

**Allyl *m*-Trifluoromethyldiazirine Mephobarbital: An Unusually Potent
Enantioselective and Photoreactive Barbiturate General Anesthetic**

Supporting Information

Pavel Y. Savechenkov[§]

Xi Zhang[‡]

David C. Chiara[†]

Deirdre S. Stewart[‡]

Rile Ge[‡]

Xiaojuan Zhou[‡]

Douglas E. Raines[‡]

Jonathan B. Cohen[†]

Stuart A. Forman[‡]

Keith W. Miller^{‡#}

Karol S. Bruzik^{*§}

[‡]Department of Anesthesia, Critical Care and Pain Medicine, Massachusetts General Hospital, 32 Fruit Street, Boston, Massachusetts 02114, [#]Department of Biological Chemistry and [#]Molecular Pharmacology, and [†]Department of Neurobiology, 220 Longwood Avenue, Harvard Medical School, Boston, Massachusetts 02115, and [§]Department of Medicinal Chemistry and Pharmacognosy, University of Illinois at Chicago, 833 S. Wood Street (M/C 781), Chicago, IL 60612-7231

Supporting information available: (1) Chiral chromatography of (\pm)-**14**, (2) HPLC of the mixture after hydrogenation of compound **15**, (3) determination of pK_a value of **14**, (4) NMR data for all new synthesized compounds.

Figure 1s. HPLC separation of enantiomers of compound **14**. Conditions: Daicell Chiralpack AD[®] column, 20x200 mm eluted with isocratic 15% ethanol in hexane as an eluent at 6.5 mL/min flow rate.

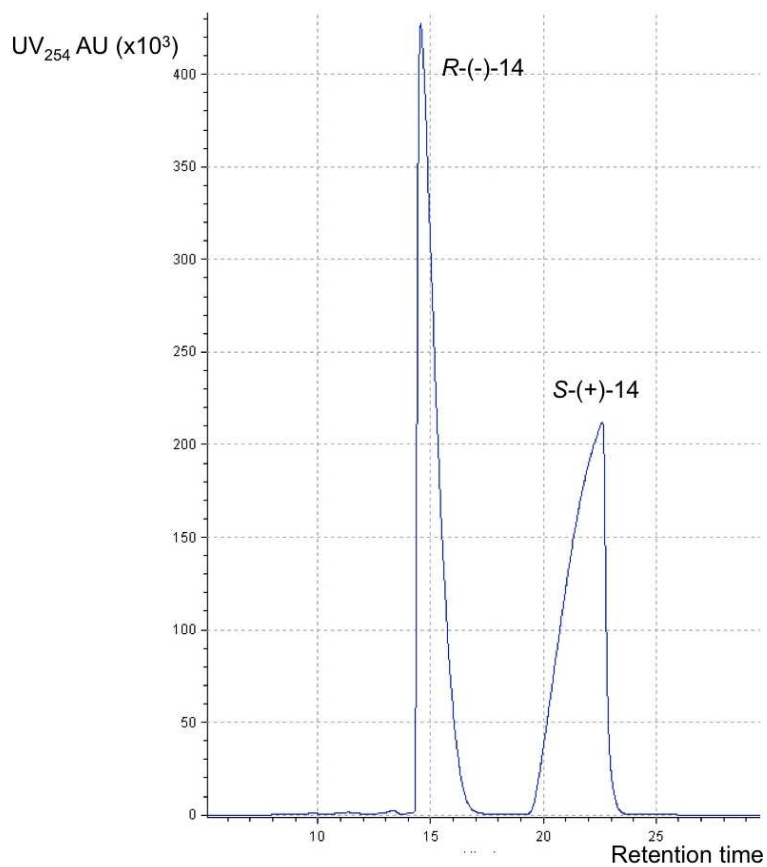
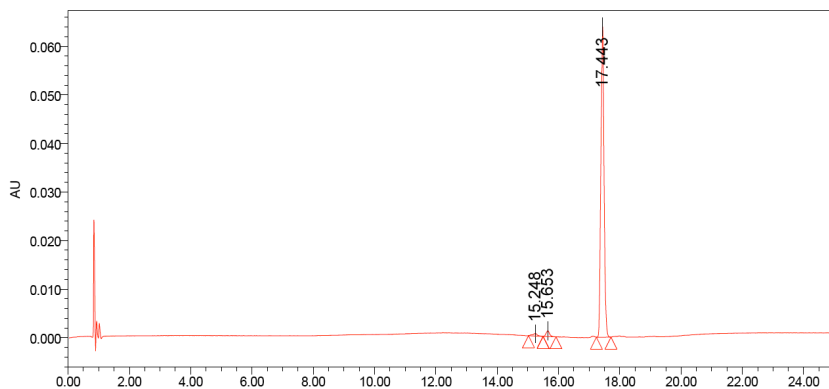
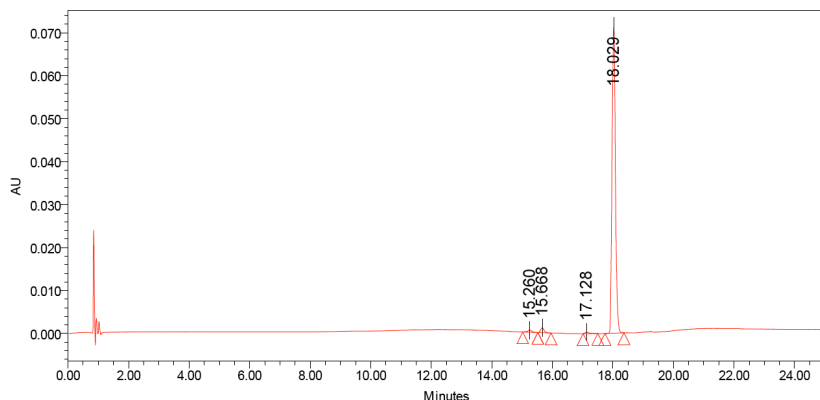


Figure 2s. Separation of compounds **14** and **15** by reverse phase HPLC. HPLC method: Luna 5 μm C18 100 \AA column, 150 x 3.00 mm from Phenomenex. During the first 2 minutes isocratic 0.05 M TFA was used as an eluent; starting at 2 min the gradient of 0.05 M TFA/MeOH (0-100%) was used to reach 100% at 17 min, and elution was continued with isocratic MeOH. Flow rate: 1 mL/min. Detection – UV array detector at 254 nm. (A) starting propargyl barbiturate **15**; (B) product allyl barbiturate **14**; (C) the mixture of **14** and **15**.

(A)



(B)



(C)

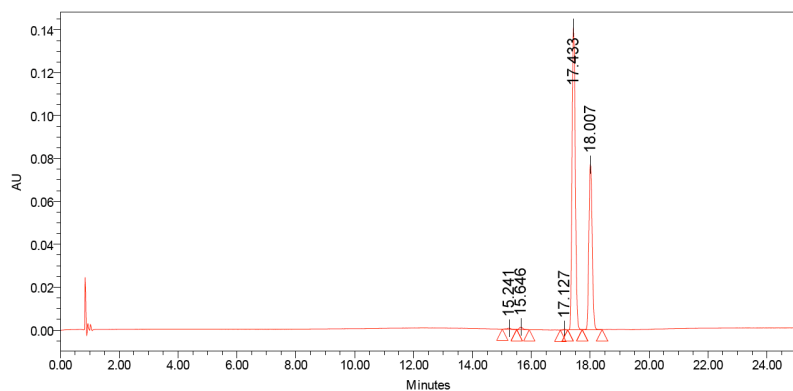


Figure 3s. Determination of pK_a of barbiturate (\pm)-**14**.

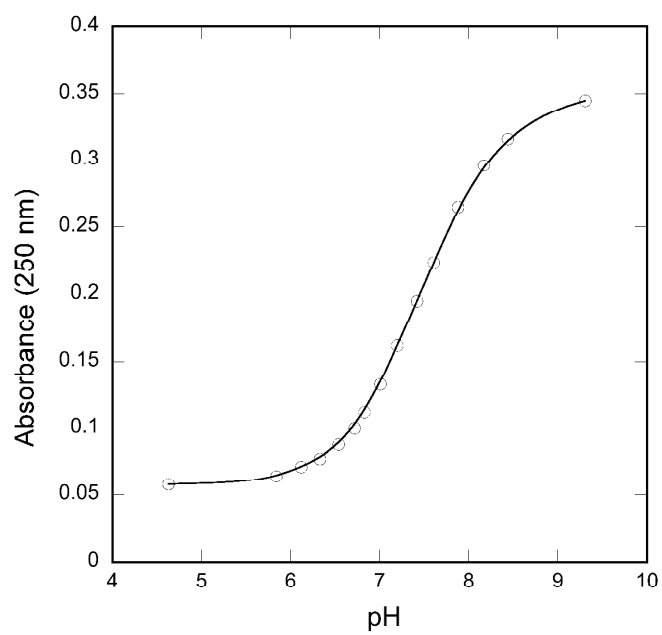
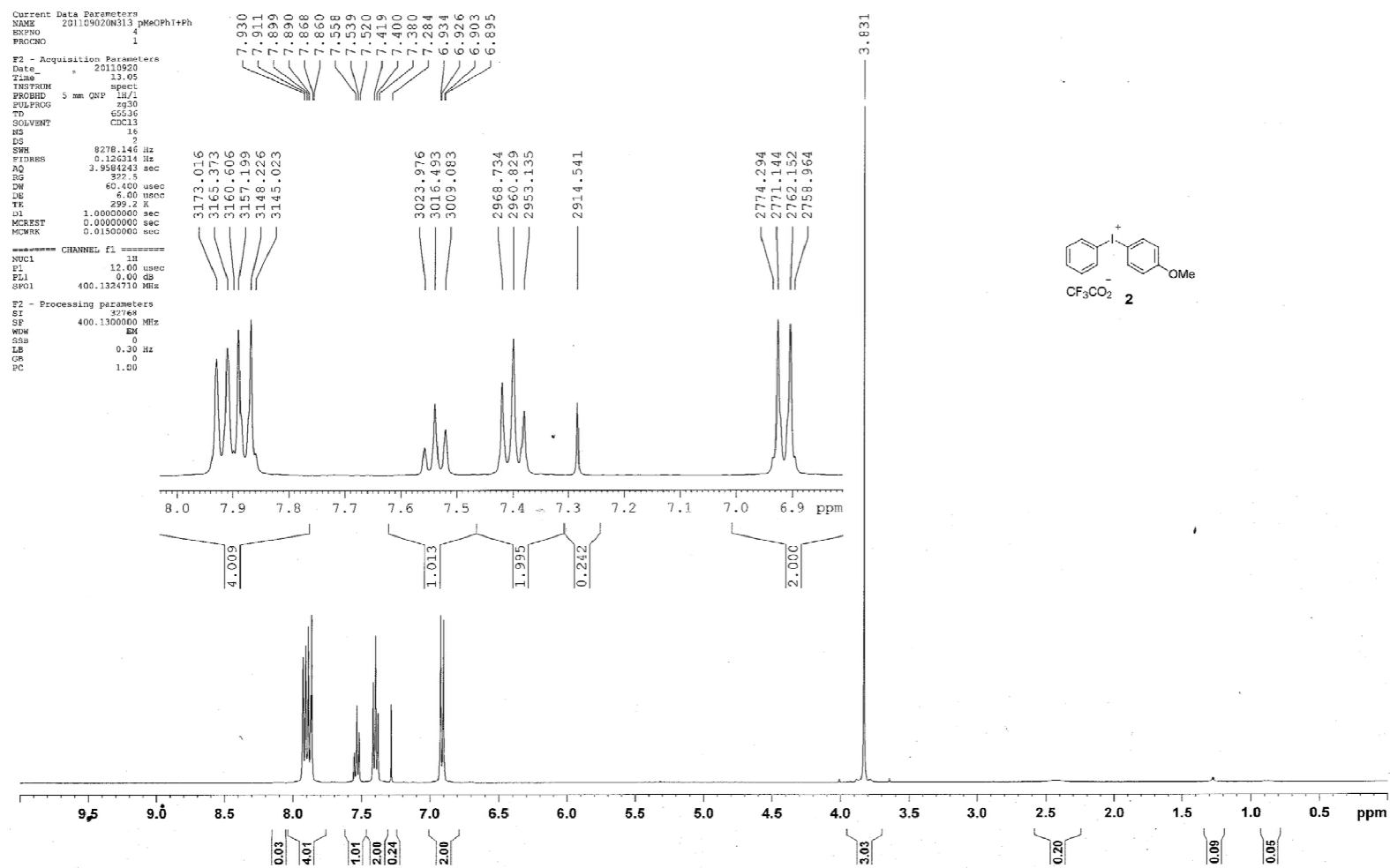


Figure 4s. NMR Data for Synthetic Intermediates and Products.



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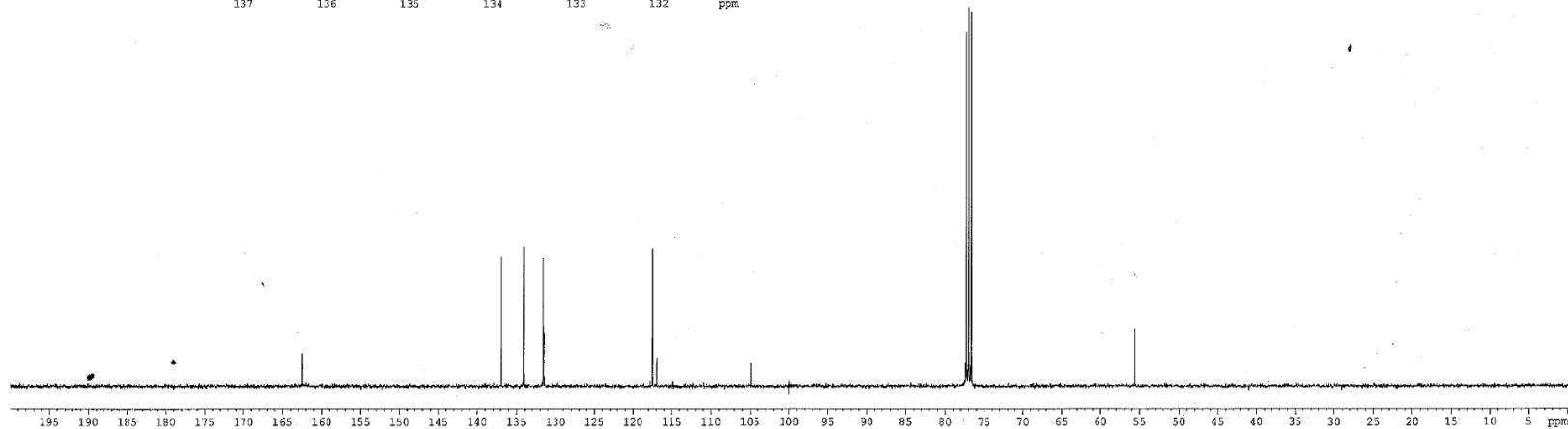
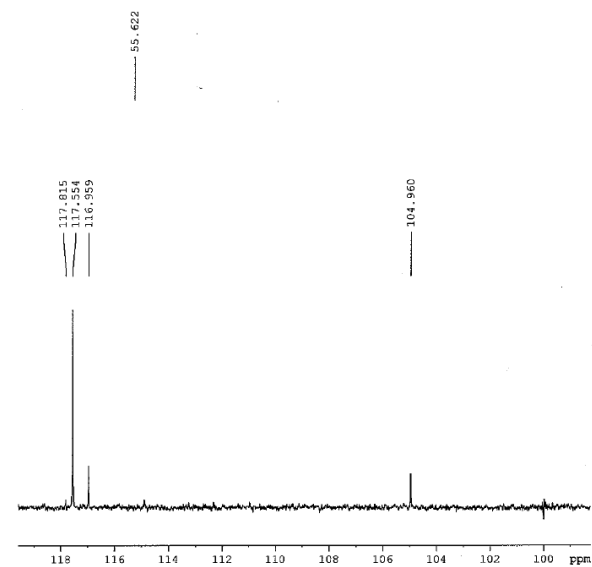
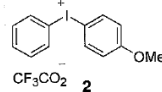
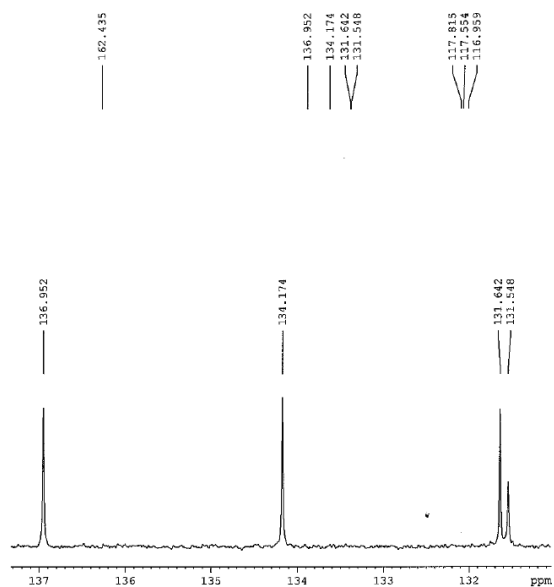
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SFO1 100.6228298 MHz

