

## Palladium-catalyzed cross-coupling in the synthesis of pyridinyl boxazomycin C analogues

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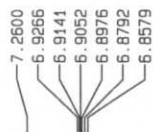
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g.rewcastle@auckland.ac.nz

## Contents

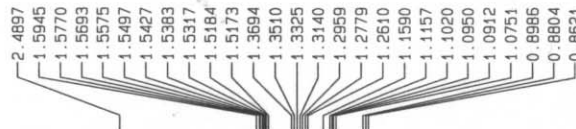
Spectral data for compound 4	S2-S4
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Spectral data for compound 8	S8-S10
Spectral data for compound 10	S11-S13
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S2

CR 1\_083\_1



3.7412



## Current Data Parameters

NAME cr1030721  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20030721  
Time 8.29  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT cdc13  
NS 16  
DS 2  
SWH 8389.262 Hz  
FIDRES 0.128010 Hz  
AQ 3.9059956 sec  
RG 181  
DW 59.600 usec  
DE 10.00 usec  
TE 300.0 K  
D1 2.00000000 sec

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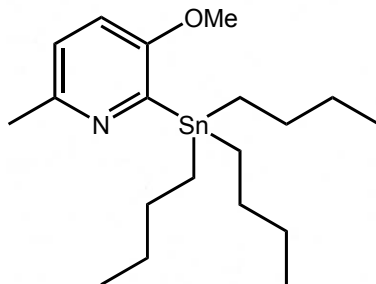
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## 1D NMR plot parameters

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CY 12.00 cm  
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F1 4001.30 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.50000 ppm/cm  
HZCM 200.06497 Hz/cm

# <sup>1</sup>H NMR spectrum of Compound 4



1.3187

1.9706

1.9840

4.8282

4.4912

0.4813

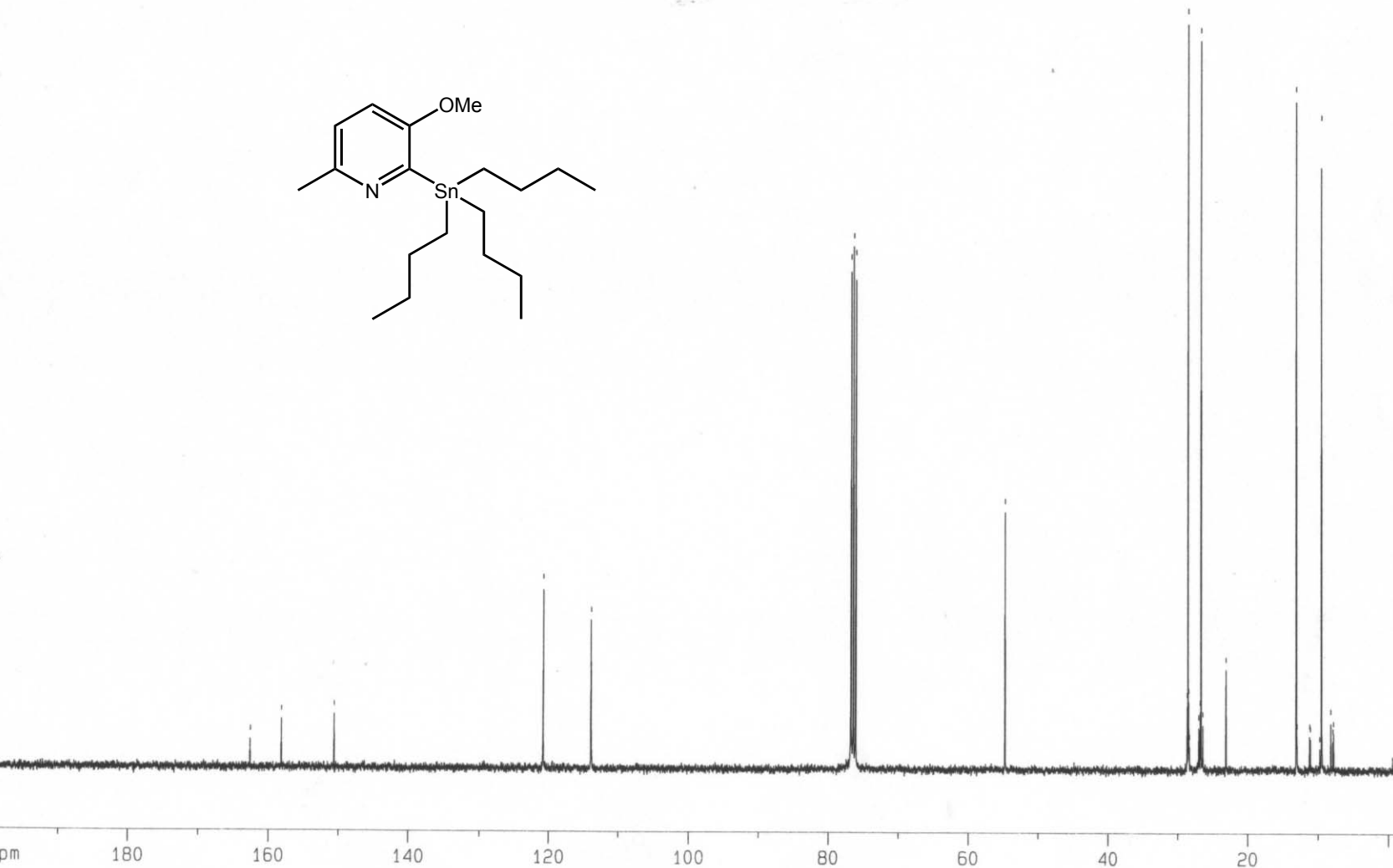
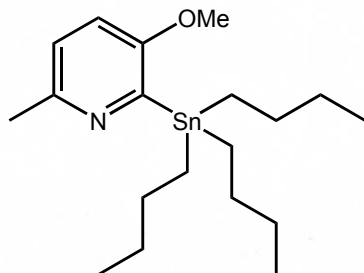
3.3623

0.4348

6.1816

S3

# <sup>13</sup>C NMR spectrum of Compound 4



Current Data Parameters  
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EXPNO 23  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050214  
Time 12.34  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg  
TD 65536  
SOLVENT dmsd  
NS 2200  
DS 0  
SWH 23980.814 Hz  
FIDRES 0.365918 Hz  
AQ 1.3664756 sec  
RG 1625.5  
DW 20.850 usec  
DE 6.50 usec  
TE 300.0 K  
D1 0.75000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

