

Supplementary Material

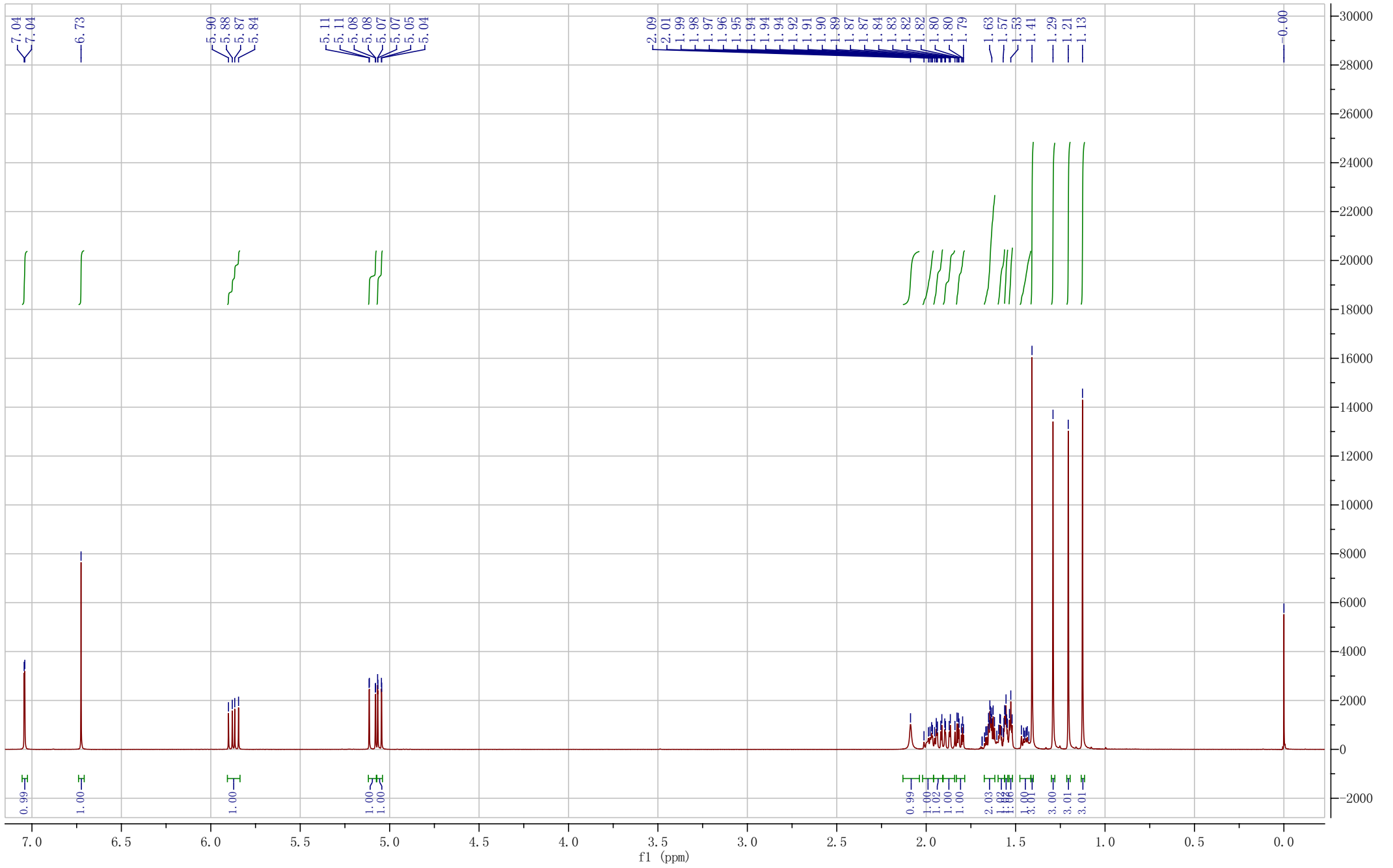
Manuscript ID: marinedrugs-13384

Type of manuscript: Article

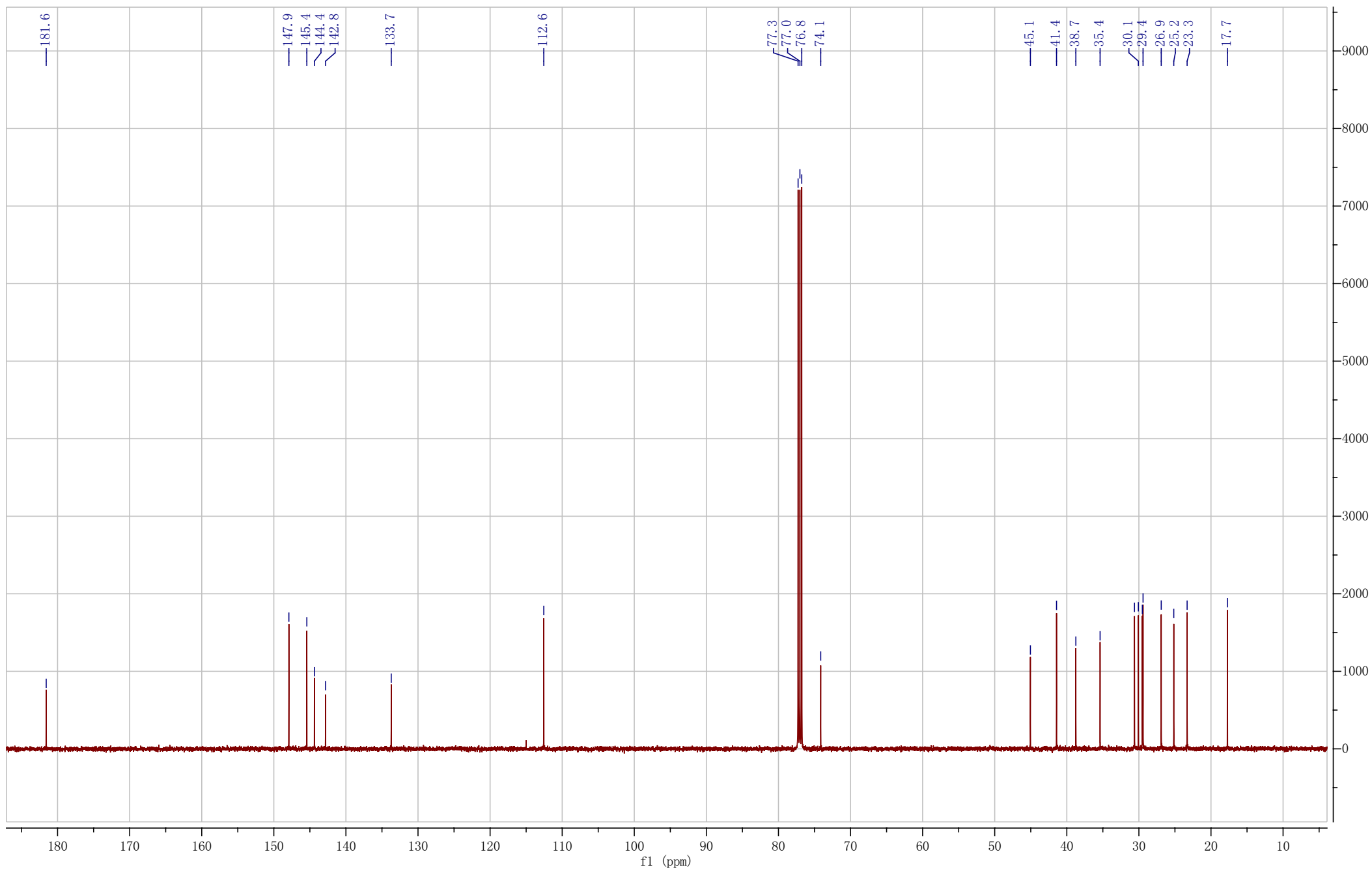
Title: Scopararanes C–G: New Oxygenated Pimarane Diterpenes from the marine sediment-derived fungus *Eutypella scoparia* FS26

