SUPPORTING INFORMATION

FOR

Total synthesis of monosporascone and dihydromonosporascone

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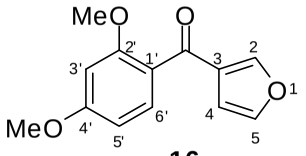
CONTENTS: NMR spectra

300 MHz ^{1}H NMR spectrum in $CDCl_3$



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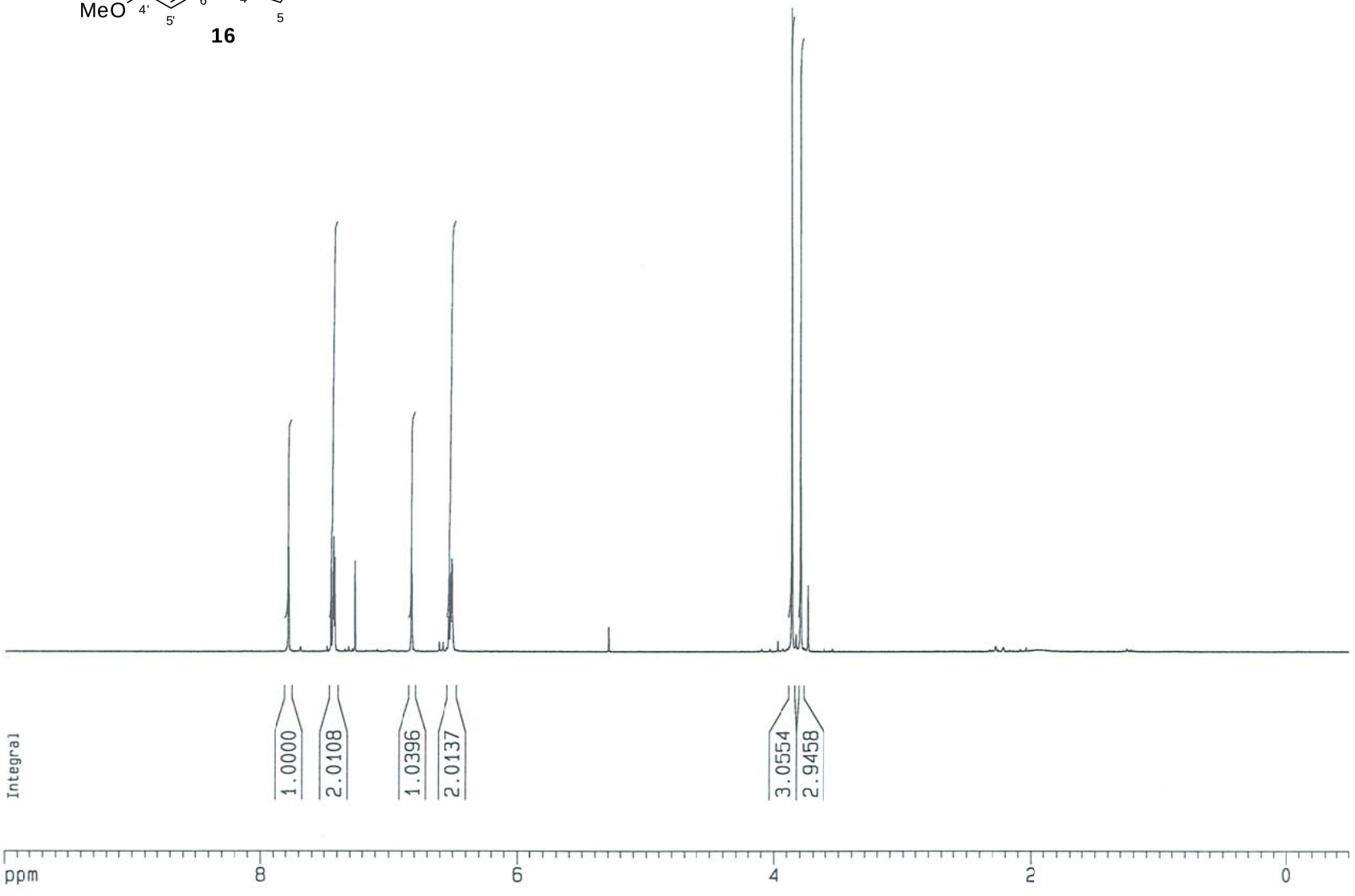
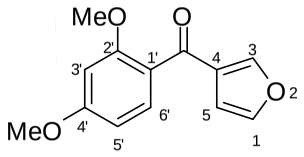


75.5 MHz ^{13}C NMR spectrum in $CDCl_3$



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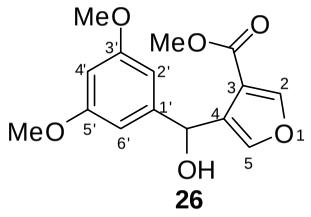
300 MHz ^{1}H NMR spectrum in $CDCl_3$



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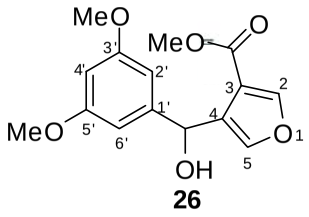
75.5 MHz ^{13}C NMR spectrum in $CDCl_3$



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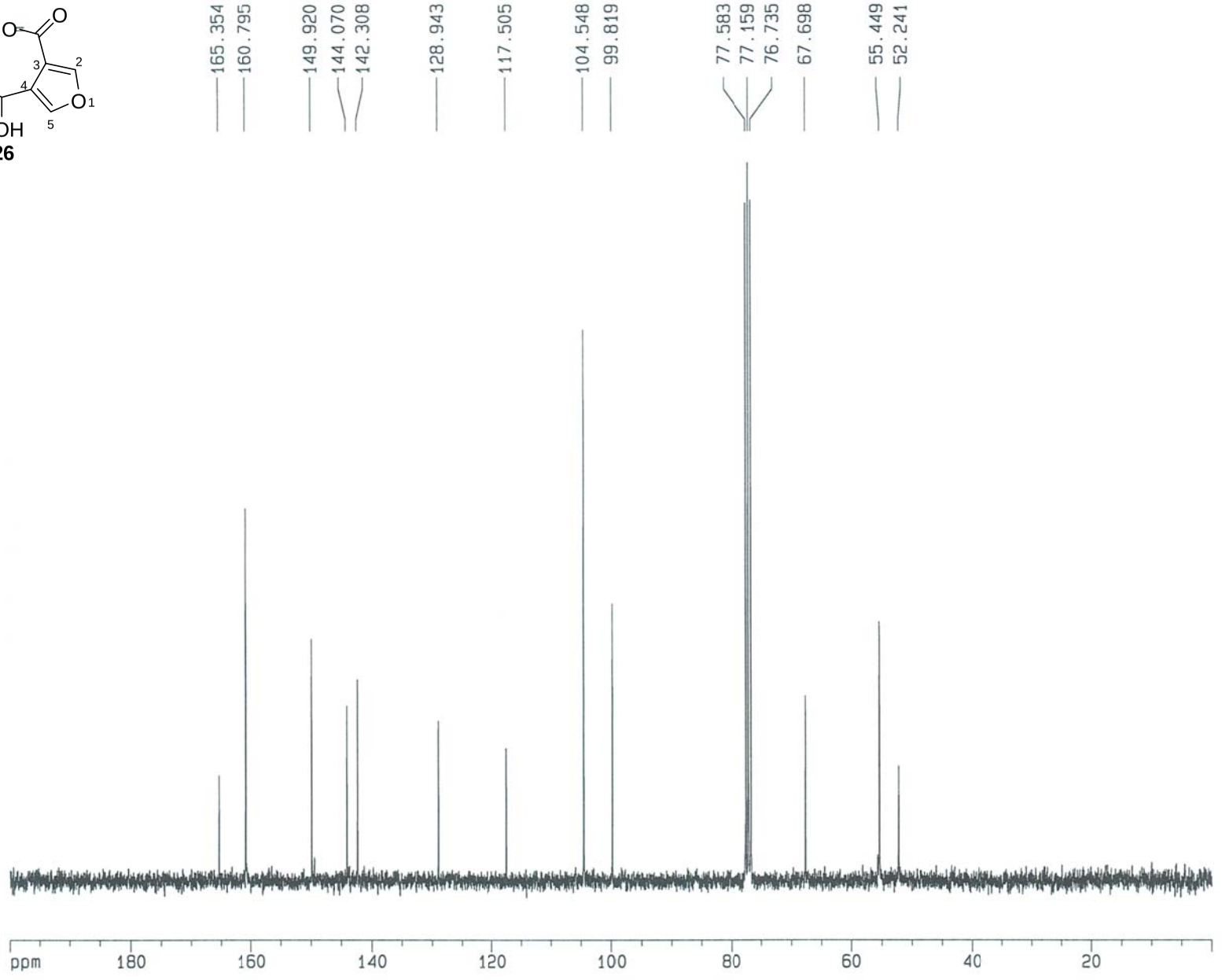
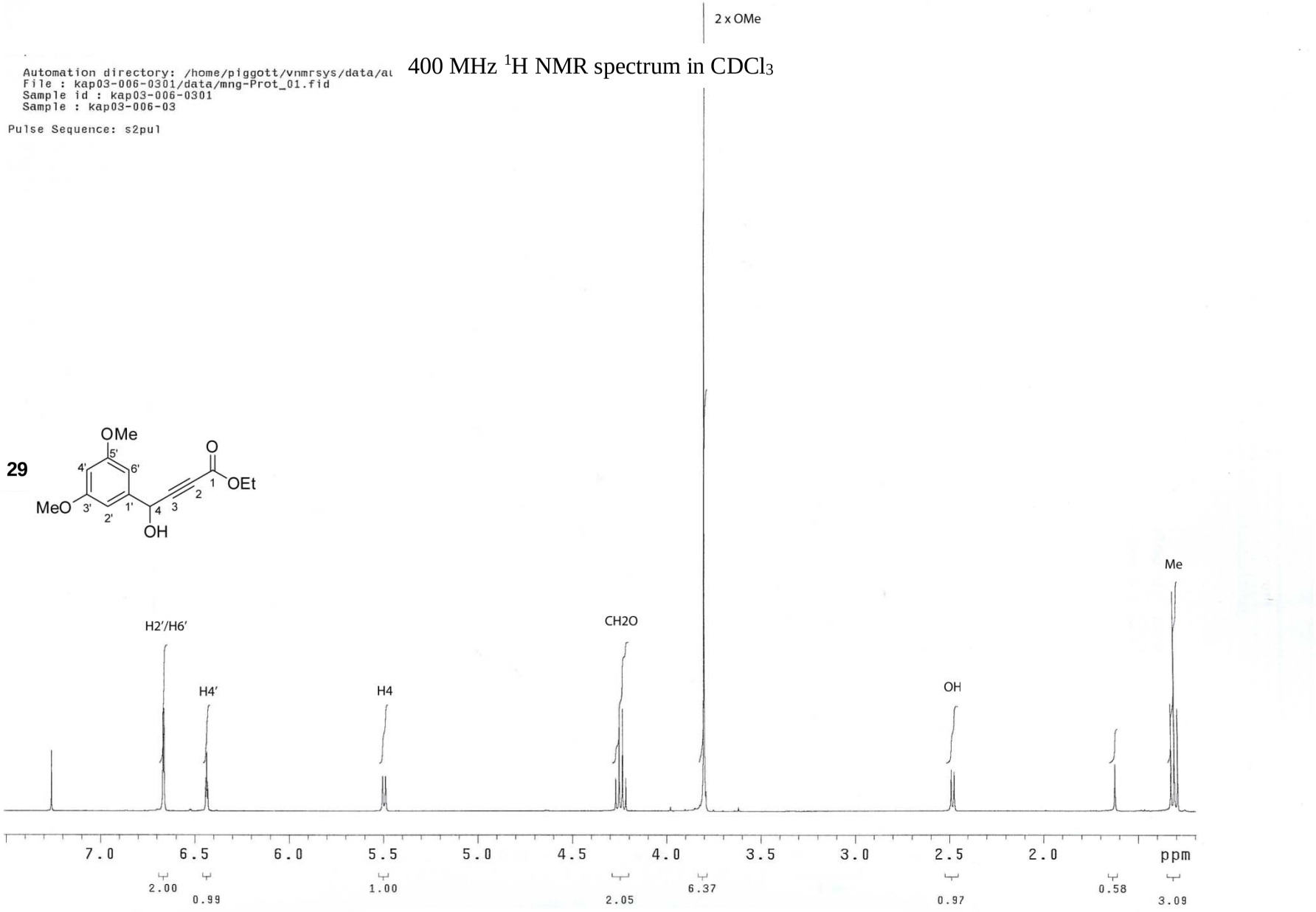