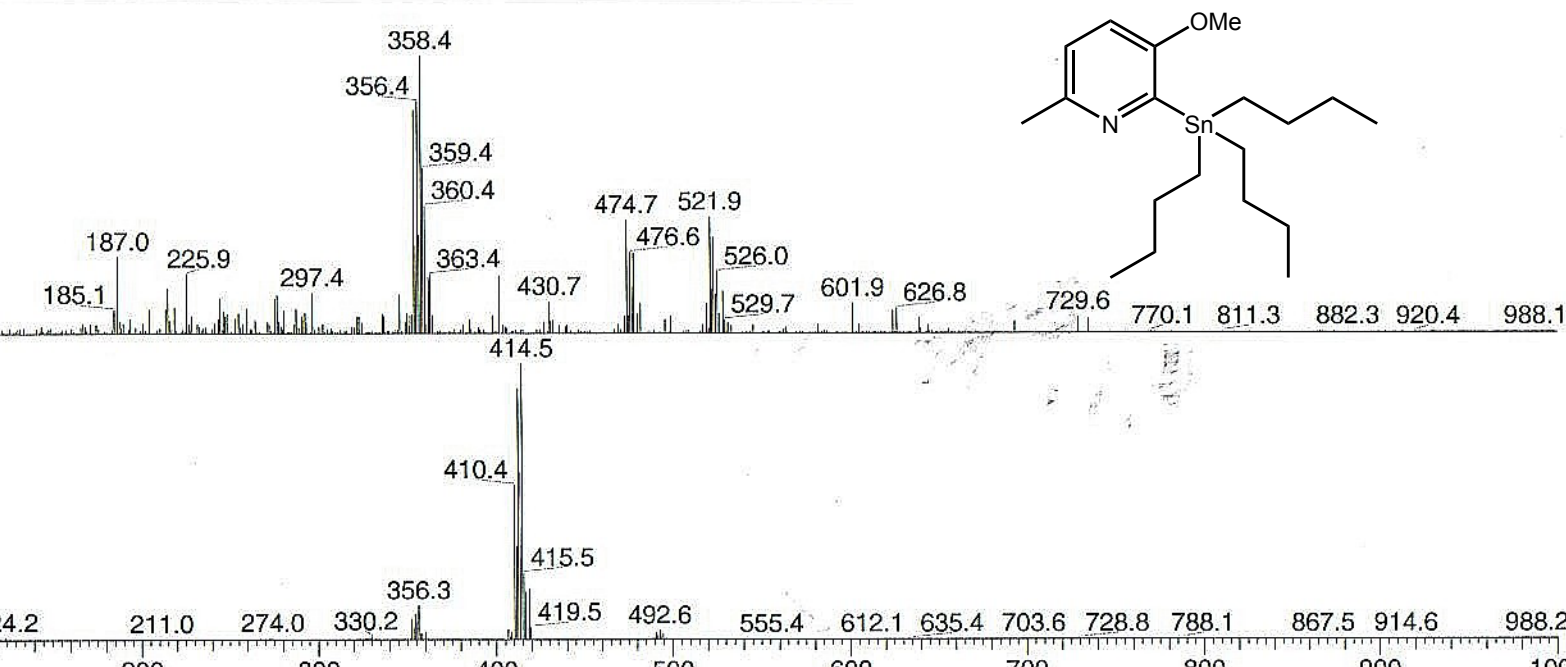
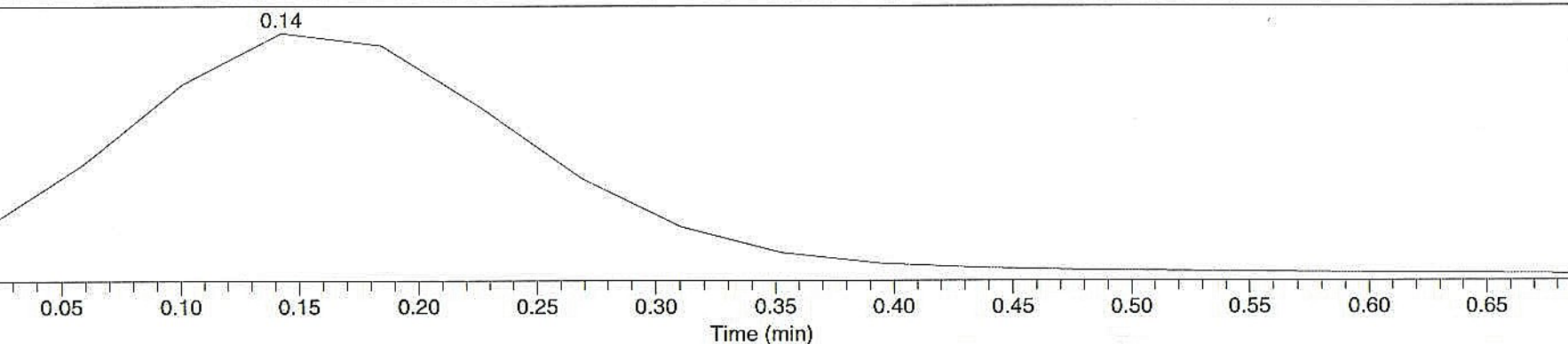
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NUC1 13C  
P1 6.26 usec  
PL1 -1.00 dB  
SF01 100.6239378 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 100.00 usec  
PL2 -3.00 dB  
PL12 17.50 dB  
PL13 19.00 dB  
SF02 400.1318006 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6128242 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 22.00 cm  
CY 11.87 cm  
F1P 200.000 ppm  
F1 20122.56 Hz  
F2P -1.000 ppm  
F2 -100.61 Hz  
PPMCM 9.13636 ppm/  
HZCM 919.23529 Hz/c

# Mass spectrum of Compound 4

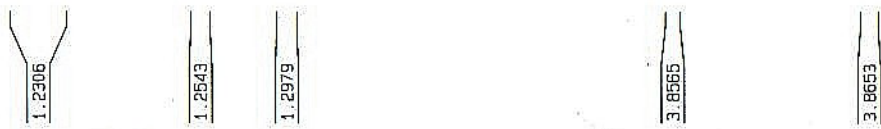
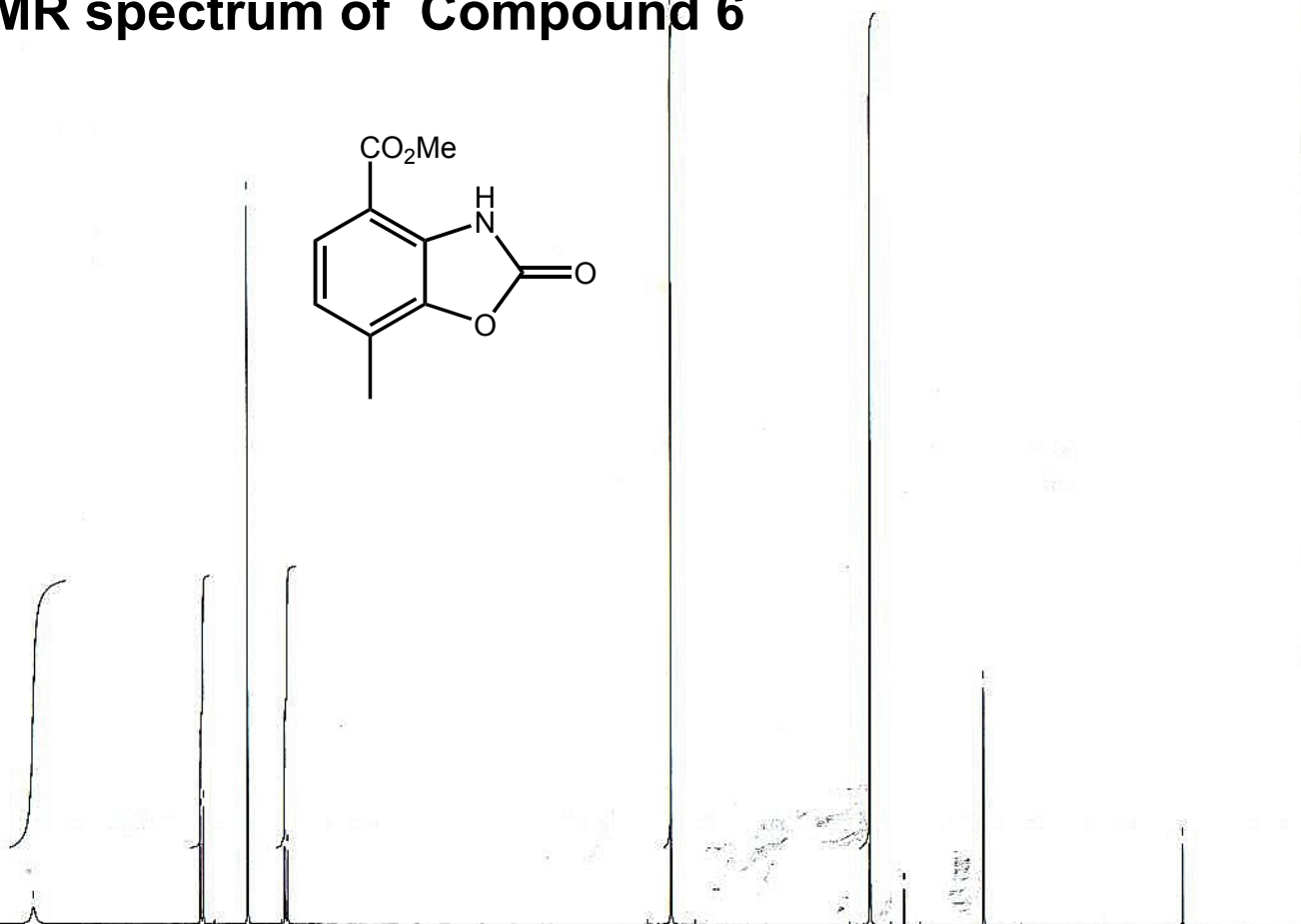
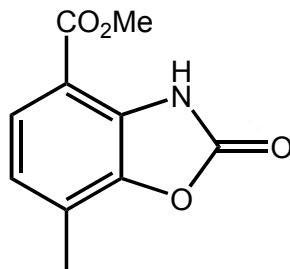