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PL2 0.00 dB
PL12 17.00 dB
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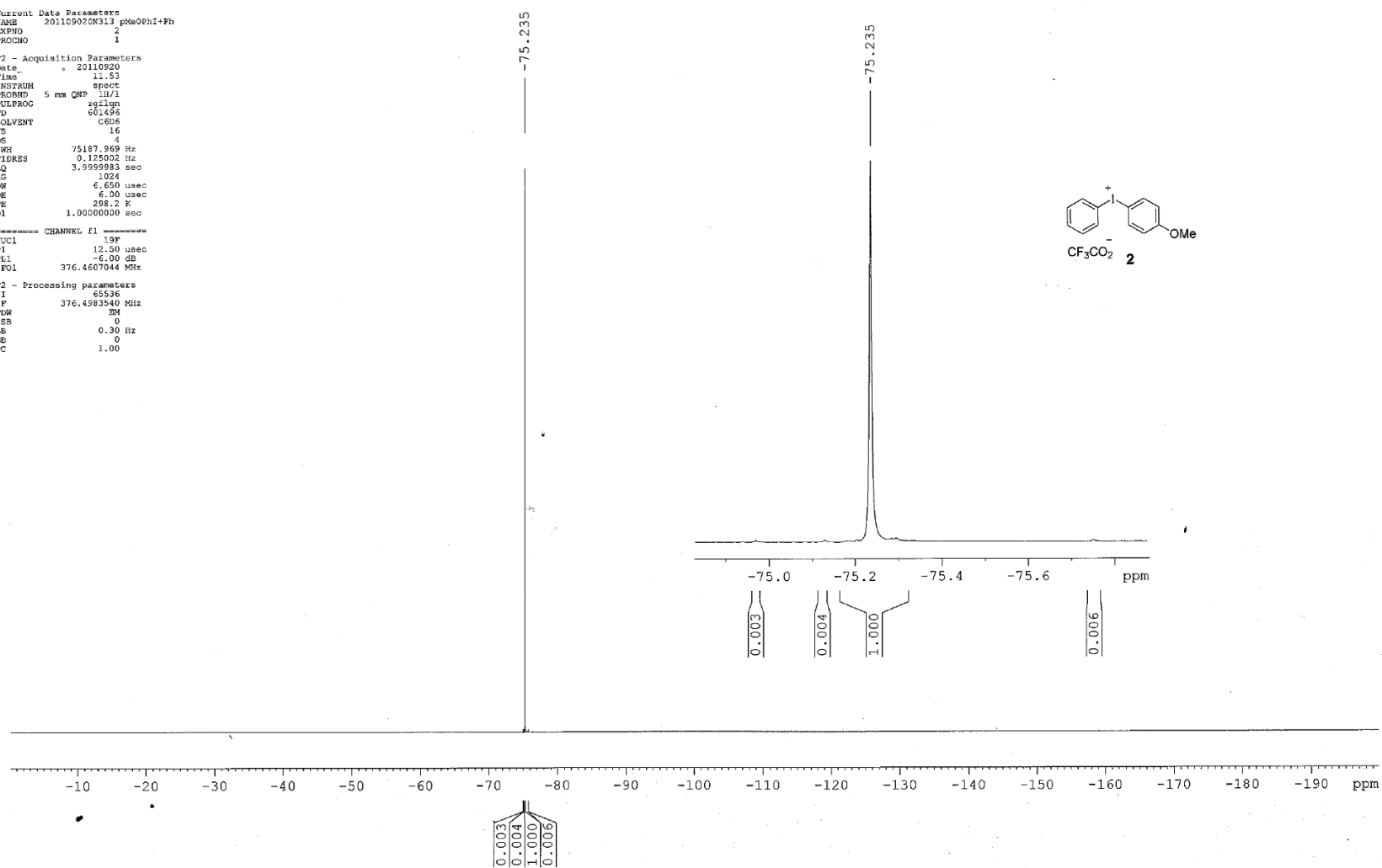


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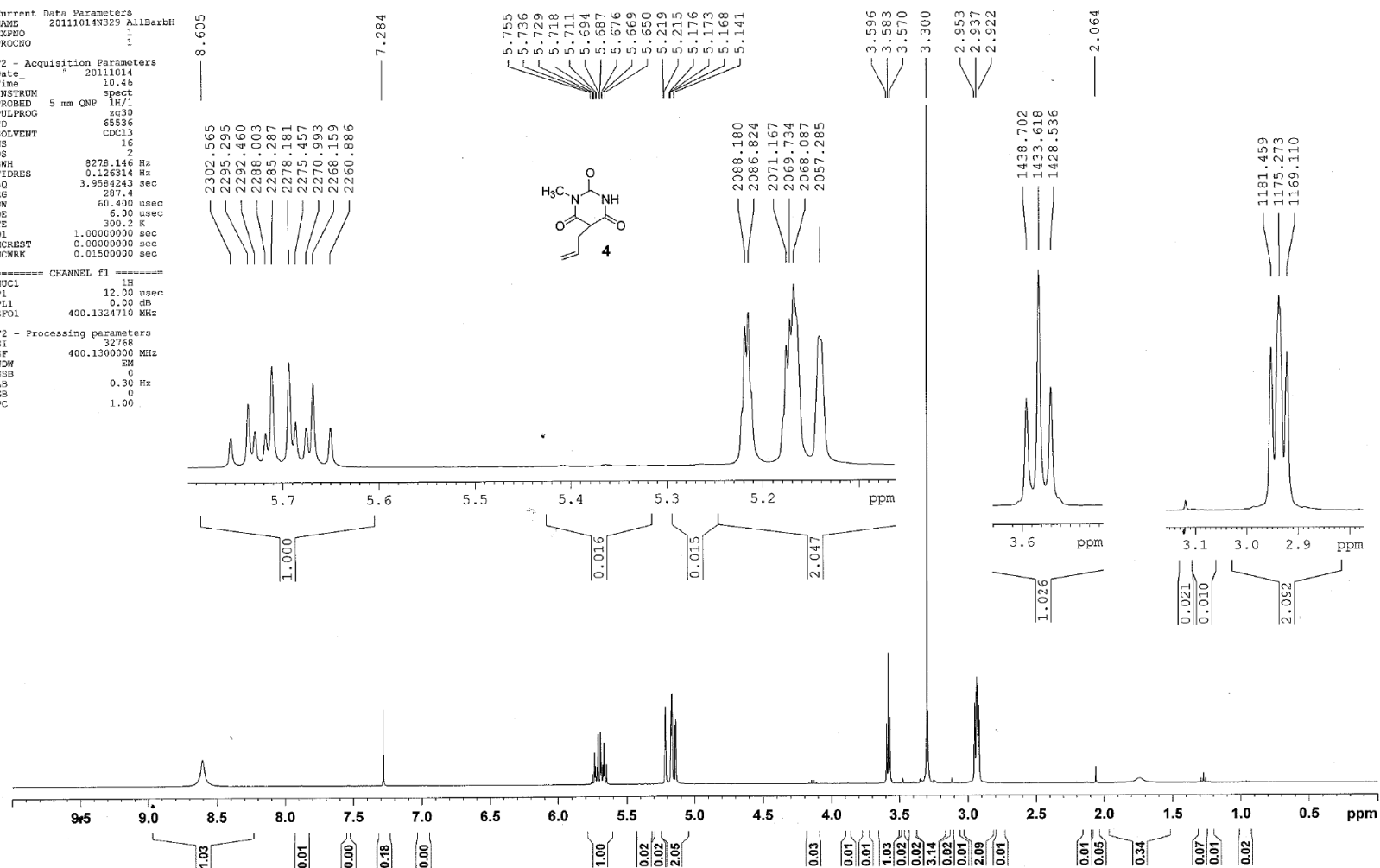


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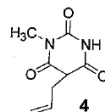
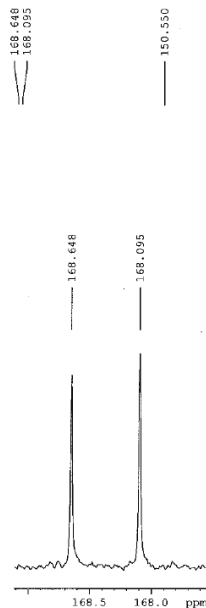
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FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 912.3
SW 20.850 usec
DE 6.00 usec
TE 297.4 K
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PL1 -2.00 dB
SFO1 100.6328888 MHz

CHANNEL F2
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PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.30 dB
PL13 14.00 dB
SFO2 400.1716007 MHz

F2 - Processing parameters
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SSB 0
LB 1.00 Hz
GB 0
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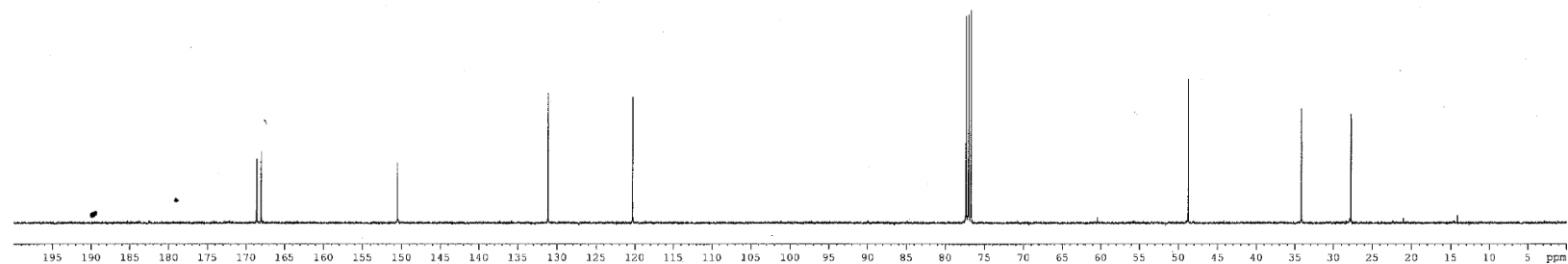
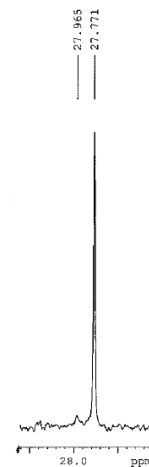
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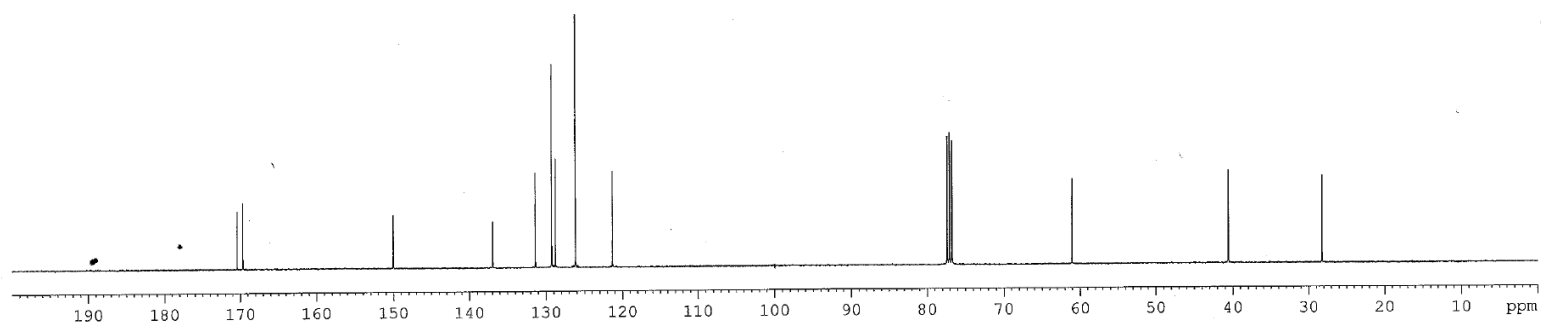
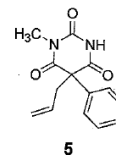
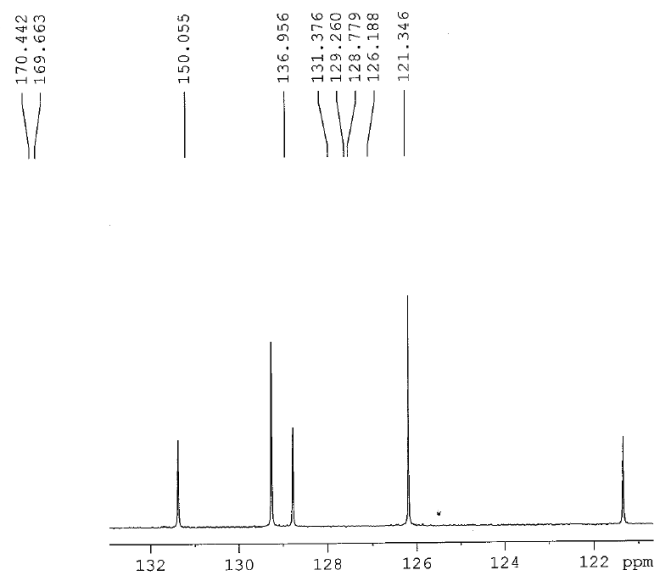
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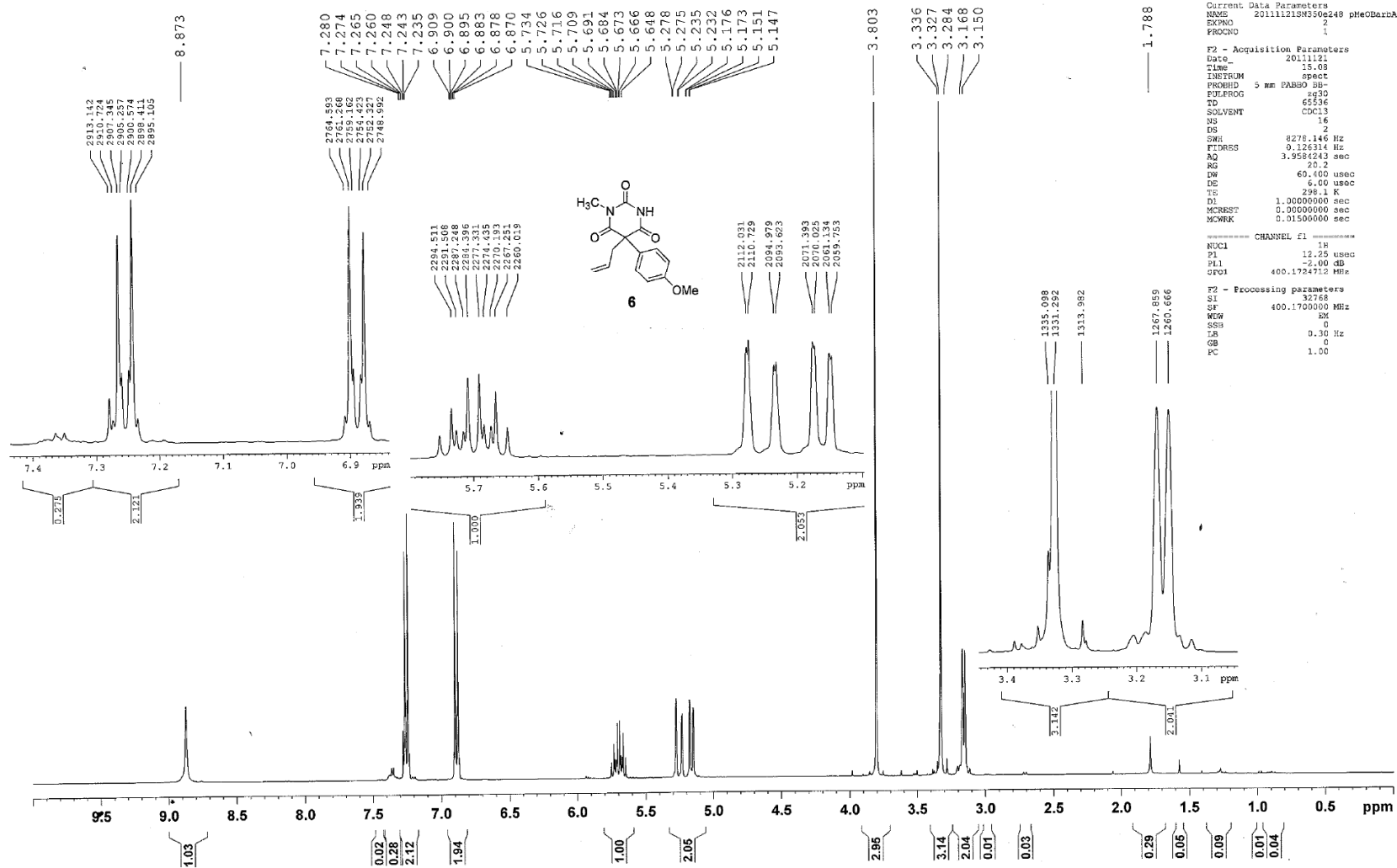
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 FIDRES 0.365918 Hz
 AQ 1.3664758 sec
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 DE 6.00 usec
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 DL 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWEX 0.01500000 sec

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 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 100.628298 MHz