400 MHz ^{1}H NMR spectrum in $CDCl_3$



100 MHz ^{13}C NMR spectrum in $CDCl_3$

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File : exp
Sample id : tmpstudy
Sample : kap03-006-03

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: piggott
VNMRS-400  "var400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
192 repetitions
OBSERVE  C13, 100.5437342 MHz
DECOUPLE  H1, 399.8575230 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 29 sec
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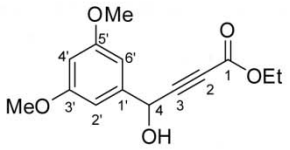
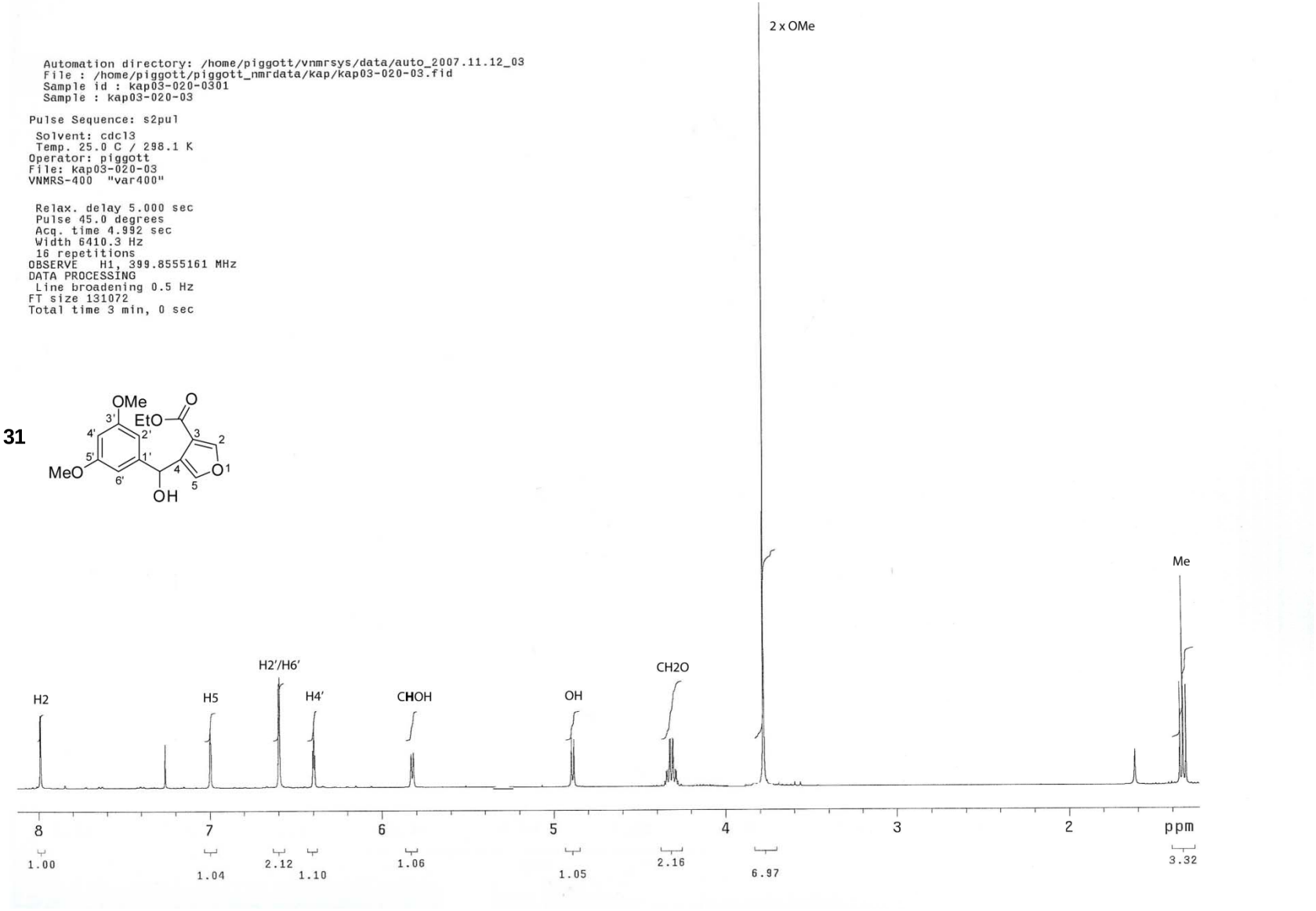


400 MHz ^{1}H NMR spectrum in $CDCl_3$



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100 MHz ^{13}C NMR spectrum in $CDCl_3$

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File : exp
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Sample : kap03-020-03

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: piggott
VNMRS-400  "var400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
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OBSERVE  C13, 100.5437350 MHz
DECOUPLE  H1, 399.8575230 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
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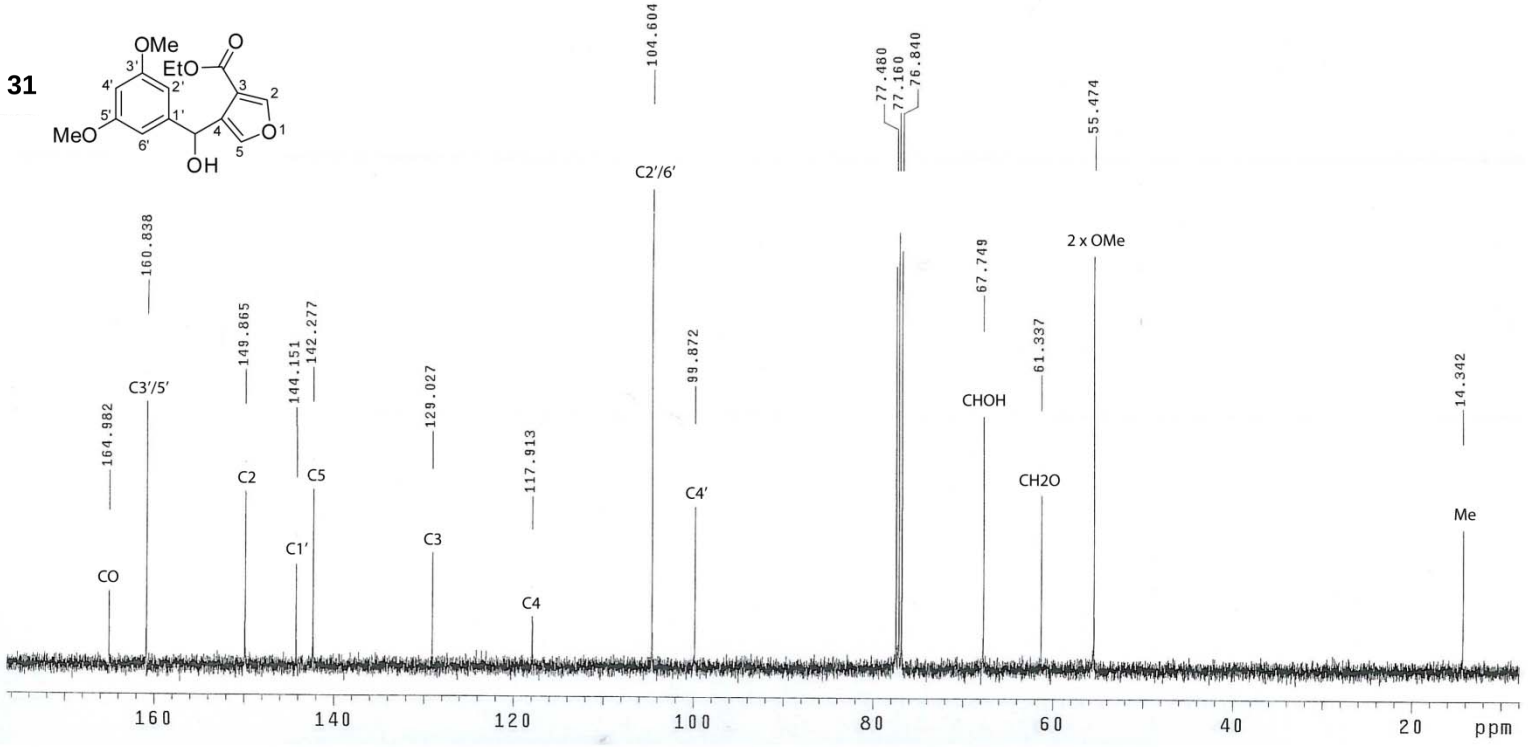
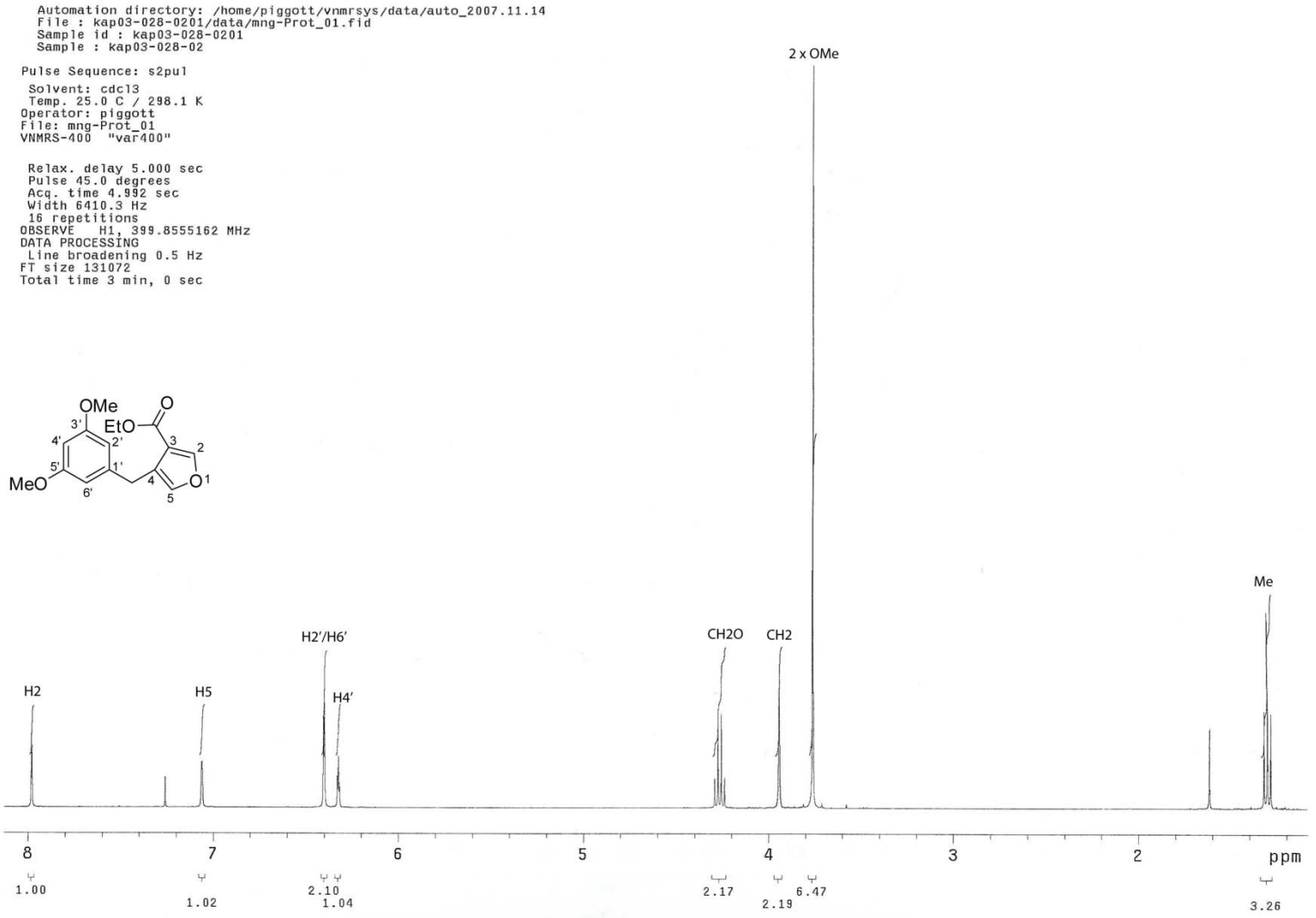