Authors: Li Sun, Dongli Li, Meihua Tao, Yuchan Chen, Feijun Dan, Weimin Zhang

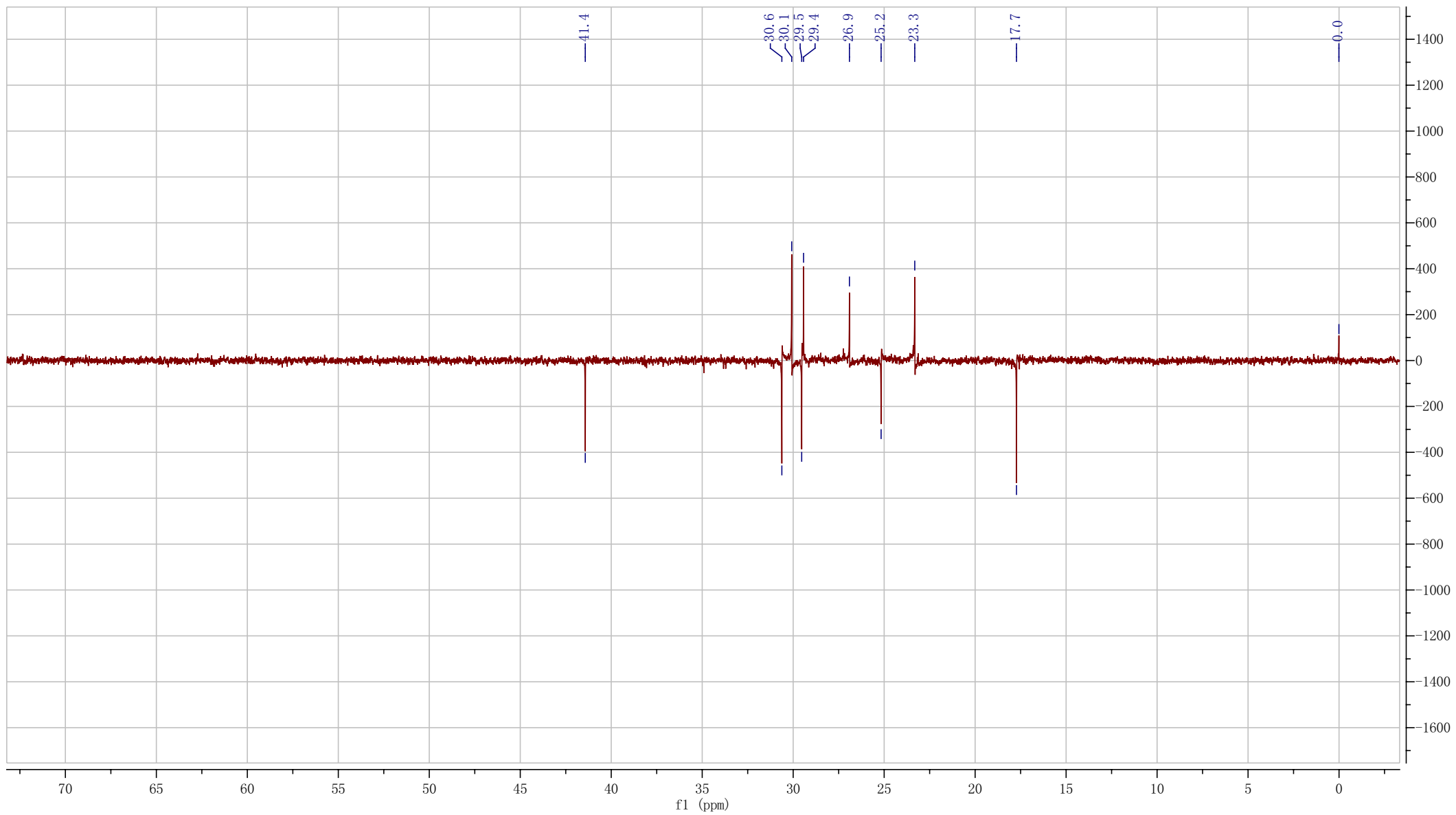
1H-NMR of scopararane C



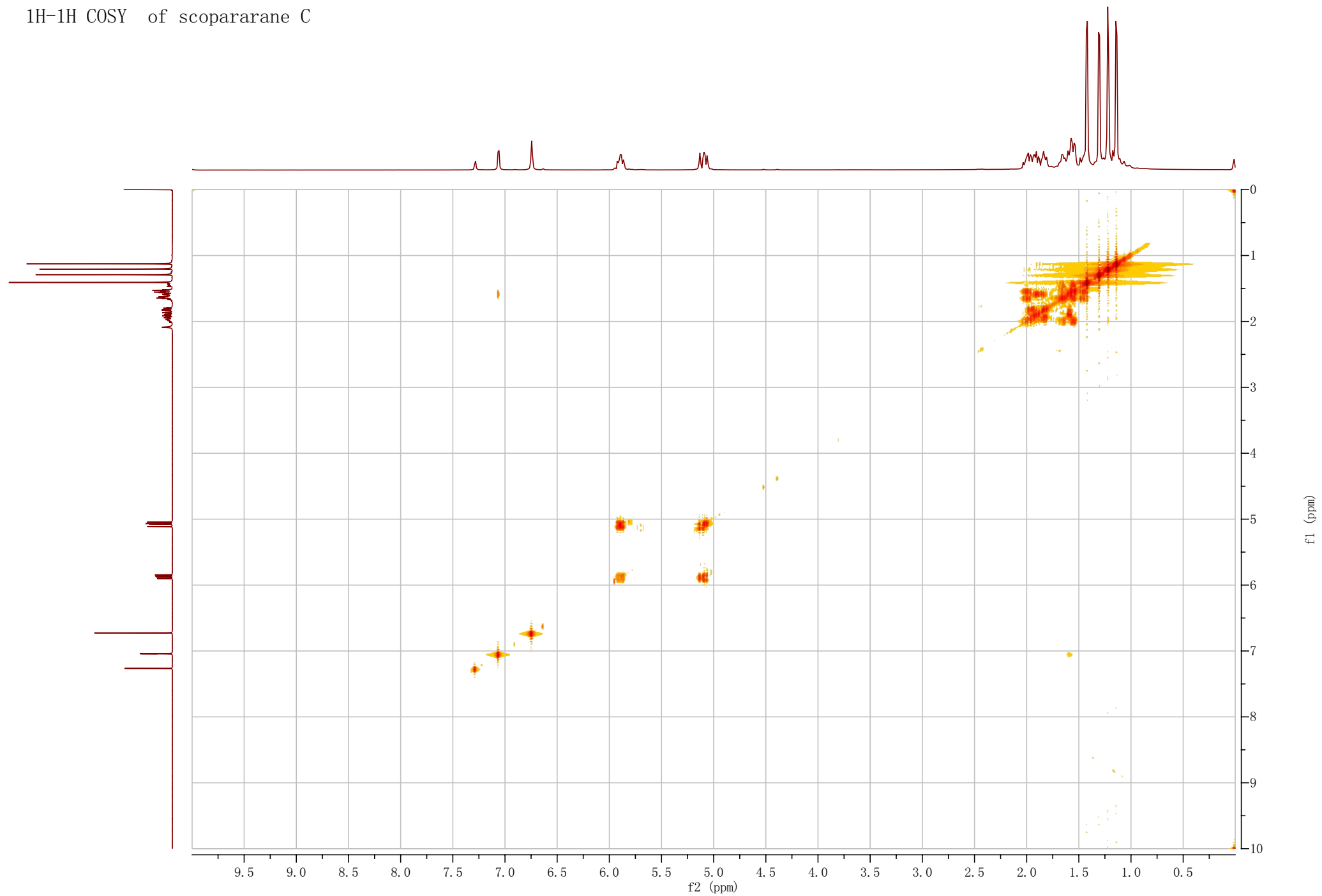
¹³C-NMR of scopararane C



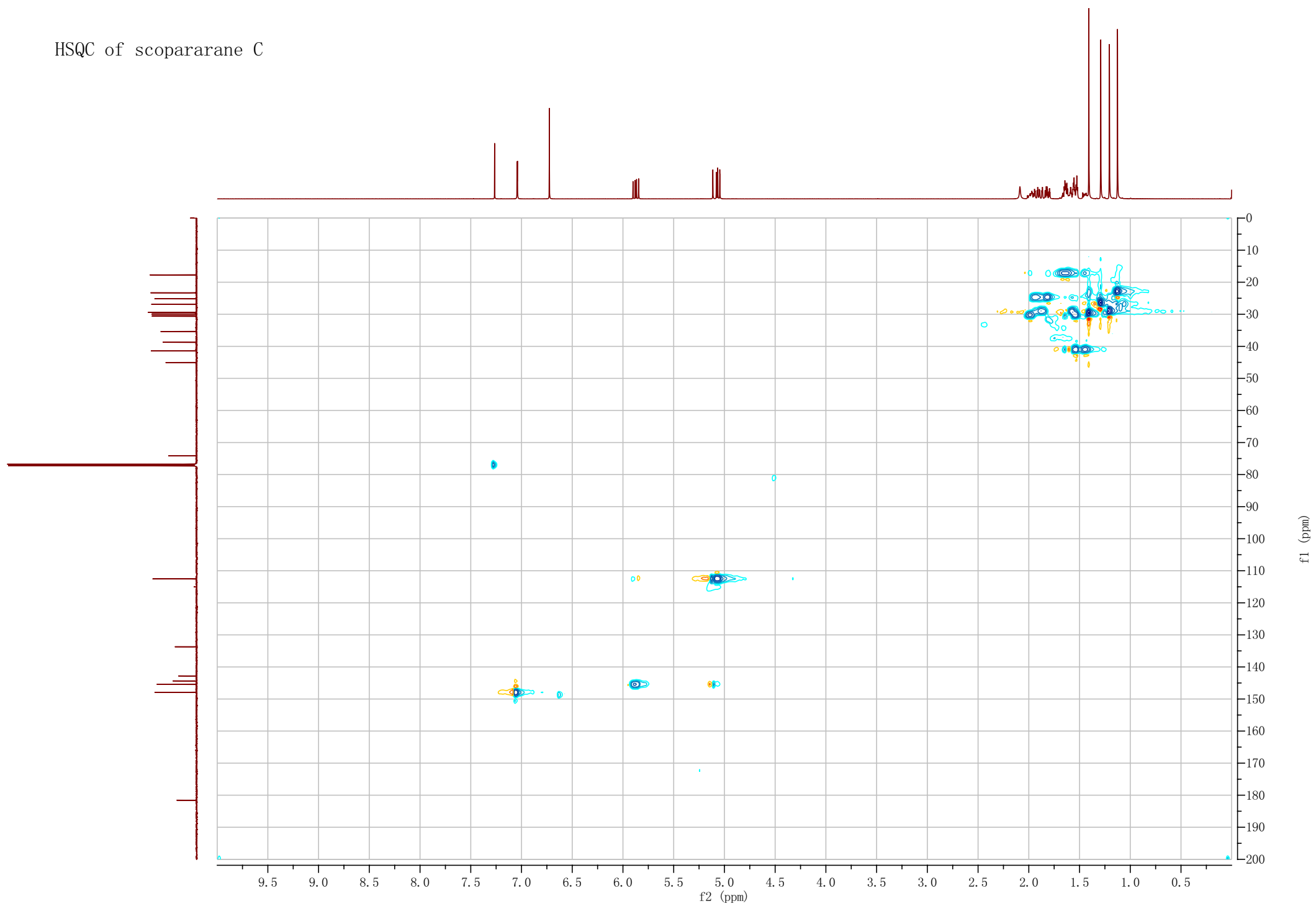
DEPT 135 of scopararane C



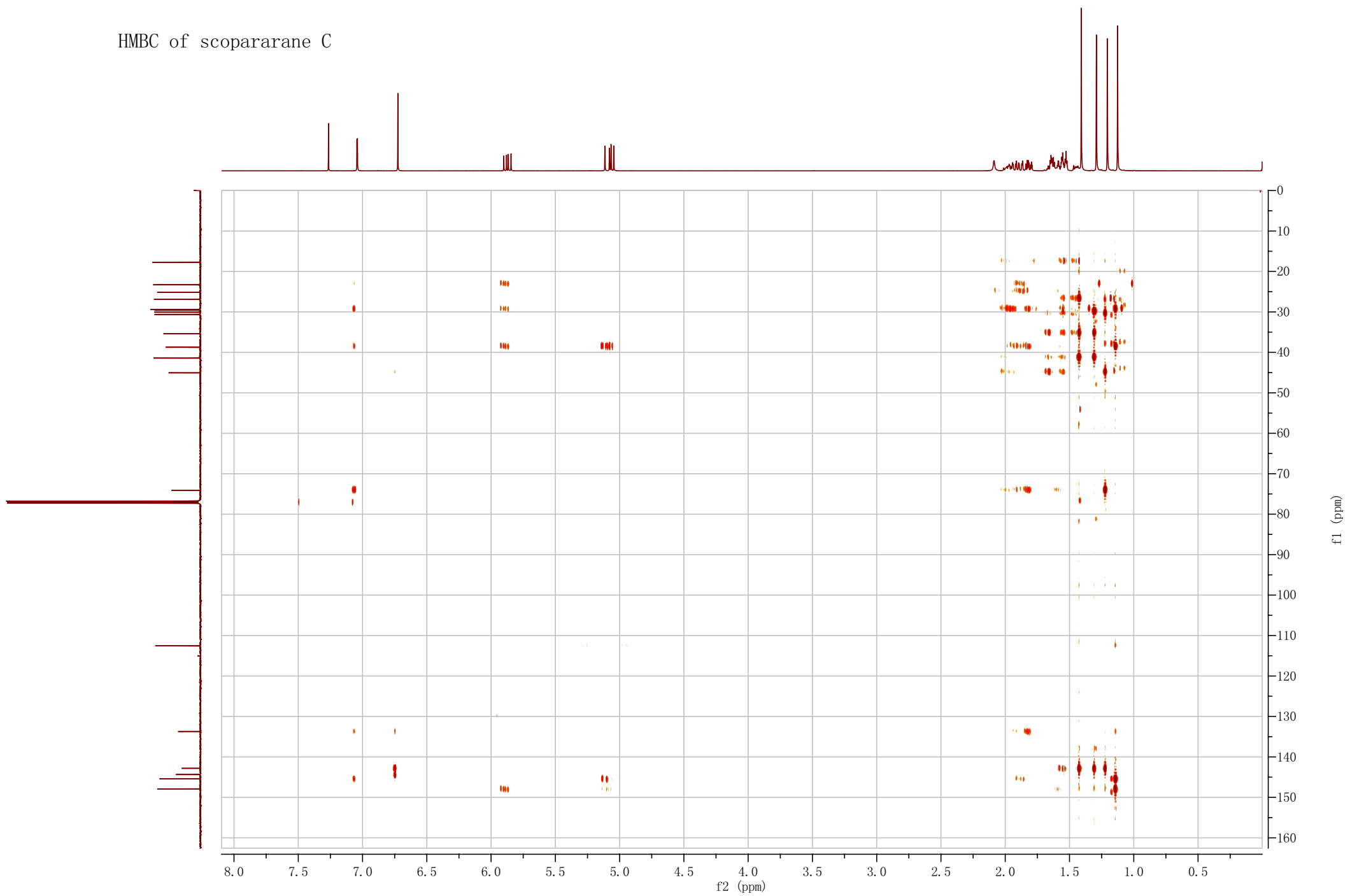
¹H-¹H COSY of scopararane C



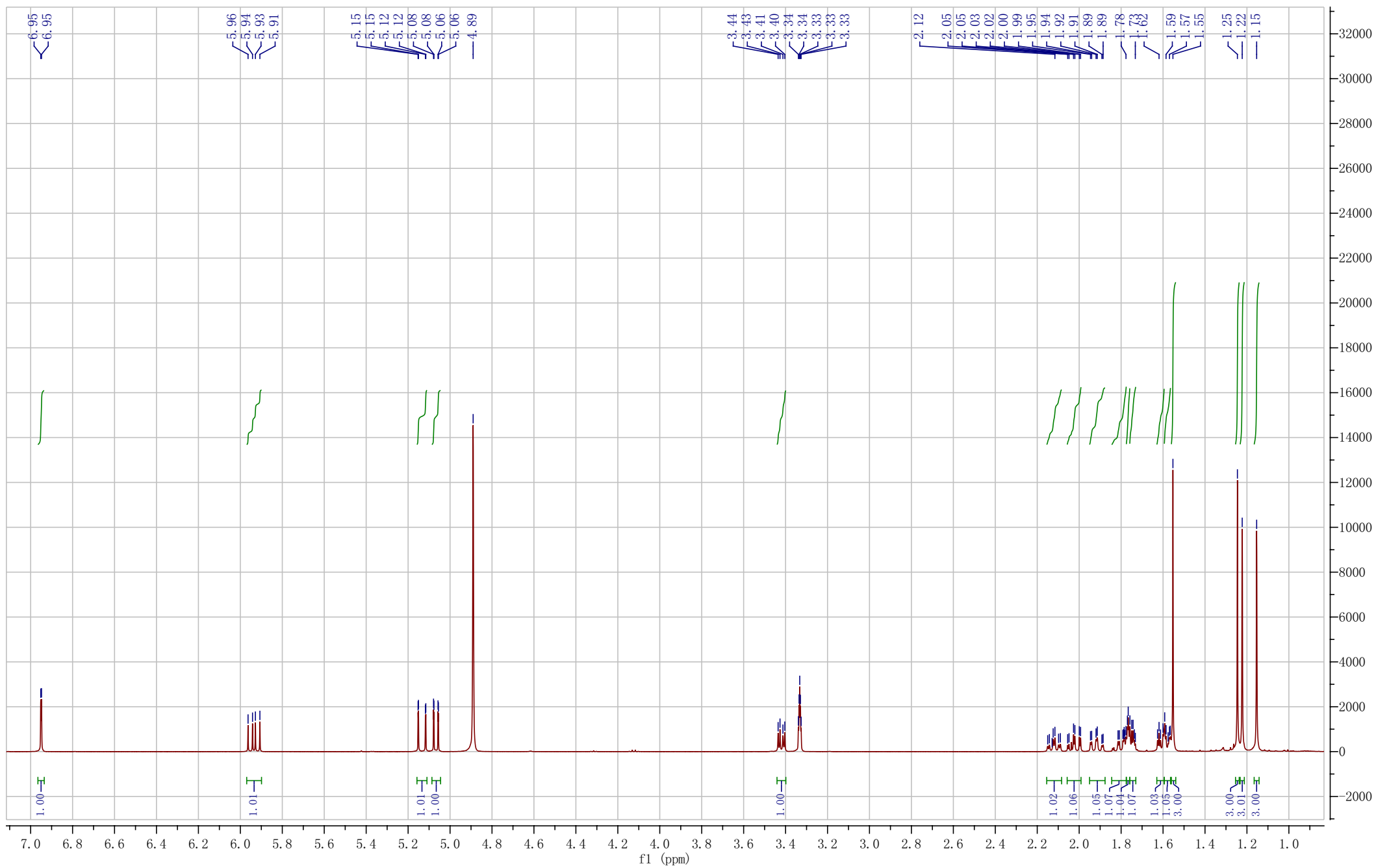
HSQC of scopararane C



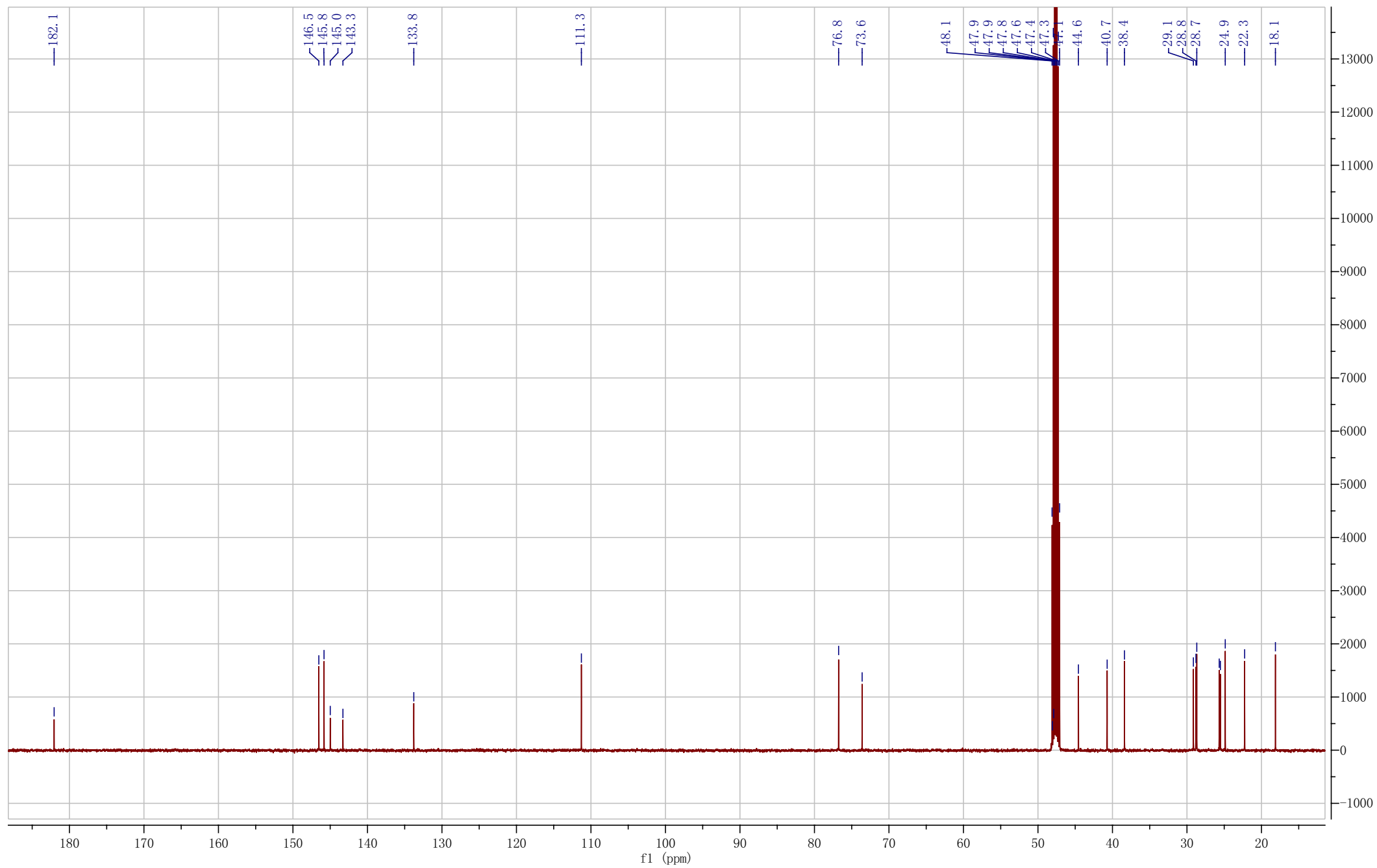
HMBC of scopararane C



