0 (C=$N_2$), 28.8 ppm; HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{16}H_{13}N_2O_2$: 265.0977; found: 265.0974.

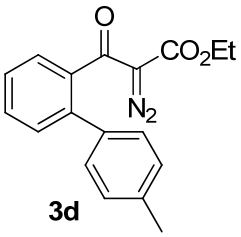
**1-([1,1'-Biphenyl]-2-yl)-2-diazo-3-phenylpropane-1,3-dione (3c)**




**3c**

The titled compound was synthesized from **2c** by following the typical procedure. The reaction proceeded for 6 hours, and the chromatographic purification (hexane-ethyl acetate 30:1, 10:1, 5:1) gave **3c** as a yellow viscous oil (93%). IR (neat) 3060, 2108, 1652, 1598, 1293, 1268, 778, 745, 701 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.57-7.27 (m, 12 H), 7.18 (d, *J* = 7.4 Hz, 2 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 188.9, 184.8, 140.0, 139.5, 137.6, 136.5, 132.5, 131.1, 129.8, 129.3, 128.5, 128.1, 128.1, 128.0, 127.9, 127.8, 85.0 (C=$N_2$) ppm; HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{21}H_{15}N_2O_2$: 327.1134; found: 327.1122.

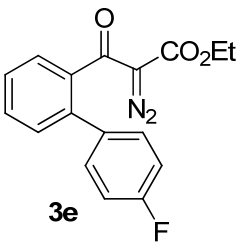
**Ethyl 2-diazo-3-(4'-methyl-[1,1'-biphenyl]-2-yl)-3-oxopropanoate (3d)**




**3d**

The titled compound was synthesized from **2d** by following the typical procedure. The reaction proceeded for 6 hours, and the chromatographic purification (hexane-ethyl acetate 40:1, 20:1, 10:1) gave **3d** as a yellow oil (quant.). IR (neat) 3022, 2138, 1725, 1700, 1304, 758 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.53-7.48 (m, 1 H), 7.42-7.36 (m, 3 H), 7.24 (d, *J* = 8.0 Hz, 2 H), 7.92 (d, *J* = 7.9 Hz, 2 H), 4.02 (q, *J* = 7.1 Hz, 2 H), 2.38 (s, 3 H), 1.07 (t, *J* = 7.1 Hz, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 189.2, 160.3, 140.1, 137.7, 137.5, 136.7, 130.6, 129.5, 129.0, 128.9, 127.1, 127.0, 61.3, 60.1 (C=$N_2$), 21.2, 14.0 ppm; HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{18}H_{17}N_2O_3$: 309.1239; found: 309.1235.

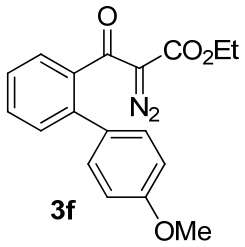
**Ethyl 2-diazo-3-(4'-fluoro-[1,1'-biphenyl]-2-yl)-3-oxopropanoate (3e)**




**3e**

The titled compound was synthesized from **2e** by following the typical procedure. The reaction proceeded for 1 hour, and the chromatographic purification (hexane-ethyl acetate 30:1, 20:1, 10:1) gave **3e** as a yellow oil (92%). IR (neat) 3064, 2141, 1725, 1697, 1631, 1315, 1273, 841, 762 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 7.52 (td, $J$ = 7.3, 1.5 Hz, 1 H), 7.45-7.38 (m, 2 H), 7.36-7.29 (m, 3 H), 7.07 (dd, $J$ = 8.6 Hz, $J_{H-F}$ = 8.6 Hz, 2 H), 4.03 (q, $J$ = 7.1 Hz, 2 H), 1.09 (t, $J$ = 7.1 Hz, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 189.1, 162.5 (d, $J_{C-F}$ = 245.8 Hz), 160.1, 139.0, 137.8, 135.7 (d, $J_{C-F}$ = 3.2 Hz), 130.7 (d, $J_{C-F}$ = 8.0 Hz), 130.6, 129.5, 127.4, 127.1, 115.2 (d, $J_{C-F}$ = 21.4 Hz), 65.1, 61.4 (C=$N_2$), 14.0 ppm; $^{19}F$ NMR (376 MHz, $CDCl_3$) δ -114.6 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{13}FN_2O_3$: 312.0910; found: 312.0905.

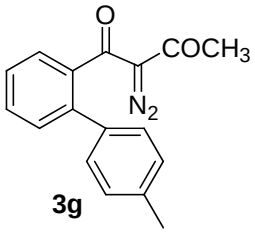
**Ethyl 2-diazo-3-(4'-methoxy-[1,1'-biphenyl]-2-yl)-3-oxopropanoate (3f)**



**3f**

The titled compound was synthesized from **2f** by following the typical procedure. The reaction proceeded for 3 hours, and the chromatographic purification (hexane-ethyl acetate 30:1, 20:1, 10:1) gave **3f** as a yellow oil (quant.). IR (neat) 3012, 2139, 1726, 1694, 1306, 1250, 835, 762 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 7.53-7.47 (m, 1H), 7.41-7.37 (m, 2 H), 7.35 (d, $J$ = 7.7 Hz, 1 H), 7.27 (d, $J$ = 9.0 Hz, 2 H), 6.91 (d, $J$ = 9.0 Hz, 2 H), 4.02 (q, $J$ = 7.1 Hz, 2 H), 3.83 (s, 3 H), 1.08 (t, $J$ = 7.1 Hz, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 189.3, 160.2, 159.3, 139.7, 137.6, 131.9, 130.5, 130.1, 129.4, 127.0, 126.8, 113.7, 61.2, 61.1, 55.2, 14.0 ppm.

**2-Diazo-1-(4'-methyl-[1,1'-biphenyl]-2-yl)butane-1,3-dione (3g)**

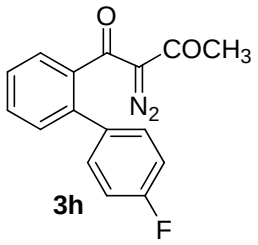


**3g**

The titled compound was synthesized from **2g** by following the typical procedure. The reaction proceeded for 6 hours, and the chromatographic purification (hexane-ethyl acetate 60:1, 15:1, 10:1) gave **3g** as a yellow oil (84%). IR (neat) 3024, 2120, 1660, 1652, 759, 721 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 7.55 (m, 1 H), 7.50-7.41 (m, 3 H), 7.30 (d, $J$ = 7.9 Hz, 2 H), 7.20 (d, $J$ = 7.8 Hz, 2 H), 2.40 (s, 3 H), 2.38 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 190.0, 187.4, 139.6, 138.4, 137.3, 135.8, 131.2, 130.0, 129.4, 128.6, 127.8, 127.8, 83.1 (C=$N_2$), 28.0, 21.2 ppm;

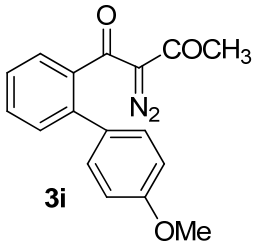
HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{17}H_{15}N_2O_2$: 279.1134; found: 279.1129.

**2-Diazo-1-(4'-fluoro-[1,1'-biphenyl]-2-yl)butane-1,3-dione (3h)**



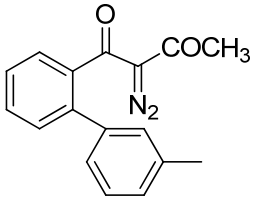

The titled compound was synthesized from **2h** by following the typical procedure. The reaction proceeded for 20 hours, and the chromatographic purification (hexane-ethyl acetate 30:1, 10:1) gave **3h** as a yellow oil (quant.). IR (neat) 3068, 2121, 1659, 1652, 841, 762, 711 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.58-7.54 (m, 1 H), 7.50-7.45 (m, 2 H), 7.41-7.36 (m, 3 H), 7.10 (dd, $J$ = 8.6 Hz, $J_{H-F}$ = 8.6 Hz, 2 H), 2.39 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 189.6, 187.1, 162.9 (d, $J_{C-F}$ = 247.3 Hz), 138.5, 137.3, 134.8 (d, $J_{C-F}$ = 3.3 Hz), 131.3, 130.4 (d, $J_{C-F}$ = 8.1 Hz), 130.1, 128.2, 127.7, 115.8 (d, $J_{C-F}$ = 21.5 Hz), 86.7 (C=$N_2$), 28.9 ppm; $^{19}F$ NMR (376 MHz, $CDCl_3$) δ -113.1 ppm; HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{16}H_{12}FN_2O_2$: 283.0883; found: 283.0875.

**2-Diazo-1-(4'-methoxy-[1,1'-biphenyl]-2-yl)butane-1,3-dione (3i)**




The titled compound was synthesized from **2i** by following the typical procedure. The reaction proceeded for 1 hour, and the chromatographic purification (hexane-ethyl acetate 40:1, 30:1, 10:1) gave **3i** as a pale yellow solid (50%). IR (neat) 3008, 2121, 1660, 1652, 1316, 836, 763 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.54 (t, $J$ = 7.4 Hz, 1 H), 7.48-7.39 (m, 3 H), 7.33 (d, $J$ = 8.6 Hz, 2 H), 6.92 (d, $J$ = 8.6 Hz, 2 H), 3.83 (s, 3 H), 2.41 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 190.0, 187.5, 159.9, 139.2, 137.2, 131.3, 130.9, 129.9, 129.9, 127.8, 127.6, 114.2, 86.8 (C=$N_2$), 55.3, 28.9 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{14}N_2O$: 294.1004; found: 294.0998.

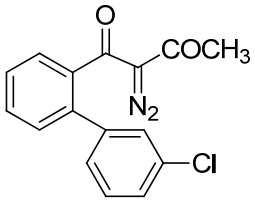
**2-Diazo-1-(3'-methyl-[1,1'-biphenyl]-2-yl)butane-1,3-dione (3j)**



**3j**

The titled compound was synthesized from **2j** by following the typical procedure. The reaction proceeded for 1.5 hours, and the chromatographic purification (hexane-ethyl acetate 50:1, 30:1, 10:1) gave **3j** as a yellow oil (72%). IR (neat) 3063, 2121, 1655, 1314, 774, 700 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.56 (td, *J* = 7.3, 1.7 Hz, 1 H), 7.51-7.43 (m, 4 H), 7.22-7.19 (m, 3 H), 2.40 (s, 3 H), 2.37 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 190.1, 187.5, 139.7, 138.6, 138.4, 137.3, 131.3, 130.0, 129.4, 129.3, 128.6, 128.0, 127.8, 125.9, 86.5 ($C=N_2$), 28.9, 21.4 ppm; HRMS-ESI: *m/z* [M + Na]$^+$ calcd. for $C_{17}H_{14}N_2O_2Na$: 301.0953; found: 301.0954.

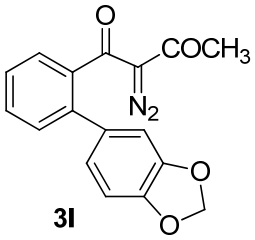
**1-(3'-Chloro-[1,1'-biphenyl]-2-yl)-2-diazobutane-1,3-dione (3k)**



**3k**

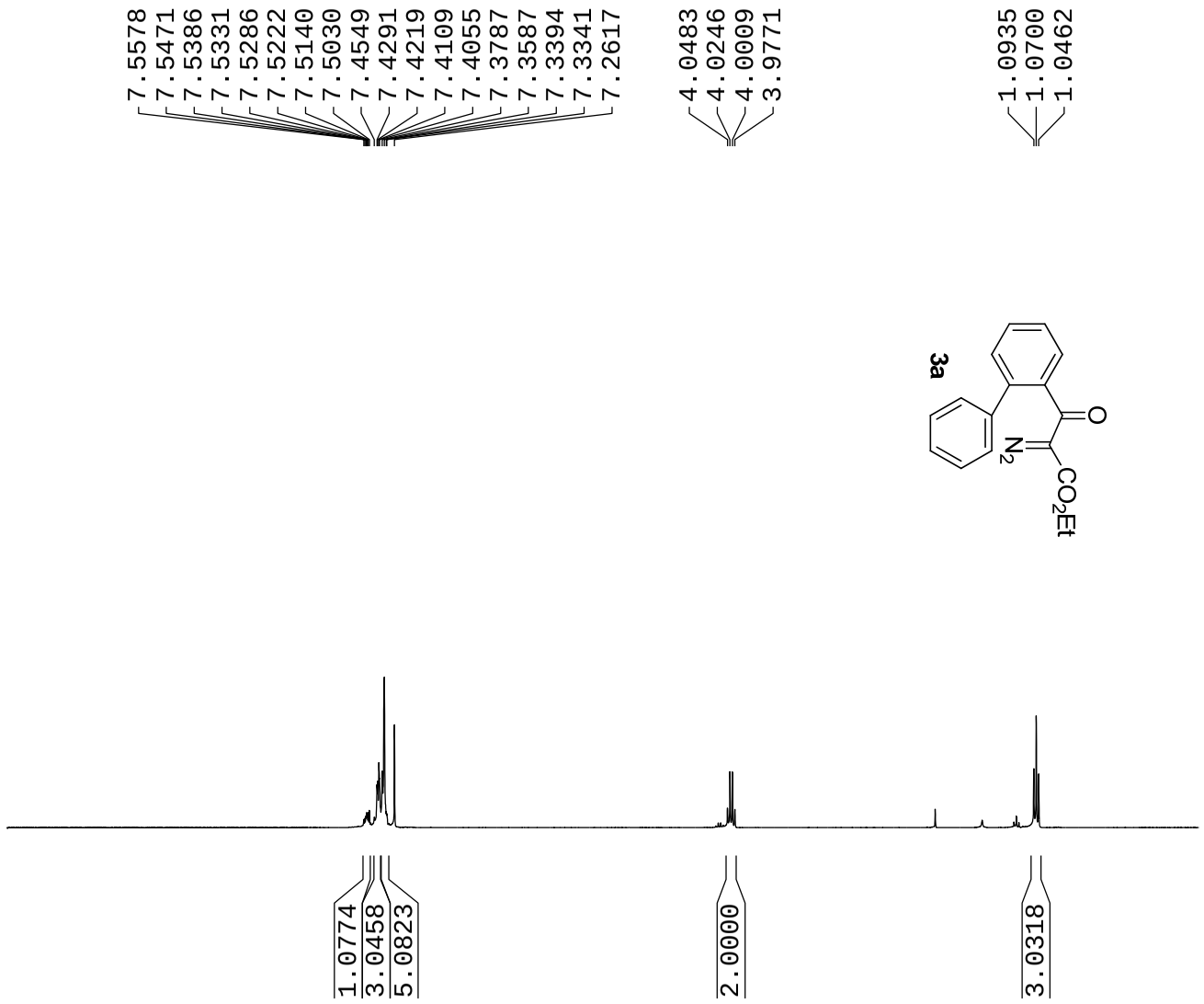
The titled compound was synthesized from **2k** by following the typical procedure. The reaction proceeded for 1 hour, and the chromatographic purification (hexane-ethyl acetate 50:1, 30:1, 10:1) gave **3k** as a pale yellow oil (93%). IR (neat) 3061, 2121, 1652, 1314, 773, 757, 693 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 1 H), 7.51 (s, 1 H), 7.50-7.48 (m, 1 H), 7.44 (s, 1 H), 7.42-7.41 (m, 1 H), 7.38-7.28 (m, 3 H), 2.41 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 189.7, 186.8, 140.5, 138.1, 137.3, 134.6, 131.4, 130.0, 129.9, 128.7, 128.6, 128.6, 127.8, 126.9, 86.6 ($C=N_2$), 28.8 ppm. HRMS-ESI: *m/z* [M + Na]$^+$ calcd. for $C_{16}H_{11}ClN_2O_2Na$: 321.0407; found: 321.0407.

**1-(2-(Benzo[*d*][1,3]dioxol-5-yl)phenyl)-2-diazobutane-1,3-dione (3l)**



**3l**

The titled compound was synthesized from **2l** by following the typical procedure. The reaction proceeded for 2 hours, and the chromatographic purification (hexane-ethyl acetate 40:1, 30:1, 10:1) gave **3l** as a yellow oil (61%). IR (neat) 3064, 2122, 1658, 1652, 1225, 872, 761 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 7.56-7.52 (m, 1 H), 7.48-7.38 (m, 3 H), 6.89 (s, 1 H), 6.86 (d, *J* = 8.0 Hz, 1 H), 6.82 (d, *J* = 8.0 Hz, 1 H), 6.02 (s, 2 H), 2.44 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 190.1, 187.4, 148.0, 148.0, 139.2, 137.2, 132.5, 131.3, 129.9, 127.8, 127.8, 122.7, 108.9, 108.5, 101.5, 86.4 ($C=N_2$), 28.9 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{17}H_{12}N_2O_4$: 308.0797; found: 308.0803.

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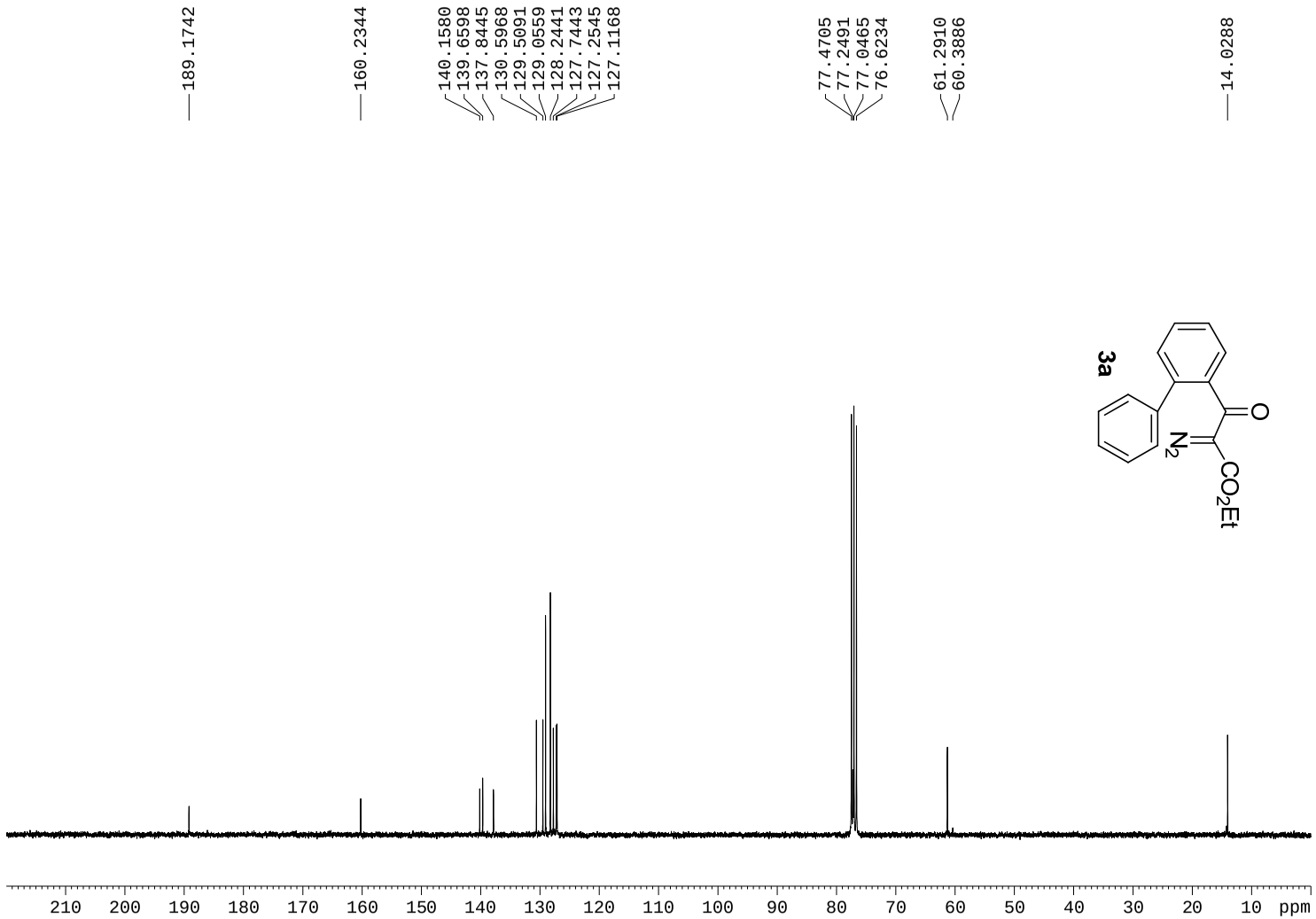


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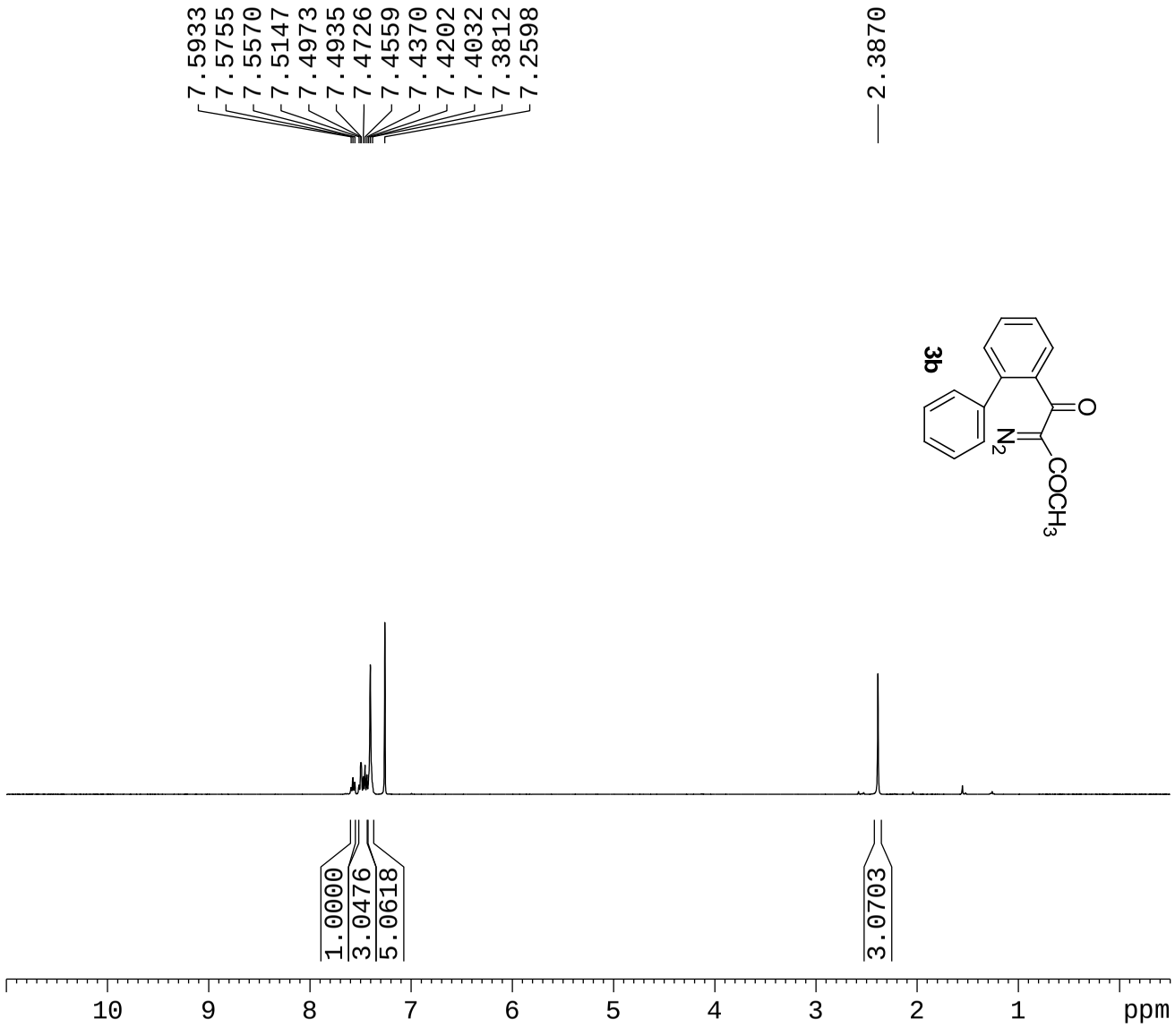


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PROCNO                 1

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TD0                    1

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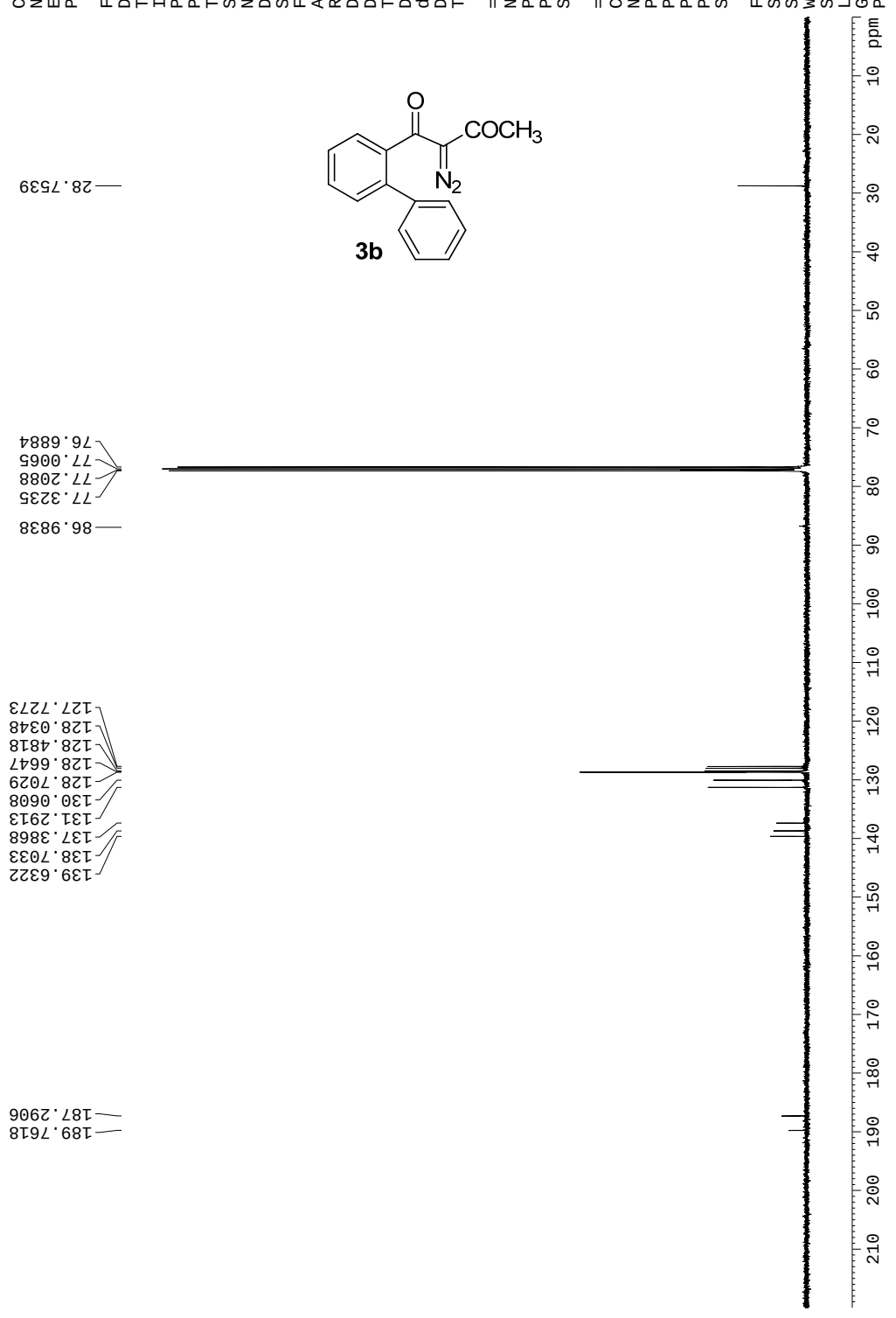
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SFO1 100.6243395 MHz
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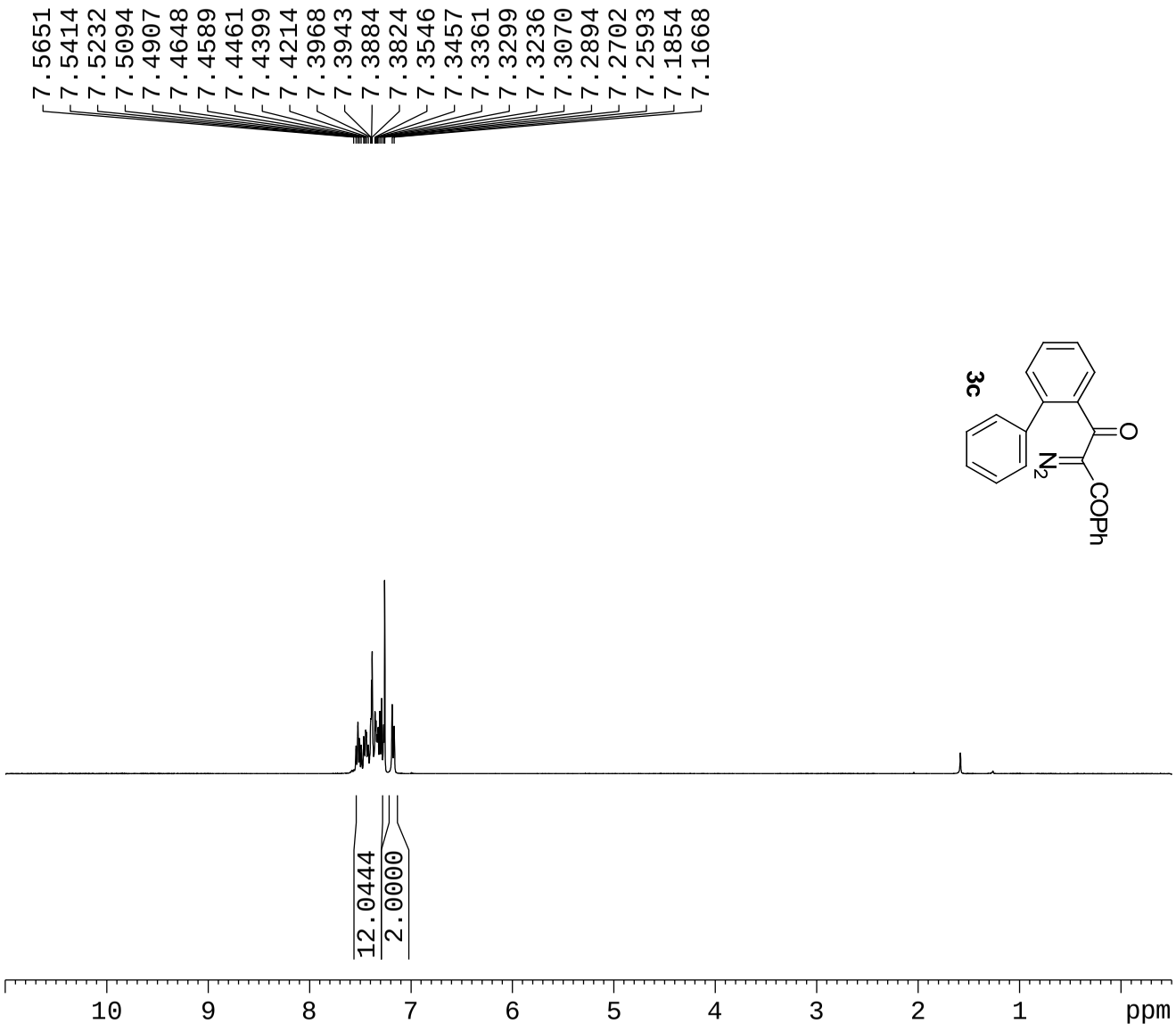


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TE                302.2 K
D1           0.10000000 sec
TD0                    1

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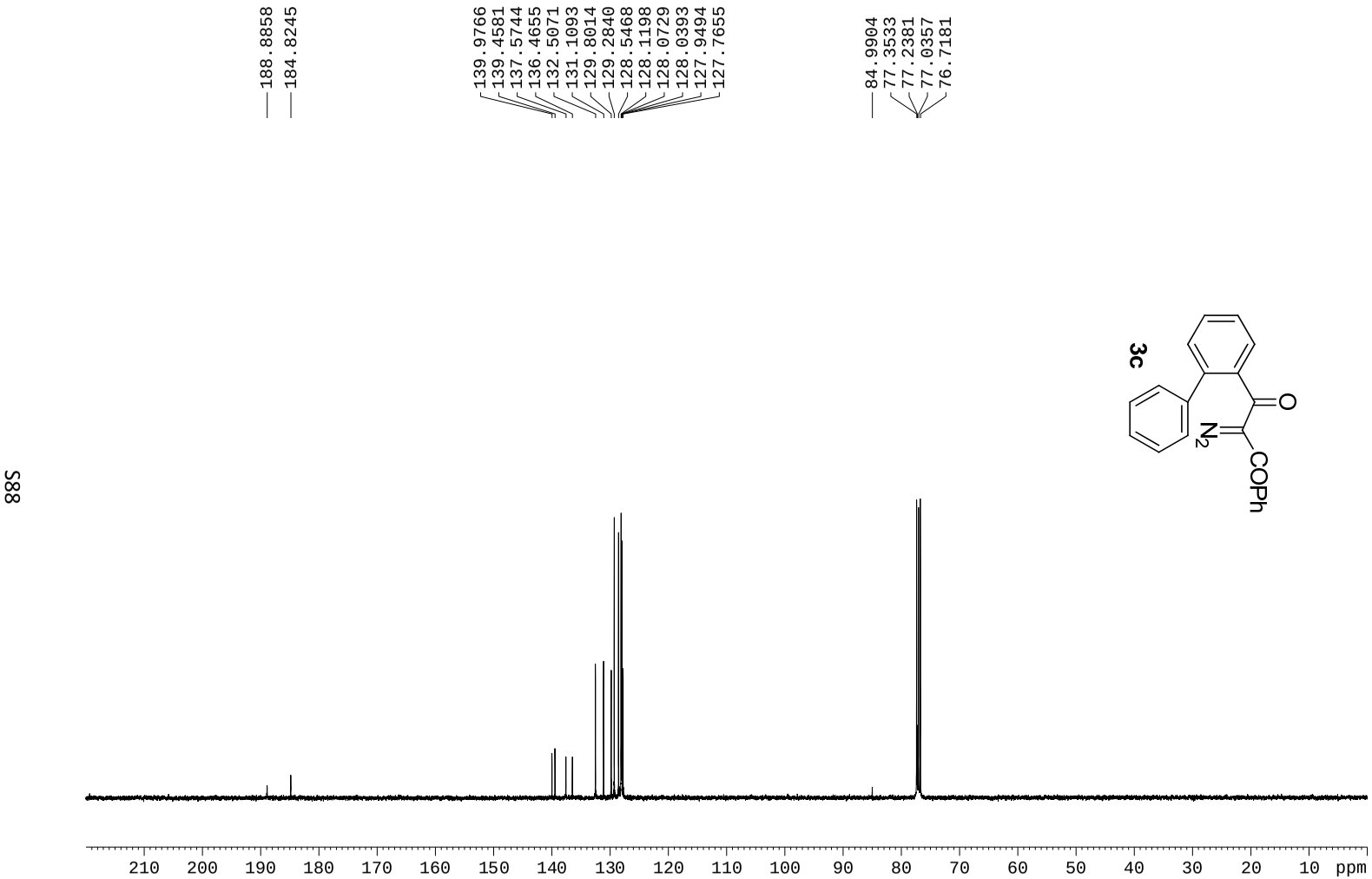


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TD0                    1

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P1                10.00 usec
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SFO1        100.6243395 MHz

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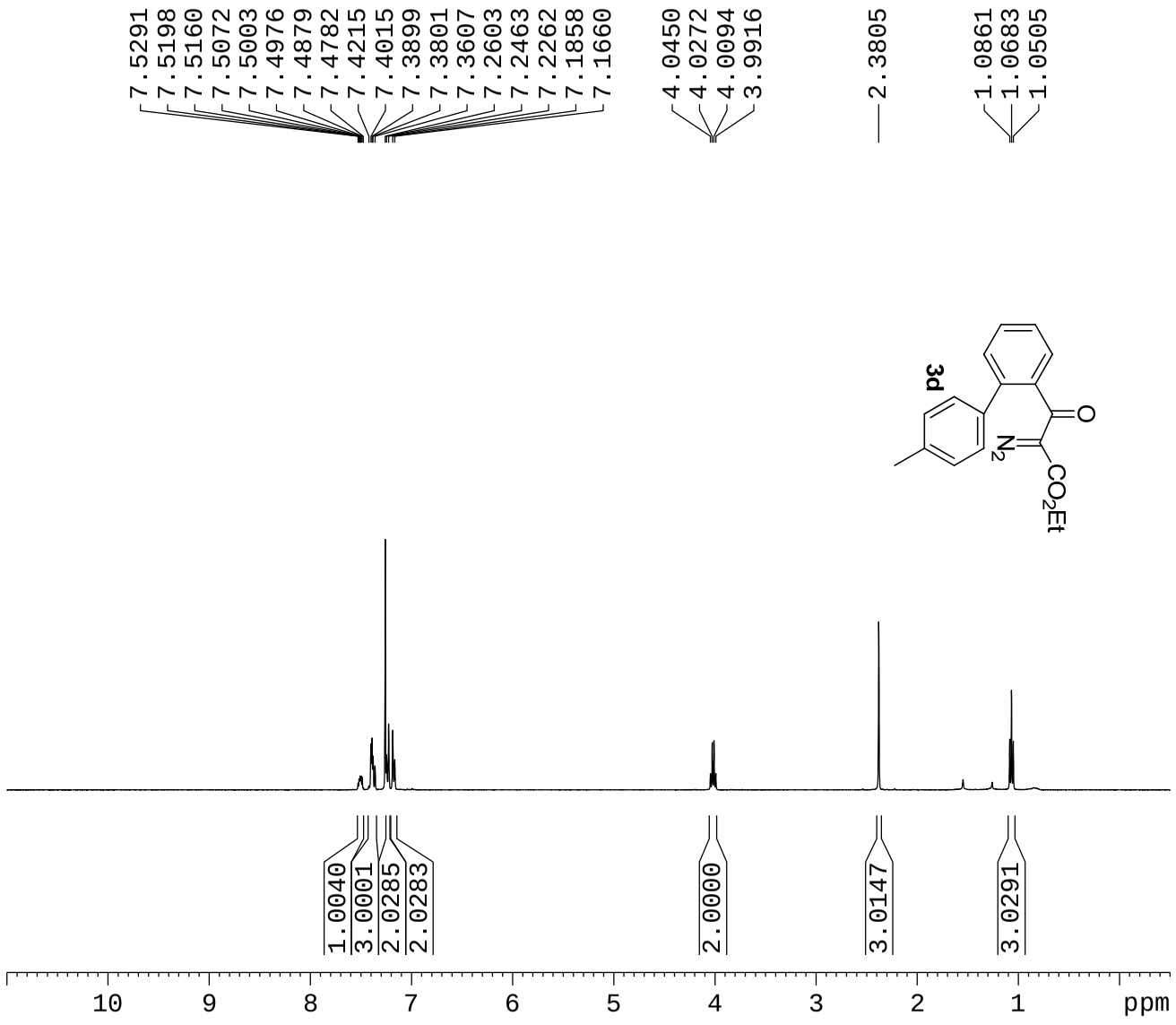


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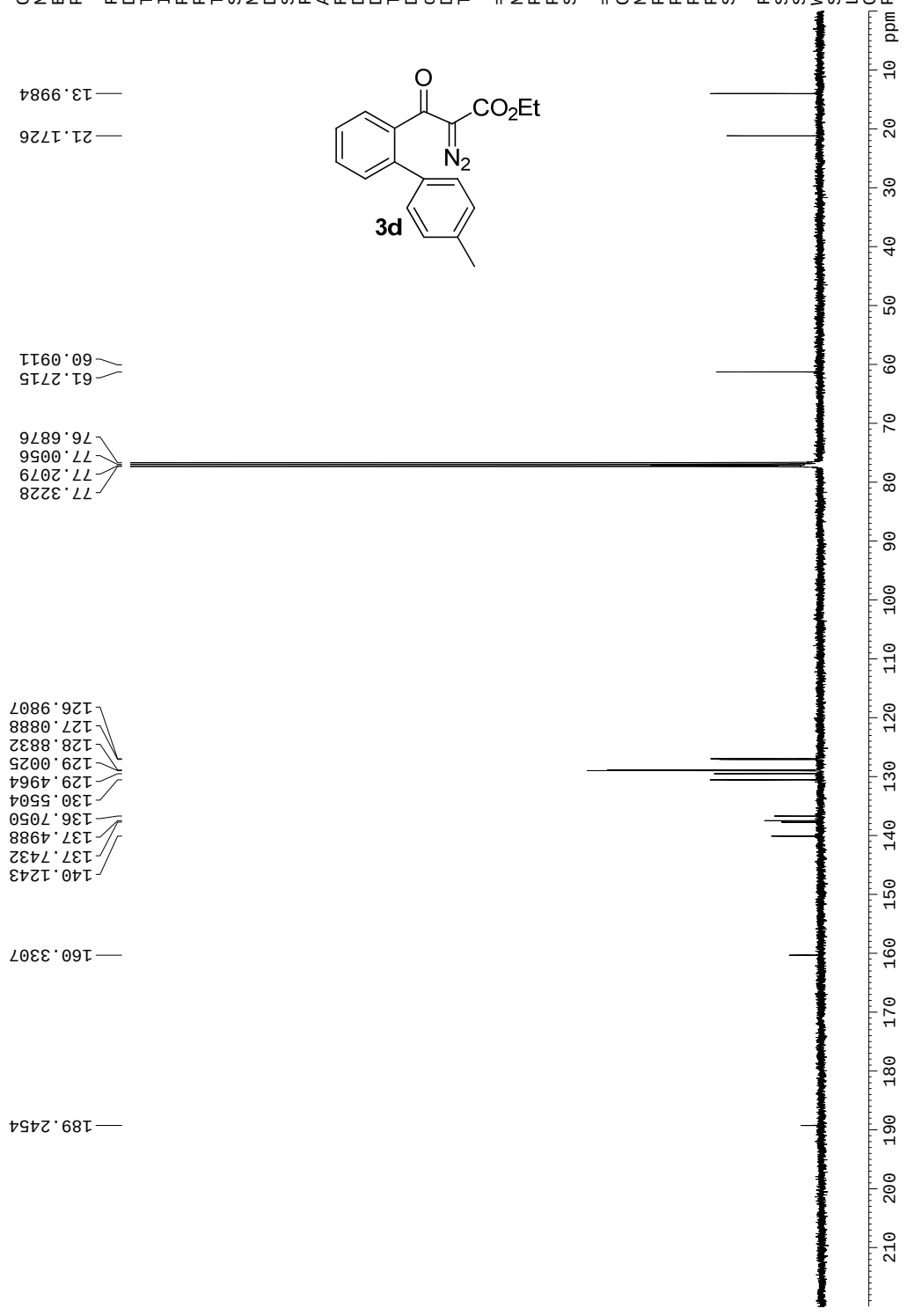
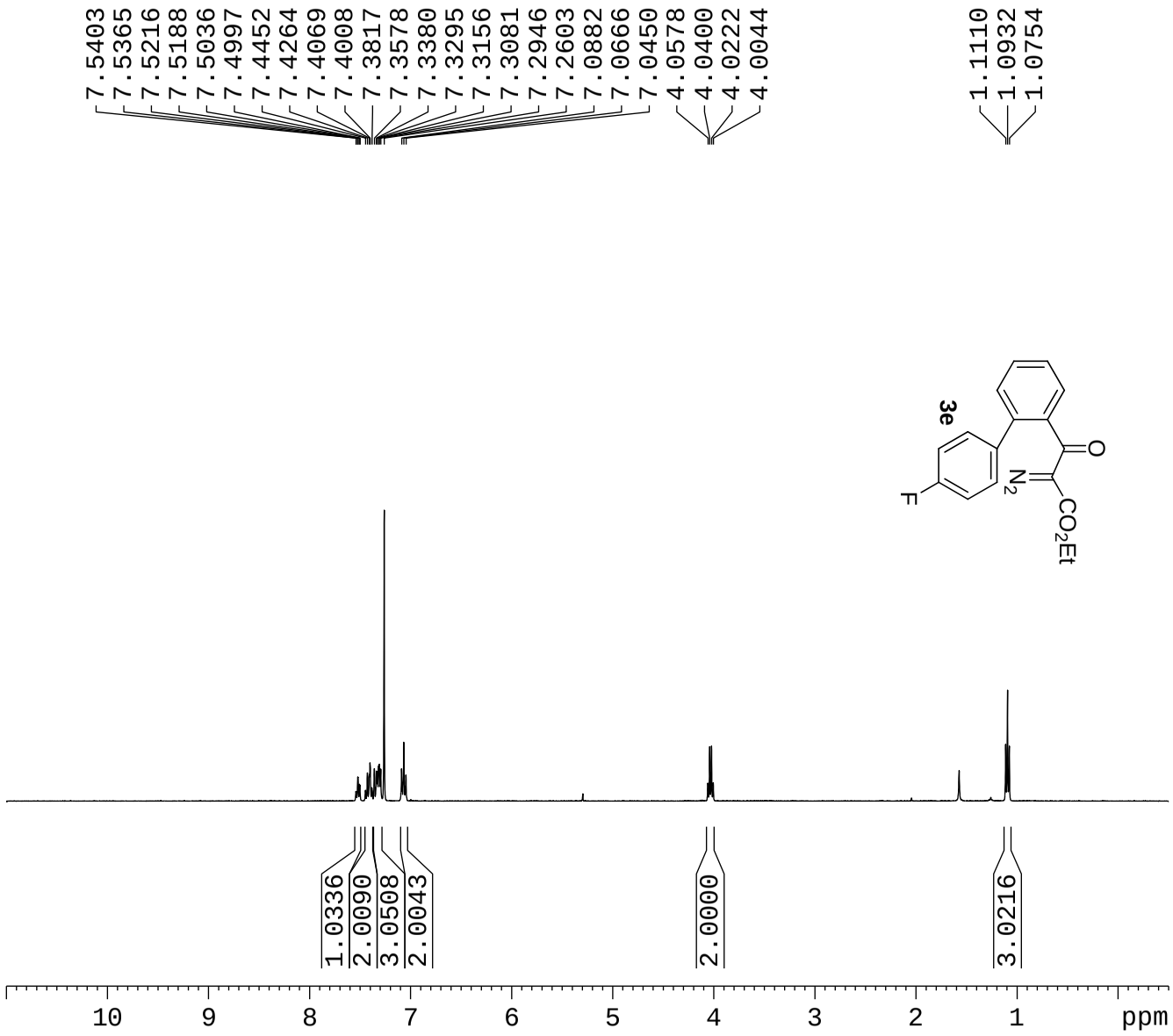
Time               11.52
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     10
DS                      0
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ               2.7263477 sec
RG                     406
DW                83.200 usec
DE                 6.50 usec
TE                 301.7 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PL1                 0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300092 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2017
EXPNO 927041
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170926
Time 12.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 50502
SOLVENT CDCl3
NS 1200
DS 0
SWH 25252.525 Hz
FIDRES 0.500030 Hz
AQ 0.9999896 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 301.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
1.40 PC
13.9984
21.1726
60.0911
61.2715
76.6876
77.0056
77.2079
77.3228
126.9807
127.0888
128.8832
129.0025
129.4964
130.5504
136.7050
137.4988
137.7432
140.1243
160.3307
189.2454
O
CO2Et
N2
3d
ppm




Current Data Parameters

| | |
|---|---|
| NAME | chiang2017 |
| EXPNO | 1109011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

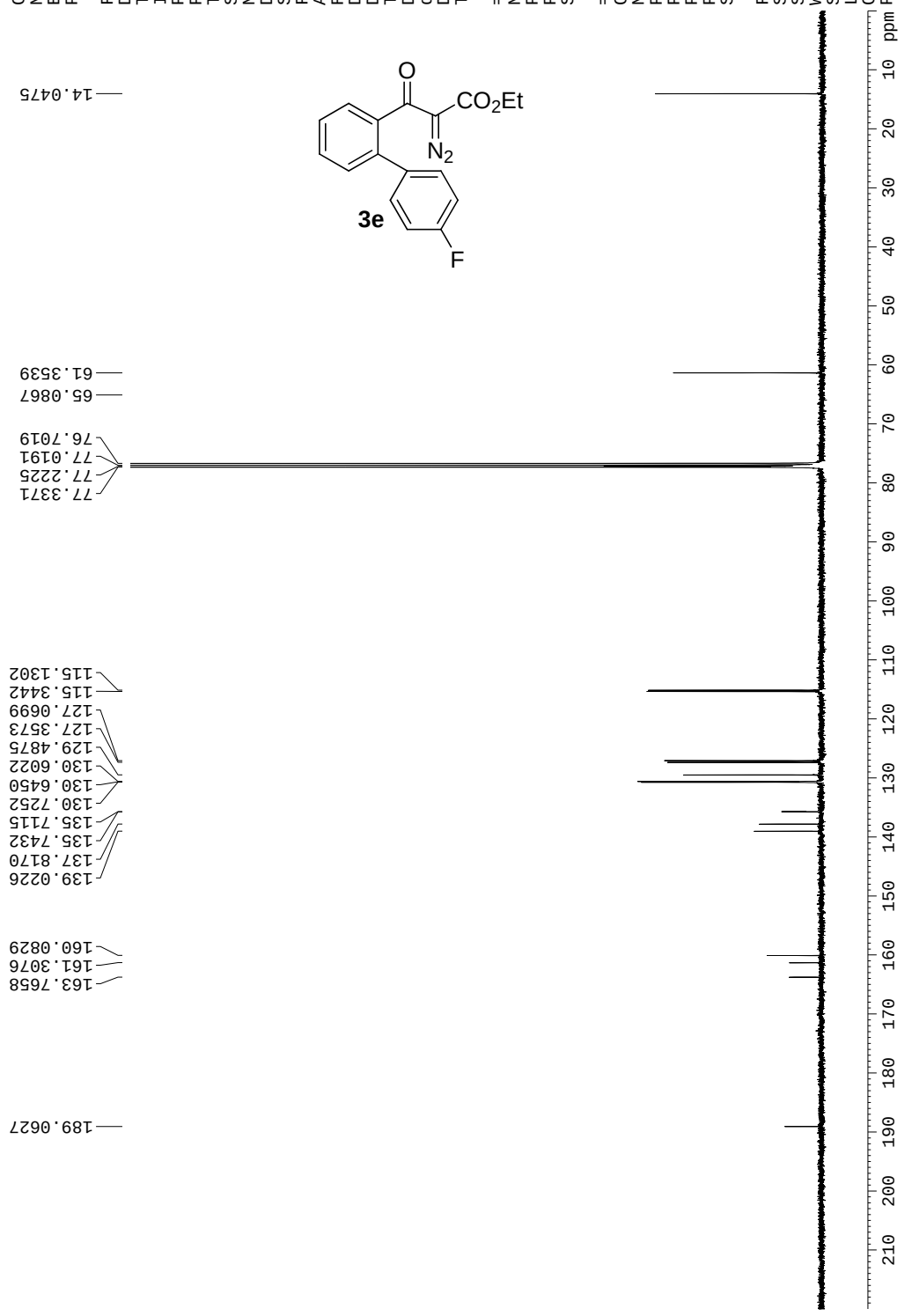
| | |
|---|---|
| Date_ | 20171109 |
| Time | 9.52 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 26 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 645 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 300.8 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

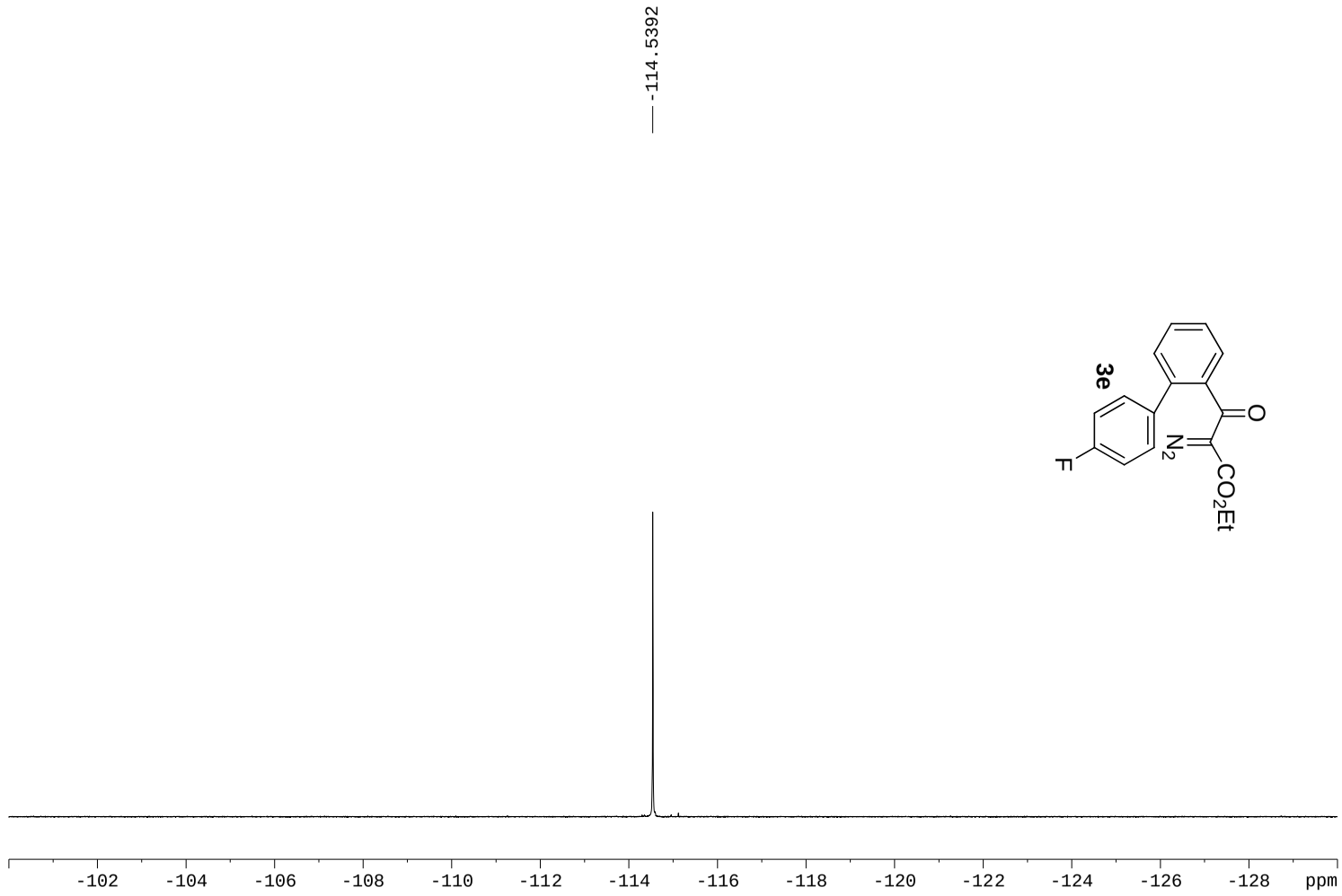
======== CHANNEL f1 ========

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300091 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |


Current Data Parameters
NAME chiang2017
EXPNO 1109021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171109
Time 9.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2202
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 300.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
14.0475
61.3539
65.0867
76.7019
77.0191
77.2225
77.3371
115.1302
115.3442
127.0699
127.3573
129.4875
130.6022
130.6450
130.7252
135.7115
135.7432
137.8170
139.0226
160.0829
161.3076
163.7658
189.0627
O
CO2Et
N2
3e
F
ppm



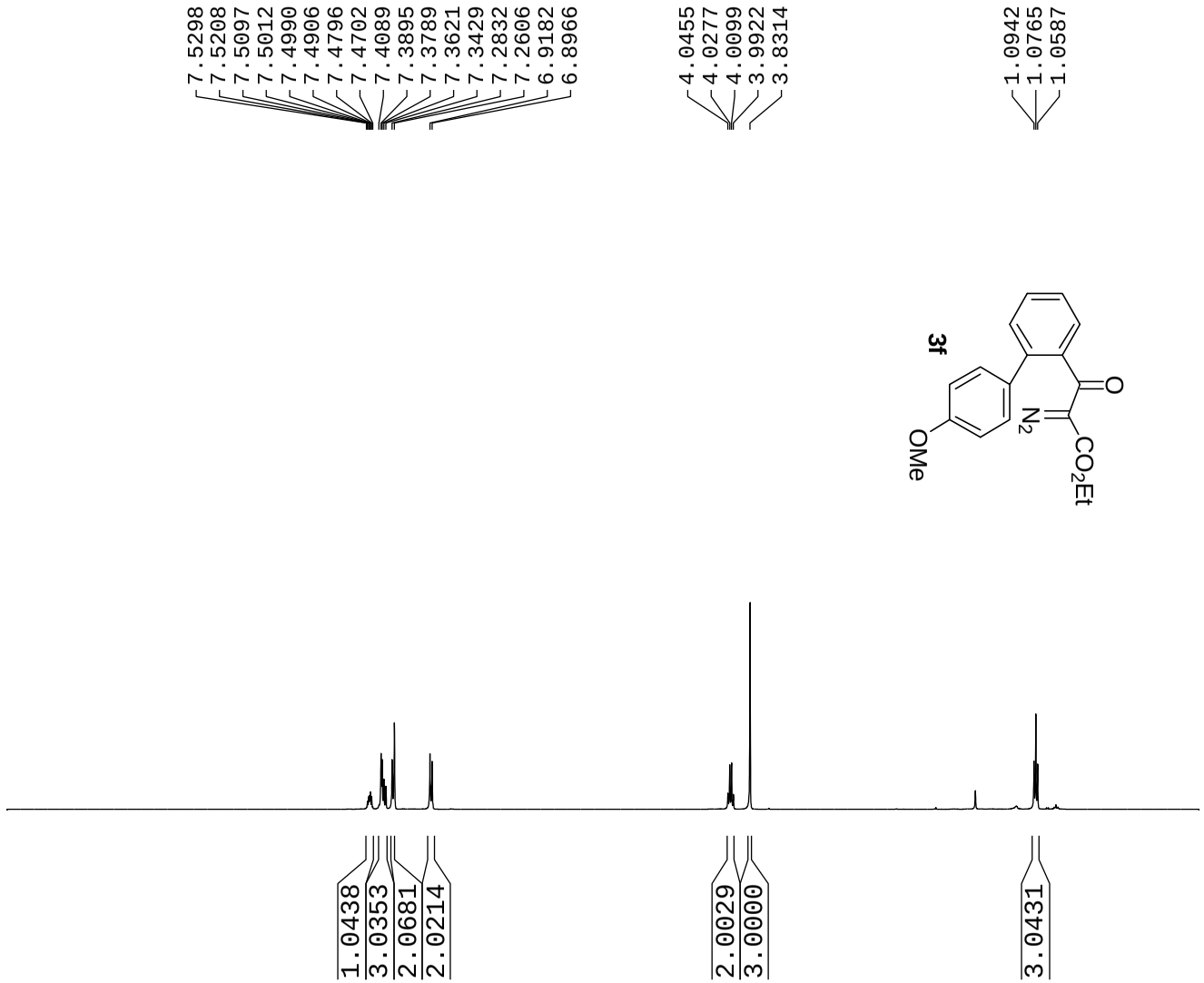

```
Current Data Parameters
NAME         chiang2017
EXPNO           1109031
PROCNO                1

F2 - Acquisition Parameters
Date_          20171109
Time              12.02
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG        zgfhigqn
TD              131072
SOLVENT            CDCl3
NS                    12
DS                     0
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340532 sec
RG                 2050
DW                 5.600 usec
DE                 6.50 usec
TE                301.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                 20.00 usec
PL1                 2.50 dB
SFO1         376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF           376.4983660 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

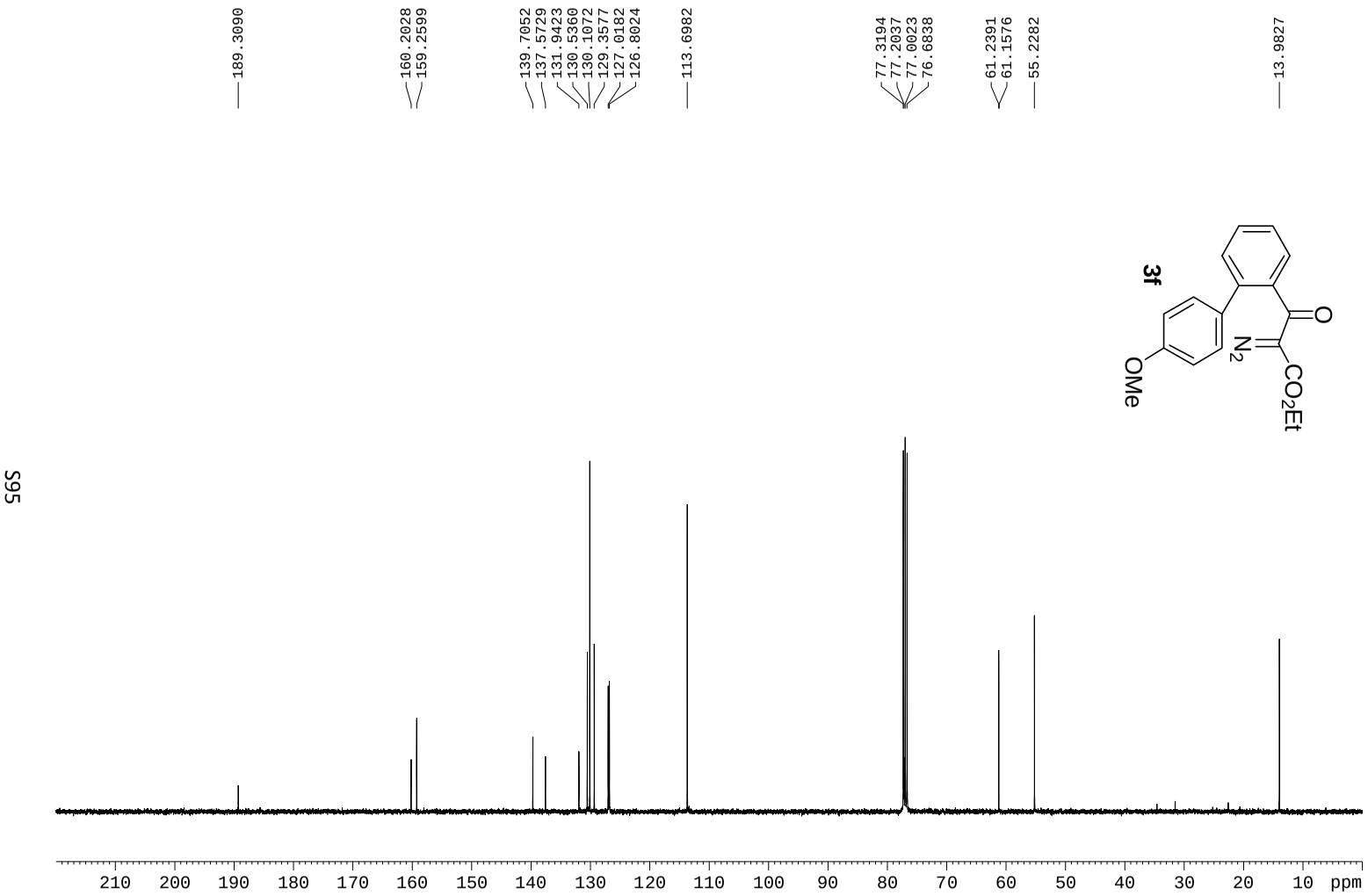



```
Current Data Parameters
NAME                  zhu2018
EXPNO                  806011
PROCNO                      1

F2 - Acquisition Parameters
Date_               20180806
Time                   16.12
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         0
SWH                 6009.615 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                       161
DW                    83.200 usec
DE                     6.50 usec
TE                     296.4 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PL1                     0.90 dB
SFO1             400.1326008 MHz

F2 - Processing parameters
SI                     16384
SF               400.1300090 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



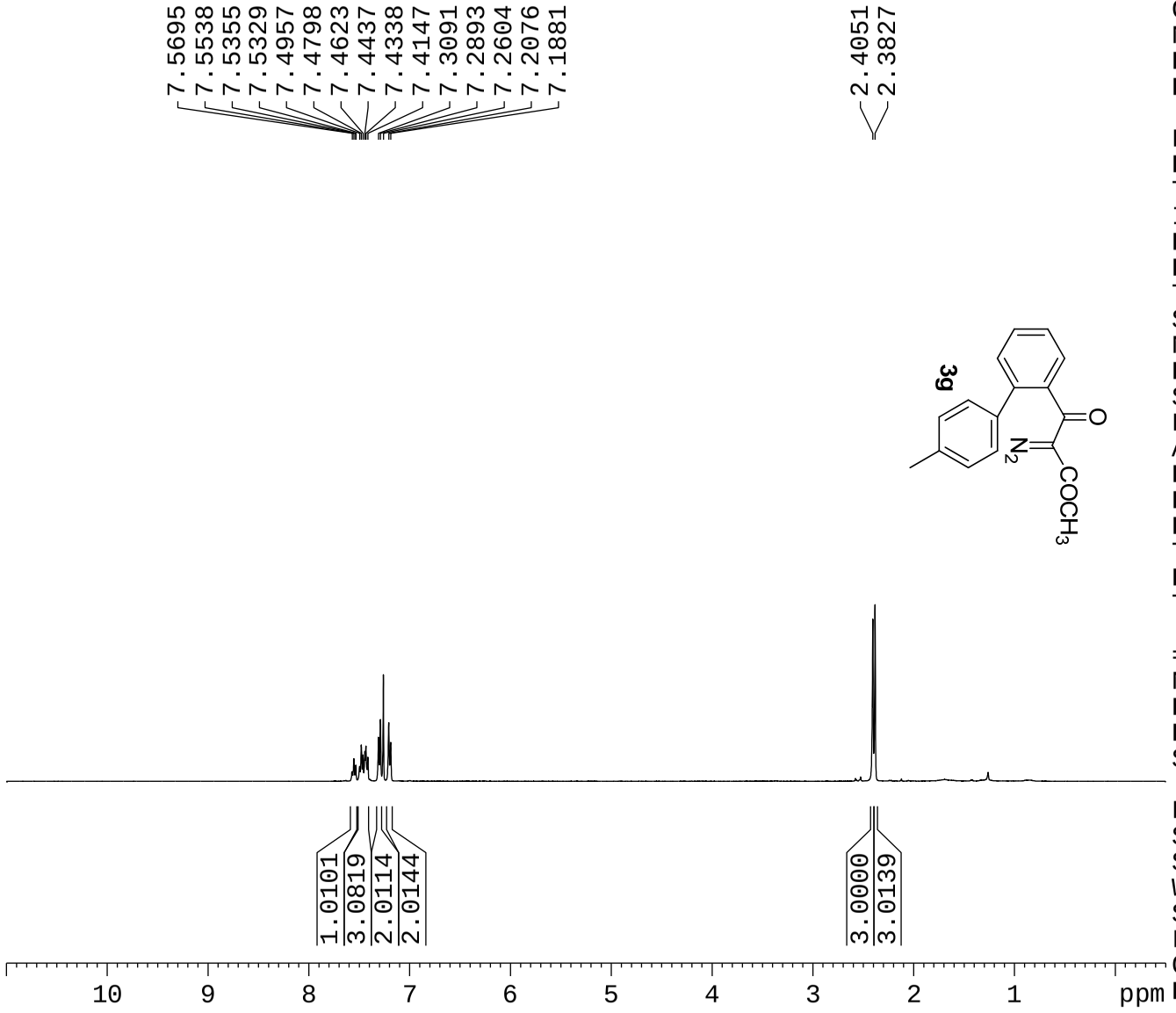

```
Current Data Parameters
NAME                  zhu2018
EXPNO                  806012
PROCNO                      1

F2 - Acquisition Parameters
Date_               20180806
Time                   16.19
INSTRUM                spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       602
DS                         0
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                 1.2976629 sec
RG                      1290
DW                    19.800 usec
DE                     6.50 usec
TE                    296.7 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     10.00 usec
PL1                     6.20 dB
SFO1             100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                  90.00 usec
PL2                    -0.40 dB
PL12                   15.80 dB
PL13                   18.50 dB
SFO2             400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF               100.6127757 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

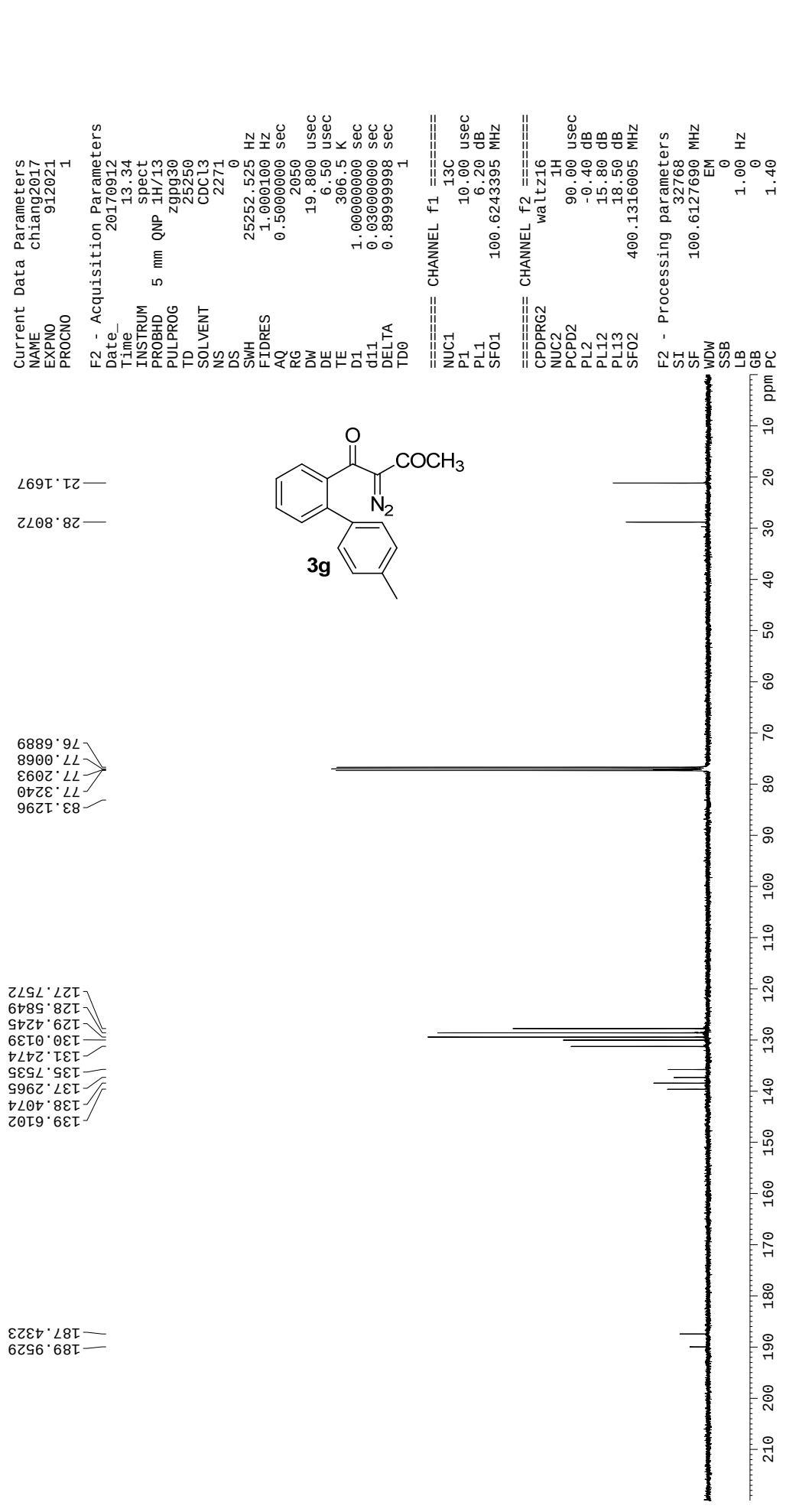


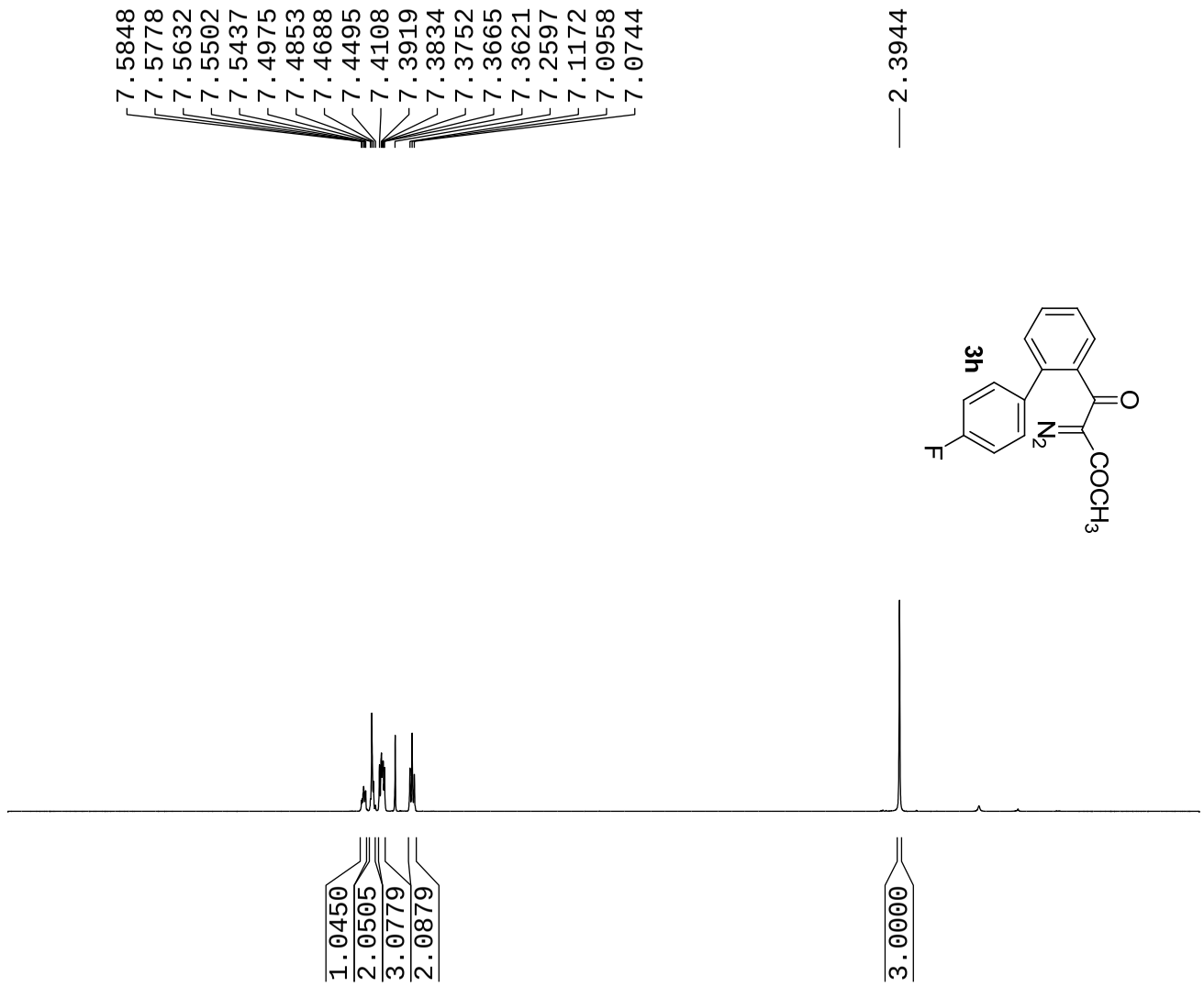

```
Current Data Parameters
NAME          chiang2017
EXPNO             912011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170912
Time               13.28
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    17
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                306.5 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300089 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2017
EXPNO 912021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170912
Time 13.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 2271
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 306.5 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
COCH3
N2
3g
21.1697
28.8072
76.6889
77.0068
77.2093
77.3240
83.1296
127.7572
128.5849
129.4245
130.0139
131.2474
135.7535
137.2965
138.4074
139.6102
187.4323
189.9529
ppm

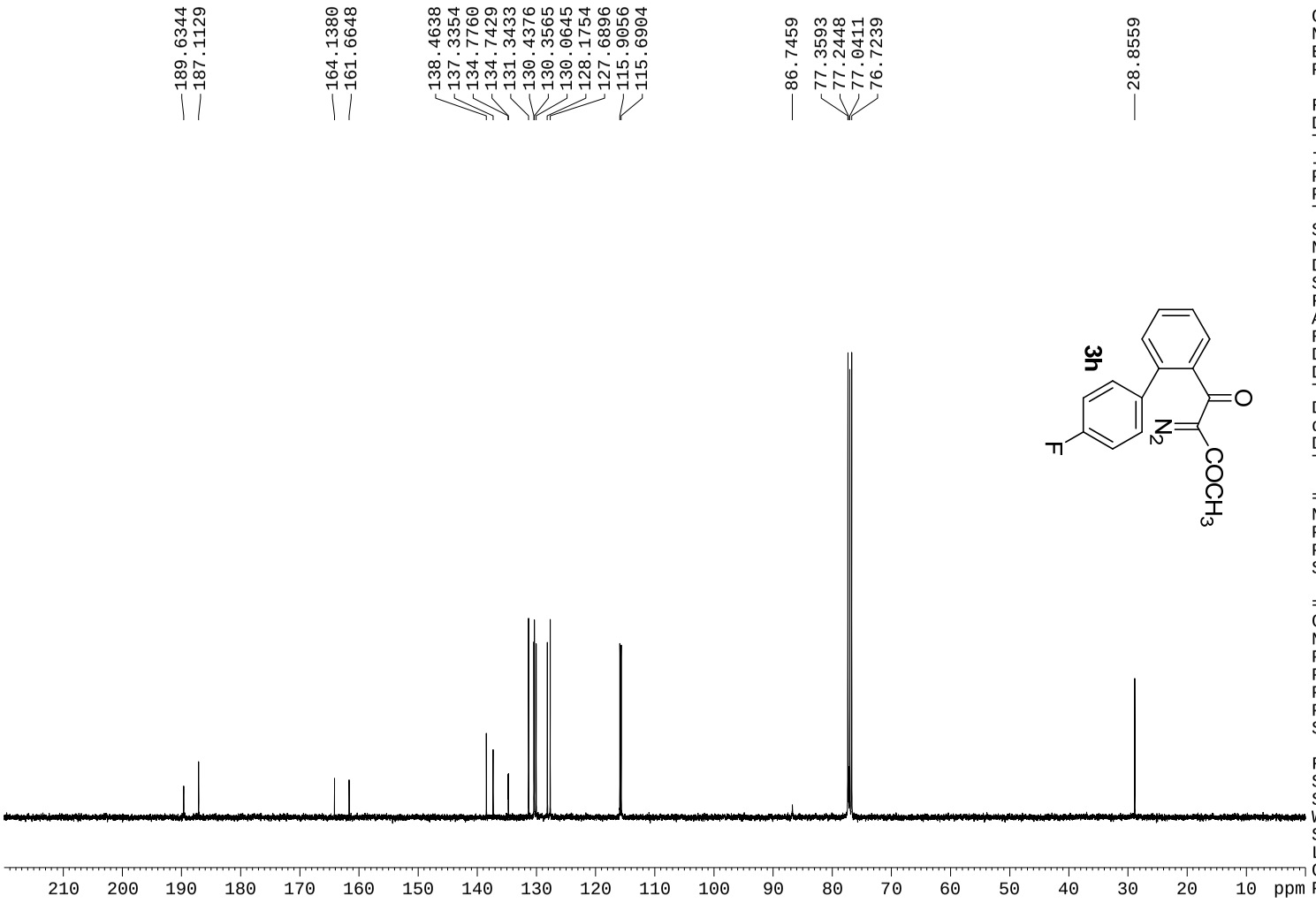



```
Current Data Parameters
NAME          chiang2017
EXPNO            1028011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171028
Time               10.45
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    17
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   406
DW                83.200 usec
DE                 6.50 usec
TE                299.2 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300093 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



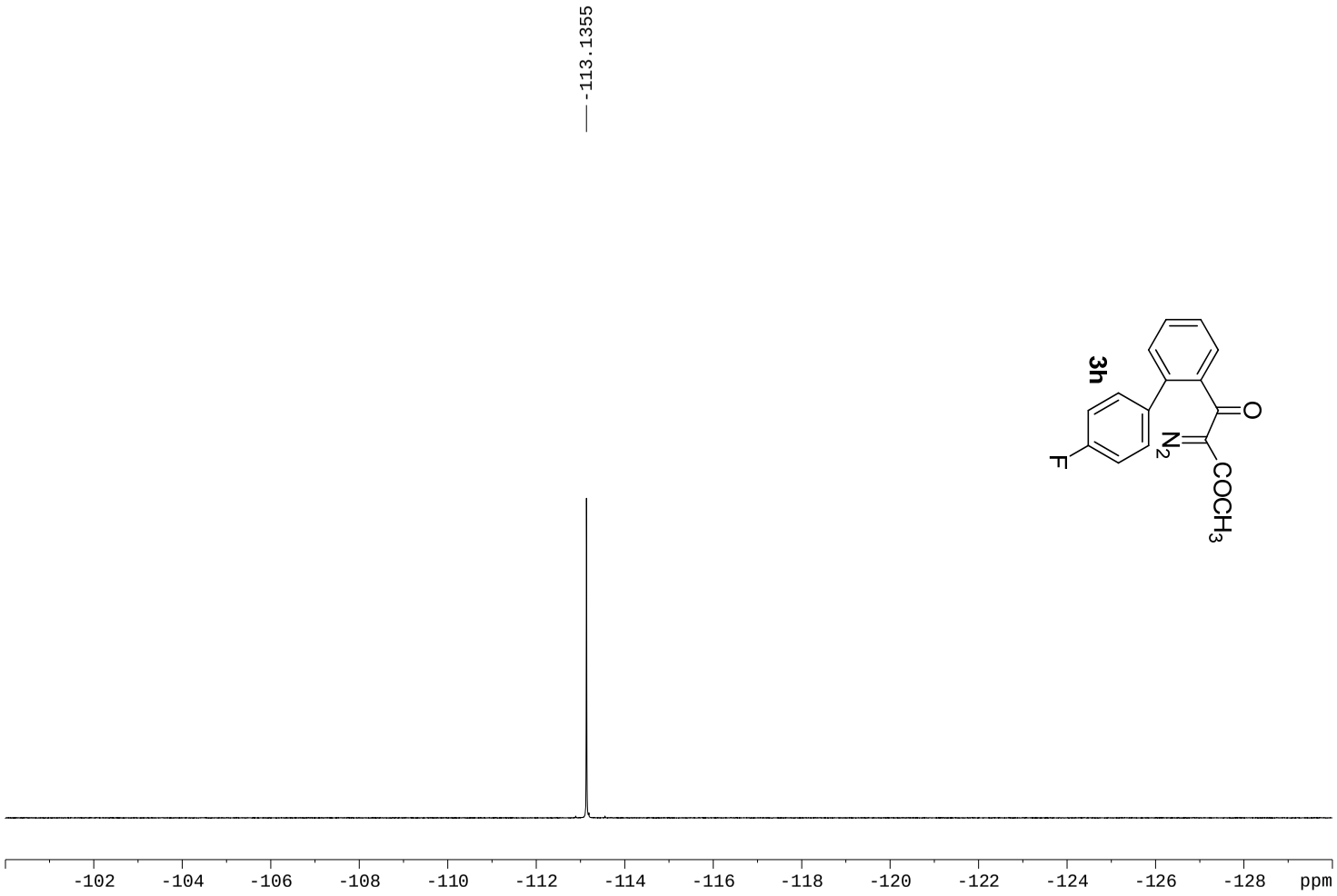

```
Current Data Parameters
NAME          chiang2017
EXPNO            1028021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171028
Time               10.50
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                299.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```







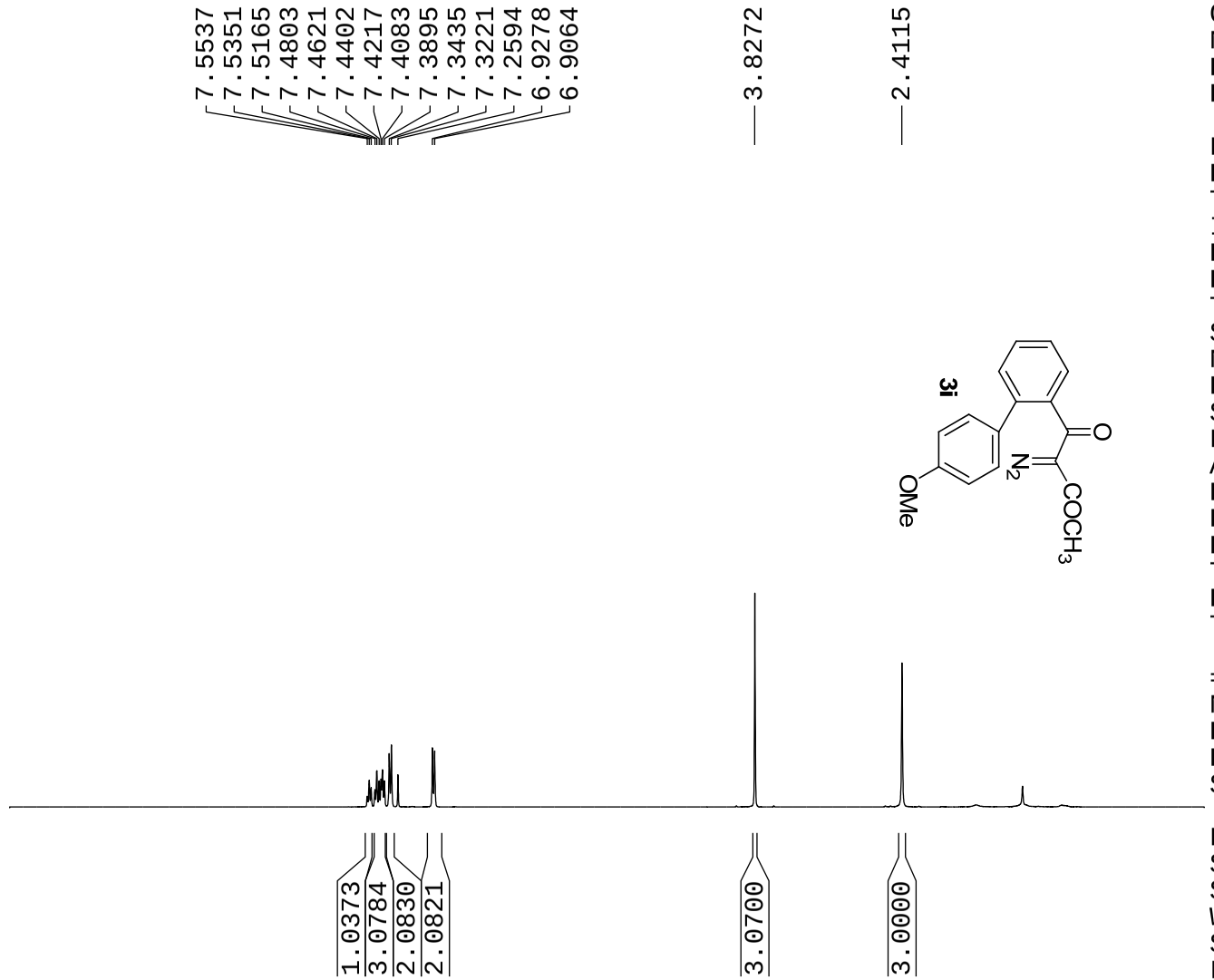
```
Current Data Parameters
NAME         chiang2017
EXPNO           1028031
PROCNO                1

F2 - Acquisition Parameters
Date_          20171028
Time              11.18
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG        zgfhigqn
TD              131072
SOLVENT            CDCl3
NS                    14
DS                     0
SWH           89285.711 Hz
FIDRES         0.681196 Hz
AQ            0.7340532 sec
RG                 2050
DW                5.600 usec
DE                6.50 usec
TE                298.2 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                  20.00 usec
PL1                  2.50 dB
SFO1        376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          376.4983660 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```







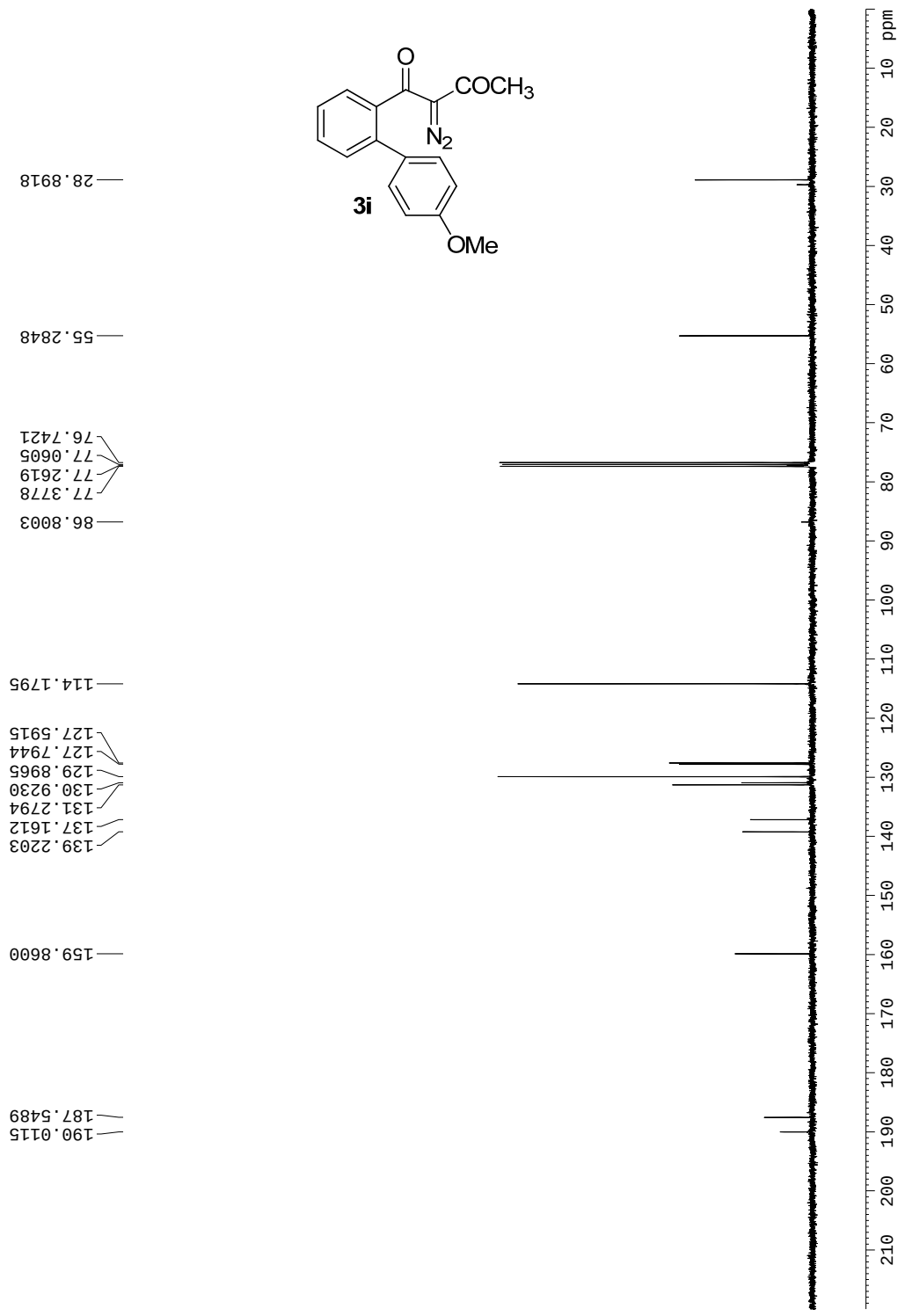
S101

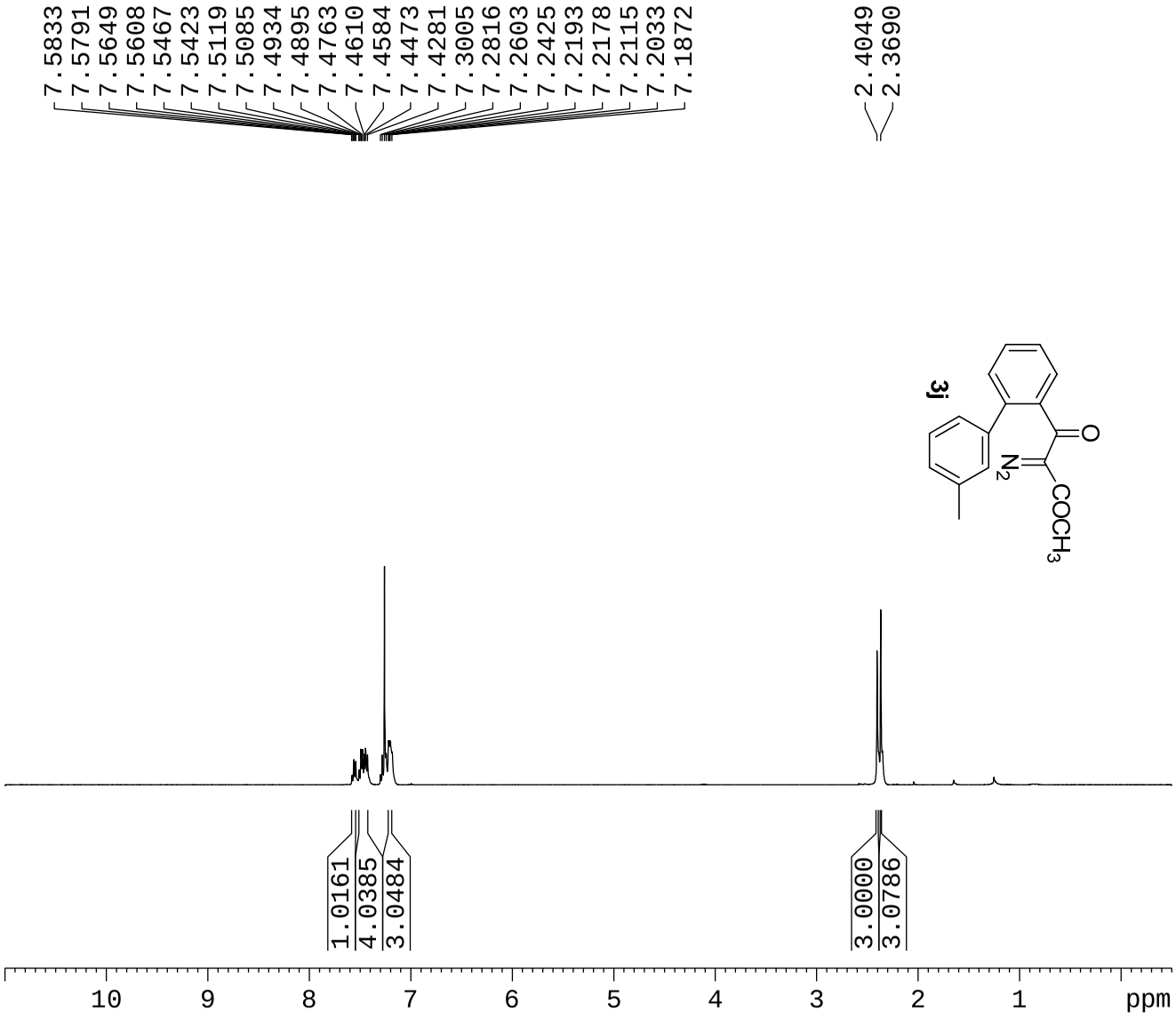
Current Data Parameters
NAME chiang2017
EXPNO 1215011
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171215
Time 14.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 287
DW 83.200 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME chiang2017
EXPNO 1215021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171215
Time_ 14.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 165
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
COCH3
N2
3i
OMe
28.8918
55.2848
76.7421
77.0605
77.2619
77.3778
86.8003
114.1795
127.5915
127.7944
129.8965
130.9230
131.2794
137.1612
139.2203
159.8600
187.5489
190.0115
ppm

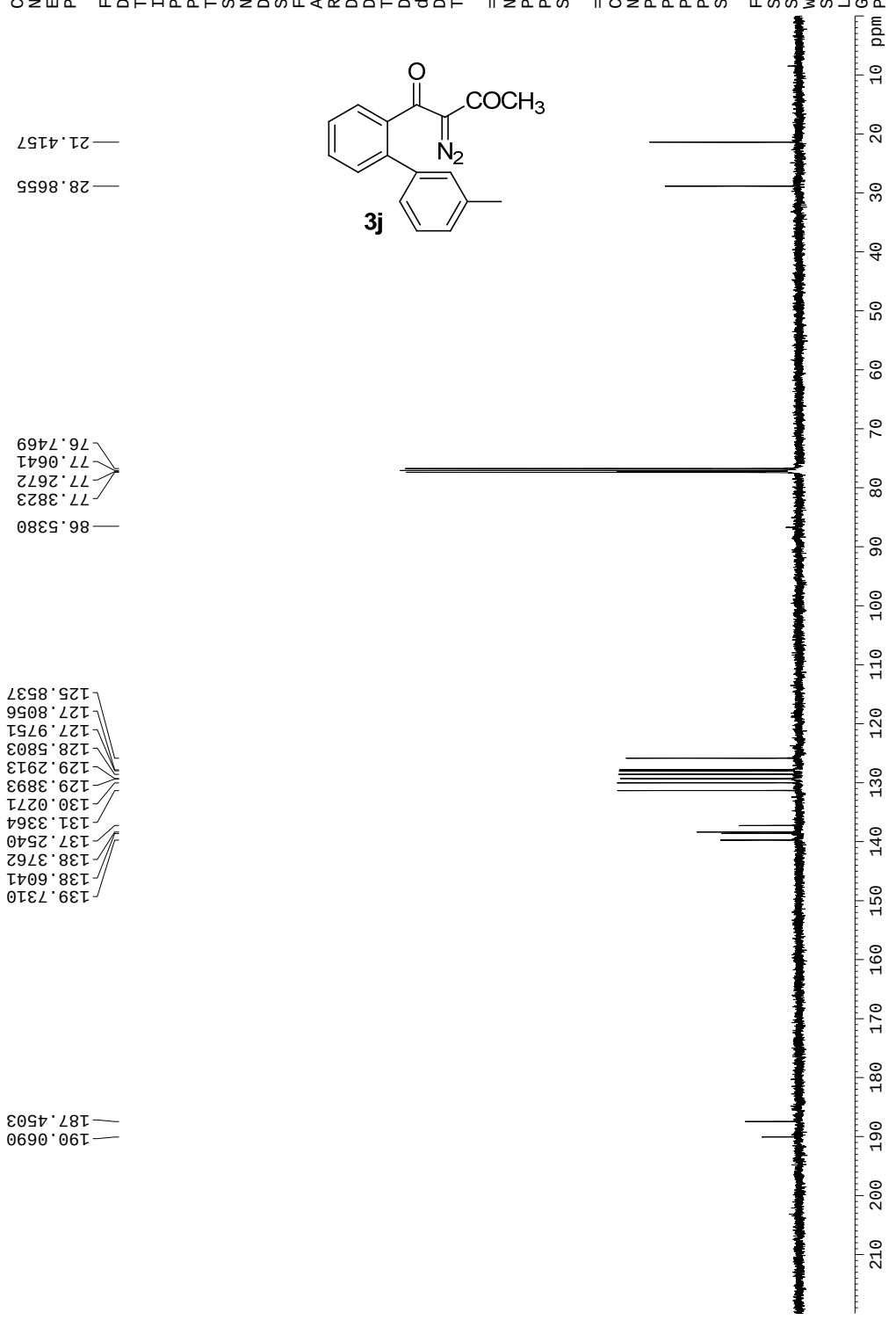


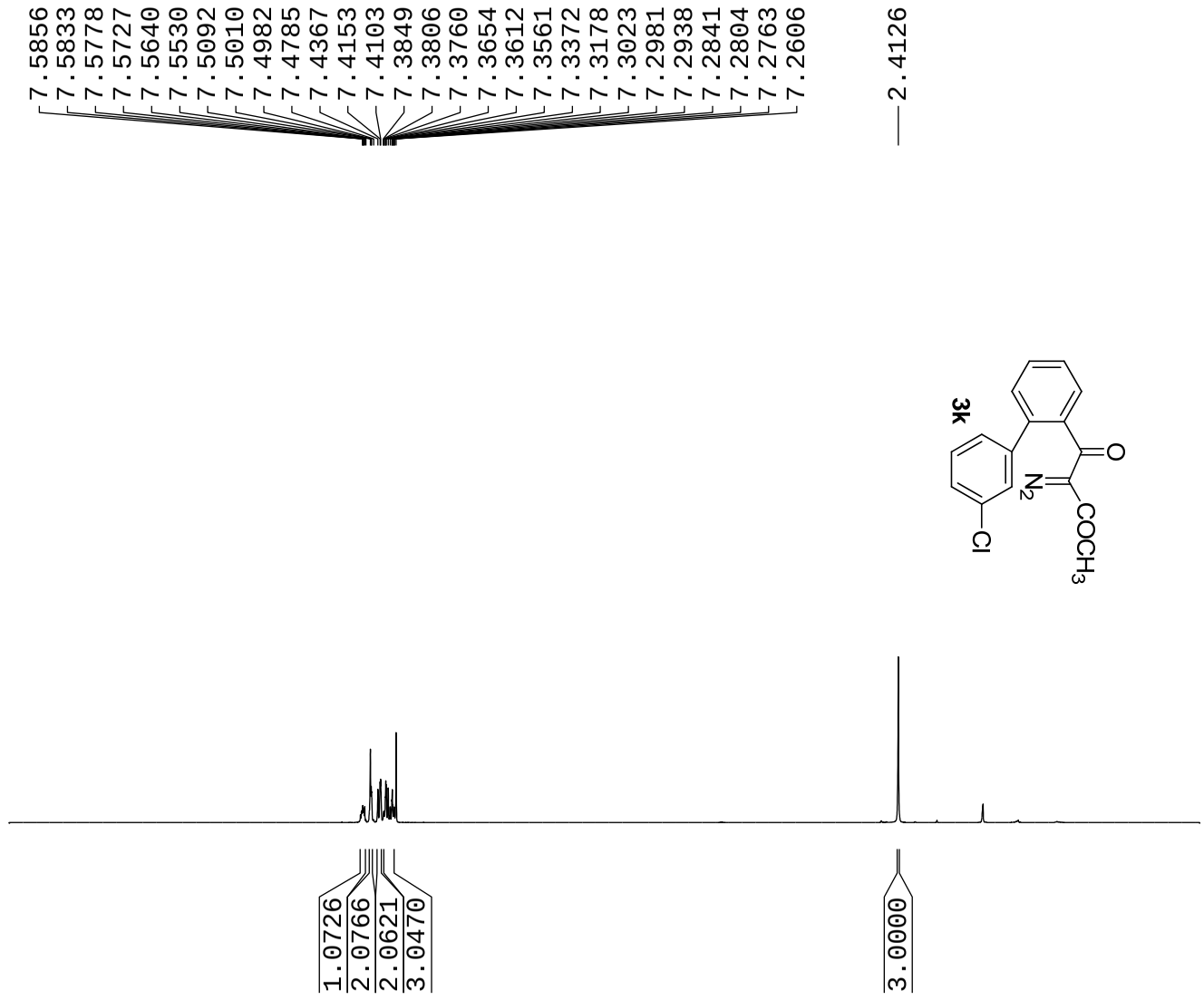

```
Current Data Parameters
NAME          chiang2018
EXPNO             208011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180208
Time               11.24
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7263477 sec
RG                   287
DW                83.200 usec
DE                 6.50 usec
TE                291.9 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2018
EXPNO 208021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180208
Time 11.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 250
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 292.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
COCH3
N2
3j
21.4157
28.8655
76.7469
77.0641
77.2672
77.3823
86.5380
125.8537
127.8056
127.9751
128.5803
129.2913
129.3893
130.0271
131.3364
137.2540
138.3762
138.6041
139.7310
187.4503
190.0690
ppm




Current Data Parameters

| | |
|---|---|
| NAME | chiang2018 |
| EXPNO | 303011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20180303 |
| Time | 14.27 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 23 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 575 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 300.3 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

| | |
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| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
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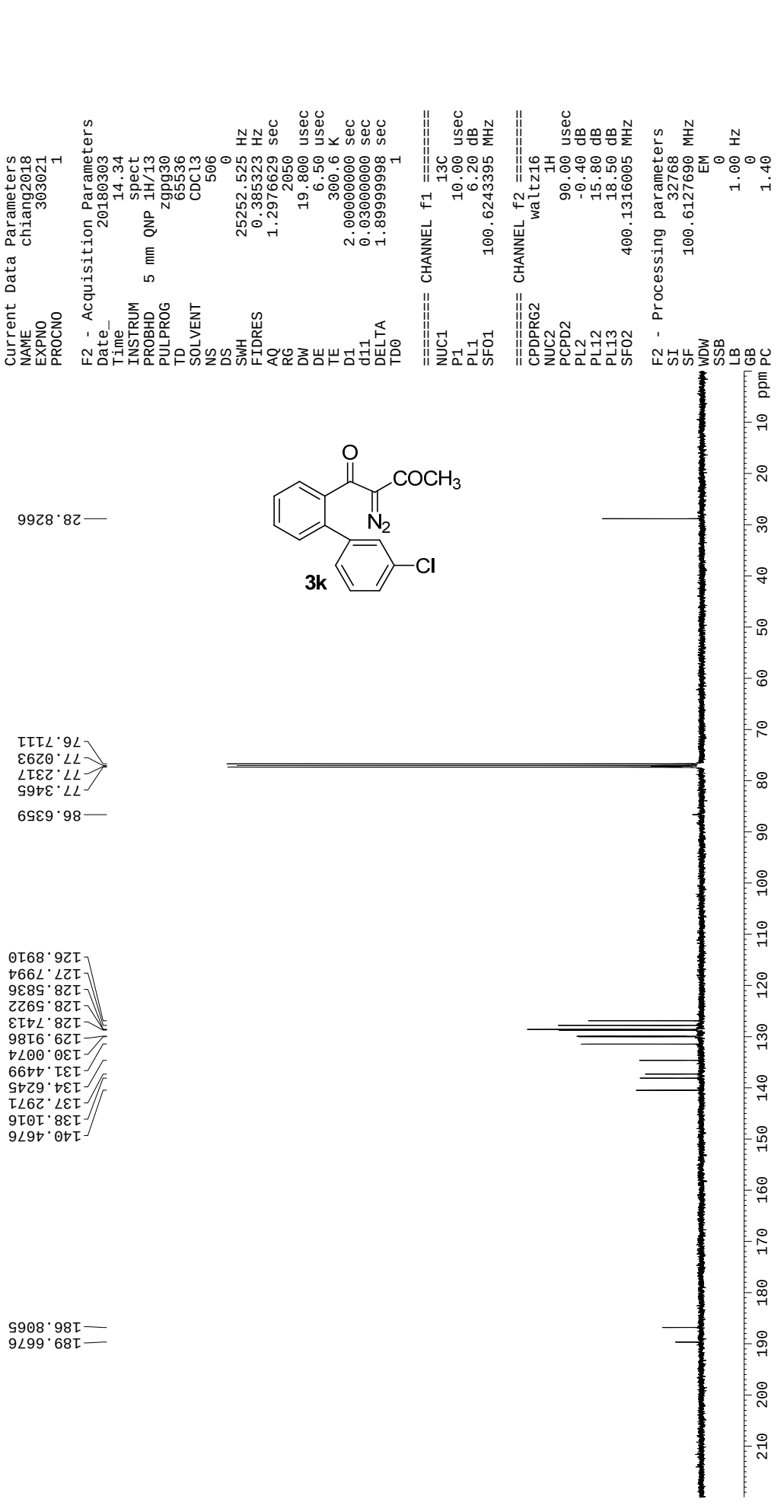
| SF | 400.1300089 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |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NAME chiang2018
EXPNO 303021
PROCNO 1
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Time 14.34
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 506
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 300.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
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P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
COCH3
N2
Cl
3k
28.8266
76.7111
77.0293
77.2317
77.3465
86.6359
126.8910
127.7994
128.5836
128.5922
128.7413
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130.0074
131.4499
134.6245
137.2971
138.1016
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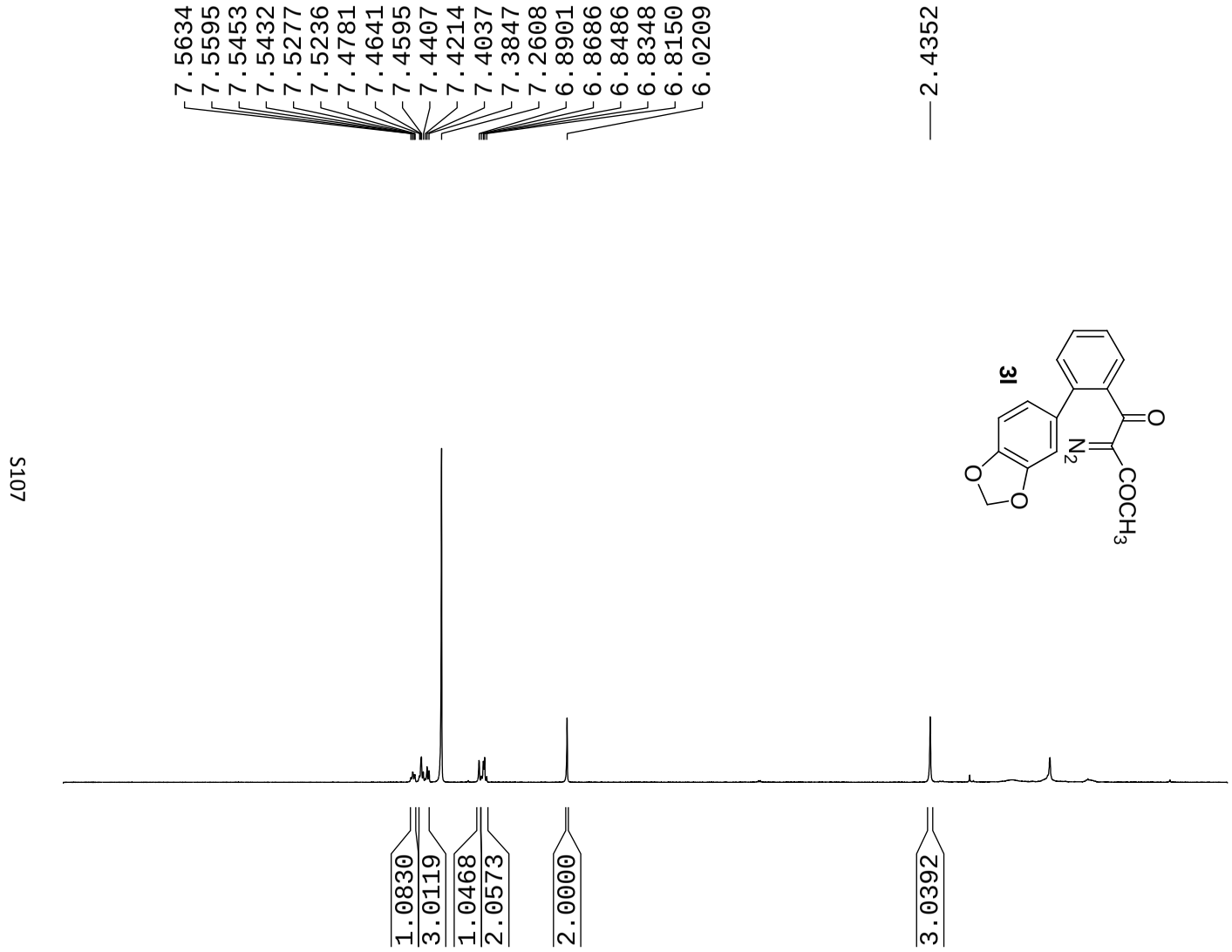
189.6676
ppm

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EXPNO             402011
PROCNO                 1

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INSTRUM            spect
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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    52
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7263477 sec
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TE                296.9 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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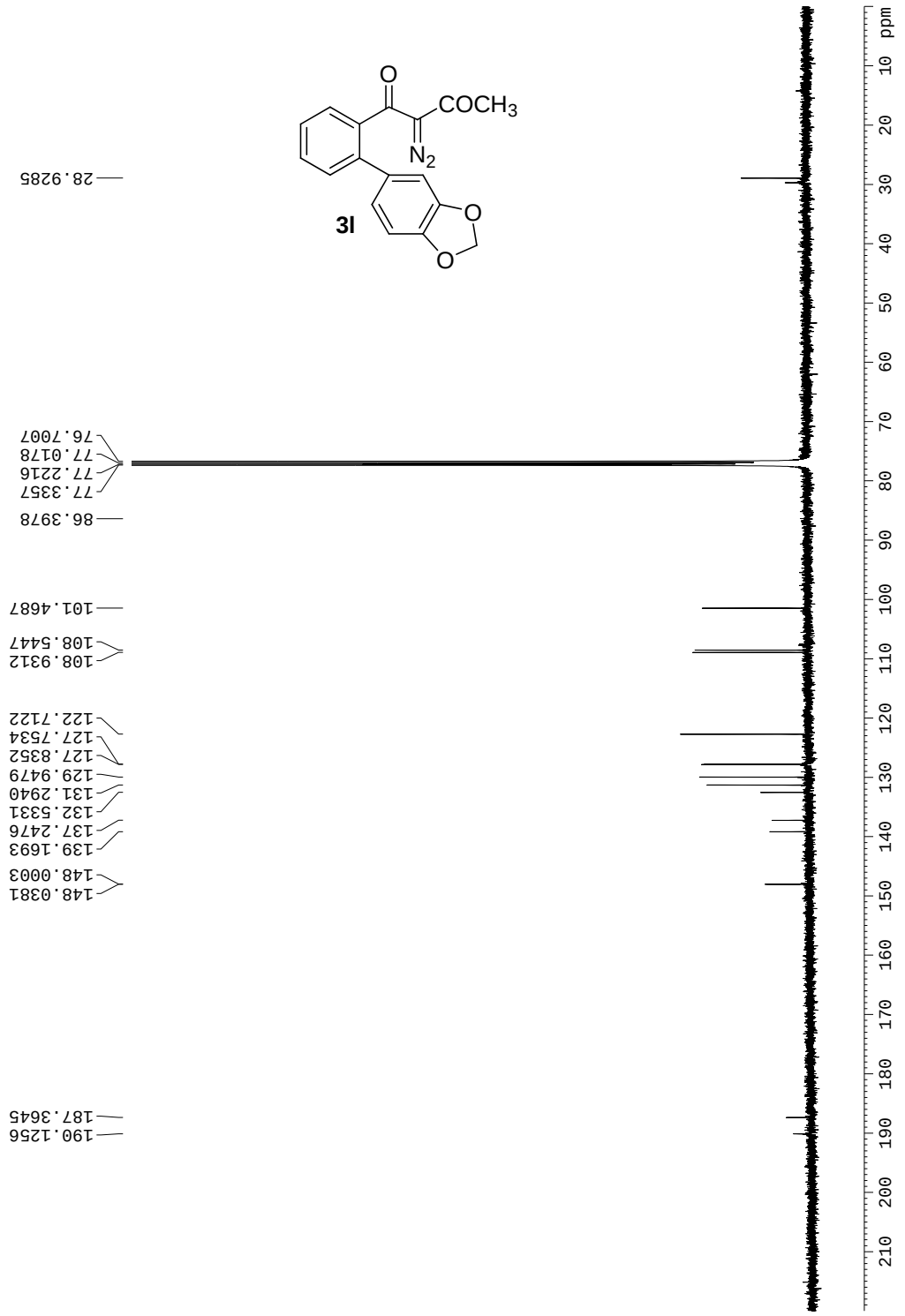
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TD 65536
SOLVENT CDCl3
NS 12850
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 724
DW 19.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
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NUC1 13C
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PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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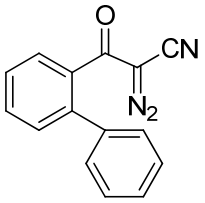
COCH3
N2
O
O
3l
28.9285
76.7007
77.0178
77.2216
77.3357
86.3978
101.4687
108.5447
108.9312
122.7122
127.7534
127.8352
129.9479
131.2940
132.5331
137.2476
139.1693
148.0003
148.0381
187.3645
190.1256
ppm

**ii) Preparation and NMR spectra of 3m-s**

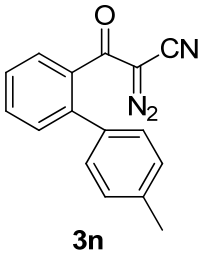
**3-([1,1'-Biphenyl]-2-yl)-2-diazo-3-oxopropanenitrile (3m)**




**3m**

Under $N_2$ protection, NaH (63.5 mg, 60%, 1.59 mmol) and *p*-ABSA (518.5 mg, 98%, 2.12 mmol) were successively added to a stirred solution of **2m** (195 mg, 0.88 mmol) in THF (18 mL) pre-cooled at 0 ºC. The mixture was stirred in dark at 0 ºC for 10 min, and then diluted by ethyl acetate (150 mL). The resulting brown solution was washed with water (30 mL x 2) and brine (30 mL), and concentrated under reduced pressure. The crude residue was packed on silica gel and quickly eluted with hexane-ethyl acetate (8:1) to afford **3m** as a yellow solid (71.3 mg, 33%). IR (neat) 3062, 2225, 2126, 1652, 778, 745, 699 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.60 (ddd, *J* = 7.2, 7.2, 1.5 Hz, 1 H), 7.52 (dd, *J* = 7.8, 1.5 Hz, 1 H), 7.50-7.40 (m, 7 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 187.3, 140.6, 138.7, 135.1, 132.0, 130.5, 129.3, 128.7, 128.4, 127.8, 127.8, 107.7 (CN), 60.2 (C=$N_2$) ppm; HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{15}H_{10}N_3O$: 248.0824; found: 248.0821.