----- CHANNEL f2 -----
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
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F2 - Processing parameters
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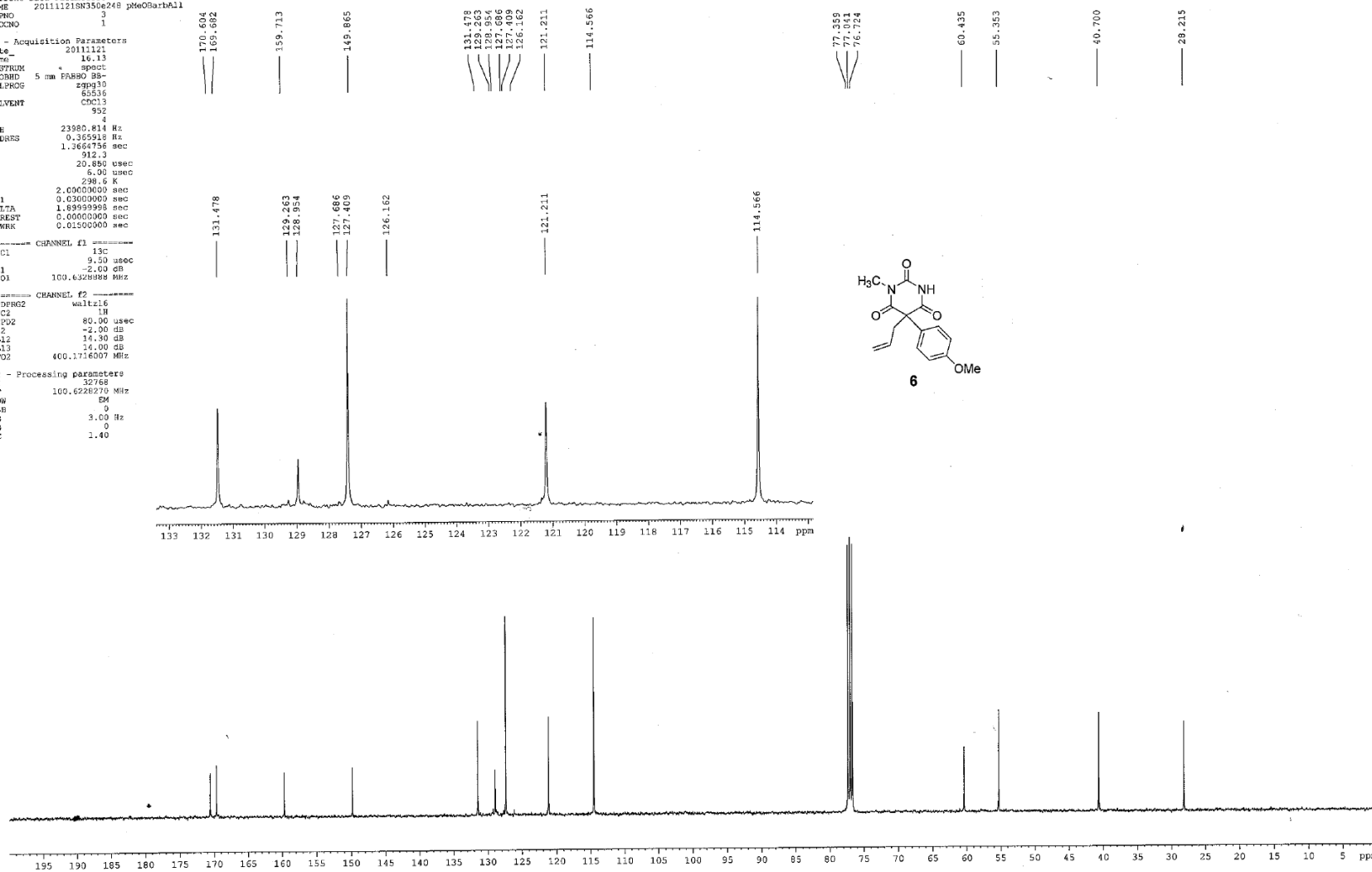
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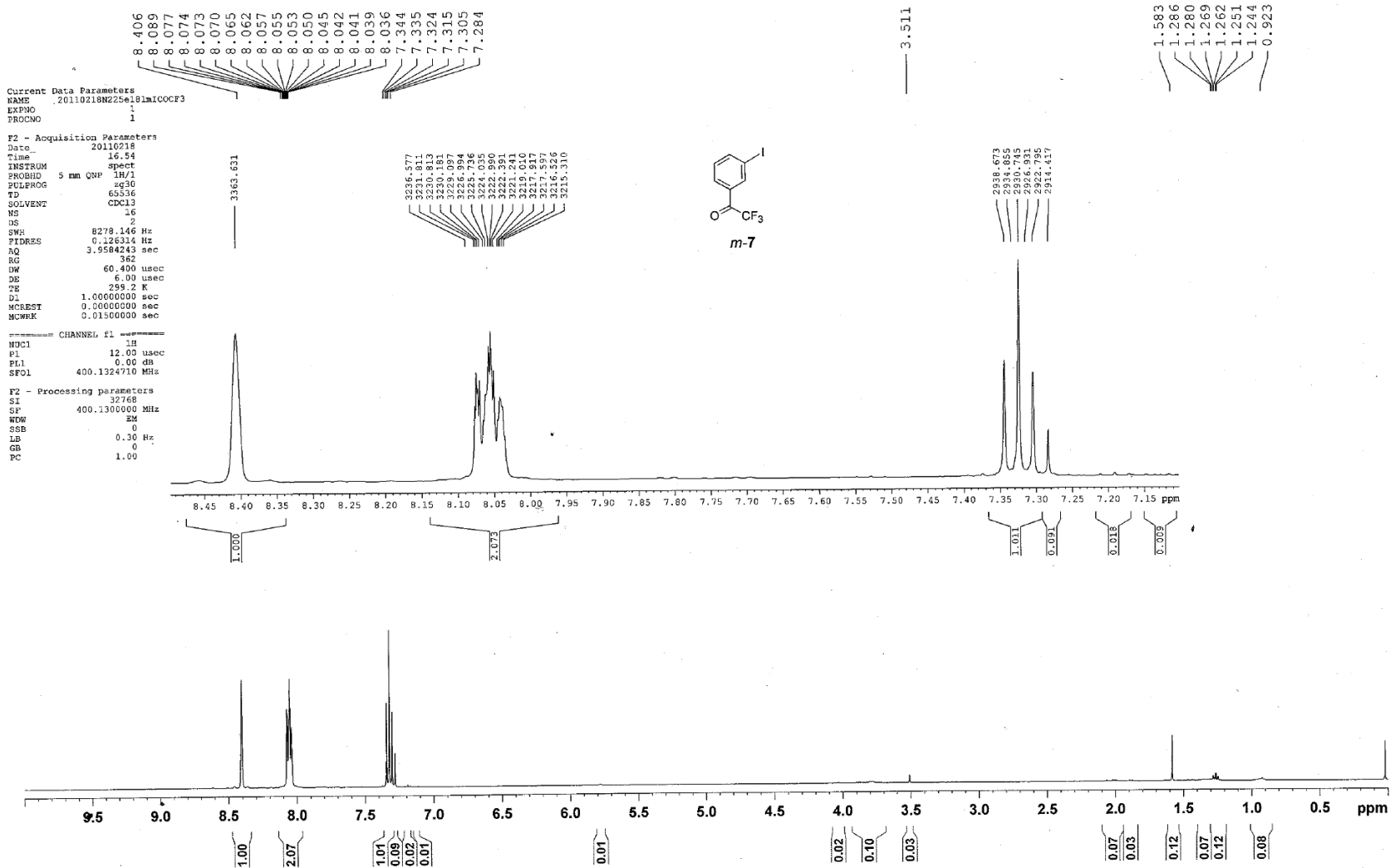
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 RG 912.3
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 PL1 2.00 dB
 SFO1 100.628270 MHz

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 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 14.30 dB
 PL13 14.00 dB
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F2 - Processing parameters
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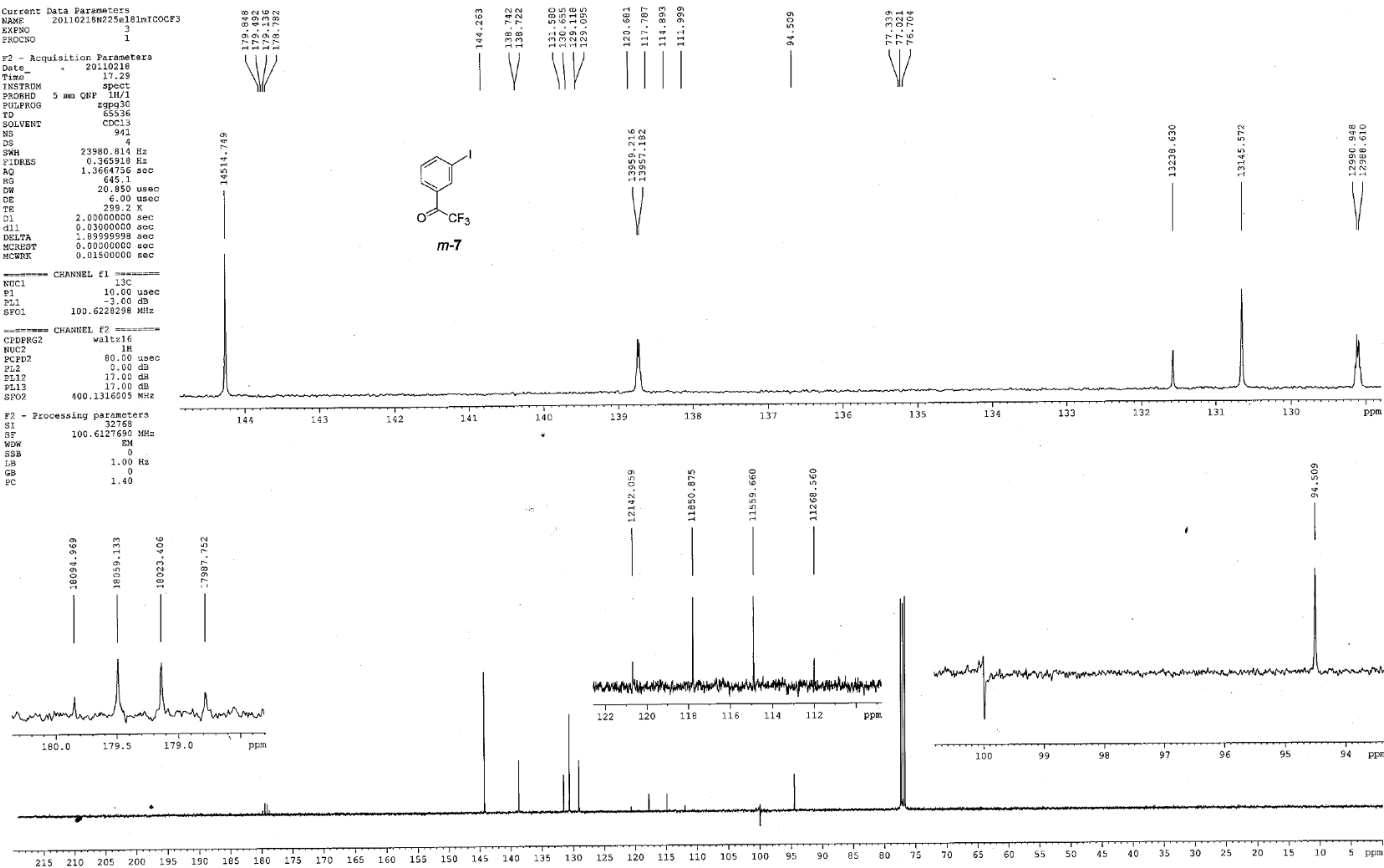
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SOLVENT CDCl3
NS 941
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FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 645.1
DW 20.850 usec
DE 6.00 usec
TE 298.2 K
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d11 0.03000000 sec
DELTA 1.89999999 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

CHANNEL f1
NUC1 13C
P1 10.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

CHANNEL f2
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NUC2 1H
PCPD2 80.30 usec
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PL12 17.00 dB
PL13 17.00 dB
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F2 - Processing parameters
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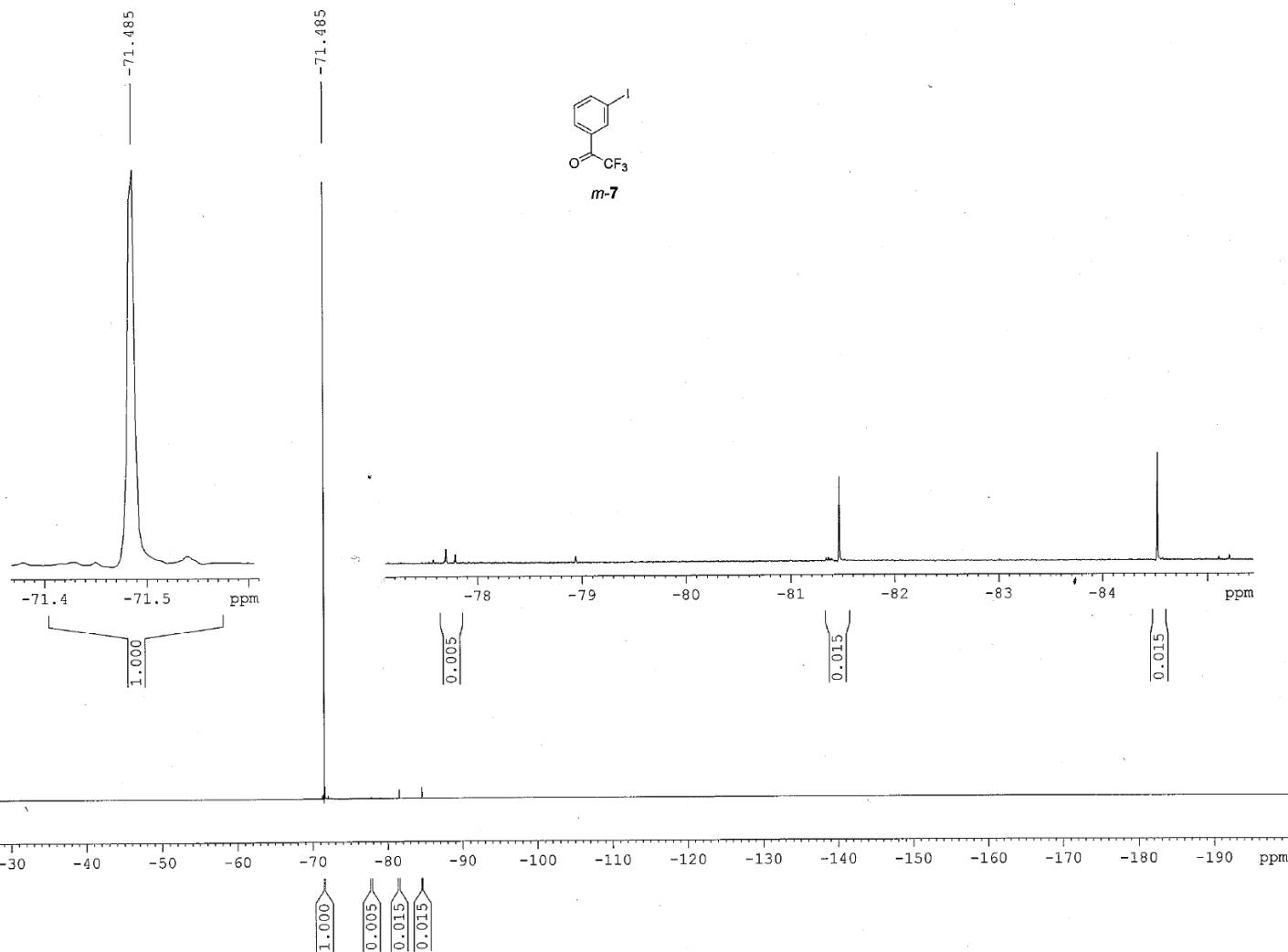
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SOLVENT C6D6
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FIDRES 0.125002 Hz
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RG 574.7
DW 6.650 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec

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PL1 -6.00 dB
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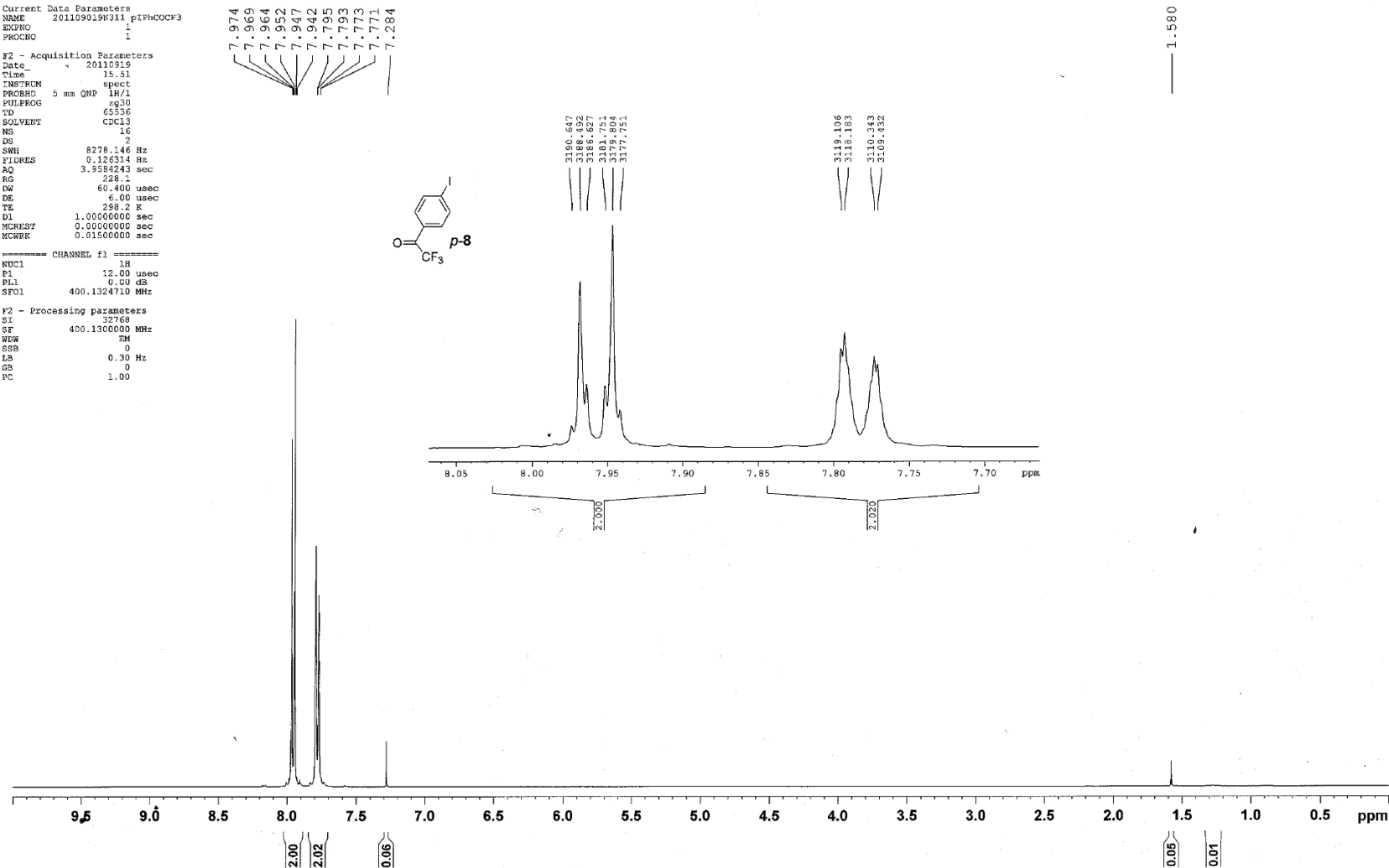


