

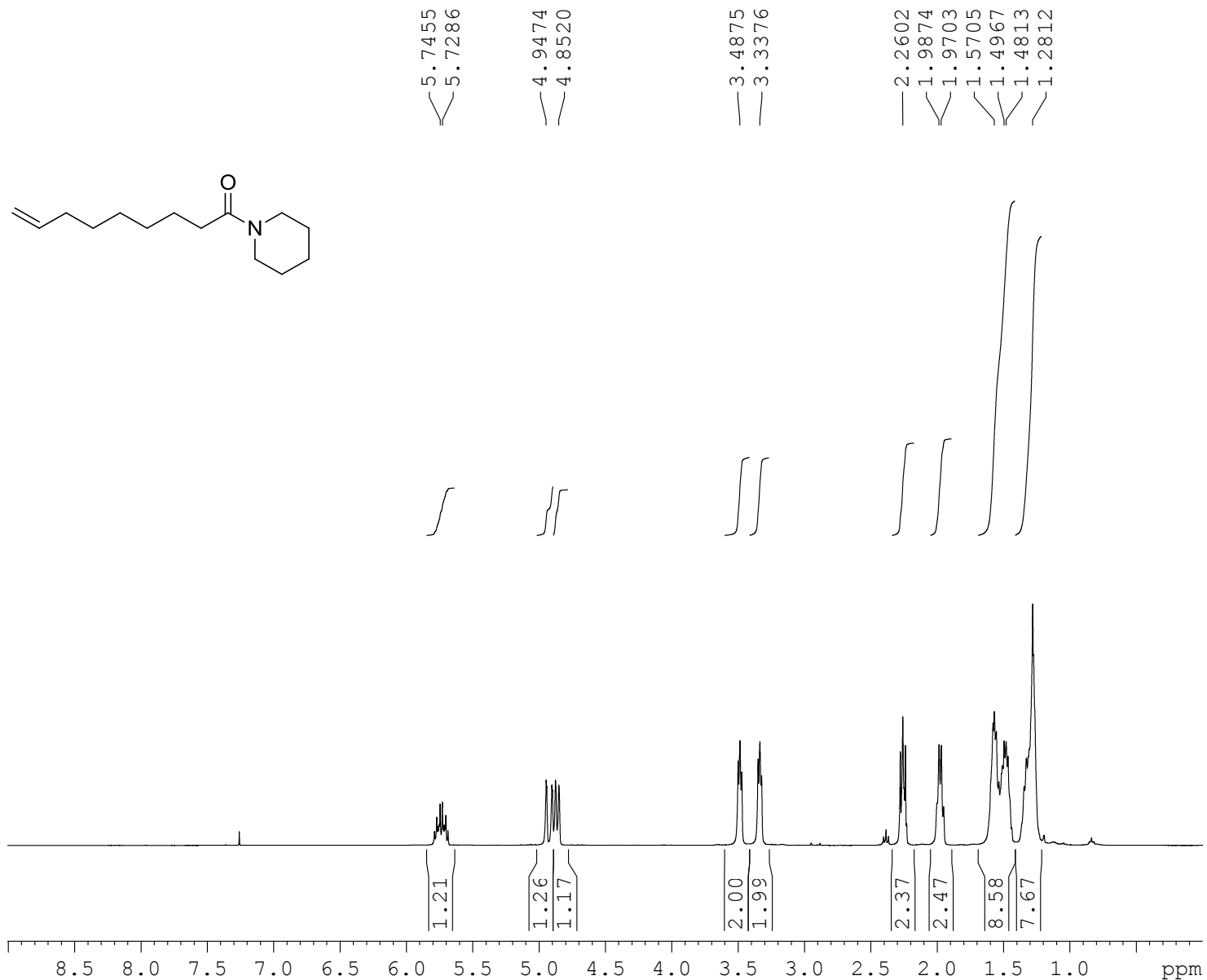
## **Piperolein B, isopiperolein B and piperamide C9:1(8E): total synthesis and cytotoxicities**

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<sup>1</sup>H NMR spectrum of 1-(piperidin-1-yl)non-8-en-1-one, **6**



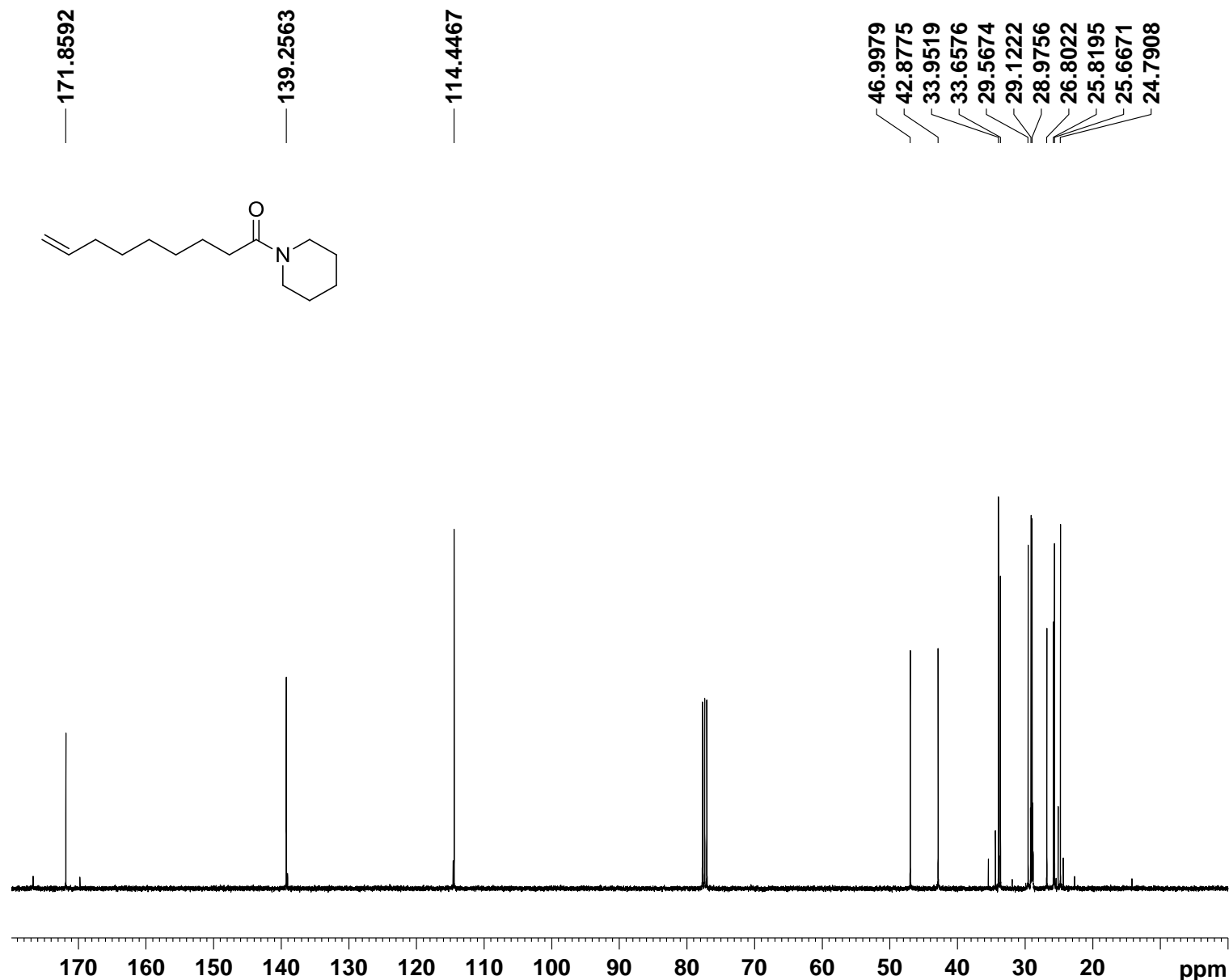
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NAME WMFK-45  
EXPNO 20  
PROCNO 1