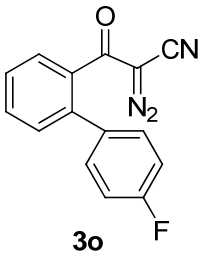
**2-Diazo-3-(4'-methyl-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (3n)**




**3n**

In typical procedure, the titled compound was synthesized from **2n**. Chromatography (hexane-ethyl acetate 16:1, 6:1) afforded **3n** as a yellow oil (13%). IR (neat) 3025, 2224, 2125, 1655, 823, 759 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 7.5, 7.5 Hz, 1 H), 7.51 (d, *J* = 7.5 Hz, 1 H), 7.47-7.42 (m, 2 H), 7.31 (d, *J* = 8.0 Hz, 2 H), 7.25 (d, *J* = 8.0 Hz, 2 H), 2.42 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 187.5, 140.6, 138.3, 135.8, 135.0, 132.0, 130.4, 129.4, 129.2, 127.8, 127.5, 107.8 (CN), 60.2 (C=$N_2$), 21.3 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{11}N_3O$: 261.0902; found: 261.0908.

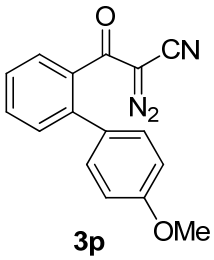
**2-Diazo-3-(4'-fluoro-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (3o)**




**3o**

In typical procedure, the titled compound was synthesized from **2o**. Chromatography (hexane-ethyl acetate 10:1, 5:1) afforded **3o** as a yellow solid (10%). IR (neat) 3064, 2224, 2127, 1654, 840, 761, 722 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 7.59 (ddd, $J$ = 7.8, 7.8, 1.0 Hz, 1 H), 7.52 (dd, $J$ = 6.9, 0.9 Hz, 1 H), 7.47 (ddd, $J$ = 7.8, 6.9, 0.9 Hz, 1 H), 7.43 (d, $J$ = 7.8 Hz, 1 H), 7.41-7.35 (m, 2 H), 7.14 (dd, $J_{H-F}$ = 8.6, $J$ = 8.6 Hz, 2 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 187.1, 162.9 (d, $J_{C-F}$ = 246.9 Hz), 139.5, 135.0, 134.8, 132.1, 131.0 (d, $J_{C-F}$ = 8.1 Hz), 130.5, 127.9, 127.8, 115.7 (d, $J_{C-F}$ = 21.5 Hz), 107.7 (CN), 60.3 ($C=N_2$) ppm; $^{19}F$ NMR (376 MHz, $CDCl_3$) δ -113.5 ppm; HRMS-ESI: *m/z* $[M + Na]^+$ calcd. for $C_{15}H_8FN_3ONa$: 288.0549; found: 288.0550.

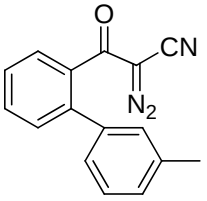
**2-Diazo-3-(4'-methoxy-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (3p)**




**3p**

In typical procedure, the titled compound was synthesized from **2p**. Chromatography (hexane-ethyl acetate 5:1) afforded **3p** as a yellow solid (27%). IR (neat) 3060, 2926, 2224, 2124, 1654, 1249, 835, 762 $cm^{-1}$; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 7.57 (ddd, $J$ = 8.0, 7.0 ,1.0 Hz, 1 H), 7.49 (d, $J$ = 7.2 Hz, 1 H), 7.45-7.40 (m, 2 H), 7.34 (d, $J$ = 8.6 Hz, 2 H), 6.98 (d, $J$ = 8.6 Hz, 2 H), 3.86 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 187.6, 159.9, 140.2, 134.9, 132.0, 131.0, 130.5, 130.3, 127.8, 127.3, 114.2, 107.8 (CN), 60.2 ($C=N_2$), 55.3 (OMe) ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{11}N_3O_2$: 277.0851; found: 277.0855.

**2-Diazo-3-(3'-methyl-[1,1'-biphenyl]-2-yl)-3-oxopropanenitrile (3q)**

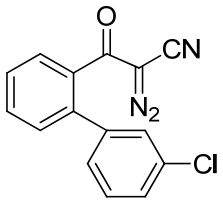



**3q**

In typical procedure, the titled compound was synthesized from **2q**. Chromatography

(hexane-ethyl acetate 8:1) afforded **3q** as a yellow oil (27%). IR (neat) 3060, 2224, 2125, 1654, 756, 698 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.59 (ddd, *J* = 7.4, 7.4, 1.5 Hz, 1 H), 7.53-7.49 (m, 1 H), 7.49-7.43 (m, 2 H), 7.33 (dd, *J* = 7.8, 7.8 Hz, 1 H), 7.26-7.22 (m, 2 H), 7.19 (d, *J* = 7.8 Hz, 1 H), 2.42 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 187.4, 140.7, 138.7, 138.4, 135.0, 132.0, 130.5, 129.9, 129.2, 128.5, 127.8, 127.7, 126.4, 107.7 (CN), 60.1 (C=$N_2$), 21.5 ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_{11}N_3O$: 261.0902; found: 261.0909.

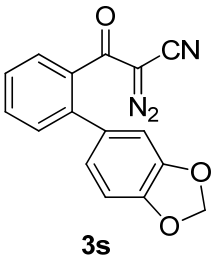
**3-(3'-Chloro-[1,1'-biphenyl]-2-yl)-2-diazo-3-oxopropanenitrile (3r)**




**3r**

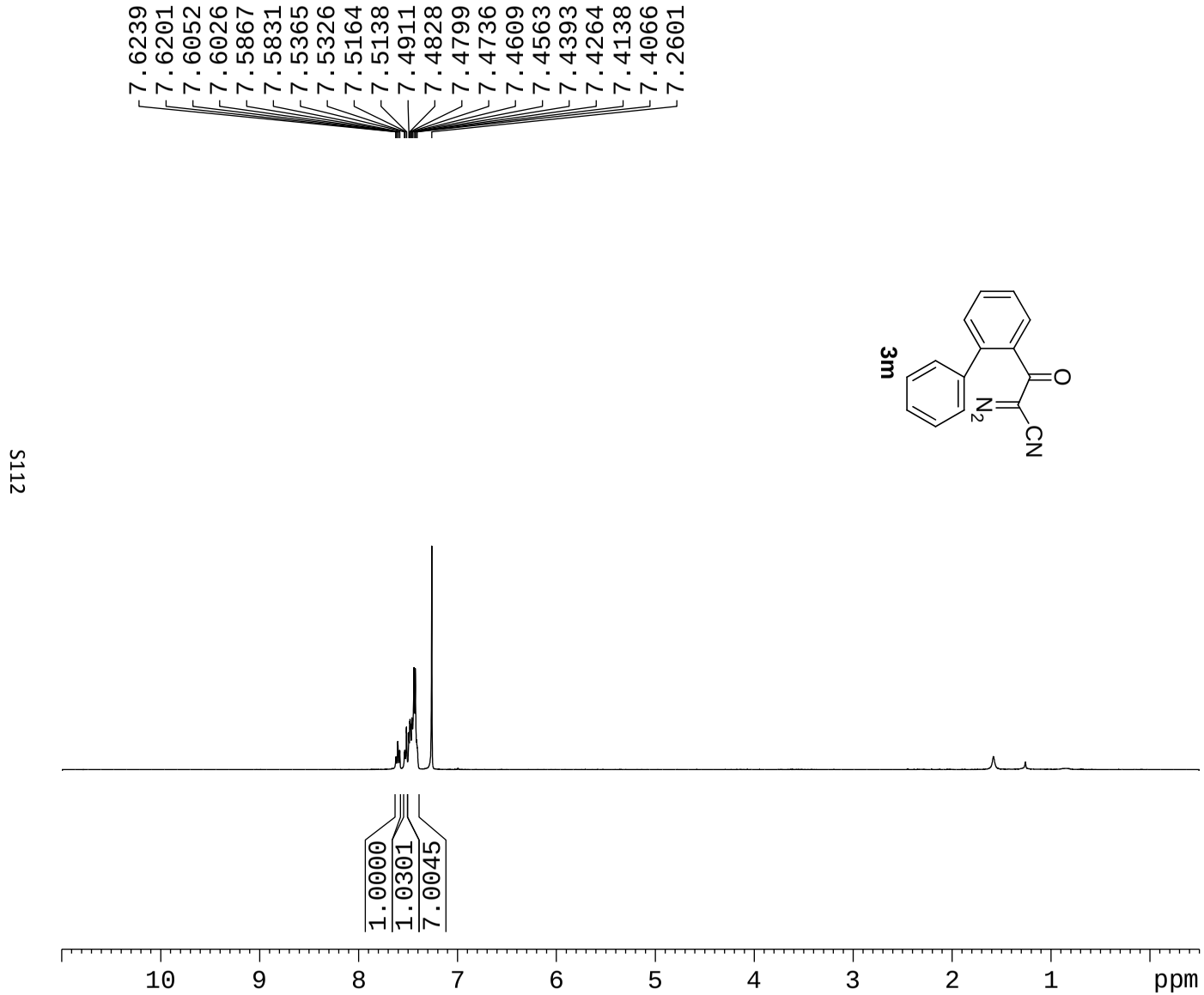
In typical procedure, the titled compound was synthesized from **2r**. Chromatography (hexane-ethyl acetate 5:1) afforded **3r** as a yellow oil (19%). IR (neat) 3063, 2224, 2127, 1649, 792, 757, 691 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.61 (ddd, *J* = 7.4, 7.4, 1.5 Hz, 1 H), 7.56-7.47 (m, 2 H), 7.46-7.34 (m, 4 H), 7.29-7.25 (m, 1 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 186.6, 140.6, 139.2, 134.9, 134.6, 132.2, 130.5, 129.8, 129.2, 128.5, 128.3, 127.9, 127.5, 107.7 (CN), 60.2 (C=$N_2$) ppm.

**3-(2-(Benzo[*d*][1,3]dioxol-5-yl)phenyl)-2-diazo-3-oxopropanenitrile (3s)**




**3s**

In typical procedure, the titled compound was synthesized from **2s**. Chromatography (hexane-ethyl acetate 5:1) afforded **3s** as a yellow oil (55%). IR (neat) 3063, 2901, 2224, 2126, 1652, 1238, 1038, 815, 760 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.56 (ddd, *J* = 7.5, 7.5, 1.2 Hz, 1 H), 7.50-7.39 (m, 3 H), 6.91 (d, *J* = 1.4 Hz, 1 H), 6.86 (d, *J* = 8.0 Hz, 1 H), 6.82 (dd, *J* = 8.0, 1.4 Hz, 1 H), 6.03 (s, 2 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 187.4, 148.0, 148.0, 140.2, 135.0, 132.7, 132.0, 130.4, 127.8, 127.5, 123.3, 109.6, 108.5, 107.9 (CN), 101.5 (-$CH_2$-), 60.2 (C=$N_2$) ppm; HRMS-EI: *m/z* $[M]^+$ calcd. for $C_{16}H_9N_3O_3$: 291.0644; found: 291.0645.

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| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 406 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 300.5 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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| | |
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| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

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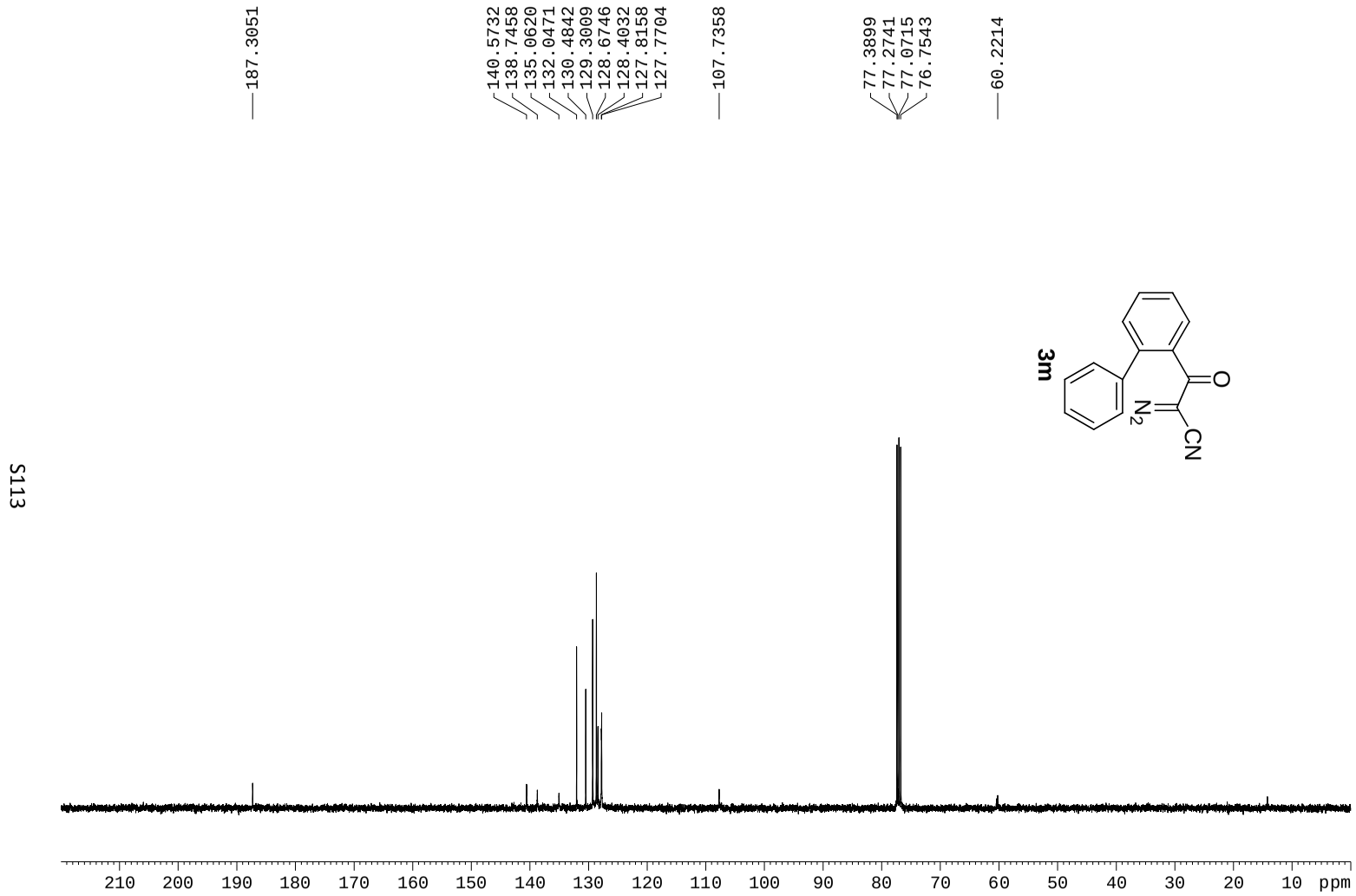




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DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
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DW               19.800 usec
DE                6.50 usec
TE                299.6 K
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DELTA         0.89999998 sec
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P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
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PCPD2             90.00 usec
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PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

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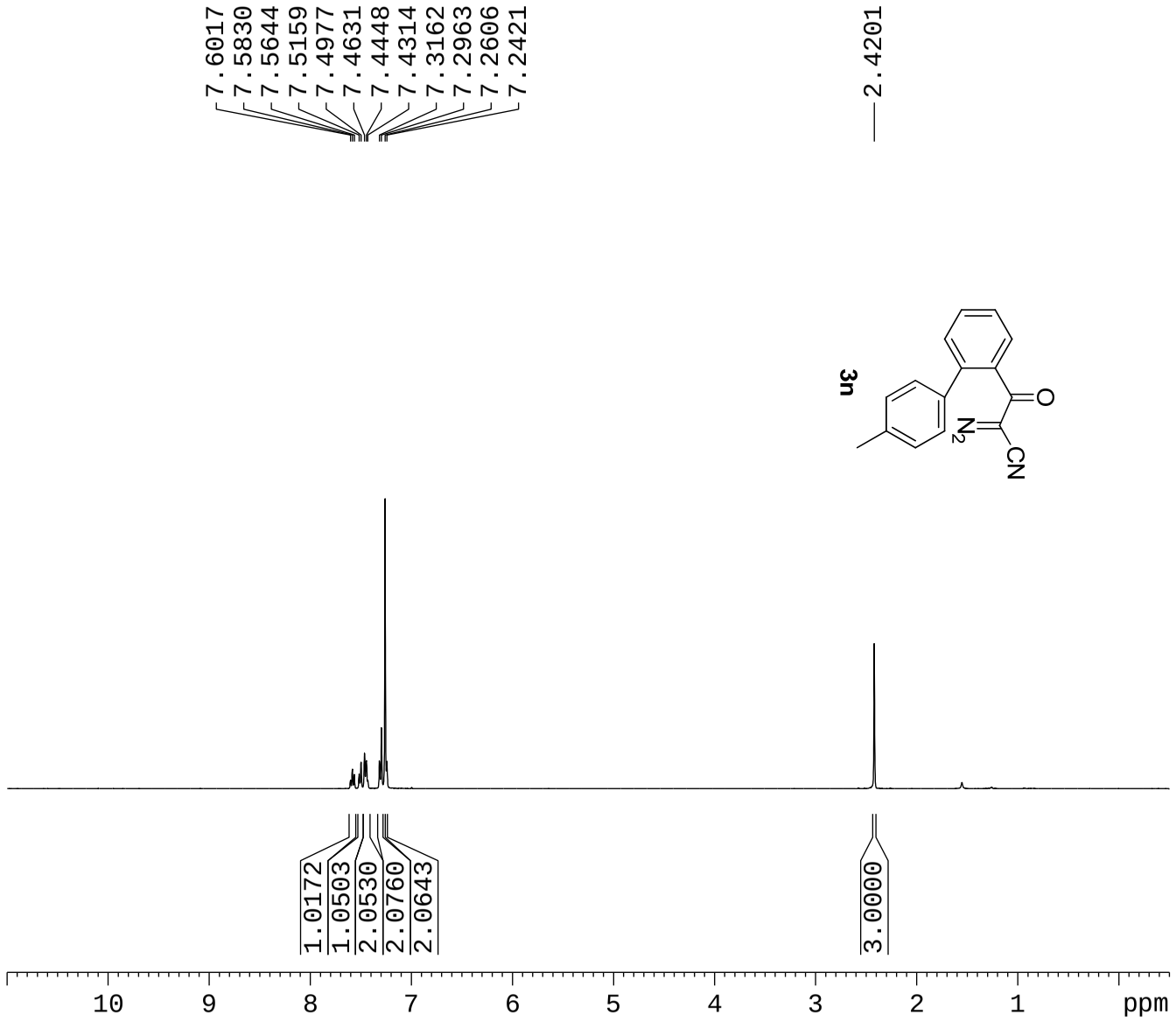


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EXPNO                  1122011
PROCNO                       1

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PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                         28
DS                          0
SWH                  6009.615 Hz
FIDRES               0.183399 Hz
AQ                   2.7263477 sec
RG                         575
DW                      83.200 usec
DE                       6.50 usec
TE                      299.5 K
D1                   2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PL1                      0.90 dB
SFO1             400.1326008 MHz

F2 - Processing parameters
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WDW                        EM
SSB                         0
LB                       0.30 Hz
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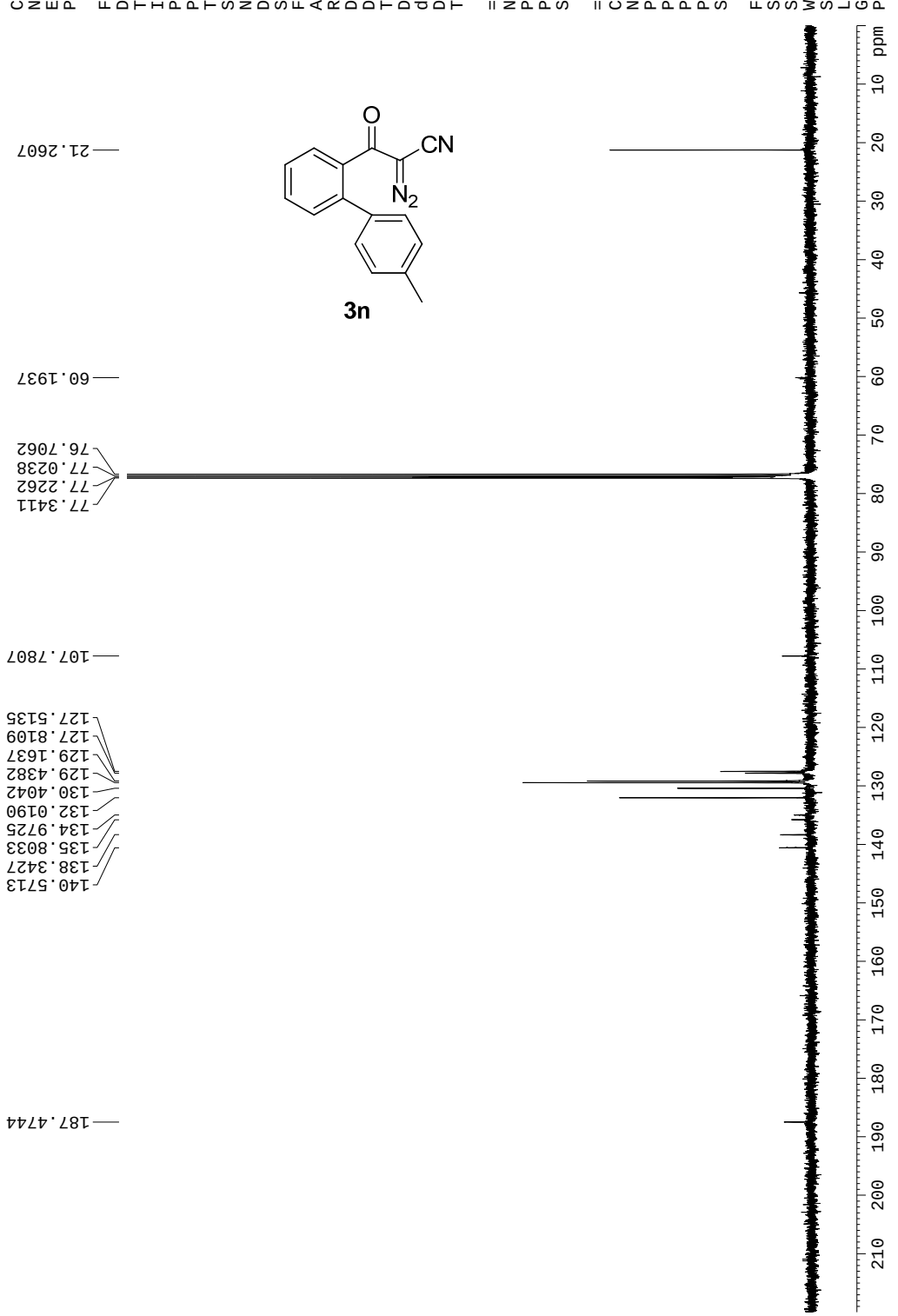
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1302
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
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PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
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SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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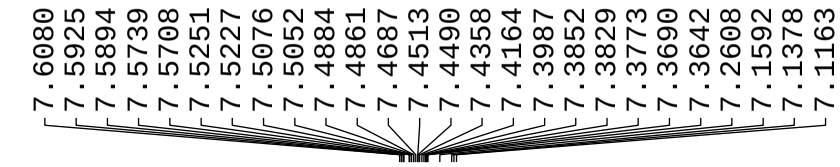
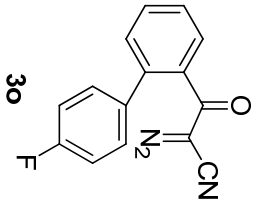
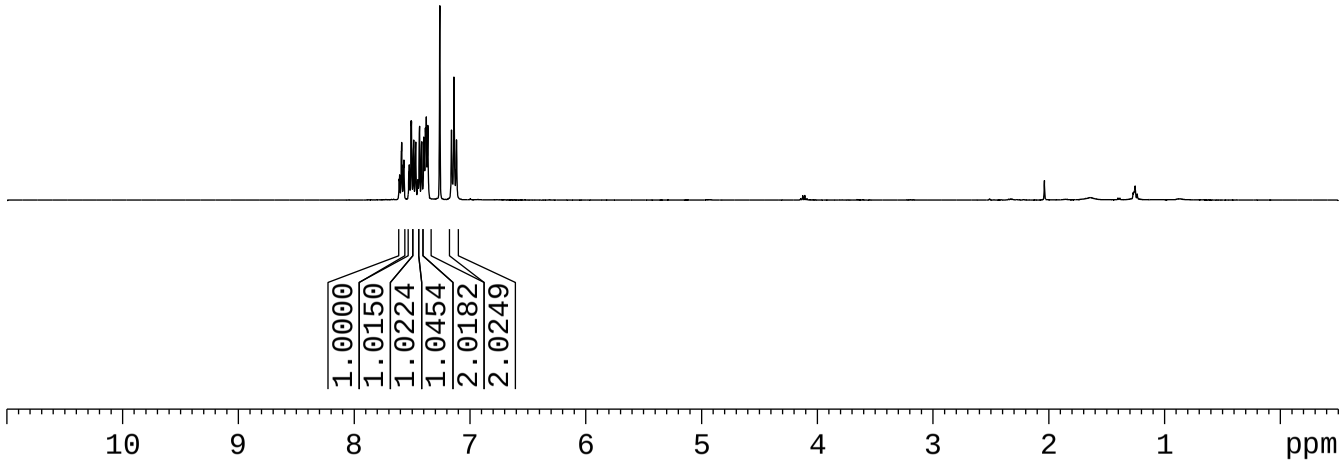



S116

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PROCNO                 1

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TD                 32768
SOLVENT            CDCl3
NS                    24
DS                     0
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AQ             2.0447731 sec
RG                   287
DW                62.400 usec
DE                 6.50 usec
TE                297.4 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300077 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```
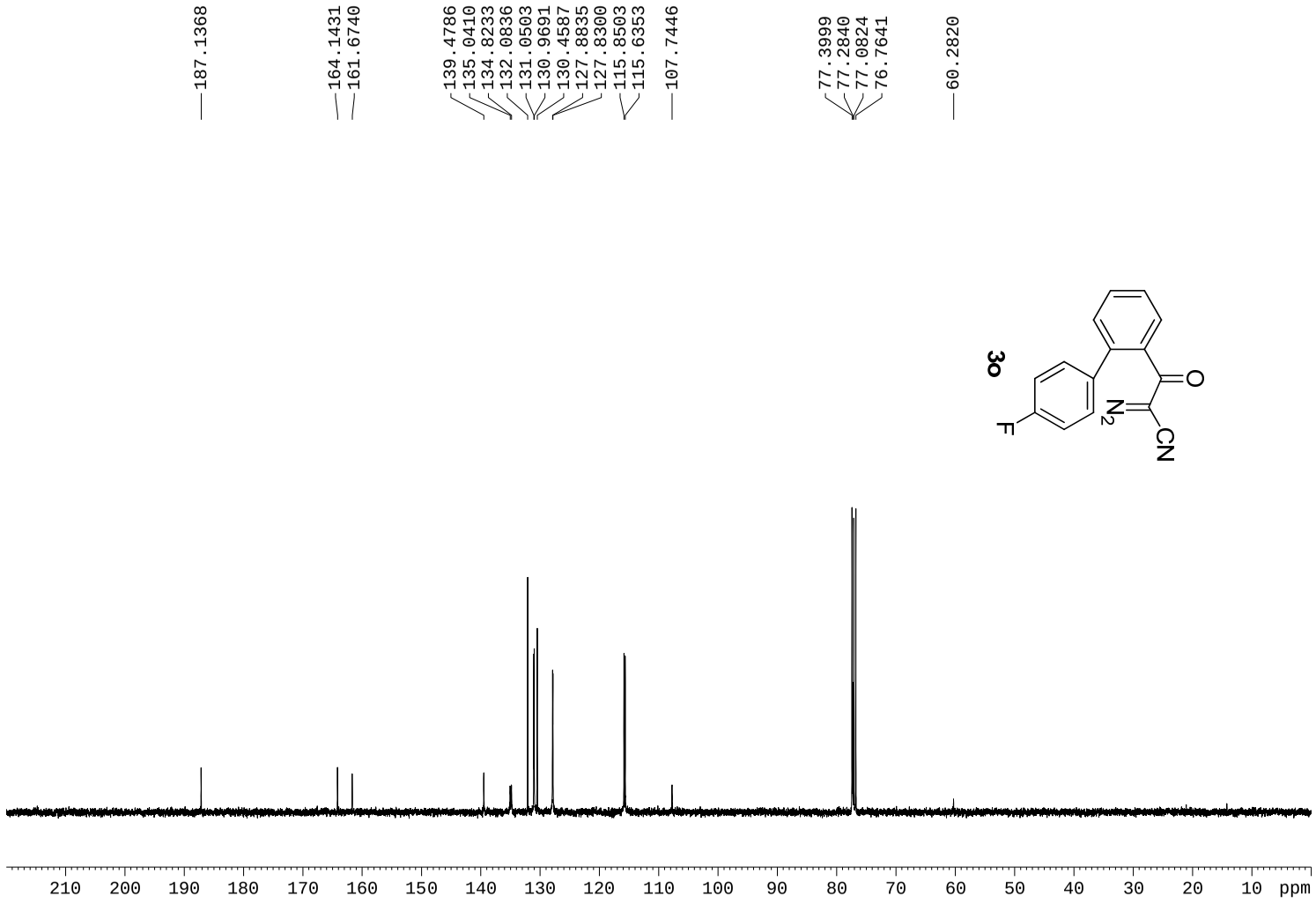
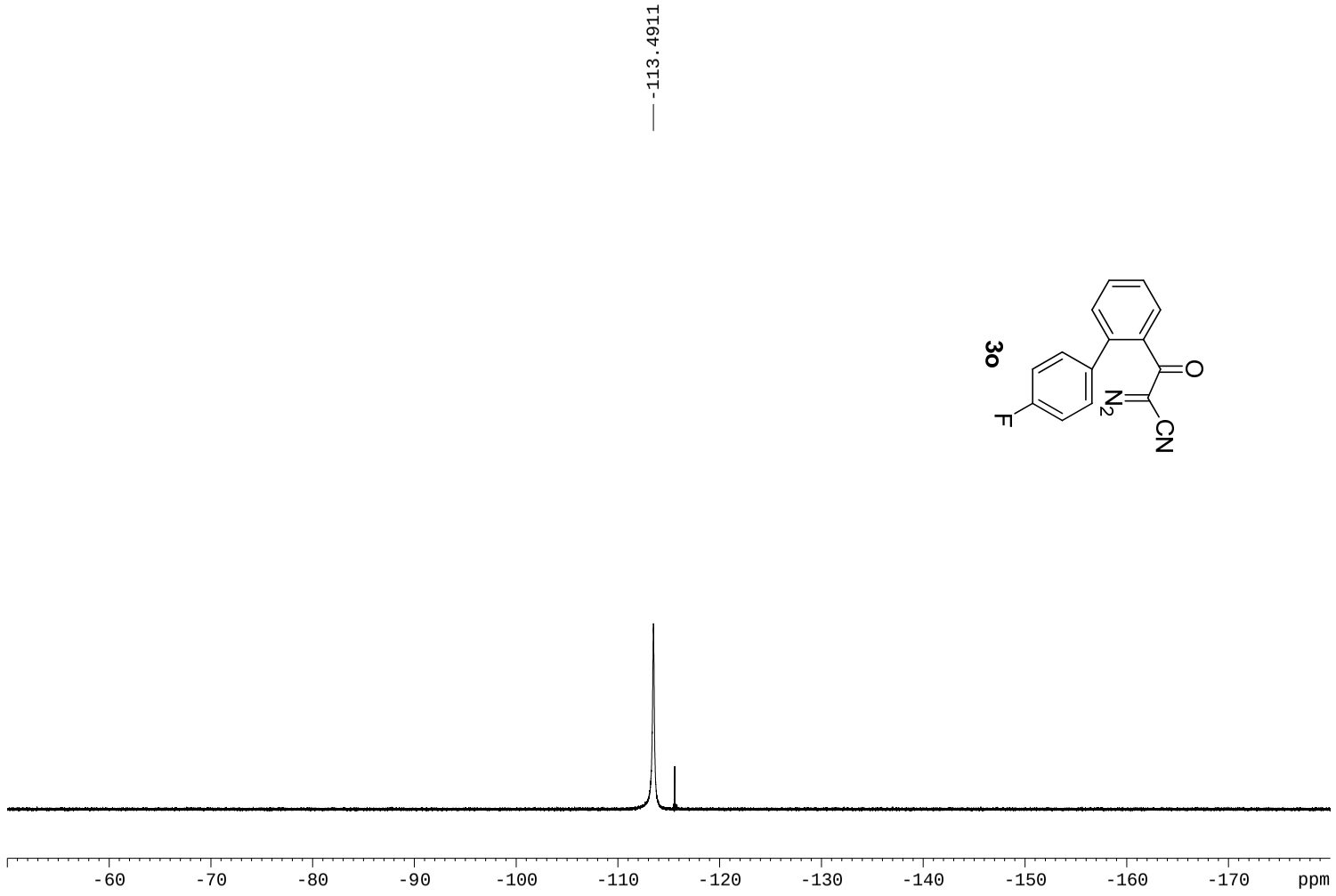



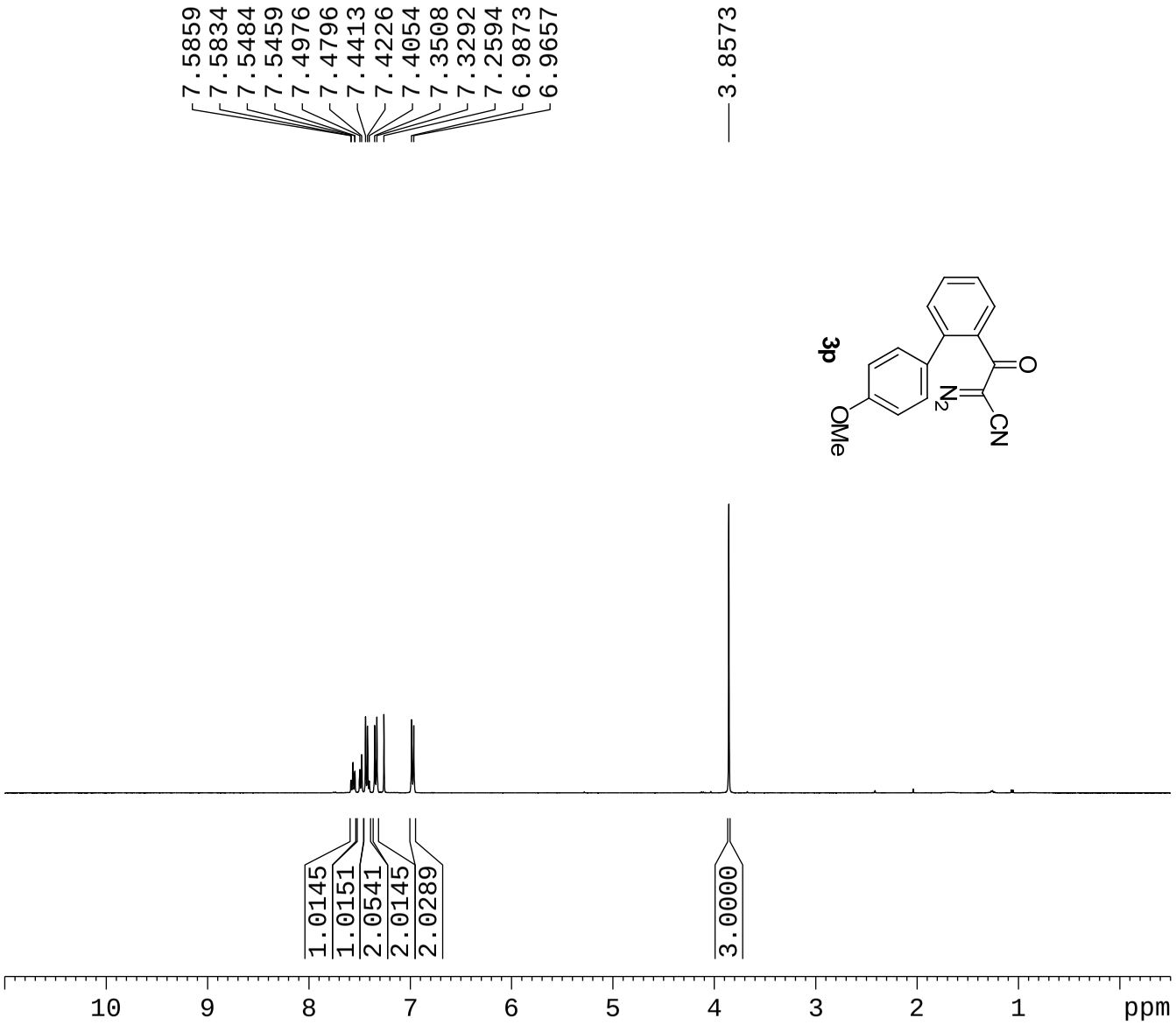

```
Current Data Parameters
NAME            chen2017
EXPNO            1213012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171213
Time               15.35
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  209
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               297.5 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```







```
Current Data Parameters
NAME            chen2017
EXPNO            1213014
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171213
Time               15.53
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgfhigqn
TD                131072
SOLVENT             CDCl3
NS                     10
DS                      0
SWH             89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340532 sec
RG                   2050
DW                  5.600 usec
DE                  6.50 usec
TE                 297.5 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                  20.00 usec
PL1                  2.50 dB
SFO1         376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -0.40 dB
PL12                15.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          376.4983660 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



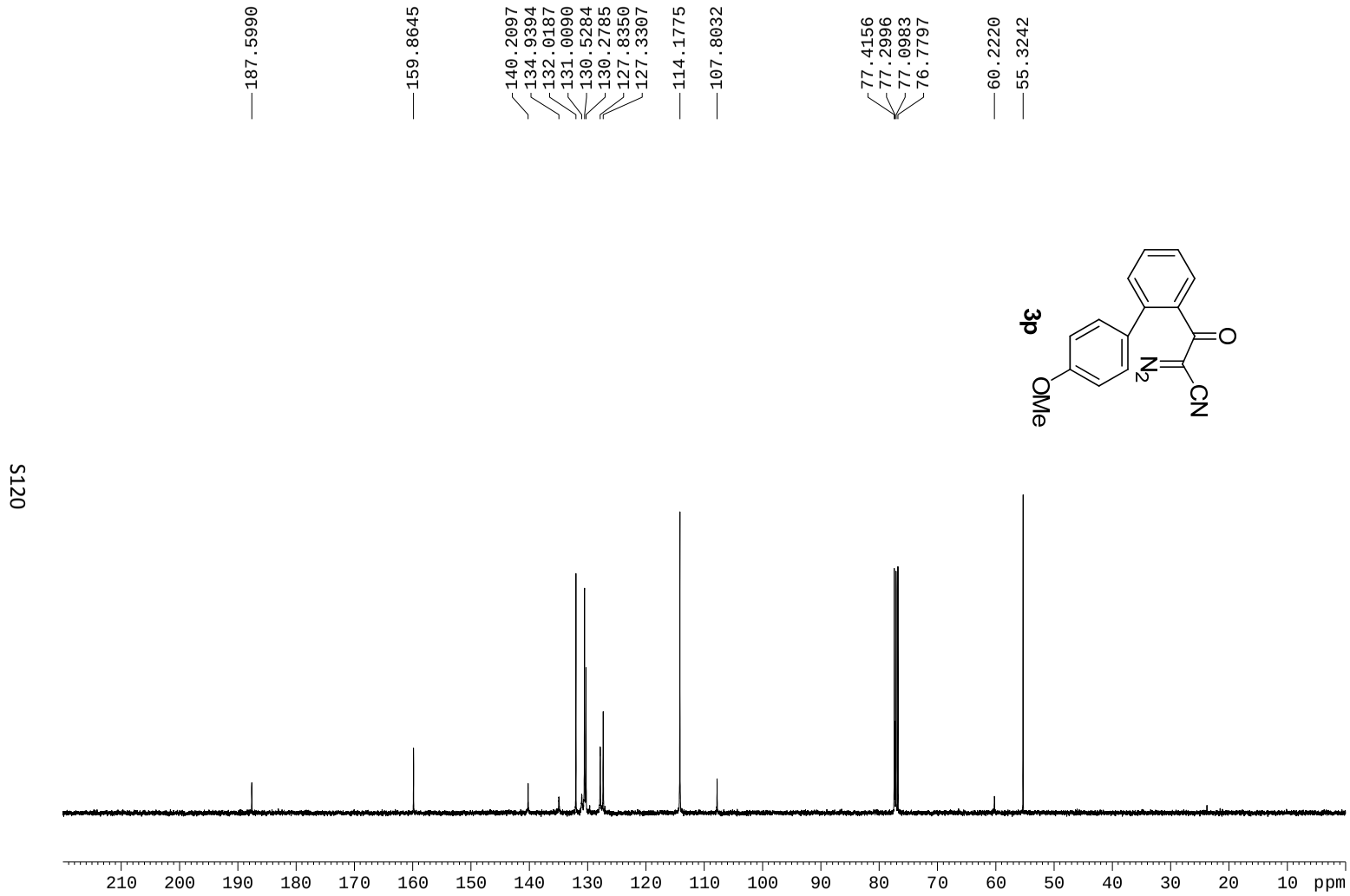

S119

Current Data Parameters
NAME chen2018
EXPNO 103011
PROCNO 1

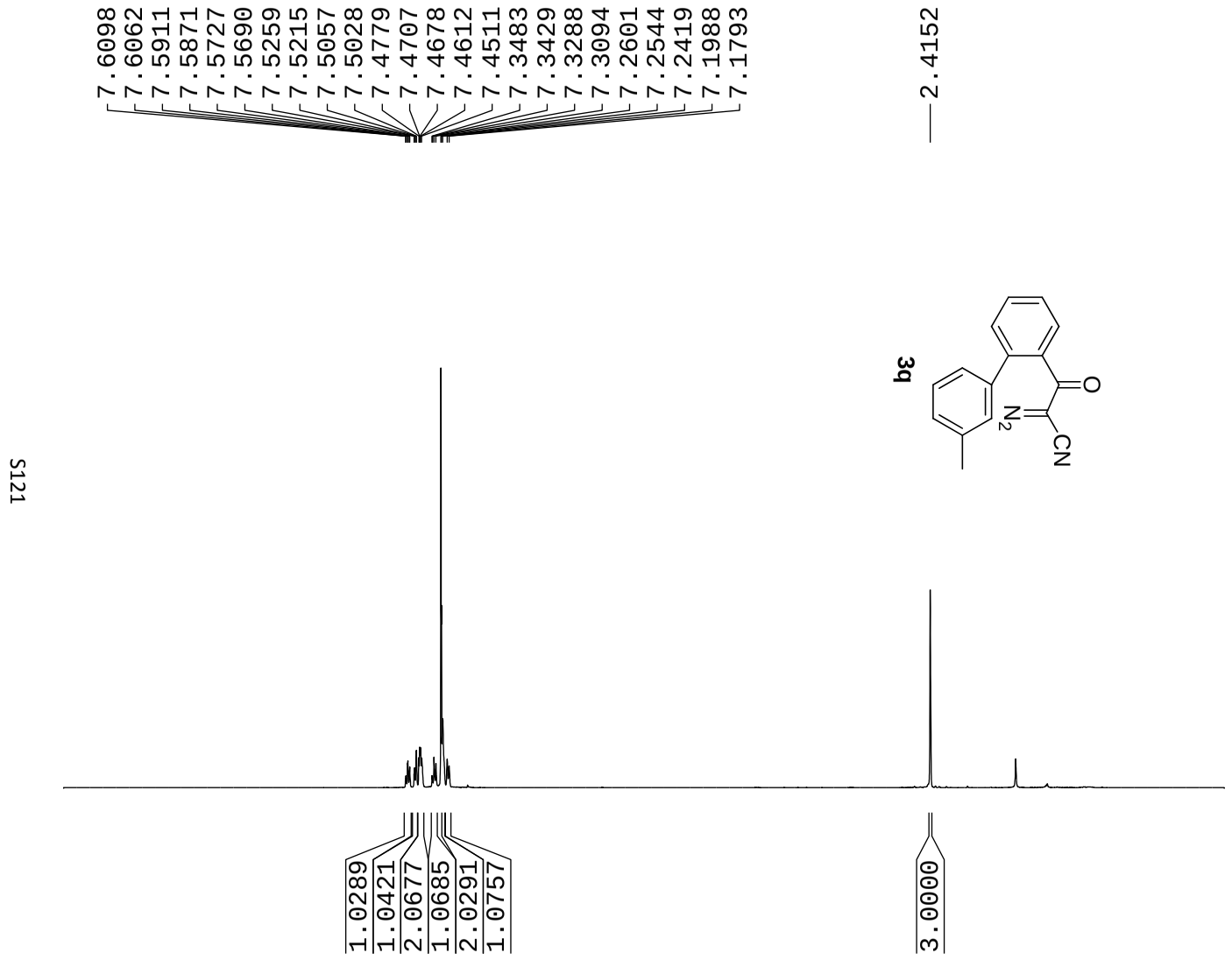
F2 - Acquisition Parameters
Date_ 20180103
Time 10.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 22
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 297.6 K
D1 0.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME            chen2018
EXPNO             103012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180103
Time               10.47
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  647
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                297.6 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 125011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

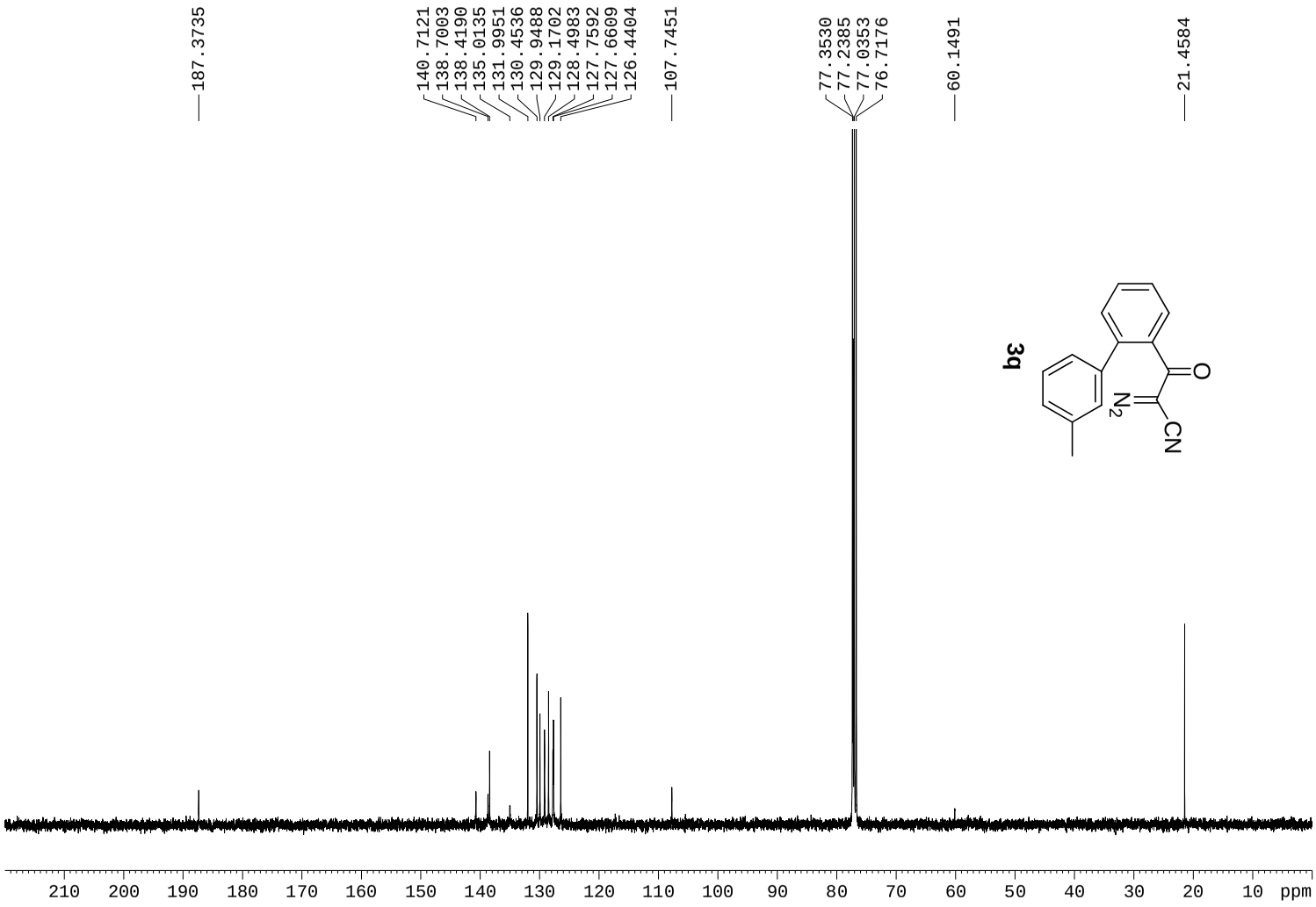
| | |
|---|---|
| Date_ | 20180125 |
| Time | 10.23 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 44 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 512 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 296.6 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