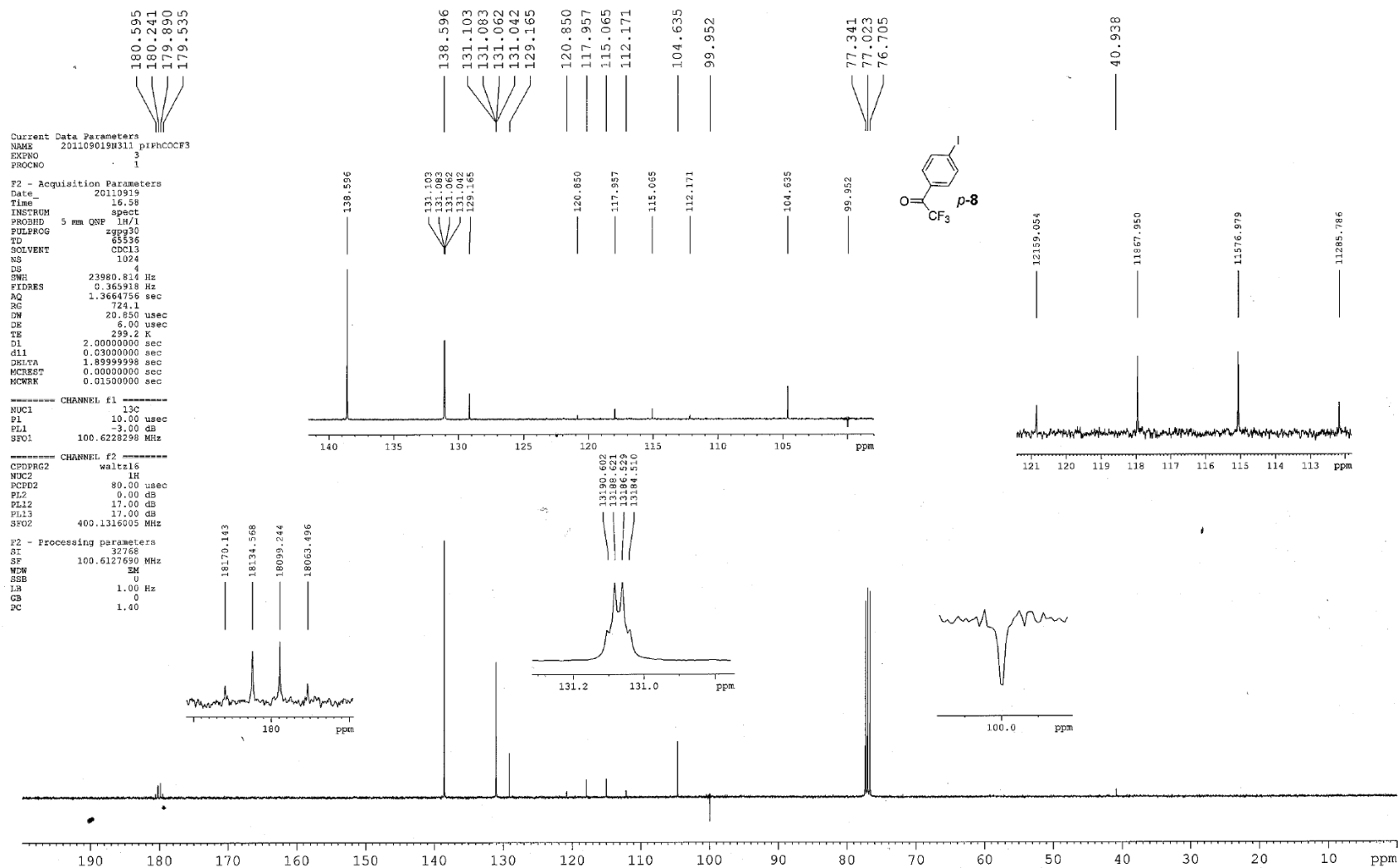
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SOLVENT CDCl3
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FIDRES 0.126314 Hz
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RG 228.1
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DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
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MCWRR 0.01500000 sec

CHANNEL f1 -----
NUC1 1H
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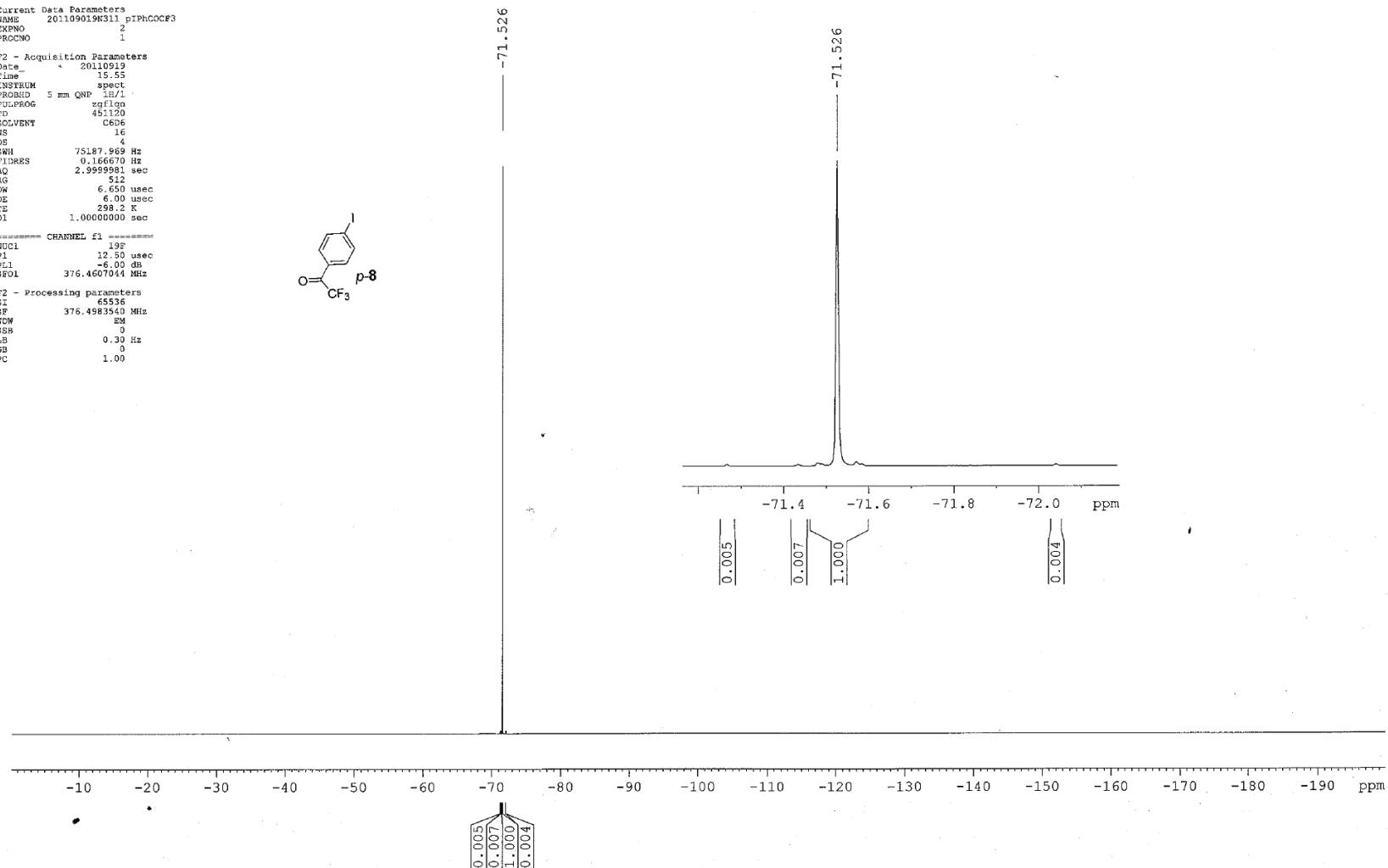
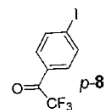


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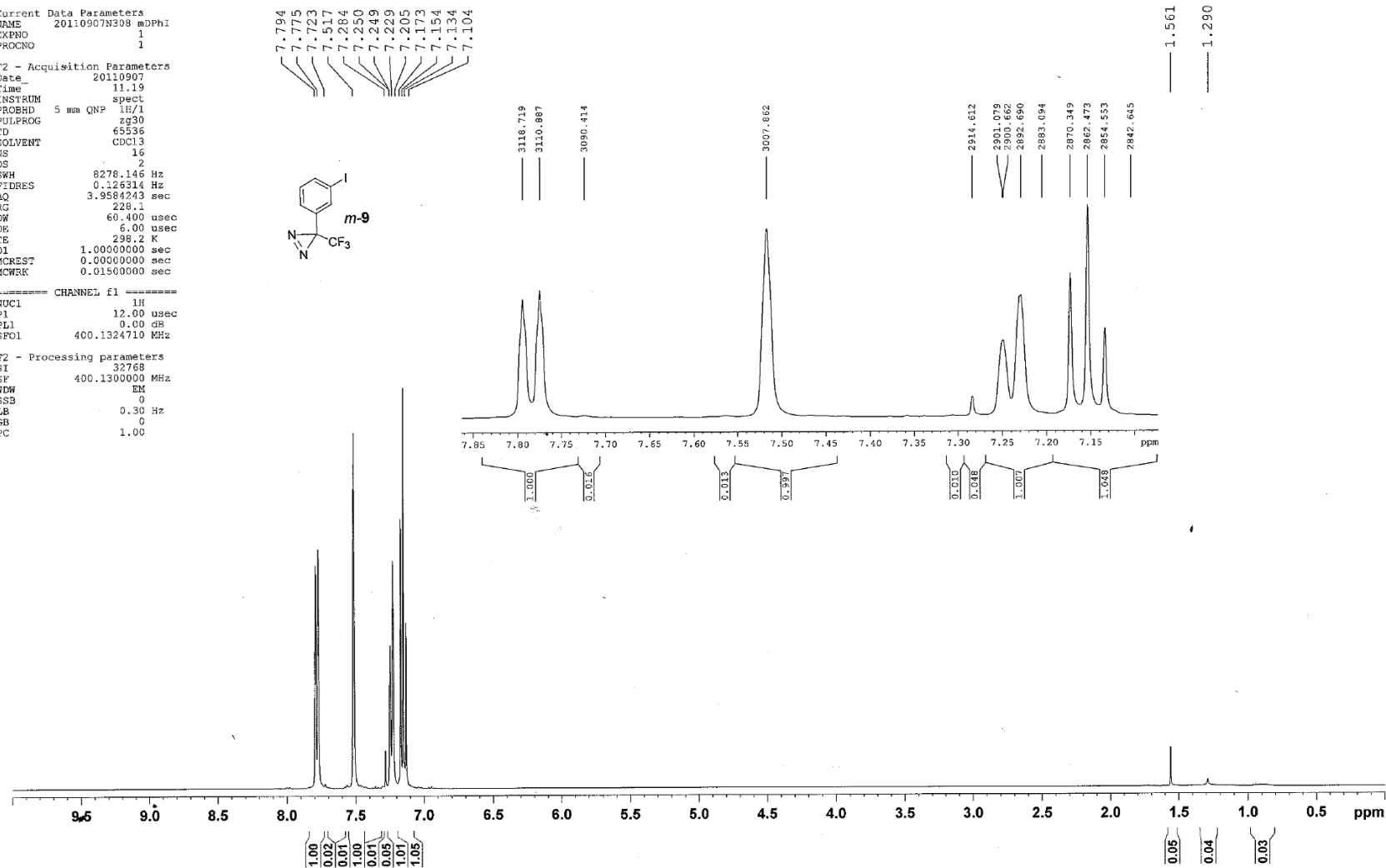


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 SOLVENT CDCl3
 NS 16
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 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
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 DE 6.00 usec
 TE 298.2 K
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 MCREST 0.00000000 sec
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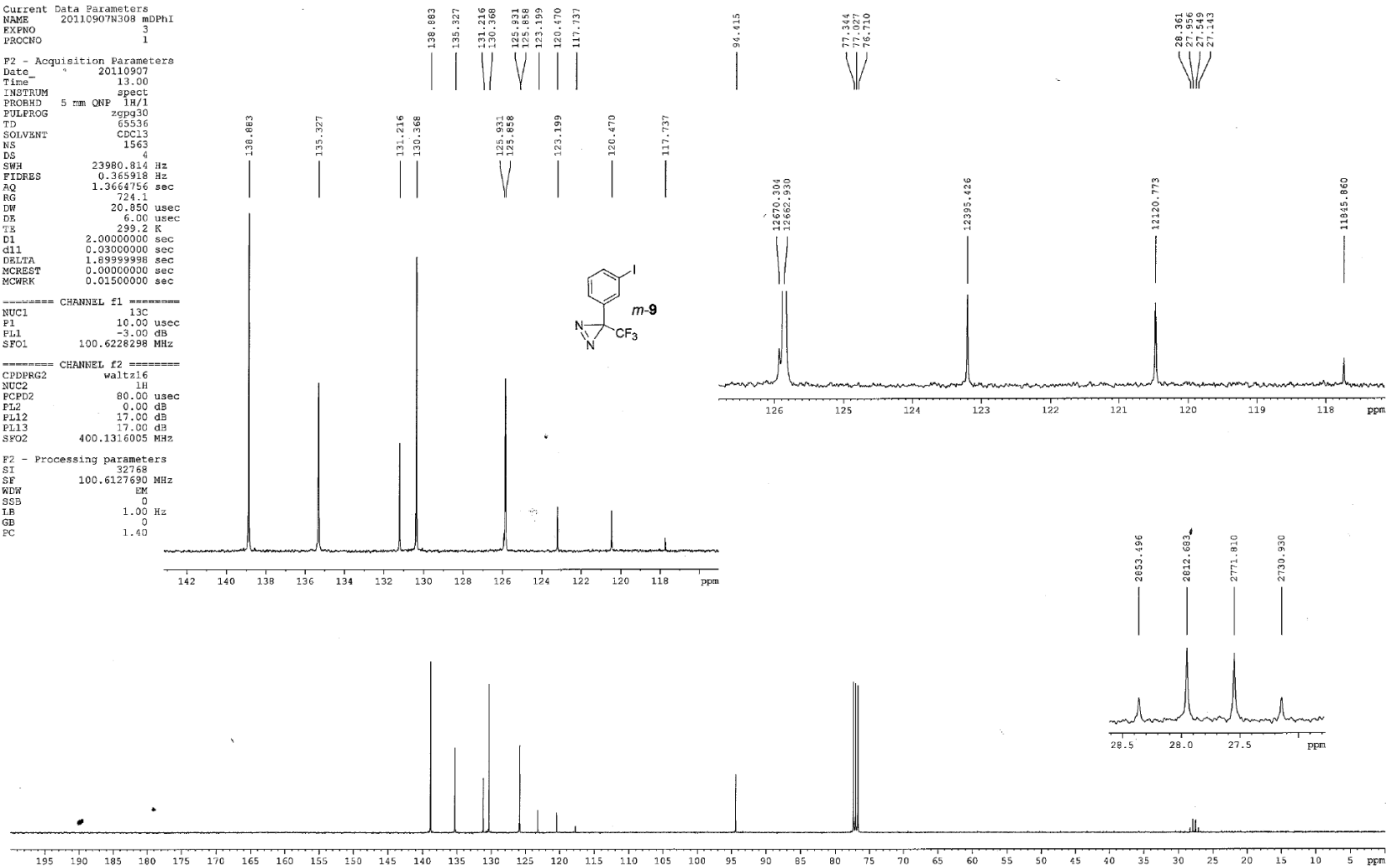
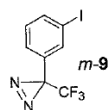
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 RG 724.1
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 DE 6.00 usec
 TE 299.2 K
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===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
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F2 - Processing parameters
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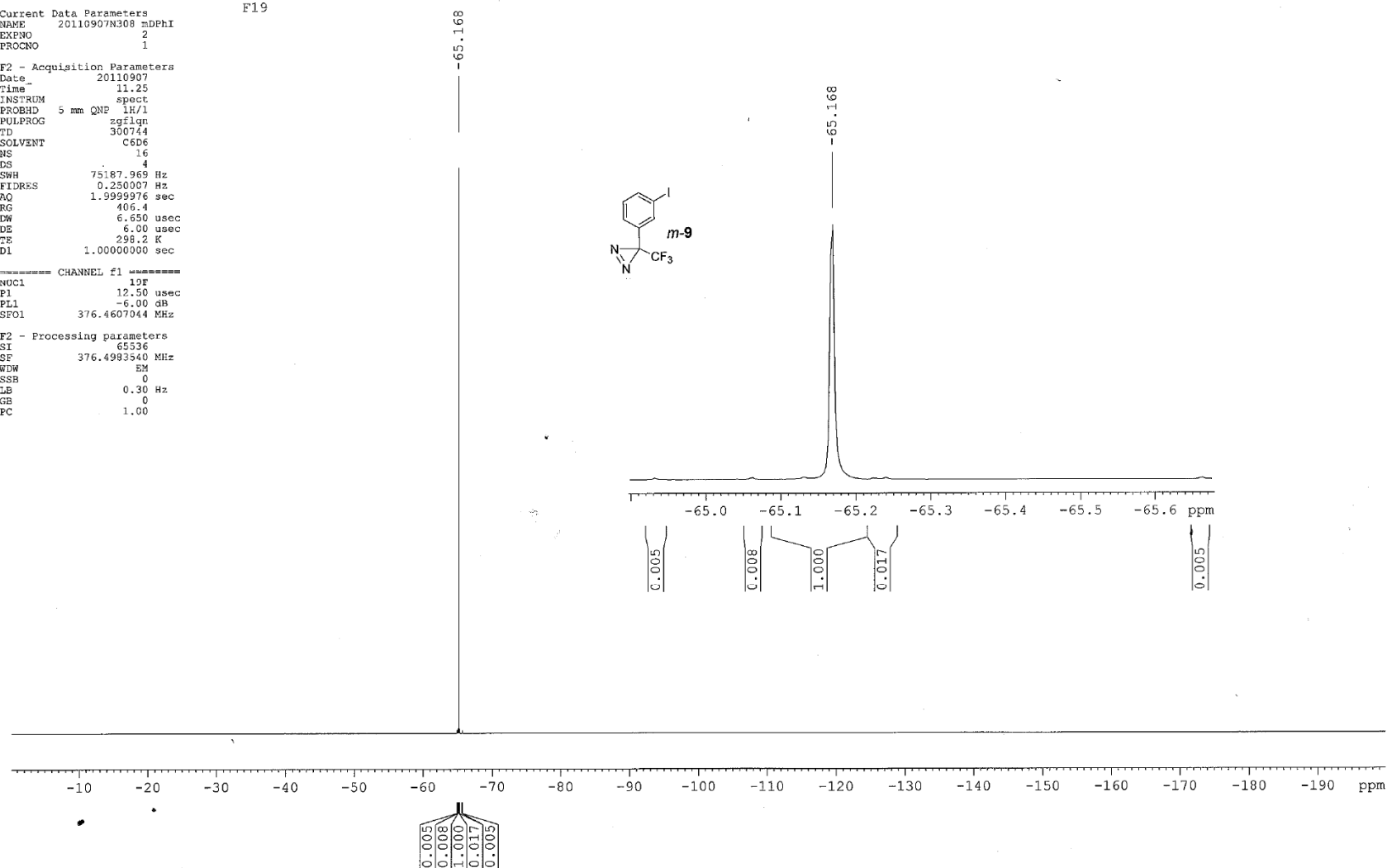
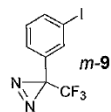
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PROCNO 1