F2 - Acquisition Parameters  
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INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 15.2  
DW 60.800 usec  
DE 10.69 usec  
TE 293.9 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1324710 MHz  
NUC1 1H  
P1 8.00 usec  
PLW1 24.00000000 W

F2 - Processing parameters  
SI 131072  
SF 400.1300103 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.50

<sup>13</sup>C NMR spectrum of 1-(piperidin-1-yl)non-8-en-1-one, **6**



Current Data Parameters  
NAME WMFK-45  
EXPNO 21  
PROCNO 1

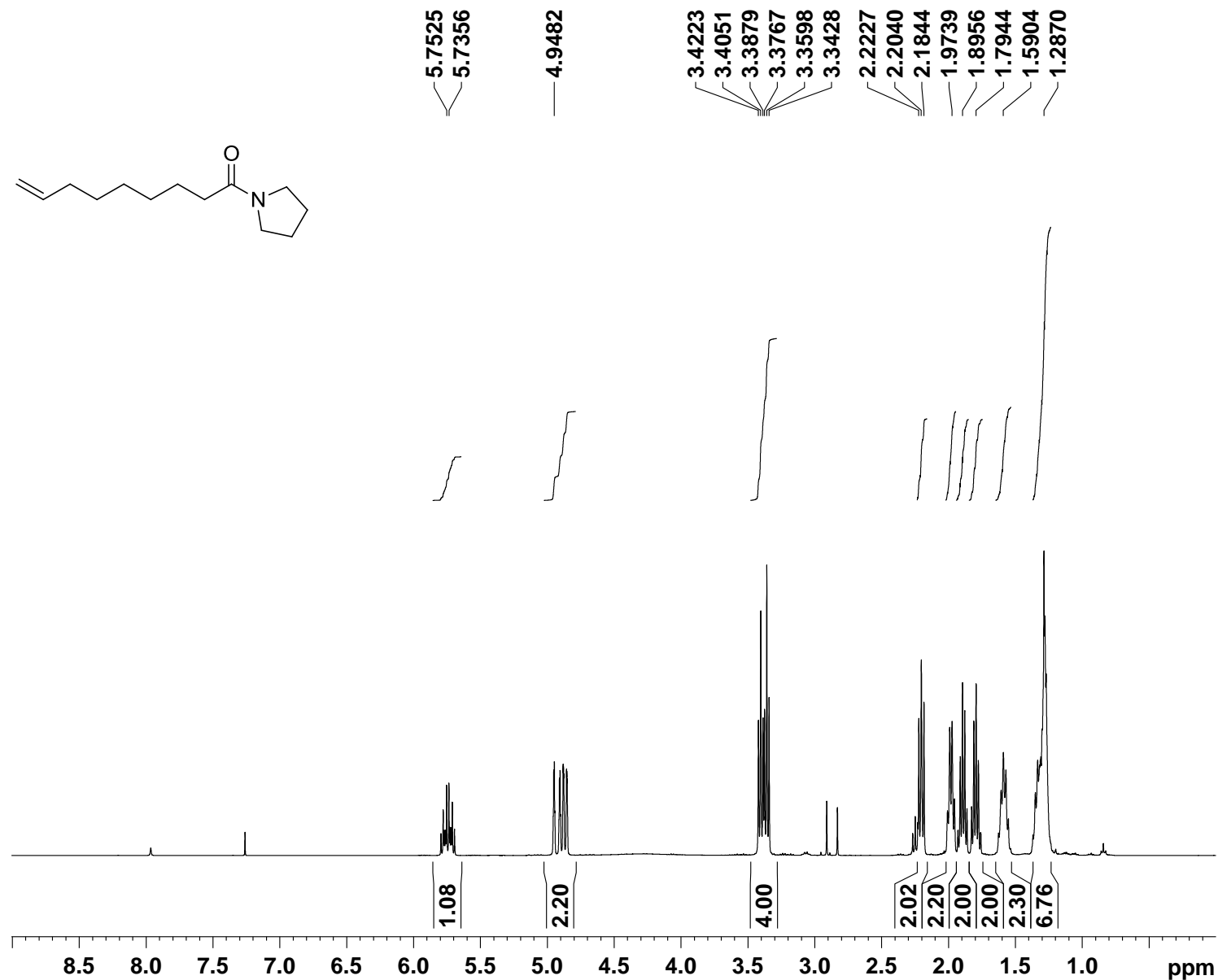
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PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 181.72  
DW 20.800 usec  
DE 8.18 usec  
TE 294.3 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6228293 MHz  
NUC1 13C  
P1 9.00 usec  
PLW1 77.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1316005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 24.00000000 W  
PLW12 0.17567000 W  
PLW13 0.14229999 W

F2 - Processing parameters  
SI 65536  
SF 100.6127429 MHz  
WDW EM  
SSB 0  
LB 0.50 Hz  
GB 0  
PC 1.40