400 MHz ^{1}H NMR spectrum in $CDCl_3$



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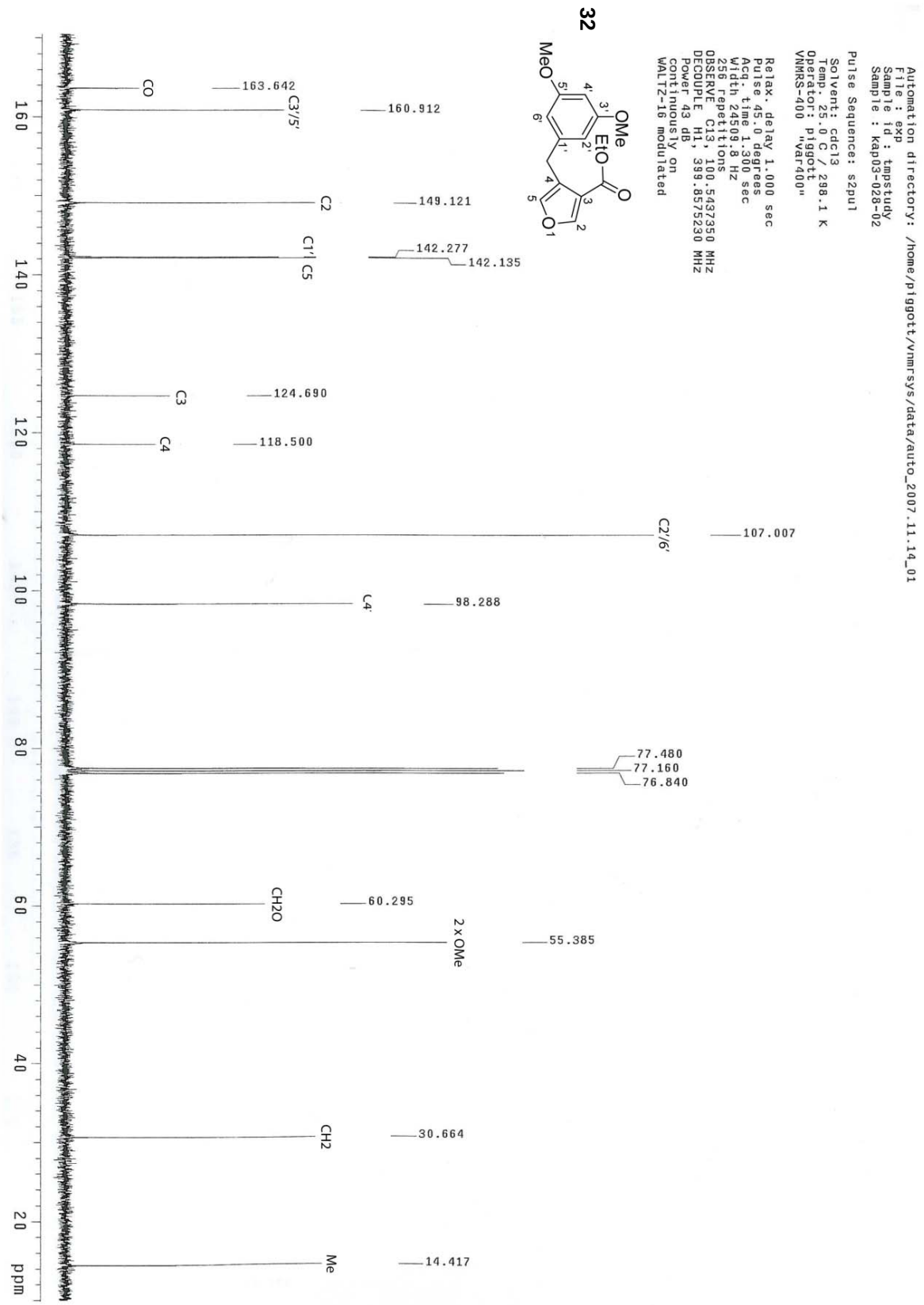
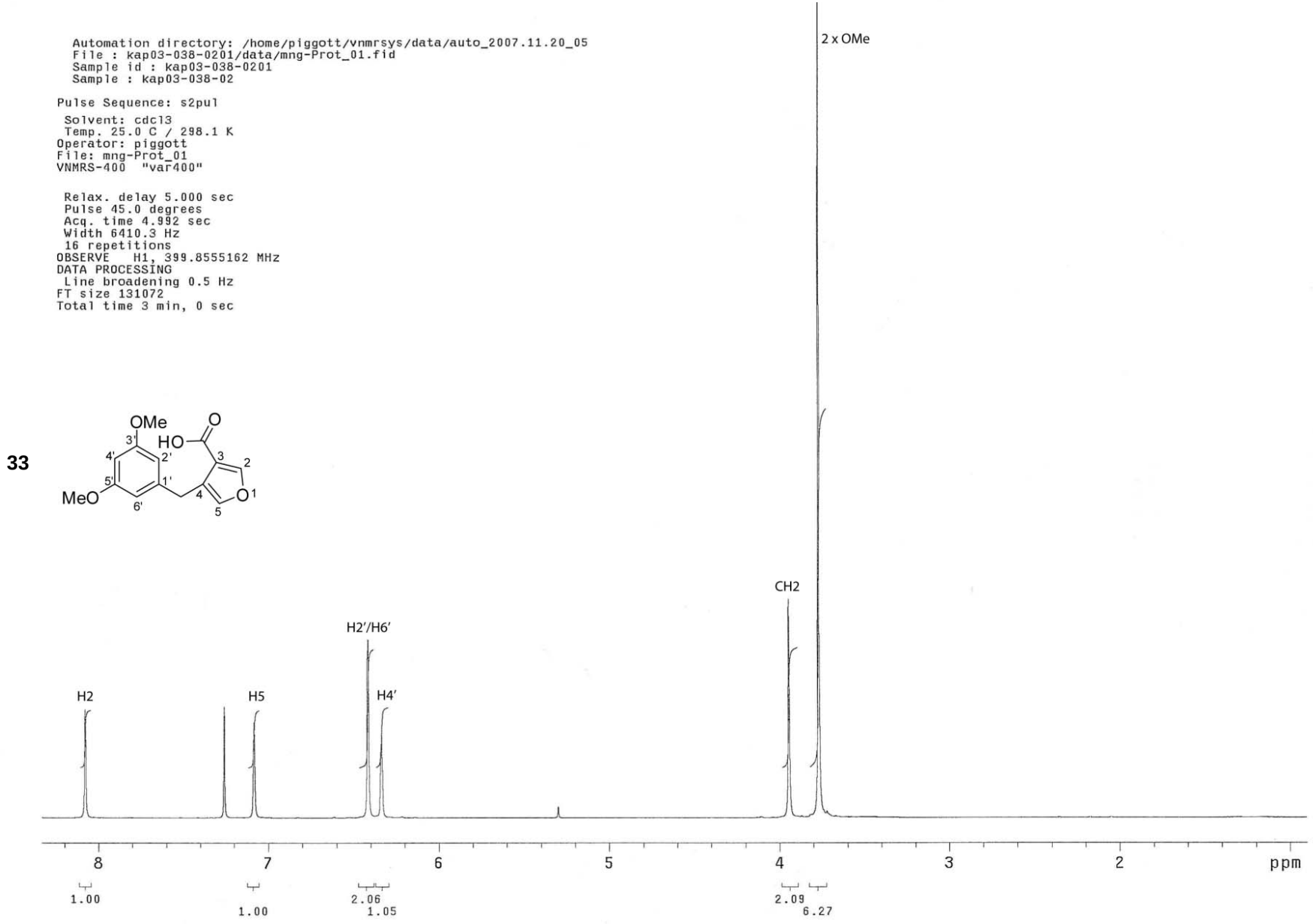
100 MHz ^{13}C NMR spectrum in $CDCl_3$



400 MHz ^{1}H NMR spectrum in $CDCl_3$



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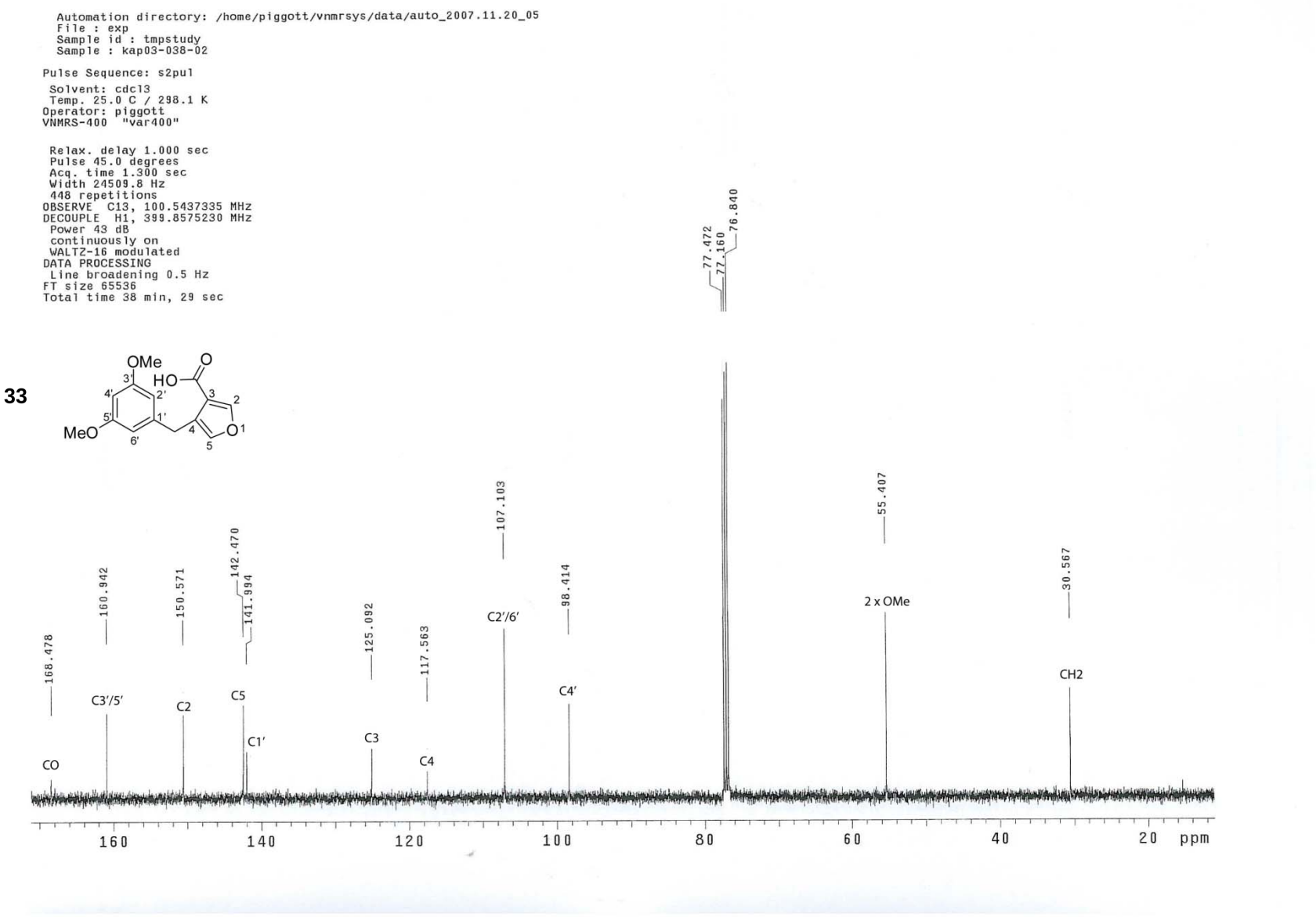
100 MHz ^{13}C NMR spectrum in $CDCl_3$