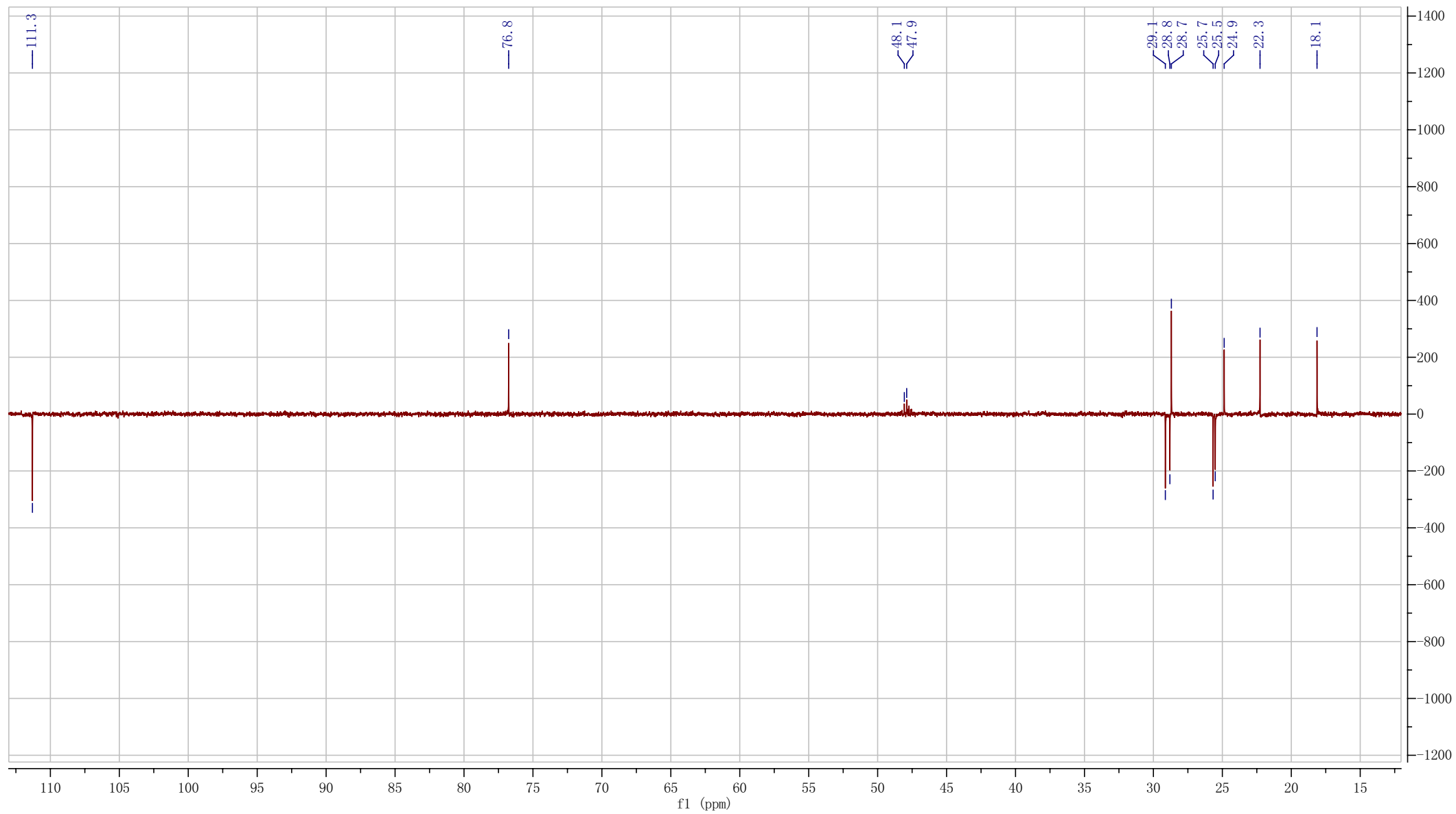
¹H-NMR of scopararane D



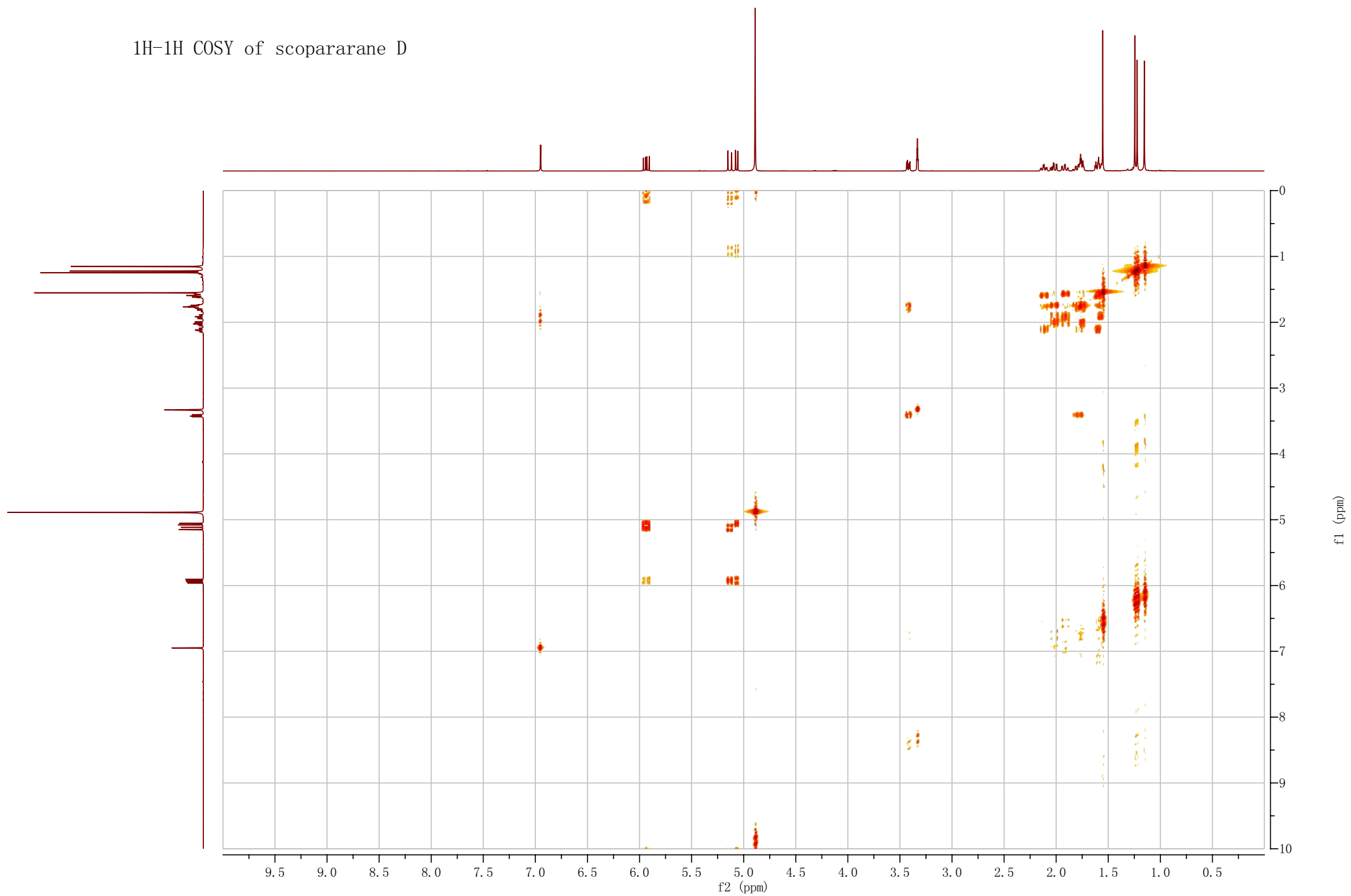
¹³C-NMR of scopararane D



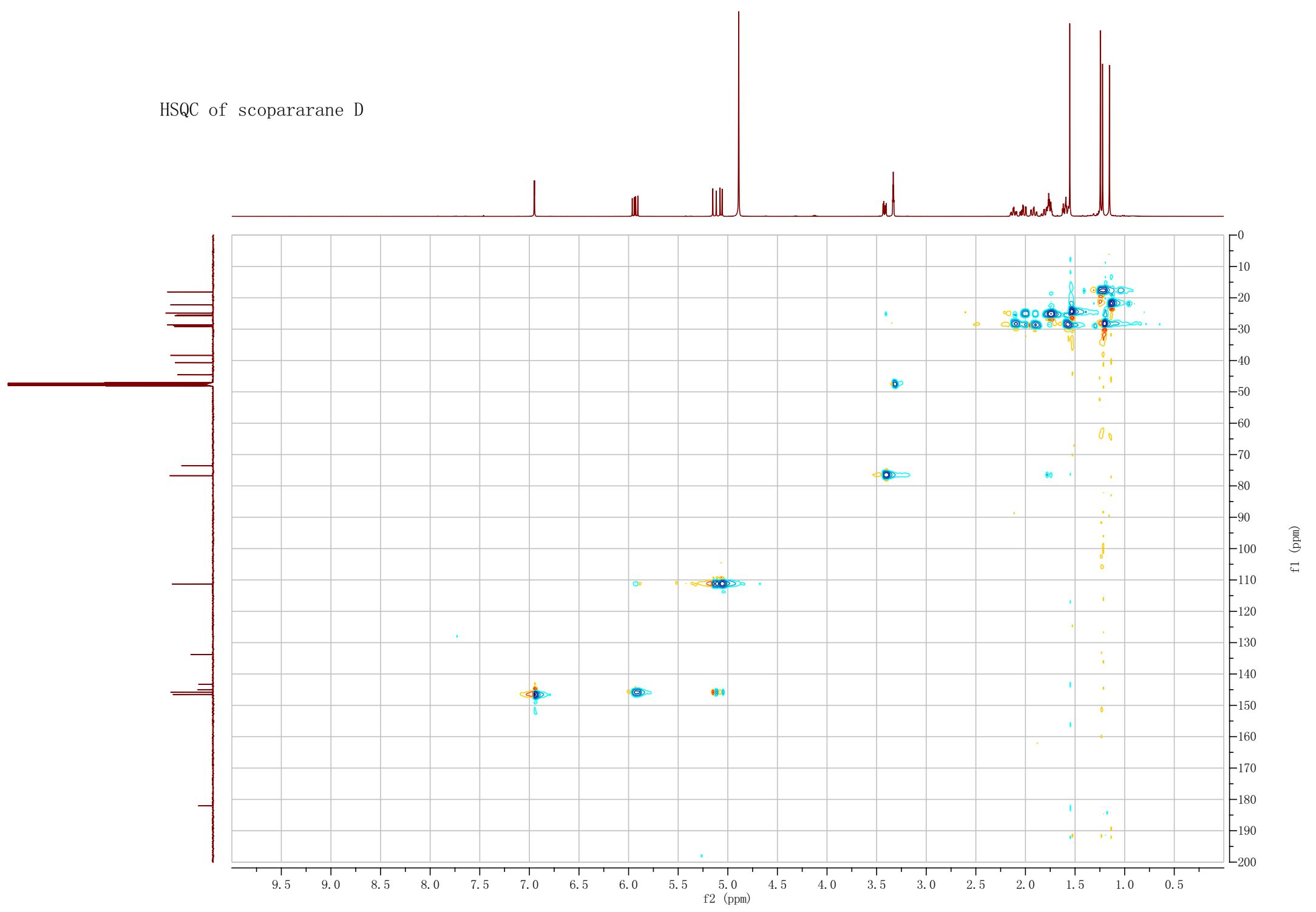
DEPT 135 of scopararane D



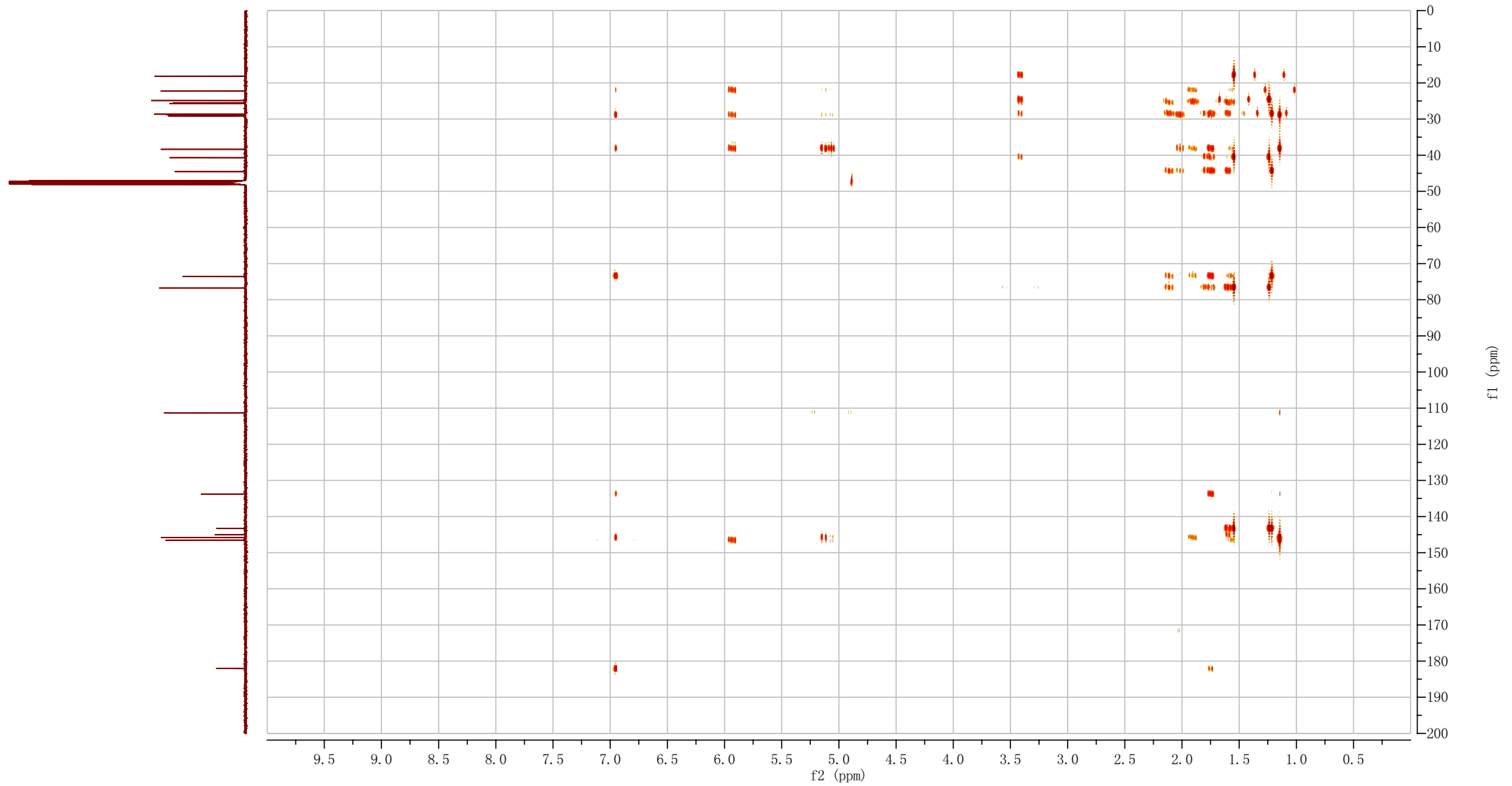
1H-1H COSY of scopararane D



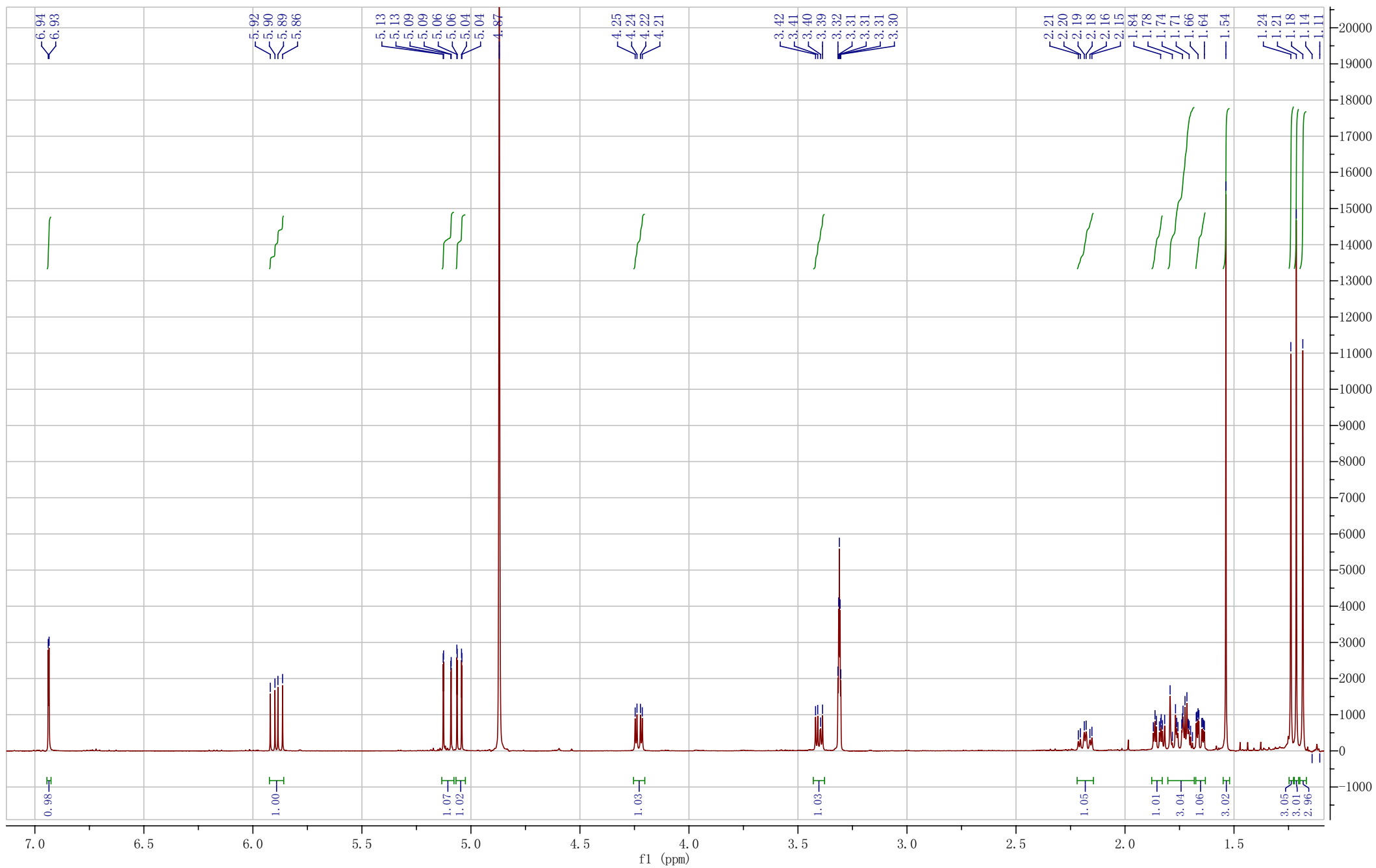
HSQC of scopararane D



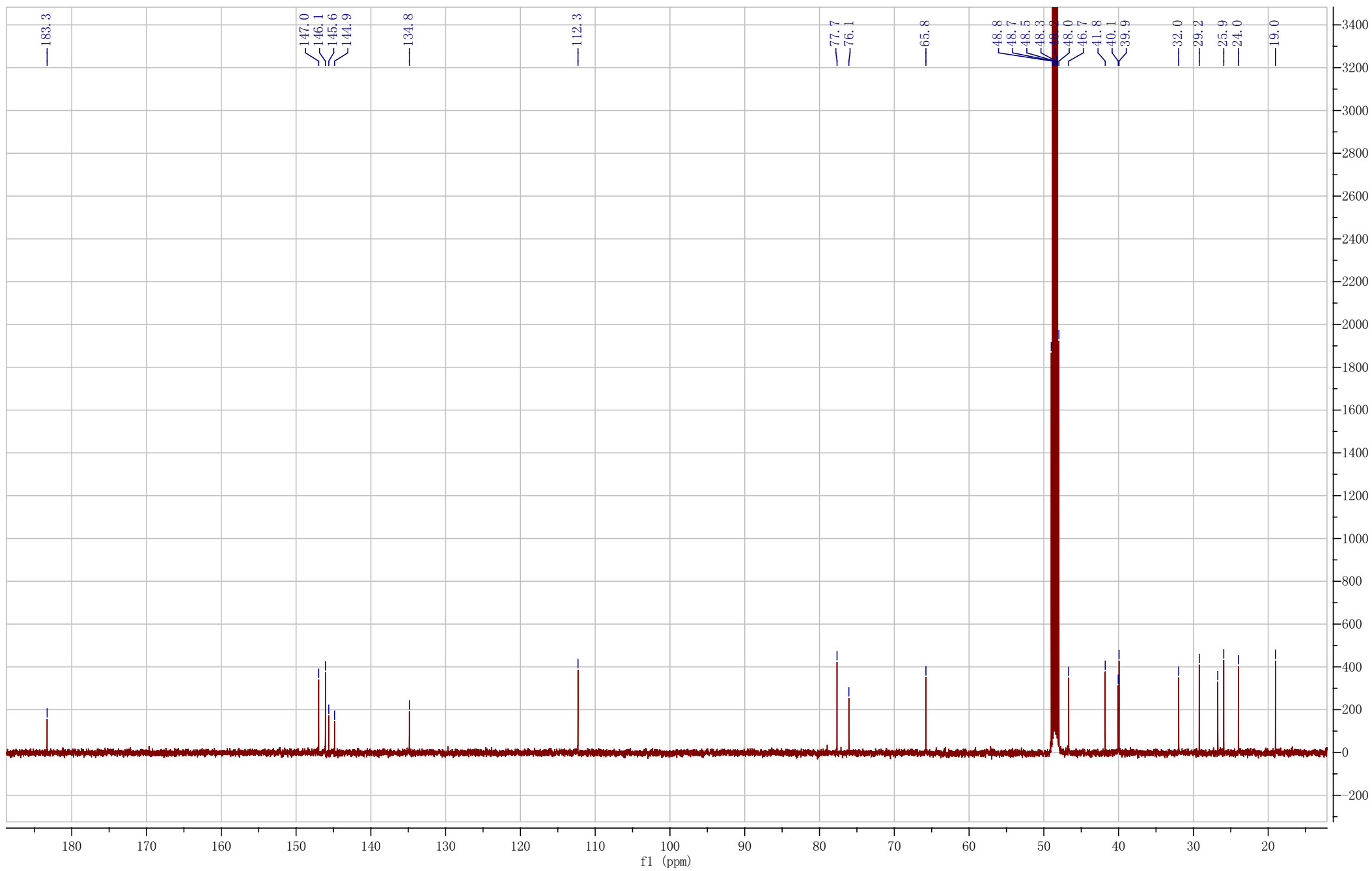
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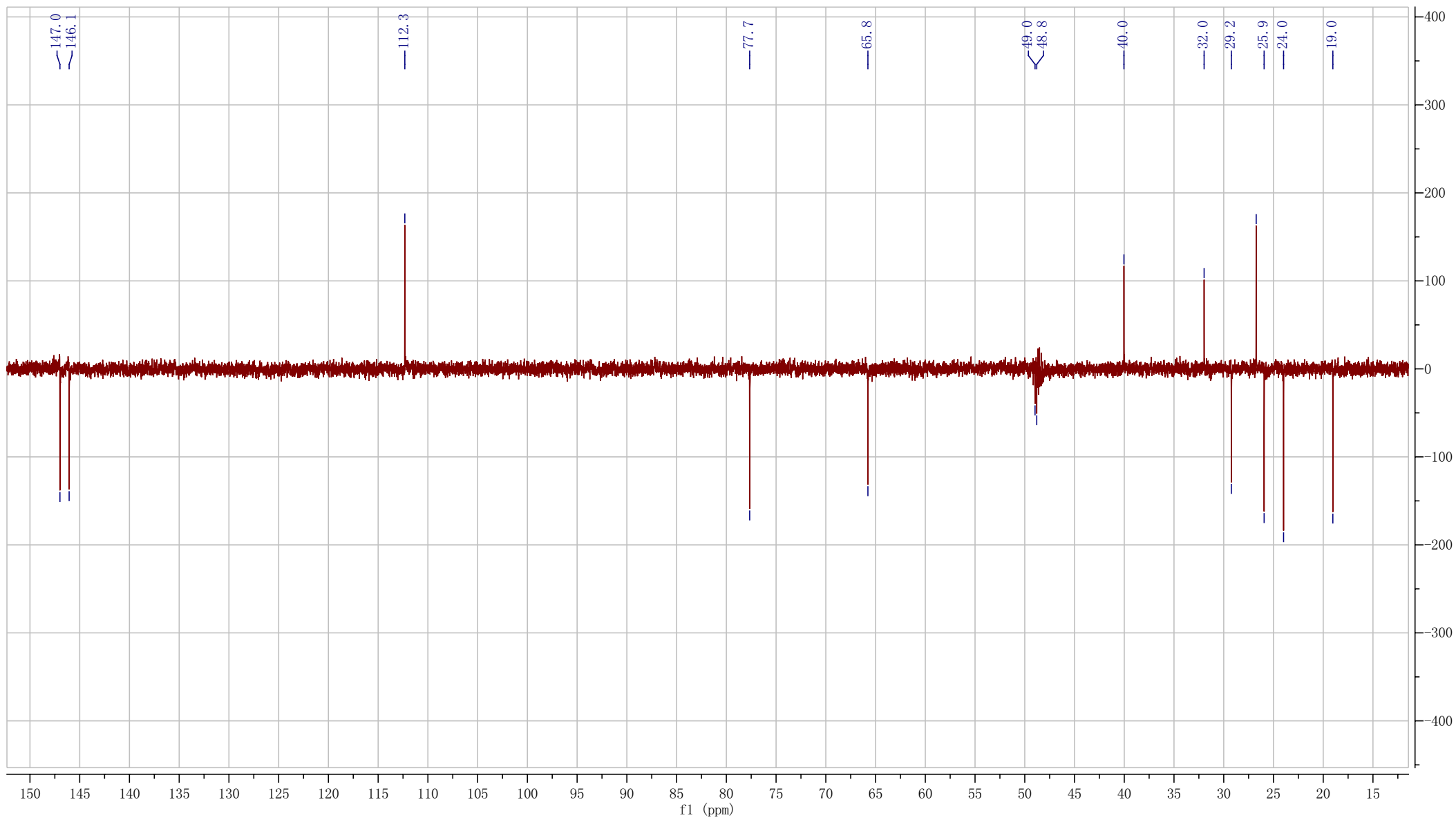
¹H-NMR of scopararane E