<sup>1</sup>H NMR spectrum of 1-(pyrrolidin-1-yl)non-8-en-1-one, 7



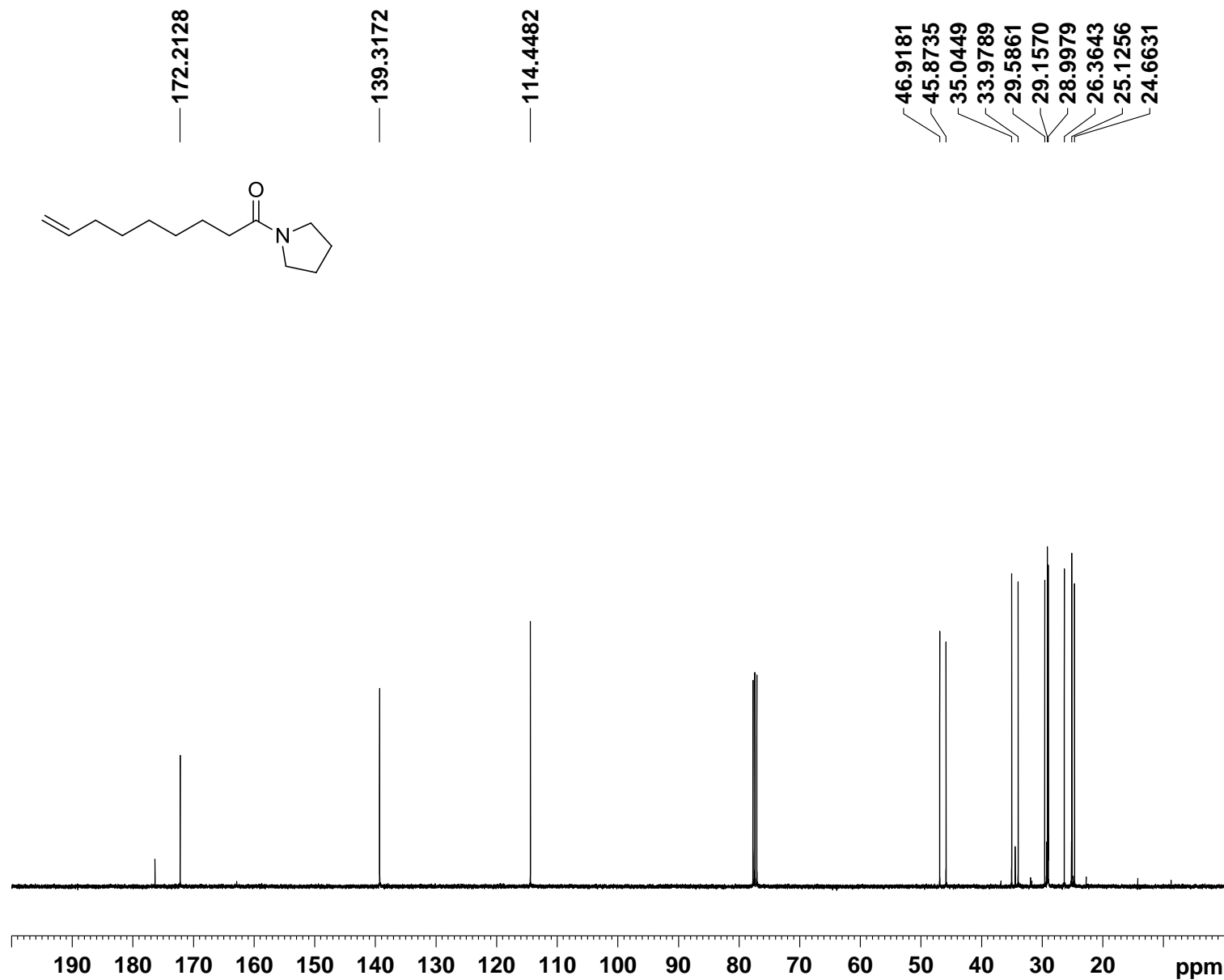
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PROCNO 1

F2 - Acquisition Parameters  
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PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.984589 sec  
RG 20.45  
DW 60.800 usec  
DE 10.69 usec  
TE 293.9 K  
D1 1.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 400.1324710 MHz  
NUC1 1H  
P1 8.00 usec  
PLW1 24.00000000 W

F2 - Processing parameters  
SI 131072  
SF 400.1300099 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.50