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400 MHz ^{1}H NMR spectrum in $CDCl_3$

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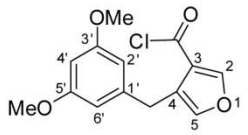

Current Data Parameters
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PROCNO 1

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INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 4
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 574.7
DW 66.600 usec
DE 7.50 usec
TE 298.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.70 usec
PL1 −1.00 dB
SFO1 500.1327500 MHz

F2 – Processing parameters
SI 65536
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WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

100 MHz ^{13}C NMR spectrum in $CDCl_3$

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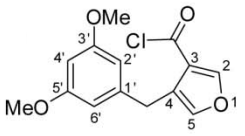

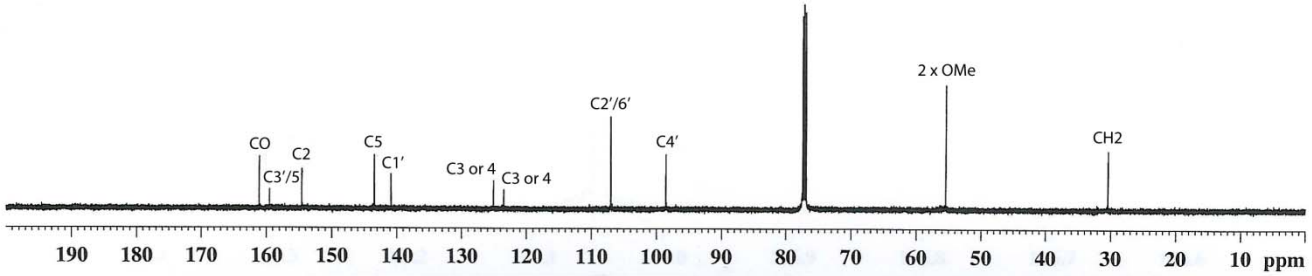

Current Data Parameters
NAME kap05–044–02
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
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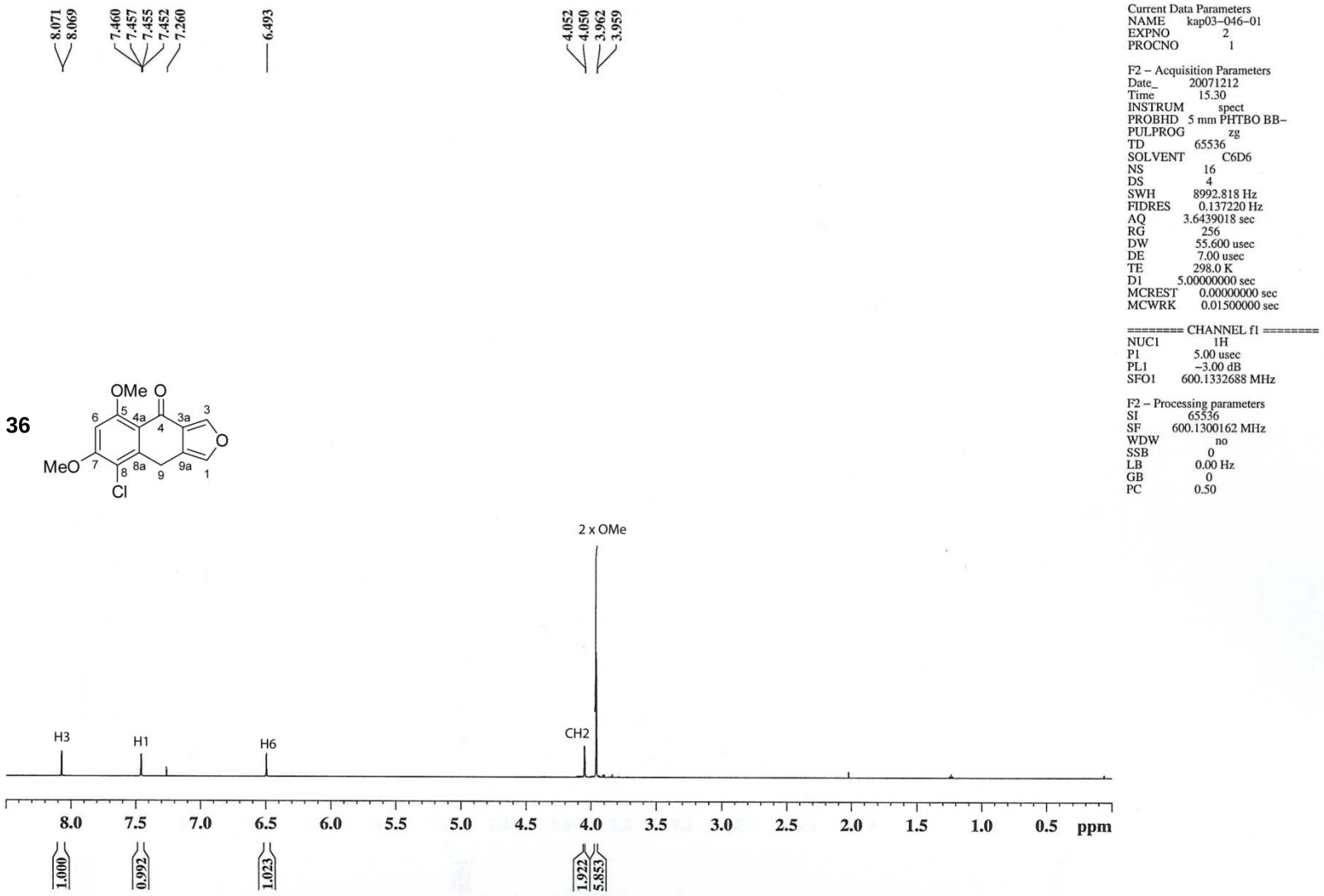
Time 14.00
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 839
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 20642.5
DW 15.900 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 125.7709890 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 500.1322500 MHz

F2 – Processing parameters
SI 65536
SF 125.7577755 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

600 MHz ^{1}H NMR spectrum in $CDCl_3$



151 MHz ^{13}C NMR spectrum in $CDCl_3$

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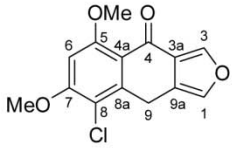

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EXPNO 3
PROCNO 1

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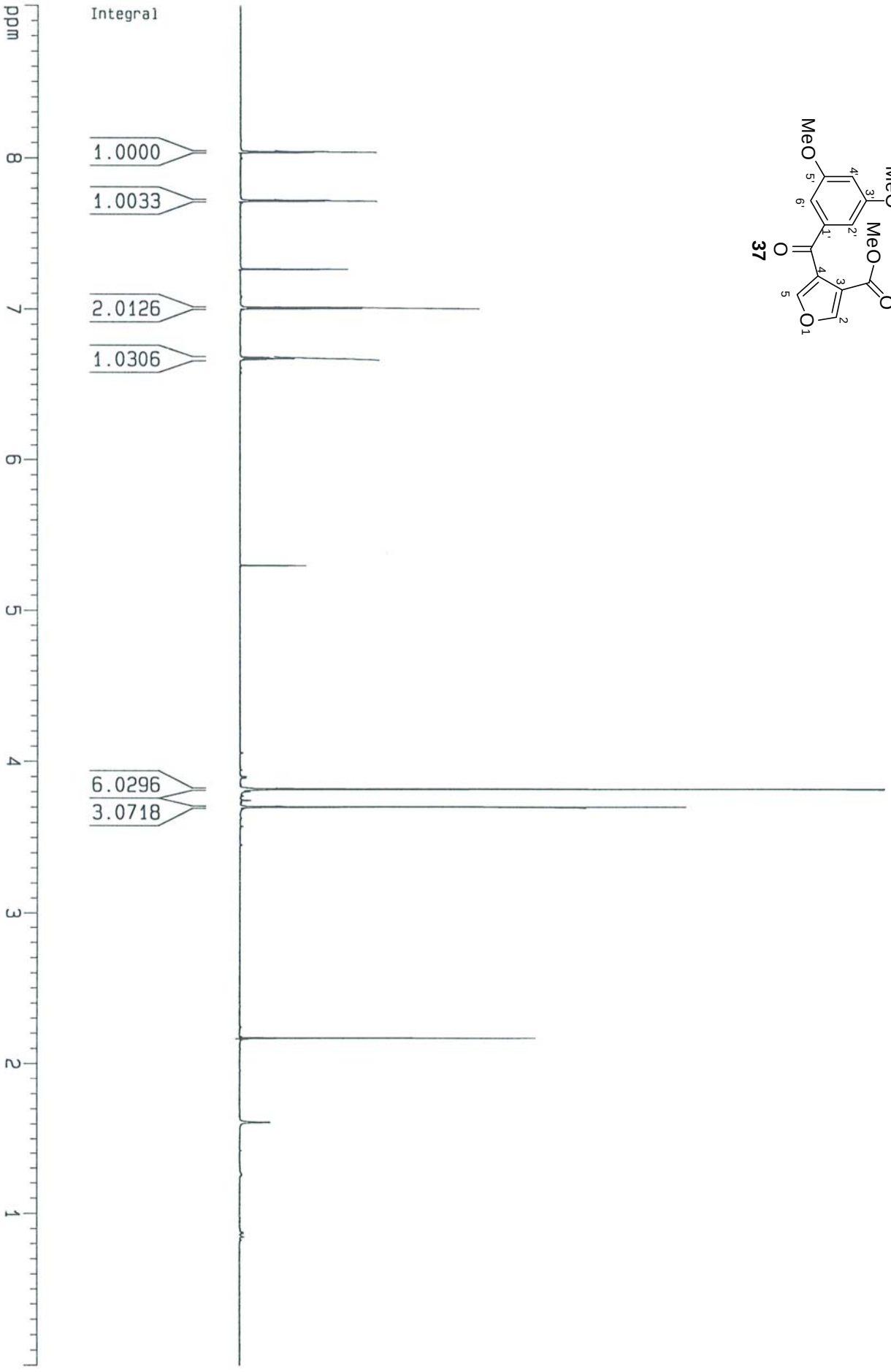
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 60
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 16384
DW 13.300 usec
DE 7.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

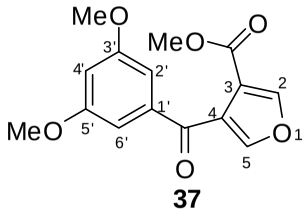
======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 –3.00 dB
SFO1 150.9147813 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 12.70 dB
PL13 22.00 dB
SFO2 600.1324500 MHz

F2 – Processing parameters
SI 65536
SF 150.9027963 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.50