Vial71#7-10 RT: 0.12-0.16  
2 0.02 , 0.54-0.60 NL: 3.90  
{0,0} - c APCI corona sid=50  
det=1400.00 Full ms [  
100.00-1000.00]

Vial71#6-10 RT: 0.10-0.18  
3 0.02 , 0.54-0.60 NL: 8.43  
{0,1} + c APCI corona sid=50  
det=1400.00 Full ms [  
100.00-1000.00]

S5

# <sup>1</sup>H NMR spectrum of Compound 6



Current Data Parameters  
NAME Apr11-2003-ACSRC  
EXPNO 8  
PROCNO 1

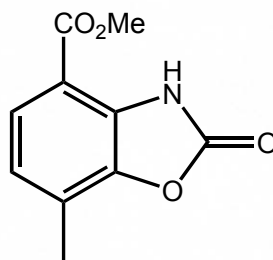
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Time 13.43  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT COC13  
NS 32  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 455.1  
DW 60.400 usec  
DE 10.00 usec  
TE 300.0 K  
D1 2.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 10.20 usec  
PL1 -3.00 dB  
SF01 400.1336412 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300996 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 22.00 cm  
CY 12.00 cm  
F1P 12.000 ppm  
F1 4801.56 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.59091 ppm/cm  
HZCM 235.44048 Hz/cm

S6

 **$^{13}\text{C}$  NMR spectrum of Compound 6**

Current Data Parameters  
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EXPNO 2  
PROCNO 1

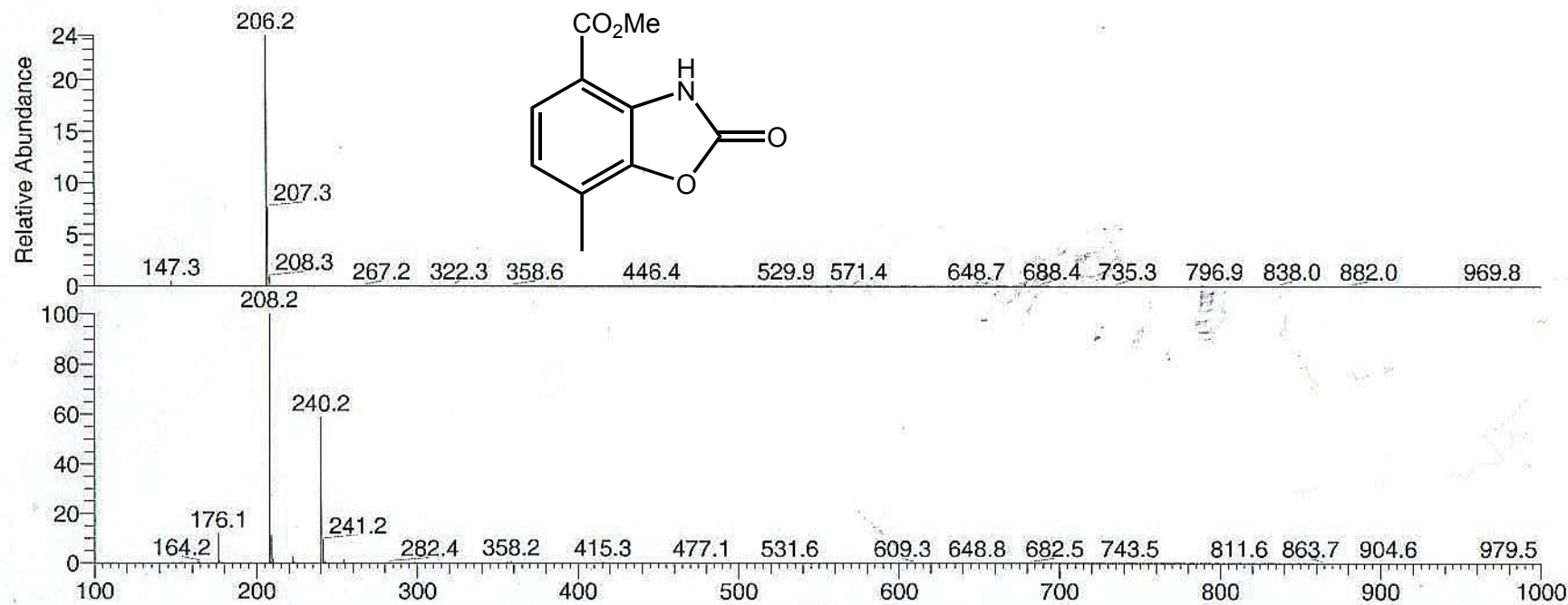
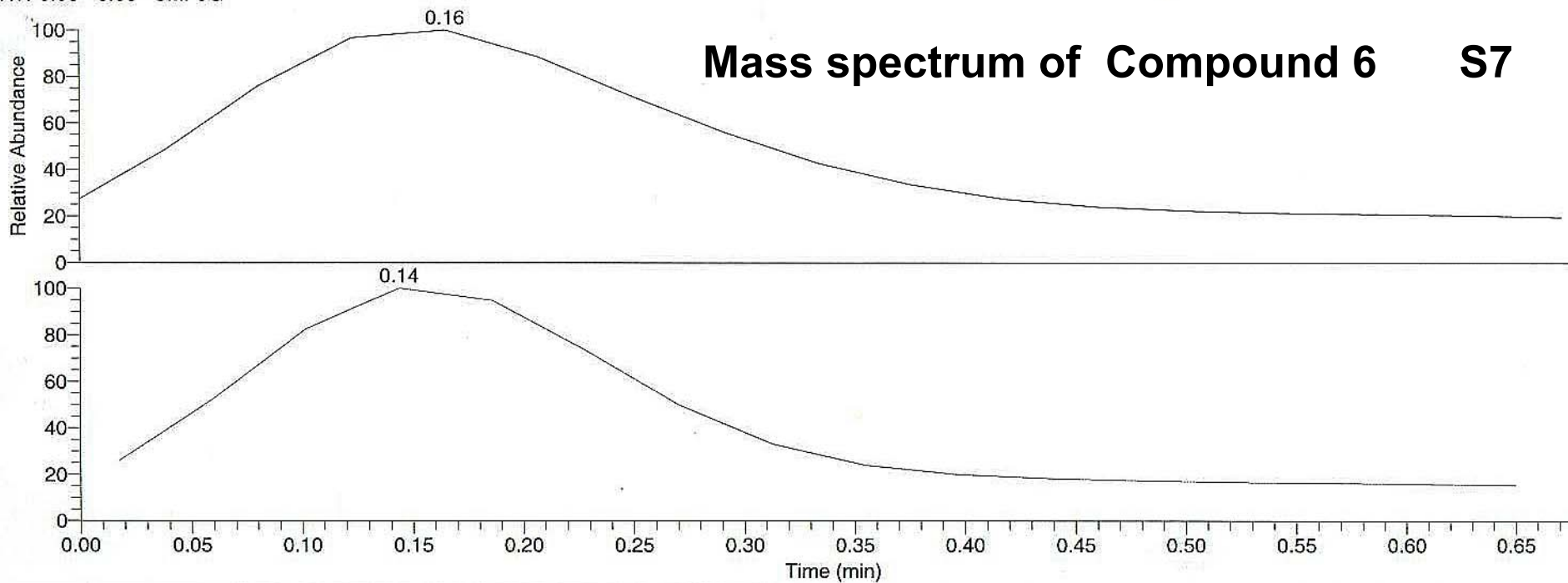
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Date\_ 20050208  
Time 14.57  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg  
TD 65536  
SOLVENT dms  
NS 1000  
DS 0  
SWH 23980.814 Hz  
FIDRES 0.365918 Hz  
AQ 1.3664756 sec  
RG 9195.2  
DM 20.850 usec  
DE 6.50 usec  
TE 300.0 K  
D1 0.75000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1  $^{13}\text{C}$   
P1 6.26 usec  
PL1 -1.00 dB  
SF01 100.6239378 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2  $^1\text{H}$   
PCPD2 100.00 usec  
PL2 -3.00 dB  
PL12 17.50 dB  
PL13 19.00 dB  
SF02 400.1318006 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127693 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 22.00 cm  
CY 15.95 cm  
F1P 200.000 ppm  
F1 20122.55 Hz  
F2P -1.000 ppm  
F2 -100.61 Hz  
PPMCH 9.13636 ppm  
HZCM 919.23480 Hz/



Vial09\_05011  
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sid=50.00 de  
100.00-1000.