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PULPROG zgpg30
TD 300744
SOLVENT C6D6
NS 16
DS 4
SWH 75187.969 Hz
FIDRES 0.250007 Hz
AQ 1.9999976 sec
RG 406.4
DW 6.650 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 12.50 usec
PL1 -6.00 dB
SFO1 376.4607044 MHz

F2 - Processing parameters
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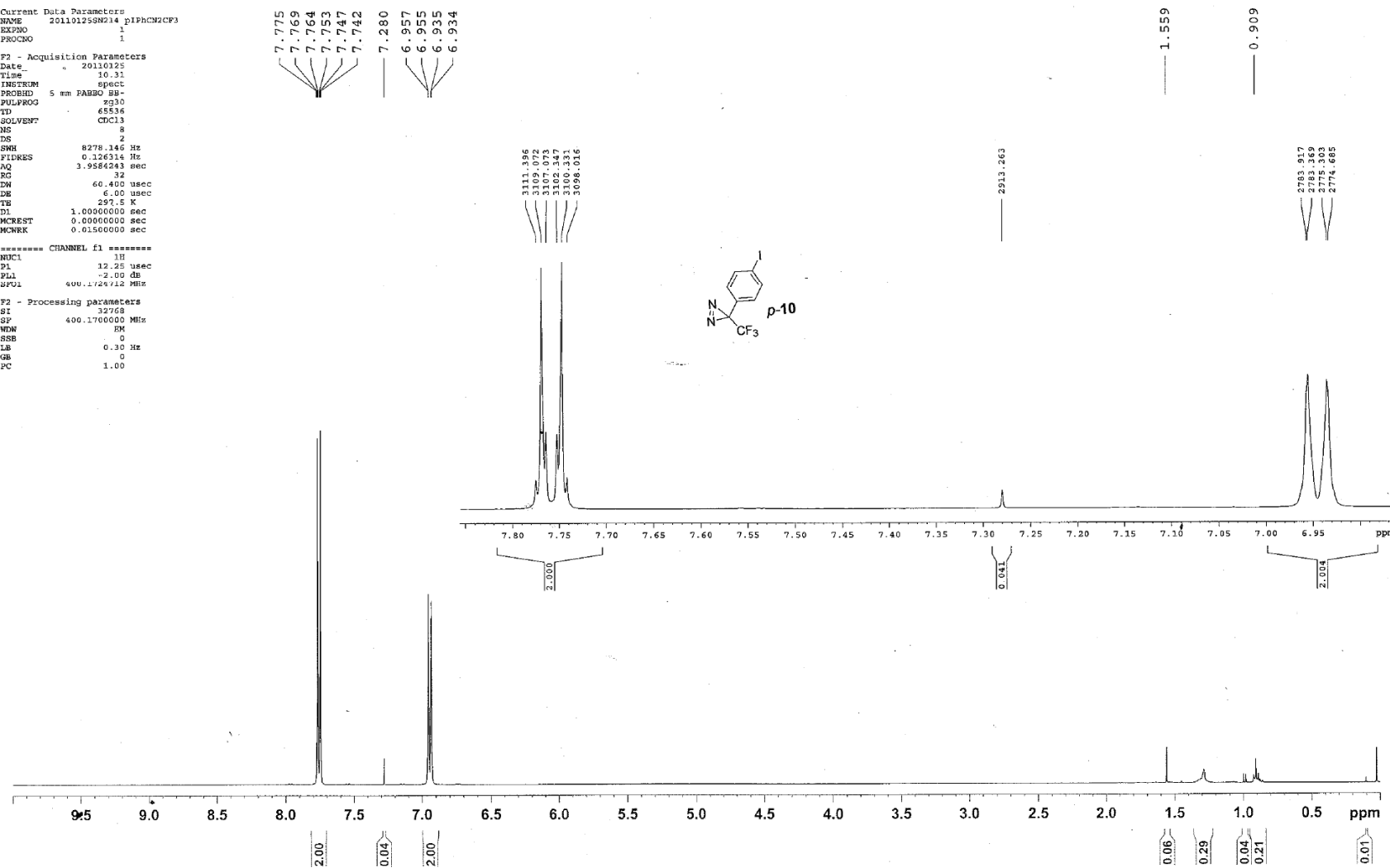


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 SOLVENT CDCl3
 NS 8
 DS 2
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 FIDRES 0.126314 Hz
 AQ 3.9564243 sec
 RG 32
 DW 60.400 usec
 DE 6.00 usec
 TE 297.5 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCNKK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 12.25 usec
 PL1 -2.00 dB
 SFO1 400.1424112 MHz

F2 - Processing parameters
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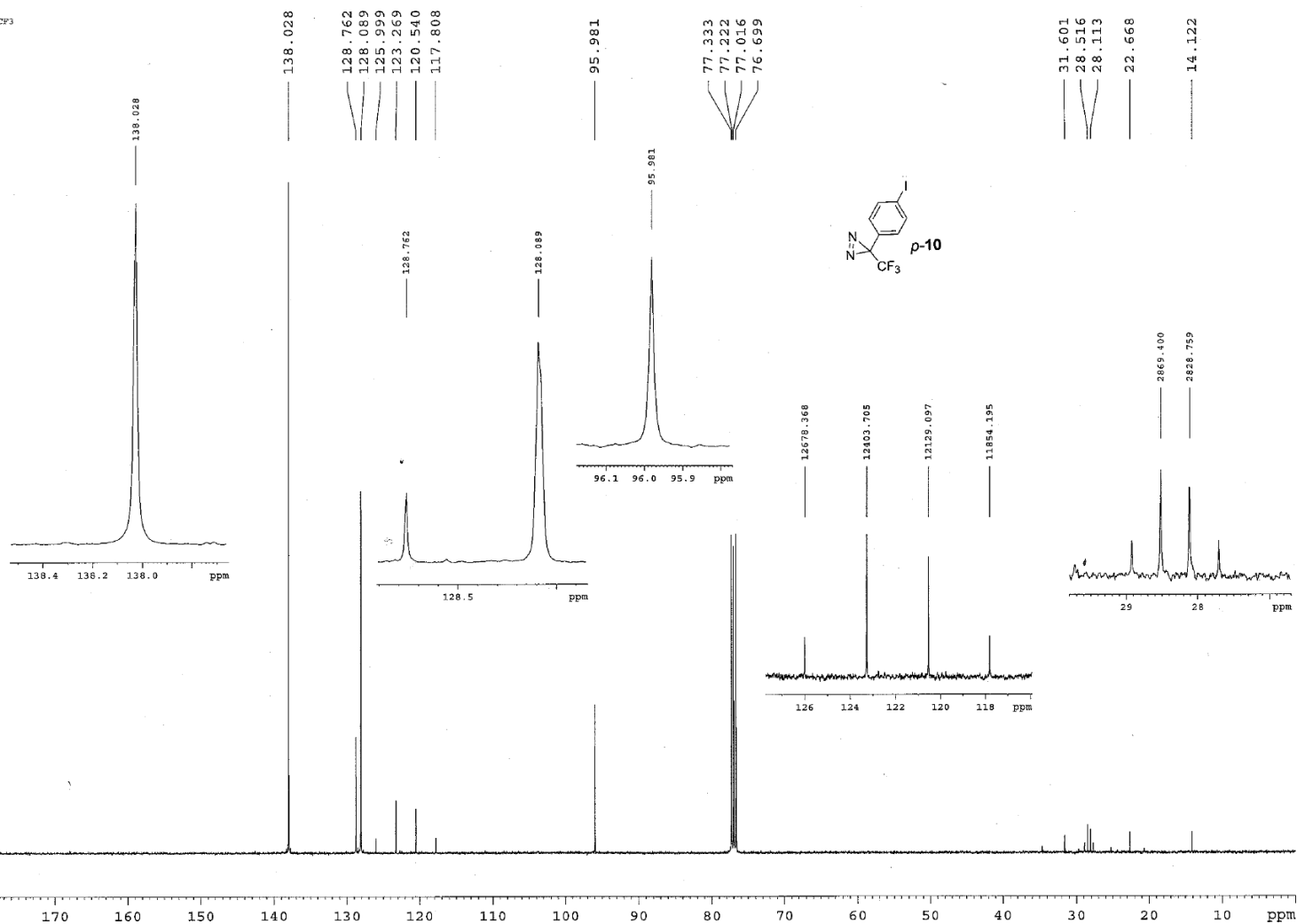
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 SOLVENT CDCl3
 NS 1934
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 RG 1290.2
 DW 20.850 usec
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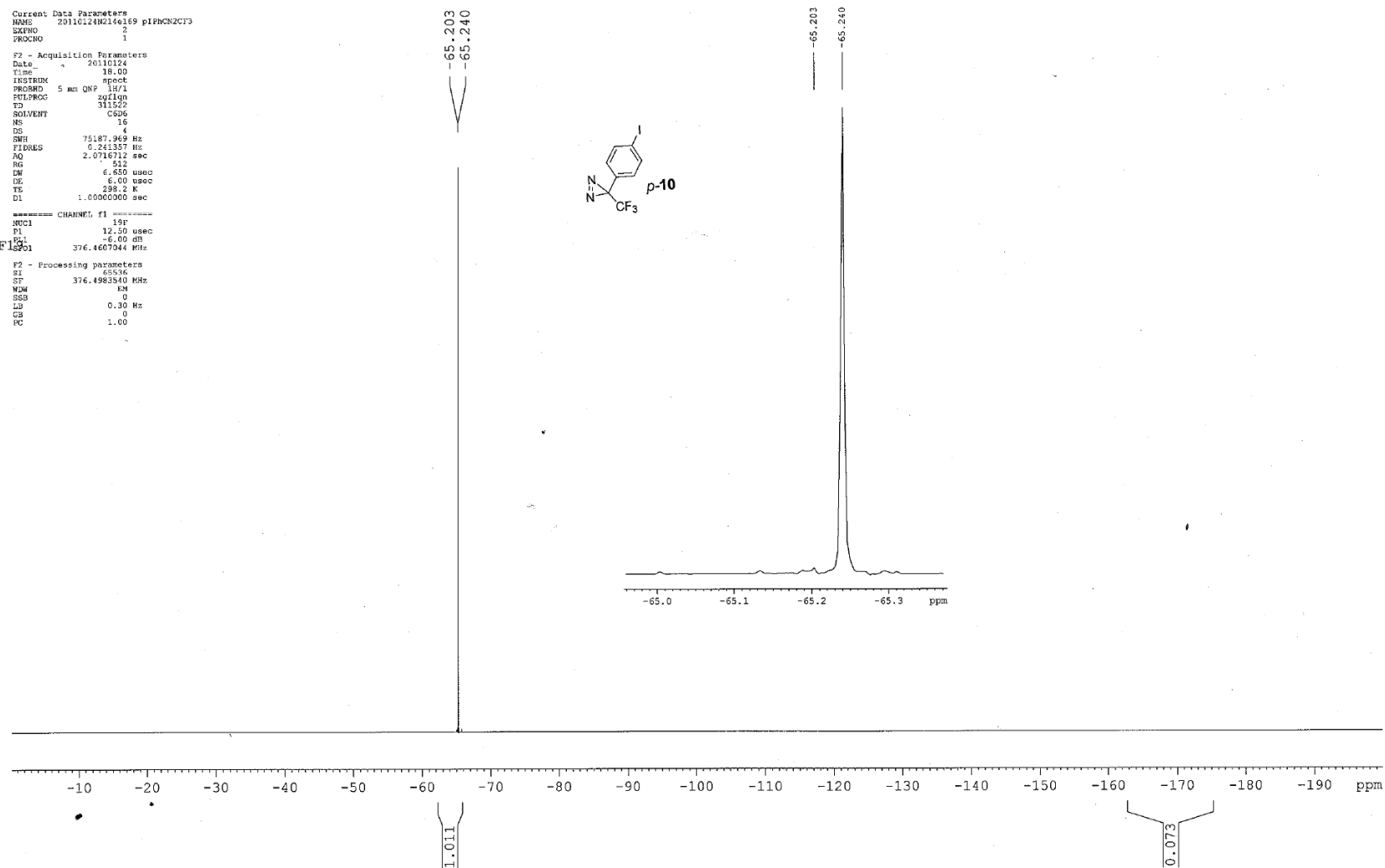
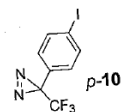
===== CHANNEL f1 =====
 NUC1 13C
 PL 9.50 usec
 PL1 -2.00 dB
 SFO1 100.628888 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCDP2 80.00 usec
 PL2 -2.00 dB
 PL12 14.30 dB
 PL13 14.00 dB
 SFO2 400.1716007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6228270 MHz
 NW 18.00 MHz
 SSB 0
 GB 0
 PC 1.40



Current Data Parameters
NAME 2011012402160169 p1PNCN2C13
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110124
Time 18.00
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 311522
SOLVENT C6D6
NS 16
DS 4
SWH 75187.969 Hz
FIDRES 0.241337 Hz
AQ 2.0716712 sec
RG 512
DM 6.650 usec
DE 6.00 usec
TE 298.2 K
D1 1.0000000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 -6.00 dB
FREQ1 376.4607044 MHz
F2 - Processing parameters
SI 65536
SF 376.4982940 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
 NAME 20110317N242e194mbiaz1+pMeO maj
 EXPNO 3
 PROCNO 1