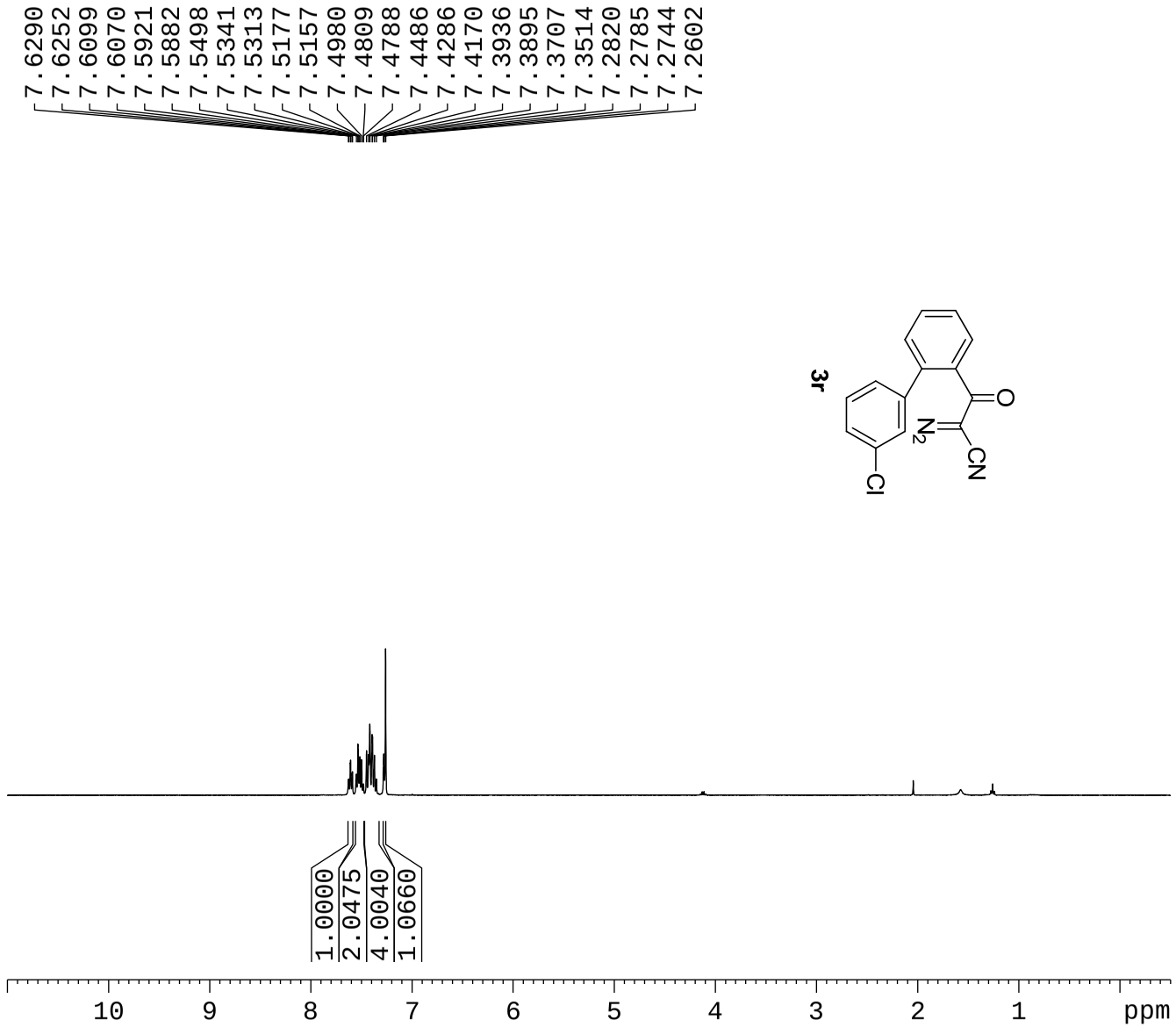
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300086 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




```
Current Data Parameters
NAME            chen2018
EXPNO             125012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180125
Time               10.35
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1455
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                296.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 306011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

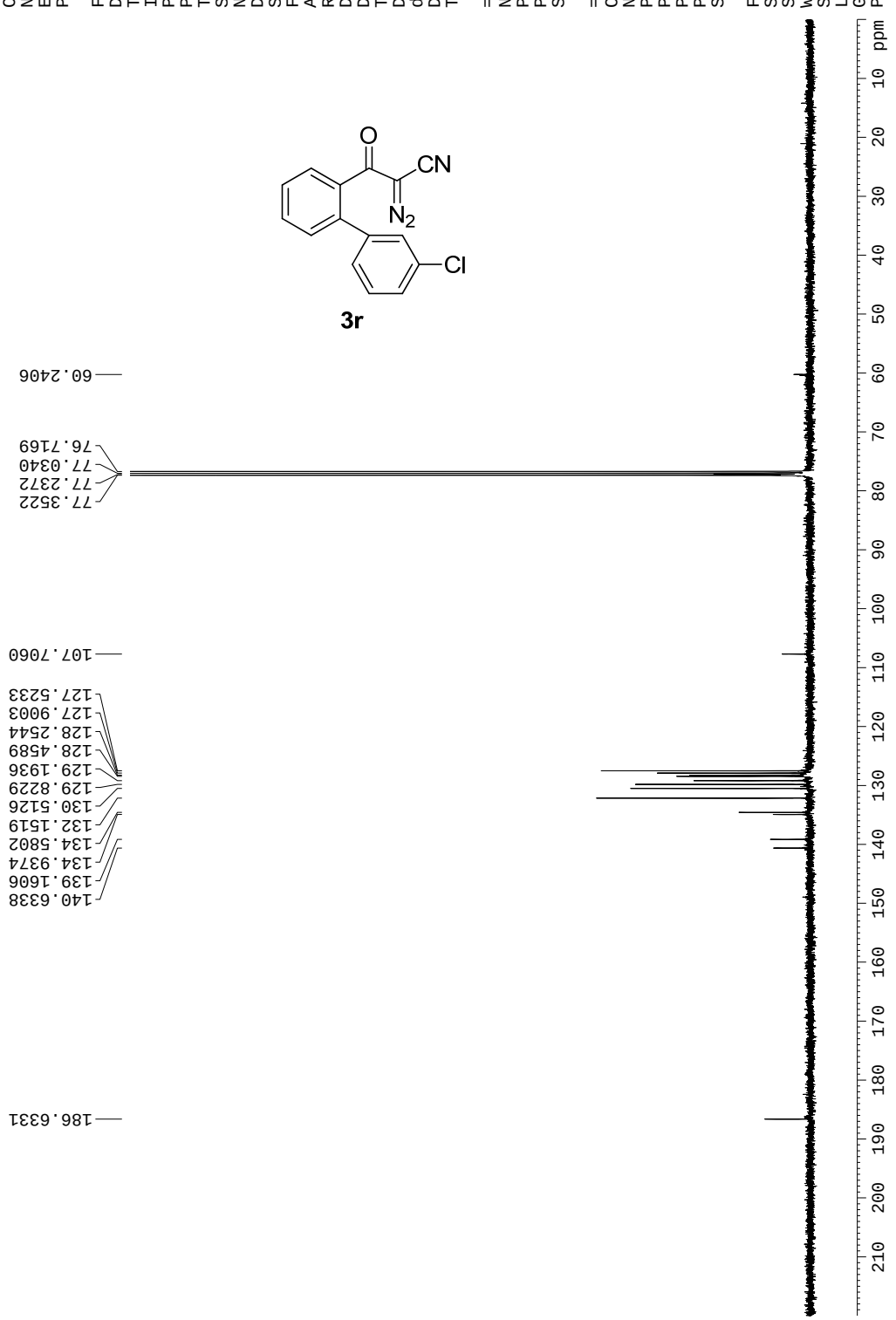
| | |
|---|---|
| Date_ | 20180306 |
| Time | 10.00 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 23 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 575 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 298.7 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

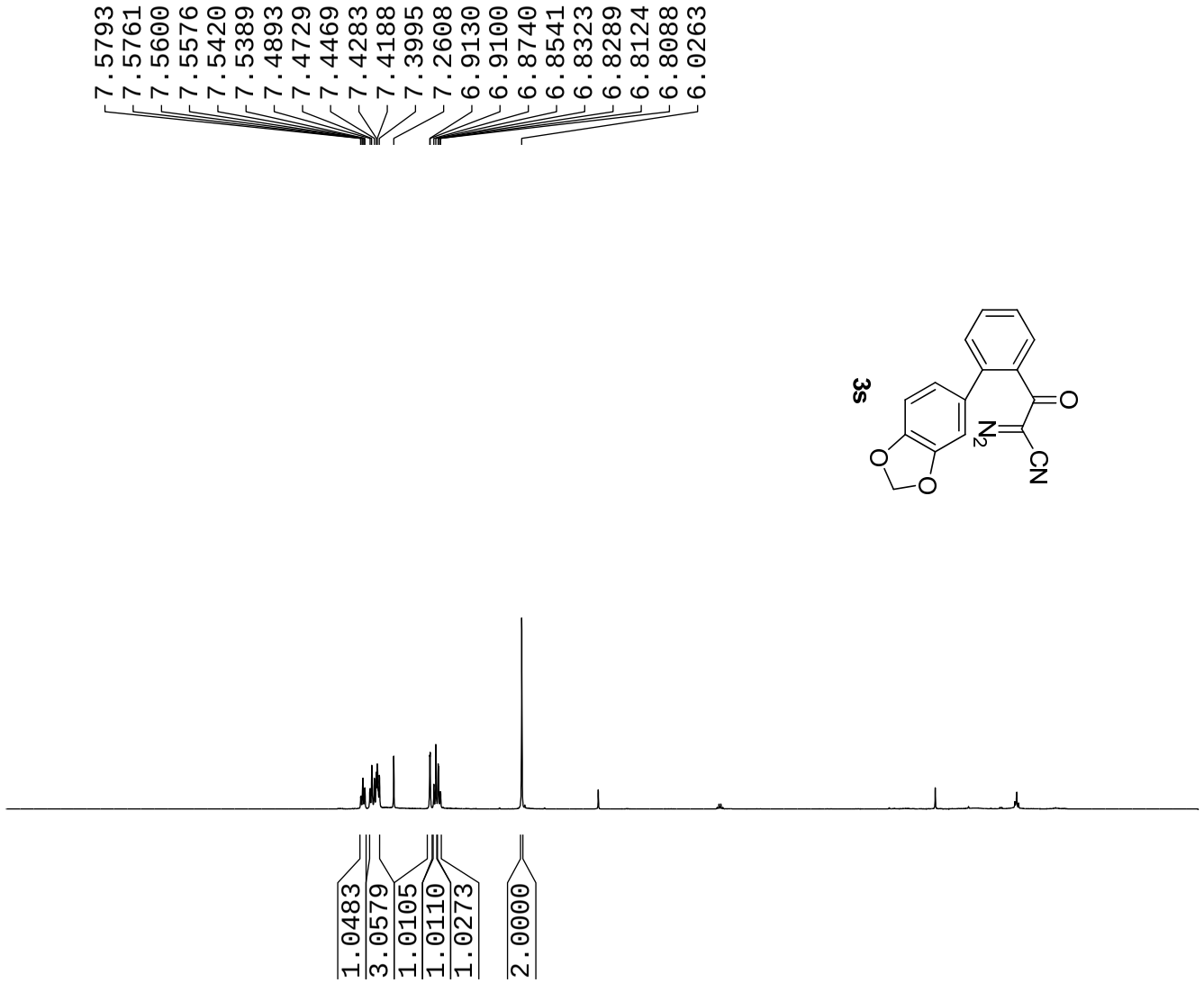
======== CHANNEL f1 ========

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300093 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |


Current Data Parameters
NAME chen2018
EXPNO 306012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180306
Time 10.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 904
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
CN
N2
Cl
3r
60.2406
77.3522
77.2372
77.0340
76.7169
107.7060
127.5233
127.9003
128.2544
128.4589
129.1936
129.8229
130.5126
132.1519
134.5802
134.9374
139.1606
140.6338
186.6331
ppm




S125

Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 409011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

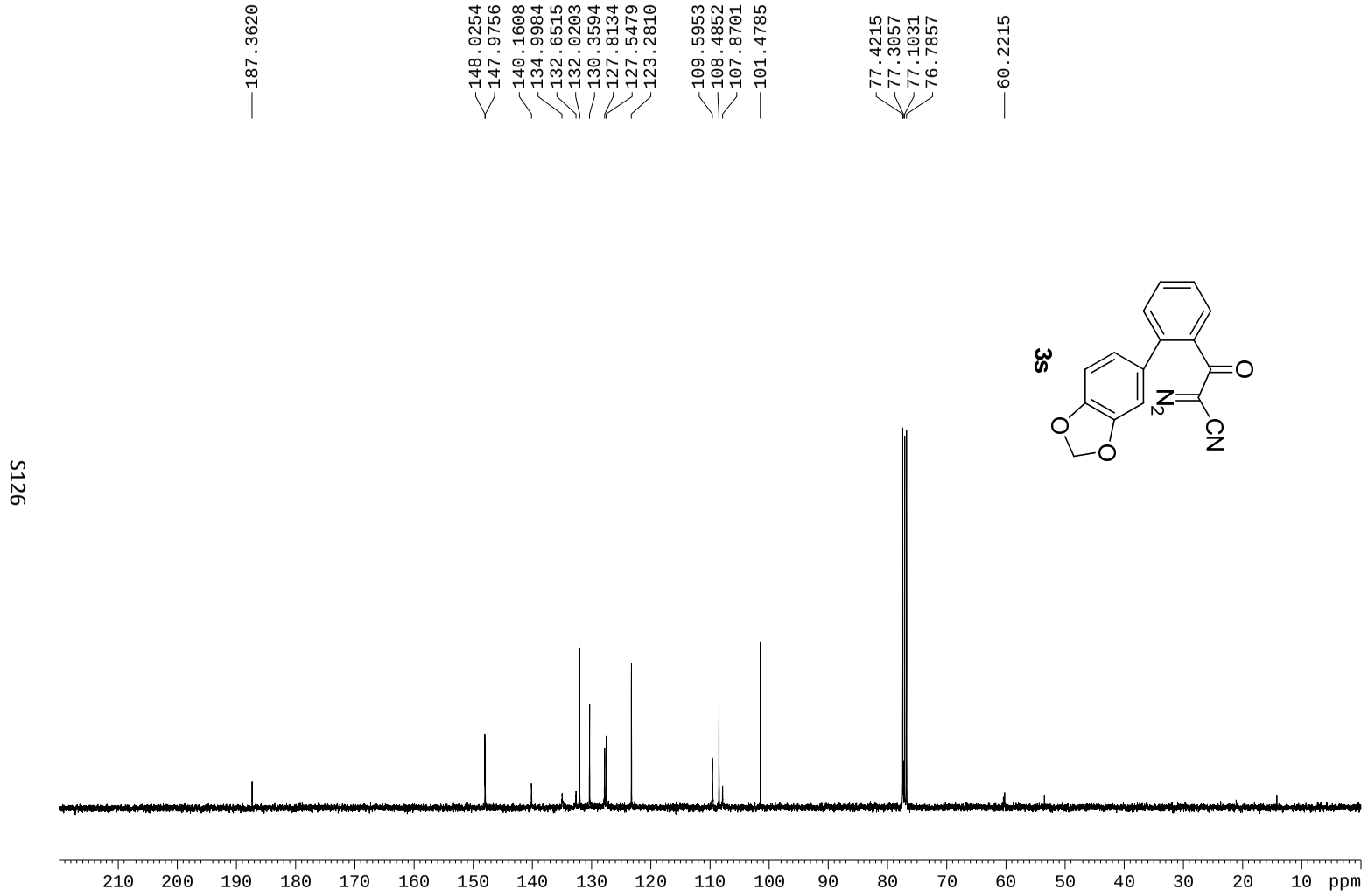
| | |
|---|---|
| Date_ | 20180409 |
| Time | 11.17 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 23 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 101 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 294.8 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |

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

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

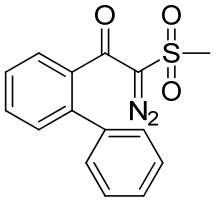
| | |
|---|---|
| SI | 16384 |
| SF | 400.1300086 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




```
Current Data Parameters
NAME            chen2018
EXPNO             409012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180409
Time               11.24
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  146
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                  406
DW               19.800 usec
DE                6.50 usec
TE                294.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

**iii) Preparation and NMR spectra of 3t/u**

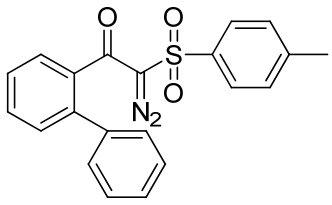
**1-([1,1'-Biphenyl]-2-yl)-2-diazo-2-(methylsulfonyl)ethanone (3t)**



**3t**

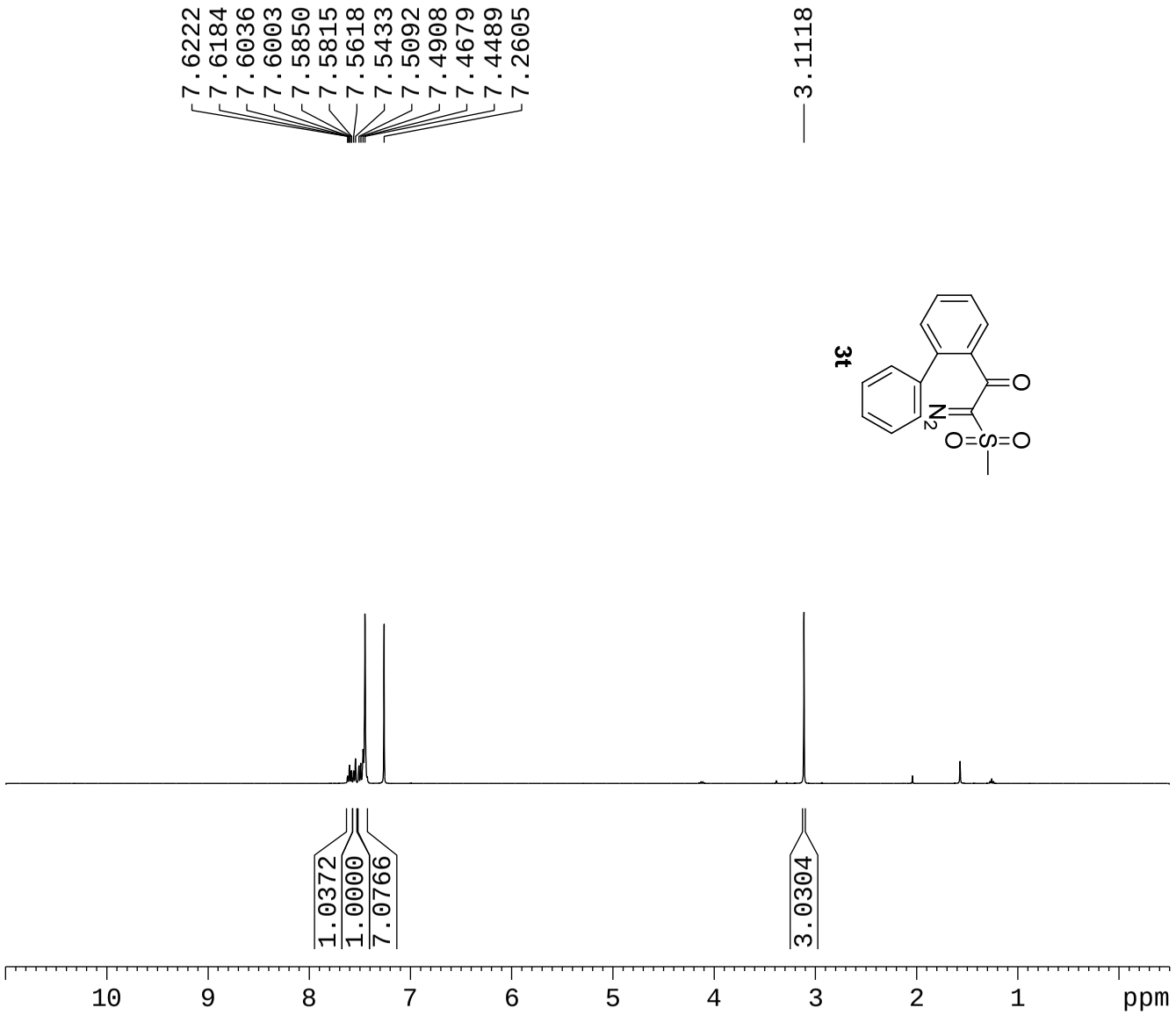
173.9 mg of $K_2CO_3$ (99.5%, 1.25 mmol) was added to a stirred solution of **2t** (229.0 mg, 0.84 mmol) in acetonitrile (14 mL). After stirring at rt for 2 min, *p*-acetamidobenzenesulfonyl azide (260 mg, 98%, 1.06 mmol) were introduced to the mixture. The reaction mixture was stirred in dark for 2.3 hours, then quenched by water (5 mL), and extracted with ethyl acetate (200 mL). The organic layer was separated and successively washed with water (30 mL x 2) and brine (30 mL). After concentration, the crude reside was purified by chromatography (hexane-ethyl acetate 4:1) to afford **3t** as a pale yellow solid (249.2 mg, 99%). IR (neat) 3027, 2925, 2118, 1644, 1332, 1149, 766, 747 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.60 (ddd, *J* = 7.4, 7.4, 1.4 Hz, 1 H), 7.55 (d, *J* = 7.4 Hz, 1 H), 7.50 (d, *J* = 7.4 Hz, 1 H), 7.48-7.42 (m, 6 H), 3.11 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 186.1, 139.6, 138.3, 135.7, 131.8, 130.0, 128.9, 128.9, 128.8, 128.3, 128.0, 84.3 ($C=N_2$), 44.1 ppm; HRMS-ESI: *m/z* $[M + Na]^+$ calcd. for $C_{15}H_{12}N_2O_3NaS$: 323.0466; found: 323.0457.

**1-([1,1'-Biphenyl]-2-yl)-2-diazo-2-tosylethanone (3u)**



**3u**

The titled compound was similarly prepared as **3t** by using **2u** as the starting material. Chromatographic purification (hexane-ethyl acetate 8:1, 4:1) provided **3u** as a yellow oil (90%). IR (neat) 3063, 2924, 2110, 1652, 1339, 1154, 745, 699, 680, 657 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.2 Hz, 2 H), 7.54 (ddd, *J* = 8.3, 7.3, 1.0 Hz, 1 H), 7.47-7.39 (m, 2 H), 7.39-7.28 (m, 4 H), 7.19-7.12 (m, 4 H), 2.50 (s, 3 H) ppm; $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 185.0, 145.1, 139.6, 138.1, 138.1, 135.9, 131.6, 130.0, 129.6, 128.7, 128.5, 128.3, 128.3, 128.1, 128.0, 85.2 ($C=N_2$), 21.7 ppm; HRMS-ESI: *m/z* $[M + H]^+$ calcd. for $C_{21}H_{17}N_2O_3S$: 377.0960; found: 377.0957.

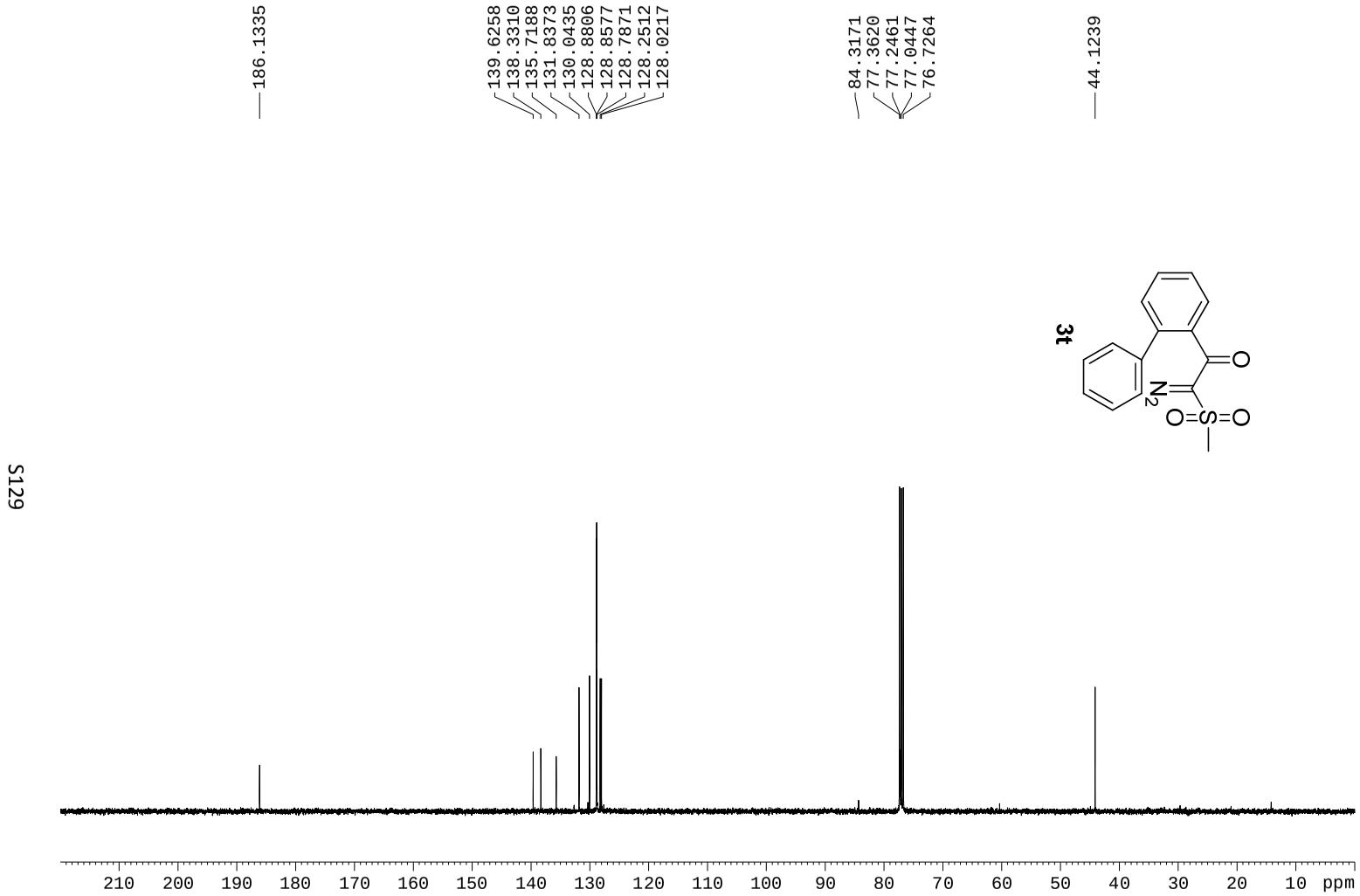



Current Data Parameters
NAME chen2017
EXPNO 803011
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170803
Time 9.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 21
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 406
DW 83.200 usec
DE 6.50 usec
TE 302.9 K
D1 1.00000000 sec
TD0 1

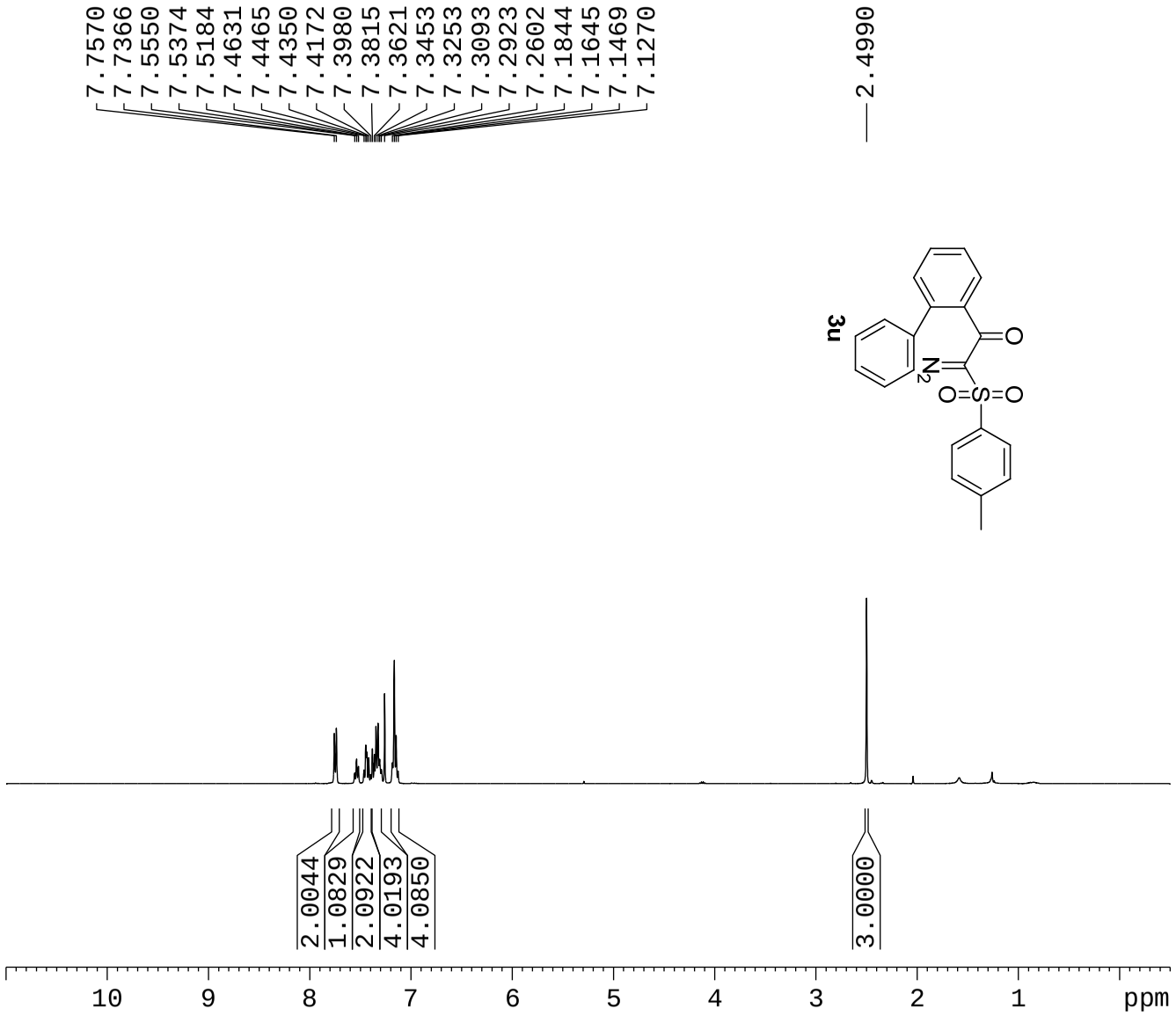
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00






```
Current Data Parameters
NAME            chen2017
EXPNO             703012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170703
Time               9.59
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  323
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               305.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




Current Data Parameters

| | |
|---|---|
| NAME | chen2017 |
| EXPNO | 912021 |
| PROCNO | 1 |

F2 - Acquisition Parameters

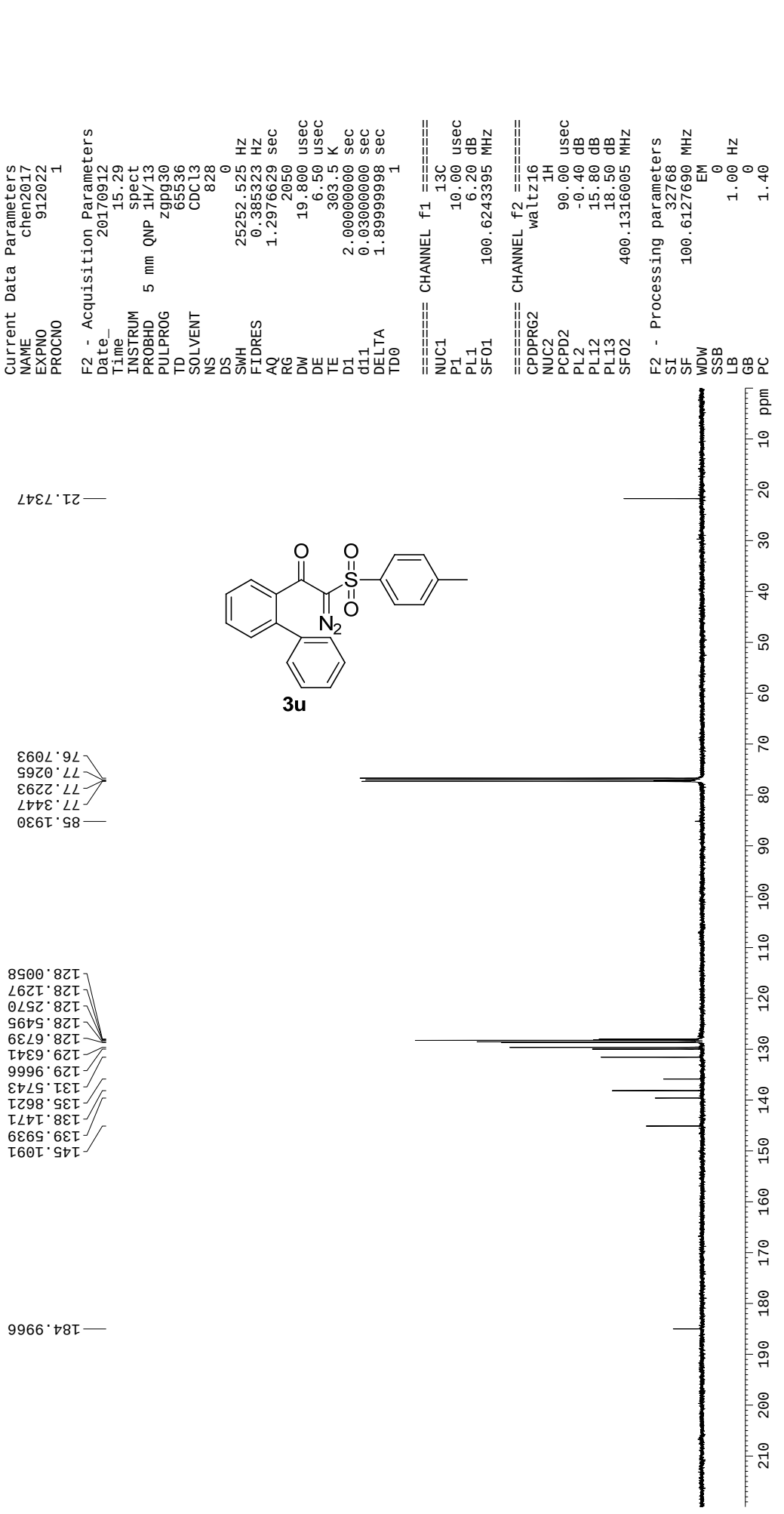
| | |
|---|---|
| Date_ | 20170912 |
| Time | 15.21 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 24 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 287 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 303.3 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

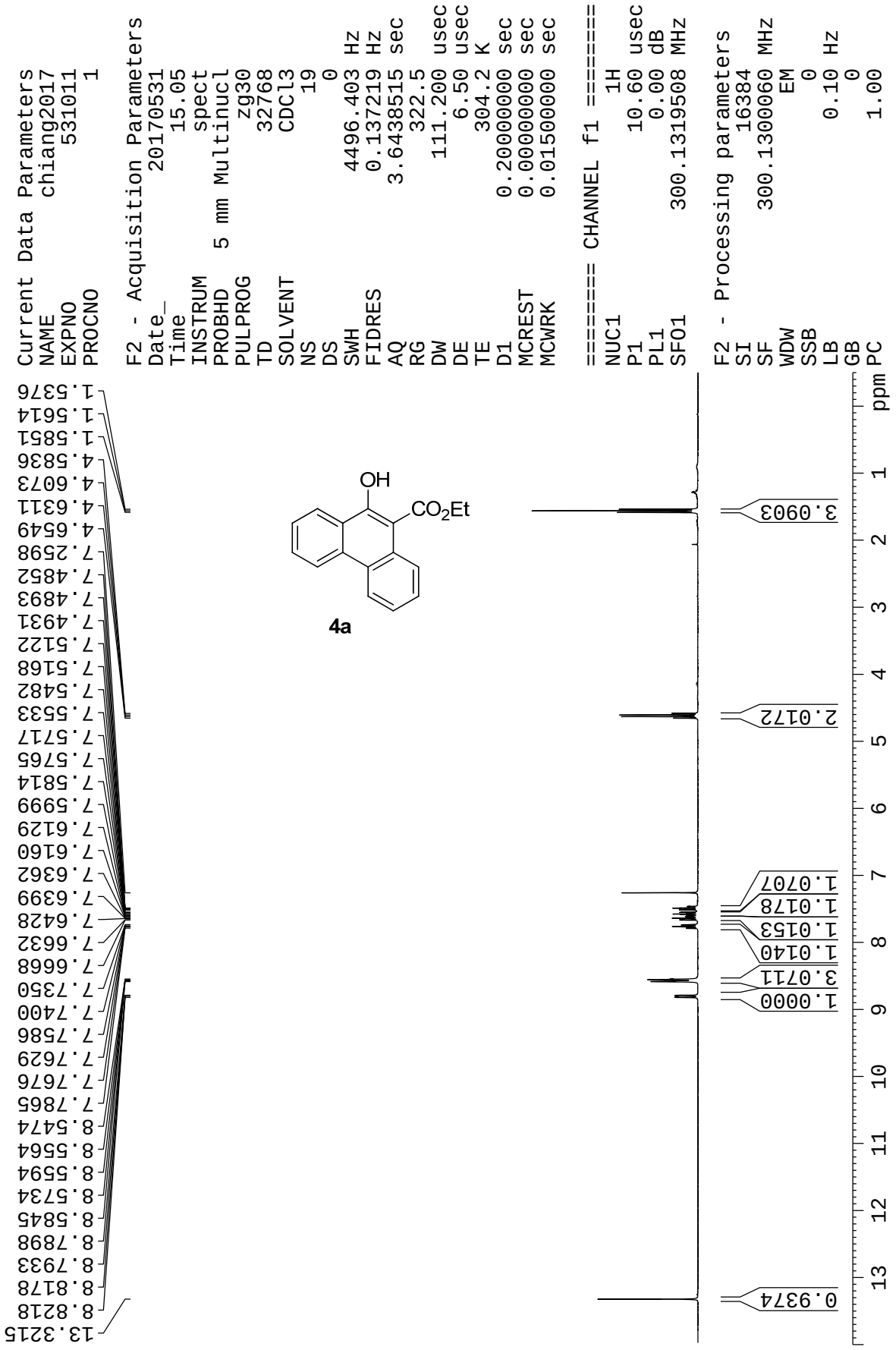
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

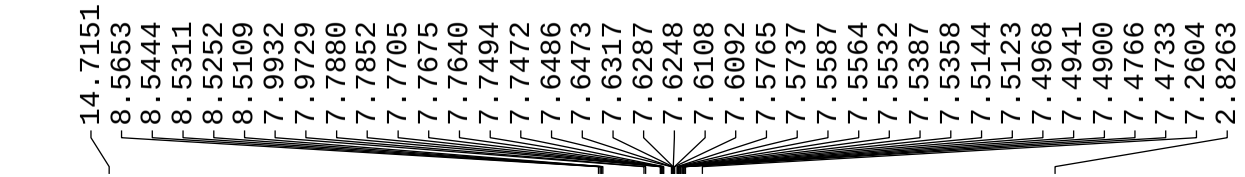
| | |
|---|---|
| SI | 16384 |
| SF | 400.1300093 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

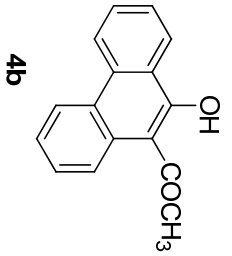

Current Data Parameters
NAME chen2017
EXPNO 912022
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170912
Time 15.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 828
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 303.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.7347
76.7093
77.0265
77.2293
77.3447
85.1930
128.0058
128.1297
128.2570
128.5495
128.6739
129.6341
129.9666
131.5743
135.8621
138.1471
139.5939
145.1091
184.9966
O
O
S
O
O
N2
3u
ppm

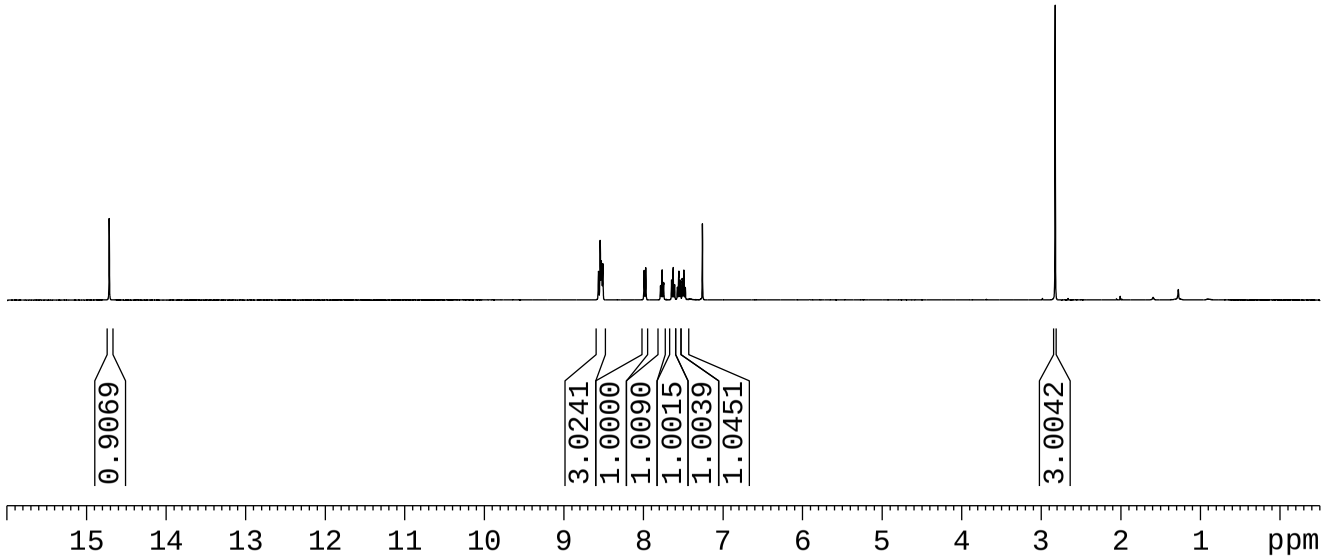
**4) NMR spectra of 4**



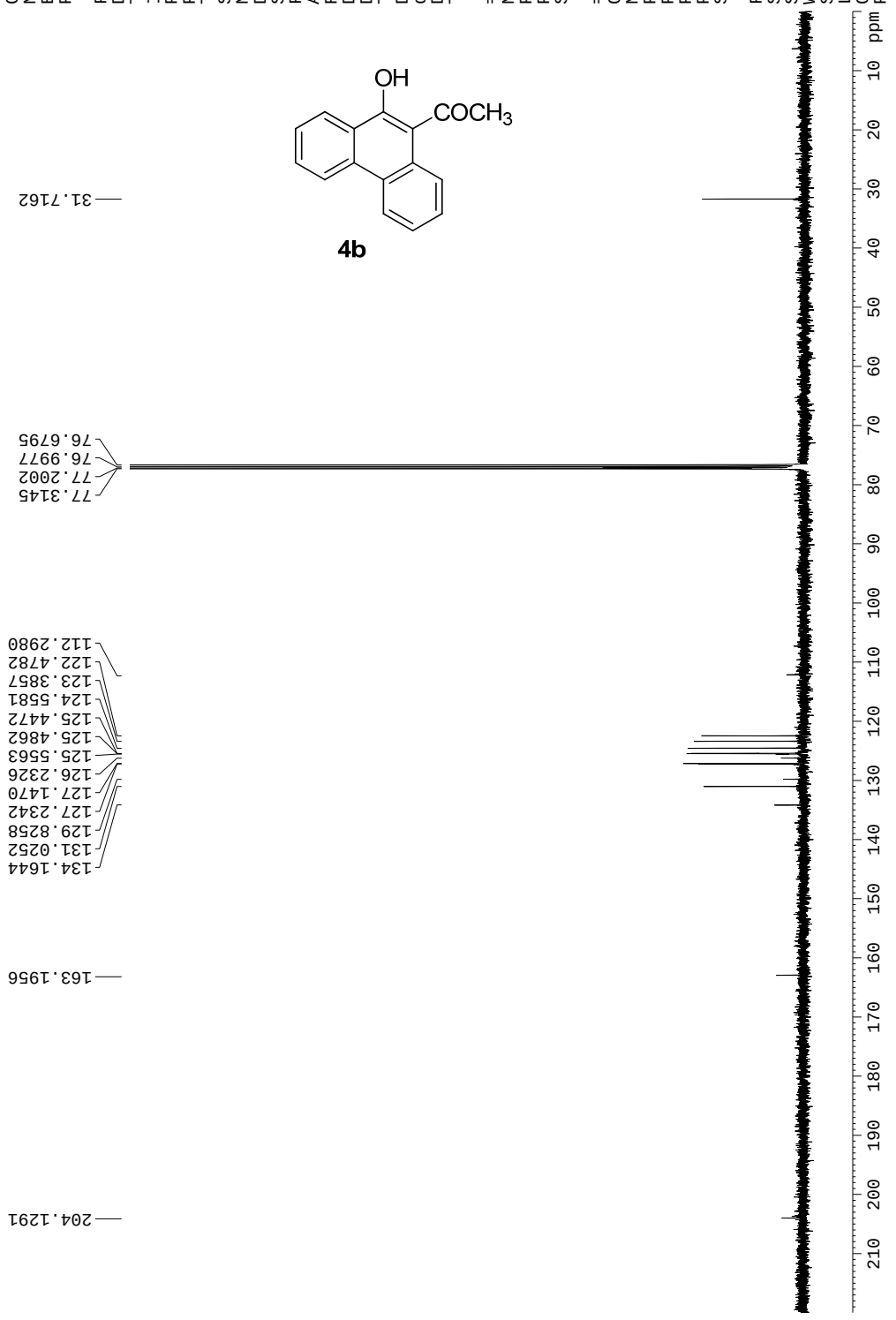
Current Data Parameters
NAME chiang2017
EXPNO 531021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170531
Time 15.09
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 803
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 18390.4
DW 26.550 usec
DE 6.50 usec
TE 304.2 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.10 usec
PL1 0.00 dB
SFO1 75.4763978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 18.80 dB
PL13 21.80 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677477 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
OH
CO2Et
4a
14.3706
62.0283
76.6330
77.0563
77.2586
77.4797
101.6129
122.4322
122.8570
124.2193
124.9922
125.3061
126.0135
126.1110
126.8472
127.5901
129.5154
130.4099
133.6973
162.7261
172.9194
ppm

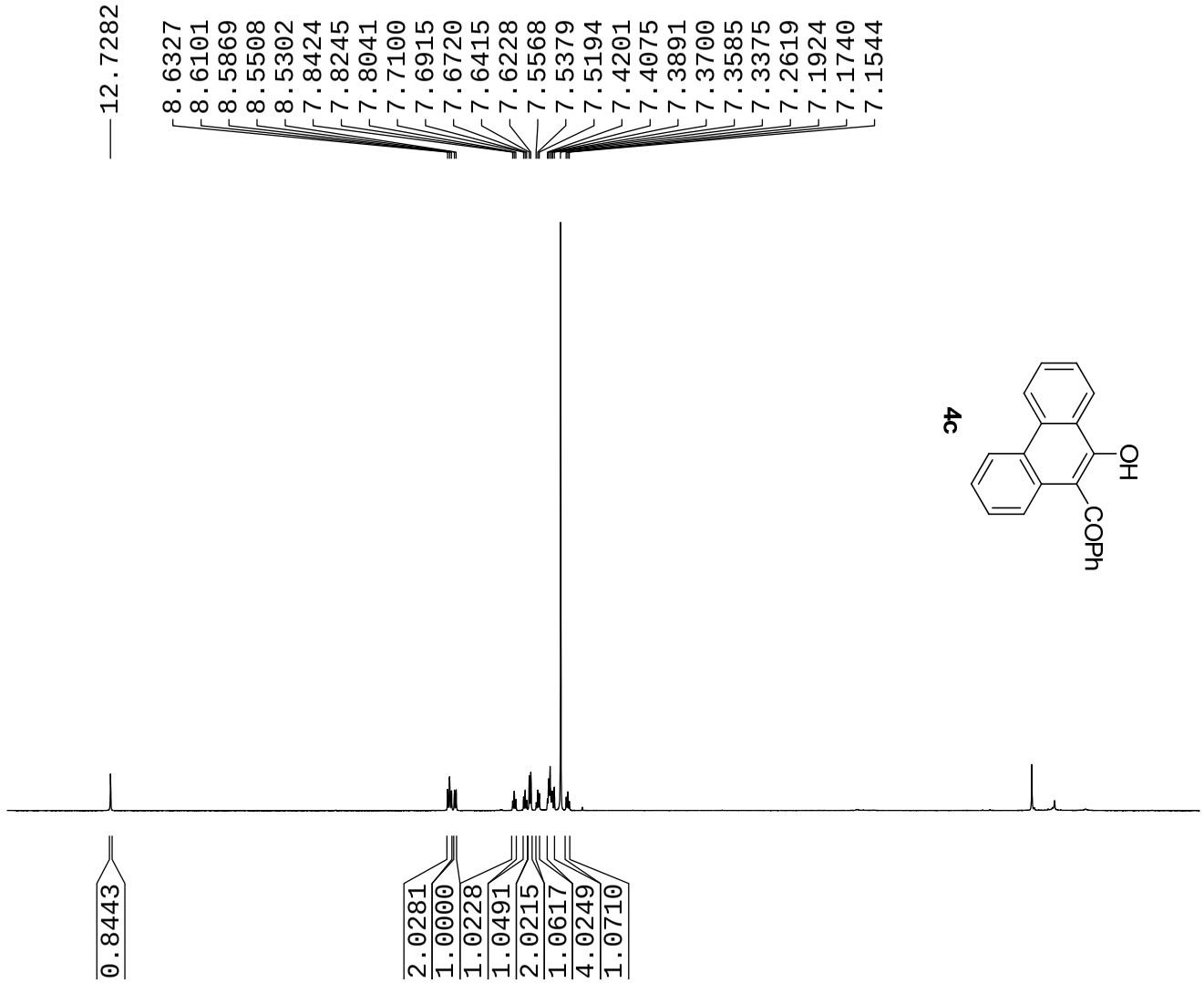



```
Current Data Parameters
NAME          chiang2017
EXPNO             713011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170713
Time                9.48
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    17
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             2.0447731 sec
RG                   203
DW                62.400 usec
DE                 6.50 usec
TE                304.9 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300087 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2017
EXPNO 710021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170710
Time_ 10.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2880
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 303.5 K
D1 0.10000000 sec
d11 0.03000000 sec
DELTA 0.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.80 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
COCH3
4b
31.7162
77.3145
77.2002
76.9977
76.6795
112.2980
122.4782
123.3857
124.5581
125.4472
125.4862
125.5563
126.2326
127.1470
127.2342
129.8258
131.0252
134.1644
163.1956
204.1291
ppm