300 MHz ^{1}H NMR spectrum in $CDCl_3$



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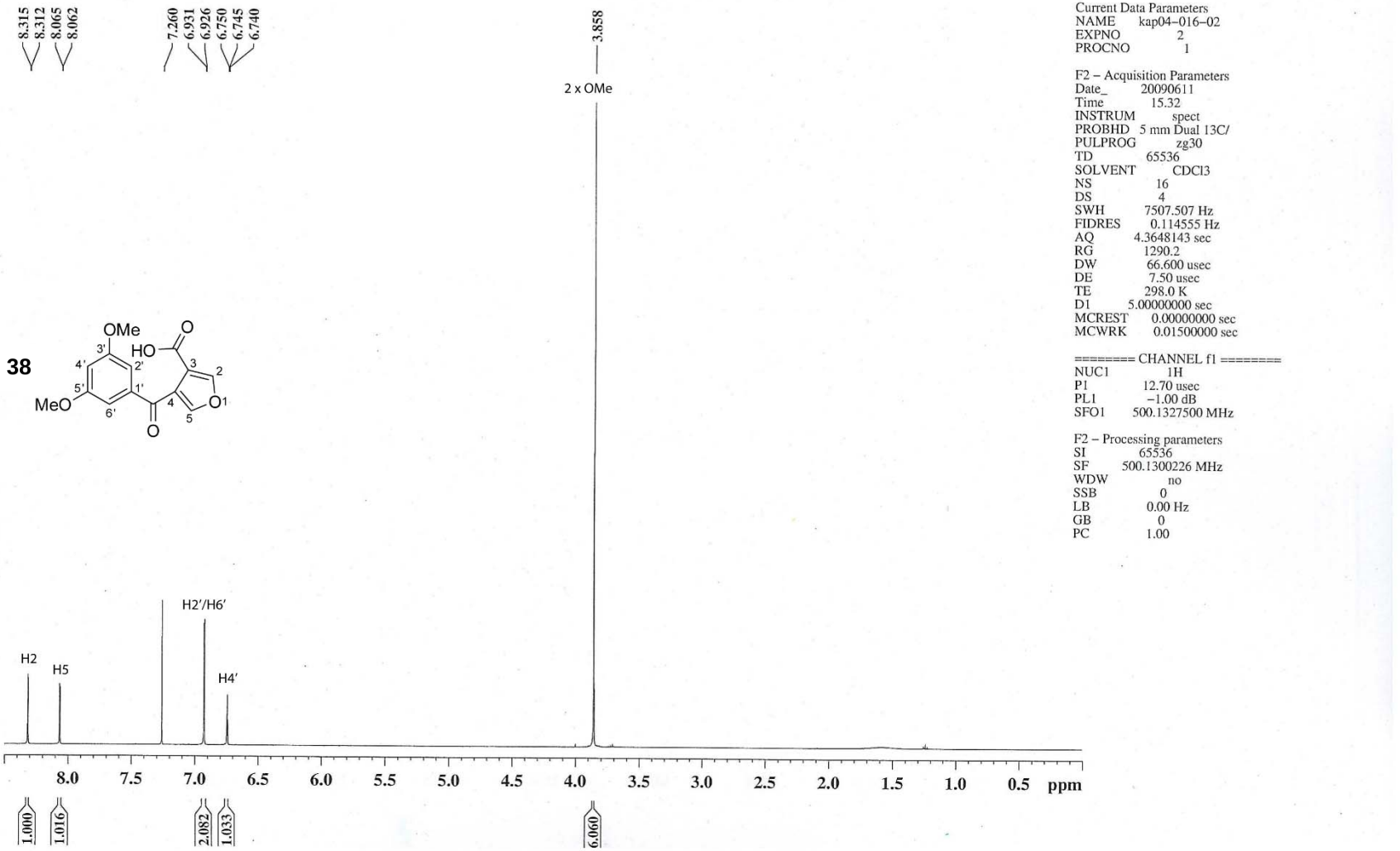
75.5 MHz ^{1}H NMR spectrum in $CDCl_3$

500 MHz ^{1}H NMR spectrum in $CDCl_3$



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EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
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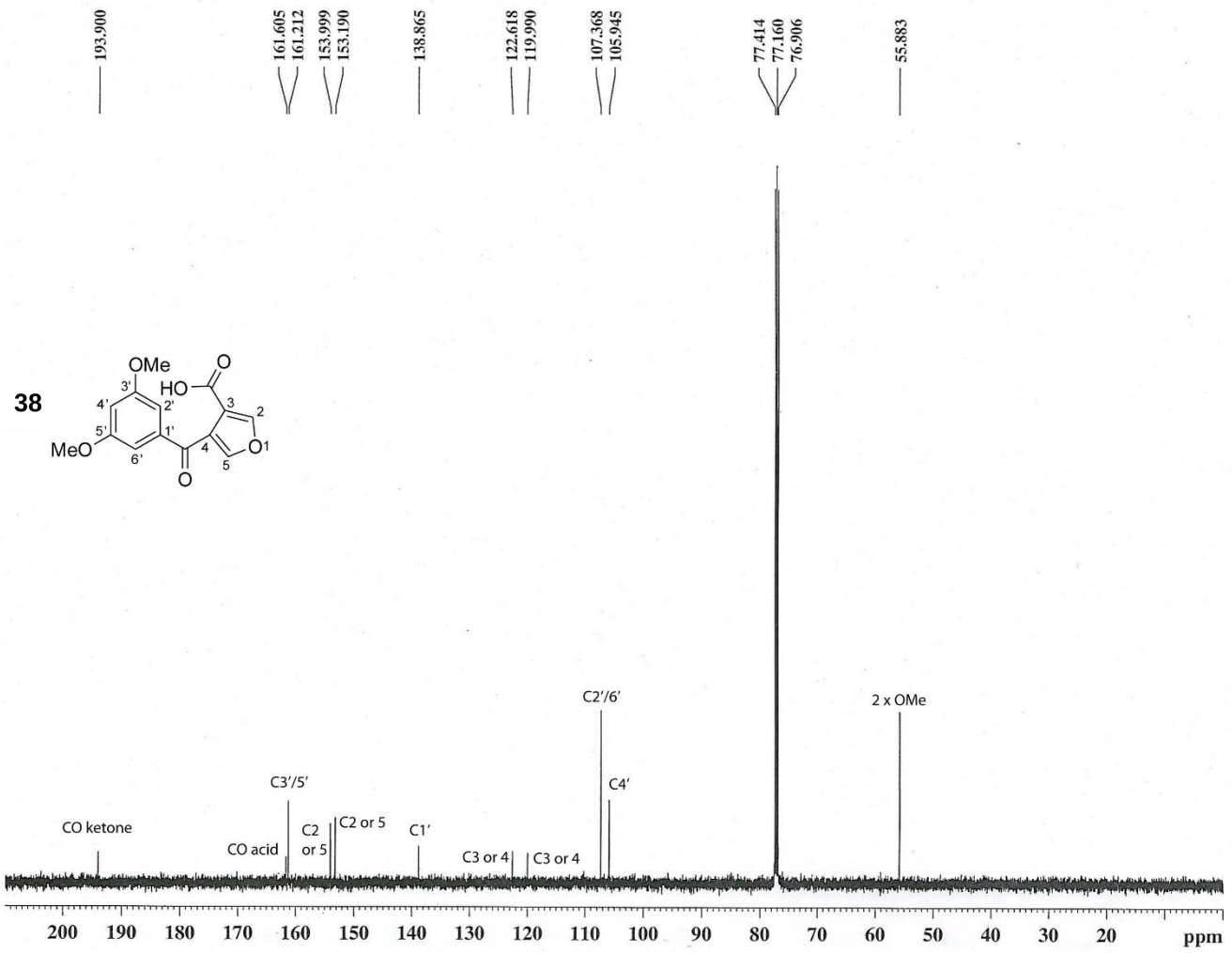
Time 15.32
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 1290.2
DW 66.600 usec
DE 7.50 usec
TE 298.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.70 usec
PL1 −1.00 dB
SFO1 500.1327500 MHz

F2 – Processing parameters
SI 65536
SF 500.1300226 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

125.7 MHz ^{13}C NMR spectrum in $CDCl_3$



Current Data Parameters
NAME kap04–016–02
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date_ 20090611
Time 15.37
INSTRUM spect
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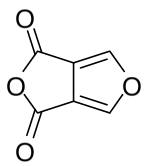
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 18390.4
DW 15.900 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 125.7709890 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 500.1322500 MHz

F2 – Processing parameters
SI 65536
SF 125.7577741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

300 MHz ^{1}H NMR spectrum in $CDCl_3$



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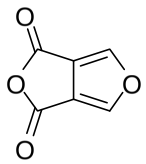
75.5 MHz ^{13}C NMR spectrum in $CDCl_3$



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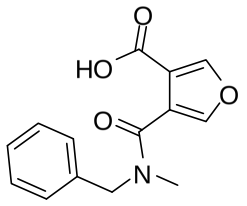
600 MHz ^{1}H NMR spectrum in d_6-DMSO



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500 MHz ^{1}H NMR spectrum in $CDCl_3$

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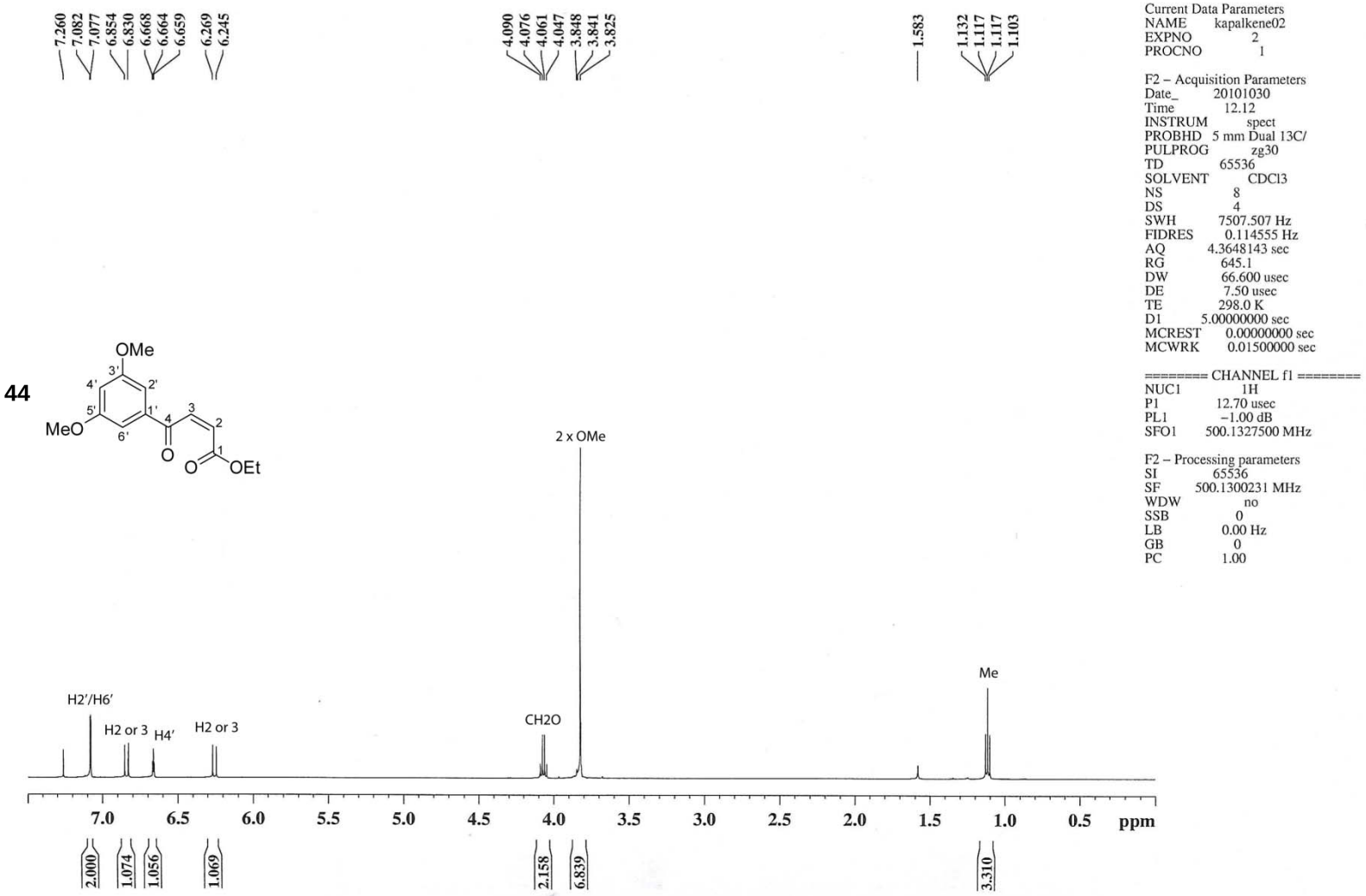