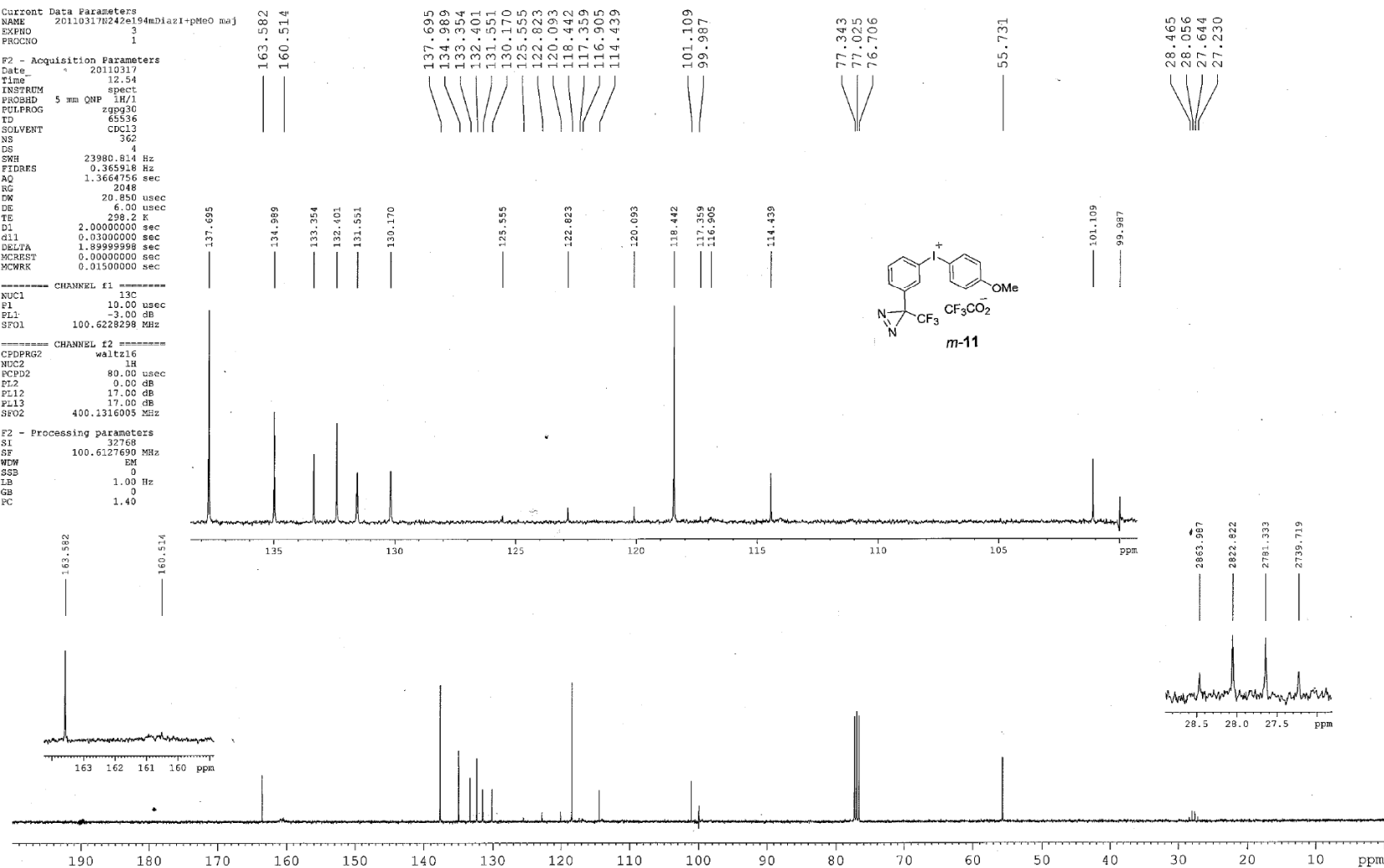
F2 - Acquisition Parameters

Date 20110317
 Time 12.54
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zgpg30
 ID 65536
 SOLVENT CDCl3
 NS 362
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 2048
 DW 20.850 usec
 DE 6.00 usec
 TE 298.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

CHANNEL f1 -----
 NUC1 13C
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PT2 0.00 dB
 PL12 17.00 dB
 PL13 17.00 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



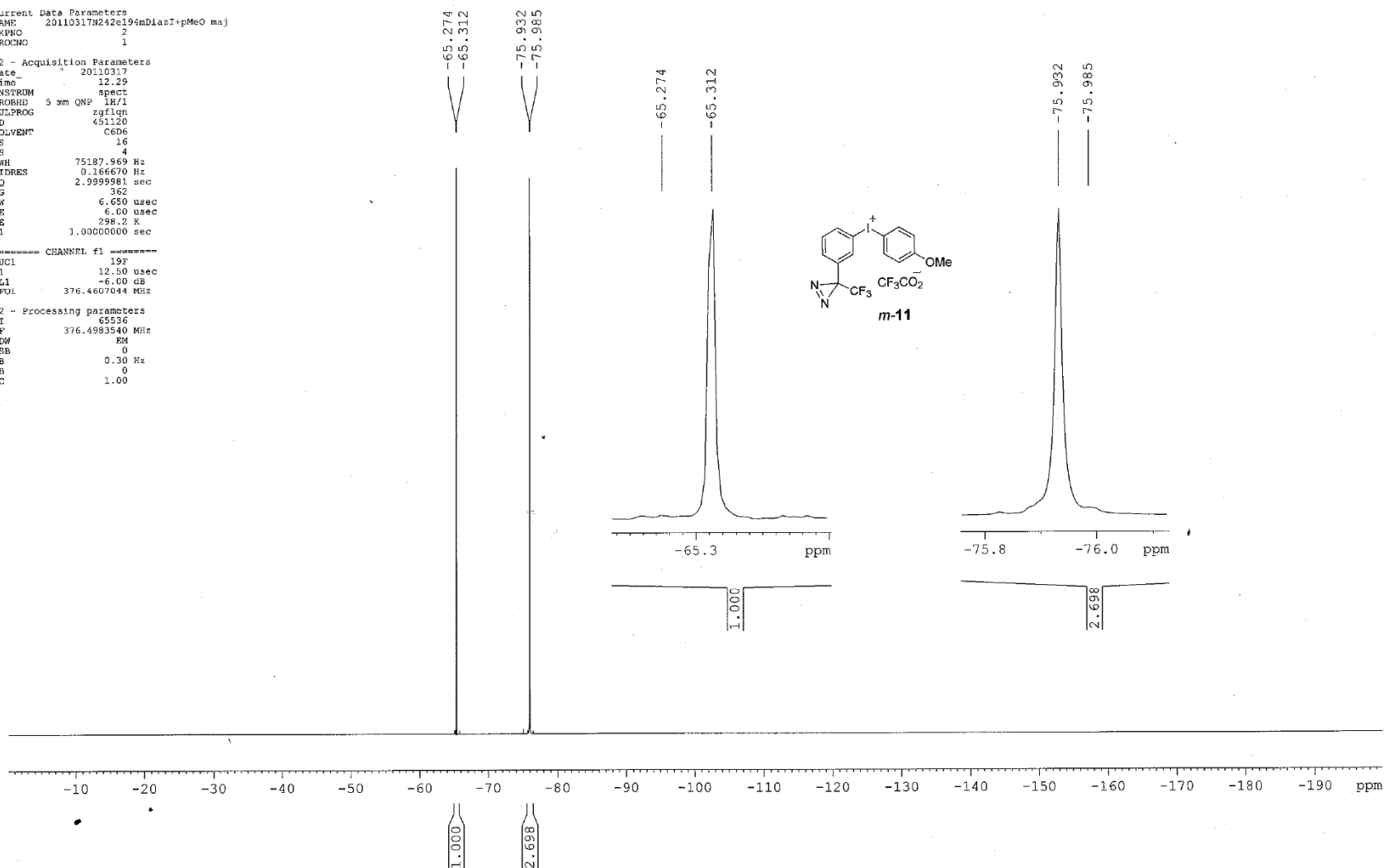
F19

Current Data Parameters
 NAME 20110317N242e19mDiazI+pMeO maj
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110317
 Time 12.29
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 451120
 SOLVENT C6D6
 NS 16
 DS 4
 SWH 75187.969 Hz
 FIDRES 0.166670 Hz
 AQ 2.9999981 sec
 RG 362
 DW 6.650 usec
 DE 6.00 usec
 TE 298.2 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 19F
 P1 12.50 usec
 PL1 -6.00 dB
 SFO1 376.4607044 MHz

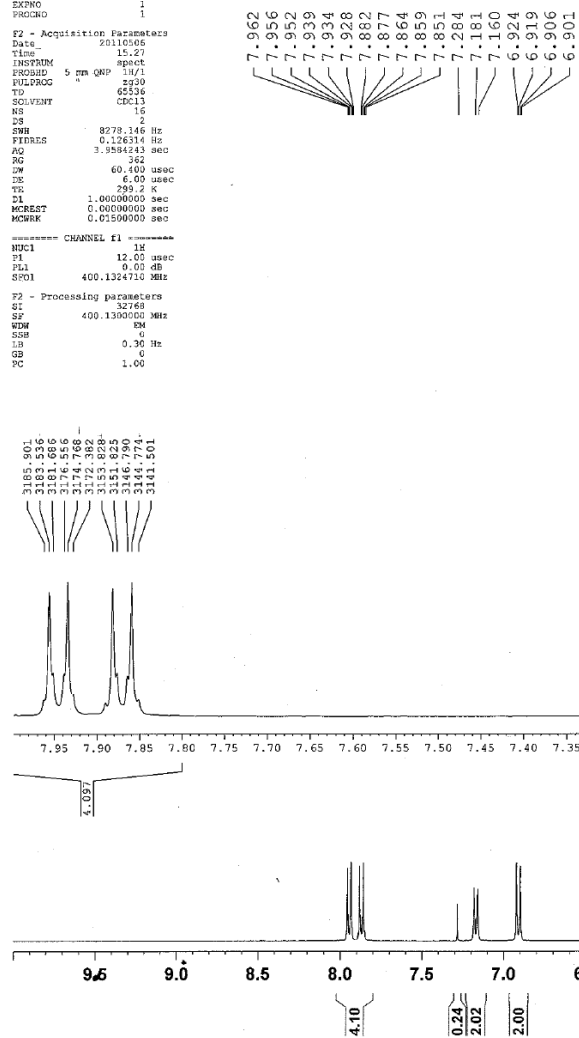
F2 - Processing parameters
 SI 65536
 SF 376.4983540 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME 20110506N276e213 pDiazil-pMeO
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110506
 Time 15.27
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8279.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.00 usec
 TE 299.2 K
 D1 1.00000000 sec
 D11 0.00000000 sec
 MCREST 0.01500000 sec
 MCHRG 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



```

F2 - Acquisition Parameters
Date_      20110517
Time_      19.23
INSTRUM    spect
PROBHD     5 mm QNP 1H/1
PULPROG    zgpg30
TD          65536
SOLVENT    CDCl3
NS          16233
DS          4
SWH         23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         1149.4
DW         20.850 usec
DE         6.60 usec
TE         295.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89399998 sec
MCKRST     0.00000000 sec
MKRKR      0.01500000 sec

```

```

===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        -3.00 dB
SF01      100.6228298 MHz

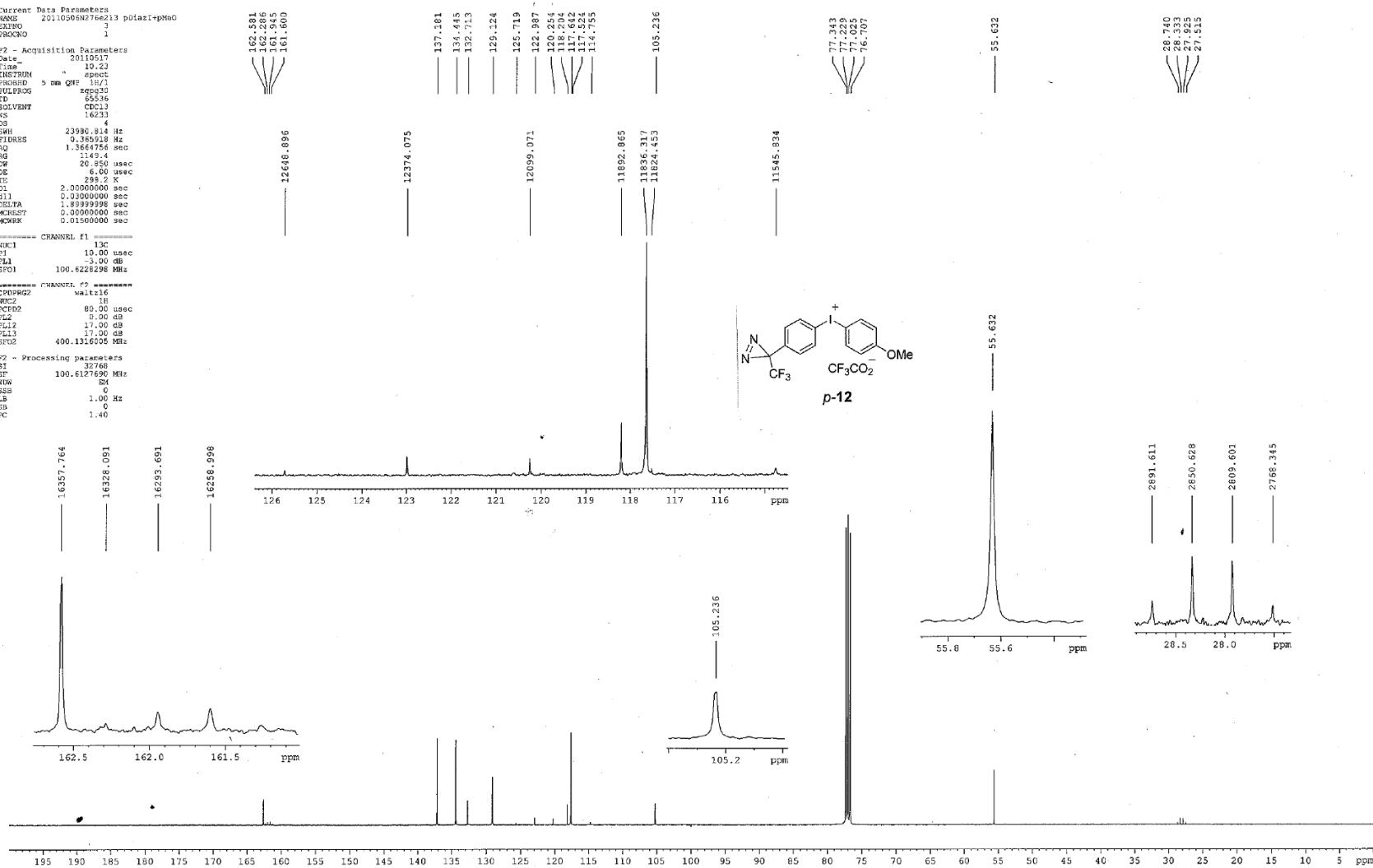
```

```

***** CHANNEL #2 *****
CPDPRG2          waltz16
NUC2              1H
PCPD2            80.00 usec
PL2              0.00 dB
PL12             17.00 dB
PL13             17.00 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
```

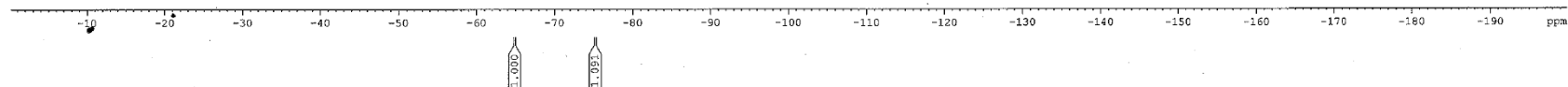
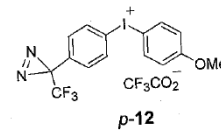
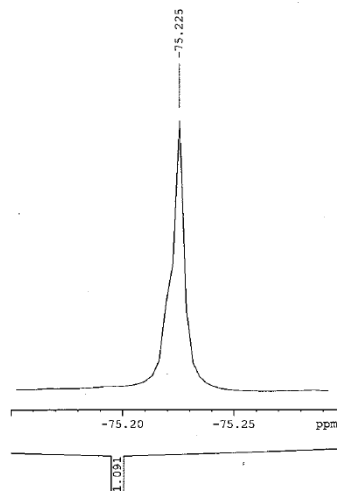
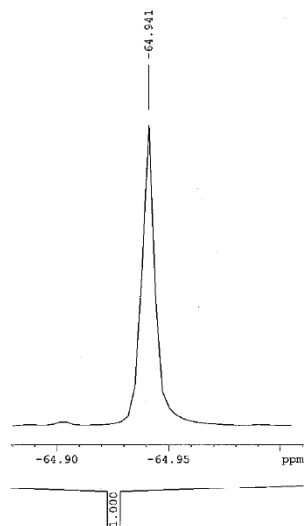