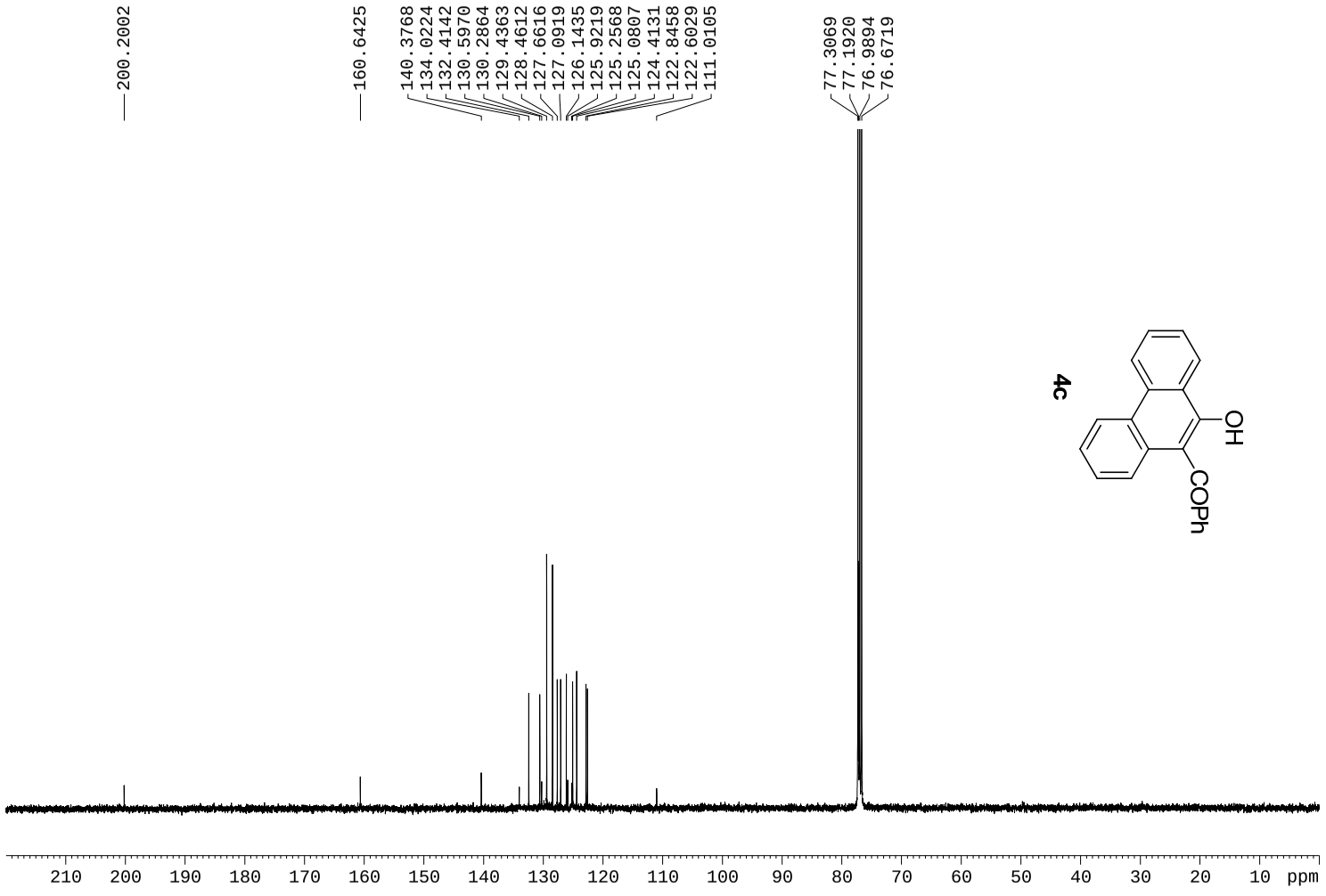






**4c**

```
Current Data Parameters
NAME          chiang2017
EXPNO             807061
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170807
Time               15.28
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                   101
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   575
DW                83.200 usec
DE                 6.50 usec
TE                302.0 K
D1           0.20000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300091 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

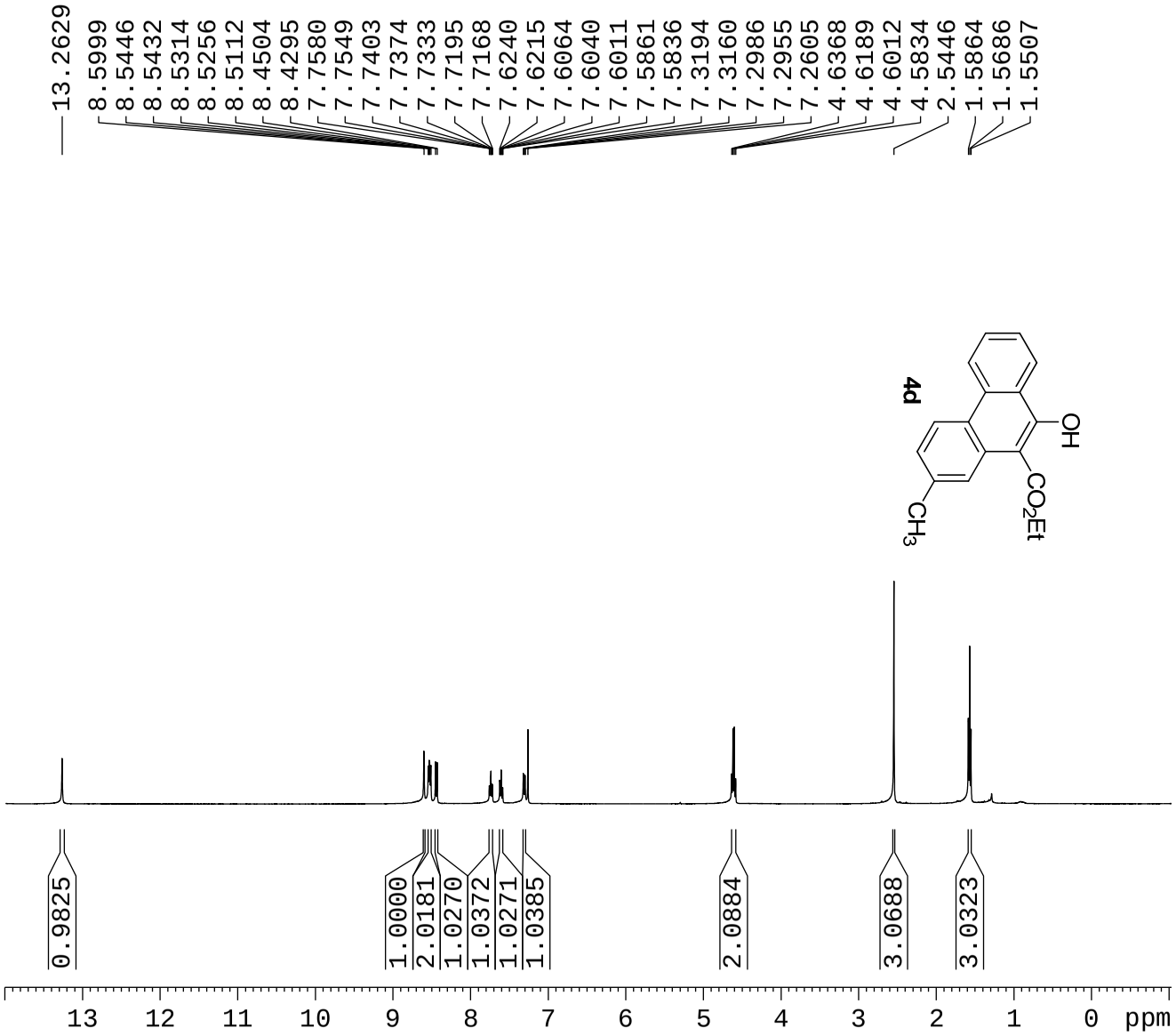






**4c**

```
Current Data Parameters
NAME          chiang2017
EXPNO             807073
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170808
Time                3.25
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                25250
SOLVENT           CDCl3
NS                15880
DS                    0
SWH           25252.525 Hz
FIDRES         1.000100 Hz
AQ            0.5000000 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               306.0 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

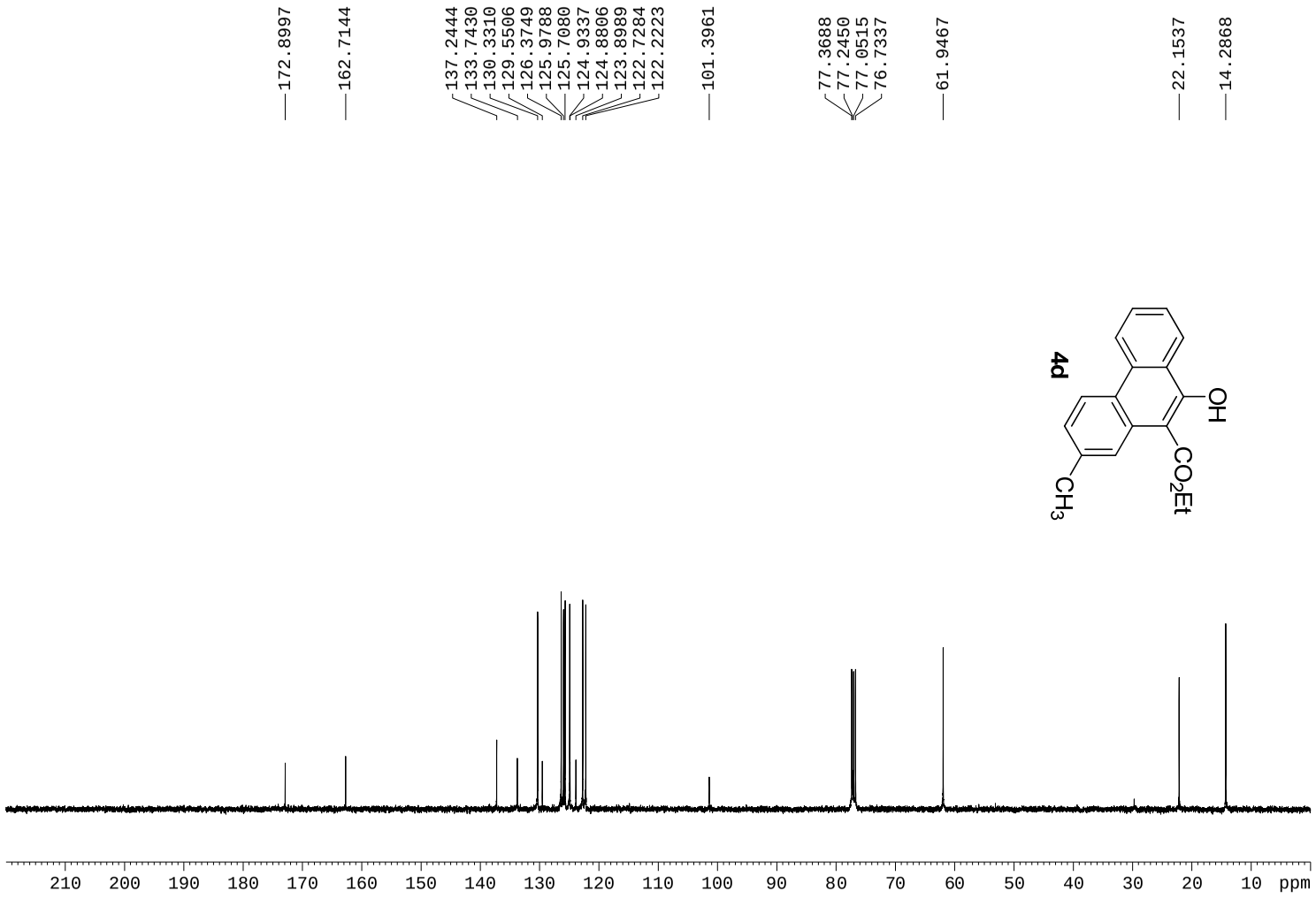



Current Data Parameters
NAME chiang2017
EXPNO 929011
PROCNO 1

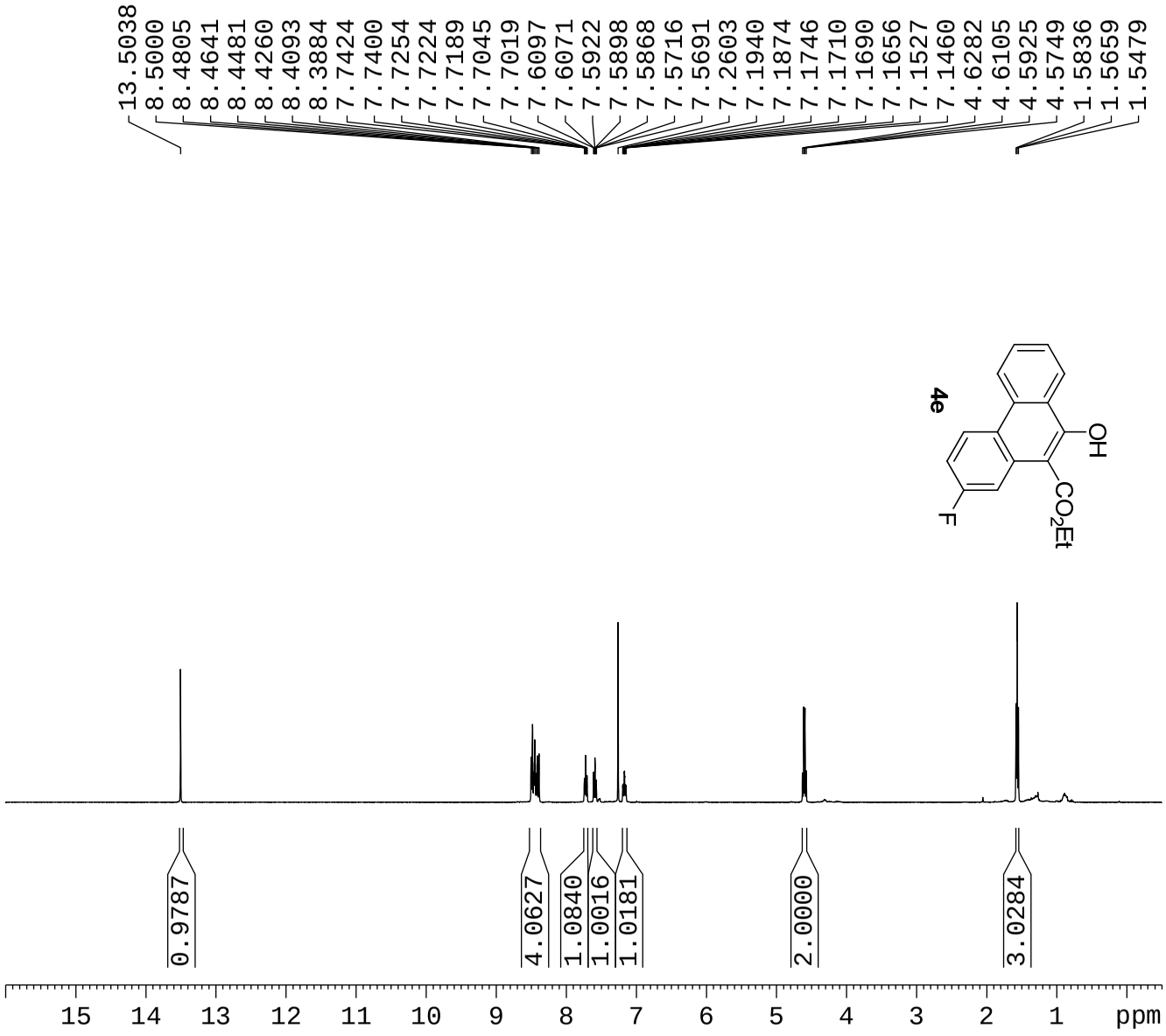
F2 - Acquisition Parameters
Date_ 20170929
Time 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 15
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 101
DW 83.200 usec
DE 6.50 usec
TE 300.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME          chiang2017
EXPNO             929021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170929
Time               14.24
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                25250
SOLVENT           CDCl3
NS                 1156
DS                    0
SWH           25252.525 Hz
FIDRES         1.000100 Hz
AQ            0.5000000 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                301.1 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




Current Data Parameters

| | |
|---|---|
| NAME | chiang2017 |
| EXPNO | 1118011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

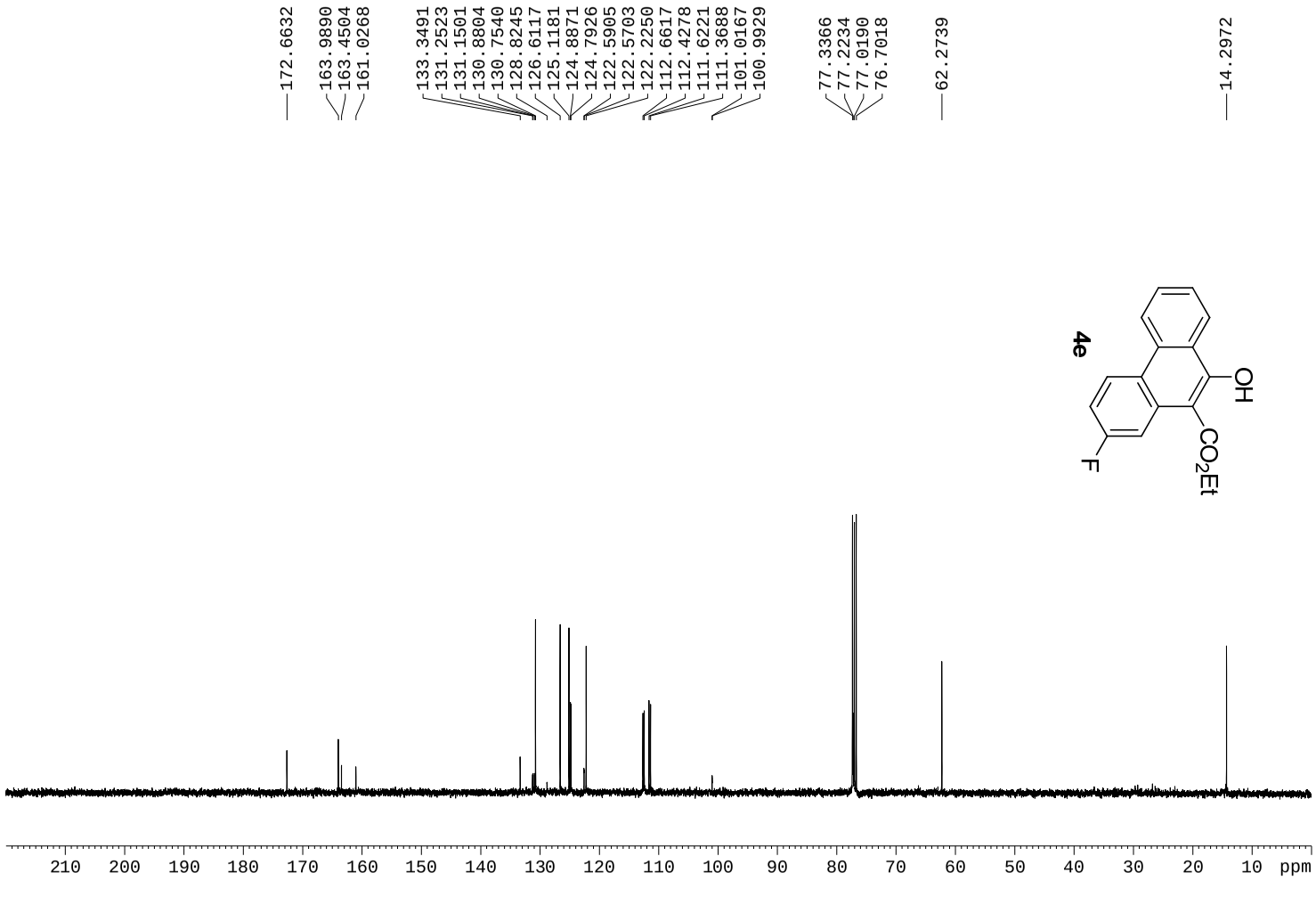
| | |
|---|---|
| Date_ | 20171118 |
| Time | 16.17 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 6 |
| DS | 0 |
| SWH | 8012.820 Hz |
| FIDRES | 0.244532 Hz |
| AQ | 2.0447731 sec |
| RG | 287 |
| DW | 62.400 usec |
| DE | 6.50 usec |
| TE | 302.3 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

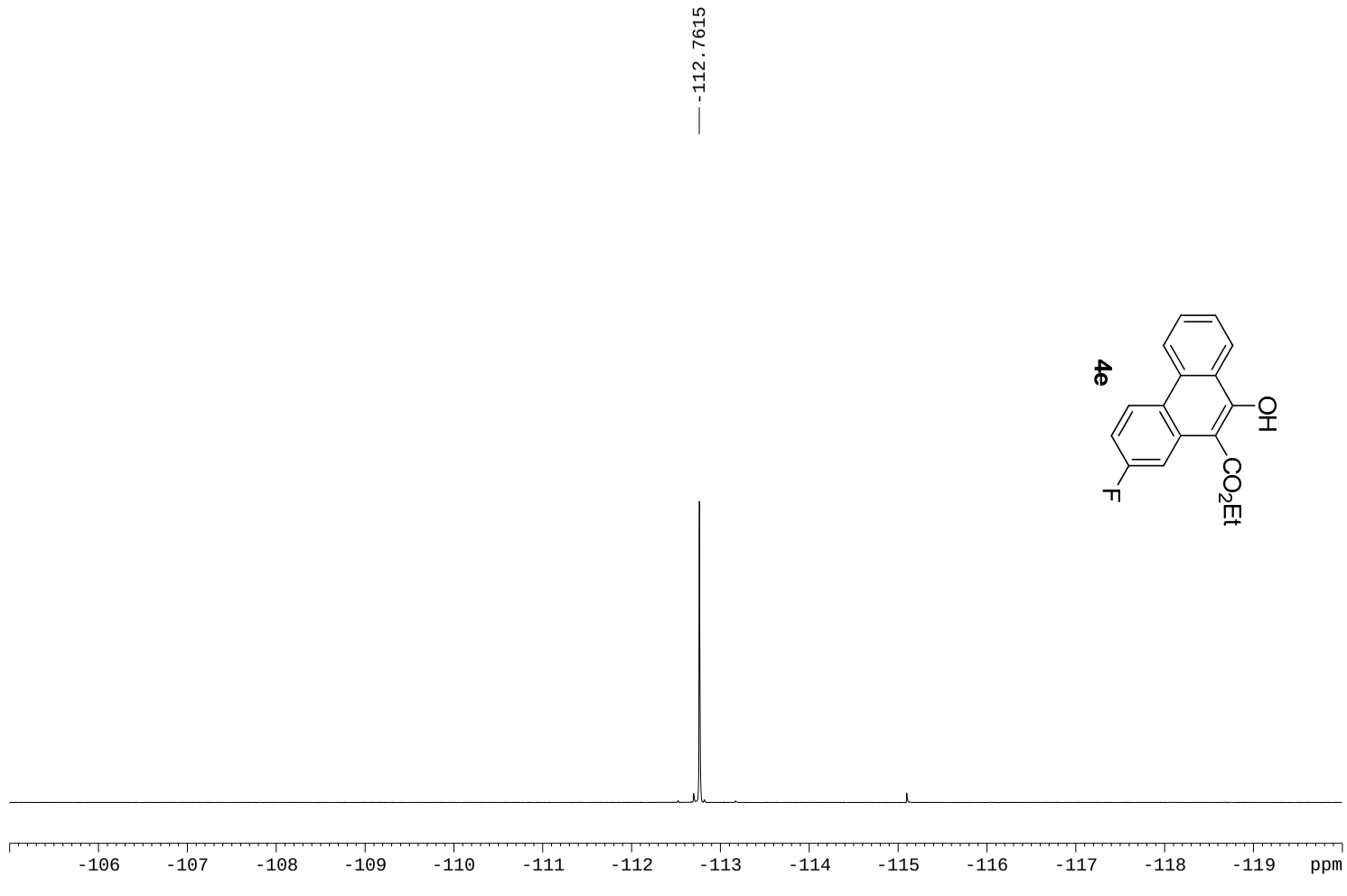
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300082 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |



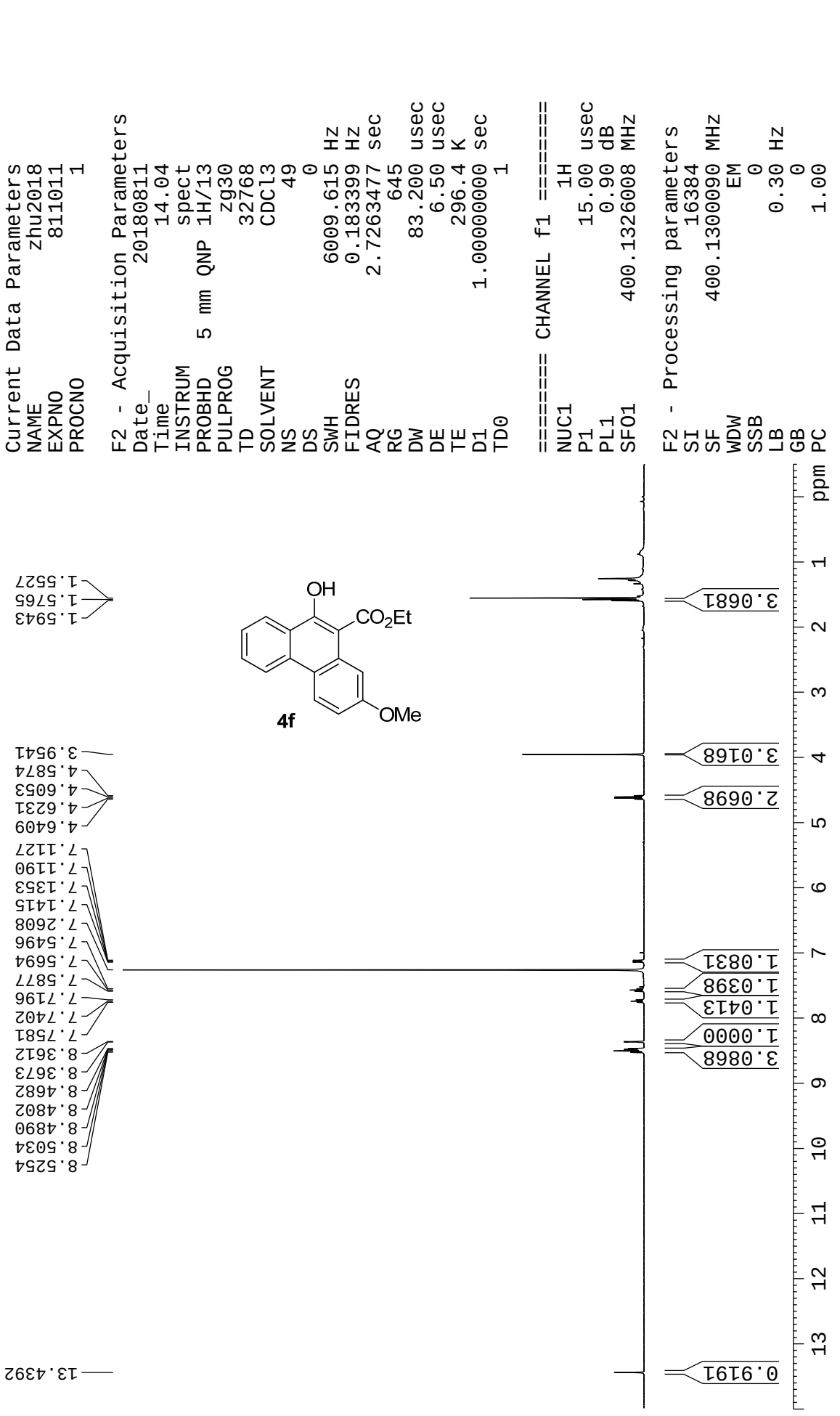

```
Current Data Parameters
NAME          chiang2017
EXPNO            1115021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171115
Time               10.09
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                25250
SOLVENT           CDCl3
NS                 1218
DS                    0
SWH           25252.525 Hz
FIDRES         1.000100 Hz
AQ            0.5000000 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               301.3 K
D1            0.20000000 sec
d11           0.03000000 sec
DELTA         0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                 6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

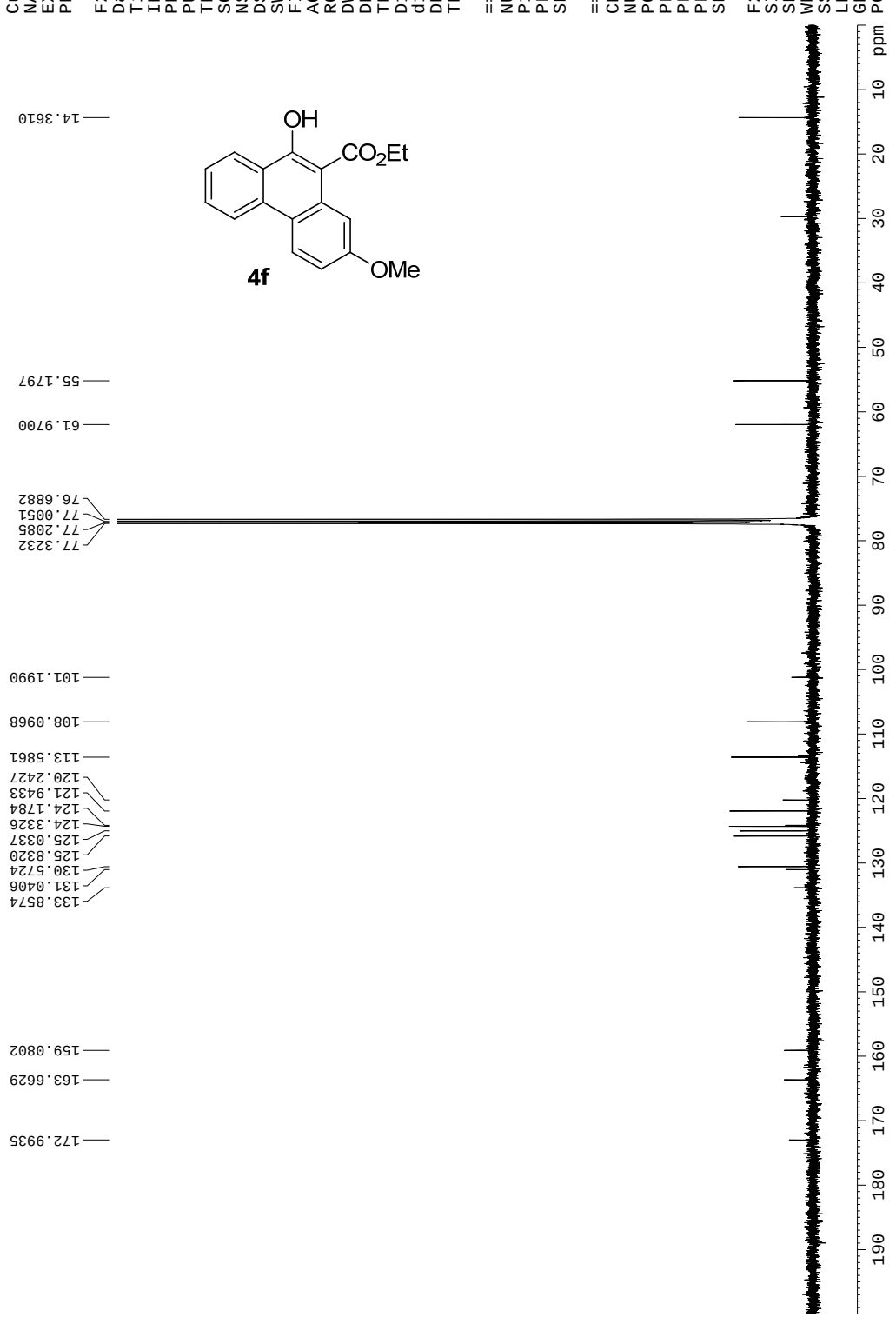


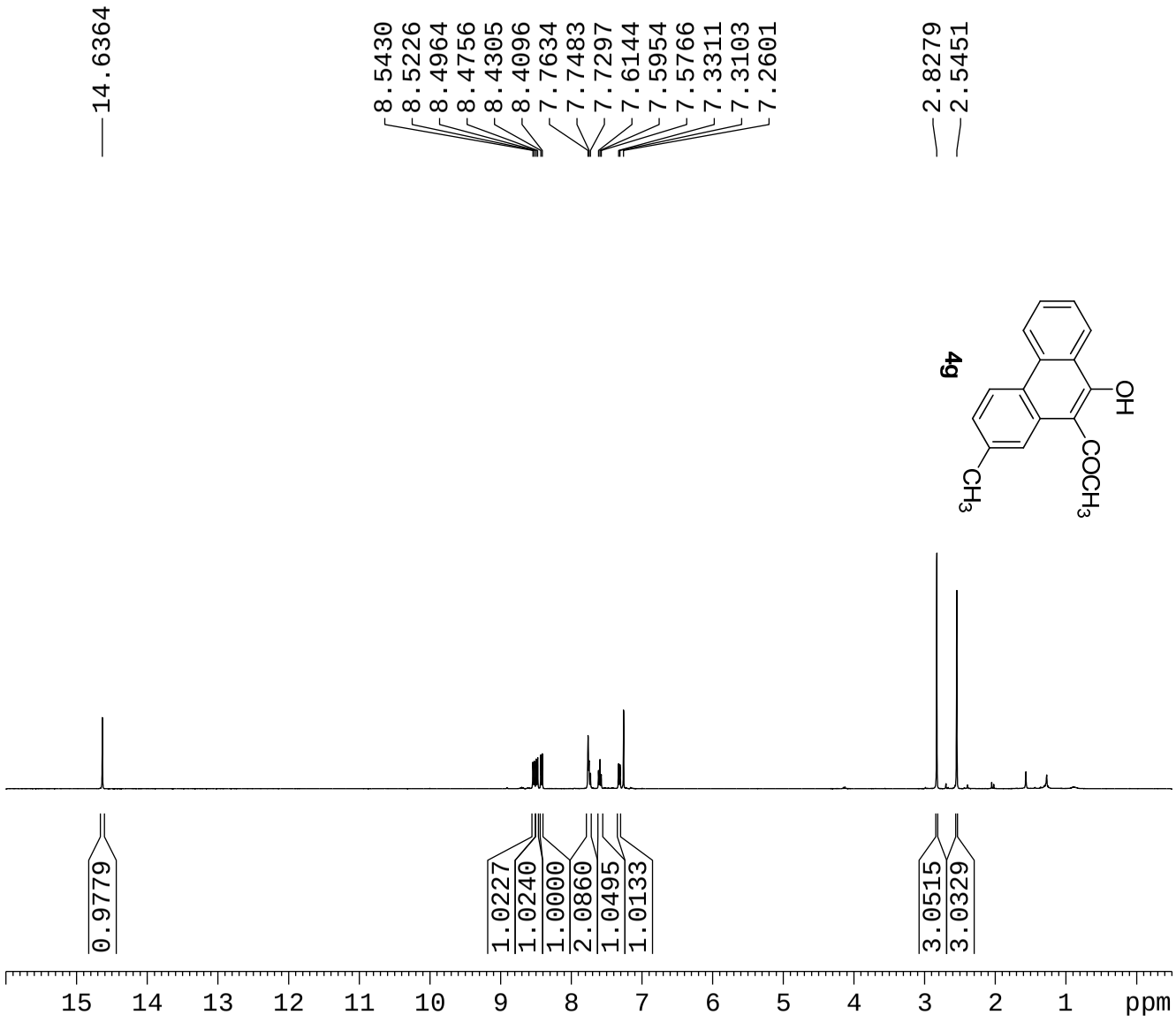




```
Current Data Parameters
NAME         chiang2017
EXPNO           1115031
PROCNO                1

F2 - Acquisition Parameters
Date_          20171115
Time              10.19
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG        zgfhigqn
TD              131072
SOLVENT           CDCl3
NS                   20
DS                    0
SWH           89285.711 Hz
FIDRES         0.681196 Hz
AQ             0.7340532 sec
RG                2050
DW                5.600 usec
DE                6.50 usec
TE                300.8 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 19F
P1                20.00 usec
PL1                2.50 dB
SFO1        376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                65536
SF          376.4983660 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```


Current Data Parameters
NAME zhu2018
EXPNO 811011
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180811
Time 14.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 49
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 645
DW 83.200 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
OMe
4f
13.4392
8.5254
8.5034
8.4890
8.4802
8.4682
8.3673
8.3612
7.7581
7.7402
7.7196
7.5877
7.5694
7.5496
7.2608
7.1415
7.1353
7.1190
7.1127
4.6409
4.6231
4.6053
4.5874
3.9541
1.5943
1.5765
1.5527
0.9191
3.0868
1.0000
1.0413
1.0398
1.0831
2.0698
3.0168
3.0681
ppm


Current Data Parameters
NAME zhu2018
EXPNO 811012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180811
Time 14.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11019
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 296.6 K
D1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000001 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
CO2Et
OMe
4f
14.3610
55.1797
61.9700
76.6882
77.0051
77.2085
77.3232
101.1990
108.0968
113.5861
120.2427
121.9433
124.1784
124.3326
125.0337
125.8320
130.5724
131.0406
133.8574
159.0802
163.6629
172.9935
ppm




S145

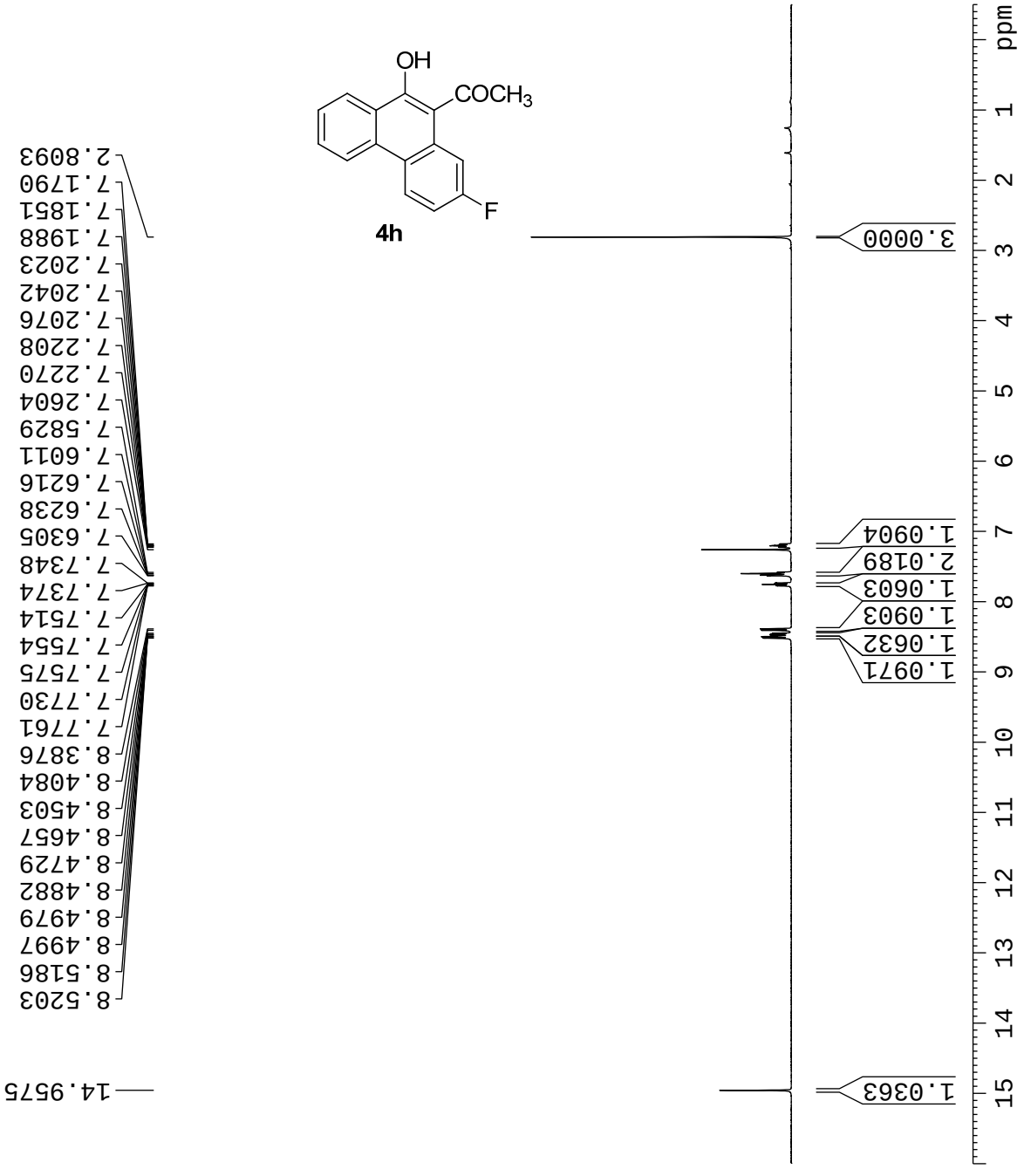
Current Data Parameters
NAME chiang2017
EXPNO 915011
PROCNO 1

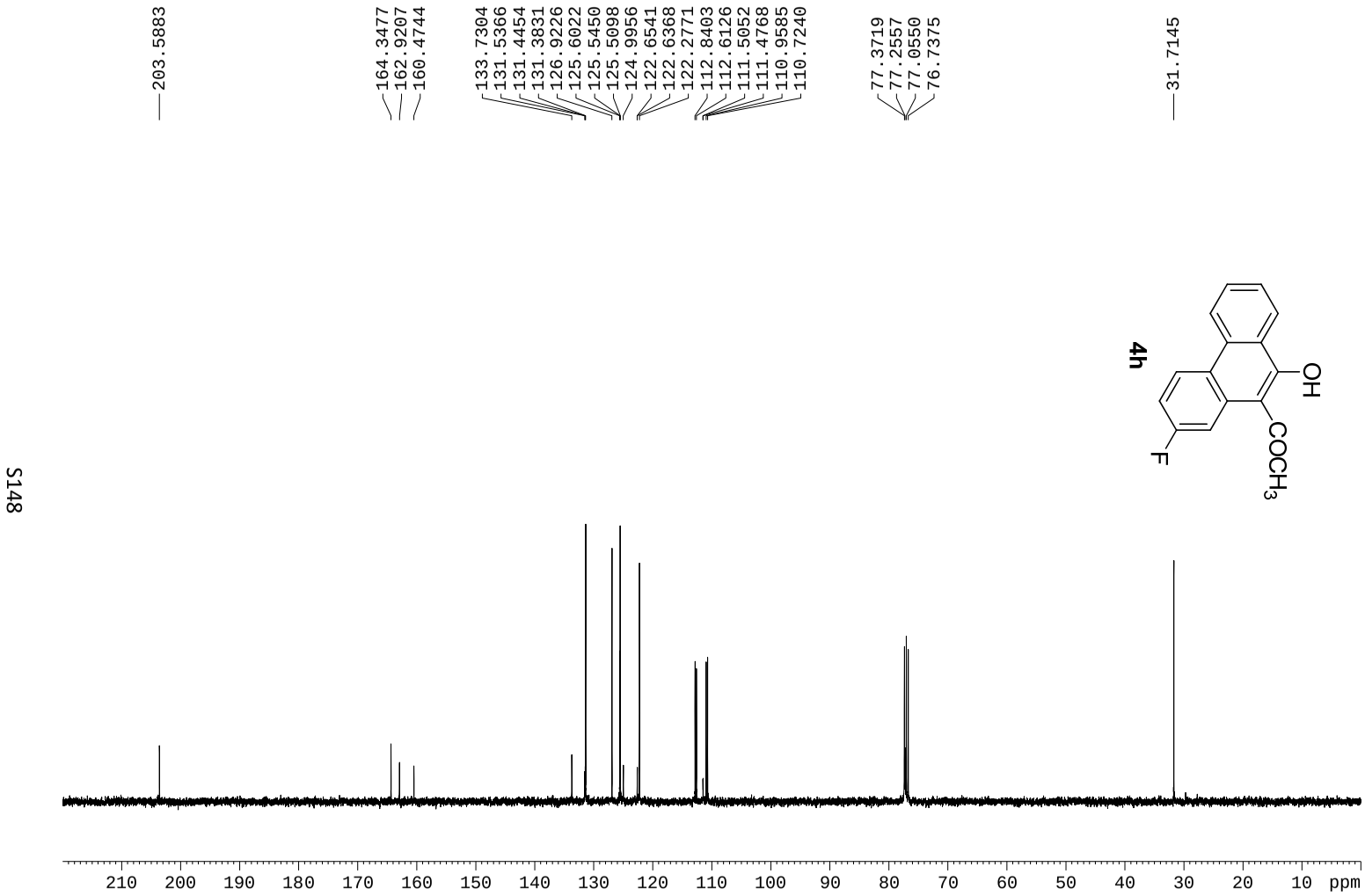
F2 - Acquisition Parameters
Date_ 20170915
Time 13.22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 11
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 256
DW 62.400 usec
DE 6.50 usec
TE 307.4 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

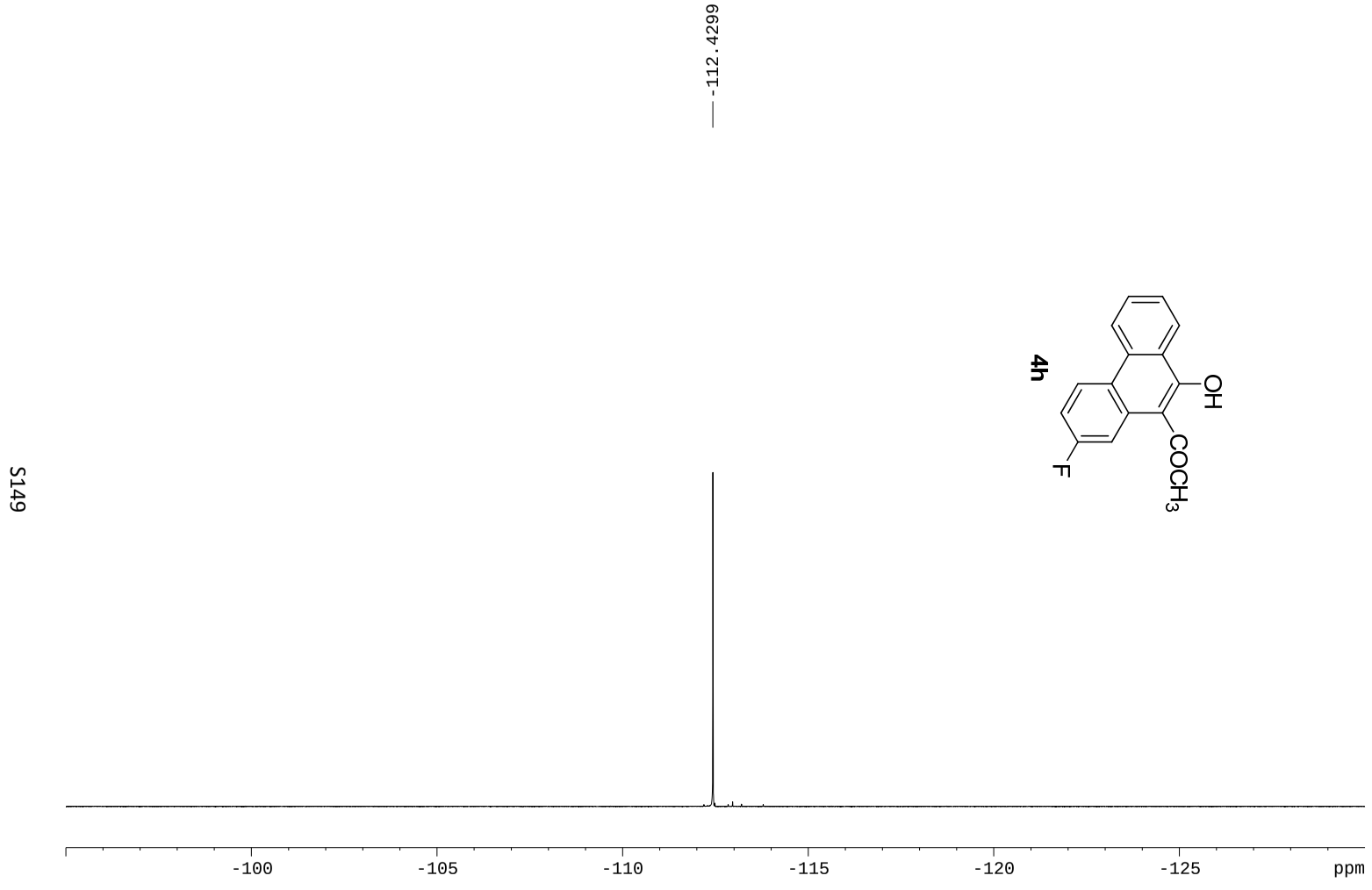
Current Data Parameters
NAME chiang2017
EXPNO 915021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170915
Time 13.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 709
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 307.4 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
COCH3
CH3
4g
21.9213
31.6514
76.6955
77.0133
77.2146
77.3306
112.0804
122.2485
123.2497
124.0127
125.1414
125.3858
125.5585
126.0066
126.6464
129.9275
130.9184
134.2236
137.0218
162.9245
203.9414
ppm


Current Data Parameters
NAME chiang2017
EXPNO 1102061
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171102
Time 16.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 22
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 362
DW 62.400 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
COCH3
F
4h
14.9575
8.5203
8.5186
8.4997
8.4979
8.4882
8.4729
8.4657
8.4503
8.4084
8.3876
7.7761
7.7730
7.7575
7.7554
7.7514
7.7374
7.7348
7.6305
7.6238
7.6216
7.6011
7.5829
7.2604
7.2270
7.2208
7.2076
7.2042
7.2023
7.1988
7.1851
7.1790
2.8093
3.0000
1.0904
2.0189
1.0603
1.0903
1.0632
1.0971
1.0363
ppm

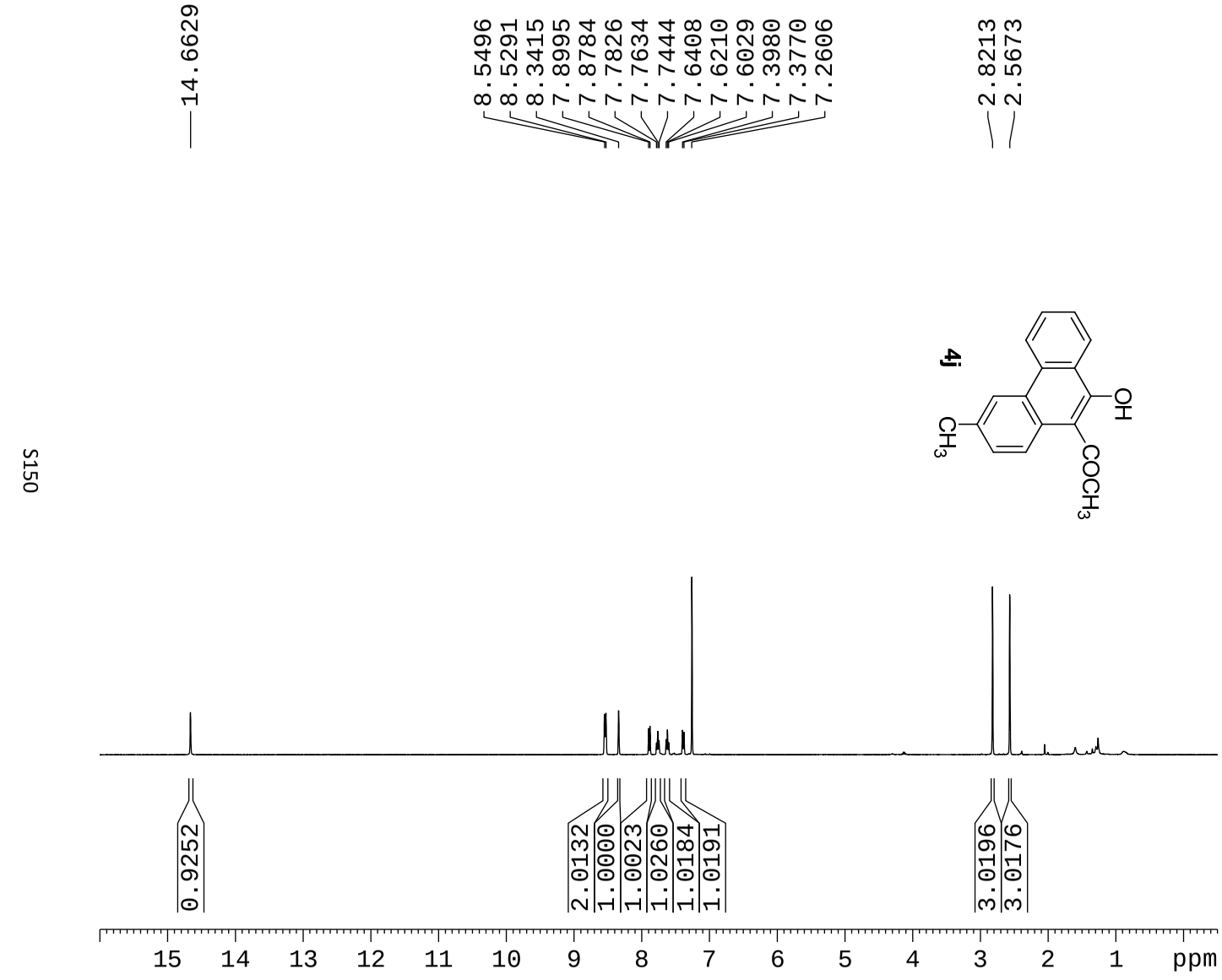




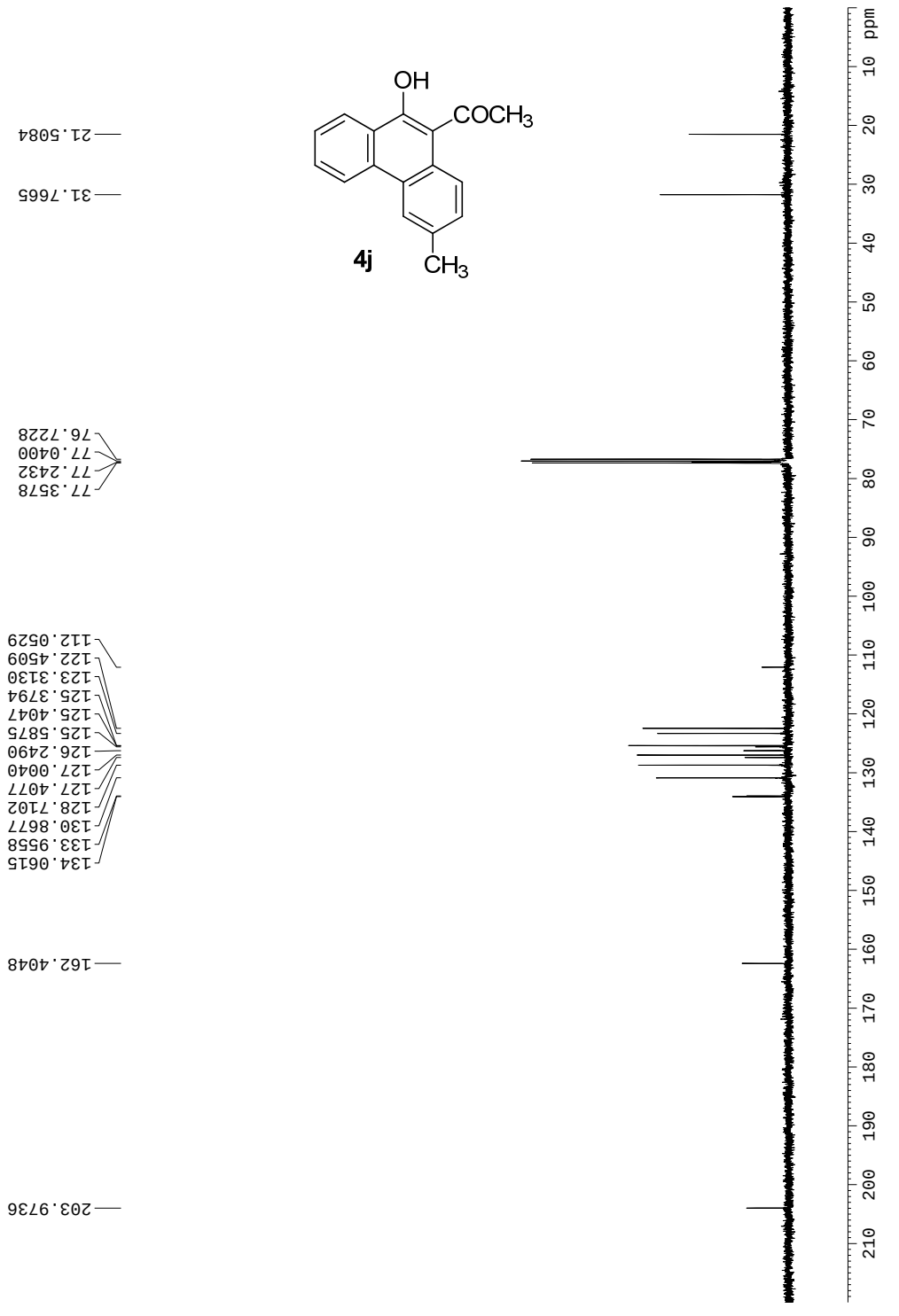

```
Current Data Parameters
NAME          chiang2017
EXPNO            1102071
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171102
Time               16.18
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                25250
SOLVENT           CDCl3
NS                  809
DS                    0
SWH           25252.525 Hz
FIDRES         1.000100 Hz
AQ            0.5000000 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE               295.1 K
D1            0.20000000 sec
d11           0.03000000 sec
DELTA         0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

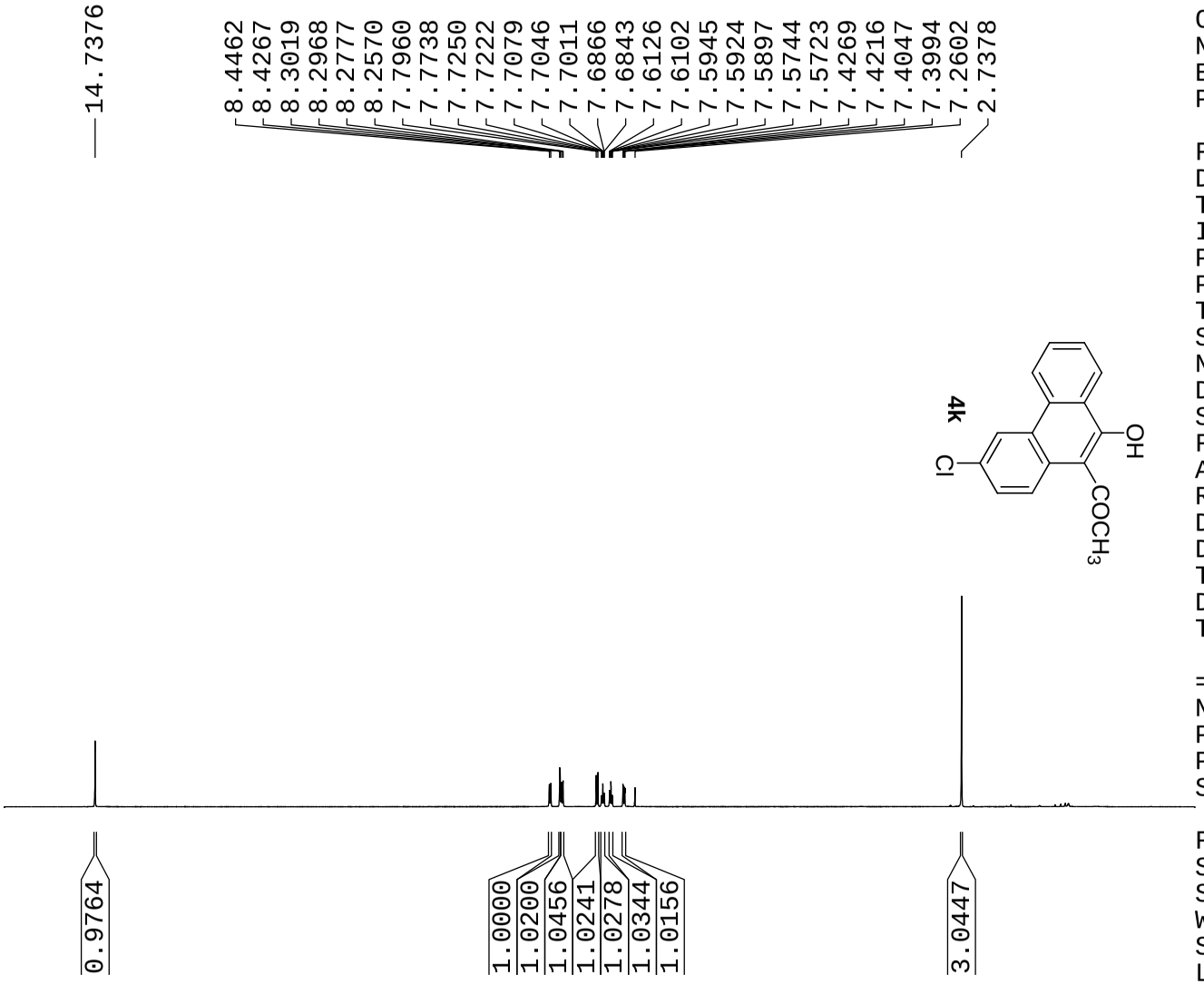