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PROCNO 1

F2 – Acquisition Parameters
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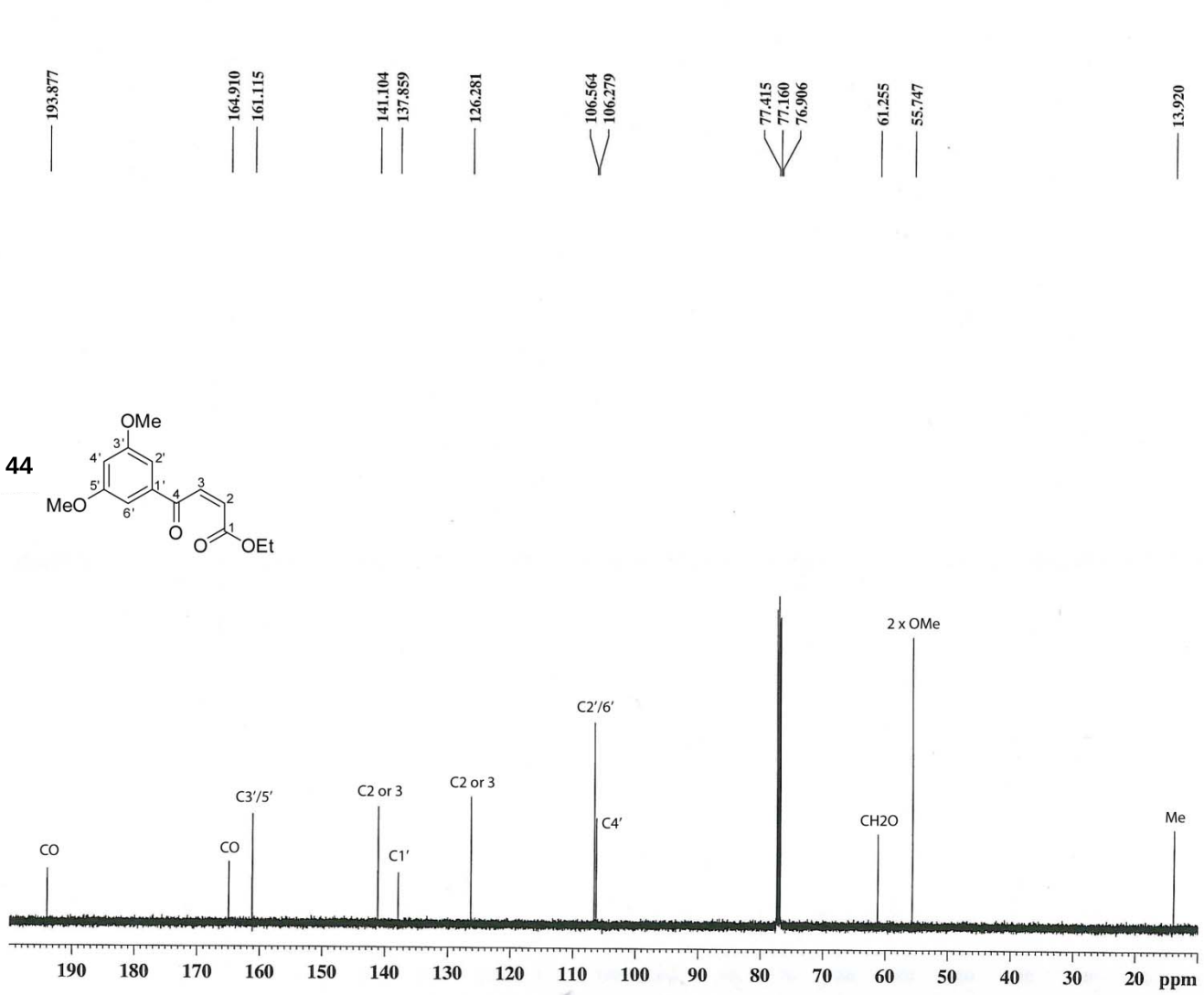
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 4
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 645.1
DW 66.600 usec
DE 7.50 usec
TE 298.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.70 usec
PL1 −1.00 dB
SFO1 500.1327500 MHz

F2 – Processing parameters
SI 65536
SF 500.1300231 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

125.8 MHz ^{13}C NMR spectrum in $CDCl_3$



Current Data Parameters
NAME kapalkene02
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date_ 20101030
Time 11.56
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 362
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 20642.5
DW 15.900 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 125.7709890 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 500.1322500 MHz

F2 – Processing parameters
SI 65536
SF 125.7577750 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

400 MHz ^{1}H NMR spectrum in $CDCl_3$

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Automation directory: /home/piggott/vnmrsys/data/auto_2007.11.18_02
File : kap02-147-0201/data/mng-Prot_01.fid
Sample id : kap02-147-0201
Sample : kap02-147-02

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: piggott
File: mng-Prot_01
VNMRS-400  "var400"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 4.992 sec
Width 6410.3 Hz
16 repetitions
OBSERVE   H1, 399.8555165 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 3 min, 0 sec
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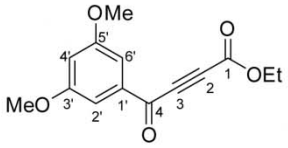
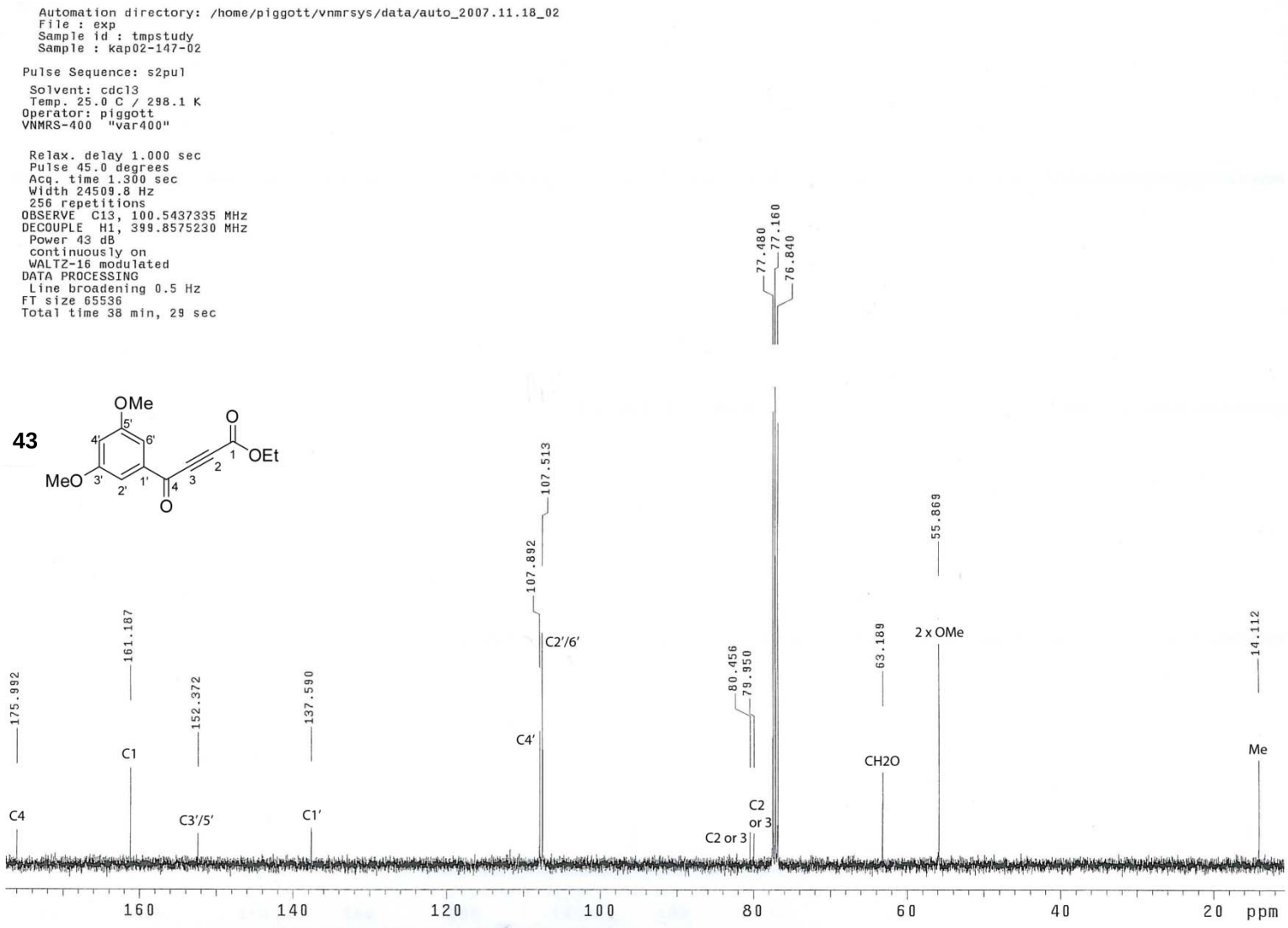


100 MHz ^{13}C NMR spectrum in $CDCl_3$



500 MHz ^{1}H NMR spectrum in $CDCl_3$

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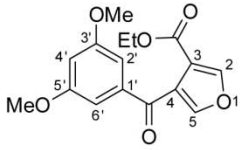