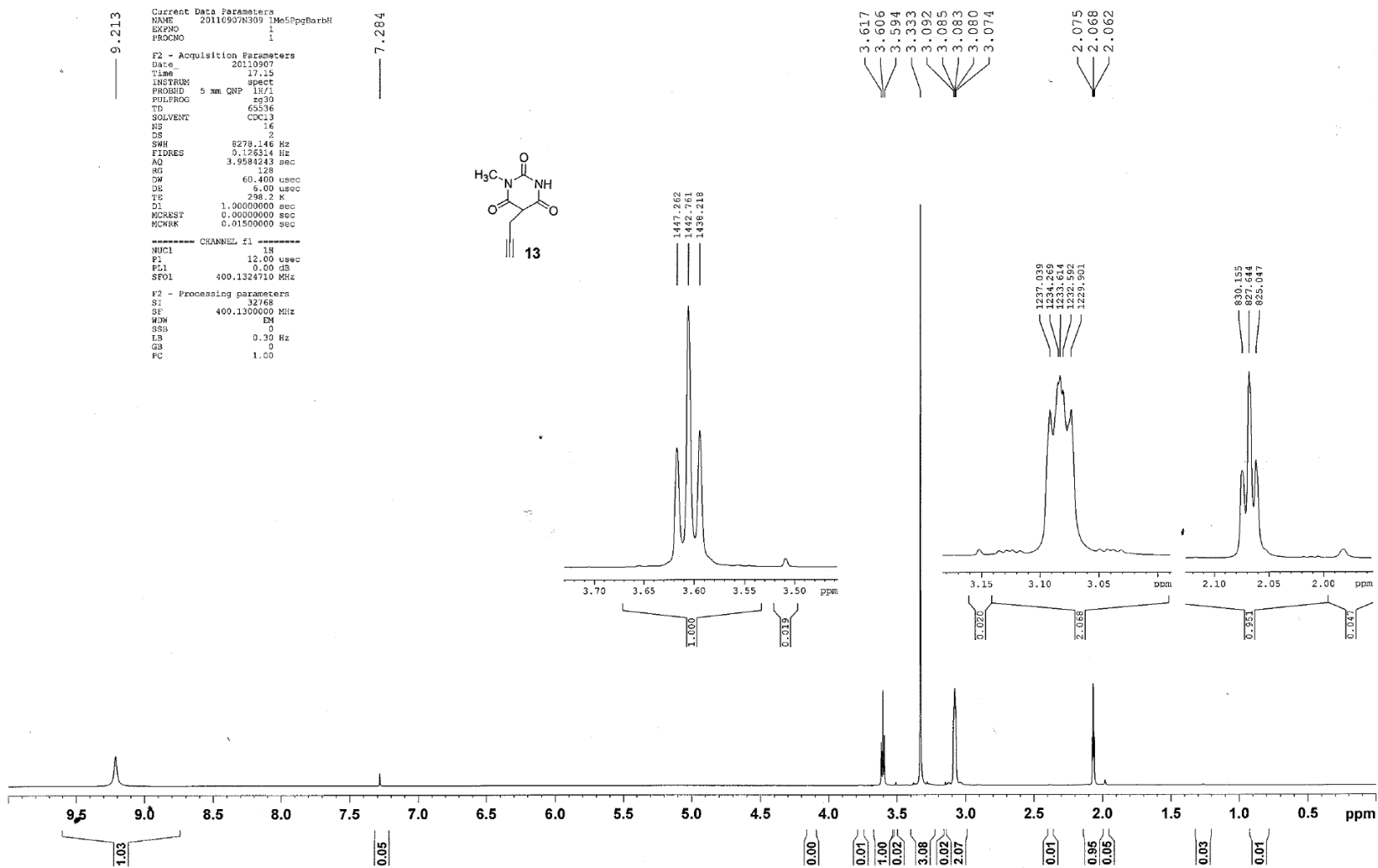
Current Data Parameters
NAME 20110506N276e213 pDiazI-pMeO
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110517
Time_ 10.40
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 300744
SOLVENT C6D6
NS 64
DS 4
SWH 75187.569 Hz
FIDRES 0.256007 Hz
AQ 1.9999976 sec
RG 812.7
DW 6.650 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 12.50 usec
PL1 -6.00 dB
SFO1 376.4607044 MHz

F2 - Processing parameters
SI 65536
SF 376.4963540 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





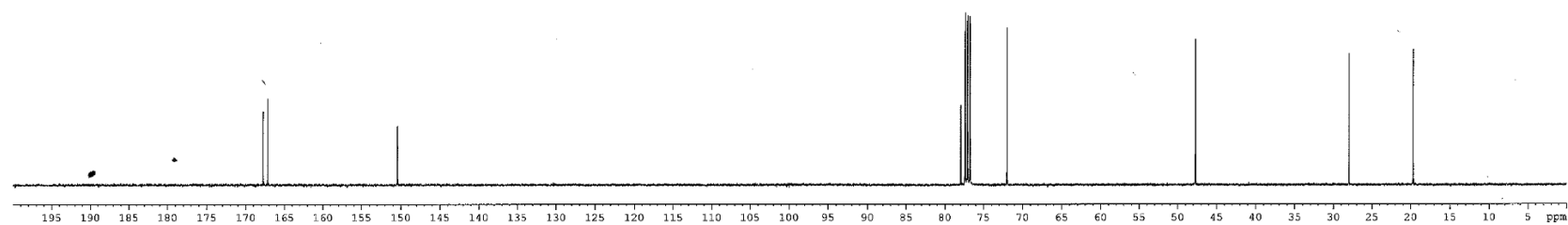
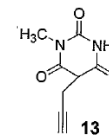
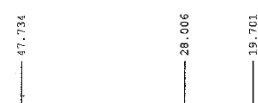
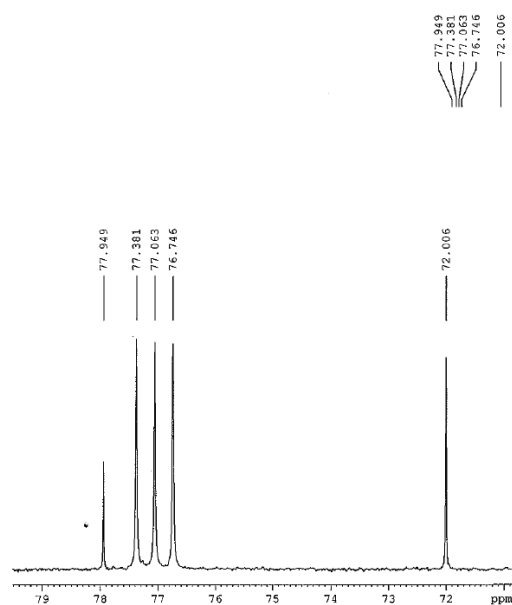
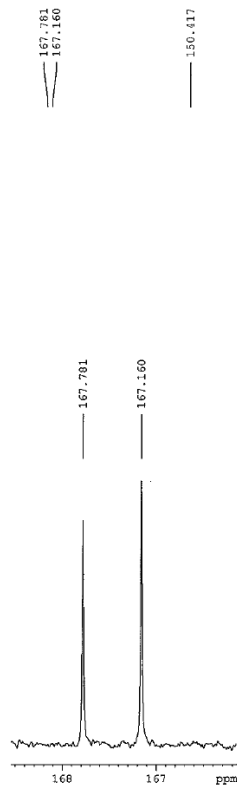
Current Data Parameters
 NAME 20110907N309 1Me5ppggharbH
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110907
 Time 17.31
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 513
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365818 Hz
 AQ 1.3664756 sec
 RG 724.1
 DR 20.850 usec
 DE 6.00 usec
 TE 299.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 NUCREST 0.00000000 sec
 NUCR1 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 100.628298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 300.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

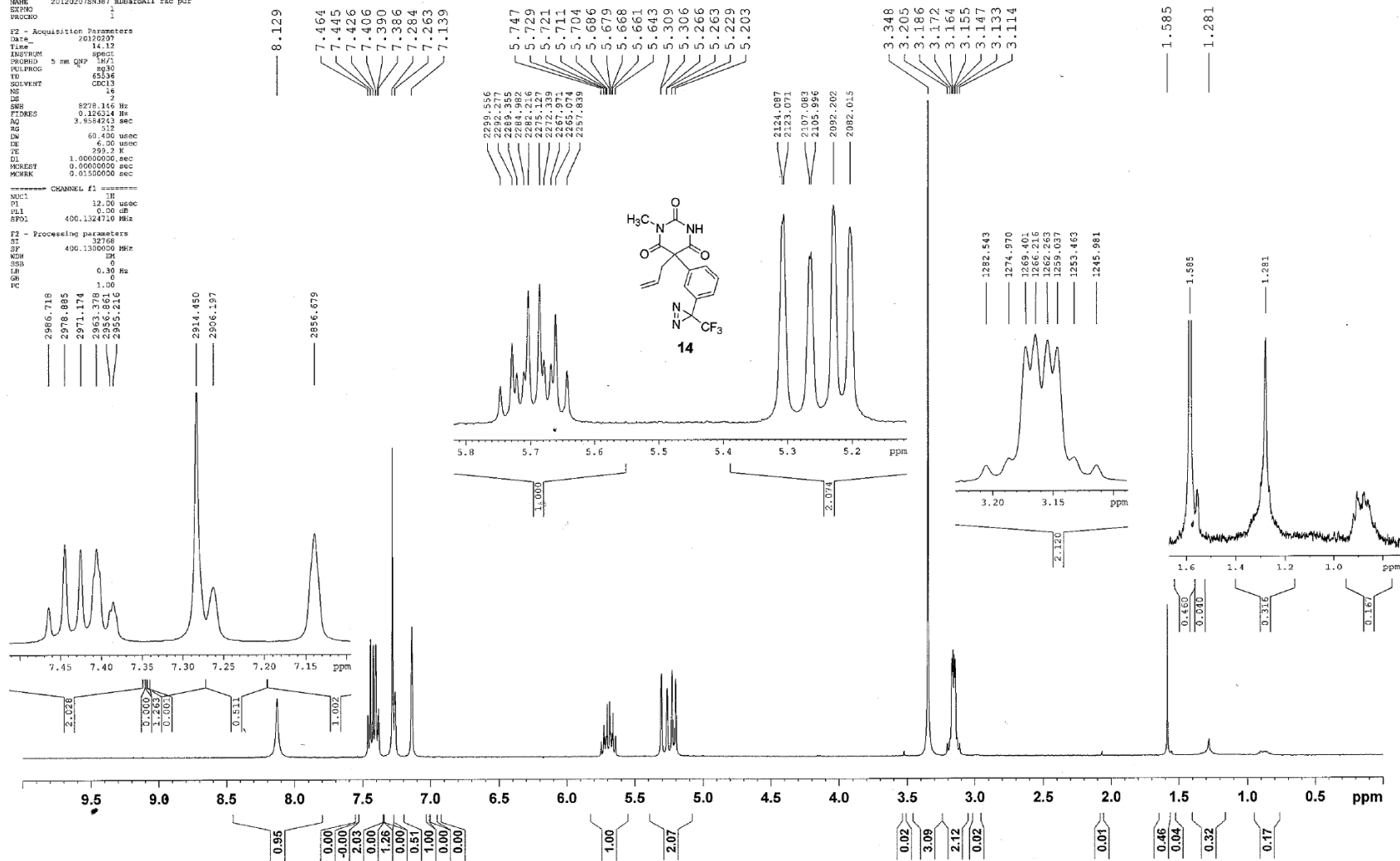


Current Data Parameters
NAME: 20120707SCH07 aDBarball rac pur
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20120707
Time: 14.12
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 4
SWH: 5278.146 Hz
FIDRES: 0.126214 Hz
AQ: 3.538421 sec
RG: 512
RW: 60.430 usec
DE: 6.00 usec
TE: 299.2 K
D1: 1.00000000 sec
MCOREST: 0.00000000 sec
MCORE: 0.01500000 sec

CHANNEL f1
NUC1: 1H
P1: 12.00 usec
PL1: 0.00 dB
SFO1: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1300000 MHz
WDW: EM
SSB: 0
GB: 0
PC: 1.00



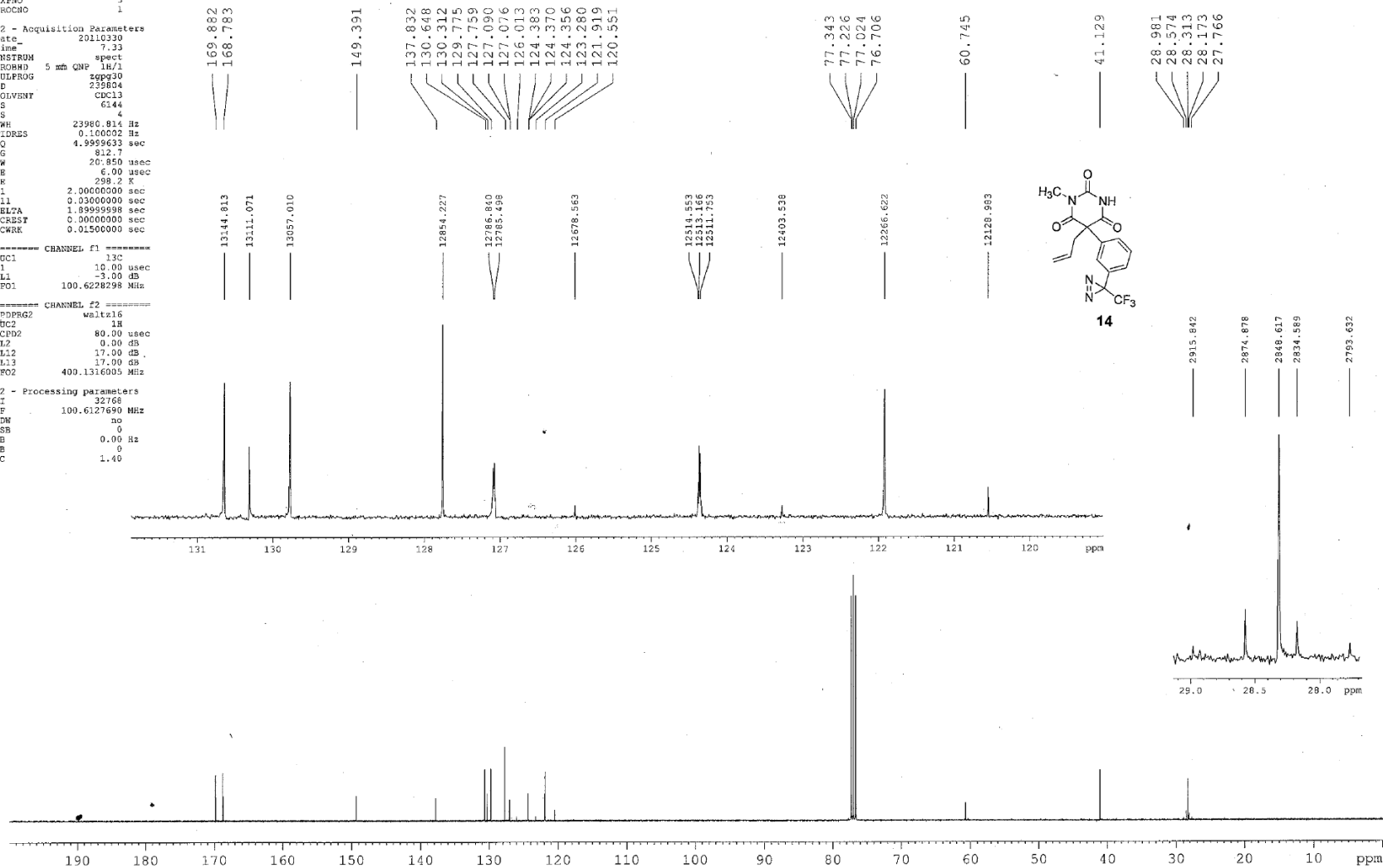
Current Data Parameters
 NAME 20110329M1508arbdiaz repur
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110330
 Time_ 7.33
 INSTRUM spect
 PROBRD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 23904
 SOLVENT CDCl3
 NS 6144
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.100002 Hz
 AQ 4.9999633 sec
 RG 812.7
 DW 20.850 usec
 DE 6.00 usec
 TE 298.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 MCREST 0.0000000 sec
 NCMR 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

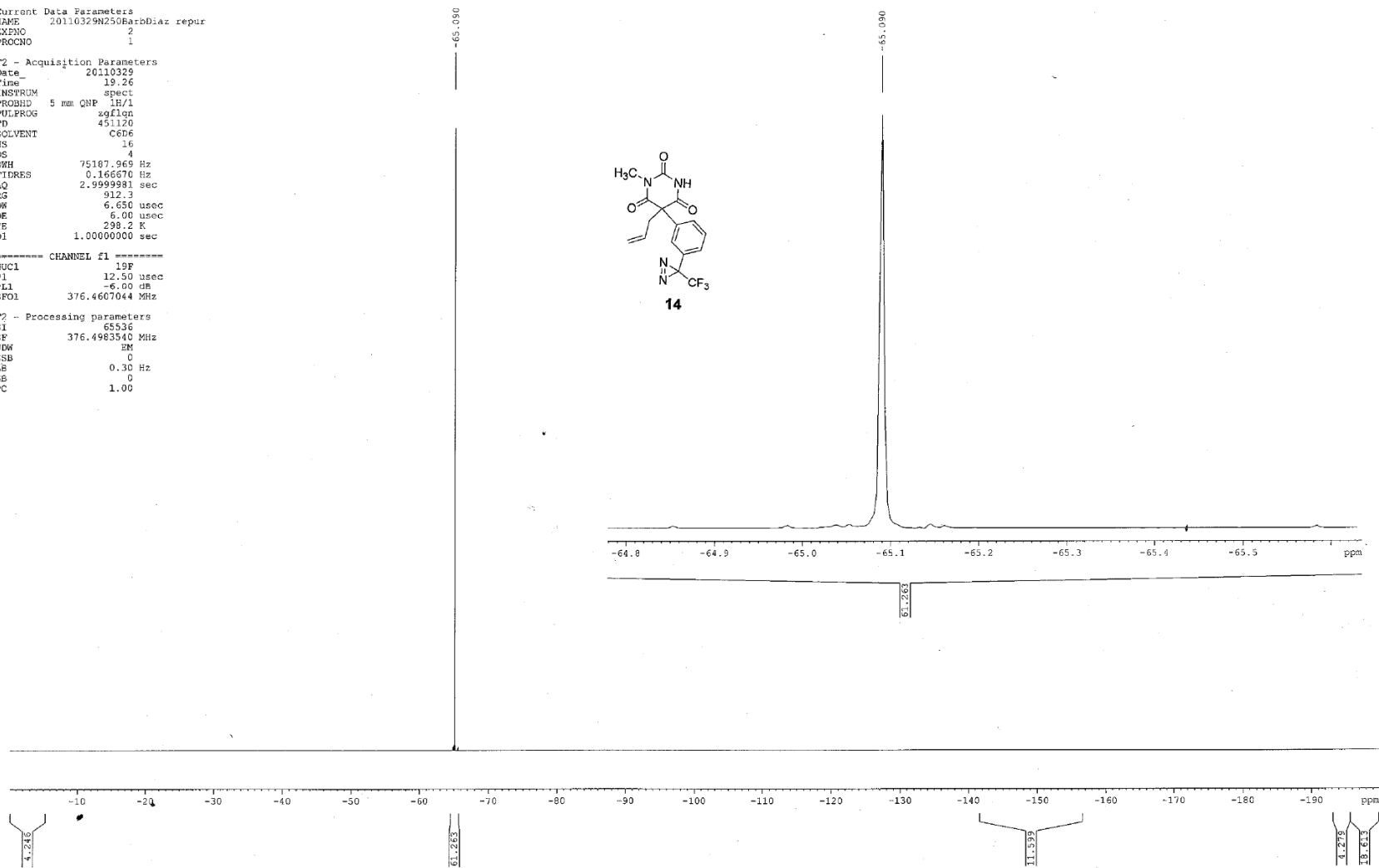
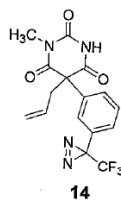