<sup>13</sup>C NMR spectrum of 1-(pyrrolidin-1-yl)non-8-en-1-one, 7



```
Current Data Parameters
NAME      WMFK-44
EXPNO     21
PROCNO    1

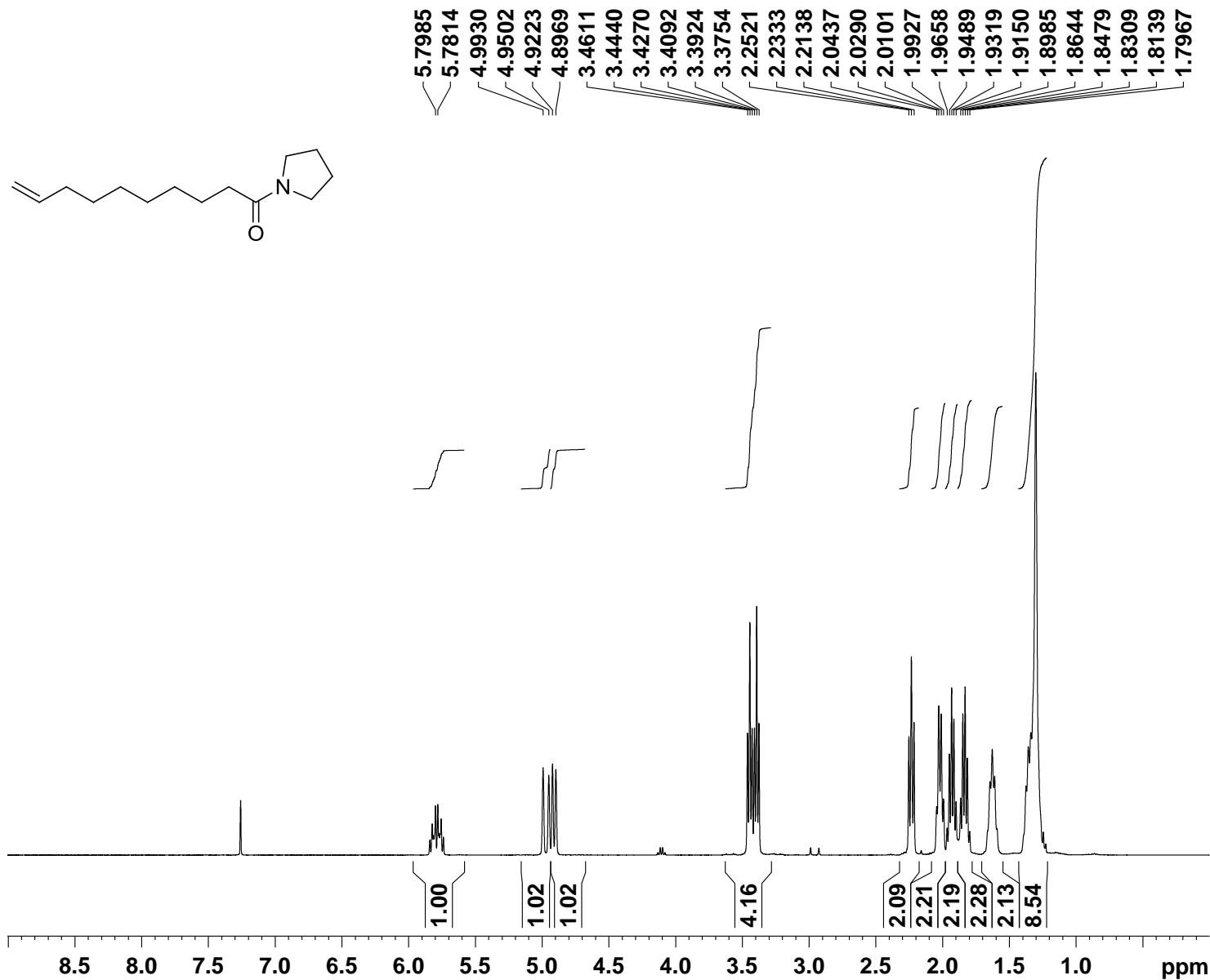
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PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
NS        256
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631488 sec
RG        181.72
DW        20.800 usec
DE        8.18 usec
TE        294.3 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1      13C
P1        9.00 usec
PLW1      77.0000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2      1H
CPDPRG[2] waltz16
PCPD2     90.00 usec
PLW2      24.0000000 W
PLW12     0.1756700 W
PLW13     0.14229999 W

F2 - Processing parameters
SI        65536
SF        100.6127412 MHz
WDW       EM
SSB       0
LB        0.50 Hz
GB        0
PC        1.40
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<sup>1</sup>H NMR spectrum of 1-(pyrrolidin-1-yl)dec-9-en-1-one, **8**



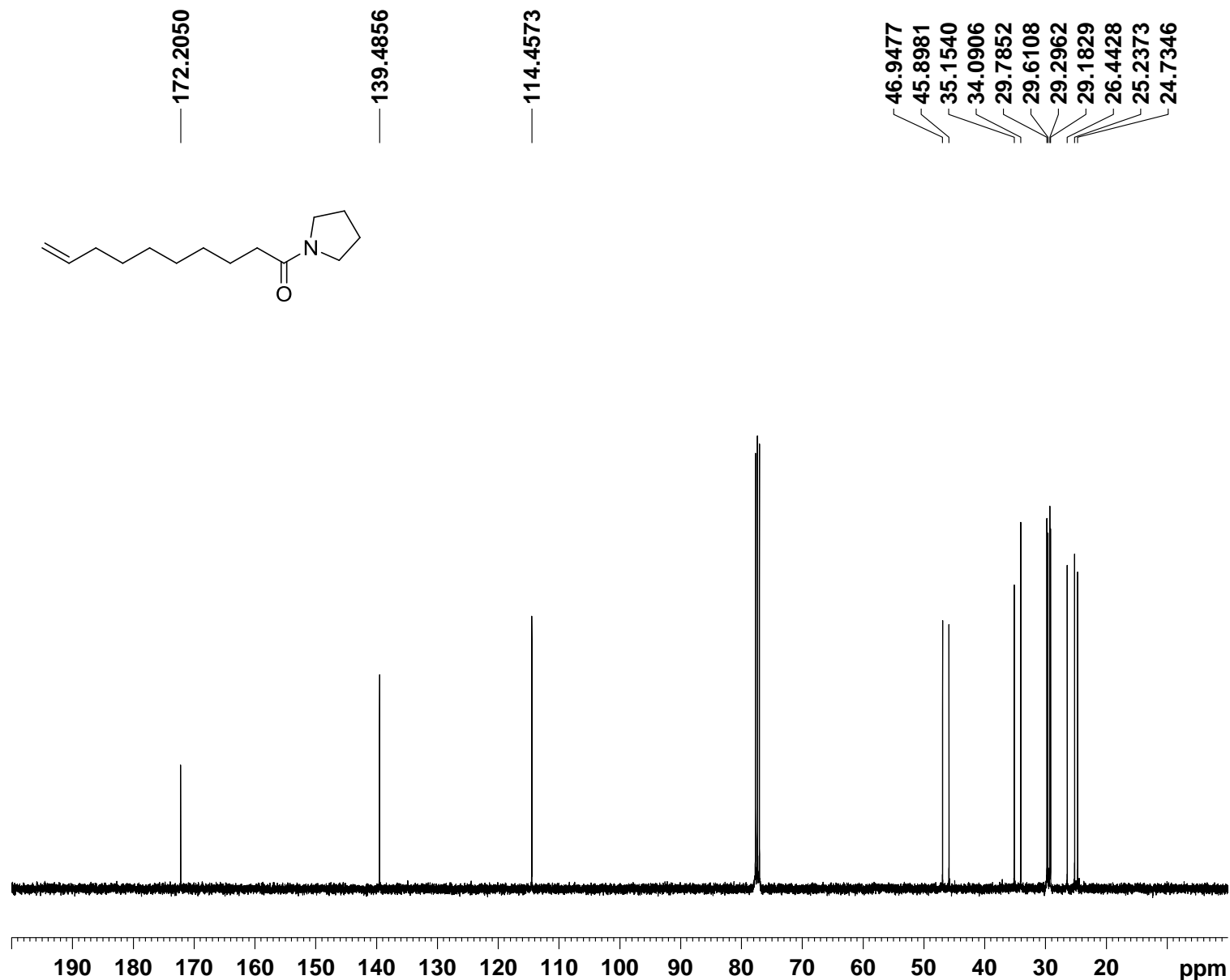
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PROCNO 1

F2 - Acquisition Parameters  
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PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 59.2  
DW 60.800 usec  
DE 10.69 usec  
TE 293.8 K  
D1 1.0000000 sec  
TDO 1

==== CHANNEL f1 =====  
SFO1 400.1324710 MHz  
NUC1 1H  
P1 8.00 usec  
PLW1 24.0000000 W

F2 - Processing parameters  
SI 131072  
SF 400.1300103 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.50

<sup>13</sup>C NMR spectrum of 1-(pyrrolidin-1-yl)dec-9-en-1-one, **8**