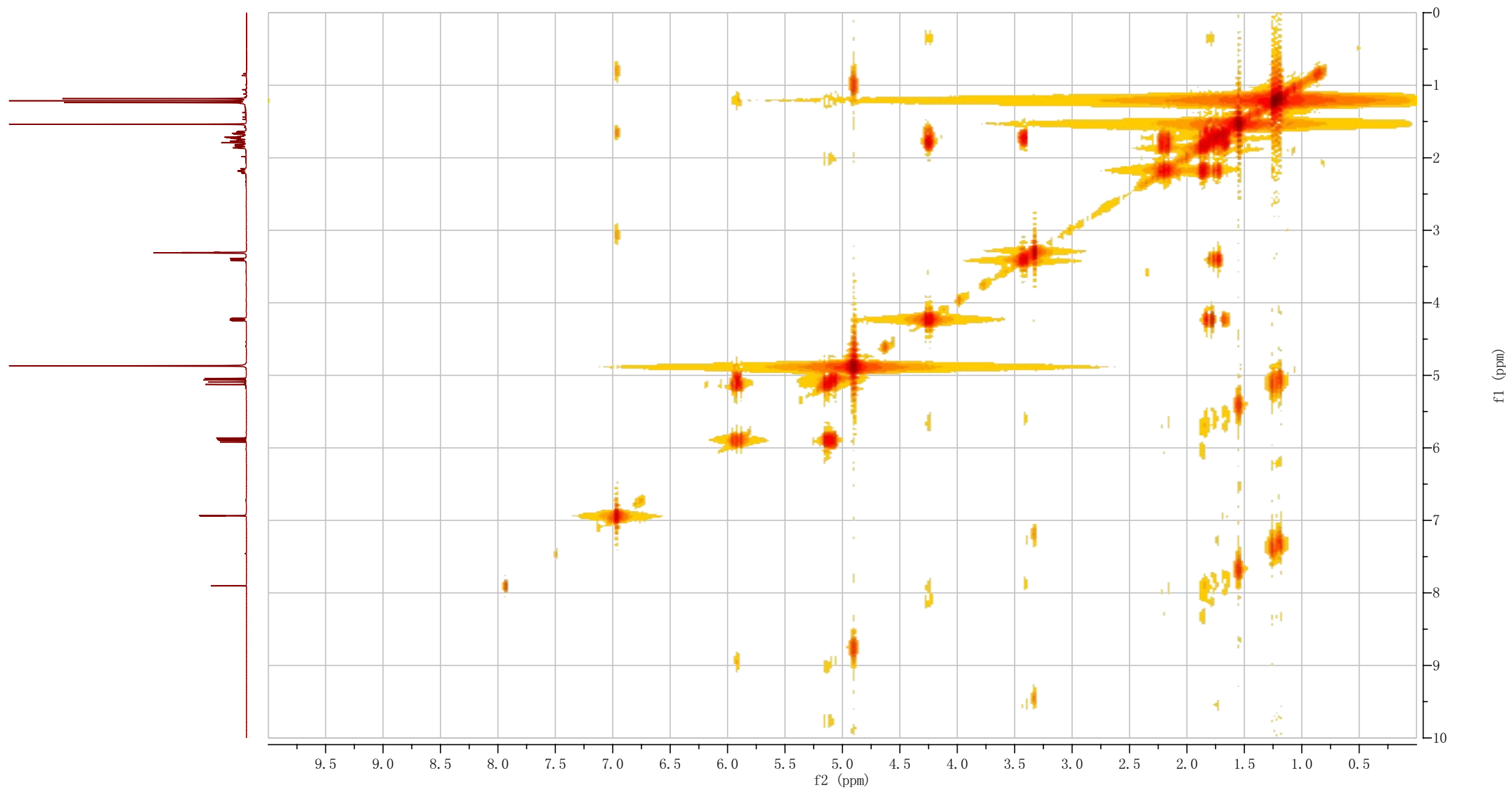
¹³C-NMR of scopararane E



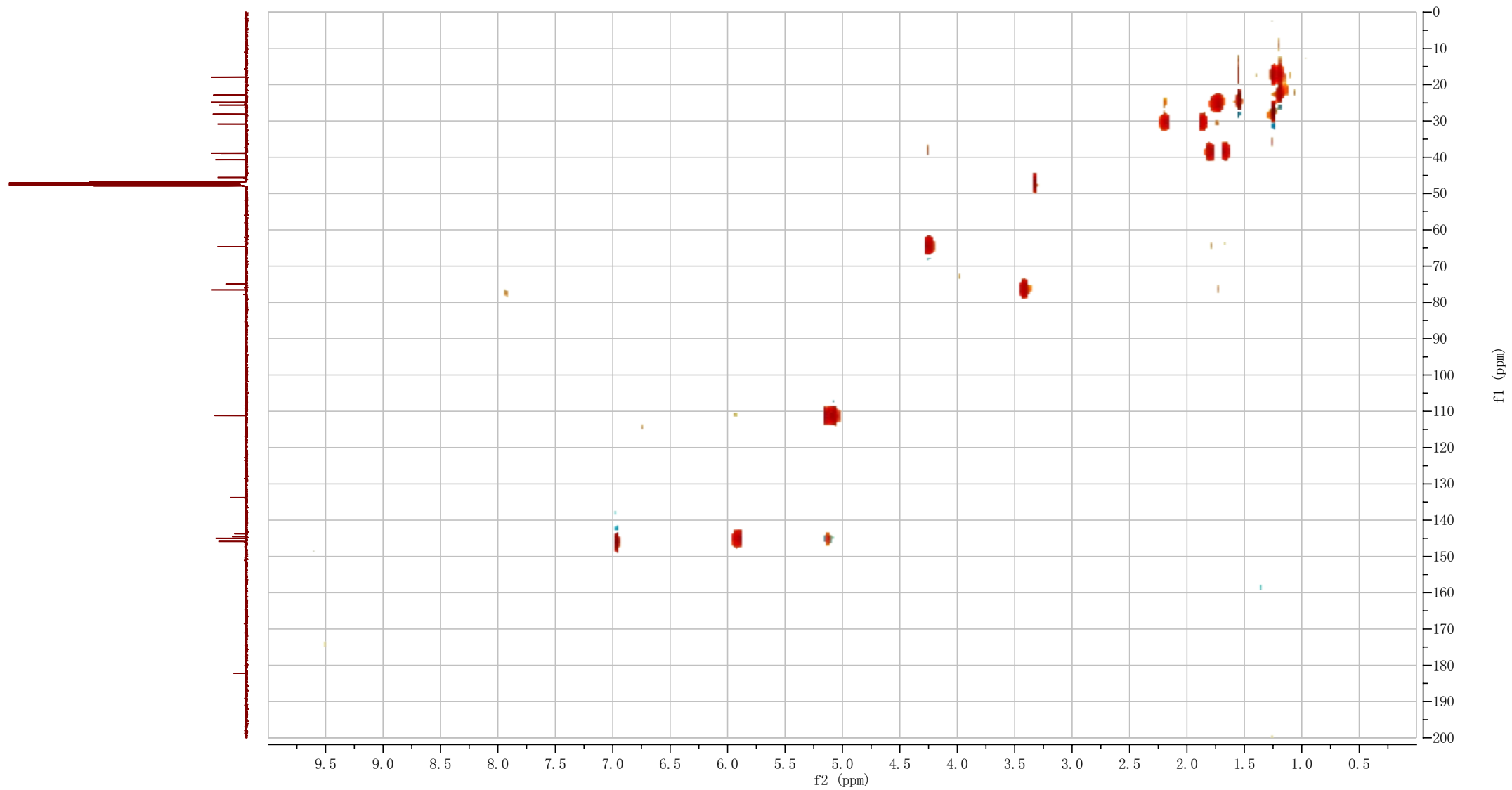
DEPT135 of scopararane E



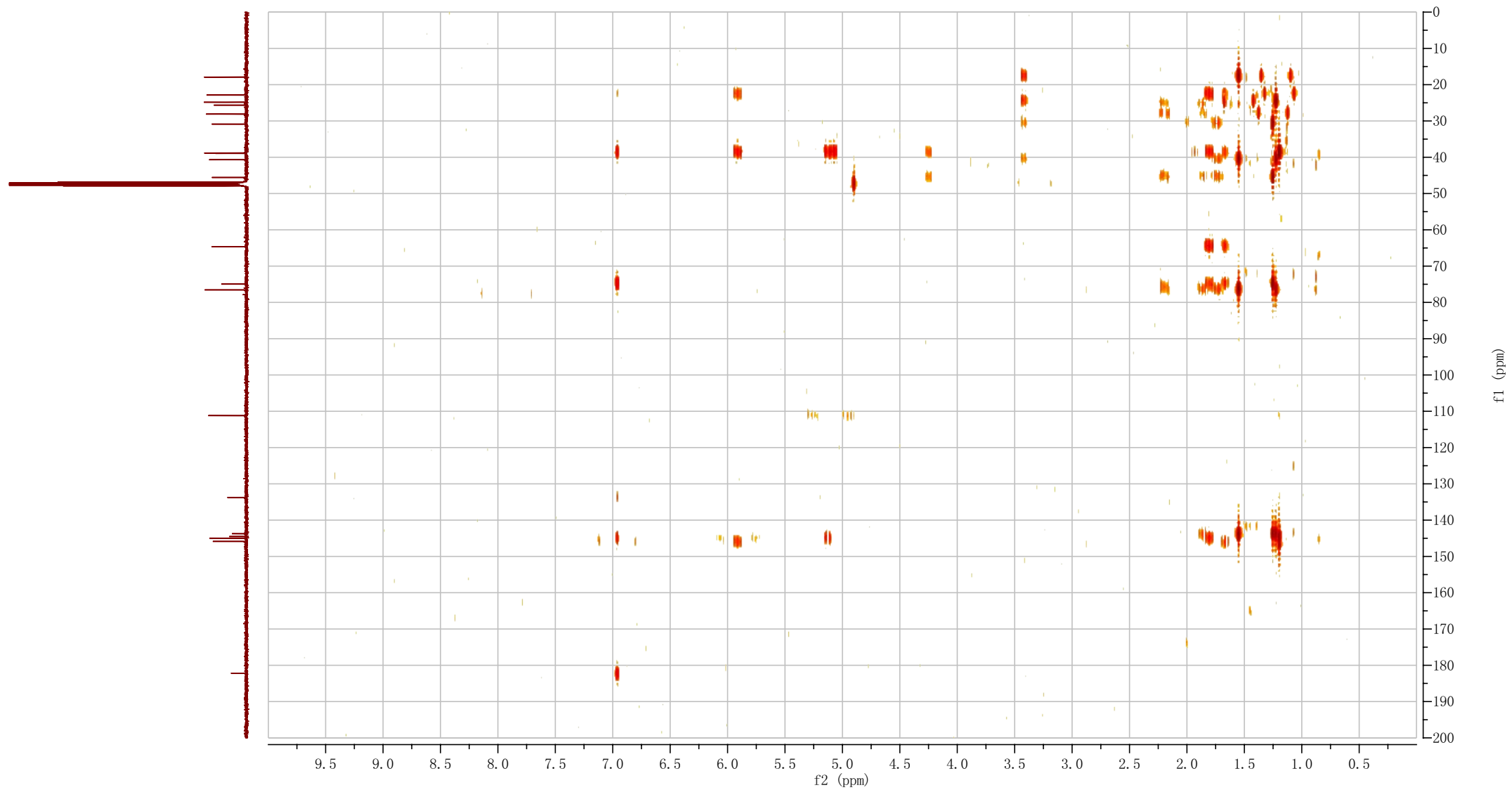
¹H-¹H COSY of scopararane E



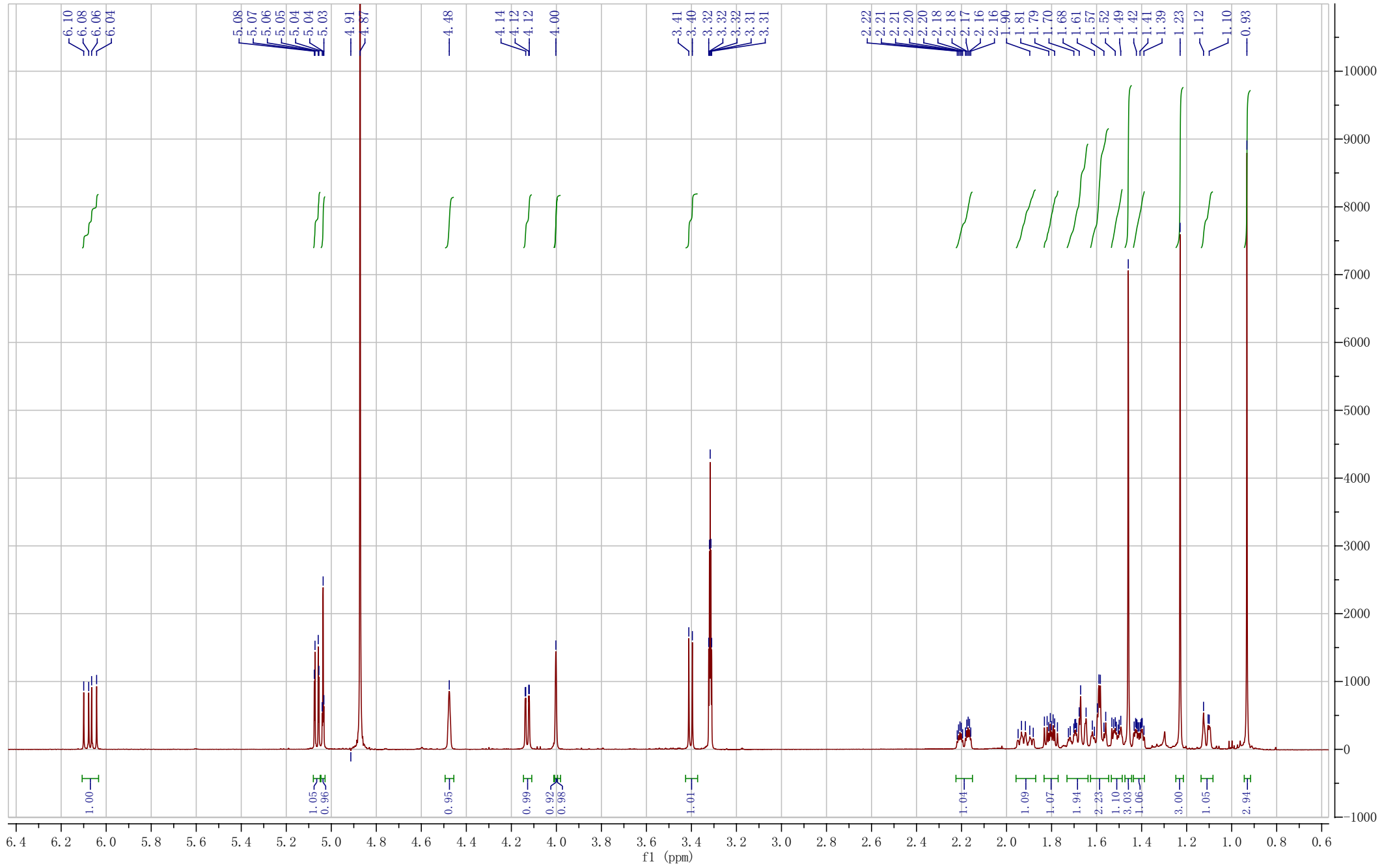
HSQC of scopararane E



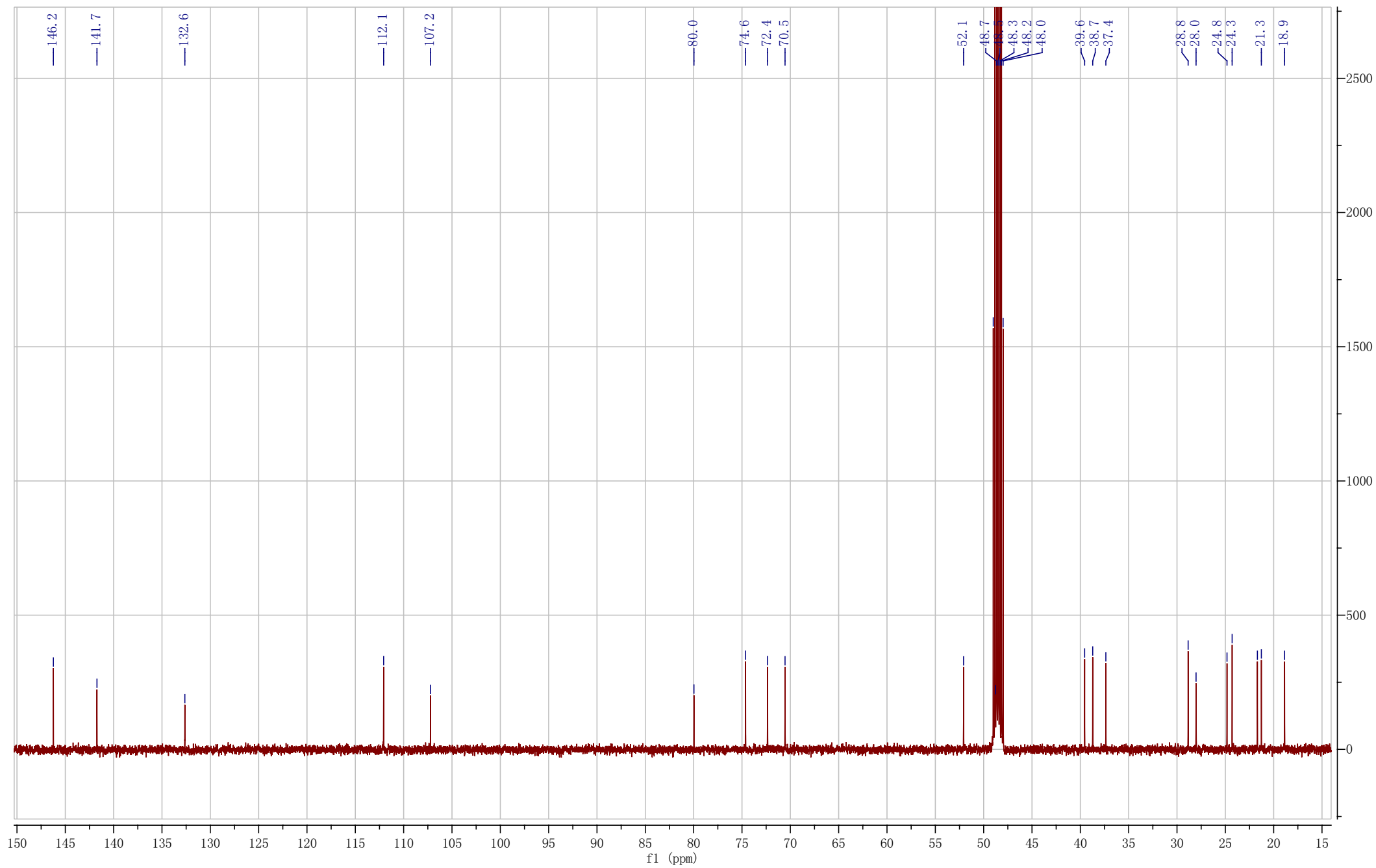
HMBC of scopararane E



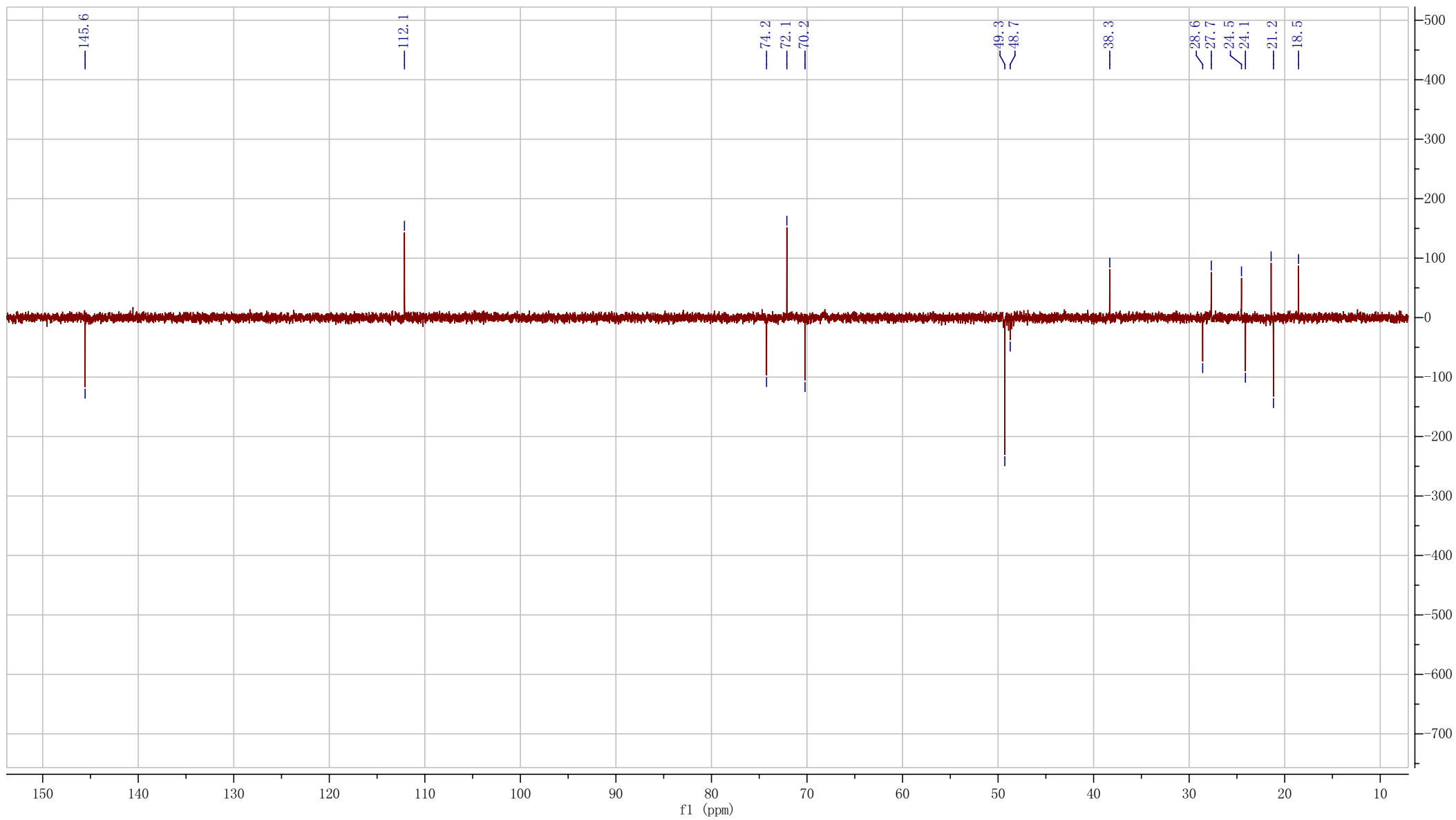
¹H-NMR of scopararane F



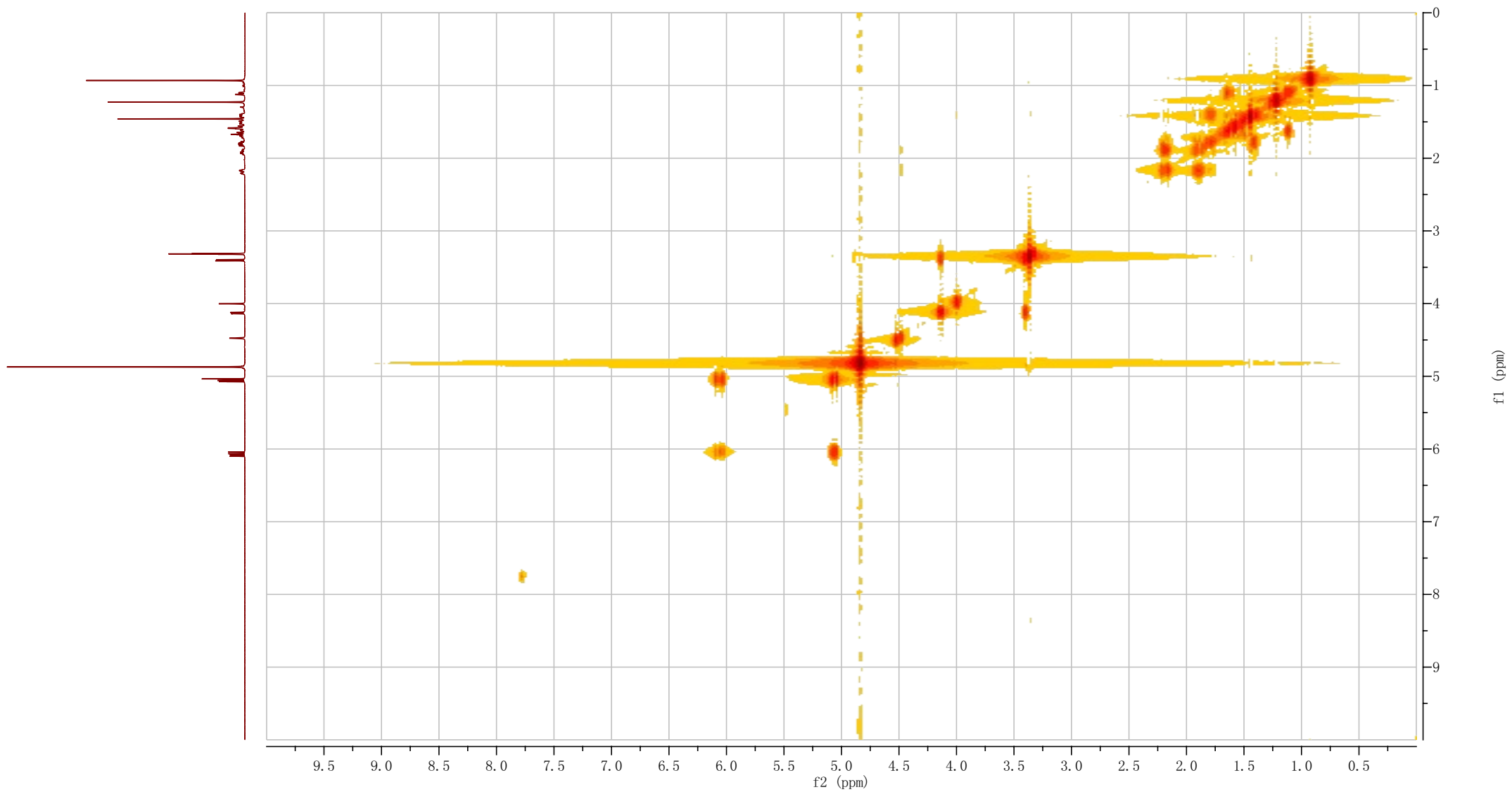
¹³C-NMR of scopararane F



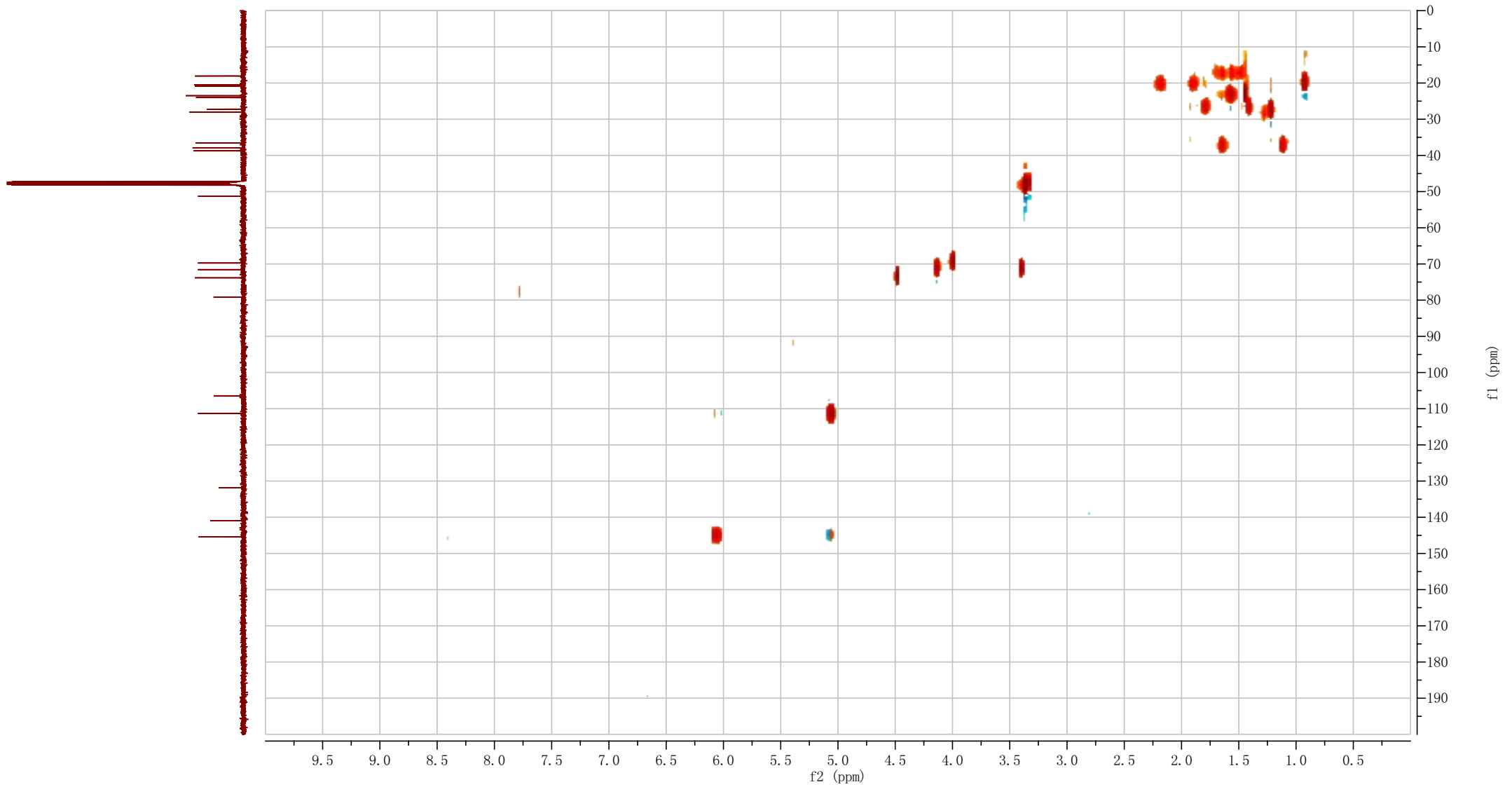
DEPT 135 of scopararane F



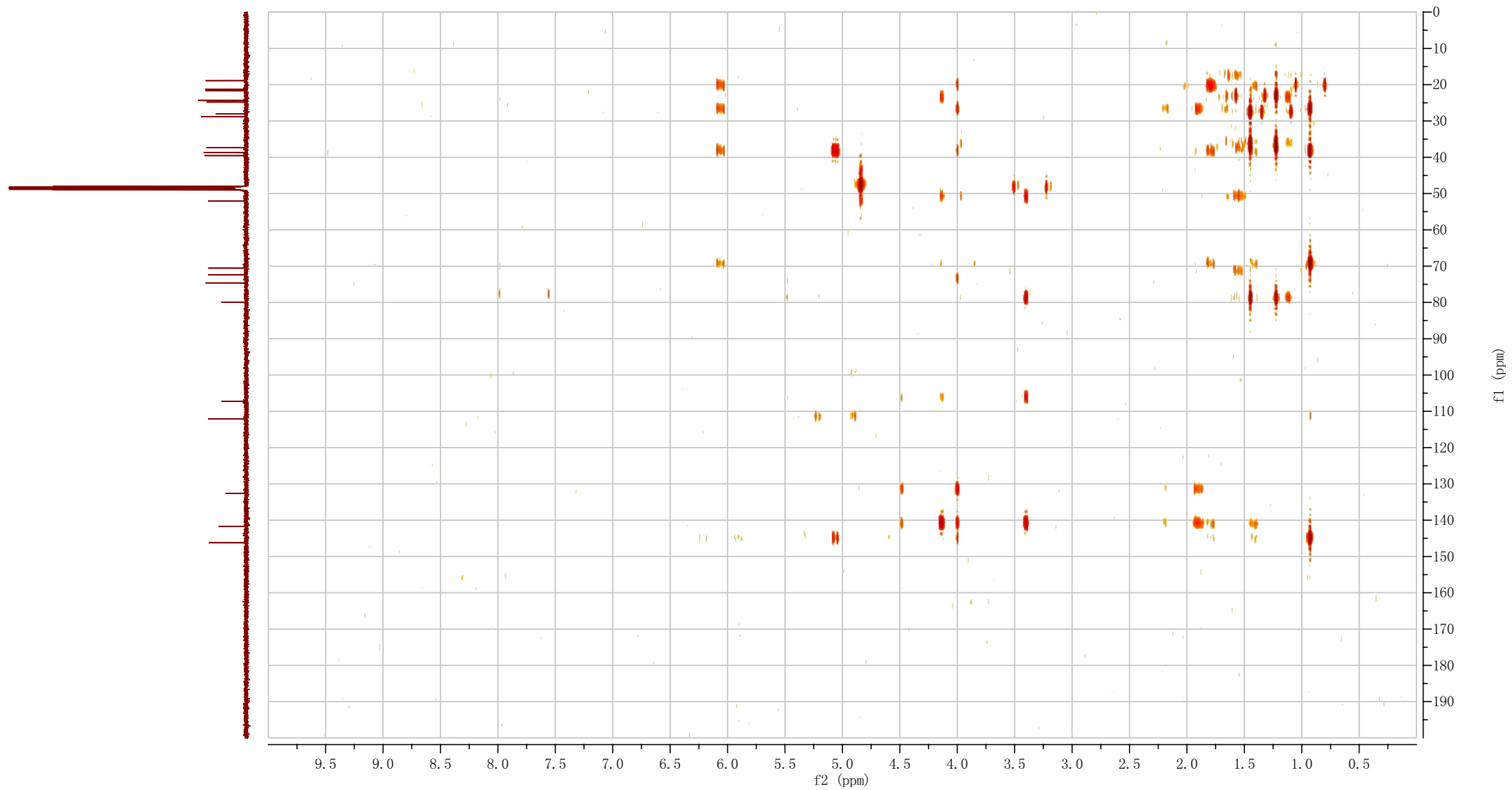
1H-1H COSY of scopararane F



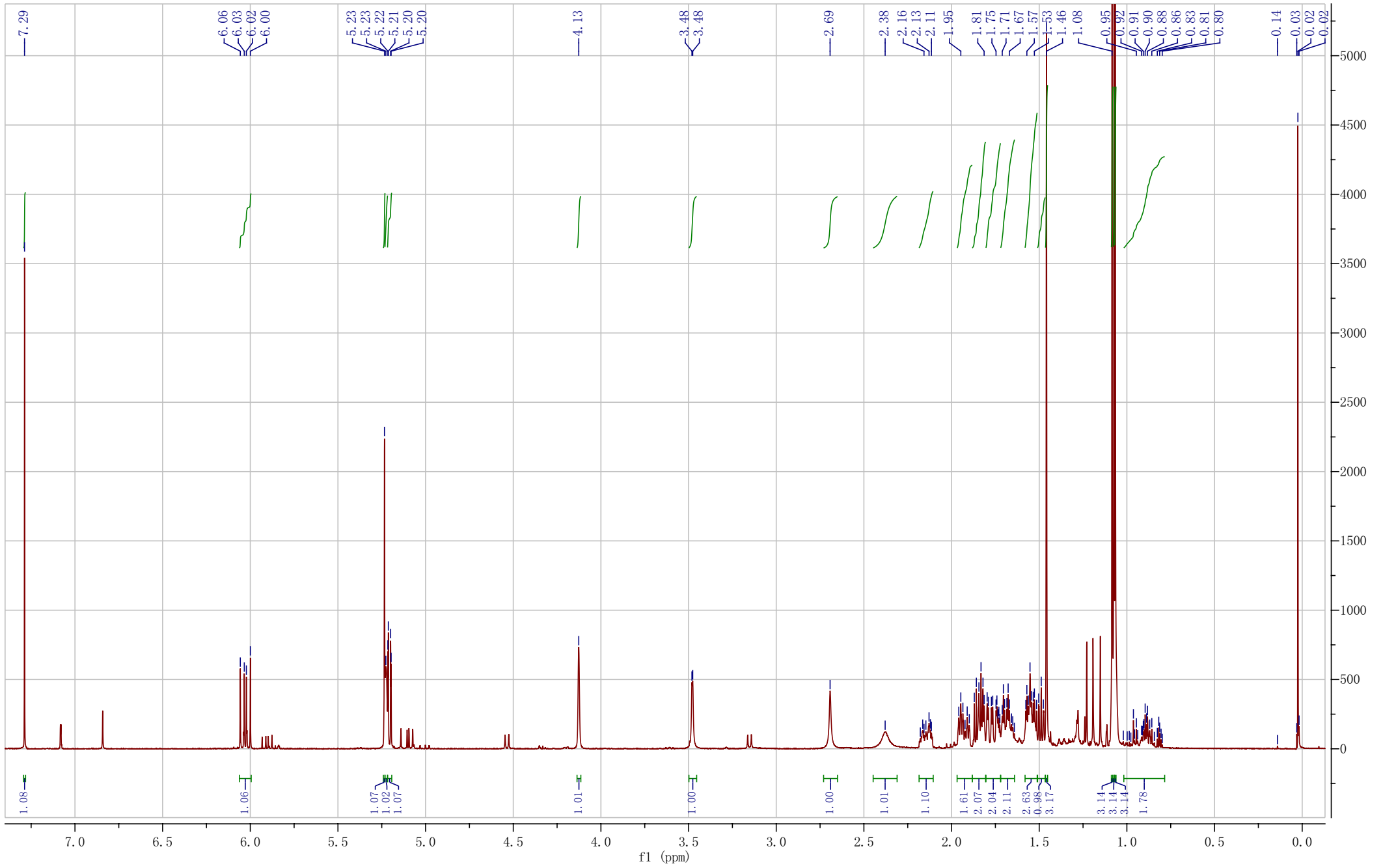
HSQC scopararane F



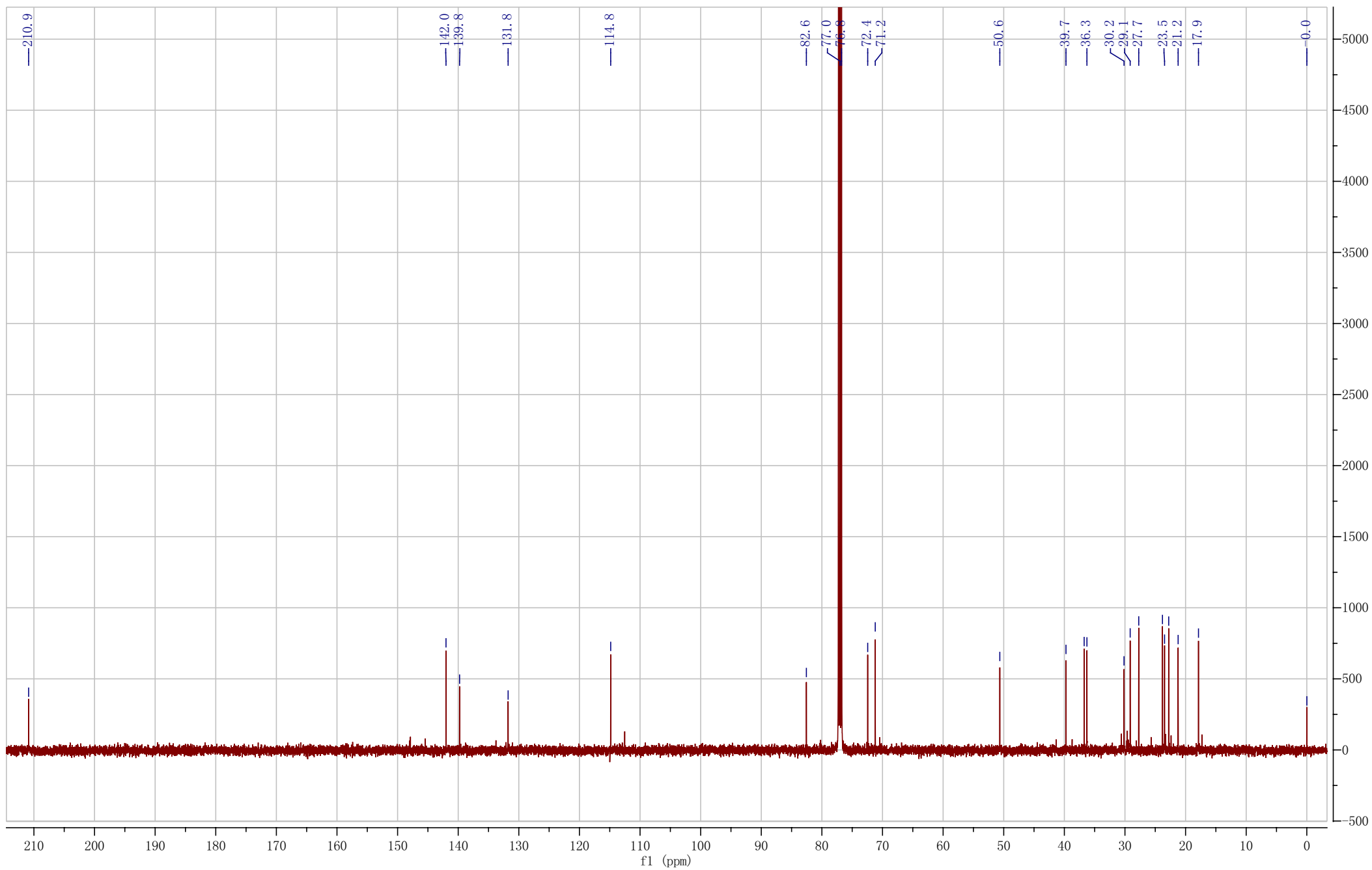
HMBC of scopararane F



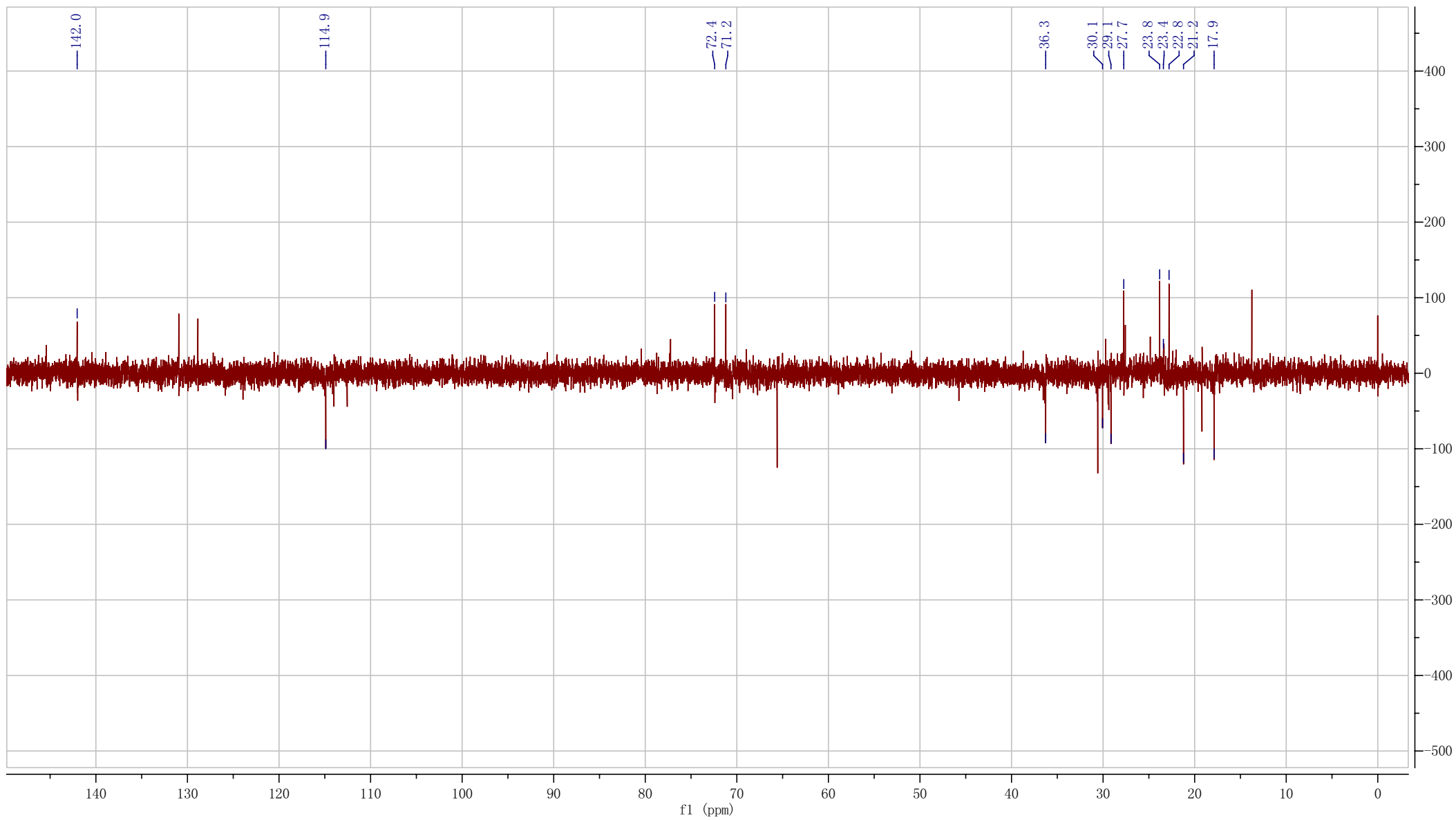
1H-NMR of scopararane G

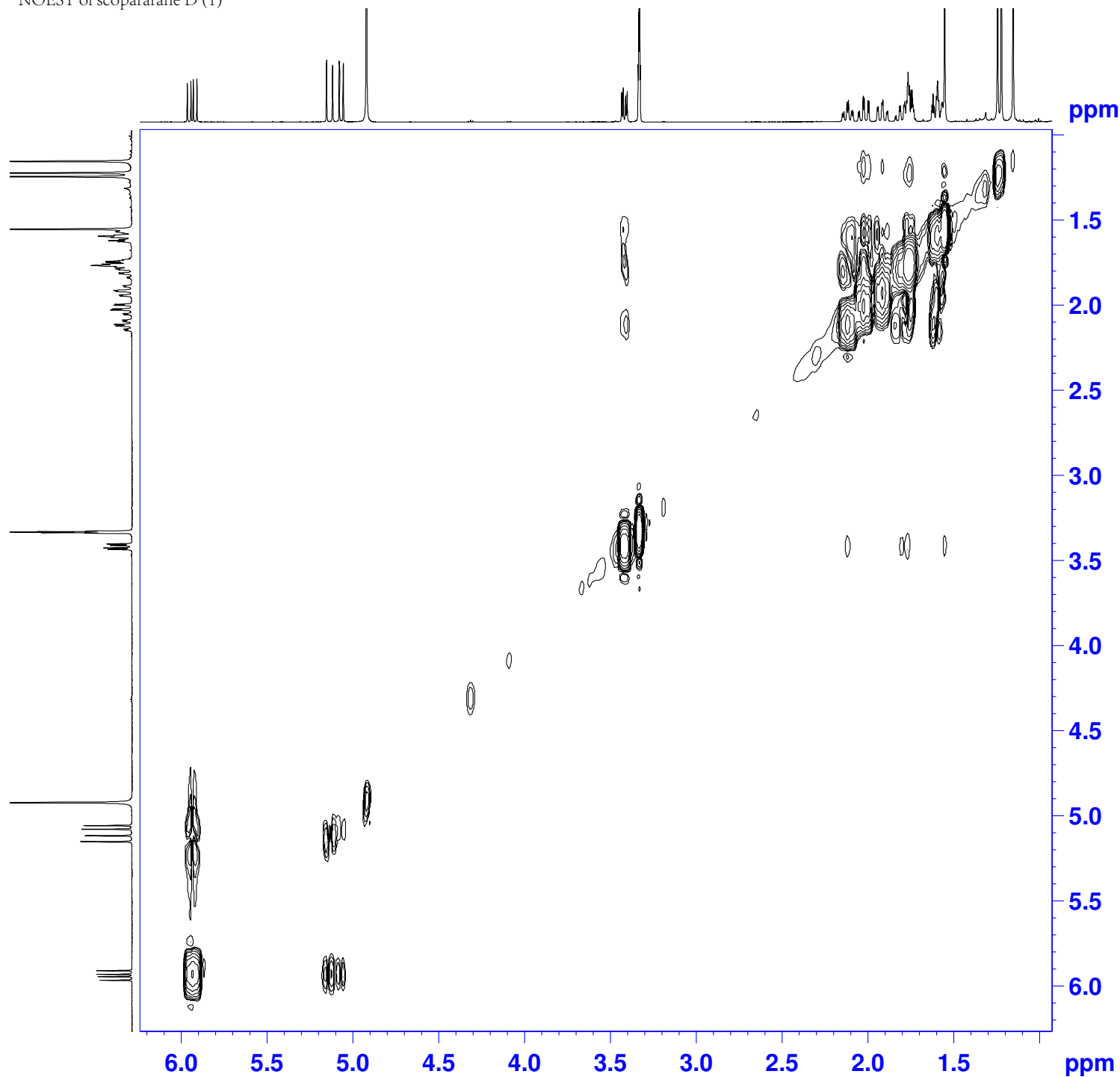


¹³C-NMR of scopararane G



DEPT 135 of scopararane G





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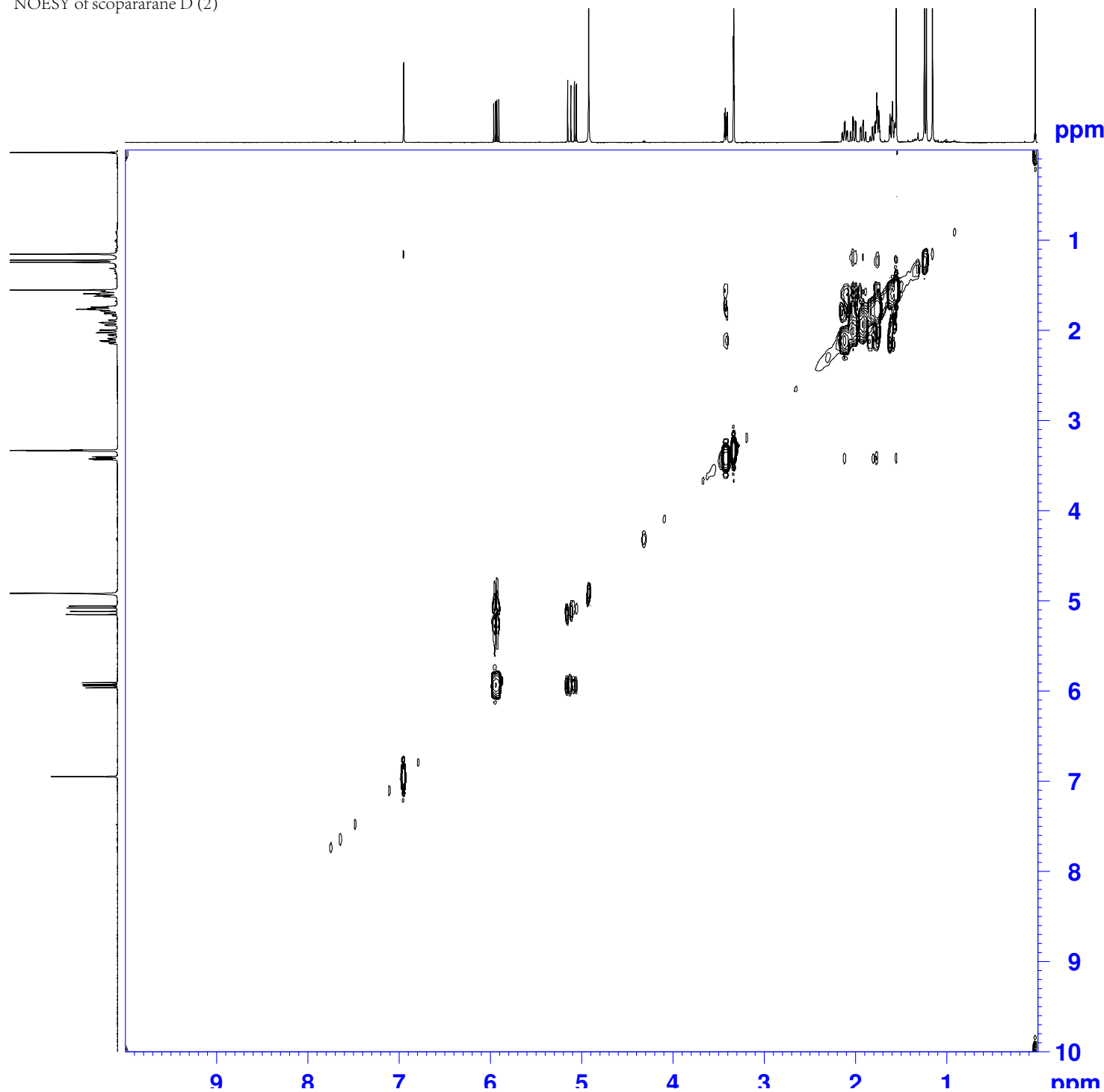
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PROCNO         1
Date_          20101214
Time           10.51
INSTRUM        spect
PROBHD         5 mm PABBO BB-
PULPROG        roesygpph19.2
TD             1024
SOLVENT        MeOD
NS              32
DS              16
SWH            5000.000
FIDRES         4.882813
AQ             0.1024500
RG              203
DW             100.000
DE              6.50
TE             293.0
D0             0.00008687
D1             2.00000000
D12            0.00002000
D16            0.00020000
D19            0.00025000
IN0            0.00019995
L4             500
P15            20000.00
    
```