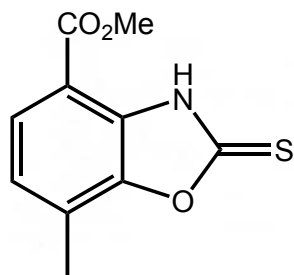
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1.87E7 F: {0,  
sid=50.00 de  
100.00-1000.

S8

13.663

7.697  
7.676  
7.207  
7.206  
7.187  
7.186

4.074  
3.909  
3.892  
3.851  
3.704  
3.450  
3.433  
3.302  
2.602  
2.511  
2.507  
2.502  
2.497  
2.493  
2.443  
2.280  
2.085  
1.332  
1.074  
1.057  
1.039

<sup>1</sup>H NMR spectrum of Compound 8

Current Data Parameters  
NAME Jun17-2003-ACSRC  
EXPNO 38  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20030617  
Time 15.26  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 287.4  
DW 60.400 usec  
DE 10.00 usec  
TE 300.0 K  
D1 2.00000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
NUC1 1H  
P1 9.50 usec  
PL1 -3.00 dB  
SF01 400.1336412 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300018 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 22.00 cm  
CY 12.00 cm  
F1P 16.000 ppm  
F1 6402.08 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.77273 ppm/cm  
HZCM 309.19138 Hz/cm

0.8777

0.8938

2.6540

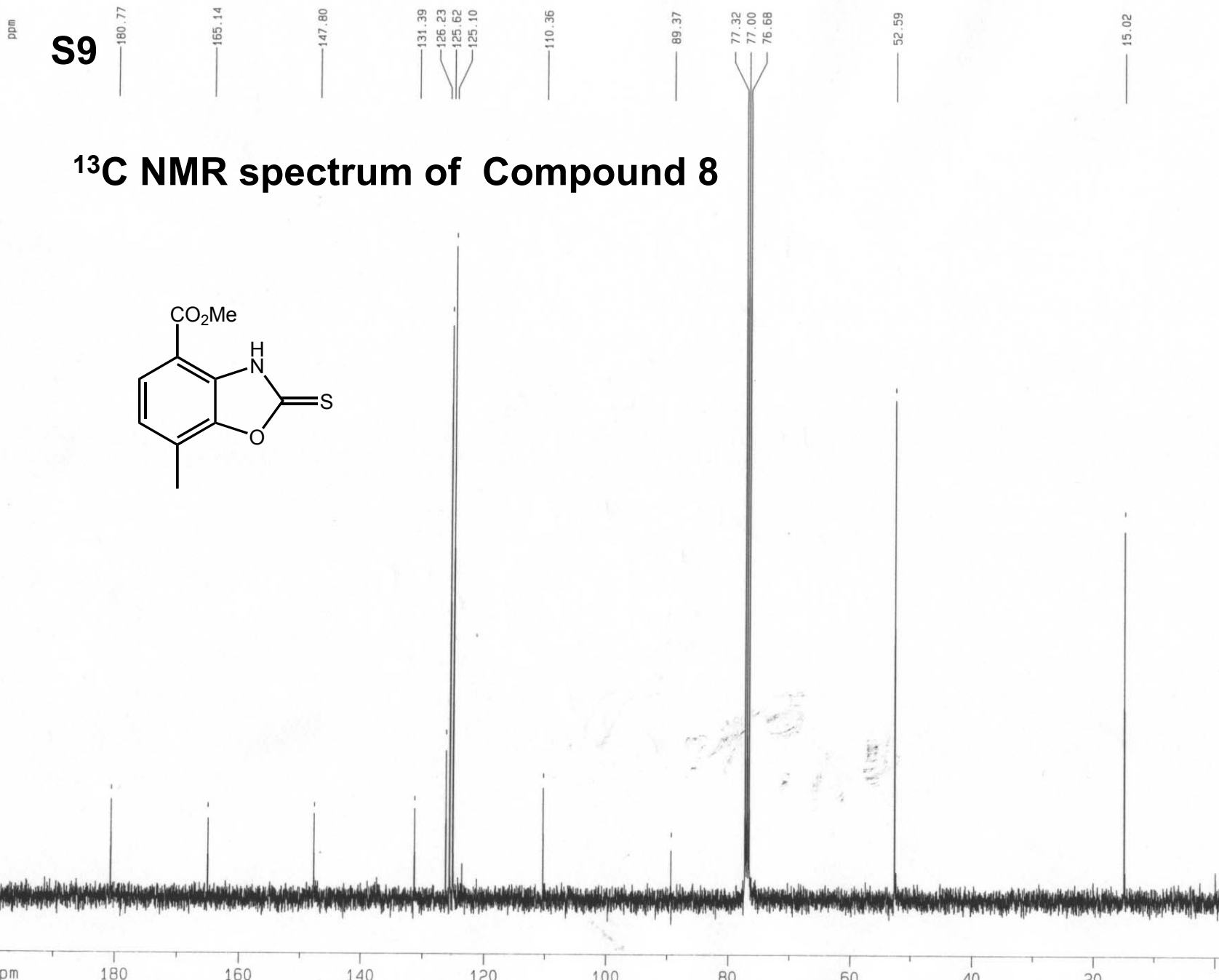
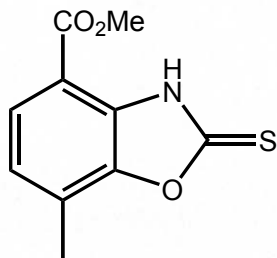
2.6512

0.0764

0.7661



S9

<sup>13</sup>C NMR spectrum of Compound 8

Current Data Parameters  
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 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050204  
 Time 15.24  
 INSTRUM spect  
 PROBHD 5 mm BBO BB-1H  
 PULPROG zgpg  
 TD 65536  
 SOLVENT dms0  
 NS 1000  
 DS 0  
 SWH 25906.736 Hz  
 FIDRES 0.395305 Hz  
 AQ 1.2648948 sec  
 RG 9195.2  
 DW 19.300 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 0.75000000 sec  
 d11 0.03000000 sec  
 d12 0.00002000 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 6.26 usec  
 PL1 -1.00 dB  
 SF01 100.6249440 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 100.00 usec  
 PL2 -3.00 dB  
 PL12 17.50 dB  
 PL13 19.00 dB  
 SF02 400.1318006 MHz