```
Current Data Parameters
NAME WM FK-11
EXPNO 10
PROCNO 1

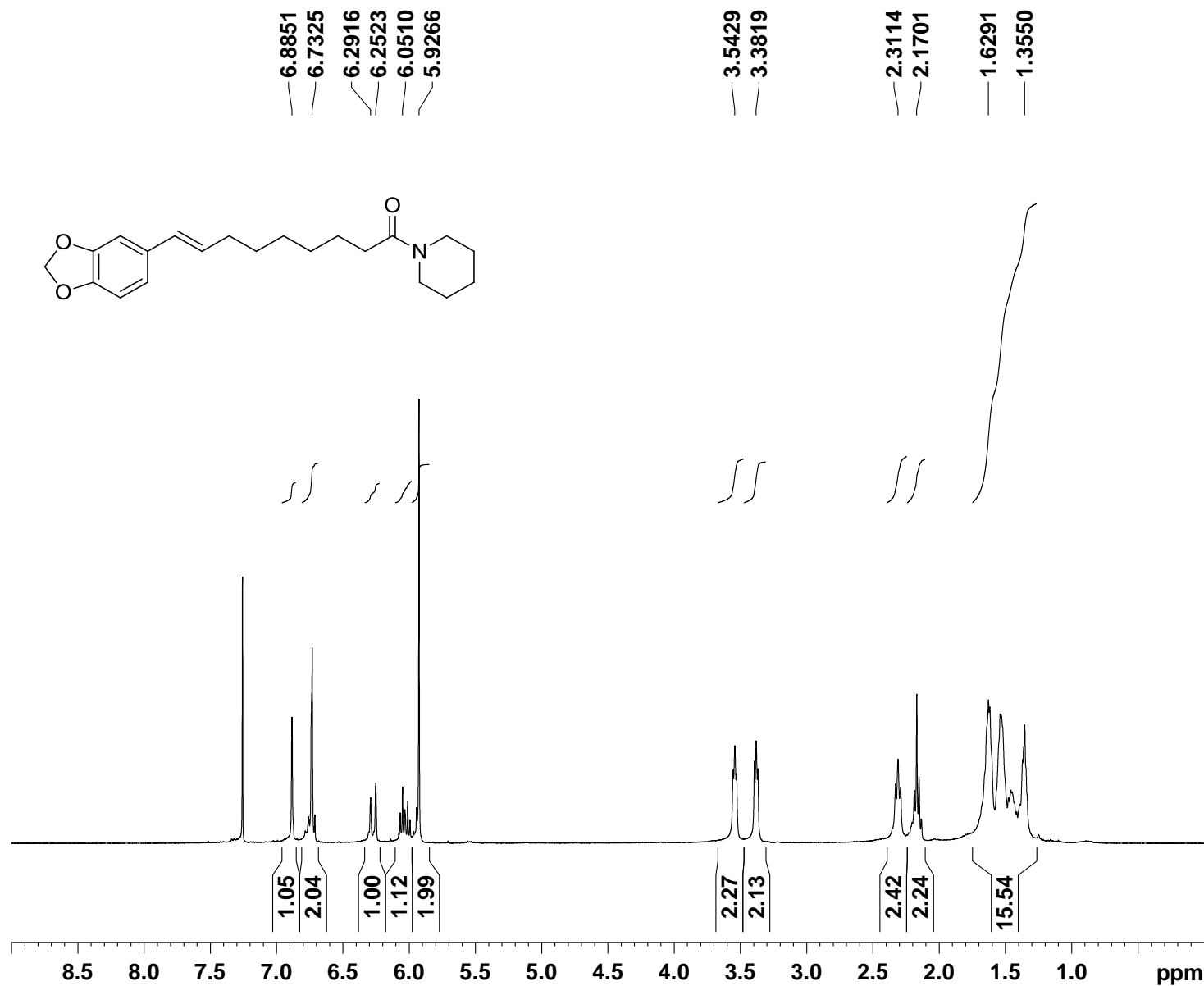
F2 - Acquisition Parameters
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Time_ 11.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 181.72
DW 20.800 usec
DE 8.18 usec
TE 294.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228293 MHz
NUC1 13C
P1 9.00 usec
PLW1 77.00000000 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 24.00000000 W
PLW12 0.17567000 W
PLW13 0.14229999 W