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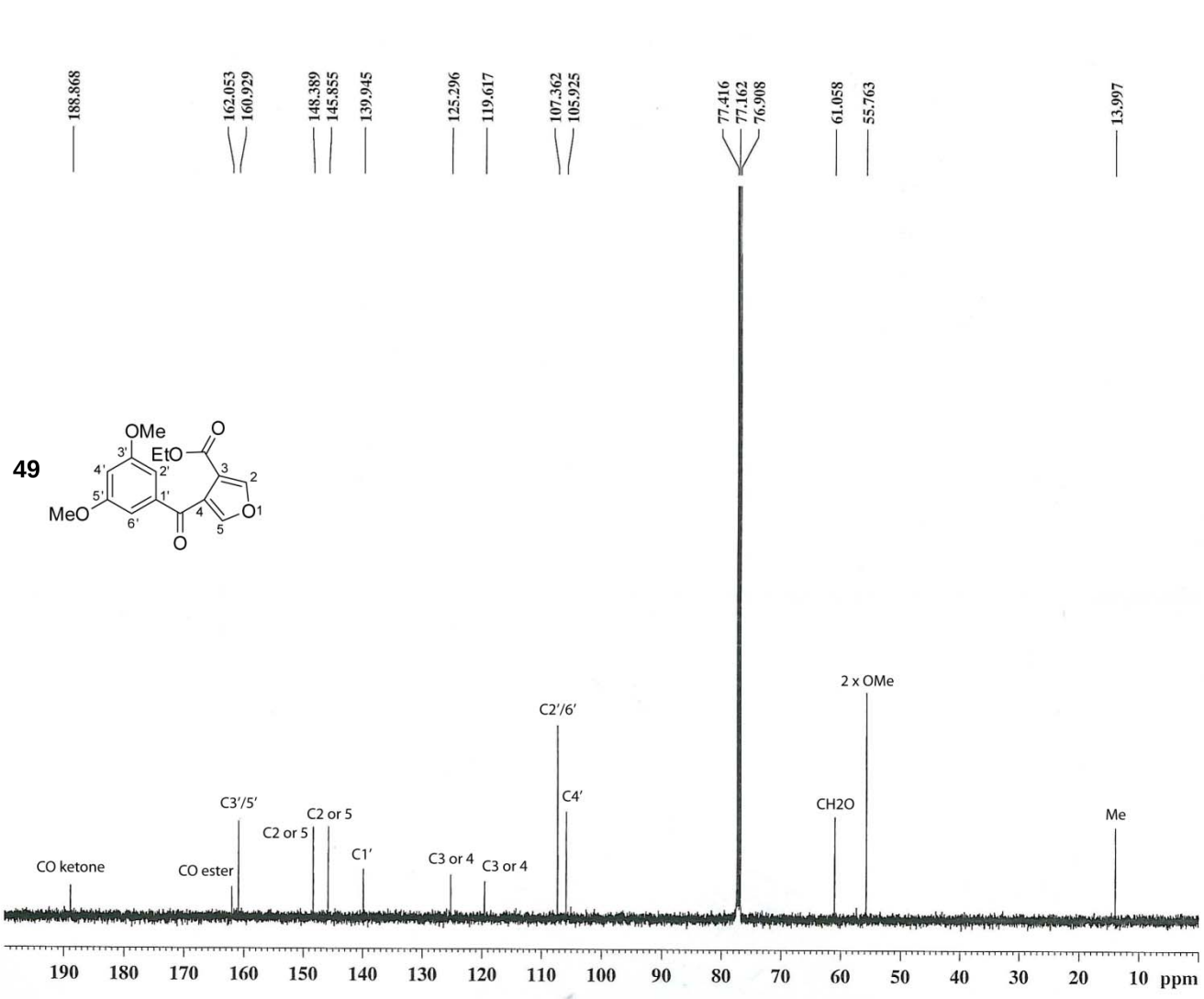
SOLVENT CDCl3
NS 8
DS 4
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 1024
DW 66.600 usec
DE 7.50 usec
TE 298.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.70 usec
PL1 -1.00 dB
SFO1 500.1327500 MHz

F2 – Processing parameters
SI 65536
SF 500.1300229 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

125.7 MHz ^{13}C NMR spectrum in $CDCl_3$



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Current Data Parameters
NAME kap03–40–02
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
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INSTRUM spect
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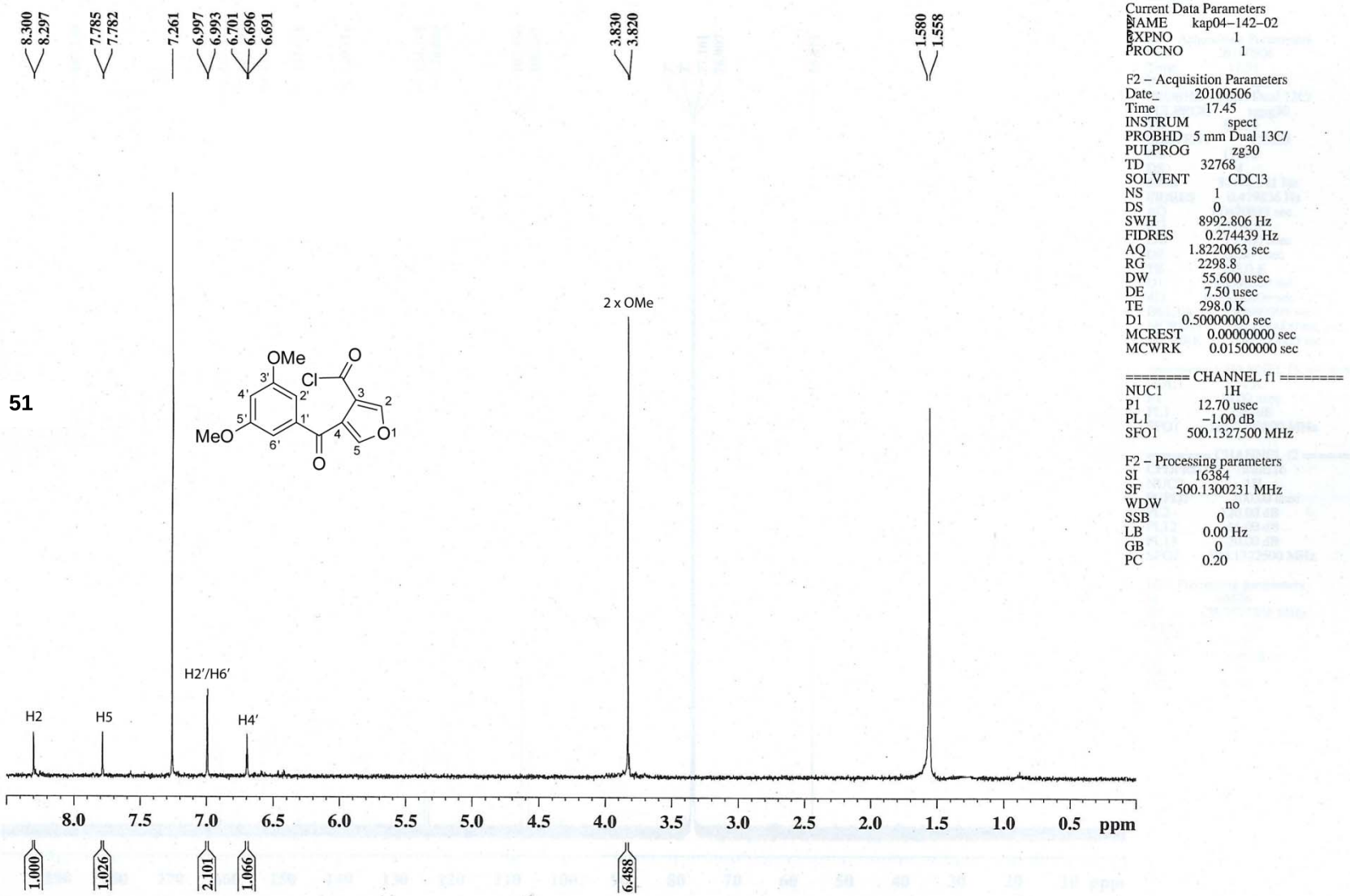
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 288
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 23170.5
DW 15.900 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 125.7709890 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 500.1322500 MHz

F2 – Processing parameters
SI 65536
SF 125.7577741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

500 MHz ^{1}H NMR spectrum in $CDCl_3$



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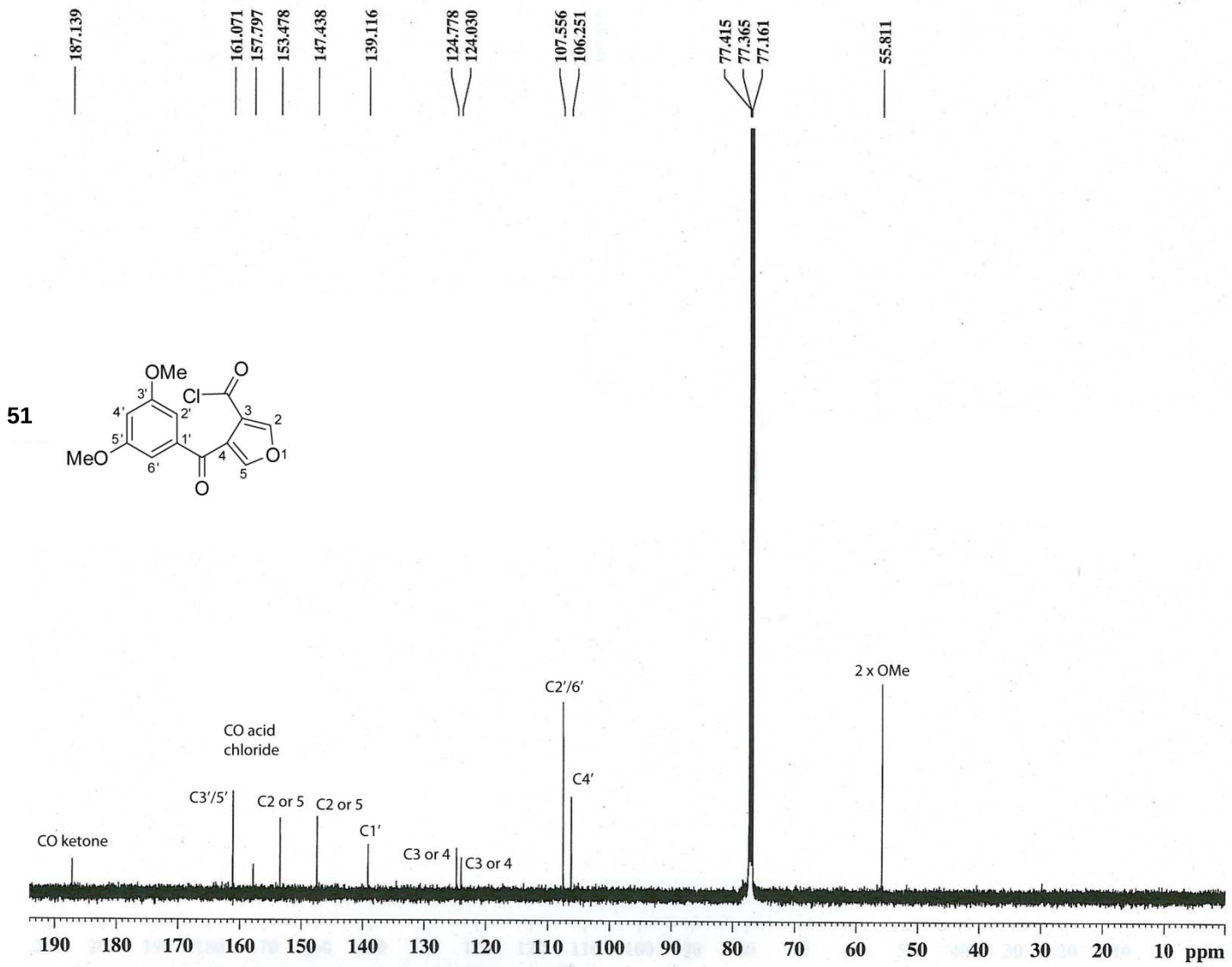
Current Data Parameters
NAME kap04–142–02
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20100506
Time 17.45
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8220063 sec
RG 2298.8
DW 55.600 usec
DE 7.50 usec
TE 298.0 K
D1 0.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.70 usec
PL1 −1.00 dB
SFO1 500.1327500 MHz

F2 – Processing parameters
SI 16384
SF 500.1300231 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.20

125.7 MHz ^{13}C NMR spectrum in $CDCl_3$



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Current Data Parameters
NAME kap04–142–02
EXPNO 3
PROCNO 1