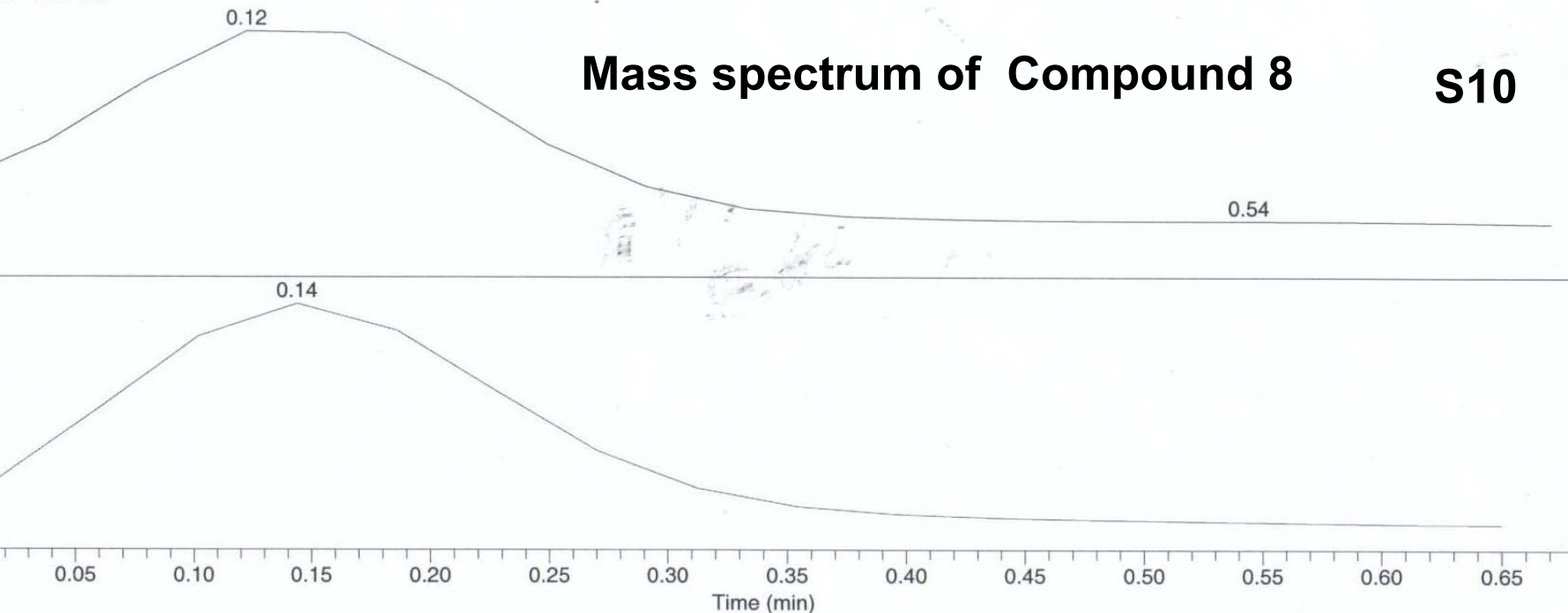
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 SF 100.6127702 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 22.00 cm  
 CY 20.17 cm  
 F1P 200.000 ppm  
 F1 20122.55 Hz  
 F2P -1.000 ppm  
 F2 -100.61 Hz  
 PPMCM 9.13636 ppm/  
 HZCM 919.23480 Hz/c

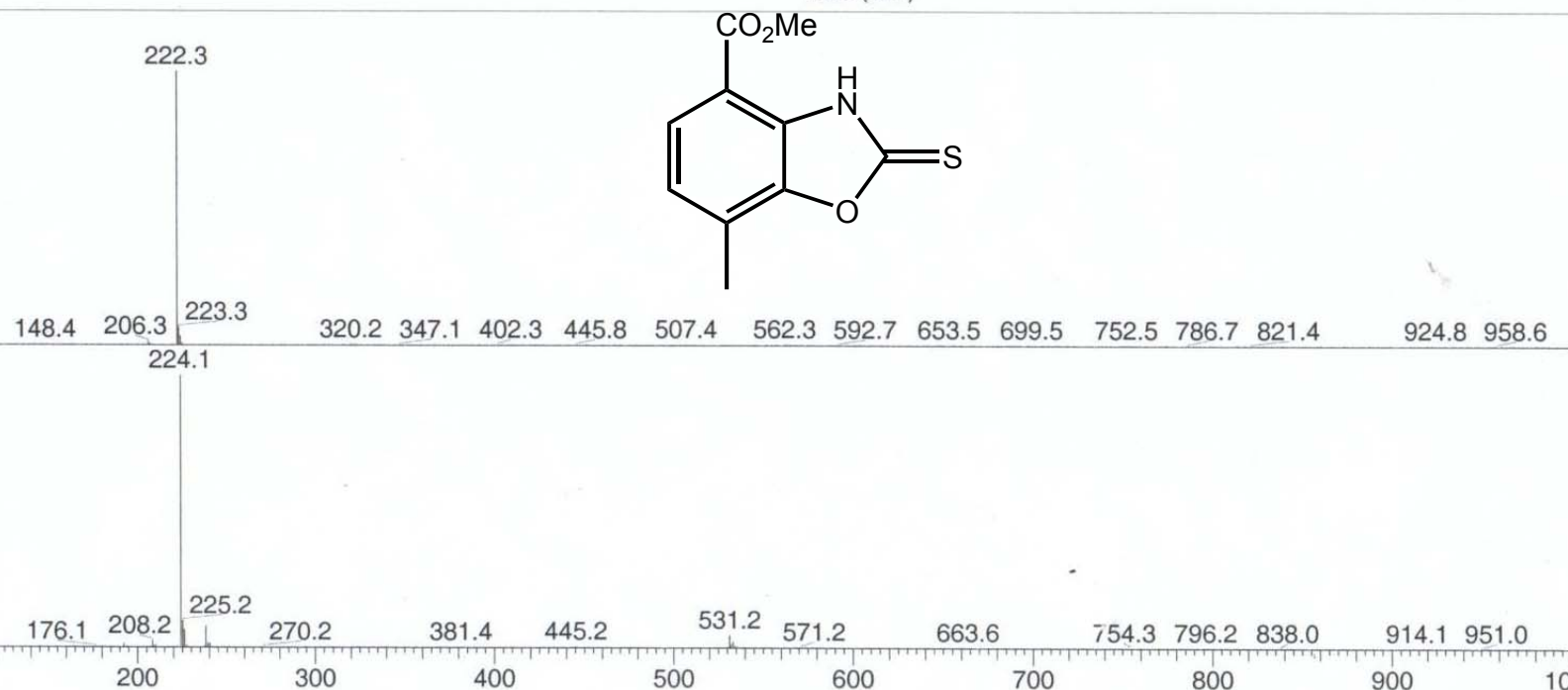
# Mass spectrum of Compound 8

S10

NL: 2.31E6  
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det=1212.00  
100.00-1000  
Vial43



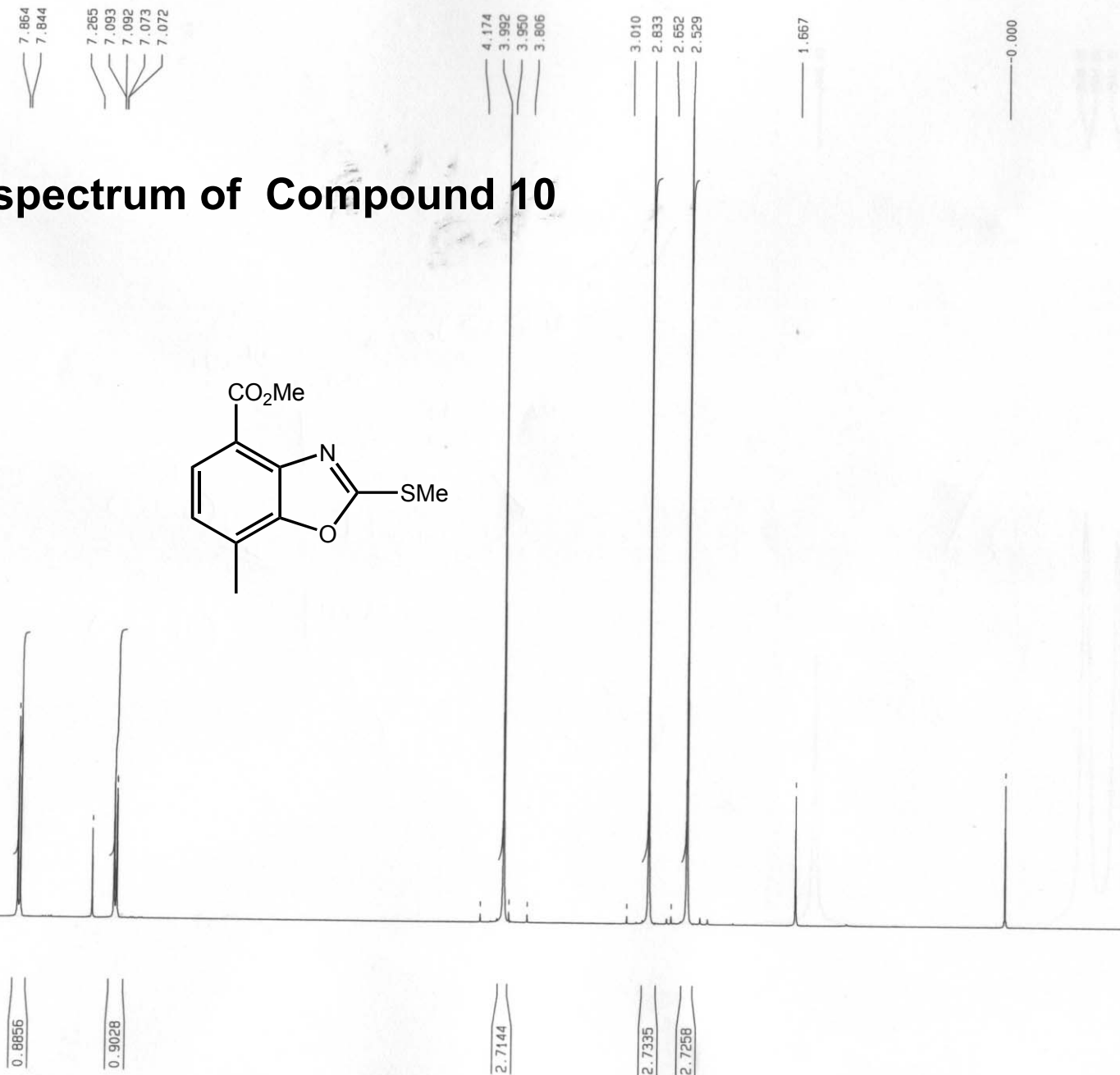
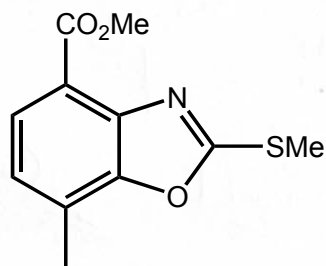
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TIC F: {0,1}  
corona sid=  
det=1212.00  
100.00-1000  
Vial43