F2 - Processing parameters
SI 65536
SF 100.6127374 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40
```

<sup>1</sup>H NMR spectrum of piperolein B, **1**



Current Data Parameters  
NAME ARM FK-50  
EXPNO 1  
PROCNO 1

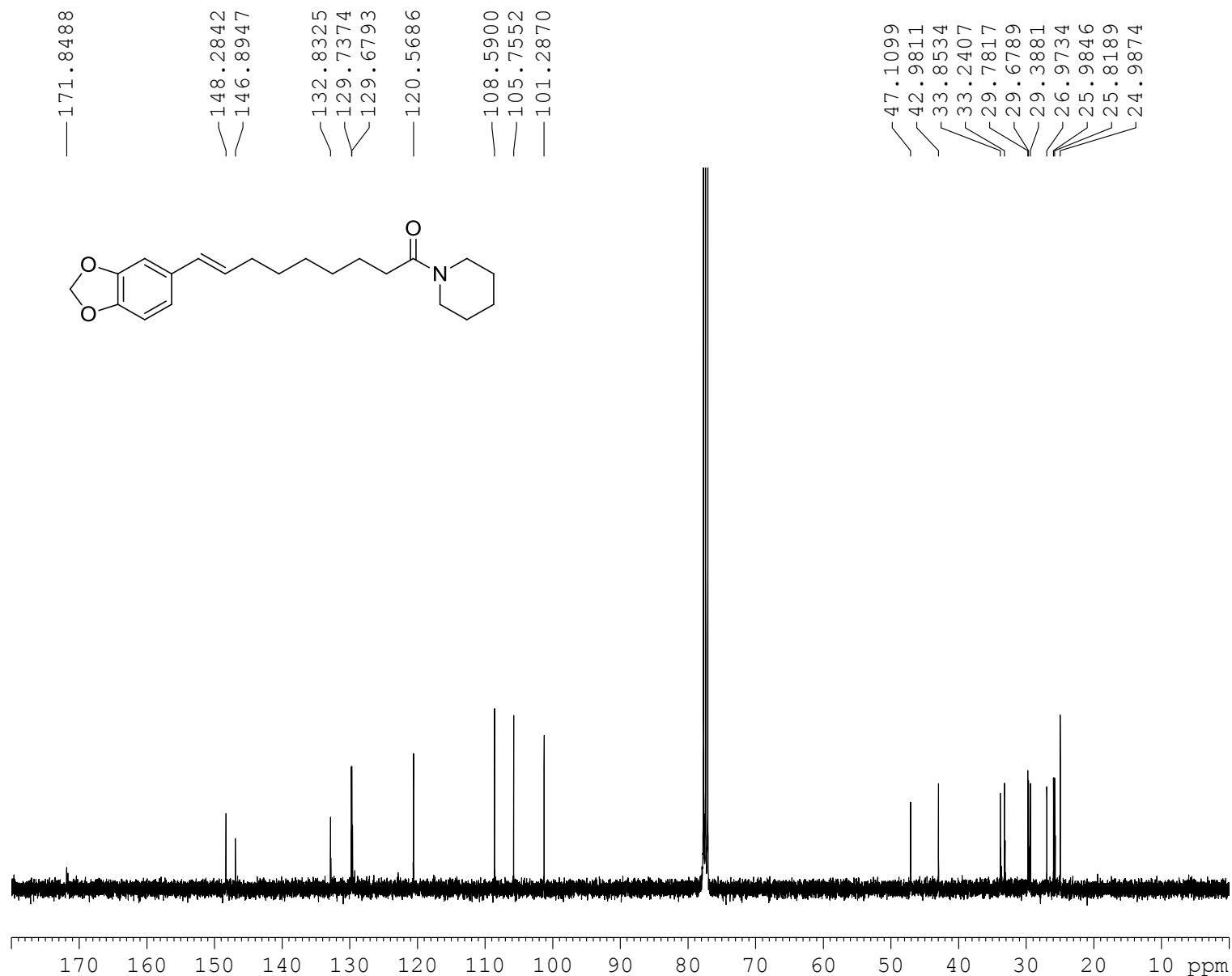
F2 - Acquisition Parameters  
Date\_ 20121218  
Time 15.31  
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PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 104.33  
DW 60.800 usec  
DE 10.69 usec  
TE 294.0 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
SFO1 400.1324710 MHz  
NUC1 1H  
P1 8.00 usec  
PLW1 24.00000000 W

F2 - Processing parameters  
SI 131072  
SF 400.1300104 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.50



<sup>13</sup>C NMR spectrum of piperolein B, **1**



Current Data Parameters  
NAME ARM FK-50  
EXPNO 3  
PROCNO 1

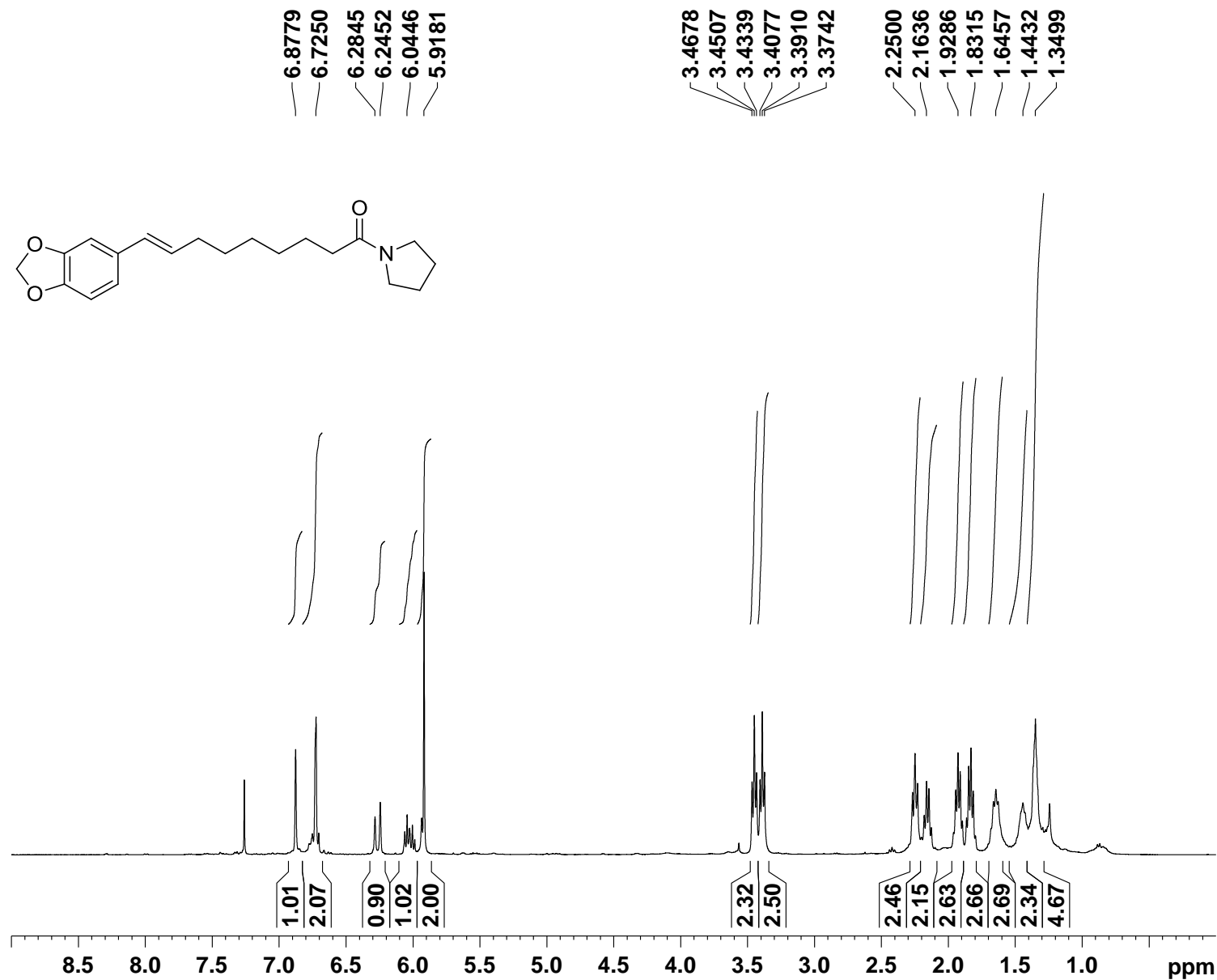
F2 - Acquisition Parameters  
Date\_ 20121218  
Time\_ 20.20  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 181.72  
DW 20.800 usec  
DE 8.18 usec  
TE 294.6 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 100.6228293 MHz  
NUC1 13C  
P1 9.00 usec  
PLW1 77.00000000 W

==== CHANNEL f2 =====  
SFO2 400.1316005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 24.00000000 W  
PLW12 0.17567000 W  
PLW13 0.14229999 W

F2 - Processing parameters  
SI 65536  
SF 100.6127312 MHz  
WDW EM  
SSB 0  
LB 0.50 Hz  
GB 0  
PC 1.40

<sup>1</sup>H NMR spectrum of piperamide C9:1(8E), **2**



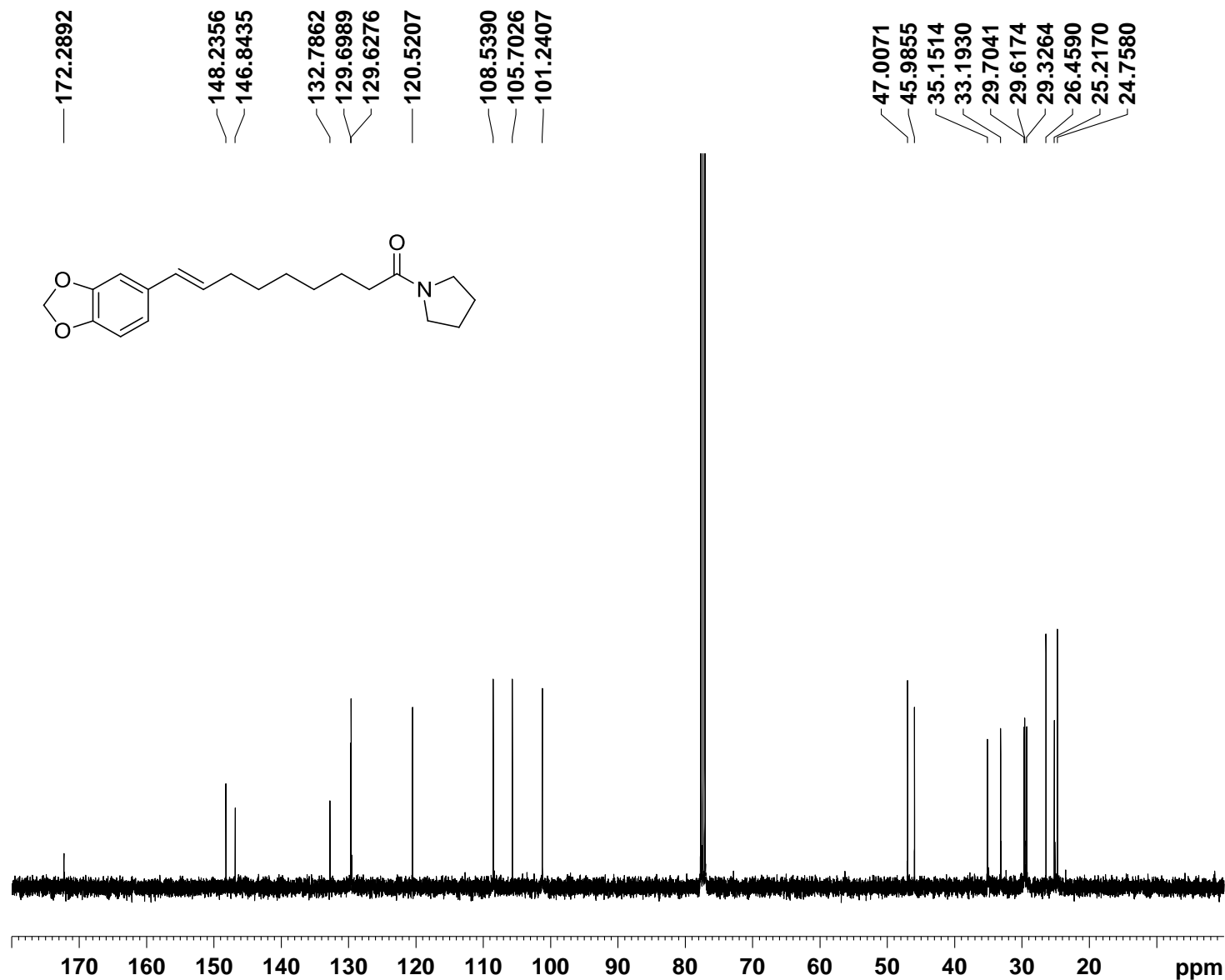