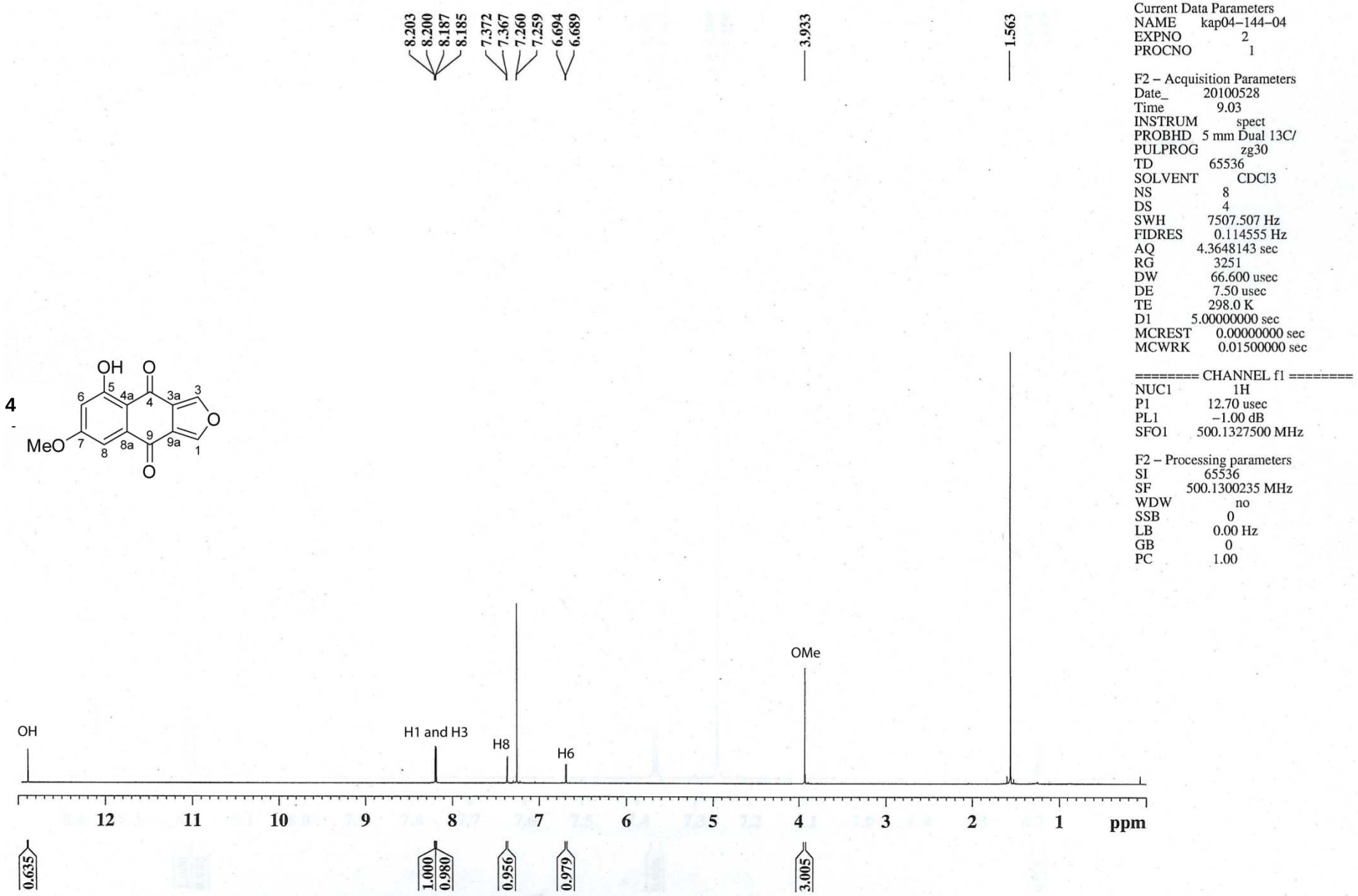
F2 – Acquisition Parameters
Date_ 20100506
Time 17.51
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 17313
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 23170.5
DW 15.900 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 125.7709890 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 500.1322500 MHz

F2 – Processing parameters
SI 65536
SF 125.7577736 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

500 MHz ^{1}H NMR spectrum in $CDCl_3$



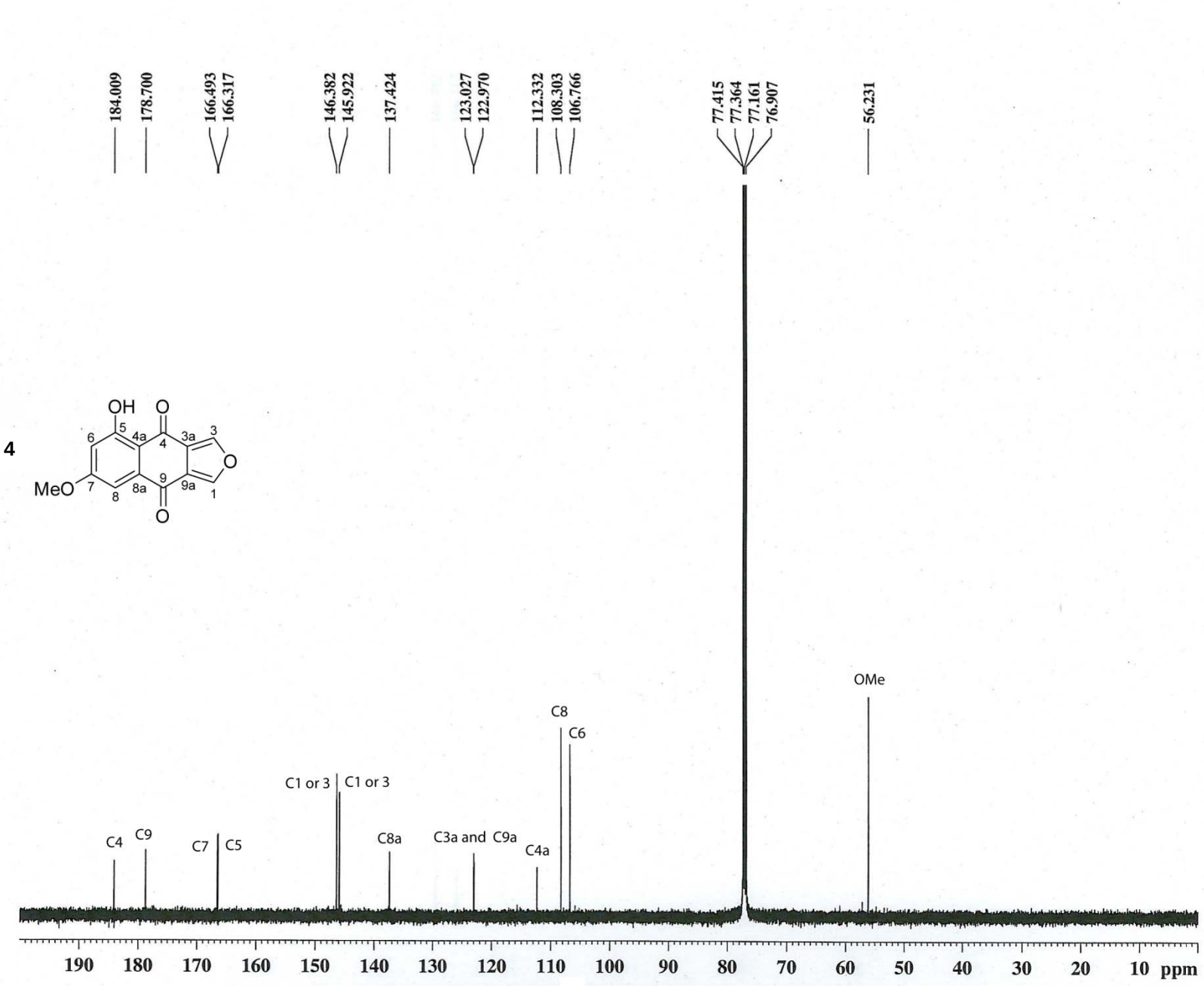
Current Data Parameters
NAME kap04–144–04
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20100528
Time 9.03
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 4
SWH 7507.507 Hz
FIDRES 0.114555 Hz
AQ 4.3648143 sec
RG 3251
DW 66.600 usec
DE 7.50 usec
TE 298.0 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.70 usec
PL1 –1.00 dB
SFO1 500.1327500 MHz

F2 – Processing parameters
SI 65536
SF 500.1300235 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

125.7 MHz ^{13}C NMR spectrum in $CDCl_3$



Current Data Parameters
NAME kap04–144–04
EXPNO 3
PROCNO 1

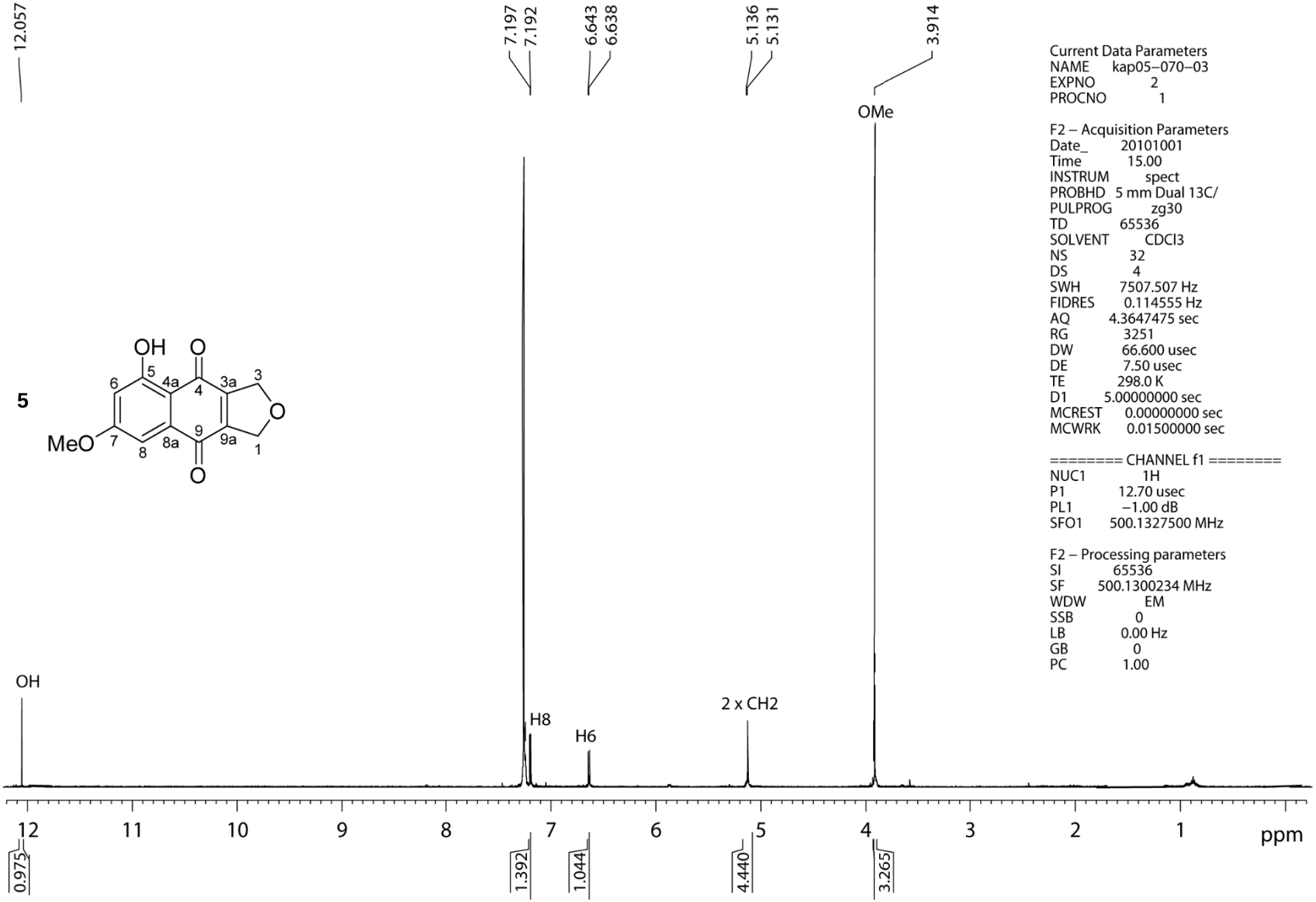
F2 – Acquisition Parameters
Date_ 20100527
Time 17.32
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 14976
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420883 sec
RG 20642.5
DW 15.900 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 125.7709890 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.00 dB
PL13 20.00 dB
SFO2 500.1322500 MHz

F2 – Processing parameters
SI 65536
SF 125.7577736 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

500 MHz ^{1}H NMR spectrum in $CDCl_3$



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