Vial43#6-11 RT: 0.12-0.2  
SB: 2 0.54-0.61 NL: 2.33  
{0,0} - c APCI corona sid=  
det=1212.00 Full ms [  
100.00-1000.00]

Vial43#6-11 RT: 0.10-0.1  
SB: 2 0.54-0.61 NL: 2.35  
{0,1} + c APCI corona sid=  
det=1212.00 Full ms [  
100.00-1000.00]

S11

<sup>1</sup>H NMR spectrum of Compound 10

Current Data Parameters  
NAME cr053  
EXPNO 1  
PROCNO 1

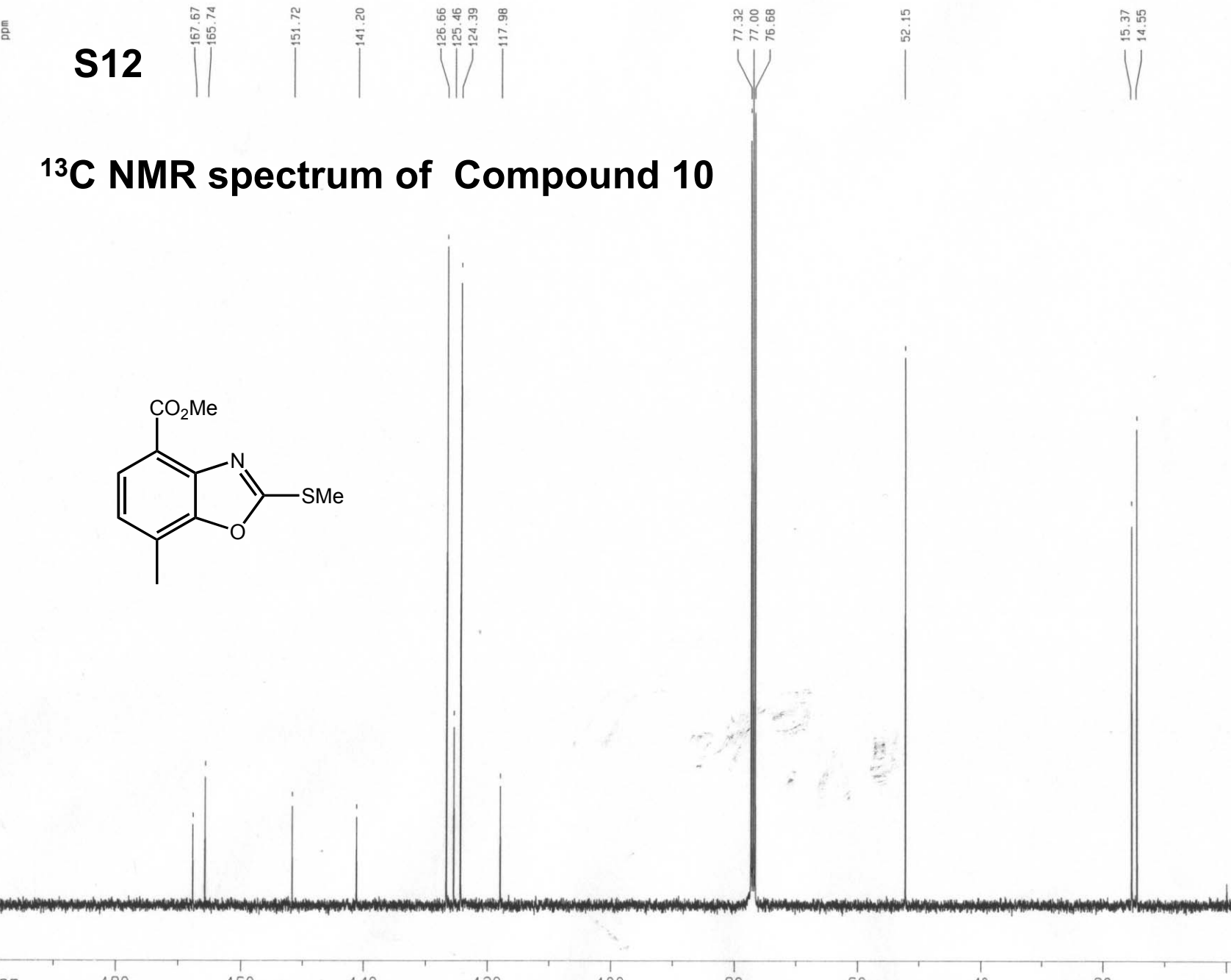
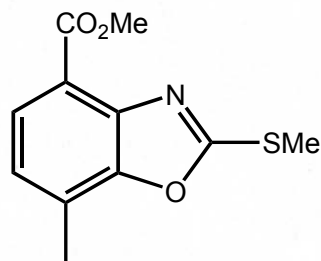
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Time 10.57  
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PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 8389.262 Hz  
FIDRES 0.128010 Hz  
AQ 3.9059956 sec  
RG 181  
DW 59.600 usec  
DE 10.00 usec  
TE 300.0 K  
D1 2.0000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 9.50 usec  
PL1 -3.00 dB  
SFO1 400.1334011 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300073 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 22.00 cm  
CY 25.00 cm  
F1P 10.000 ppm  
F1 4001.30 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.50000 ppm/c  
HZCM 200.06500 Hz/cm

S12

<sup>13</sup>C NMR spectrum of Compound 10

Current Data Parameters  
NAME cr053  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050204  
Time 12.07  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg  
TD 65536  
SOLVENT dms  
NS 2000  
DS 0  
SWH 23980.814 Hz  
FIDRES 0.365918 Hz  
AQ 1.3664756 sec  
RG 9195.2  
DW 20.850 usec  
DE 6.50 usec  
TE 300.0 K  
D1 0.75000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 6.26 usec  
PL1 -1.00 dB  
SF01 100.6239378 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 100.00 usec  
PL2 -3.00 dB  
PL12 17.50 dB  
PL13 19.00 dB  
SF02 400.1318006 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127708 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 22.00 cm  
CY 14.76 cm  
F1P 200.000 ppm  
F1 20122.55 Hz  
F2P -1.000 ppm  
F2 -100.61 Hz  
PPMCH 9.13636 ppm/  
HZCM 919.23480 Hz/c