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NAME ARM FK-49  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20121217  
Time\_ 12.25  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 56.49  
DW 60.800 usec  
DE 10.69 usec  
TE 294.0 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
SF01 400.1324710 MHz  
NUC1 1H  
P1 8.00 usec  
PLW1 24.00000000 W

F2 - Processing parameters  
SI 131072  
SF 400.1300093 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.50

<sup>13</sup>C NMR spectrum of piperamide C9:1(8E), 2



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EXPNO         11
PROCNO        1

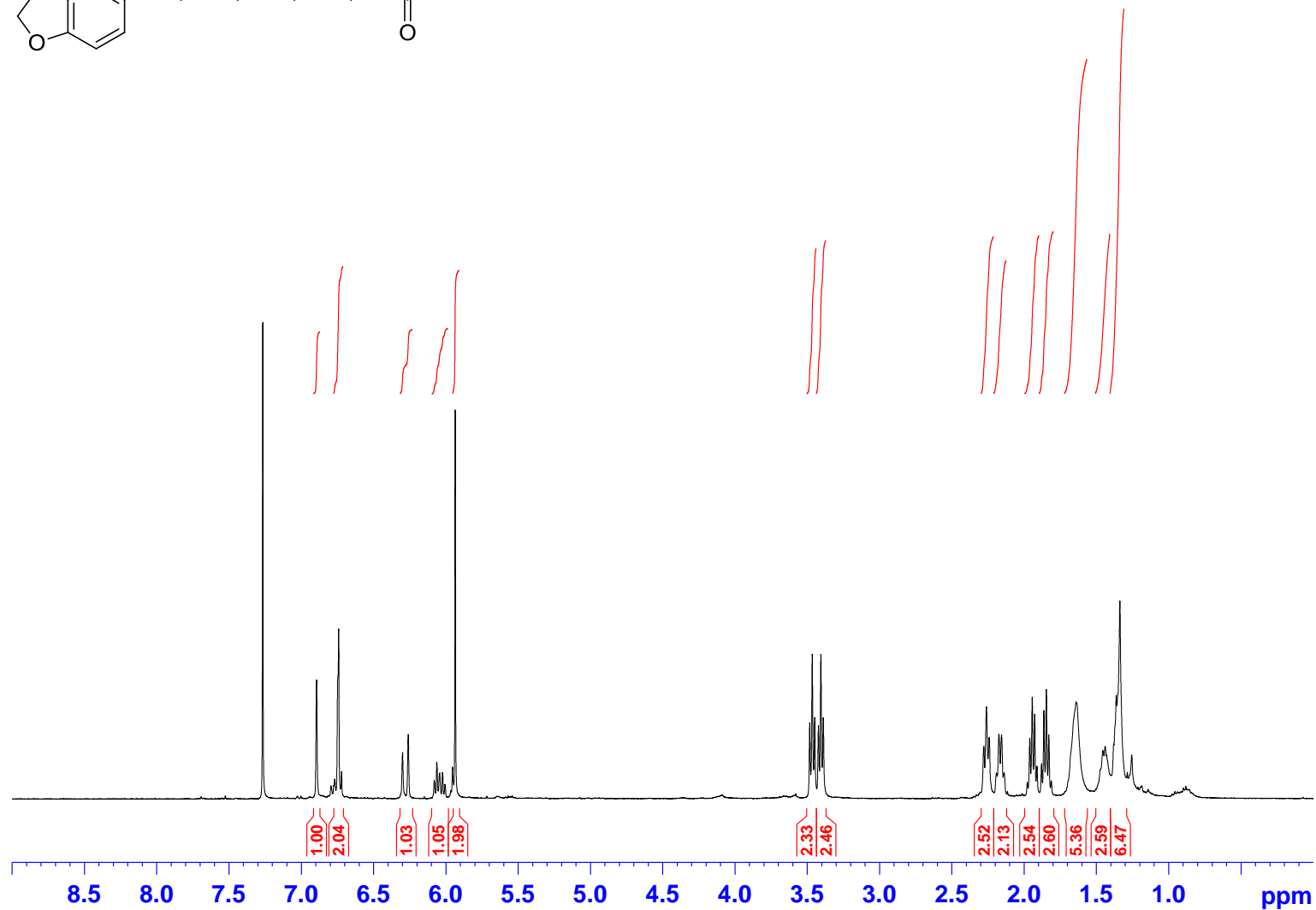
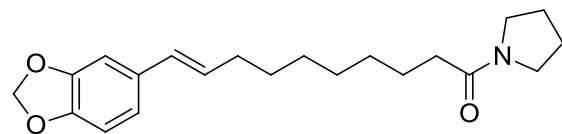
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Time          12.41
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG            181.72
DW            20.800 usec
DE            8.18 usec
TE            294.3 K
D1            2.0000000 sec
D11           0.0300000 sec
TDO           1

===== CHANNEL f1 =====
SFO1          100.6228293 MHz
NUC1          13C
P1            9.00 usec
PLW1          77.0000000 W

===== CHANNEL f2 =====
SFO2          400.1316005 MHz
NUC2          1H
CPDPRG[2]    waltz16
PCPD2        90.00 usec
PLW2          24.0000000 W
PLW12         0.1756700 W
PLW13         0.14229999 W

F2 - Processing parameters
SI            65536
SF            100.6127352 MHz
WDW           EM
SSB           0
LB            0.50 Hz
GB            0
PC            1.40
```