```

===== CHANNEL f1 =====
NUC1           1H
P0             14.30
P1             14.30
P25            200.00
P27            14.30
PL1            2.50
PL18           2.50
PL27           19.39
PL1W           14.12537575
PL18W          14.12537575
PL27W          0.28906804
SFO1           500.1325007
    
```

```

===== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPZ1           20.00
GPZ2           20.00
P16            1000.00
ND0            1
TD             128
SFO1           500.1325
FIDRES         39.072853
SW             10.000
FnMODE         States-TPPI
SI             1024
SF             500.1299996
WDW            QSINE
SSB            2
LB             0.00
    
```



```

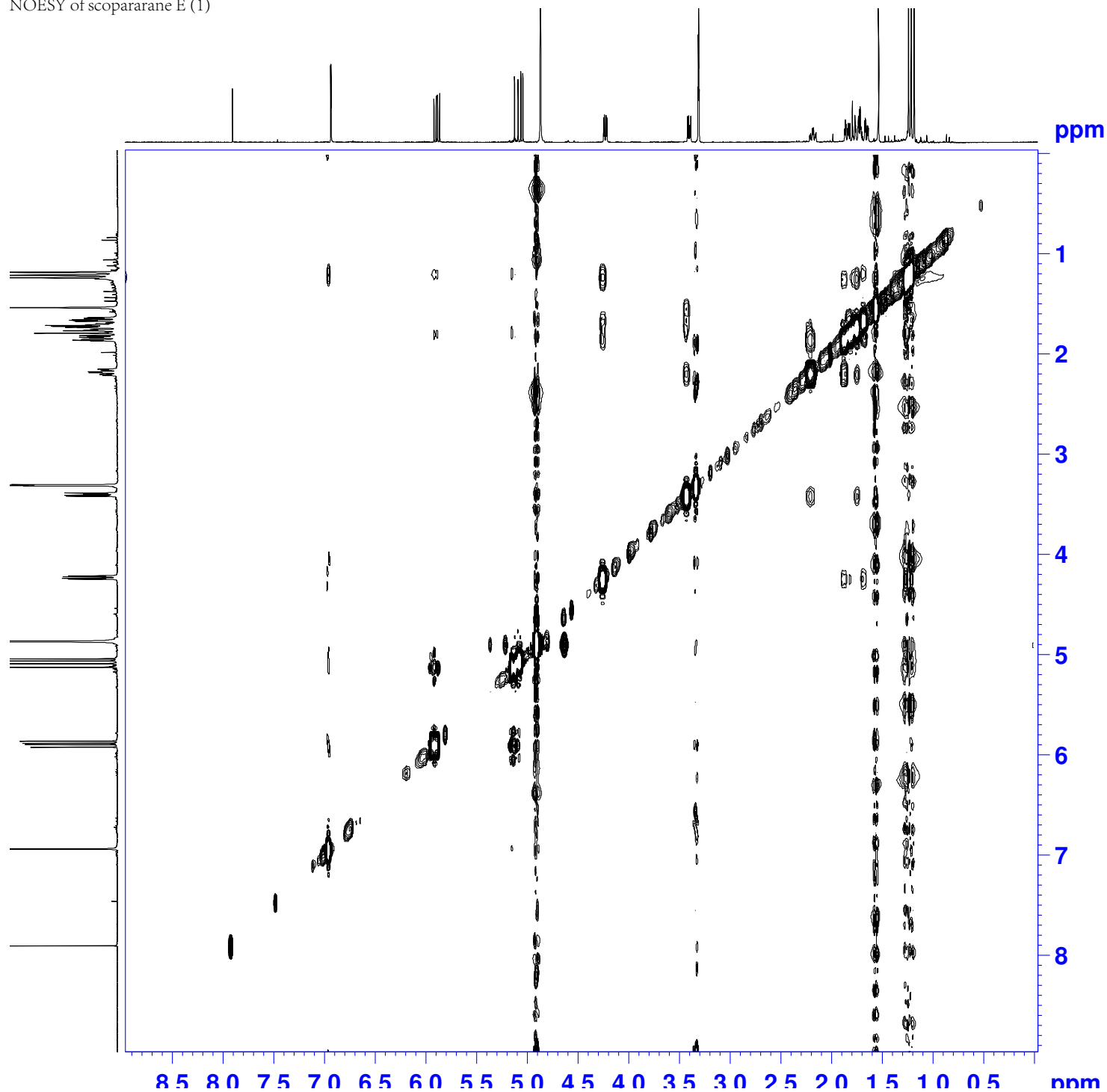
NAME          N24
EXPNO         10
PROCNO        1
Date_         20101214
Time          10.51
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       roesygpph19.2
TD            1024
SOLVENT       MeOD
NS            32
DS            16
SWH           5000.000
FIDRES        4.882813
AQ            0.1024500
RG            203
DW            100.000
DE            6.50
TE            293.0
D0            0.00008687
D1            2.00000000
D12           0.00002000
D16           0.00020000
D19           0.00025000
IN0           0.00019995
L4            500
P15           20000.00
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P0            14.30
P1            14.30
P25           200.00
P27           14.30
PL1           2.50
PL18          2.50
PL27          19.39
PL1W          14.12537575
PL18W         14.12537575
PL27W         0.28906804
SFO1          500.1325007
    
```

```