0.17

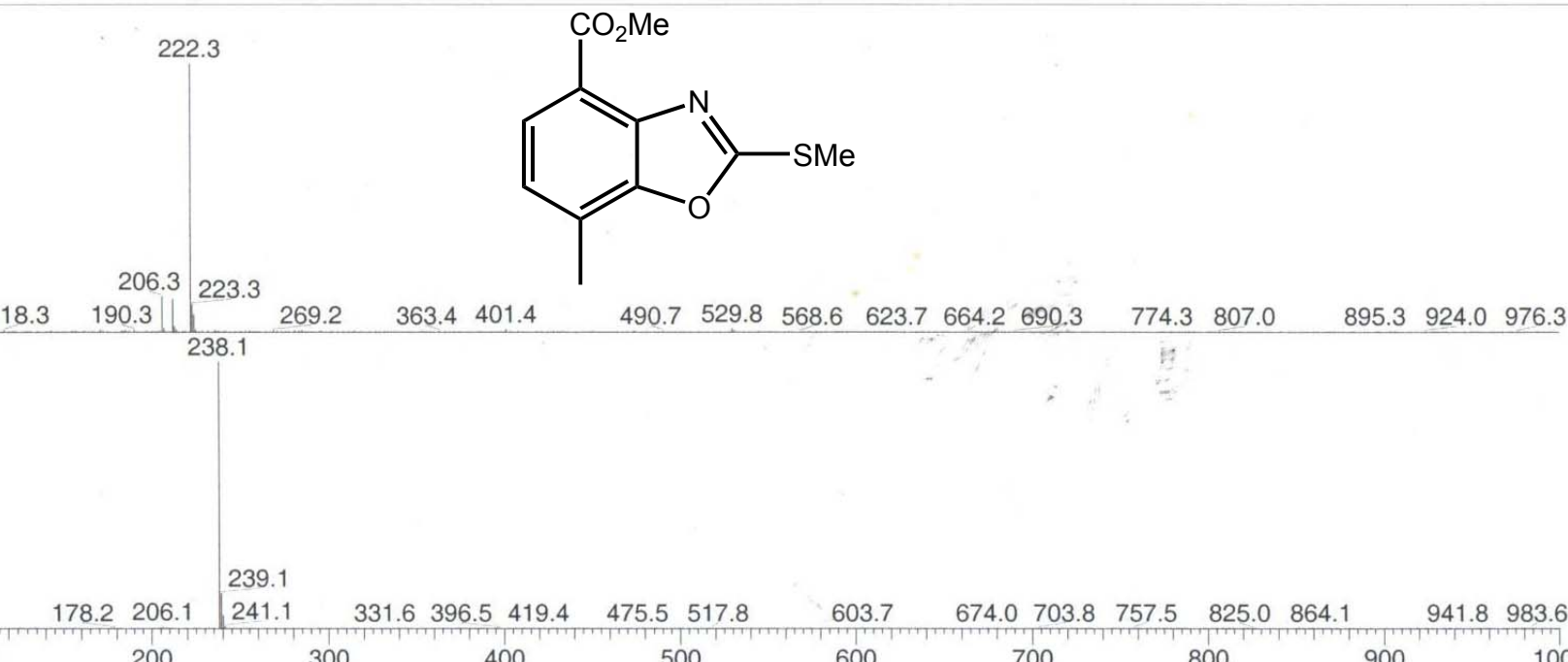
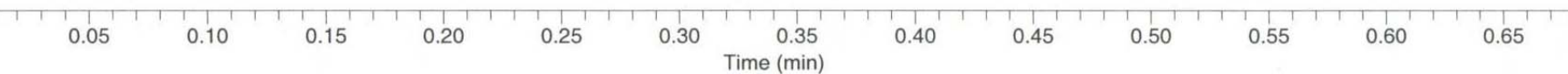
S13

NL: 9.06E5  
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det=1212.0  
100.00-100  
Vial73

# Mass spectrum of Compound 10

0.19

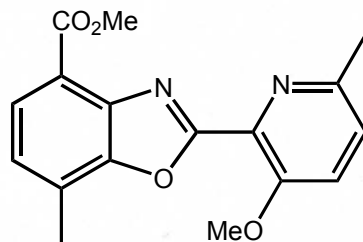
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TIC F: {0,1}  
corona sid-  
det=1212.0  
100.00-100  
Vial73



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SB: 2 0.54-0.61 NL: 3.4  
{0,0} - c APCI corona si  
det=1212.00 Full ms [  
100.00-1000.00]

Vial73#6-11 RT: 0.10-0  
SB: 2 0.54-0.61 NL: 5.2  
{0,1} + c APCI corona si  
det=1212.00 Full ms [  
100.00-1000.00]

S14

<sup>1</sup>H NMR spectrum of Compound 11

7.989  
7.969  
7.385  
7.364  
7.321  
7.299  
7.268  
7.258  
7.256  
7.239  
7.237

4.047  
4.025

2.681  
2.680  
2.640

1.873

-0.000

Current Data Parameters  
NAME cr087  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20050209  
Time 10.12  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 8389.262 Hz  
FIDRES 0.128010 Hz  
AQ 3.9059956 sec  
RG 287.4  
DW 59.600 usec  
DE 10.00 usec  
TE 300.0 K  
D1 2.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 9.50 usec  
PL1 -3.00 dB  
SF01 400.1334011 MHz

F2 - Processing parameters  
SI 32768  
SF 400.130063 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 22.00 cm  
CY 12.88 cm  
F1P 10.000 ppm  
F1 4001.30 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.50000 ppm/cm  
HZCM 200.06500 Hz/cm

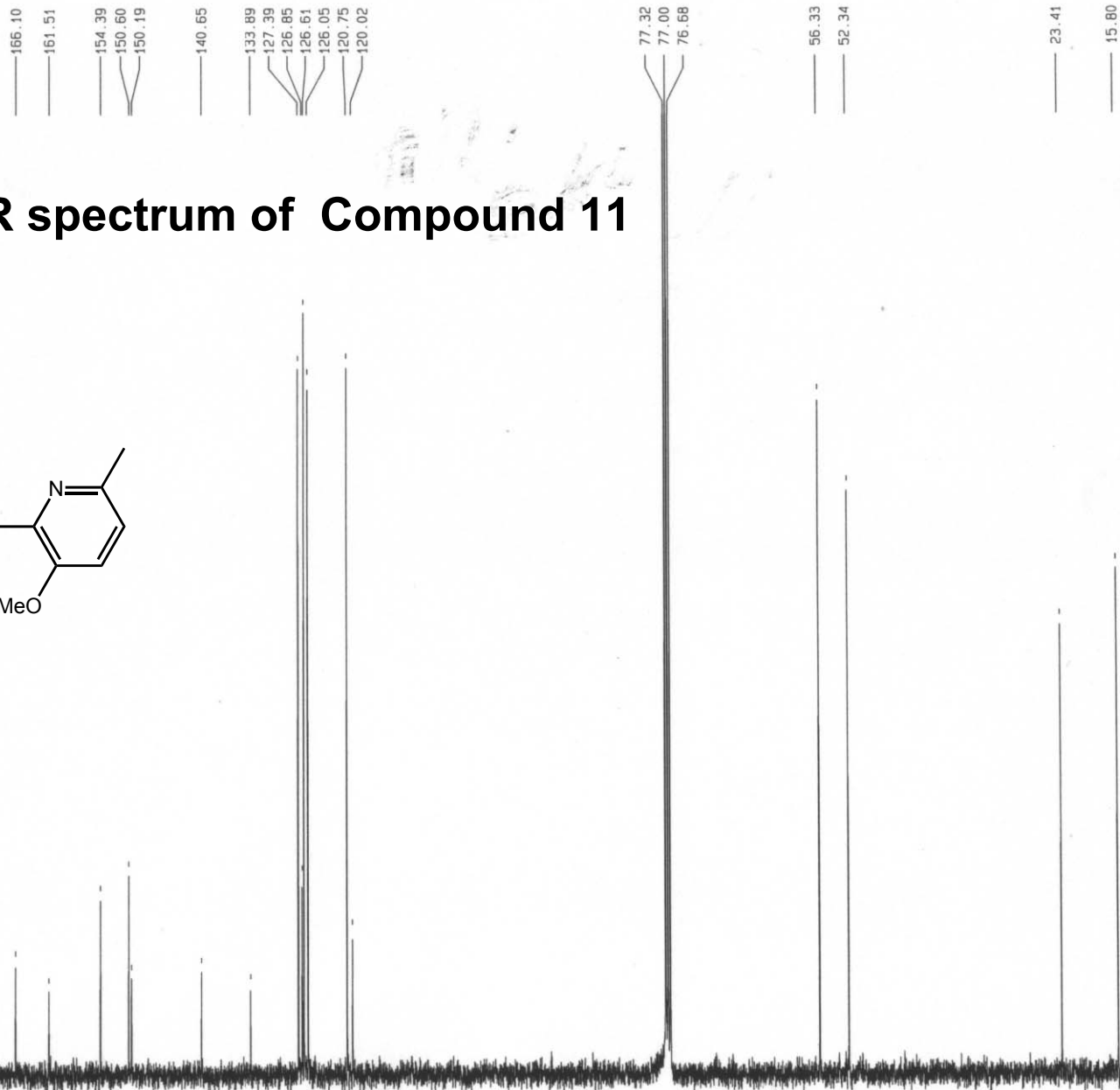
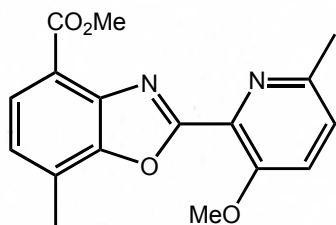
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3.0480  
3.0699