```
Current Data Parameters
NAME          chiang2017
EXPNO            1102081
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171102
Time               16.27
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgfhigqn
TD                131072
SOLVENT             CDCl3
NS                      6
DS                      0
SWH             89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340532 sec
RG                   2050
DW                  5.600 usec
DE                  6.50 usec
TE                 295.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  19F
P1                  20.00 usec
PL1                  2.50 dB
SFO1         376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -0.40 dB
PL12                15.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    65536
SF           376.4983660 MHz
WDW                      EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
Current Data Parameters
NAME          chiang2018
EXPNO             123031
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180123
Time               10.55
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    30
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              2.0447731 sec
RG                   406
DW                62.400 usec
DE                 6.50 usec
TE                296.5 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300080 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2018
EXPNO 123021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180123
Time 10.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 450
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 296.6 K
D1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000001 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
COCH3
4j
CH3
21.5084
31.7665
76.7228
77.0400
77.2432
77.3578
112.0529
122.4509
123.3130
125.3794
125.4047
125.5875
126.2490
127.0040
127.4077
128.7102
130.8677
133.9558
134.0615
162.4048
203.9736
ppm

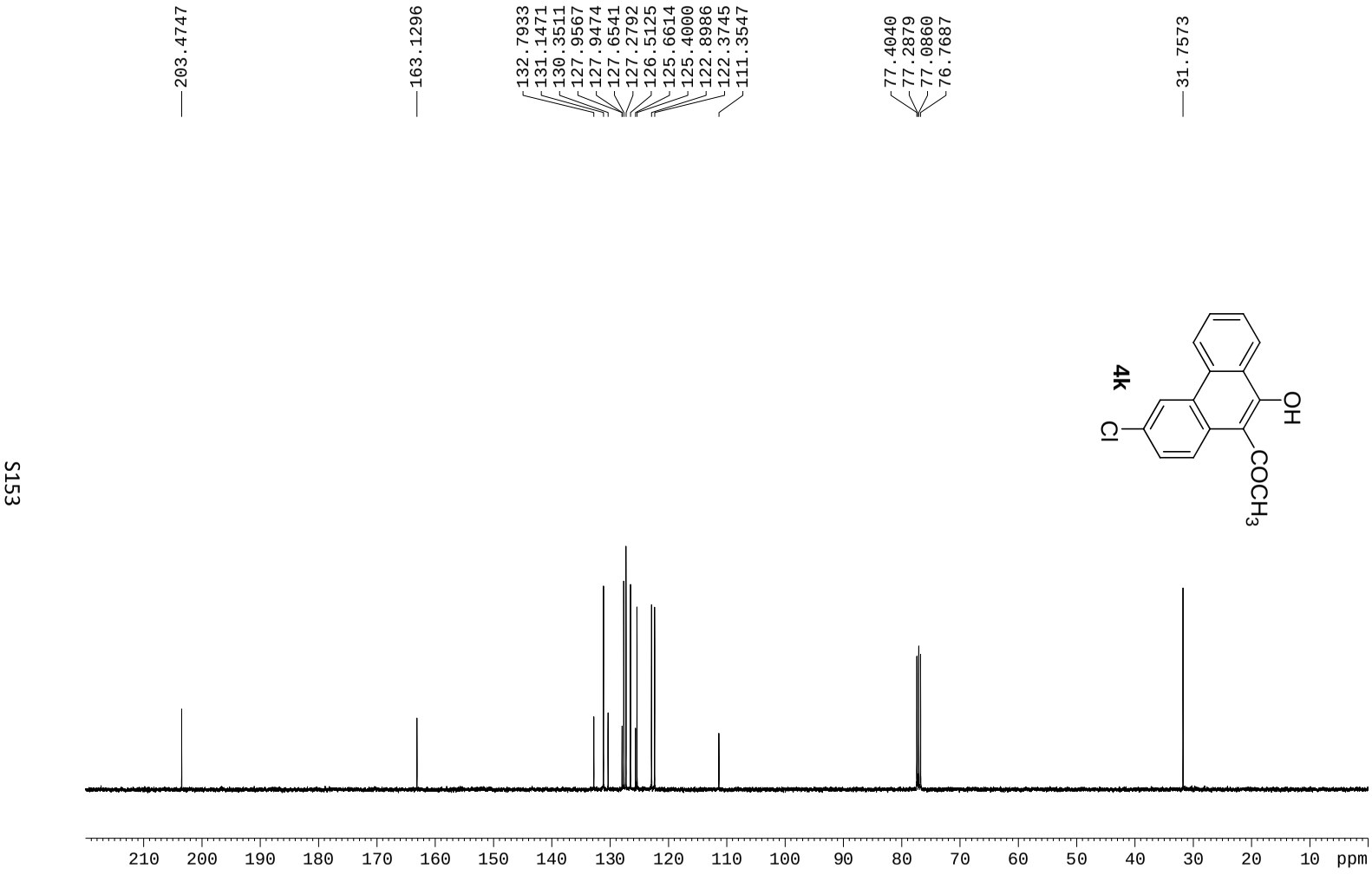



Current Data Parameters
NAME chiang2018
EXPNO 308011
PROCNO 1

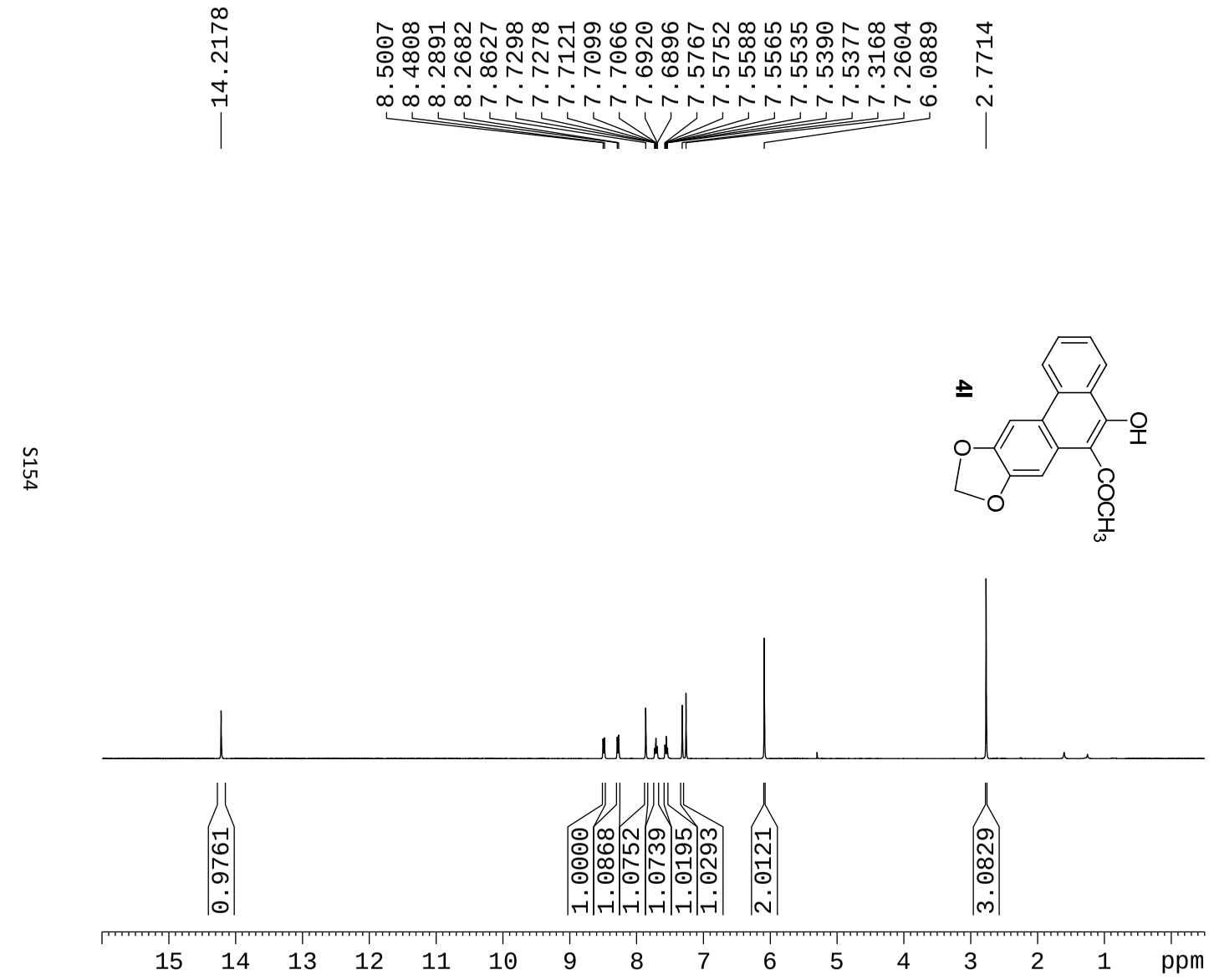
F2 - Acquisition Parameters
Date_ 20180308
Time 10.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 13
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 228
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME          chiang2018
EXPNO             308021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180308
Time               10.12
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  215
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                298.5 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

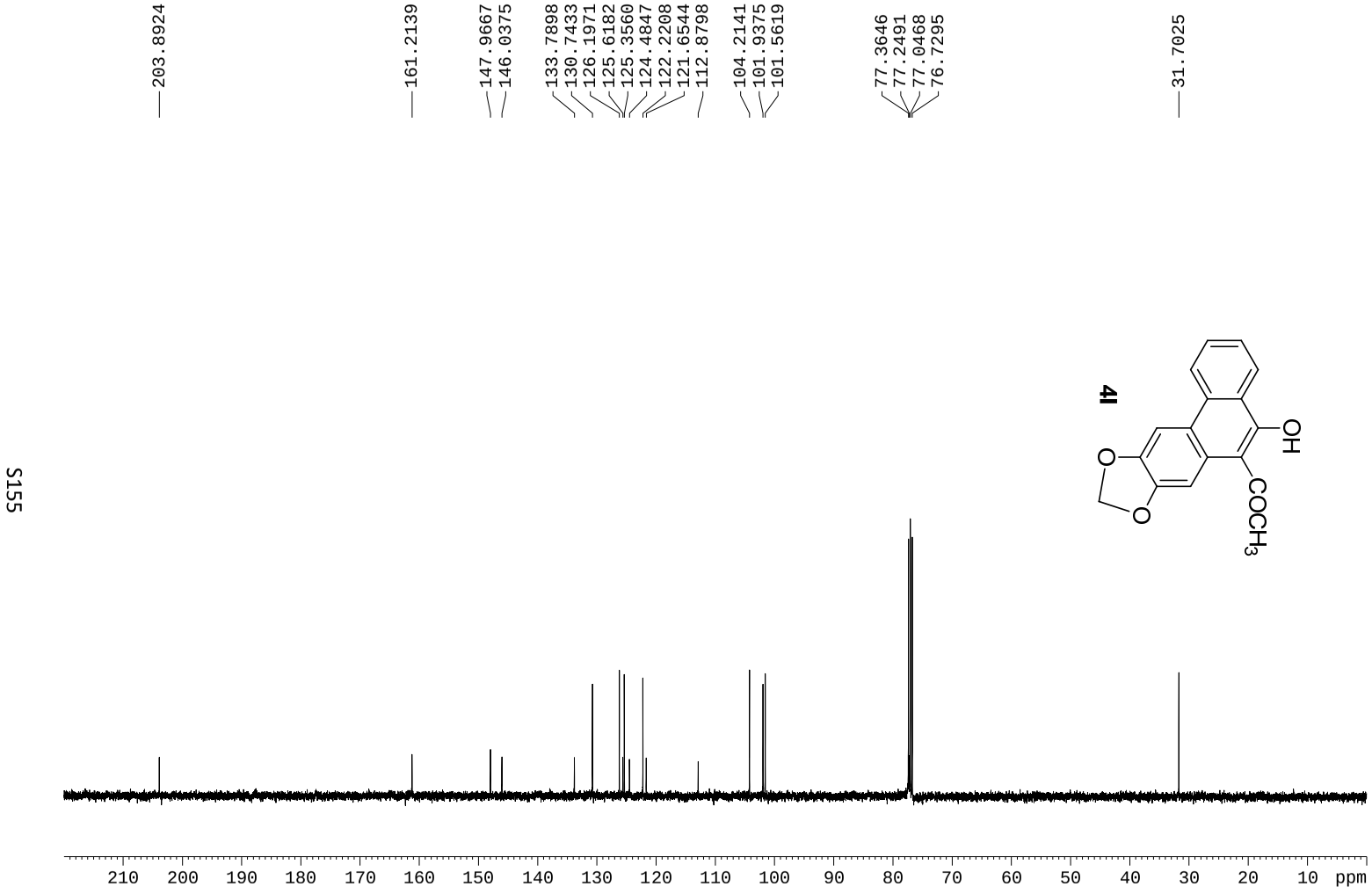



Current Data Parameters
NAME chiang2018
EXPNO 404031
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180404
Time 13.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 9
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 256
DW 62.400 usec
DE 6.50 usec
TE 294.2 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



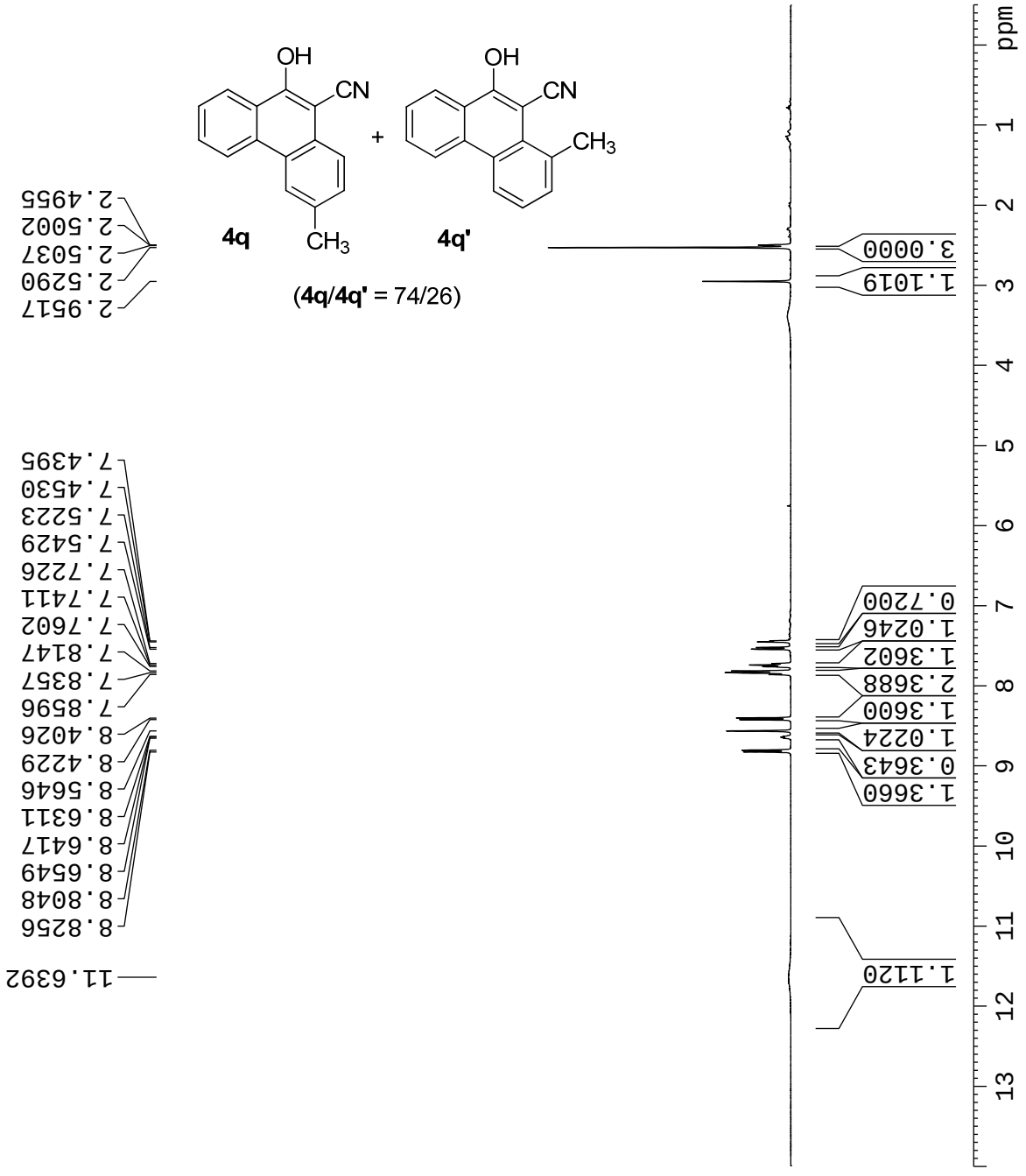

Current Data Parameters
NAME chiang2018
EXPNO 404041
PROCNO 1

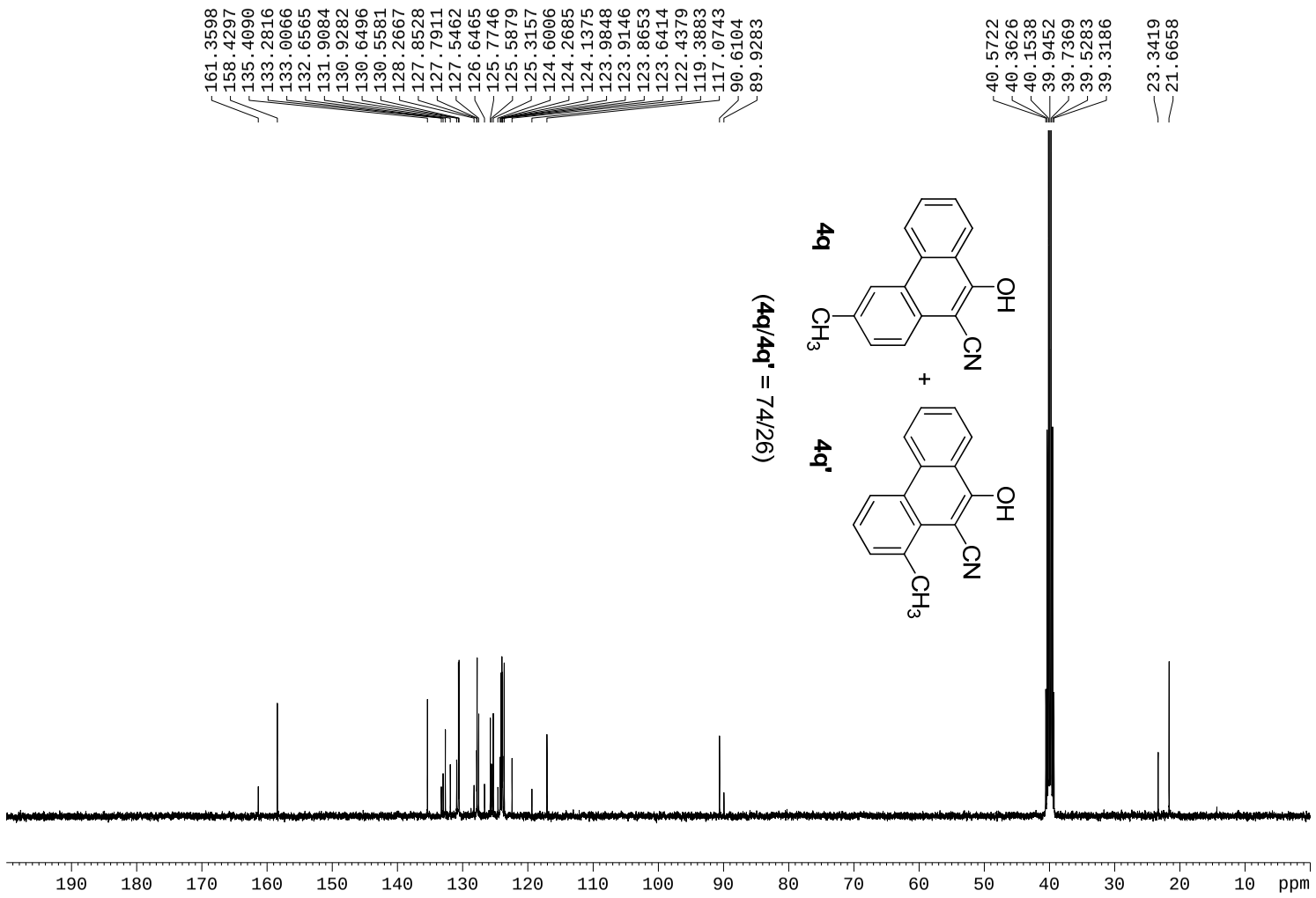
F2 - Acquisition Parameters
Date_ 20180404
Time 13.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 190
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 294.8 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz

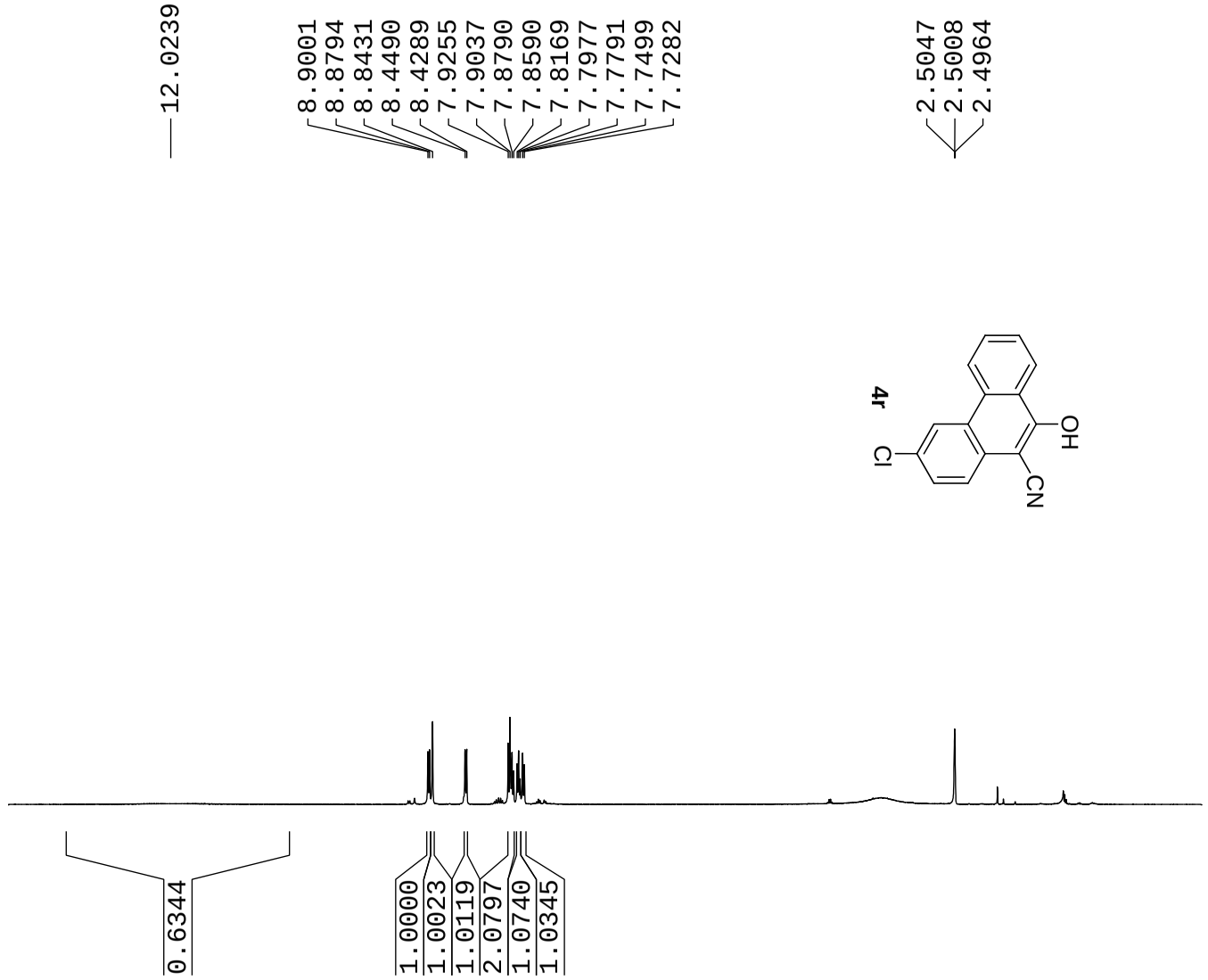
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

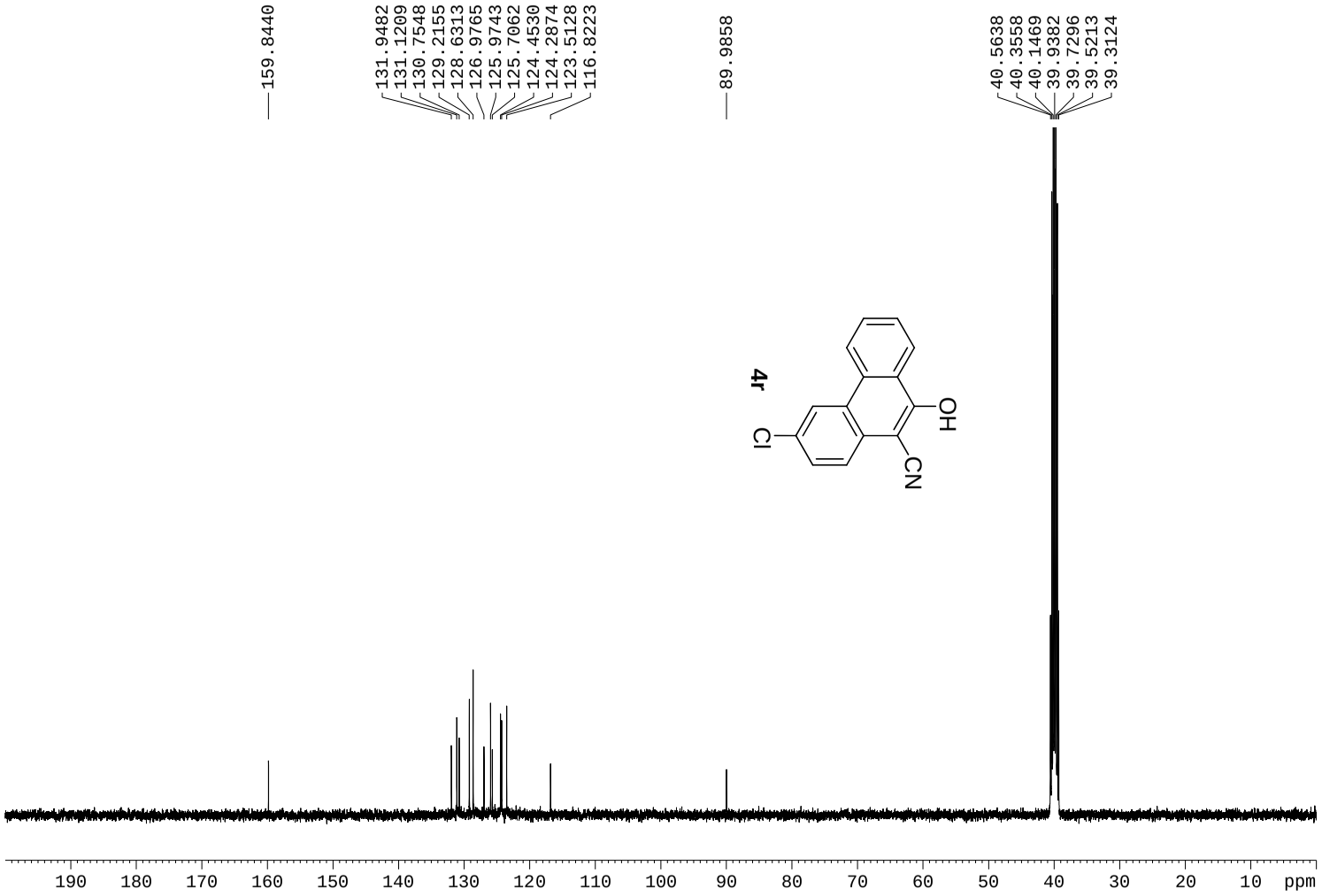

Current Data Parameters
NAME chen2018
EXPNO 201011
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180201
Time 10.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 30
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 362
DW 83.200 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300003 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CN
CH3
4q
4q'
(4q/4q' = 74/26)
2.9517
2.5290
2.5037
2.5002
2.4955
7.4395
7.4530
7.5223
7.5429
7.7226
7.7411
7.7602
7.8147
7.8357
7.8596
8.4026
8.4229
8.5646
8.6311
8.6417
8.6549
8.8048
8.8256
11.6392
3.0000
1.1019
1.3660
0.3643
1.0224
1.3600
2.3688
1.3602
1.0246
0.7200
1.1120
ppm




```
Current Data Parameters
NAME            chen2018
EXPNO             201012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180201
Time               11.11
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                    716
DS                      0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                294.6 K
D1           0.50000000 sec
d11          0.03000000 sec
DELTA        0.40000001 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```




```
Current Data Parameters
NAME            chen2018
EXPNO             312021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180312
Time               15.02
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    47
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              2.0447731 sec
RG                   645
DW                62.400 usec
DE                 6.50 usec
TE                292.7 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300020 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 312022 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20180312 |
| Time | 15.14 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | DMSO |
| NS | 1106 |
| DS | 0 |
| SWH | 25252.525 Hz |
| FIDRES | 0.385323 Hz |
| AQ | 1.2976629 sec |
| RG | 2050 |
| DW | 19.800 usec |
| DE | 6.50 usec |
| TE | 292.8 K |
| D1 | 0.50000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 0.40000001 sec |
| TD0 | 1 |

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

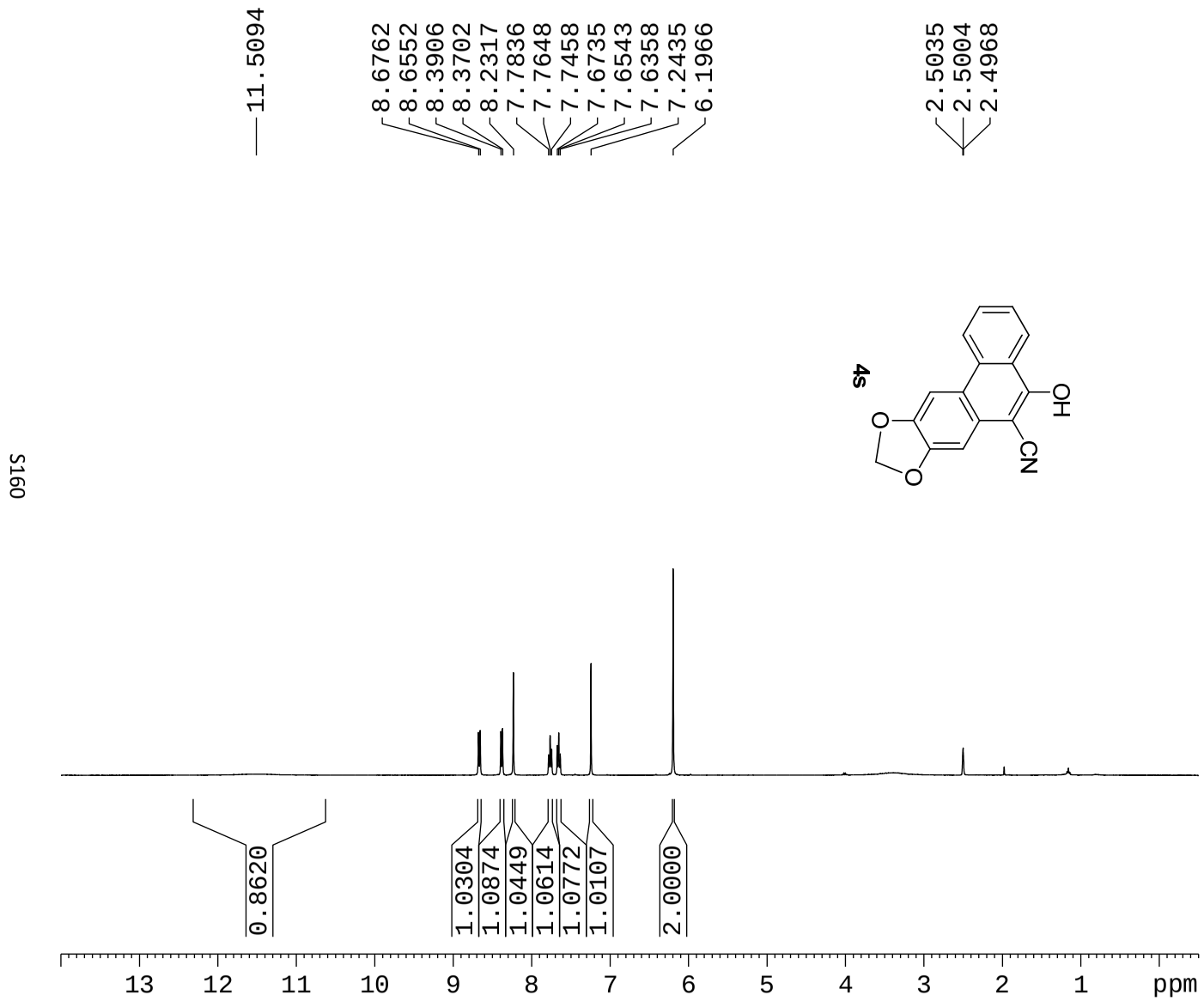
| | |
|---|---|
| NUC1 | 13C |
| P1 | 10.00 usec |
| PL1 | 6.20 dB |
| SFO1 | 100.6243395 MHz |

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

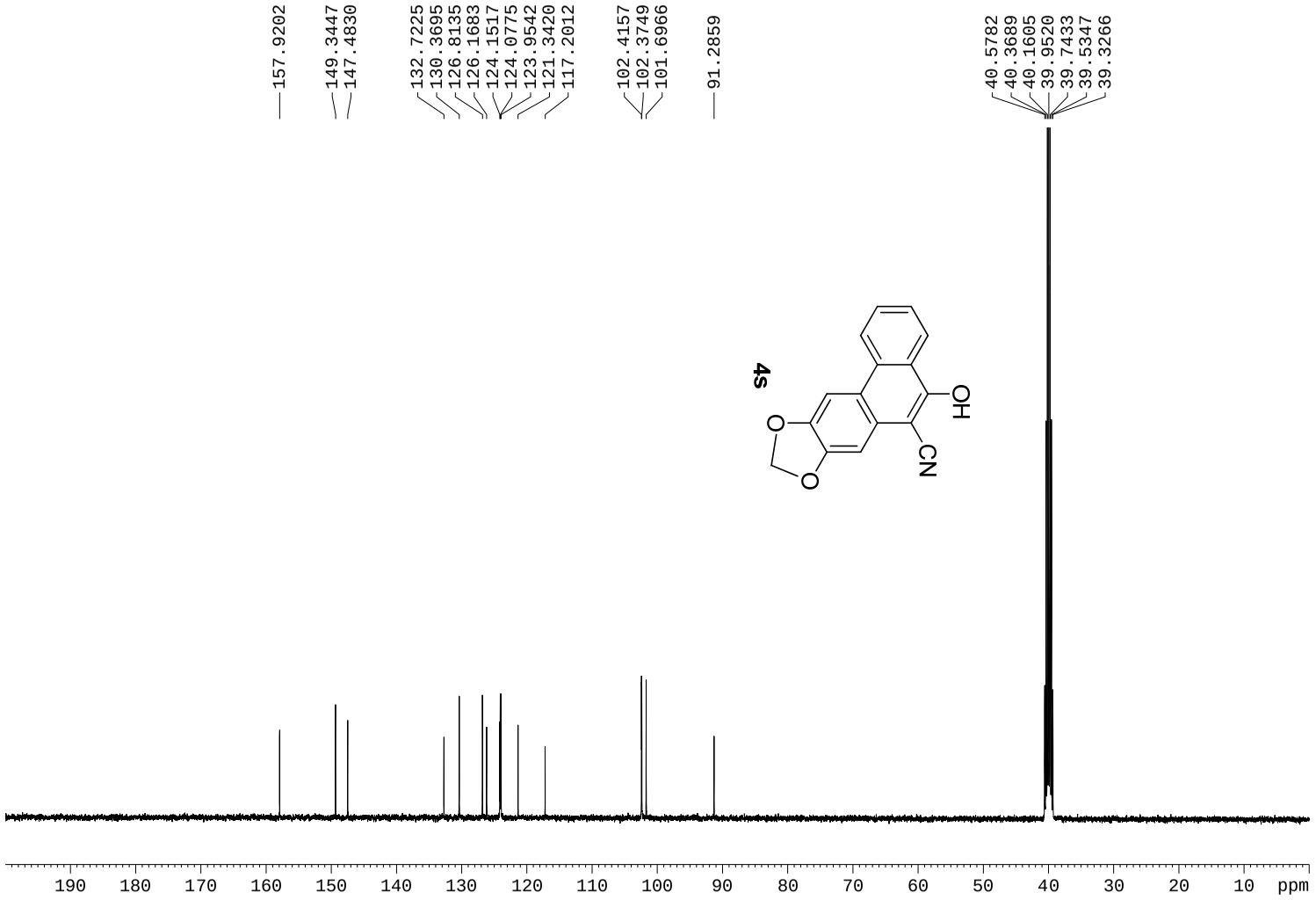
| | |
|---|---|
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 90.00 usec |
| PL2 | -0.40 dB |
| PL12 | 15.80 dB |
| PL13 | 18.50 dB |
| SFO2 | 400.1316005 MHz |

F2 - Processing parameters

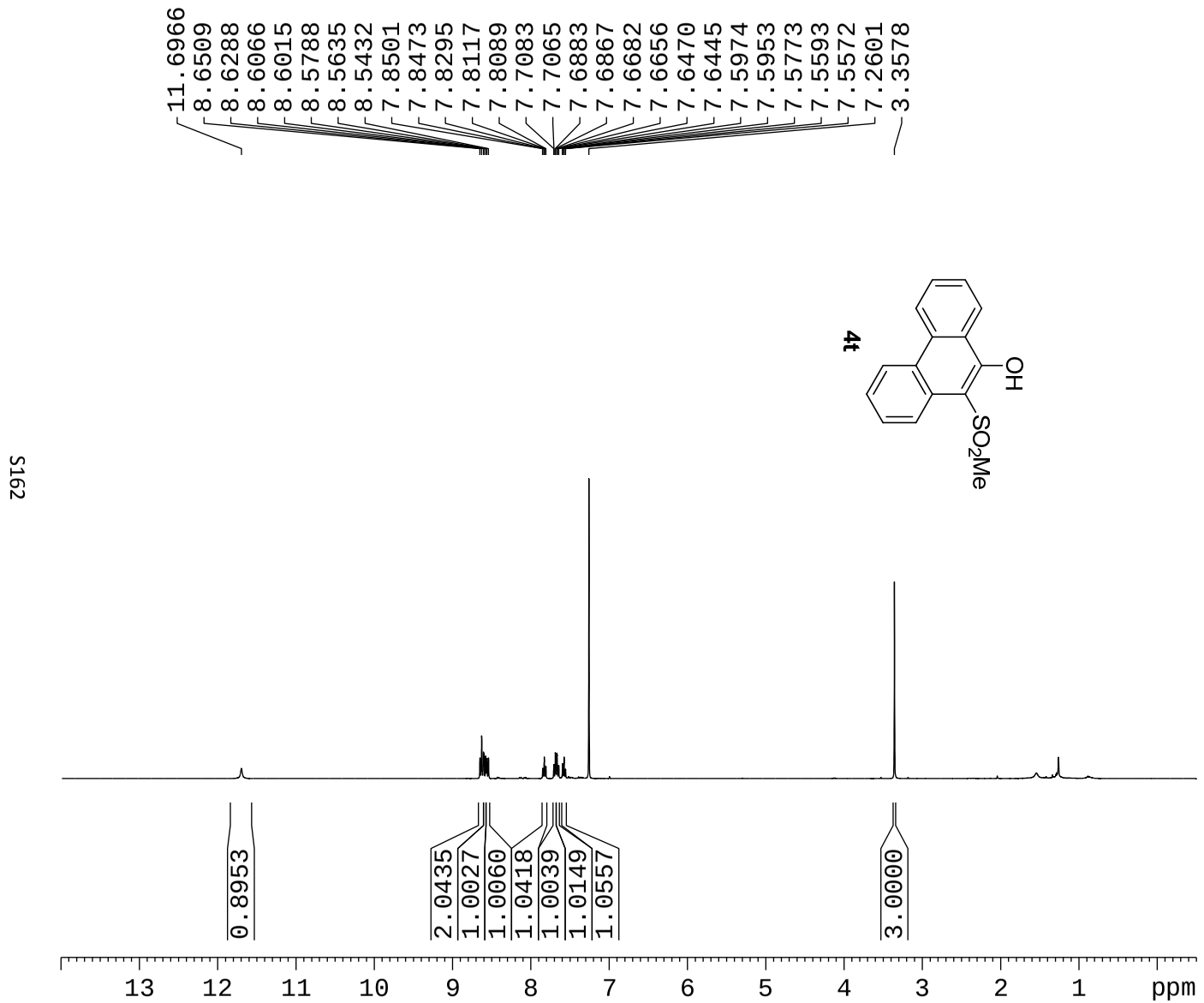
| | |
|---|---|
| SI | 32768 |
| SF | 100.6127690 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |



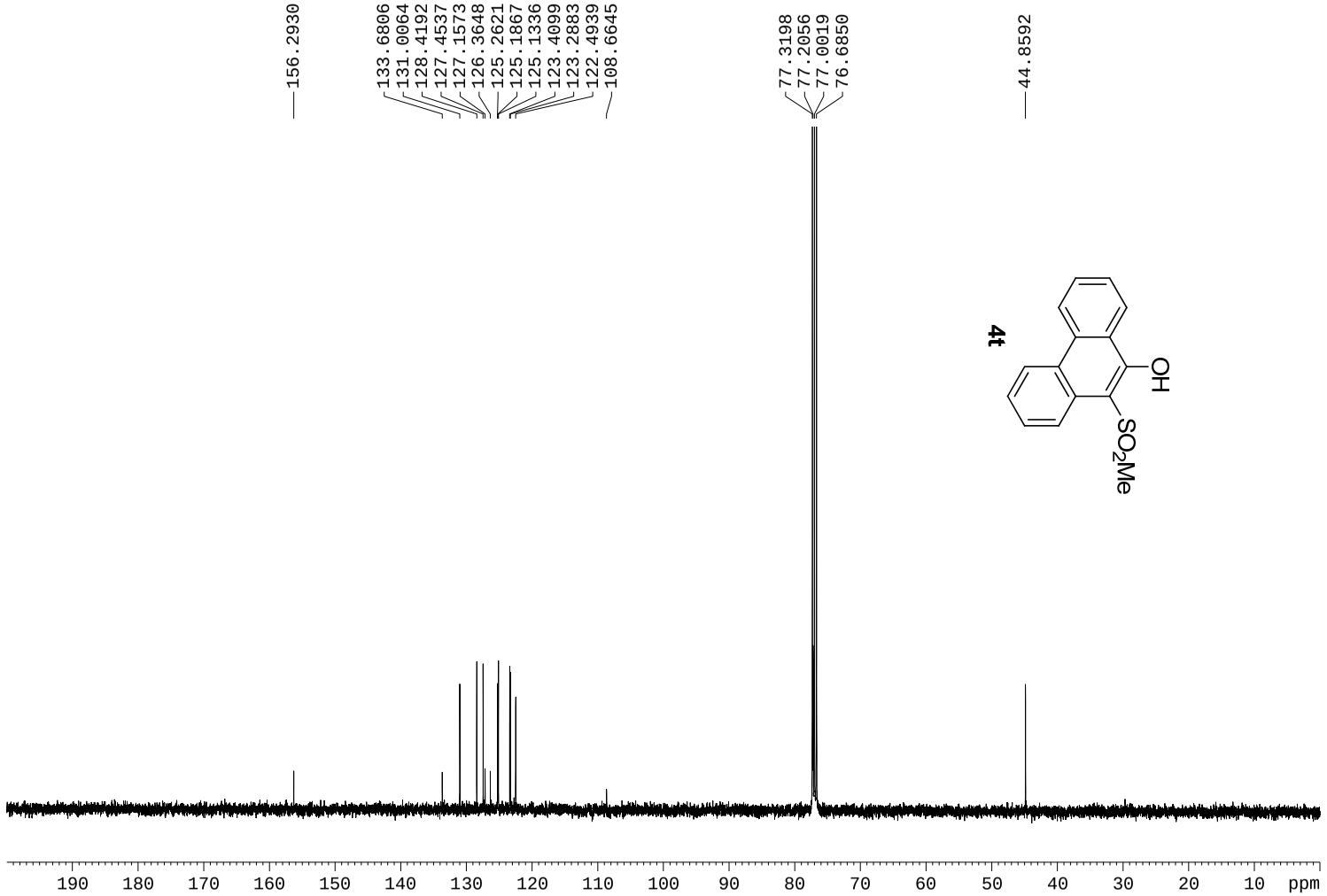

```
Current Data Parameters
NAME                  chen2018
EXPNO                   411011
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20180411
Time                     10.44
INSTRUM                  spect
PROBHD   5 mm QNP 1H/13
PULPROG                  zg30
TD                      32768
SOLVENT                  DMSO
NS                         22
DS                          0
SWH                  6009.615 Hz
FIDRES               0.183399 Hz
AQ                   2.7263477 sec
RG                         256
DW                      83.200 usec
DE                       6.50 usec
TE                      295.2 K
D1                   2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       15.00 usec
PL1                       0.90 dB
SFO1              400.1326008 MHz

F2 - Processing parameters
SI                       16384
SF                400.1300018 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```




```
Current Data Parameters
NAME            chen2018
EXPNO             411012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180411
Time               10.56
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  722
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                  456
DW               19.800 usec
DE                6.50 usec
TE                295.4 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



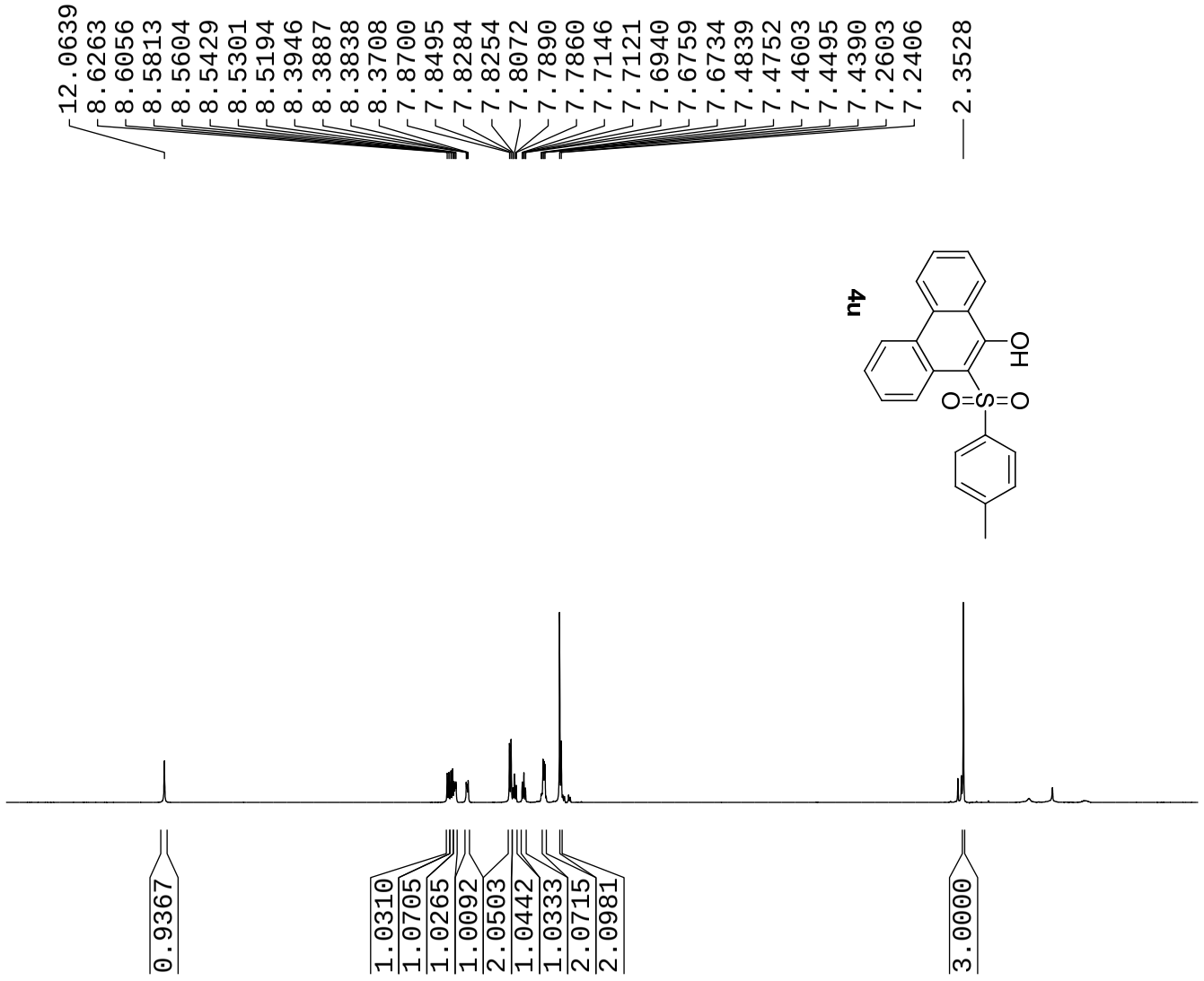

```
Current Data Parameters
NAME            chen2017
EXPNO             704011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170704
Time               14.51
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    43
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   575
DW                83.200 usec
DE                 6.50 usec
TE                305.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300090 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```







```
Current Data Parameters
NAME            chen2017
EXPNO             704015
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170704
Time               15.13
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  831
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                304.7 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.80 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




Current Data Parameters

| | |
|---|---|
| NAME | chen2017 |
| EXPNO | 920021 |
| PROCNO | 1 |

F2 - Acquisition Parameters

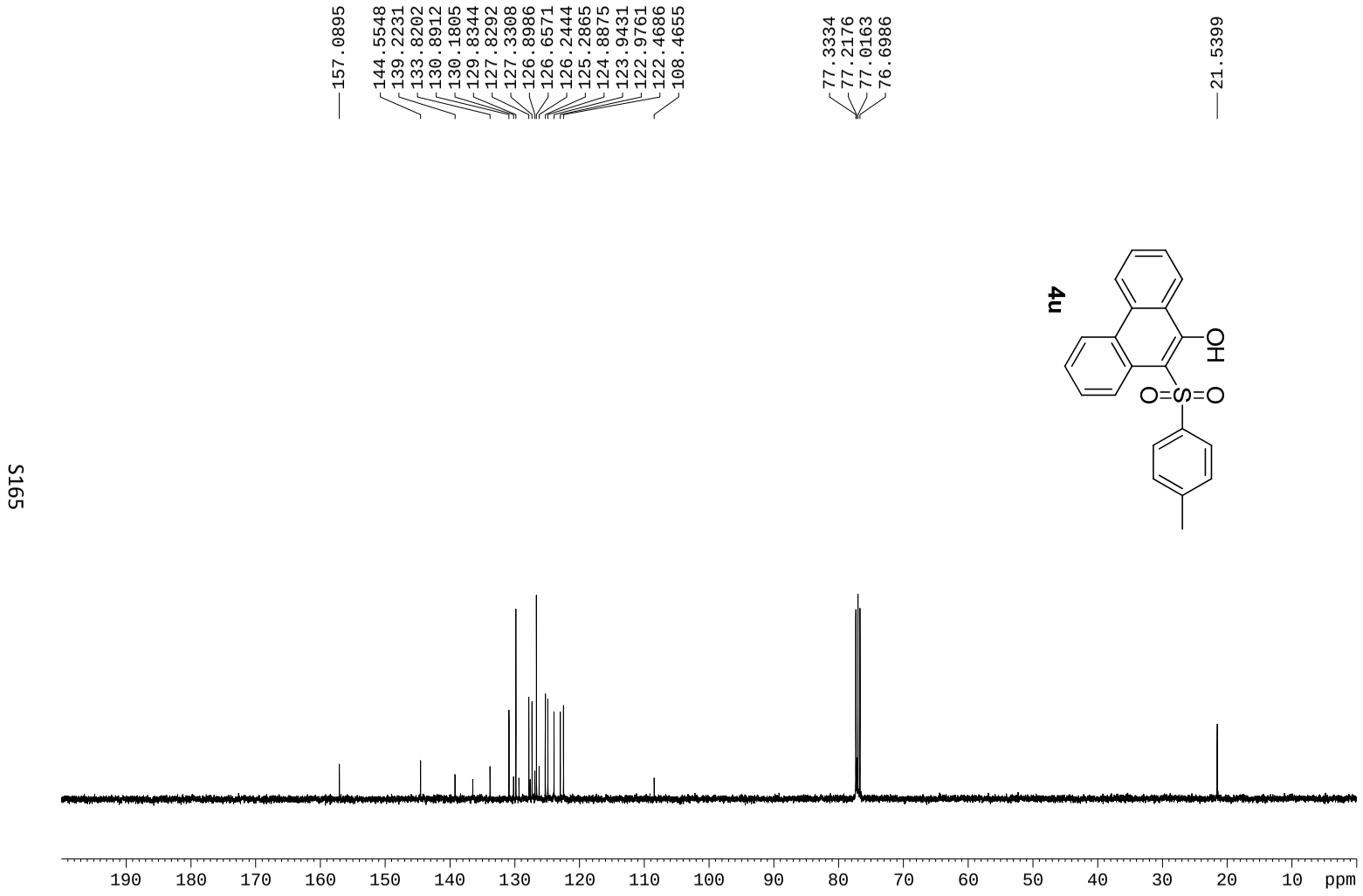
| | |
|---|---|
| Date_ | 20170920 |
| Time | 10.39 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 18 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 287 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 305.8 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