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          20.00
GPZ2          20.00
P16           1000.00
ND0           1
TD            128
SFO1          500.1325
FIDRES        39.072853
SW            10.000
FnMODE        States-TPPI
SI            1024
SF            500.1299996
WDW           QSINE
SSB           2
LB            0.00
    
```



```

NAME          P23a2
EXPNO         8
PROCNO        1
Date_         20101022
Time          9.53
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesygpph
TD            1024
SOLVENT       MeOD
NS            8
DS            16
SWH           4504.504
FIDRES        4.398930
AQ            0.1137140
RG            64
DW            111.000
DE            6.50
TE            295.0
D0            0.00009287
D1            2.00000000
D8            0.50000000
D16           0.00020000
IN0           0.00022215
    
```

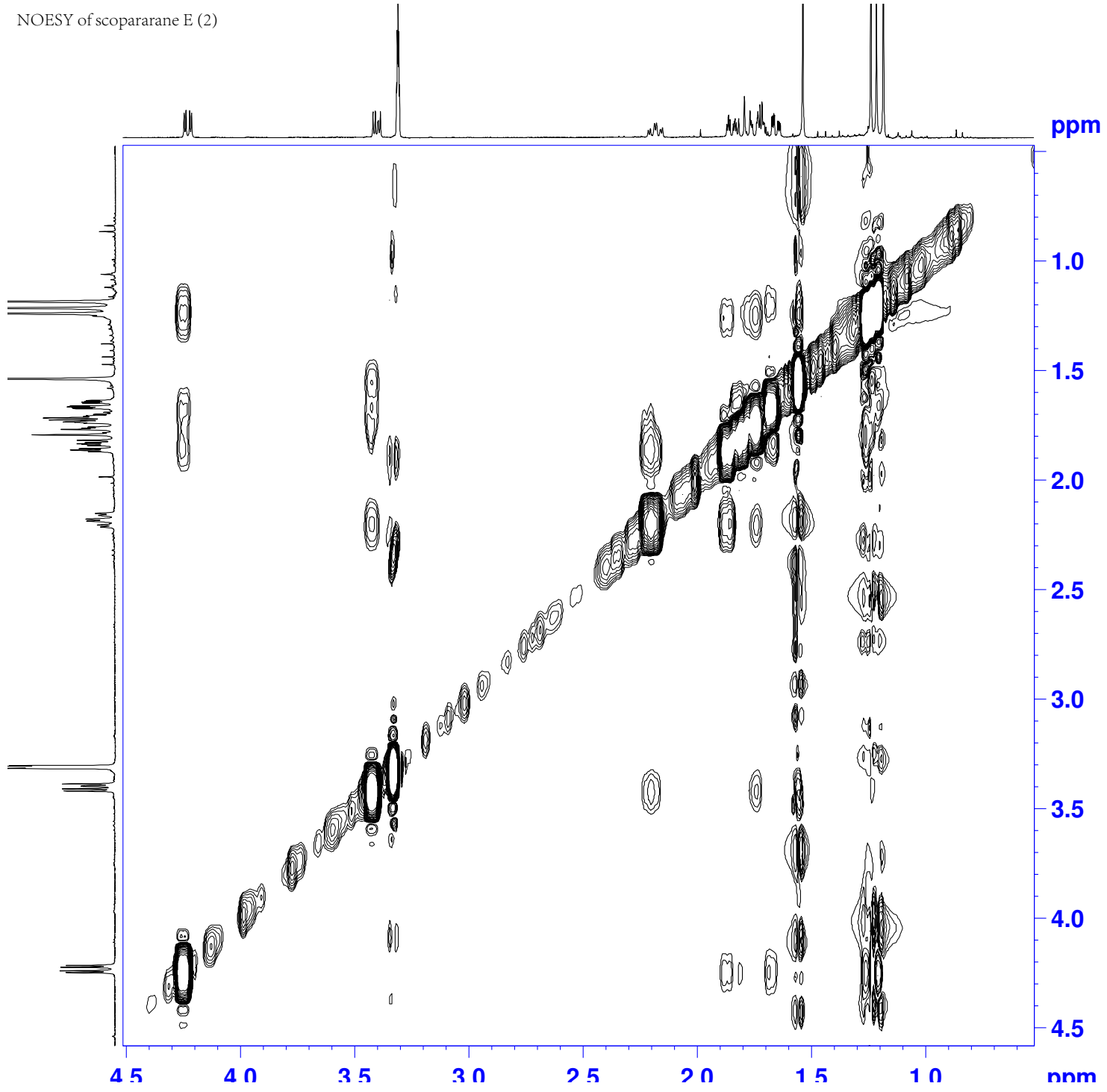
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===== CHANNEL f1 =====
NUC1           1H
P1             14.30
P2             28.60
PL1            2.50
PL1W           14.12537575
SFO1           500.1322506
    
```

```

===== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPZ1           20.00
GPZ2           20.00
P16            1000.00
ND0            1
TD             128
SFO1           500.1323
FIDRES         35.165550
SW             9.000
FnMODE         States-TPPI
SI             1024
SF             500.1300000
WDW            QSINE
SSB            2
LB             0.00
GB             0
PC             1.40
SI             1024
MC2            States-TPPI
SF             500.1300000
WDW            QSINE
SSB            2
LB             0.00
GB             0
    
```