3.0069  
3.1147

3.0480  
3.0699

S15

<sup>13</sup>C NMR spectrum of Compound 11

Current Data Parameters  
 NAME cr087  
 EXPNO 6  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050209  
 Time 14.32  
 INSTRUM spect  
 PROBHD 5 mm BBO BB-1H  
 PULPROG zgpg  
 TD 65536  
 SOLVENT cdc13  
 NS 2000  
 DS 0  
 SWH 26178.010 Hz  
 FIDRES 0.399445 Hz  
 AQ 1.2517875 sec  
 RG 9195.2  
 DW 19.100 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 0.75000000 sec  
 d11 0.03000000 sec  
 d12 0.00002000 sec

===== CHANNEL f1 =====  
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 PL1 -1.00 dB  
 SF01 100.6248442 MHz

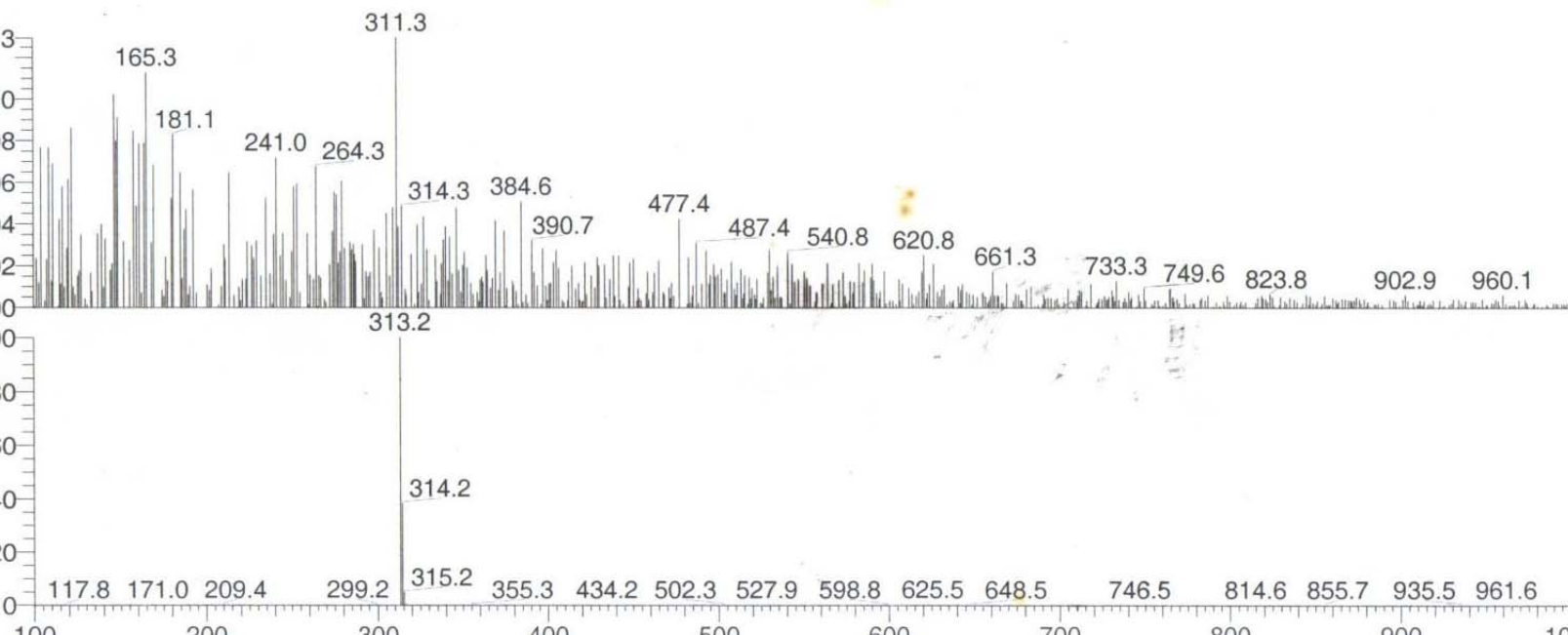
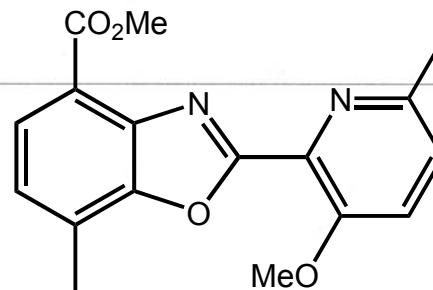
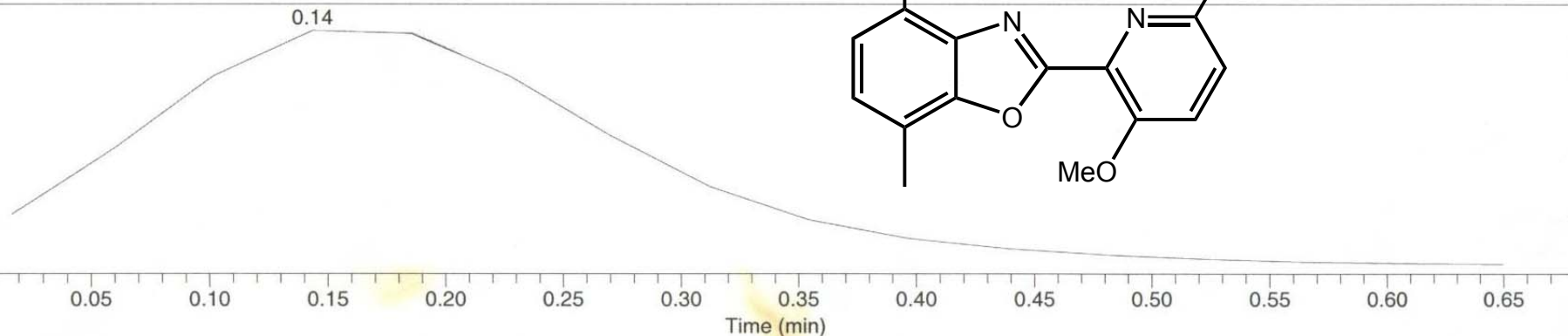
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 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 100.00 usec  
 PL2 -3.00 dB  
 PL12 17.50 dB  
 PL13 19.00 dB  
 SF02 400.1318100 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127702 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 22.00 cm  
 CY 22.51 cm  
 F1P 200.000 ppm  
 F1 20122.55 Hz  
 F2P -1.000 ppm  
 F2 -100.61 Hz  
 PPMCM 9.13636 ppm/  
 HZCM 919.23480 Hz/c

**S16**

# Mass spectrum of Compound 11



Vial26#6-11 RT: 0.12-0.14  
SB: 2 0.54-0.61 NL: 2.1  
{0,0} - c APCI corona si  
det=1212.00 Full ms [  
100.00-1000.00]

Vial26#6-11 RT: 0.10-0.12  
SB: 2 0.54-0.61 NL: 1.6  
{0,1} + c APCI corona si  
det=1212.00 Full ms [  
100.00-1000.00]