Current Data Parameters
NAME 20110329N250BarbDiaz repur
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20110329
Time 19.26
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 451120
SOLVENT C6D6
NS 16
DS 4
SWH 75107.969 Hz
FIDRES 0.166670 Hz
AQ 2.9999981 sec
RG 912.3
DW 6.650 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 -6.00 dB
SFO1 376.4607044 MHz

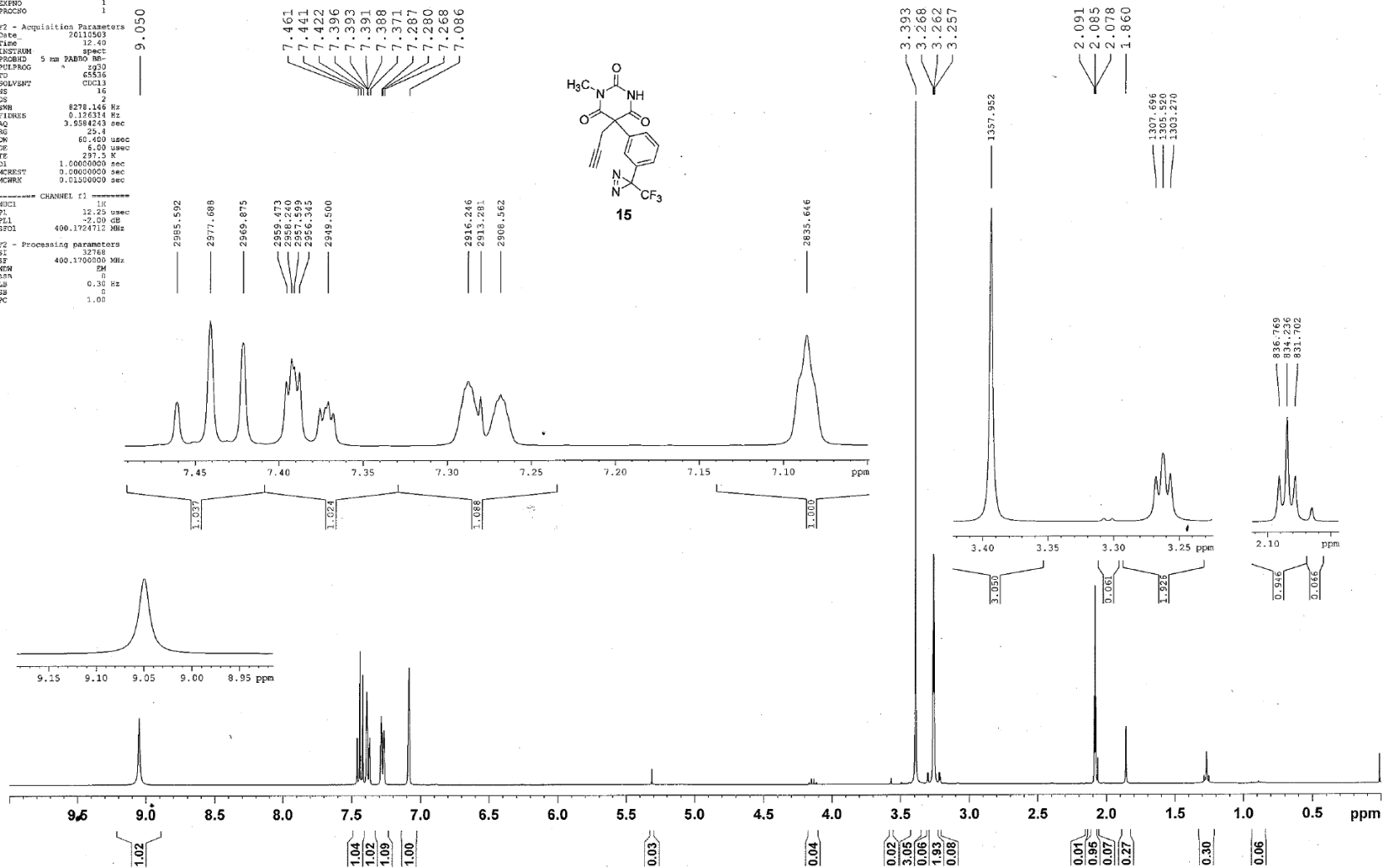
F2 - Processing parameters
SI 65536
SF 376.4983540 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 20110503M0271a209 PpgBarb-m01az
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110503
Time 12.40
INSTRUM spect
PROBHD 5 mm BBOBO MM-
PULPROG zgpg30
TD 65536
SOLVENT CCl₄
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.538423 sec
RG 25.4
DM 65.400 usec
DE 6.00 usec
TE 297.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWEX 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ¹H
P1 12.25 usec
PL1 -2.00 dB
SFO1 400.1724713 MHz
F2 - Processing parameters
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



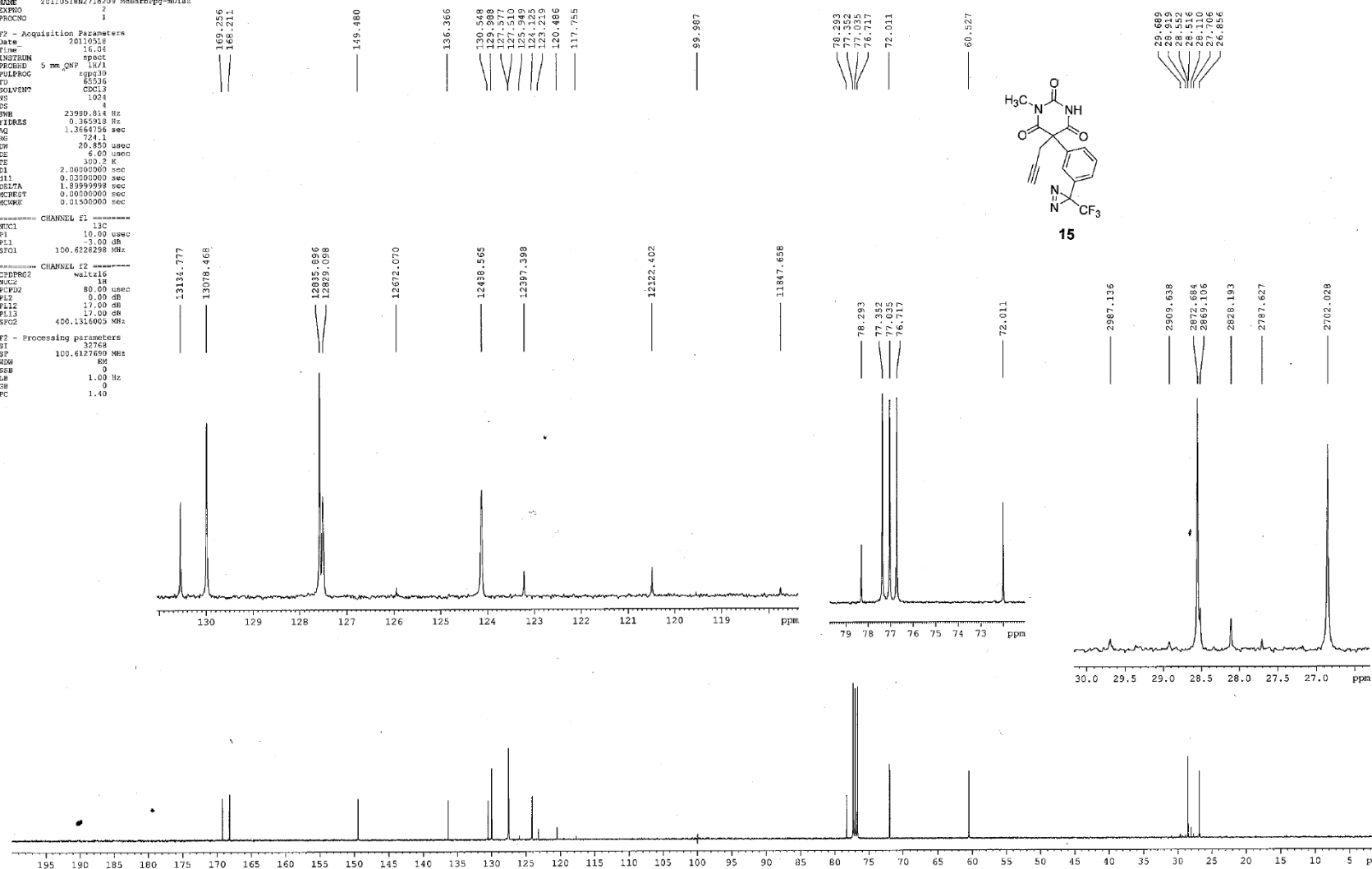
Current Data Parameters
 NAME 20110518N271a209 MeBarbPpg-mDias
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110518
 Time 16.04
 INSTRUM spect
 PROCNO 5 mm_QNP 1H/1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.3664756 Hz
 AQ 1.3664756 sec
 RG 324.1
 DW 20.850 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCHST 0.00000000 sec
 MCHW 0.01500000 sec

CHANNEL F1 -----
 NUC1 13C
 P1 10.00 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

CHANNEL F2 -----
 CDEPRG2 waltz16
 NUC2 1H
 FWD2 80.00 usec
 PL2 0.00 dB
 PL12 17.00 dB
 PL13 17.00 dB
 SFO2 400.1314005 MHz

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



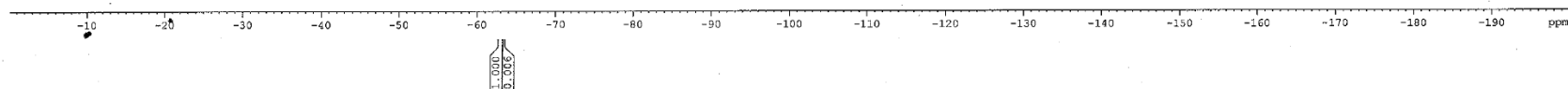
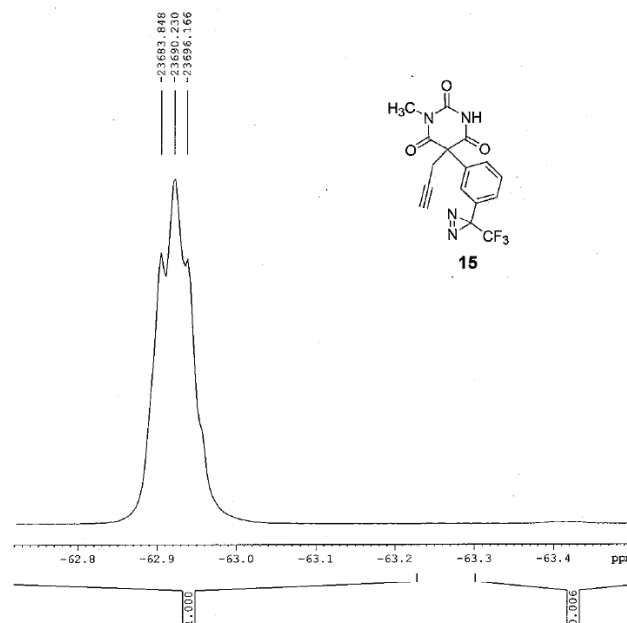
Current Data Parameters
NAME 201105180271e209 MeBarbPp-mDiaz
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110519
Time_ 12.03
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 300744
SOLVENT CDCl3
NS 16
DS 4
SWH 75187.969 Hz
FIDRES 0.250007 Hz
AQ 1.9999976 sec
RG 645.1
DW 8.650 usec
DE 5.00 usec
TE 300.2 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 12.00 usec
PL1 -6.00 dB
SFO1 376.4607044 MHz

F2 - Processing parameters
SI 65536
SF 376.4983540 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

-62.906
-62.922
-62.938



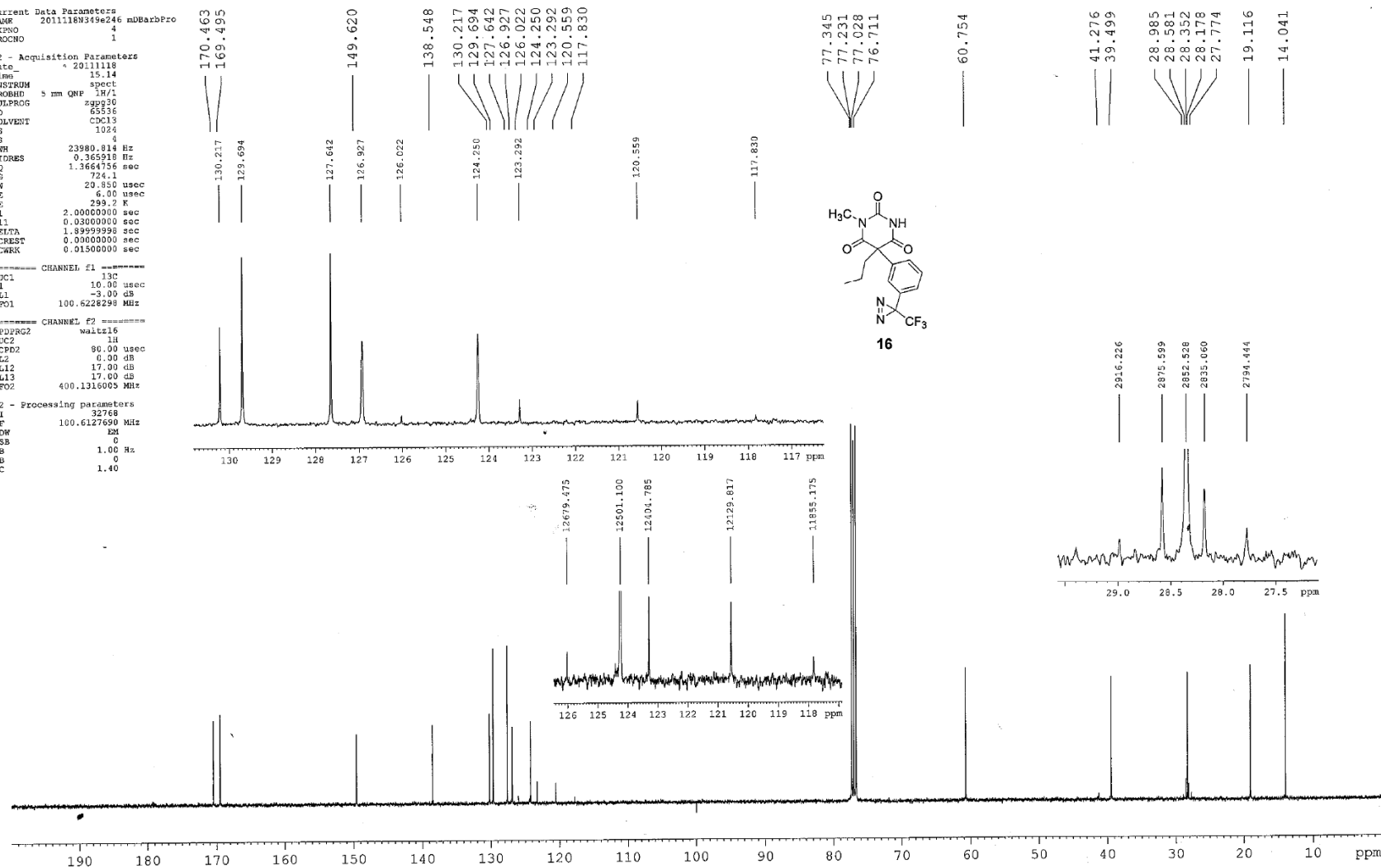
Current Data Parameters
NAME 201118N349246 mBarbPro
EXNO 4
PROCNO 1

F2 - Acquisition Parameters
Date 20111118
Time 15.14
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 724.1
LW 20.850 usec
DE 6.00 usec
TE 299.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.09000000 sec
MCWRR 0.01500000 sec

CHANNEL F1
NUC1 13C
P1 10.00 usec
PL1 -3.00 dB
SFO1 100.6228299 MHz

CHANNEL F2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 17.00 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 2011118349e246 mDBarbPro
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20111118
Time 14.57
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 451120
SOLVENT C6D6
NS 16
DS 4
SWH 75187.969 Hz
FIDRES 0.166670 Hz
AQ 2.9999981 sec
RG 645.1
DW 6.650 usec
DE 6.00 usec
TE 299.2 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 19F
P1 12.50 usec
PL1 -6.00 dB
SFO1 376.4607044 MHz

F2 - Processing parameters
SI 65536
SF 376.4983540 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