NOESY of scopararane E (2)

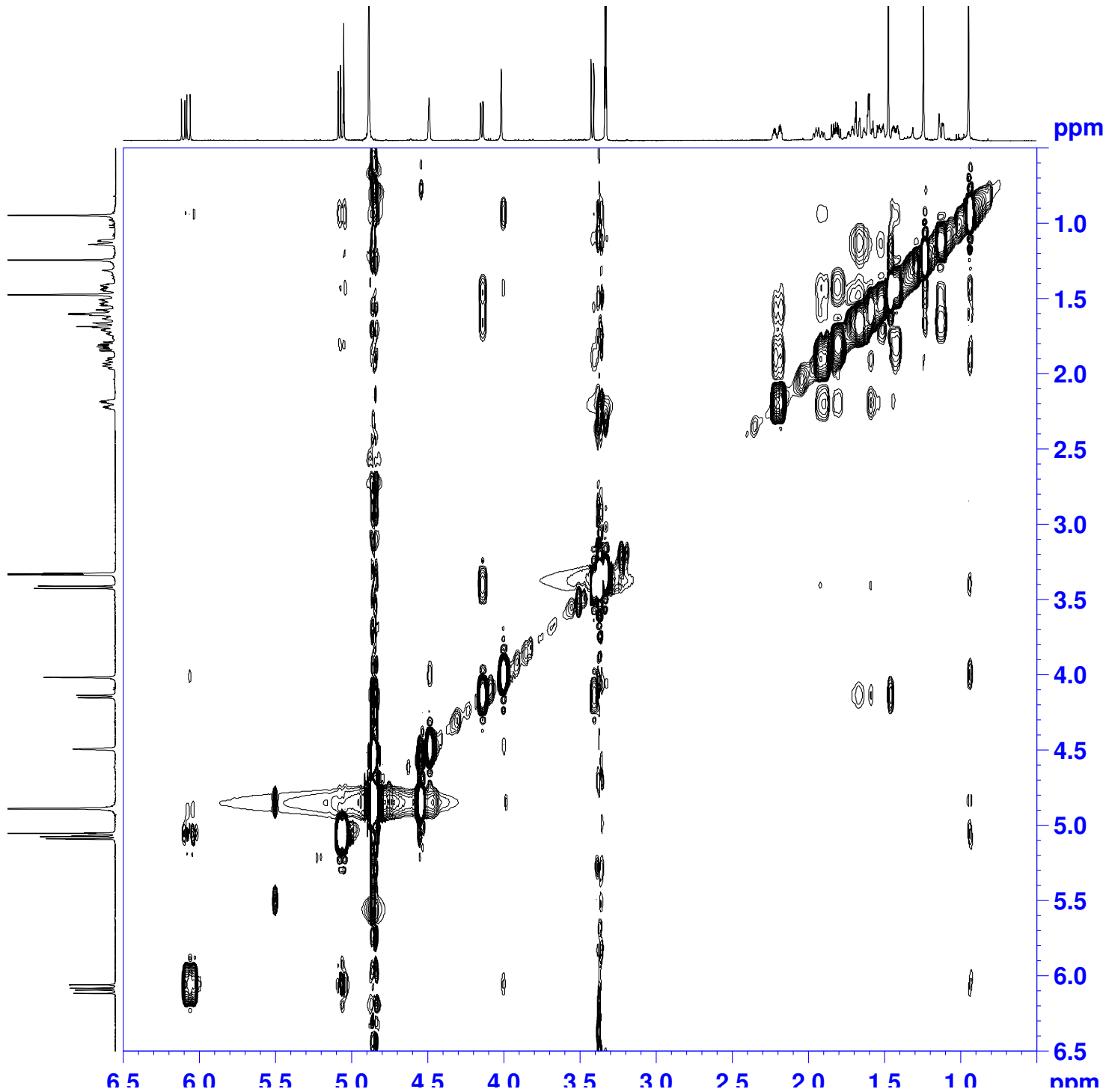


```

NAME                P23a2
EXPNO                8
PROCNO              1
Date_               20101022
Time                9.53
INSTRUM             spect
PROBHD              5 mm PABBO BB-
PULPROG             noesygpph
TD                  1024
SOLVENT             MeOD
NS                  8
DS                  16
SWH                 4504.504
FIDRES              4.398930
AQ                  0.1137140
RG                  64
DW                  111.000
DE                  6.50
TE                  295.0
D0                  0.00009287
D1                  2.00000000
D8                  0.50000000
D16                 0.00020000
IN0                 0.00022215

===== CHANNEL f1 =====
NUC1                 1H
P1                   14.30
P2                   28.60
PL1                  2.50
PL1W                 14.12537575
SFO1                 500.1322506

===== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPZ1                 20.00
GPZ2                 20.00
P16                 1000.00
ND0                  1
TD                   128
SFO1                 500.1323
FIDRES              35.165550
SW                   9.000
FnMODE              States-TPPI
SI                   1024
SF                   500.1300000
WDW                  QSINE
SSB                  2
LB                   0.00
GB                   0
PC                   1.40
SI                   1024
MC2                  States-TPPI
SF                   500.1300000
WDW                  QSINE
SSB                  2
LB                   0.00
GB                   0
    
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```

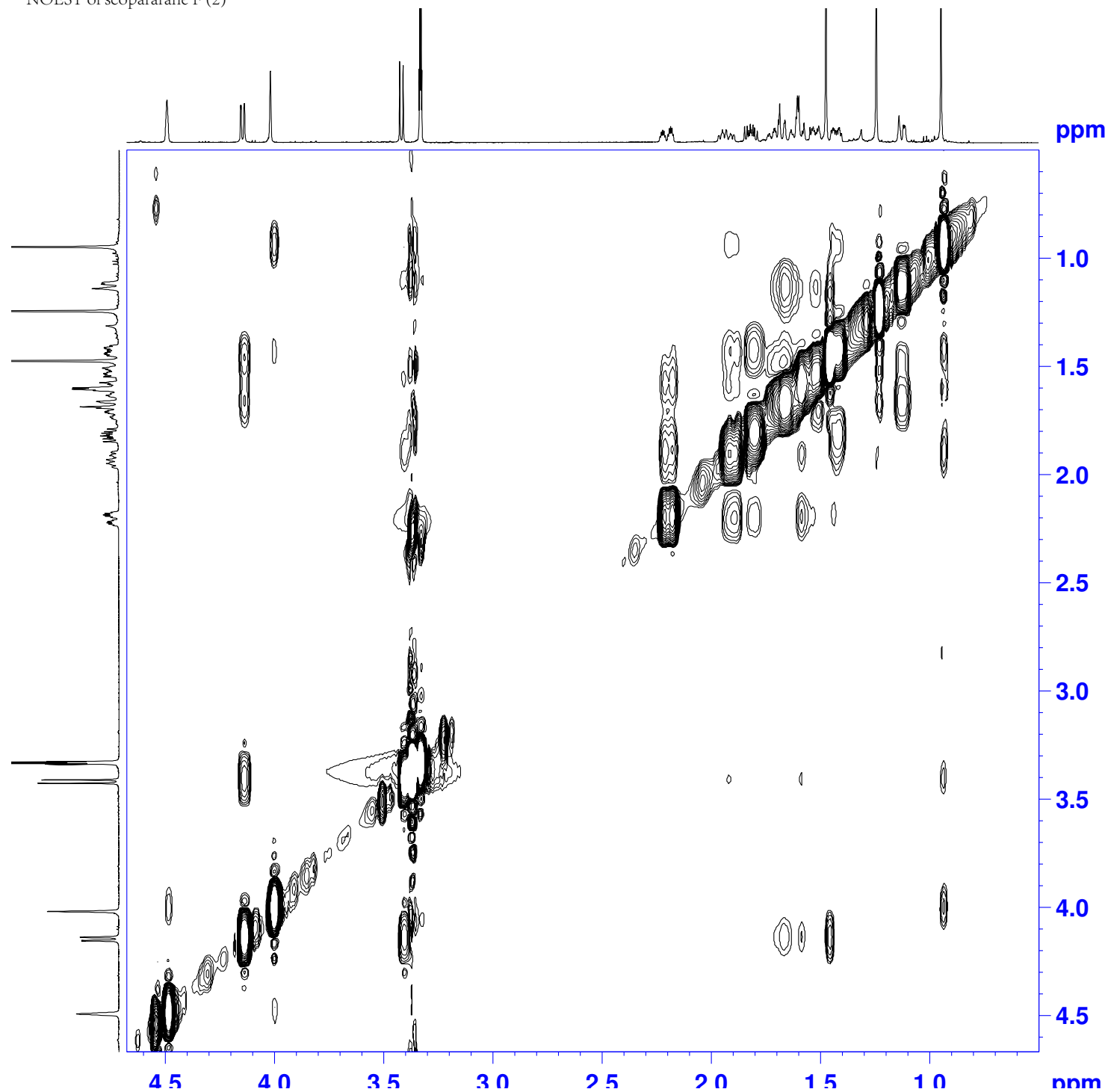
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PULPROG       noesygpph
TD            1024
SOLVENT       MeOD
NS            8
DS           16
SWH           4504.504
FIDRES        4.398930
AQ            0.1137140
RG            64
DW           111.000
DE            6.50
TE           295.1
D0            0.00009287
D1            2.00000000
D8            0.50000000
D16           0.00020000
IN0           0.00022215
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.30
P2            28.60
PL1           2.50
PL1W          14.12537575
SFO1          500.1322506
    
```

```

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          20.00
GPZ2          20.00
P16           1000.00
ND0           1
TD            128
SFO1          500.1323
FIDRES        35.165550
SW            9.000
FnMODE        States-TPPI
SI            1024
SF            500.130000
WDW           QSINE
SSB           2
LB            0.00
GB            0
PC            1.40
SI            1024
MC2           States-TPPI
SF            500.130000
WDW           QSINE
SSB           2
LB            0.00
GB            0
    
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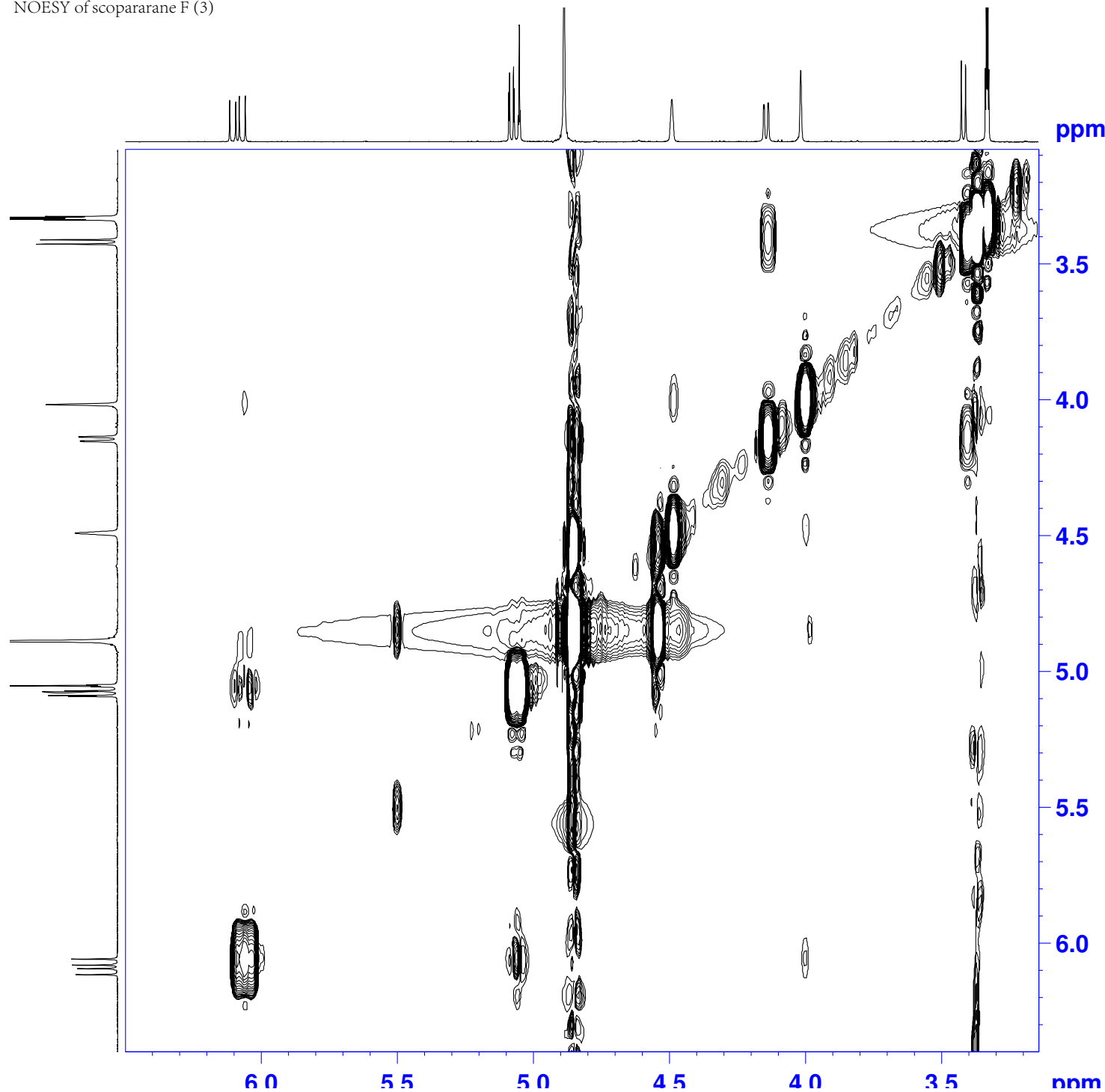
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EXPNO         8
PROCNO        1
Date_         20101022
Time          10.52
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesygpph
TD            1024
SOLVENT       MeOD
NS            8
DS            16
SWH           4504.504
FIDRES        4.398930
AQ            0.1137140
RG            64
DW            111.000
DE            6.50
TE            295.1
D0            0.00009287
D1            2.00000000
D8            0.50000000
D16           0.00020000
IN0           0.00022215
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.30
P2            28.60
PL1           2.50
PL1W          14.12537575
SFO1          500.1322506
    
```

```

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          20.00
GPZ2          20.00
P16           1000.00
ND0           1
TD            128
SFO1          500.1323
FIDRES        35.165550
SW            9.000
FnMODE        States-TPPI
SI            1024
SF            500.1300000
WDW           QSINE
SSB           2
LB            0.00
GB            0
PC            1.40
SI            1024
MC2           States-TPPI
SF            500.1300000
WDW           QSINE
SSB           2
LB            0.00
GB            0
    
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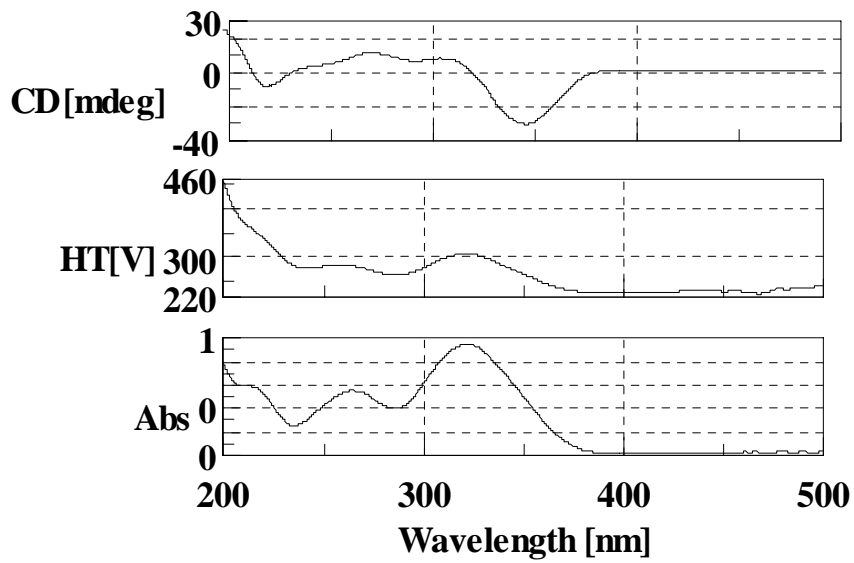
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EXPNO         8
PROCNO        1
Date_         20101022
Time          10.52
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesygpph
TD            1024
SOLVENT       MeOD
NS            8
DS            16
SWH           4504.504
FIDRES        4.398930
AQ            0.1137140
RG            64
DW            111.000
DE            6.50
TE            295.1
D0            0.00009287
D1            2.00000000
D8            0.50000000
D16           0.00020000
IN0           0.00022215
    
```

```

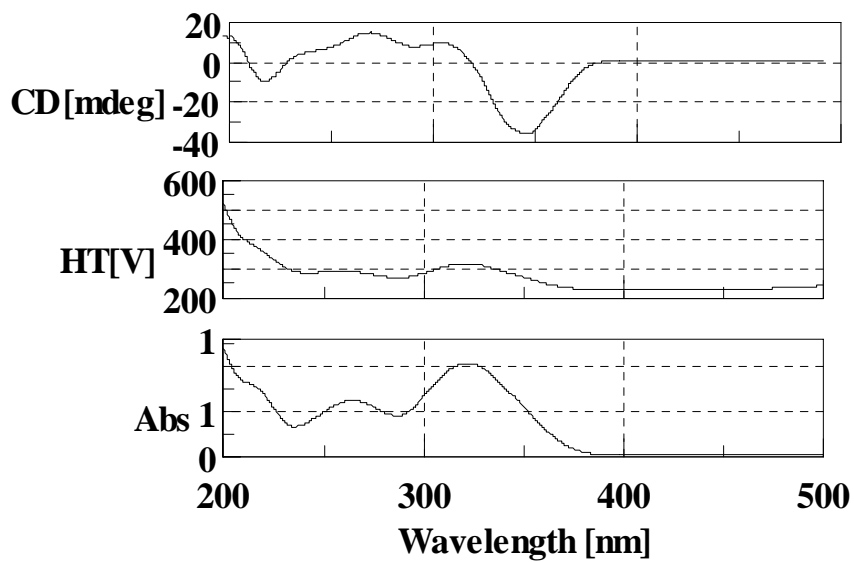
===== CHANNEL f1 =====
NUC1          1H
P1            14.30
P2            28.60
PL1           2.50
PL1W          14.12537575
SFO1          500.1322506
    
```

```

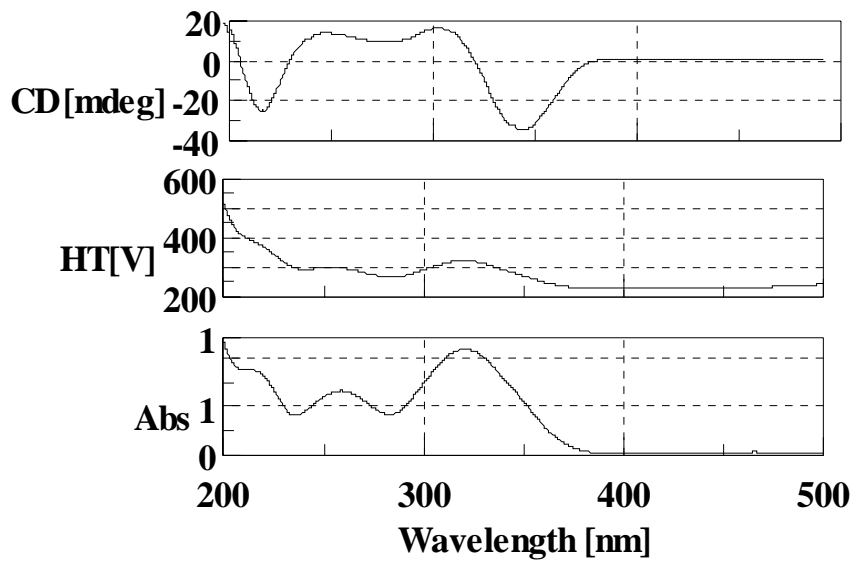
===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          20.00
GPZ2          20.00
P16           1000.00
ND0           1
TD            128
SFO1          500.1323
FIDRES        35.165550
SW            9.000
FnMODE        States-TPPI
SI            1024
SF            500.1300000
WDW           QSINE
SSB           2
LB            0.00
GB            0
PC            1.40
SI            1024
MC2           States-TPPI
SF            500.1300000
WDW           QSINE
SSB           2
LB            0.00
GB            0
    
```