<sup>1</sup>H NMR spectrum of isopiperolein B, **3**



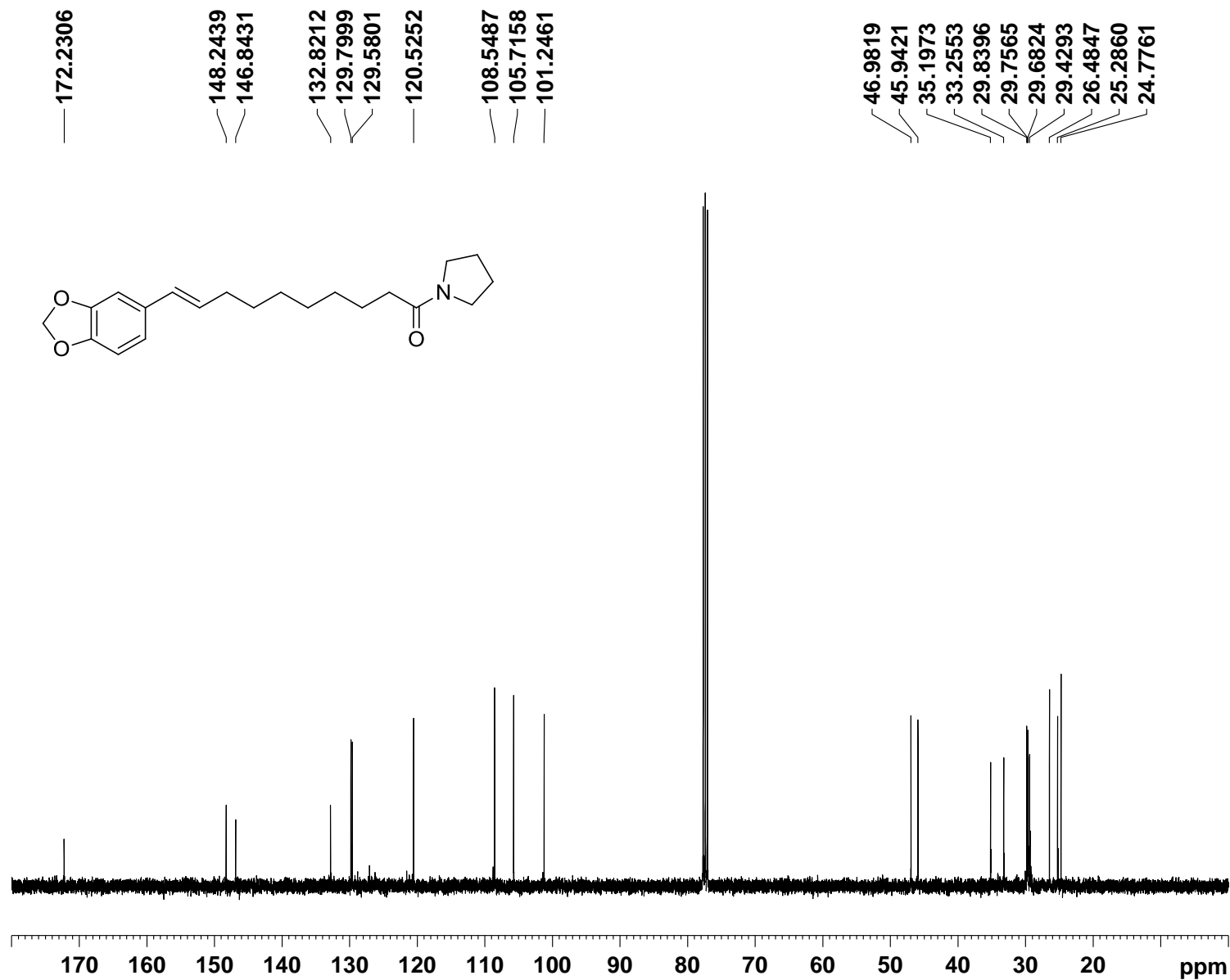
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EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20121217  
Time 12.05  
INSTRUM spect  
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FULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 134.04  
DW 60.800 usec  
DE 10.69 usec  
TE 293.8 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 400.1324710 MHz  
NUC1 1H  
P1 8.00 usec  
PLW1 24.00000000 W

F2 - Processing parameters  
SI 131072  
SF 400.1300075 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.50

<sup>13</sup>C NMR spectrum of isopiperolein B, 3



Current Data Parameters  
NAME WM FK-36  
EXPNO 90  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120802  
Time\_ 14.28  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 181.72  
DW 20.800 usec  
DE 8.18 usec  
TE 294.4 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 100.6228293 MHz  
NUC1 13C  
P1 9.00 usec  
PLW1 77.0000000 W

===== CHANNEL f2 =====  
SFO2 400.1316005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 90.00 usec  
PLW2 24.0000000 W  
PLW12 0.17567000 W  
PLW13 0.14229999 W

F2 - Processing parameters  
SI 65536  
SF 100.6127345 MHz  
WDW EM  
SSB 0  
LB 0.50 Hz  
GB 0  
PC 1.40