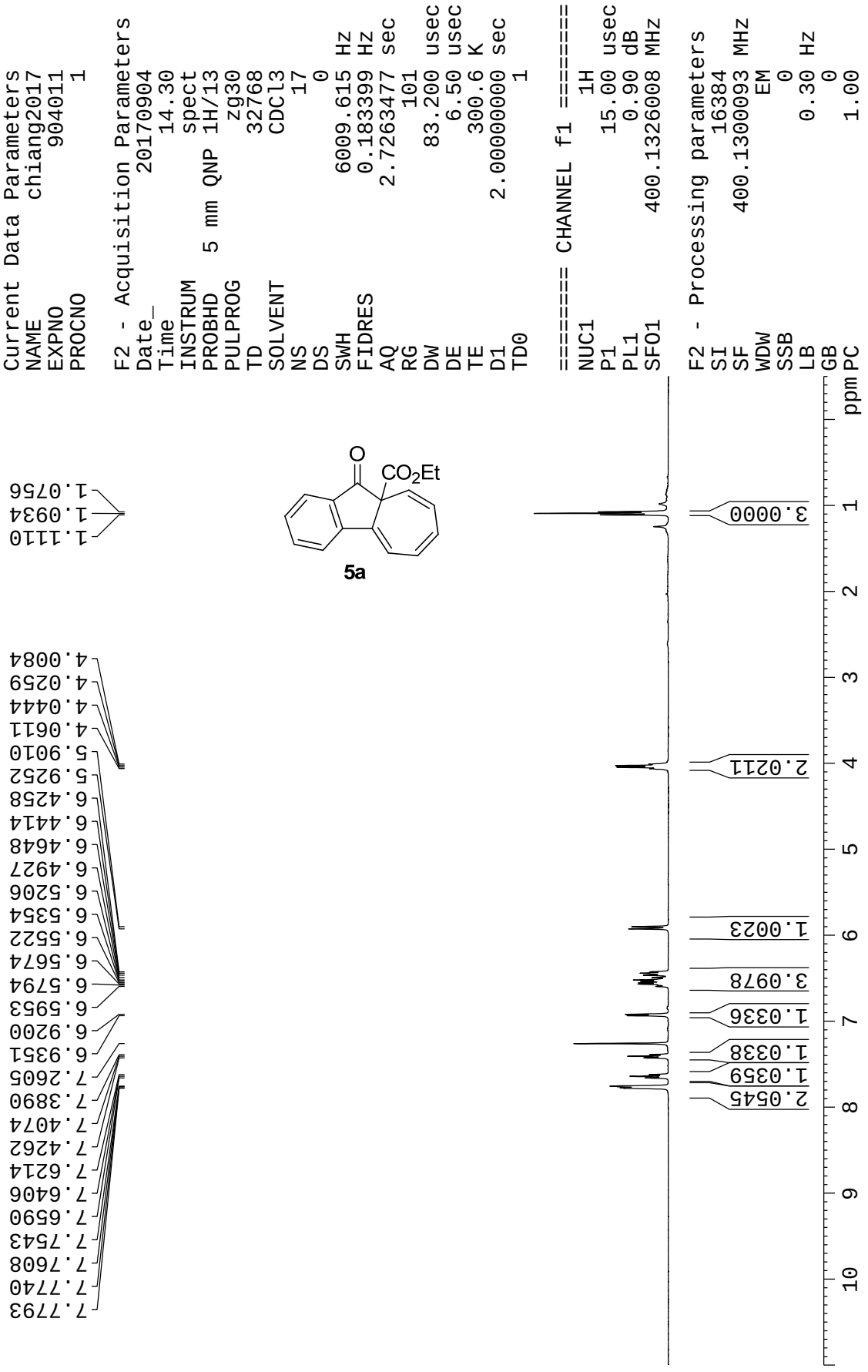
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300093 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

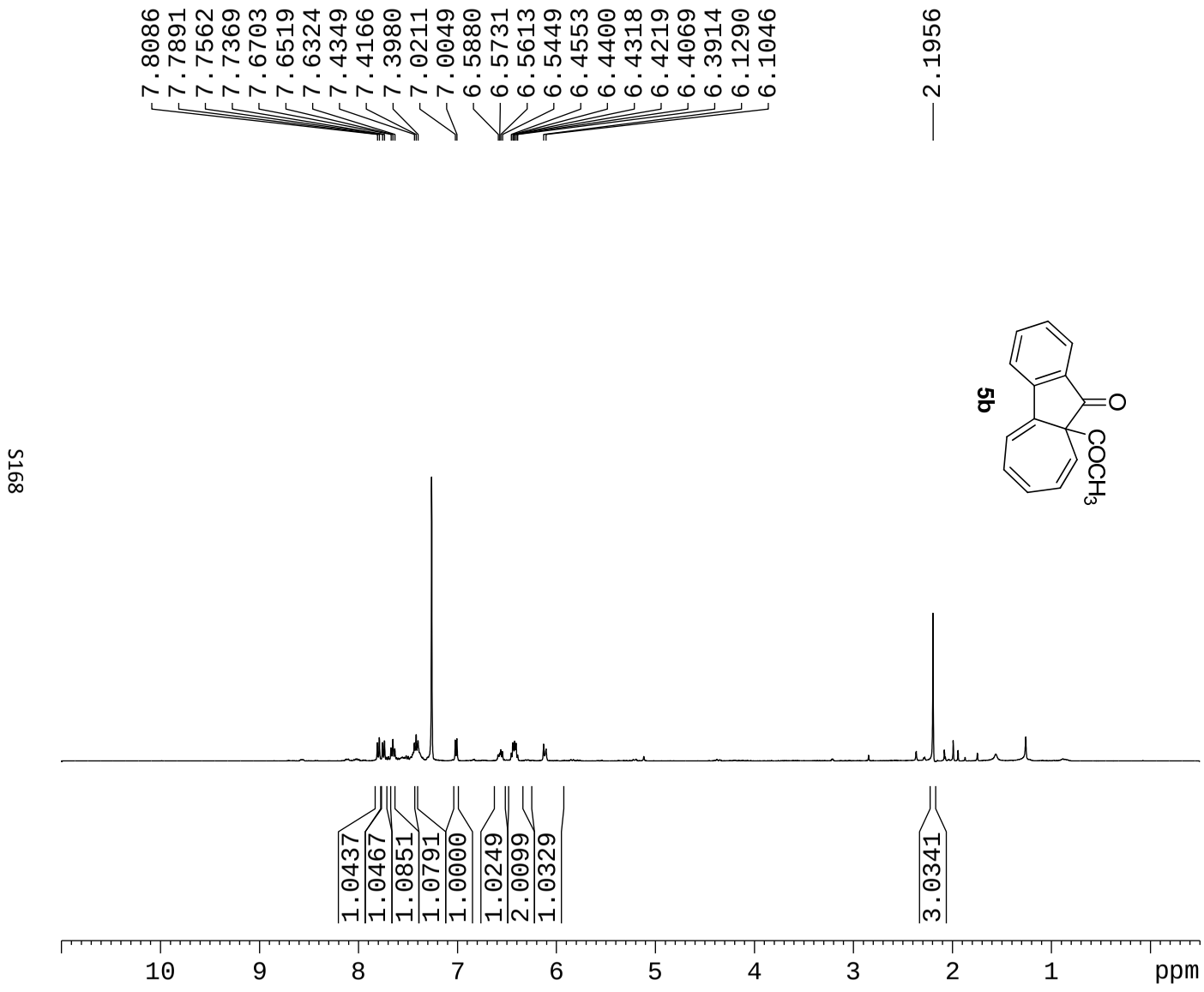



```
Current Data Parameters
NAME            chen2017
EXPNO             920022
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170920
Time               10.46
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   297
DS                     0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ             1.2976629 sec
RG                  2050
DW                19.800 usec
DE                 6.50 usec
TE                306.0 K
D1             0.20000000 sec
d11            0.03000000 sec
DELTA          0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

**5) NMR spectra of 5**



Current Data Parameters
NAME chiang2017
EXPNO 724031
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170724
Time 15.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 356
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 302.6 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.80 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
CO2Et
5a
13.9841
61.8607
64.2198
76.7440
77.0620
77.2626
77.3798
118.2703
122.0928
124.9830
125.9434
129.1878
129.2069
129.3220
131.6909
133.4588
134.0778
135.8778
149.0030
166.9954
198.4498
ppm




Current Data Parameters

| | |
|---|---|
| NAME | chiang2017 |
| EXPNO | 727011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

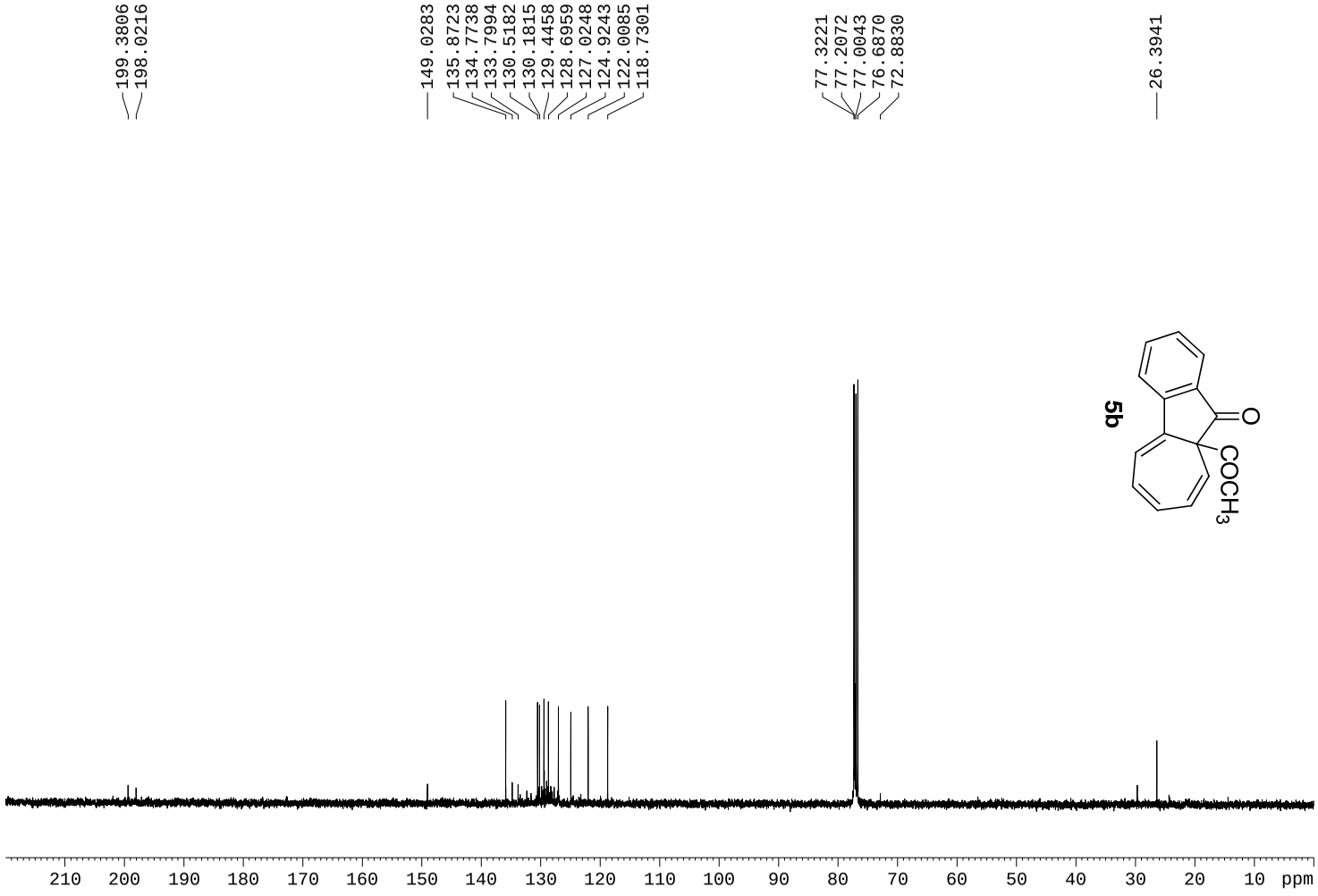
| | |
|---|---|
| Date_ | 20170727 |
| Time | 10.35 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 36 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 456 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 303.6 K |
| D1 | 0.20000000 sec |
| TD0 | 1 |

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

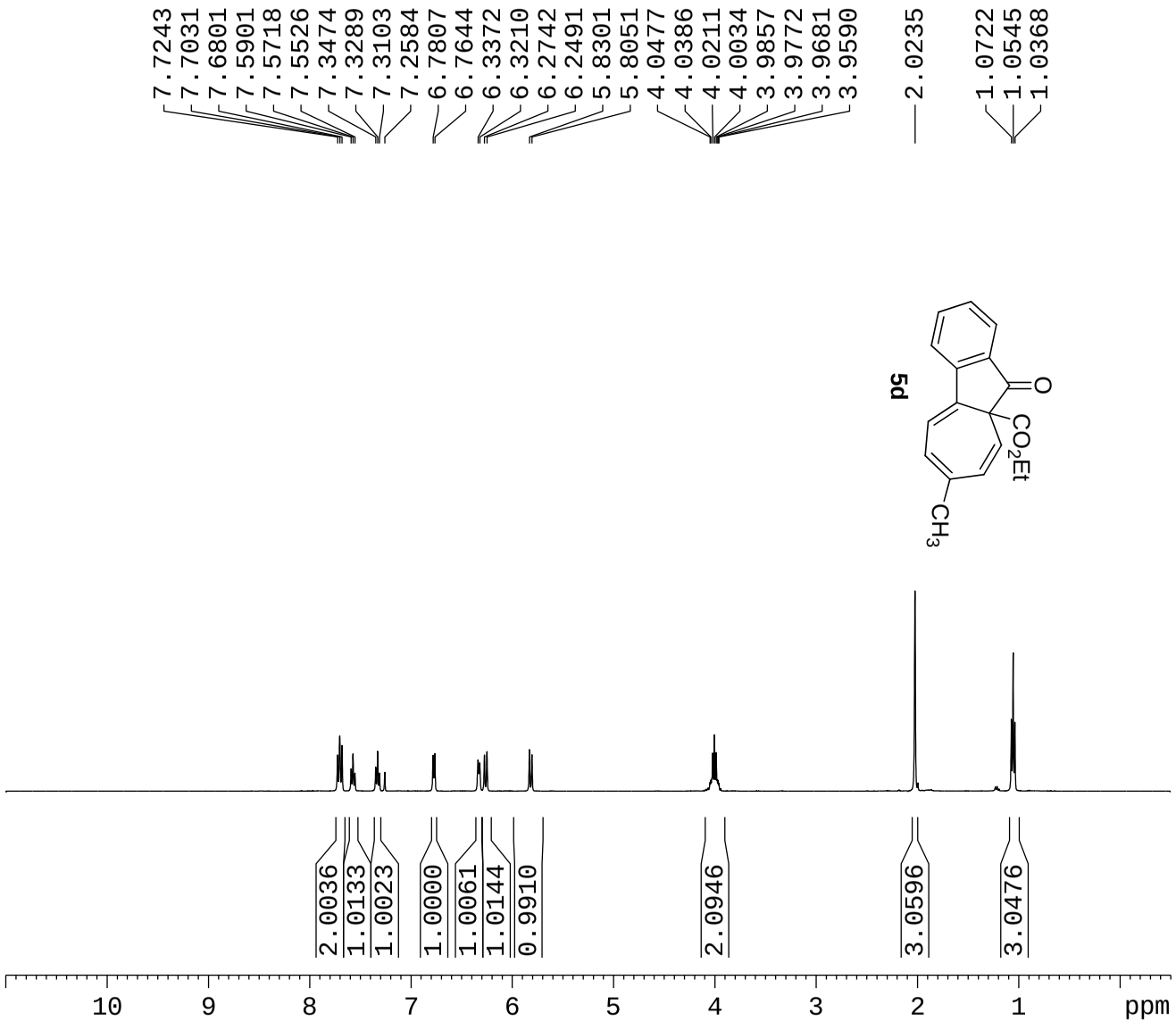
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

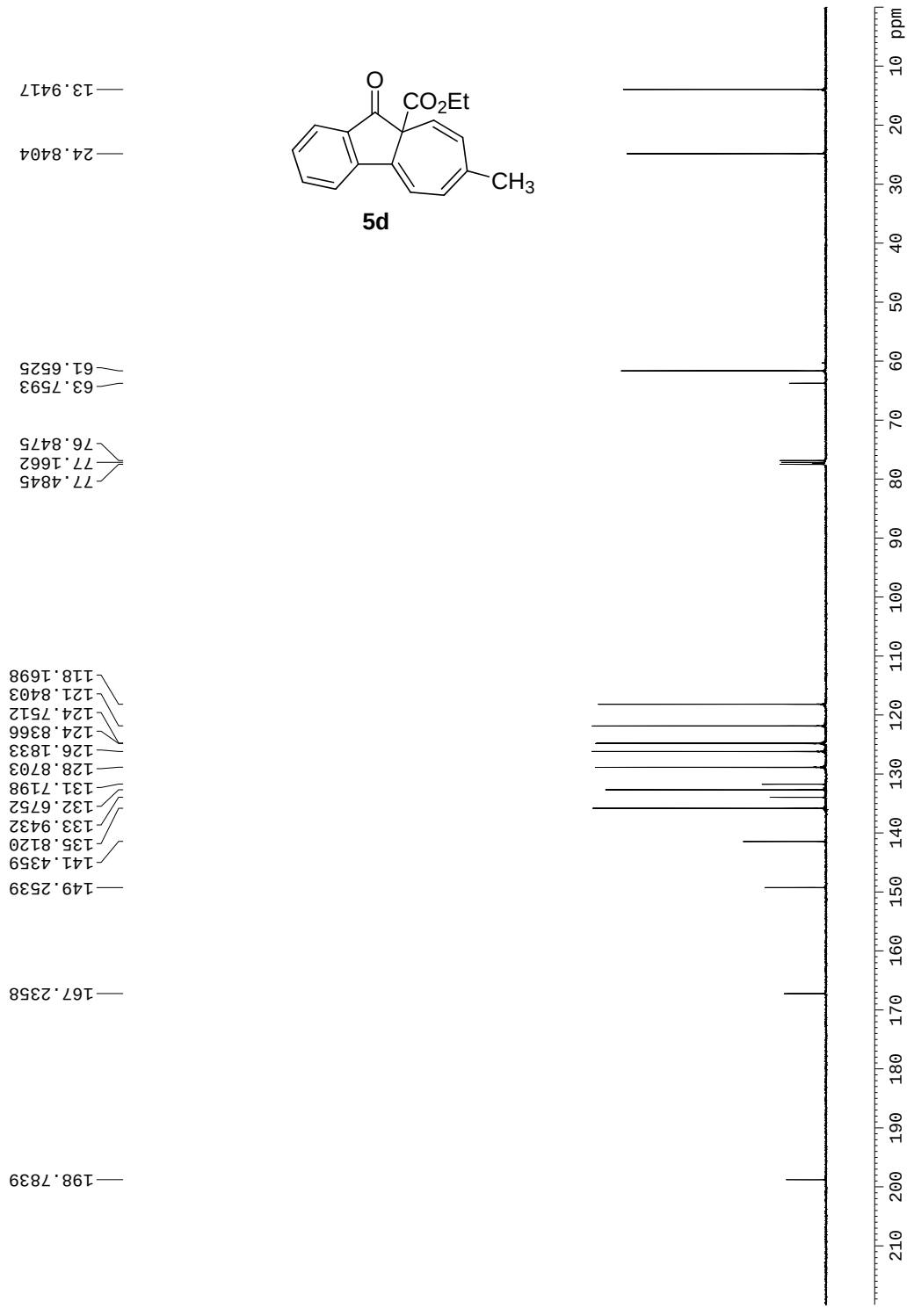
| | |
|---|---|
| SI | 16384 |
| SF | 400.1300089 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

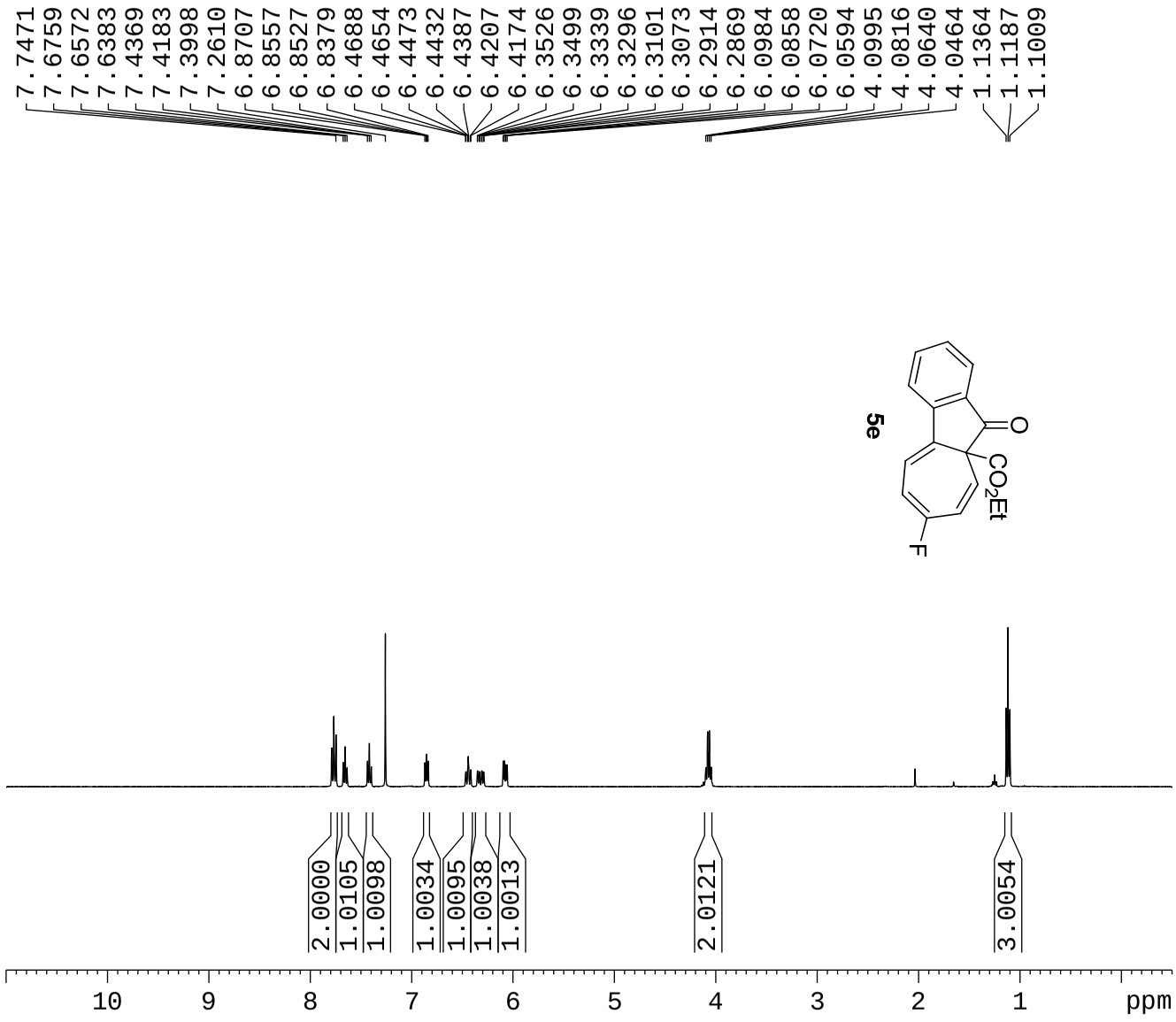



```
Current Data Parameters
NAME          chiang2017
EXPNO             727021
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170727
Time               10.44
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 25250
SOLVENT            CDCl3
NS                  2598
DS                     0
SWH            25252.525 Hz
FIDRES          1.000100 Hz
AQ             0.5000000 sec
RG                  2050
DW                19.800 usec
DE                 6.50 usec
TE                304.0 K
D1            0.20000000 sec
d11           0.03000000 sec
DELTA         0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                  6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -0.40 dB
PL12                15.80 dB
PL13                18.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
Current Data Parameters
NAME          chiang2017
EXPNO             930011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170930
Time                9.57
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                  50.8
DW                83.200 usec
DE                 6.50 usec
TE                304.3 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1         400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300094 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2017
EXPNO 930021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170930
Time 10.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 579
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 304.6 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.9417
24.8404
61.6525
63.7593
76.8475
77.1662
77.4845
118.1698
121.8403
124.7512
124.8366
126.1833
128.8703
131.7198
132.6752
133.9432
135.8120
141.4359
149.2539
167.2358
198.7839
O
CO2Et
CH3
5d
ppm




Current Data Parameters

| | |
|---|---|
| NAME | chiang2017 |
| EXPNO | 1115041 |
| PROCNO | 1 |

F2 - Acquisition Parameters

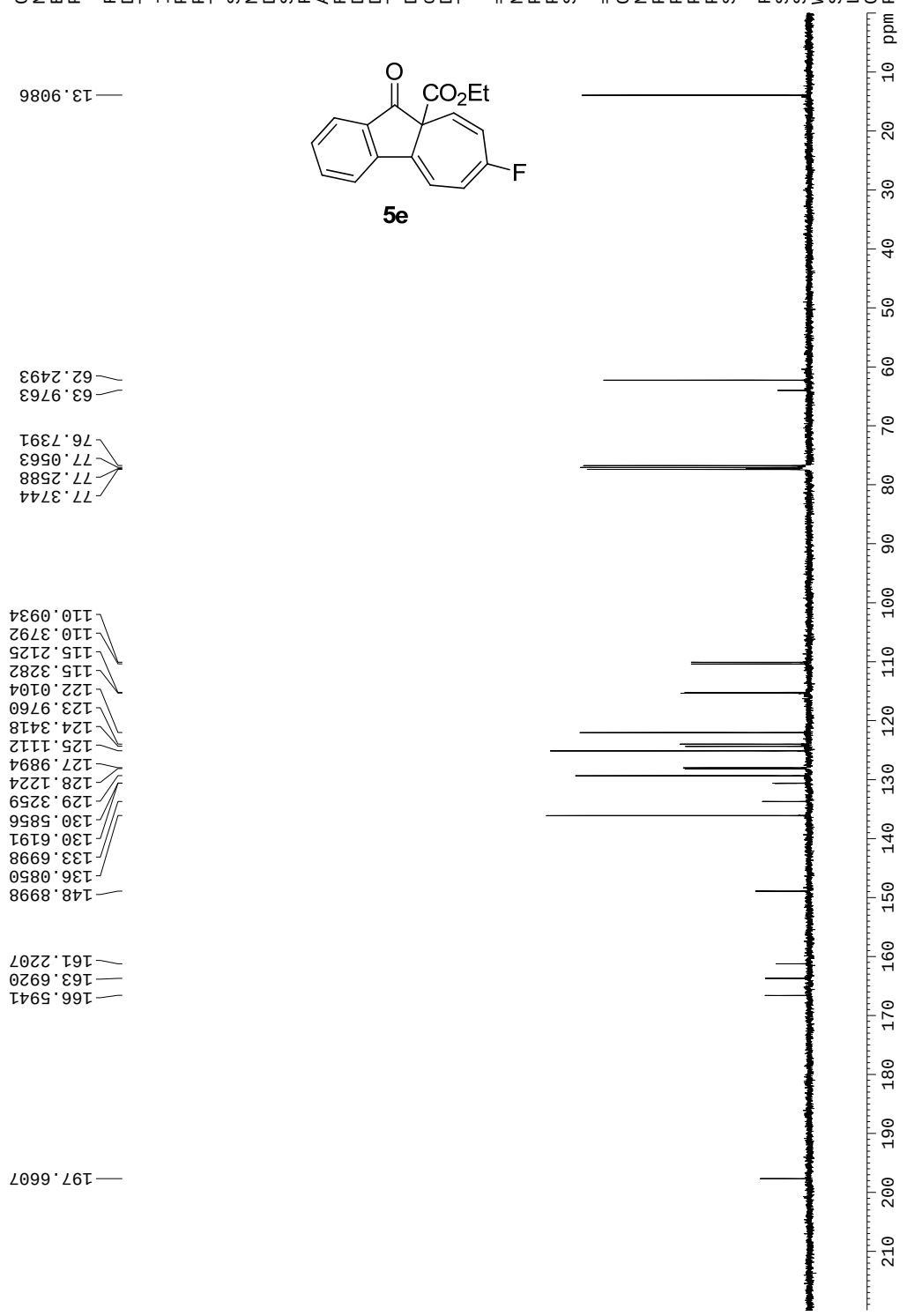
| | |
|---|---|
| Date_ | 20171115 |
| Time | 13.25 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 13 |
| DS | 0 |
| SWH | 8012.820 Hz |
| FIDRES | 0.244532 Hz |
| AQ | 2.0447731 sec |
| RG | 287 |
| DW | 62.400 usec |
| DE | 6.50 usec |
| TE | 296.9 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

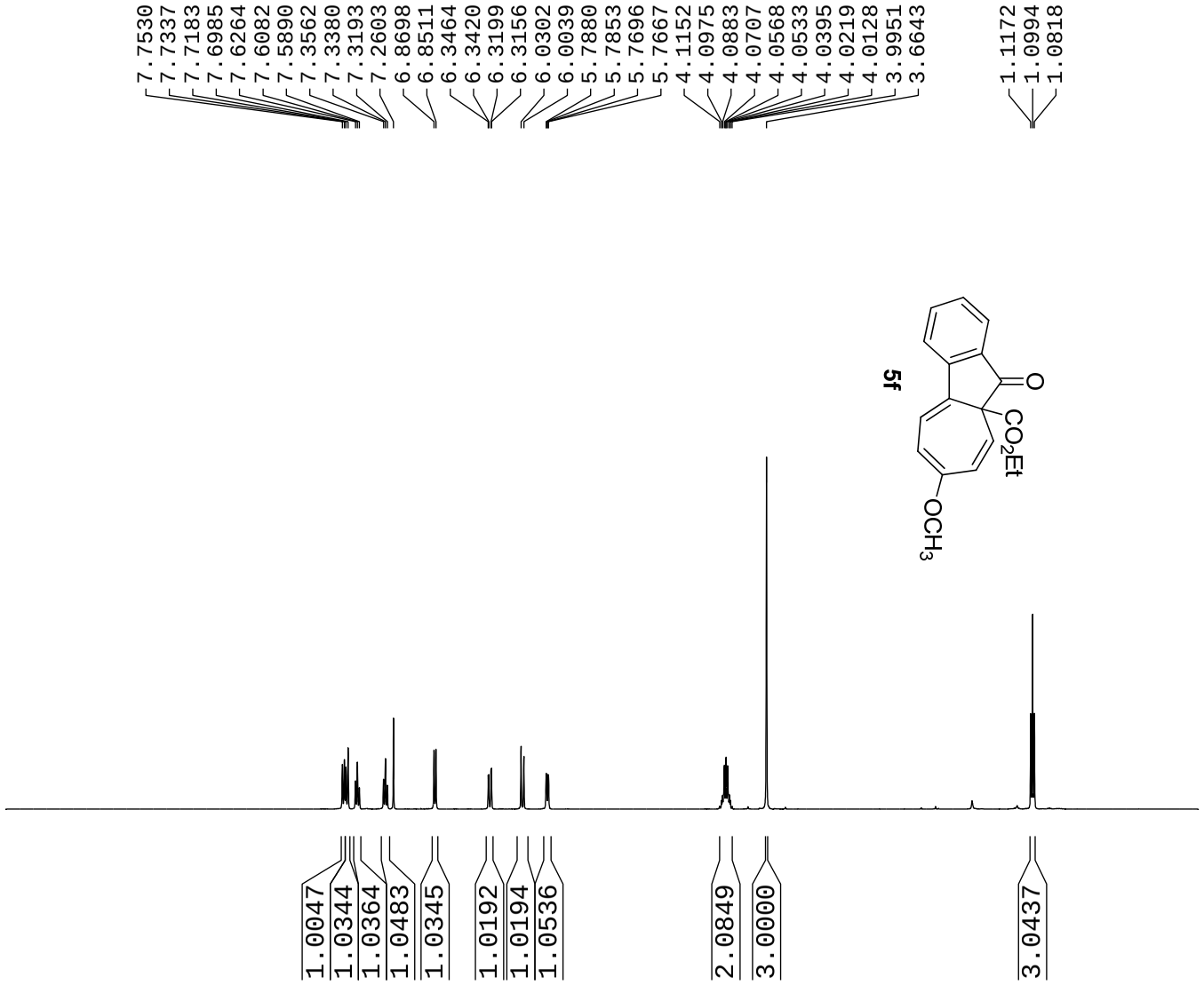
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300077 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |


Current Data Parameters
NAME chiang2017
EXPNO 1115051
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171115
Time 13.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 555
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.2 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.9086
62.2493
63.9763
76.7391
77.0563
77.2588
77.3744
110.0934
110.3792
115.2125
115.3282
122.0104
123.9760
124.3418
125.1112
127.9894
128.1224
129.3259
130.5856
130.6191
133.6998
136.0850
148.8998
161.2207
163.6920
166.5941
197.6607
O
CO2Et
F
5e
ppm

Current Data Parameters
NAME chiang2017
EXPNO 1115071
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171115
Time 13.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgfhigqn
TD 131072
SOLVENT CDCl3
NS 20
DS 0
SWH 89285.711 Hz
FIDRES 0.681196 Hz
AQ 0.7340532 sec
RG 2050
DW 5.600 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 19F
P1 20.00 usec
PL1 2.50 dB
SFO1 376.4607164 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 65536
SF 376.4983660 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
CO2Et
F
5e
-95.3219
-82
-84
-86
-88
-90
-92
-94
-96
-98
-100
-102
-104
-106
-108
ppm




S175

Current Data Parameters

| | |
|---|---|
| NAME | zhu2018 |
| EXPNO | 810011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20180810 |
| Time | 16.09 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 29 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 101 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 296.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |

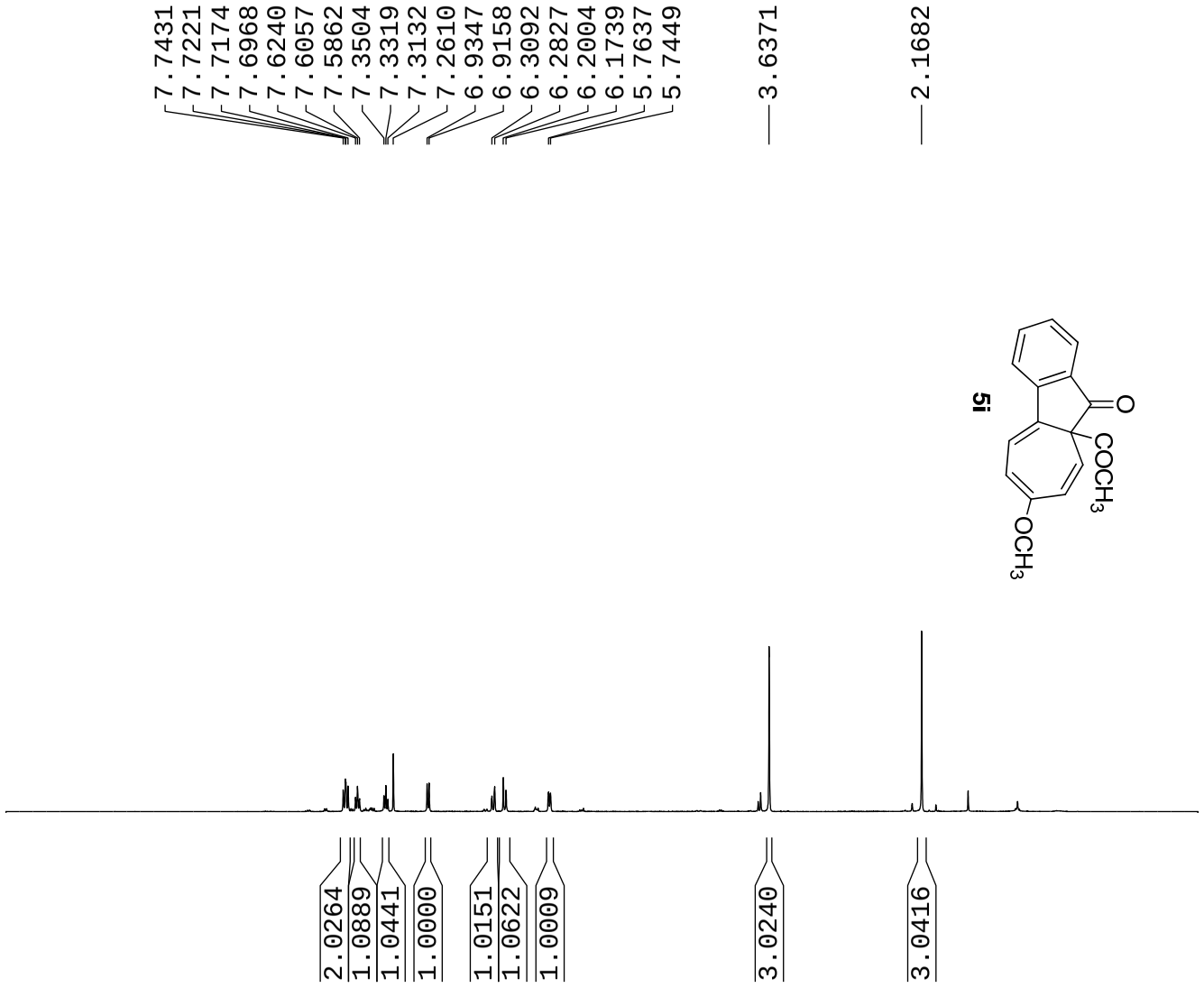
======== CHANNEL f1 ========

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300089 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

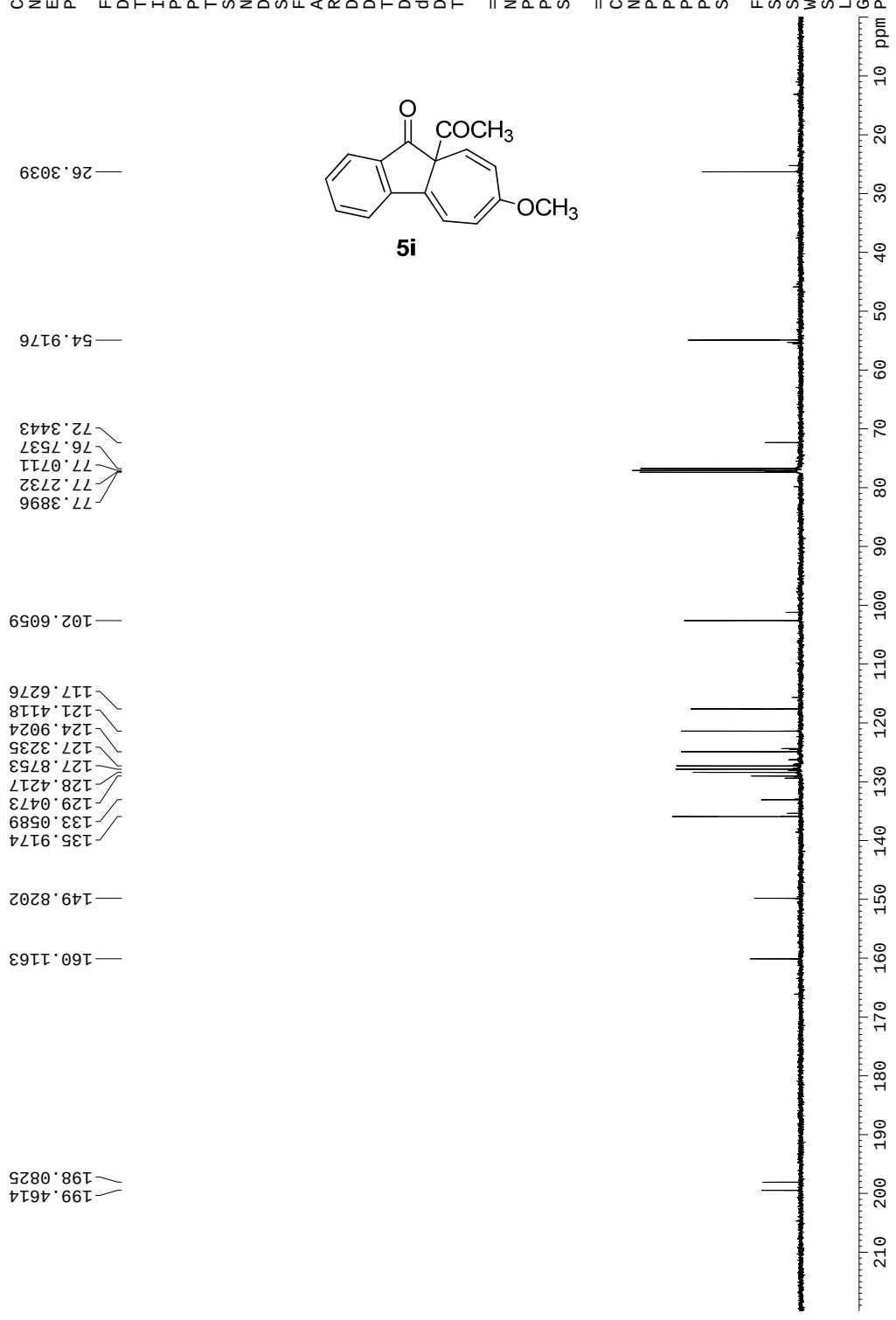
Current Data Parameters
NAME zhu2018
EXPNO 810012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180810
Time 16.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 338
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 1440
DW 19.800 usec
DE 6.50 usec
TE 296.2 K
D1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000001 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
CO2Et
OCH3
5f
13.8837
54.9368
61.8449
63.5991
76.6896
77.0071
77.2093
77.3251
102.1106
117.2251
121.4125
124.9133
126.9289
127.6421
128.1615
128.2404
133.3622
135.8221
149.7214
160.8833
167.1113
198.5584
ppm

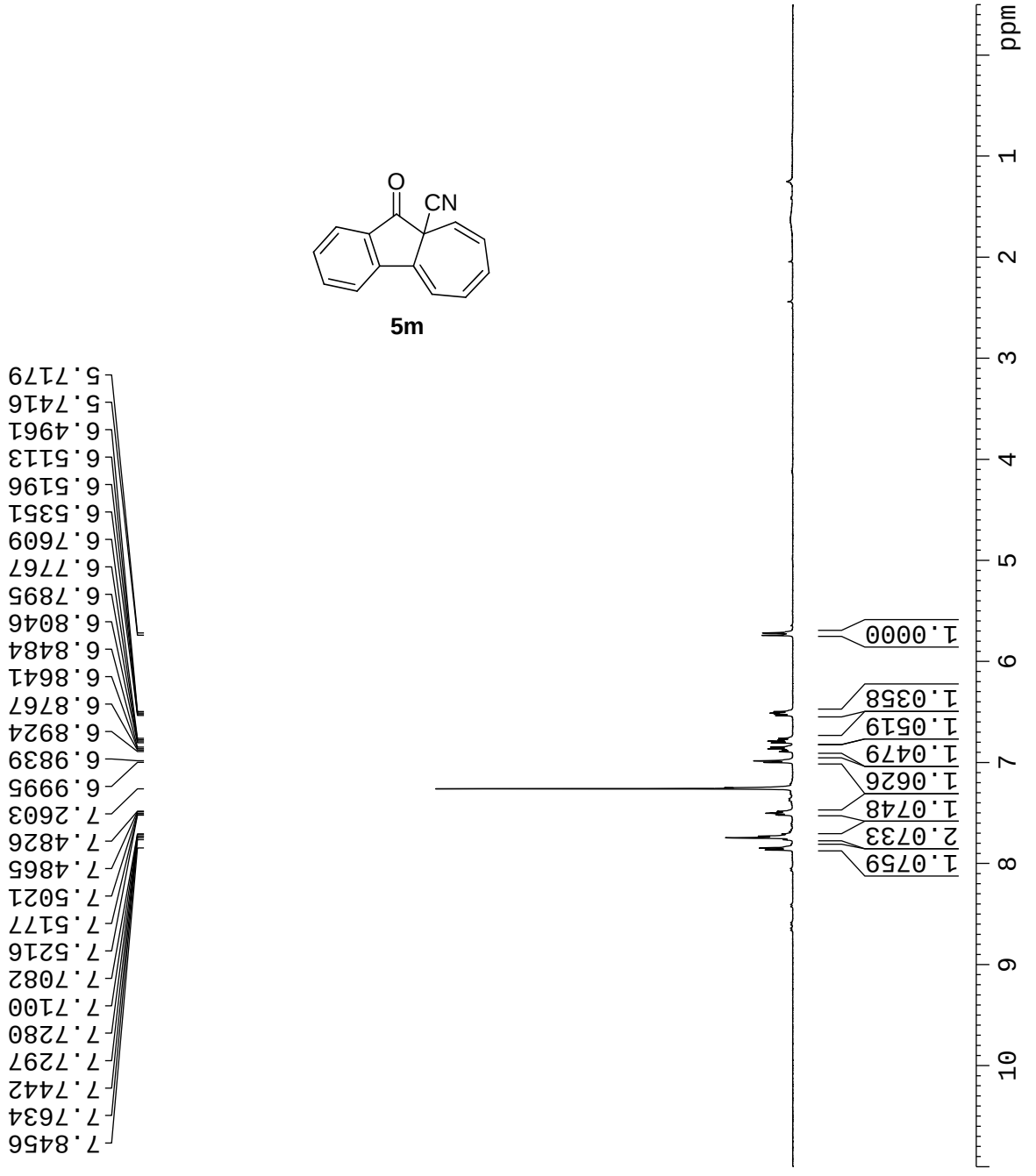


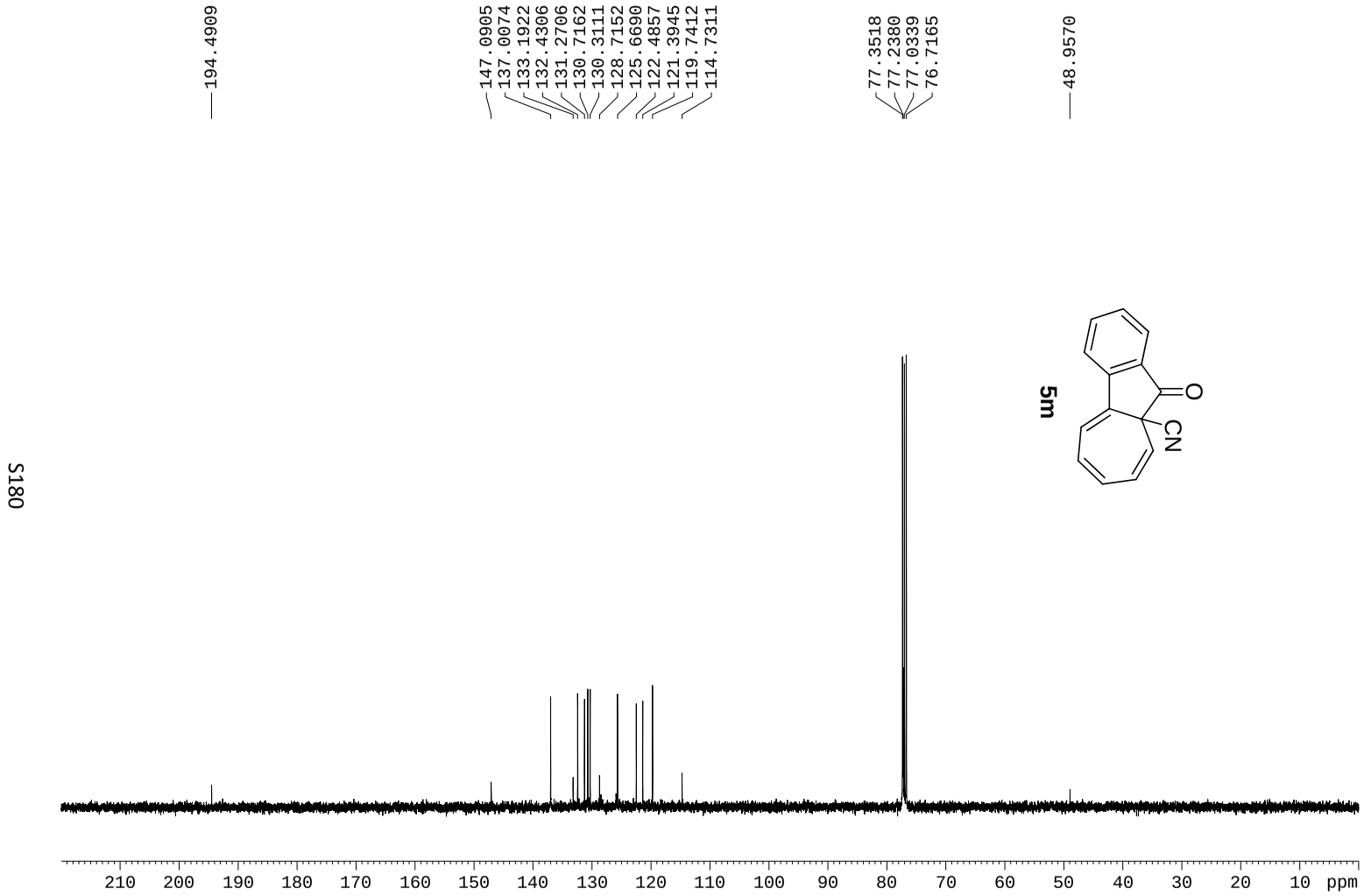

**5i**




```
Current Data Parameters
NAME          chiang2017
EXPNO            1220011
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171220
Time               10.01
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG                   256
DW                83.200 usec
DE                 6.50 usec
TE                296.9 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                0.90 dB
SFO1        400.1326008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300078 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


Current Data Parameters
NAME chiang2017
EXPNO 1220021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171220
Time_ 10.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 297.1 K
D1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000001 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
COCH3
OCH3
5i
26.3039
54.9176
72.3443
76.7537
77.0711
77.2732
77.3896
102.6059
117.6276
121.4118
124.9024
127.3235
127.8753
128.4217
129.0473
133.0589
135.9174
149.8202
160.1163
198.0825
199.4614
ppm


Current Data Parameters
NAME chen2017
EXPNO 1110011
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171110
Time 18.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 18
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 575
DW 62.400 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
CN
5m
7.8456
7.7634
7.7442
7.7297
7.7280
7.7100
7.7082
7.5216
7.5177
7.5021
7.4865
7.4826
7.2603
6.9995
6.9839
6.8924
6.8767
6.8641
6.8484
6.8046
6.7895
6.7767
6.7609
6.5351
6.5196
6.5113
6.4961
5.7416
5.7179
1.0759
2.0733
1.0748
1.0626
1.0479
1.0519
1.0358
1.0000
ppm




```
Current Data Parameters
NAME            chen2017
EXPNO            1110012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171110
Time               18.27
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   600
DS                     0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ             1.2976629 sec
RG                  2050
DW                19.800 usec
DE                 6.50 usec
TE                296.1 K
D1            0.20000000 sec
d11           0.03000000 sec
DELTA         0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                 6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


S181

Current Data Parameters

| | |
|---|---|
| NAME | chen2017 |
| EXPNO | 1126011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

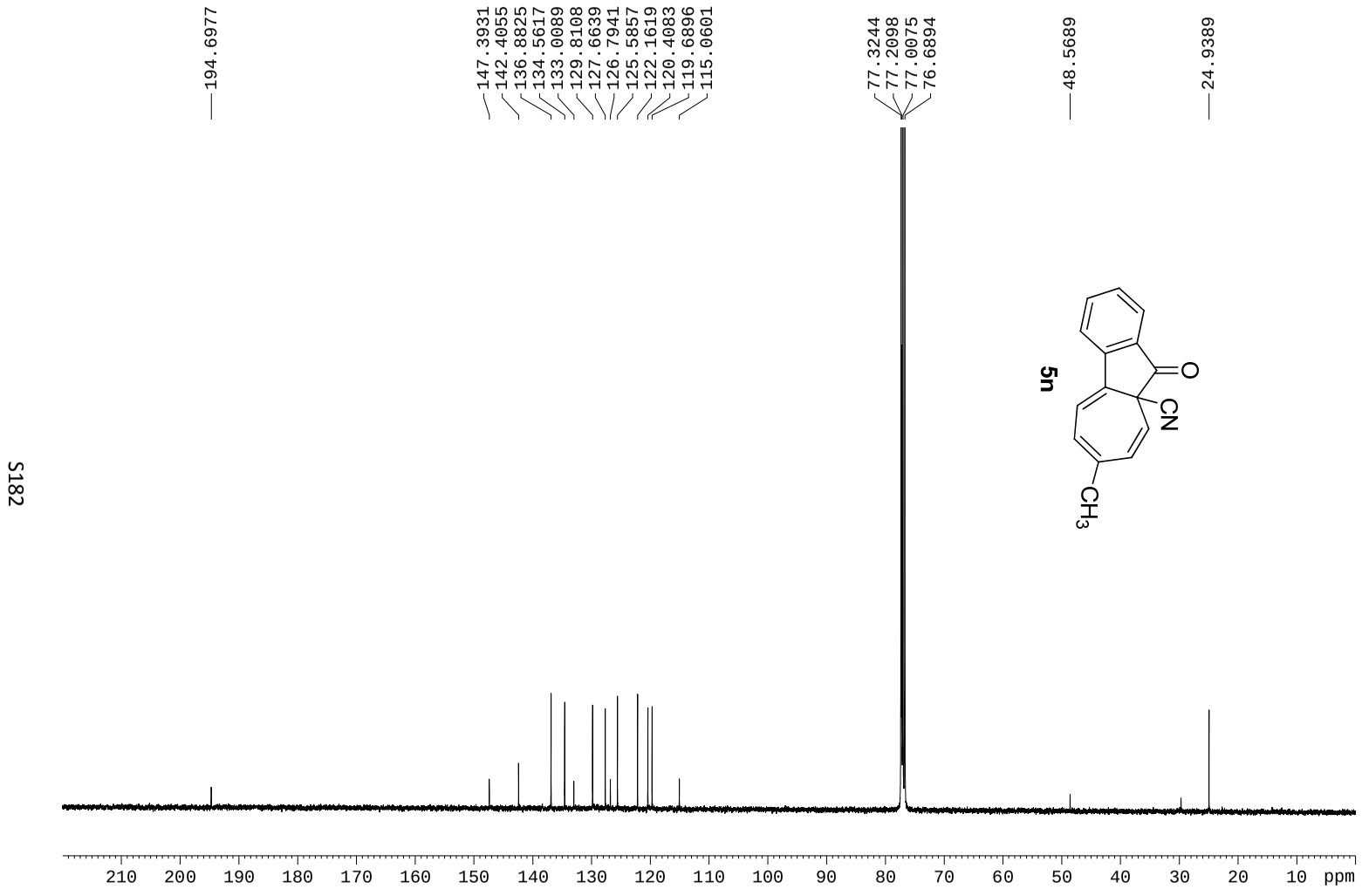
| | |
|---|---|
| Date_ | 20171126 |
| Time | 22.40 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 63 |
| DS | 0 |
| SWH | 8012.820 Hz |
| FIDRES | 0.244532 Hz |
| AQ | 2.0447731 sec |
| RG | 1290 |
| DW | 62.400 usec |
| DE | 6.50 usec |
| TE | 300.1 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

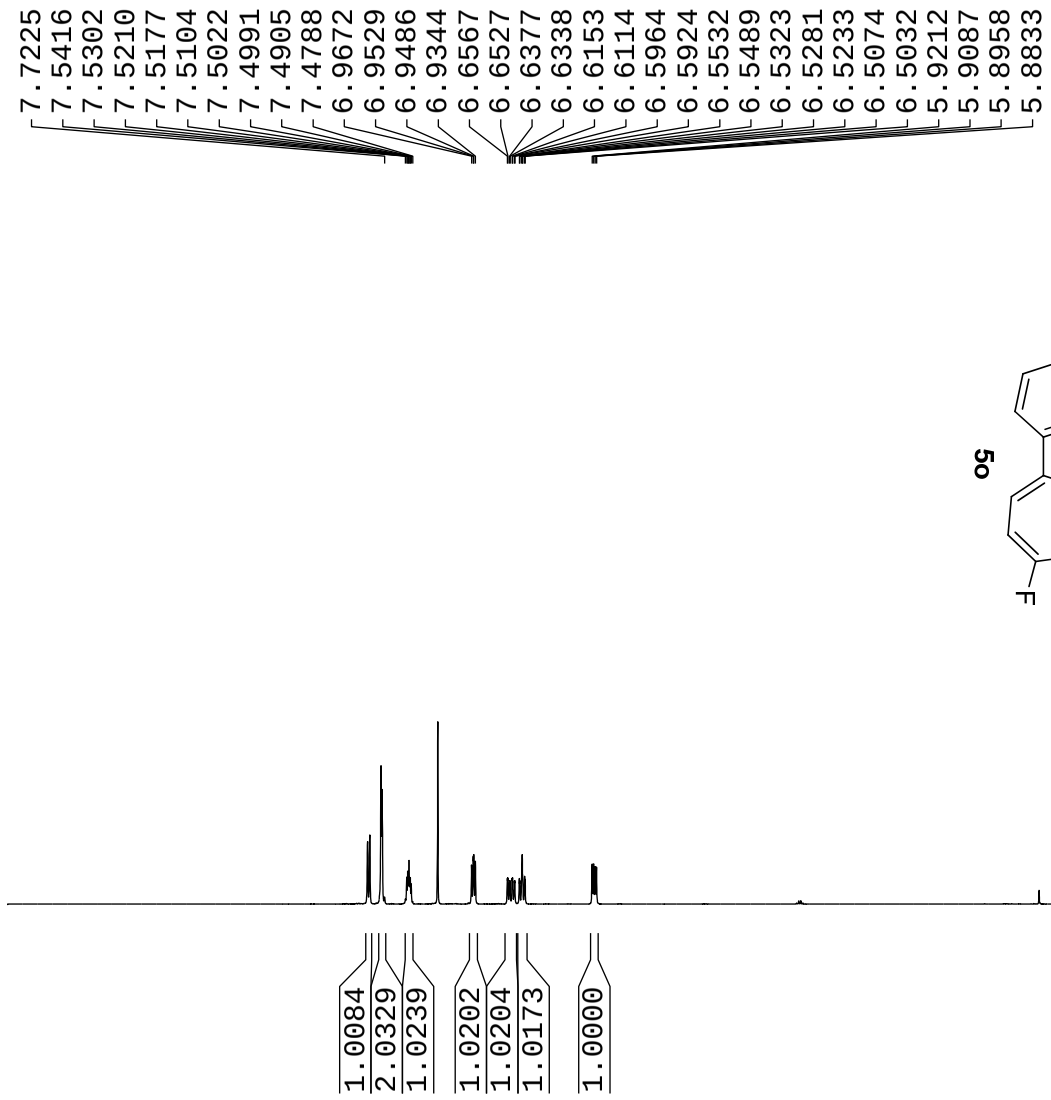
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300081 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




```
Current Data Parameters
NAME            chen2017
EXPNO            1126013
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171126
Time               22.55
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                24005
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                300.3 K
D1           0.10000000 sec
d11          0.03000000 sec
DELTA        0.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
PL13               18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```




S183

Current Data Parameters

| | |
|---|---|
| NAME | chen2017 |
| EXPNO | 1216011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

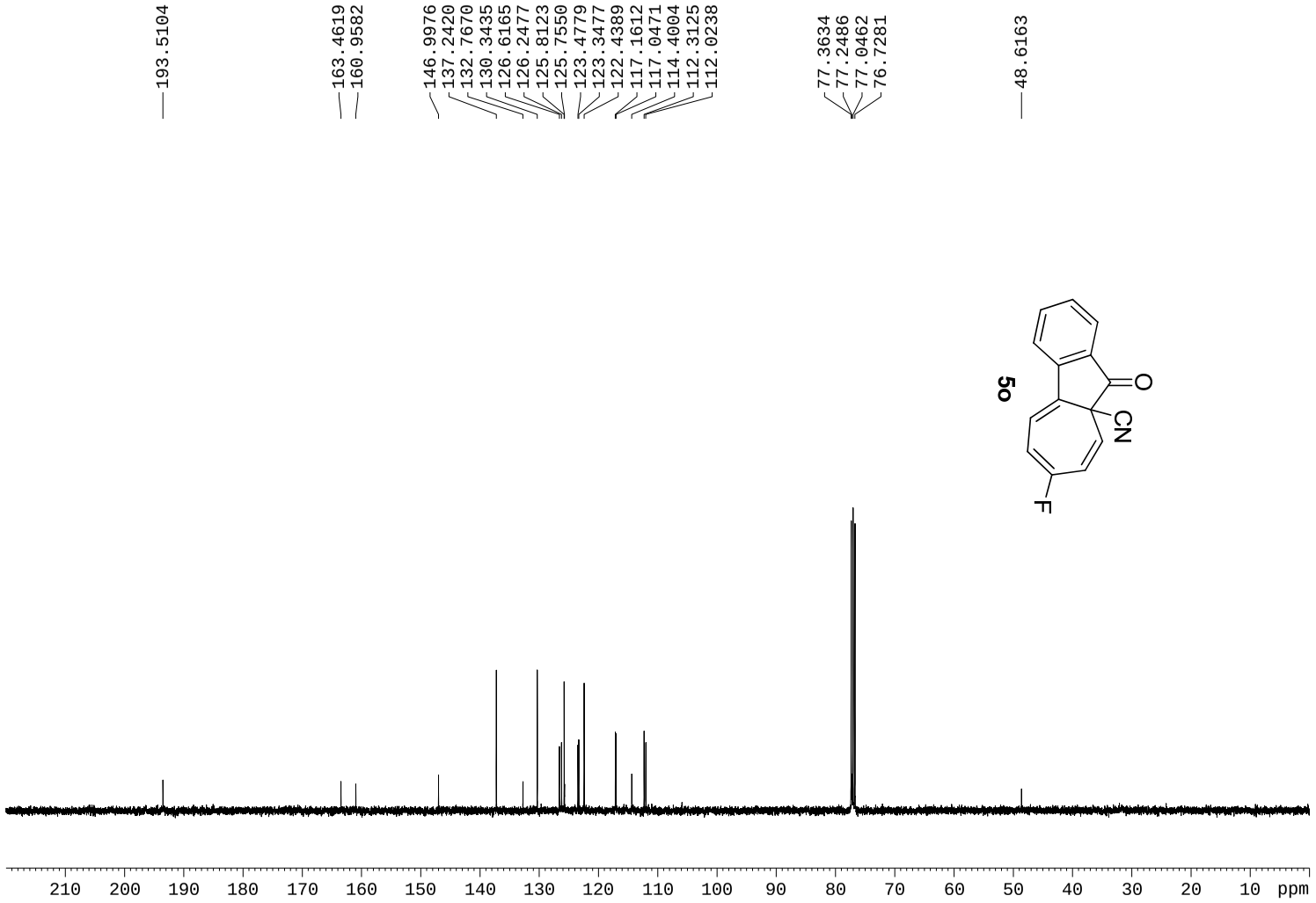
| | |
|---|---|
| Date_ | 20171216 |
| Time | 15.45 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 19 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 512 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 297.7 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

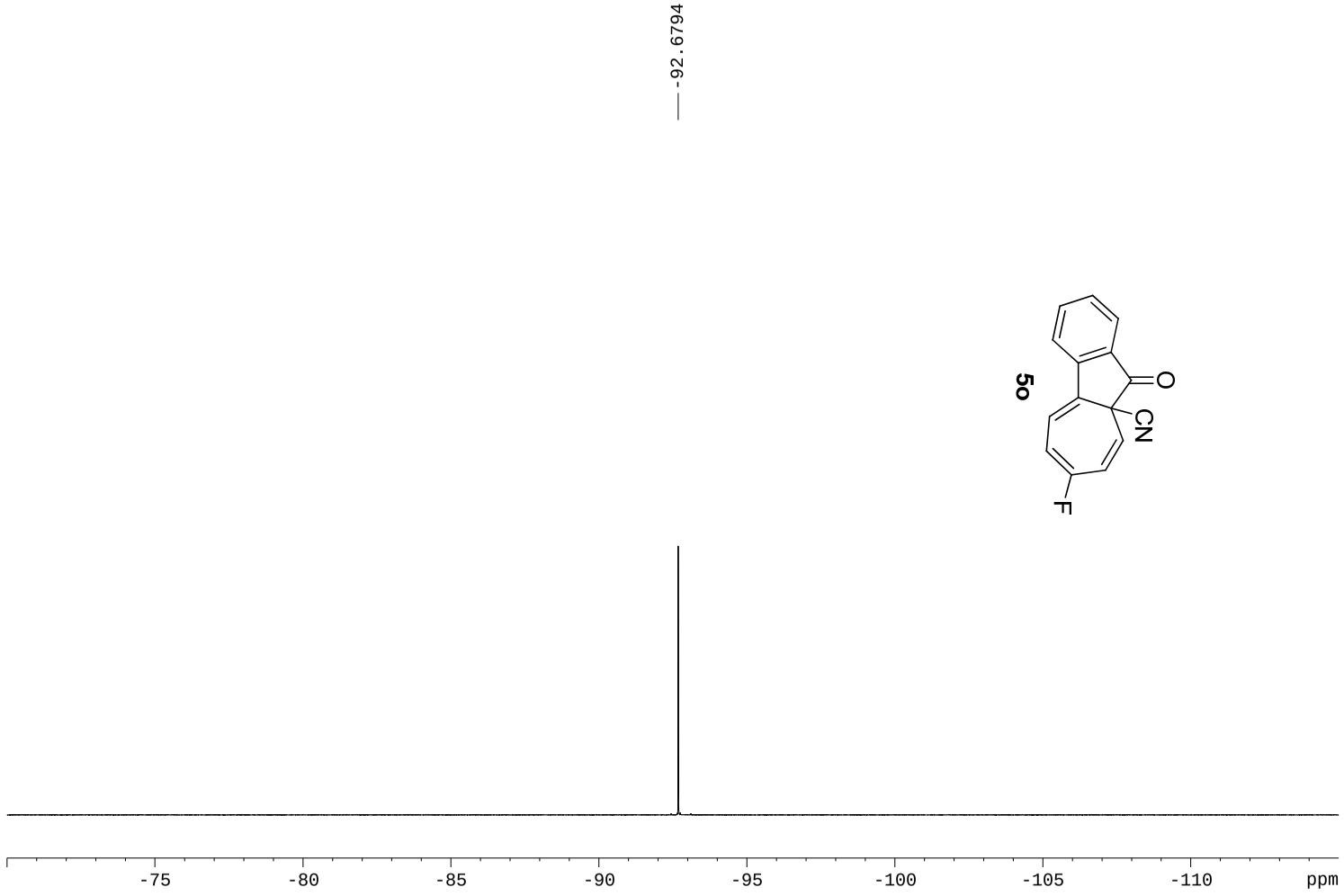
| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300079 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




```
Current Data Parameters
NAME            chen2017
EXPNO            1216013
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171216
Time               15.59
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  293
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                298.0 K
D1           0.20000000 sec
d11          0.03000000 sec
DELTA        0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

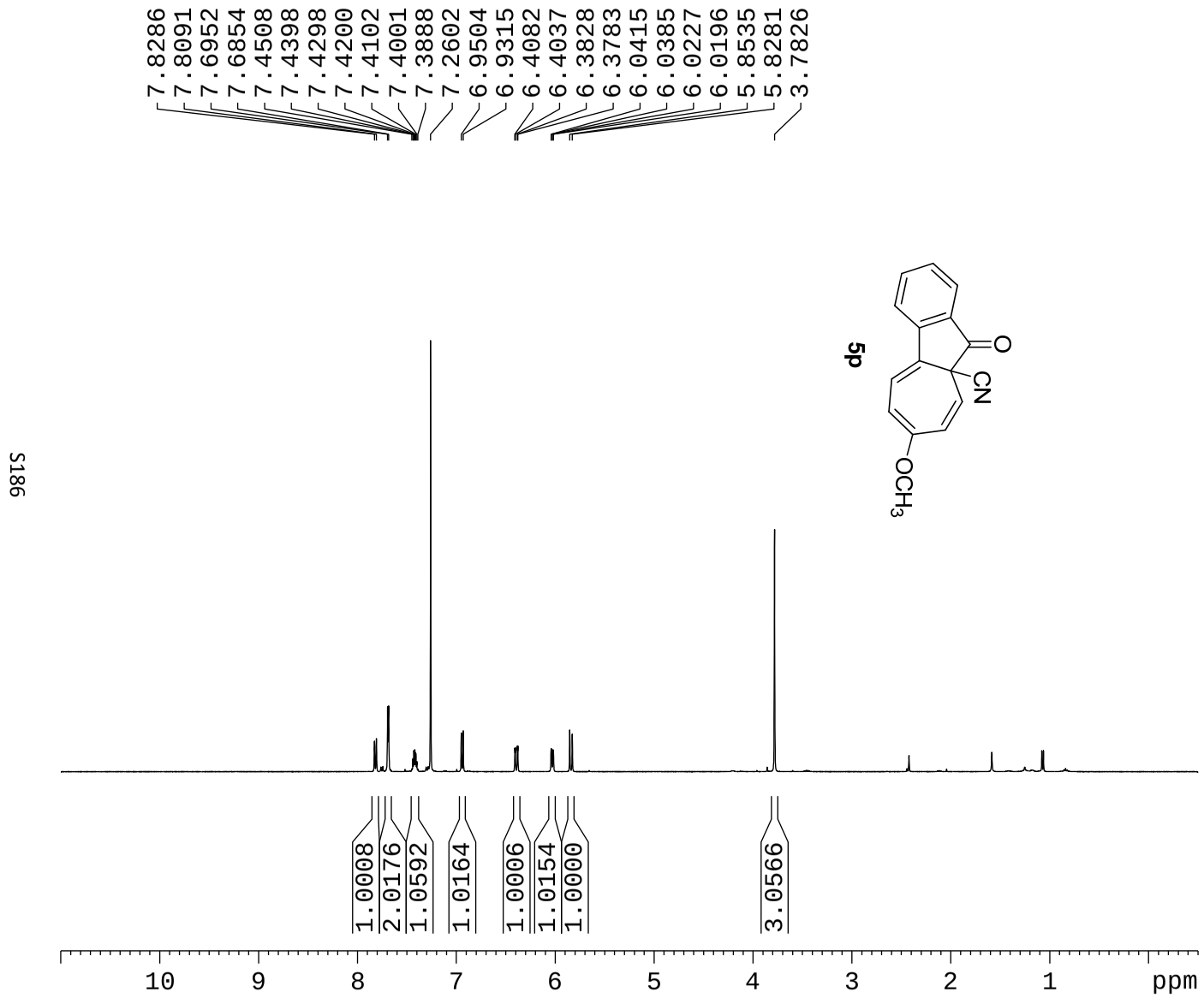






**5o**

```
Current Data Parameters
NAME            chen2017
EXPNO            1216014
PROCNO                 1

F2 - Acquisition Parameters
Date_           20171216
Time               16.08
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG         zgfhigqn
TD               131072
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340532 sec
RG                  2050
DW                 5.600 usec
DE                 6.50 usec
TE                297.9 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  19F
P1                 20.00 usec
PL1                 2.50 dB
SFO1         376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -0.40 dB
PL12               15.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF           376.4983660 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 109011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

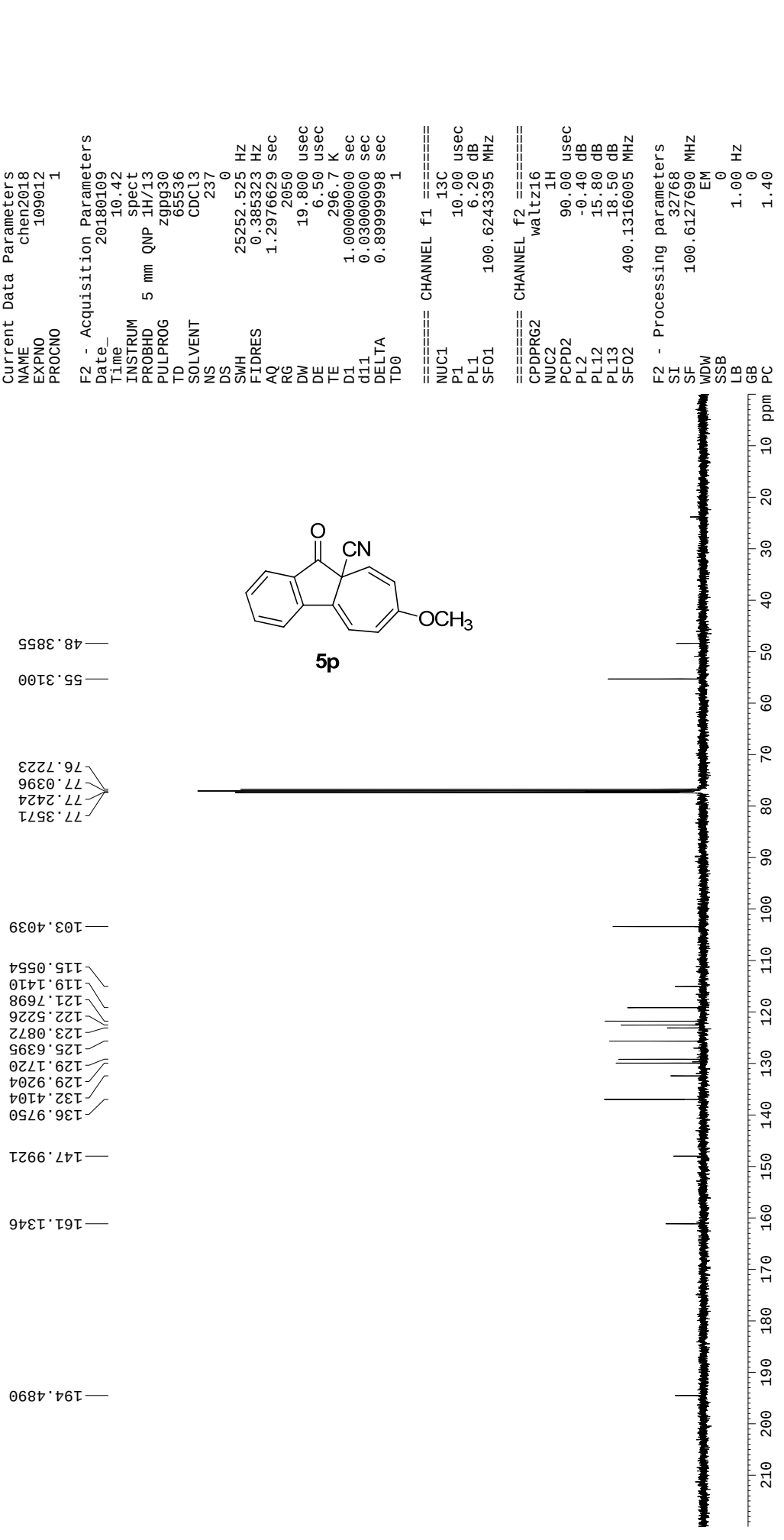
| | |
|---|---|
| Date_ | 20180109 |
| Time | 10.34 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 33 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 512 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 296.6 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

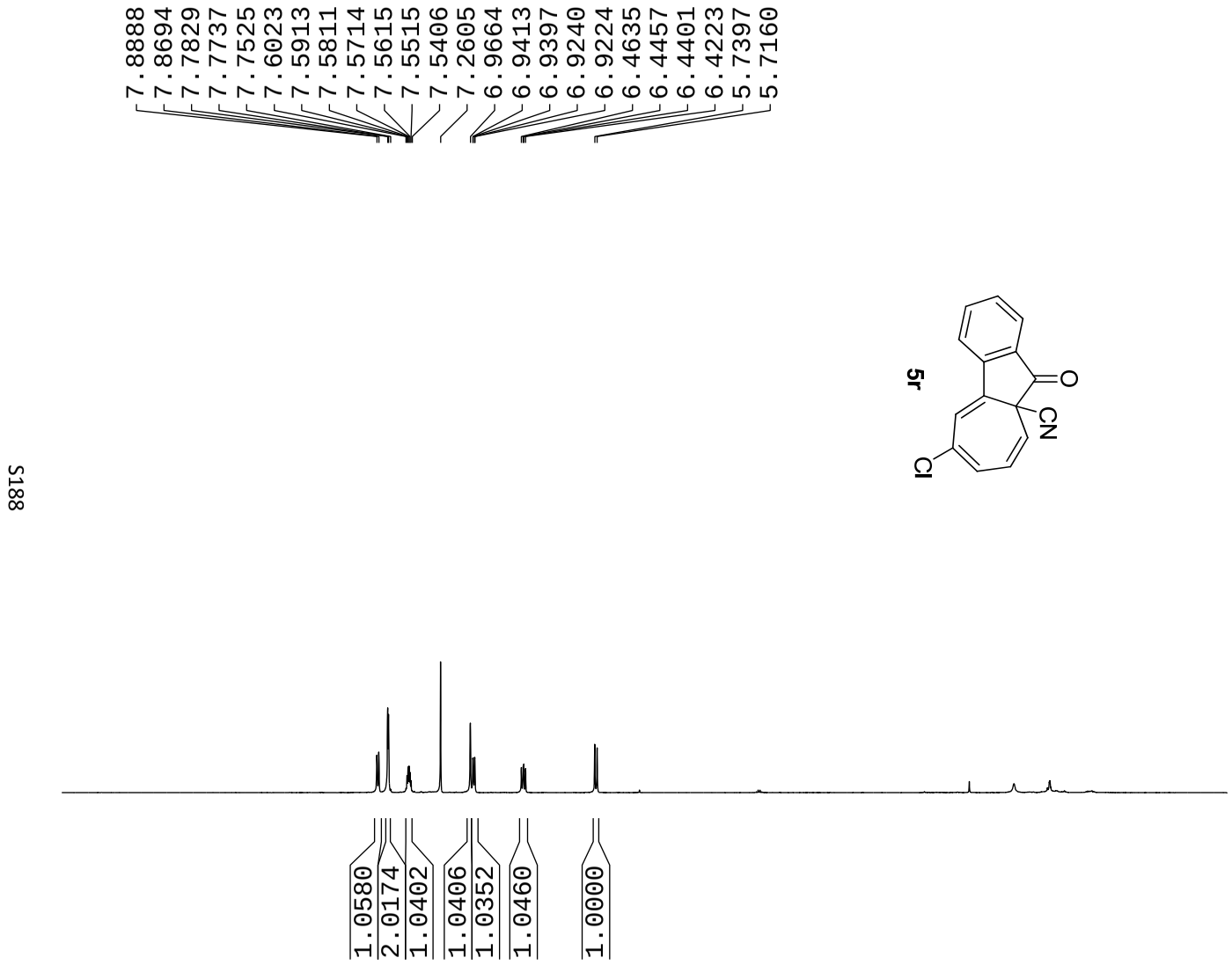
======== CHANNEL f1 ========

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300082 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |


Current Data Parameters
NAME chen2018
EXPNO 109012
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180109
Time_ 10.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 237
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
CN
OCH3
5p
48.3855
55.3100
76.7223
77.0396
77.2424
77.3571
103.4039
115.0554
119.1410
121.7698
122.5226
123.0872
125.6395
129.1720
129.9204
132.4104
136.9750
147.9921
161.1346
194.4890
ppm




Current Data Parameters

| | |
|---|---|
| NAME | chen2018 |
| EXPNO | 312011 |
| PROCNO | 1 |

F2 - Acquisition Parameters

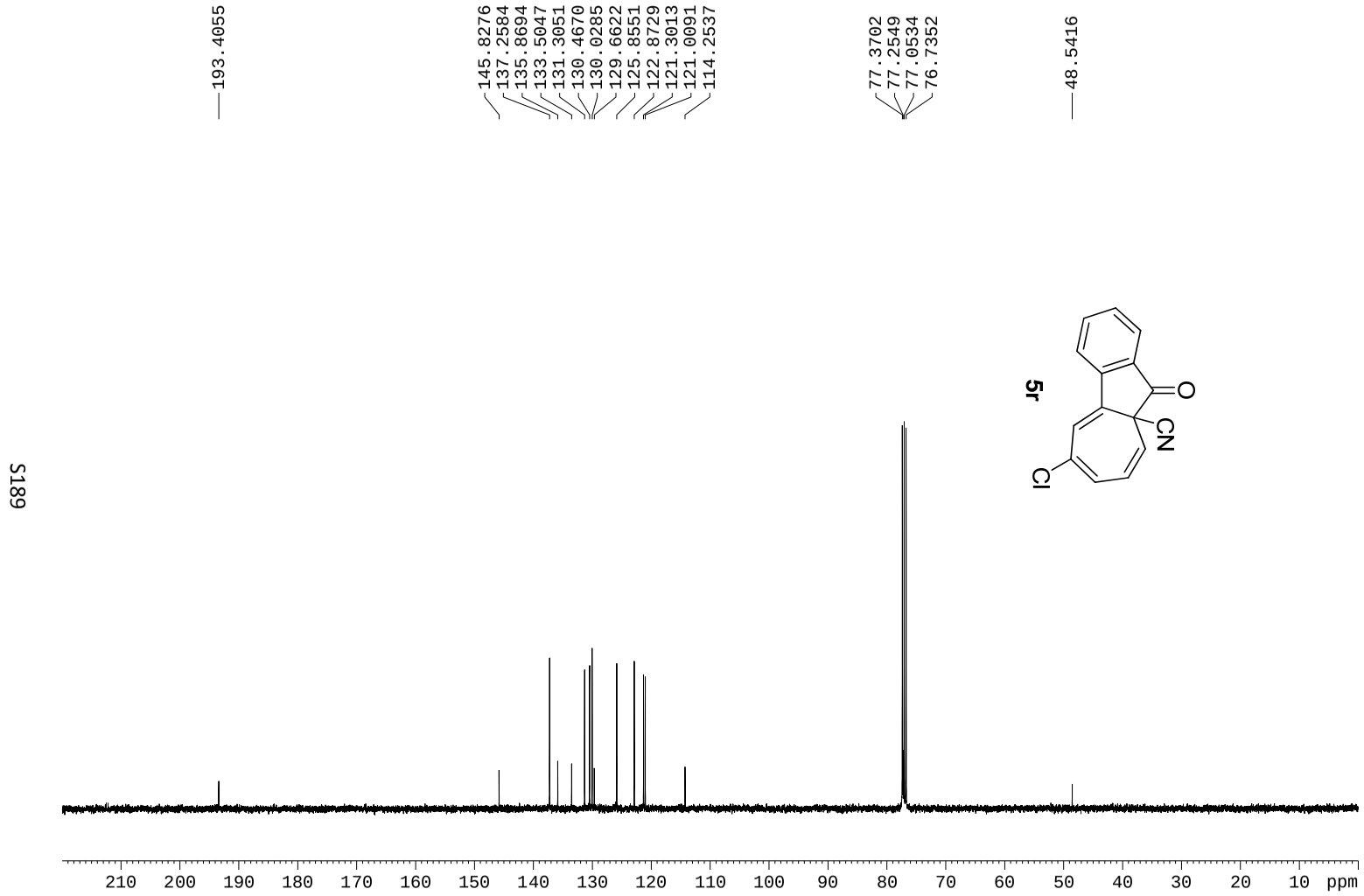
| | |
|---|---|
| Date_ | 20180312 |
| Time | 13.17 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 41 |
| DS | 0 |
| SWH | 8012.820 Hz |
| FIDRES | 0.244532 Hz |
| AQ | 2.0447731 sec |
| RG | 512 |
| DW | 62.400 usec |
| DE | 6.50 usec |
| TE | 293.8 K |
| D1 | 2.00000000 sec |
| TD0 | 1 |

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

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

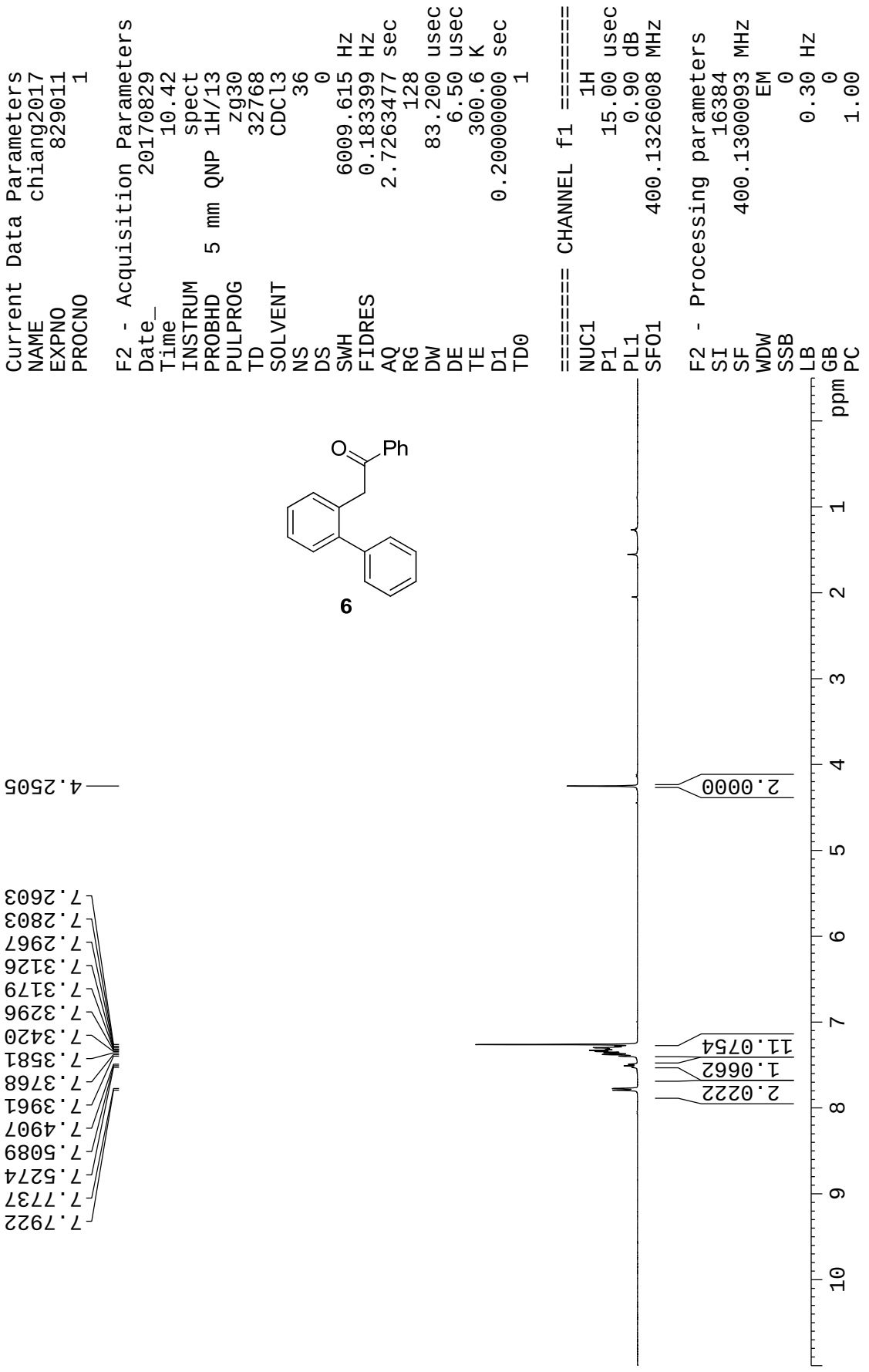
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300091 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

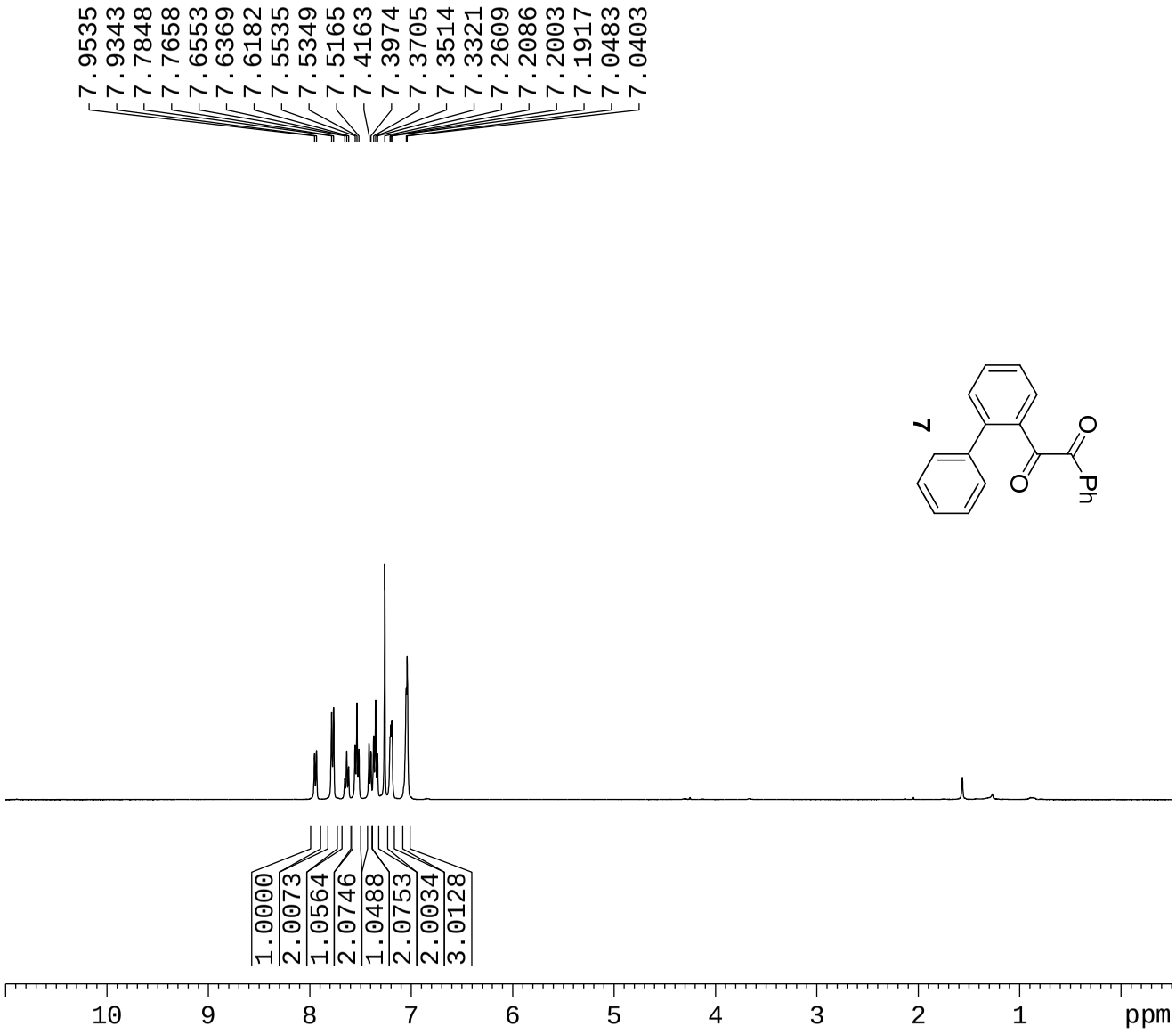



```
Current Data Parameters
NAME            chen2018
EXPNO             312012
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180312
Time               13.25
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  695
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ            1.2976629 sec
RG                 2050
DW               19.800 usec
DE                6.50 usec
TE                294.1 K
D1           0.50000000 sec
d11          0.03000000 sec
DELTA        0.40000001 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

**6) NMR spectra of 6, 7 and 8**



Current Data Parameters
NAME chiang2017
EXPNO 829021
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170829
Time 10.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 1680
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.50000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 301.0 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
Ph
6
43.2538
76.7018
77.0190
77.2216
77.3369
127.0118
127.1557
127.5669
128.2584
128.3052
128.4782
129.1518
130.1687
130.6543
132.3872
132.9681
136.7088
141.3433
142.3838
198.2755
ppm

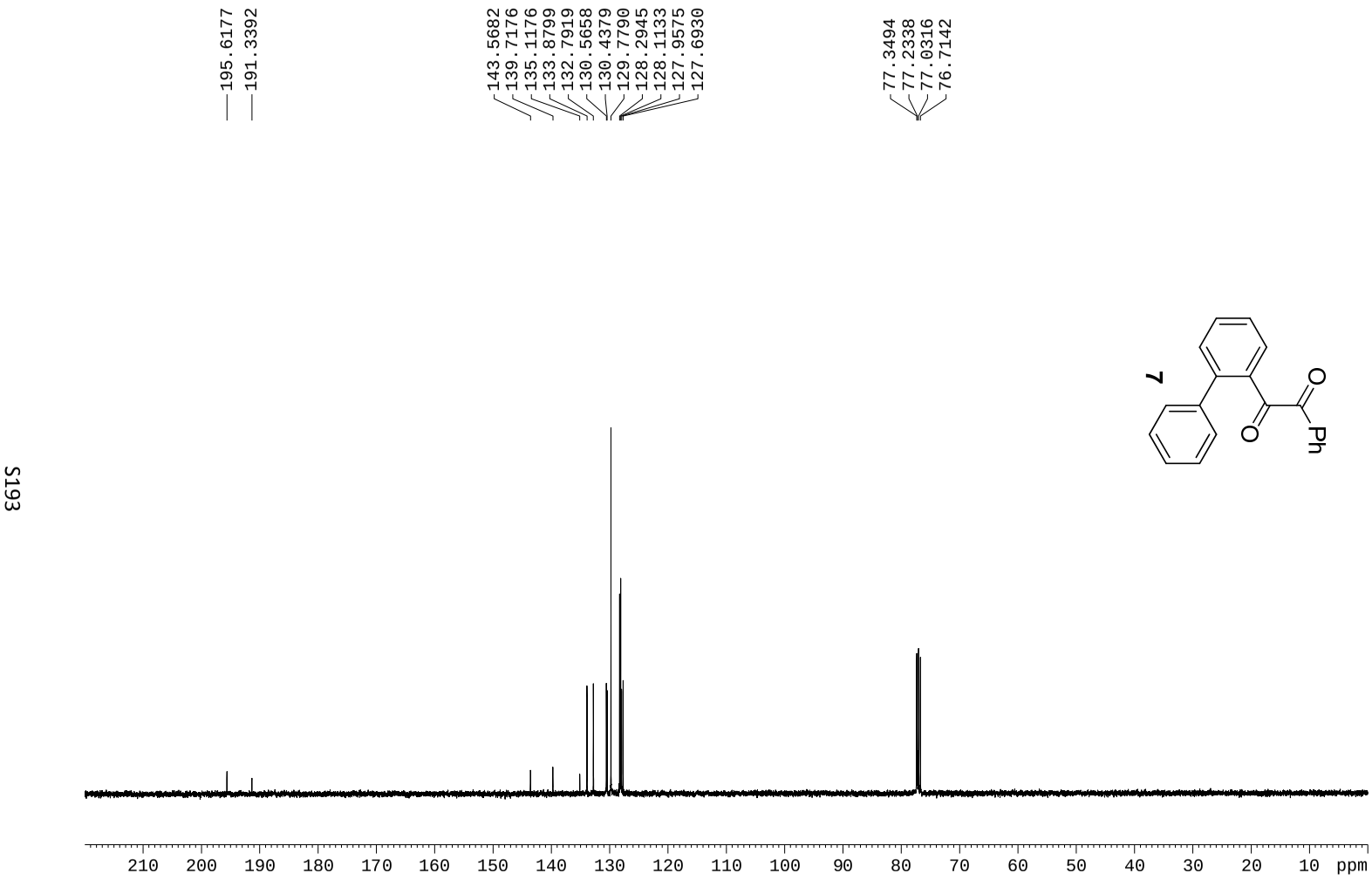



Current Data Parameters
NAME chiang2017
EXPNO 828011
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170828
Time 12.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 30
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 128
DW 83.200 usec
DE 6.50 usec
TE 302.2 K
D1 0.20000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 0.90 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



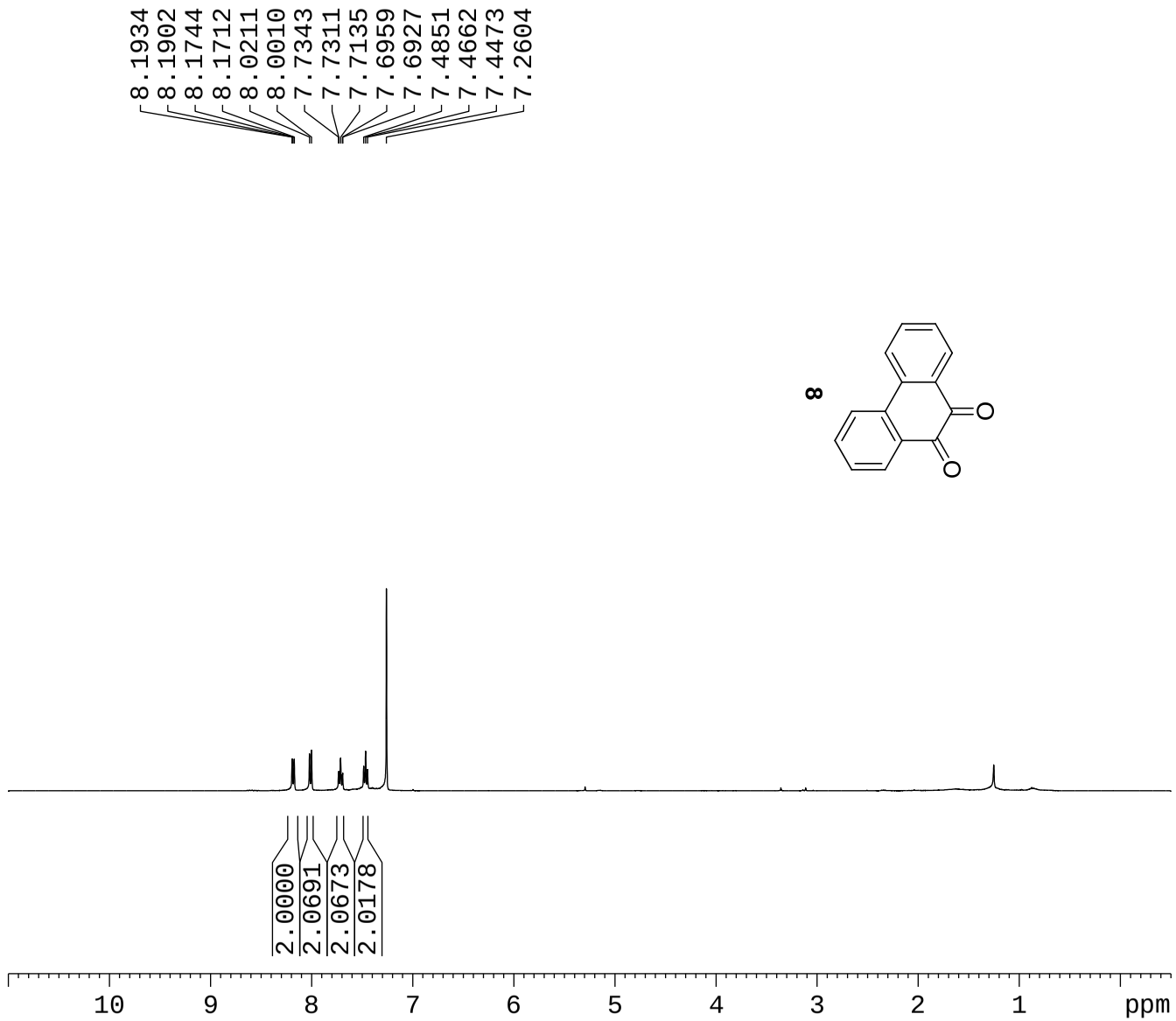

Current Data Parameters
NAME chiang2017
EXPNO 828021
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170828
Time 12.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 25250
SOLVENT CDCl3
NS 690
DS 0
SWH 25252.525 Hz
FIDRES 1.000100 Hz
AQ 0.5000000 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 302.3 K
D1 0.20000000 sec
d11 0.03000000 sec
DELTA 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 6.20 dB
SFO1 100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.40 dB
PL12 15.80 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




Current Data Parameters

| | |
|---|---|
| NAME | chen2017 |
| EXPNO | 807041 |
| PROCNO | 1 |

F2 - Acquisition Parameters

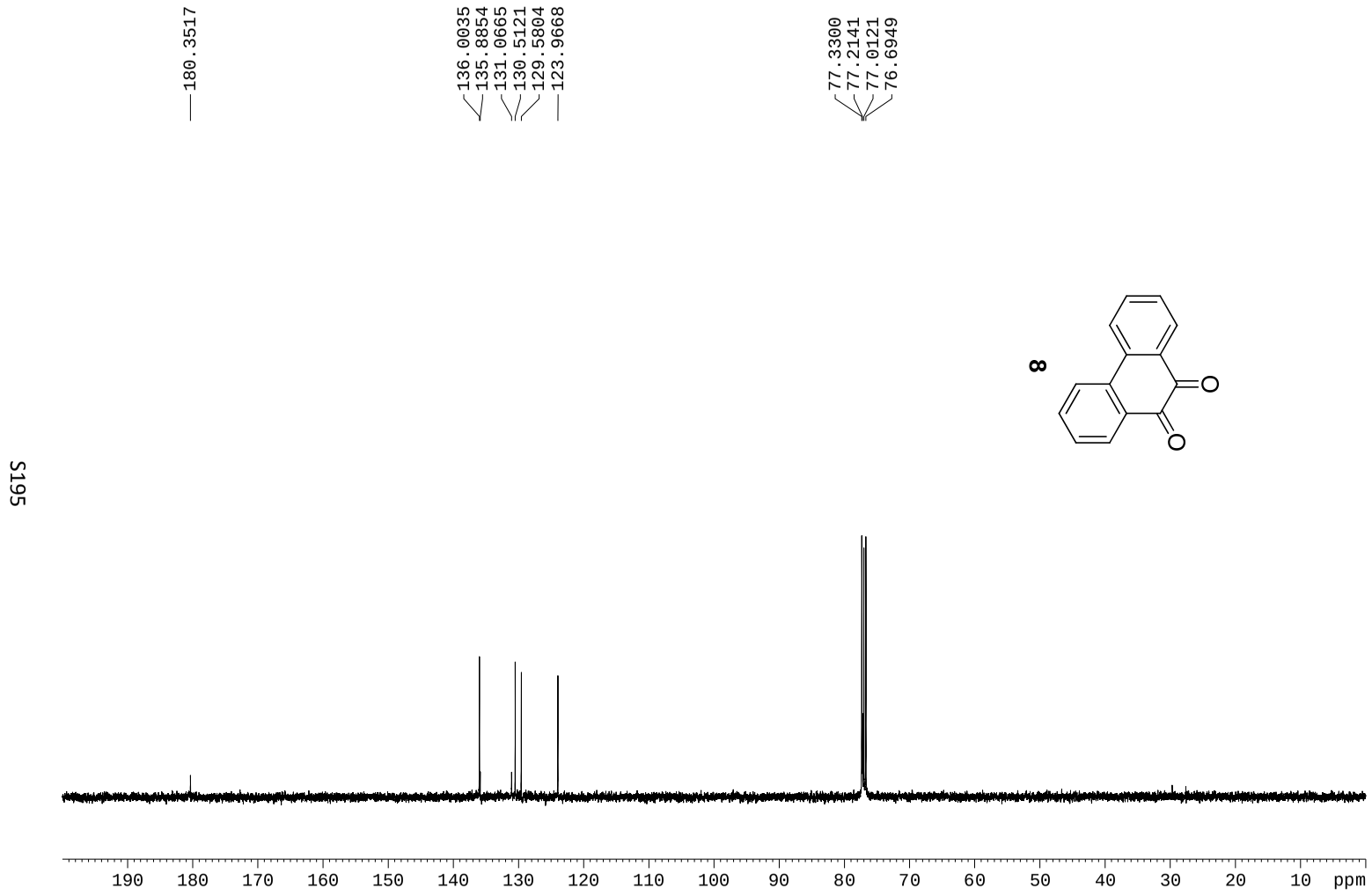
| | |
|---|---|
| Date_ | 20170807 |
| Time | 19.34 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 32768 |
| SOLVENT | CDCl3 |
| NS | 31 |
| DS | 0 |
| SWH | 6009.615 Hz |
| FIDRES | 0.183399 Hz |
| AQ | 2.7263477 sec |
| RG | 406 |
| DW | 83.200 usec |
| DE | 6.50 usec |
| TE | 301.2 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |

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

| | |
|---|---|
| NUC1 | 1H |
| P1 | 15.00 usec |
| PL1 | 0.90 dB |
| SFO1 | 400.1326008 MHz |

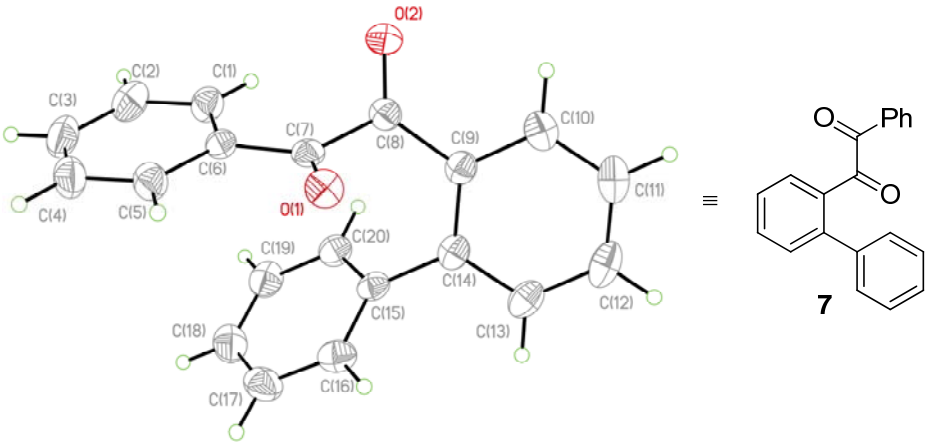
F2 - Processing parameters

| | |
|---|---|
| SI | 16384 |
| SF | 400.1300090 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

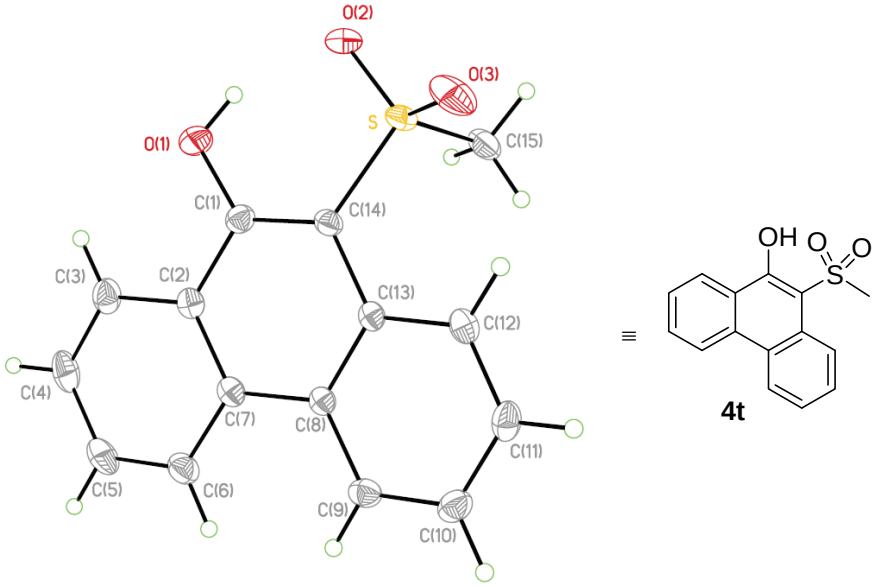



```
Current Data Parameters
NAME            chen2017
EXPNO             807042
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170807
Time               19.40
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ             1.2976629 sec
RG                  2050
DW                19.800 usec
DE                 6.50 usec
TE                301.4 K
D1            0.10000000 sec
d11           0.03000000 sec
DELTA         0.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PL1                6.20 dB
SFO1         100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.40 dB
PL12              15.80 dB
PL13              18.80 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```

## 7) X-ray crystal structures of 7 and 4t




X-ray crystal structure of compound **7** (CCDC 1813583)




X-ray crystal structure of compound **4t** (CCDC 1836777)

**References**

[S1] H. Ke, X. Chen, G. Zou, *J. Org. Chem*. **2014**, *79*, 7132-7140.

[S2] C. Zhu, Y. Zuo, R. Wang, B. Liang, X. Yue, G. Wen, N. Shang, L. Huang, Y. Chen, J. Du, and X. Bu, *J. Med. Chem*. **2014**, *57*, 6364-6382.

[S3] L.-Y. Xu, C.-Y. Liu, S.-Y. Liu, Z.-G. Ren, D. J. Young, J.-P. Lang, *Tetrahedron* **2017**, *73*, 3125-3132.

[S4] Z. Liu, H. Tan, L. Wang, T. Fu, Y. Xia, Y. Zhang, J. Wang, *Angew. Chem. Int. Ed*. **2015**, *54*, 3056 –3060.

[S5] J. Damborsky, F. Nikulenkov, A. Sisakova, S. Havel, L. Krejci, B. Carbain, J. Brezovsky, L. Daniel, K. Paruch U.S. Patent 20170197966 A1, 2017.