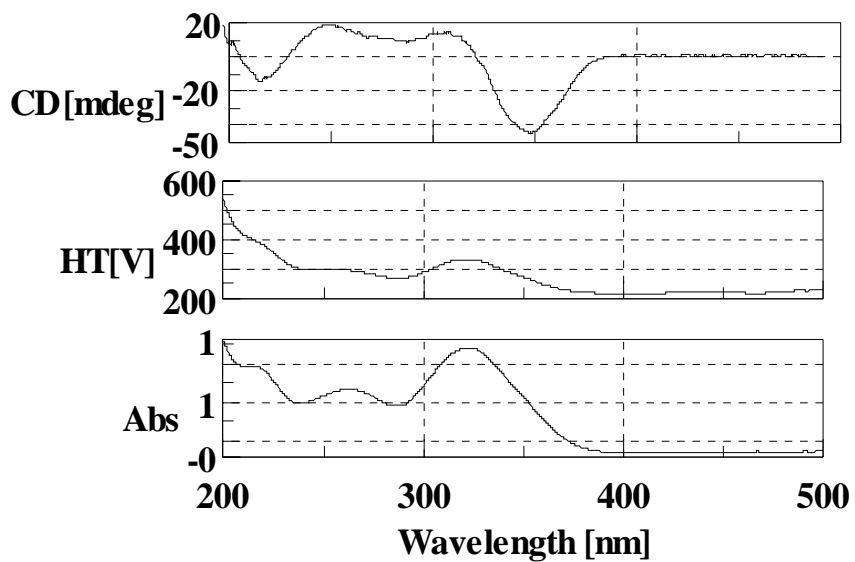
CD of scopararane C (1)



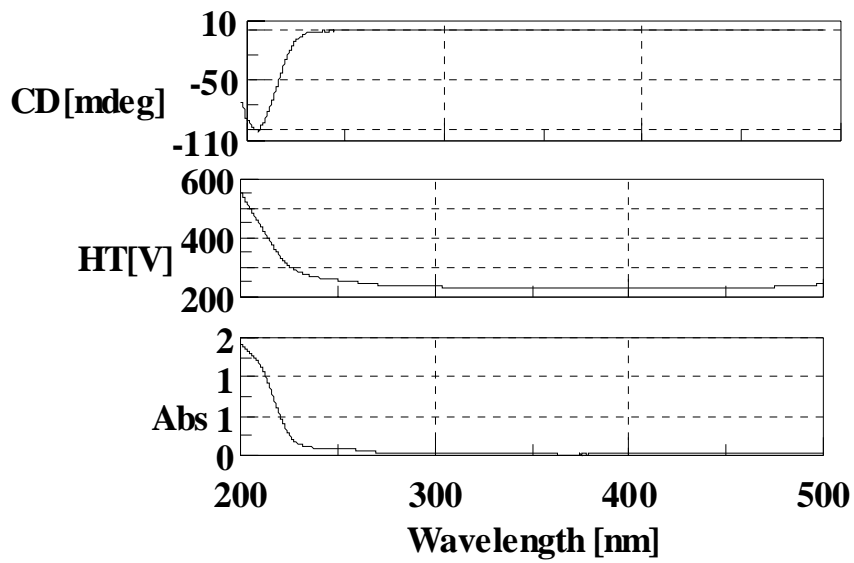
CD of scopararane D (2)



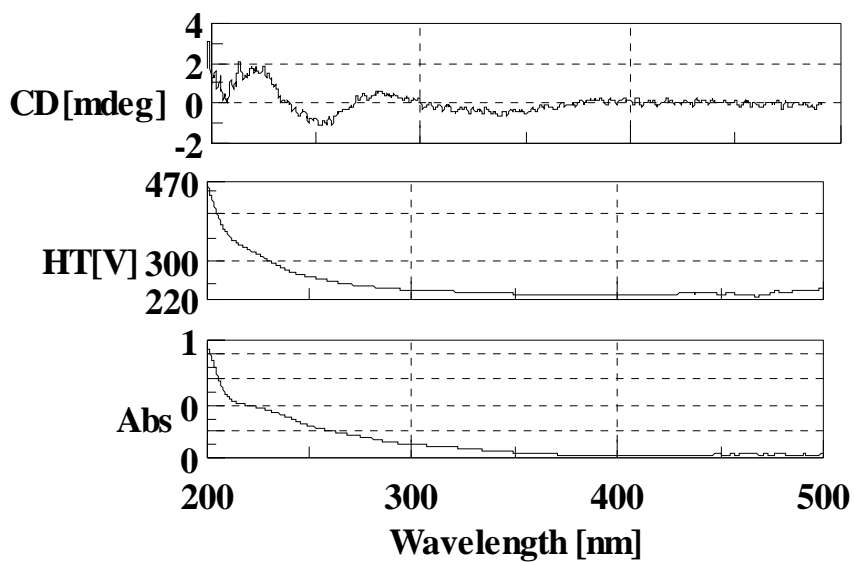
CD of scopararane E (3)



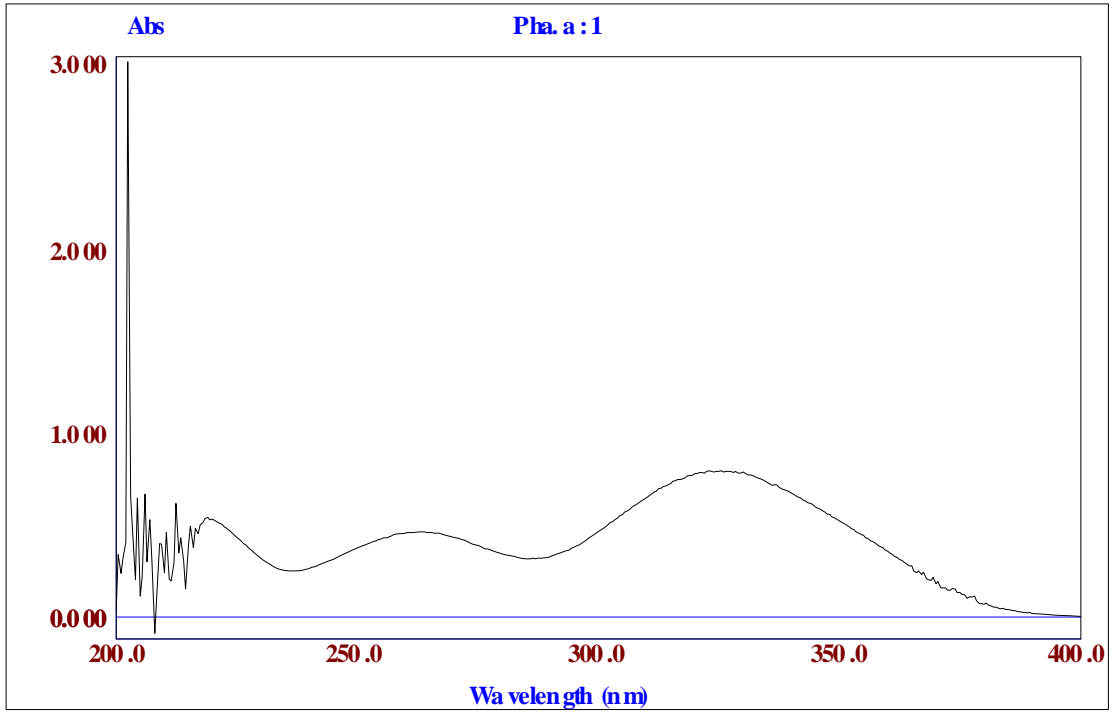
CD of Libertellenone A (6)



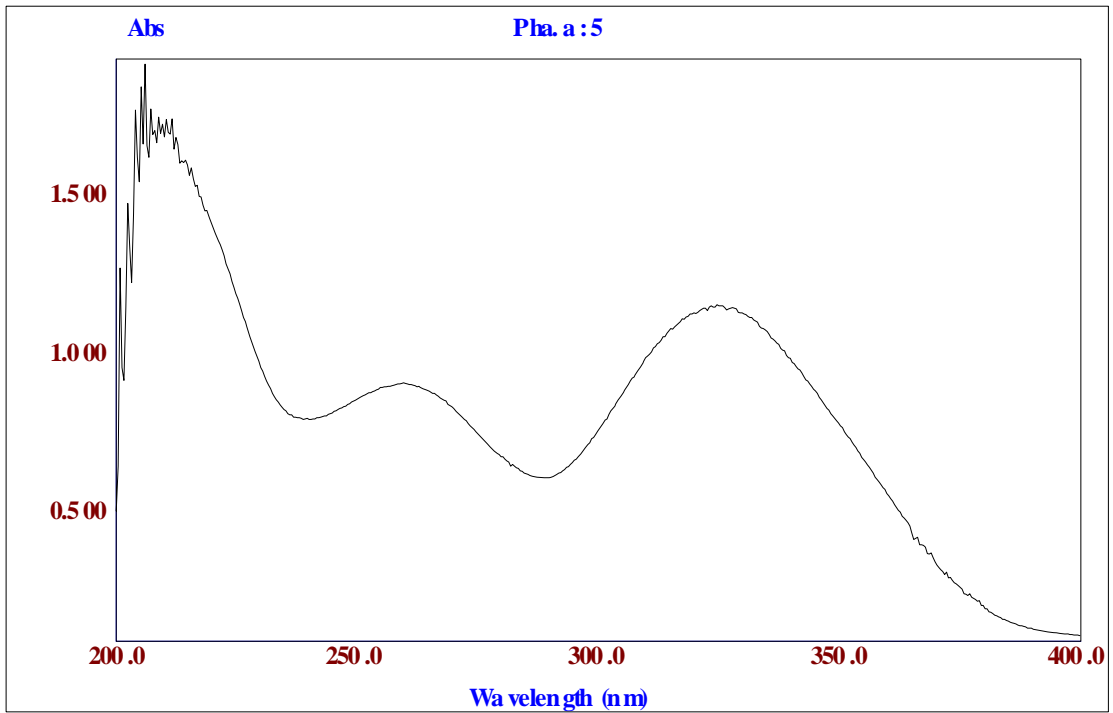
CD of scopararane F (4)



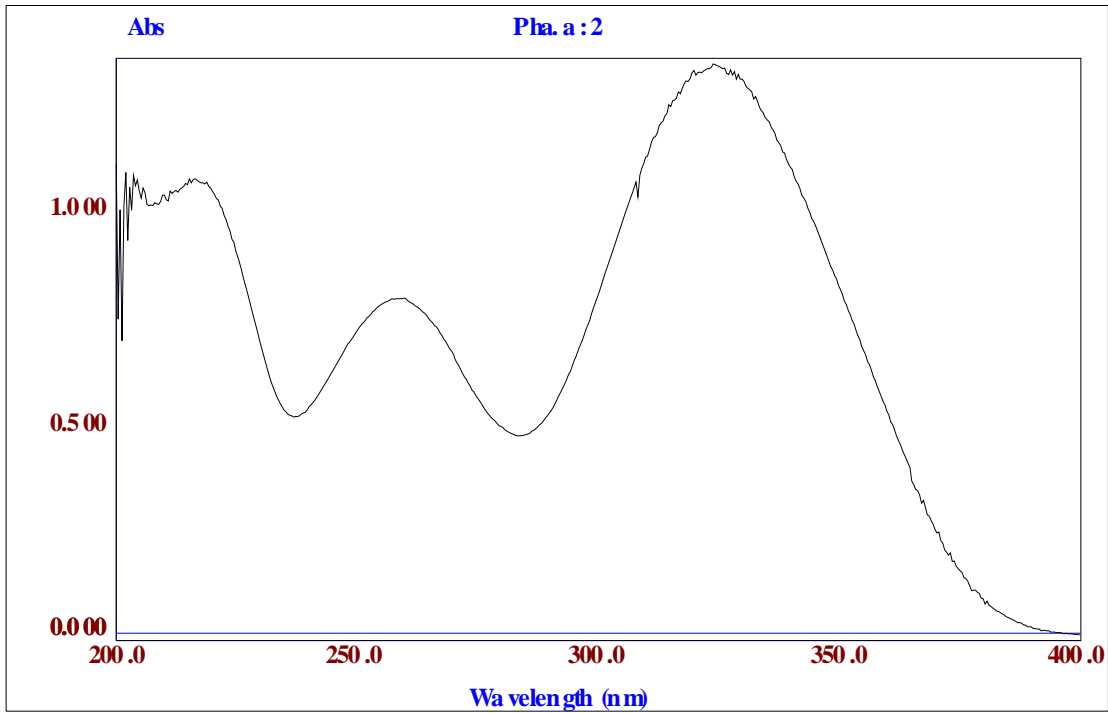
CD of scopararane G (5)



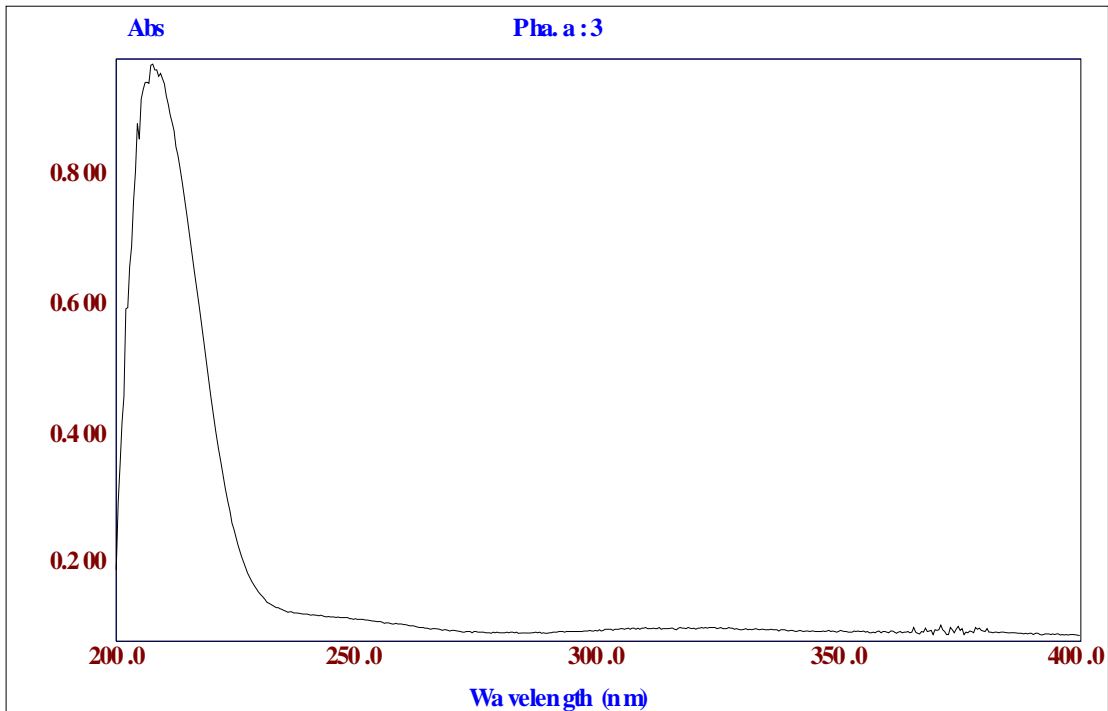
UV of scoparane C (1)



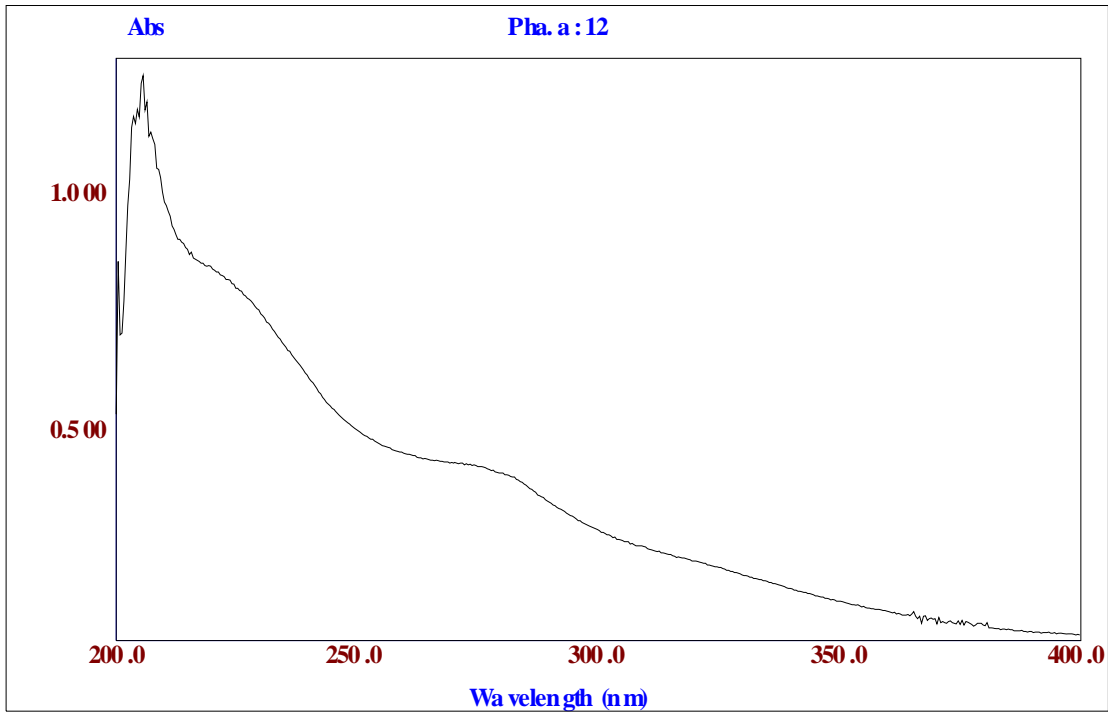
UV of scoparane D (2)



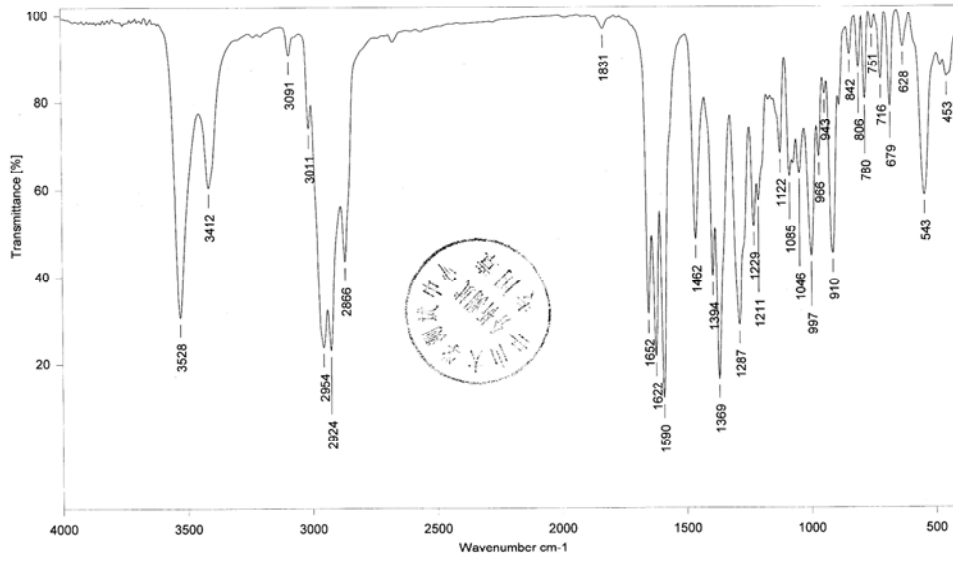
UV of scoparane E (3)



UV of scoparane F (4)



UV of scoparane G (5)

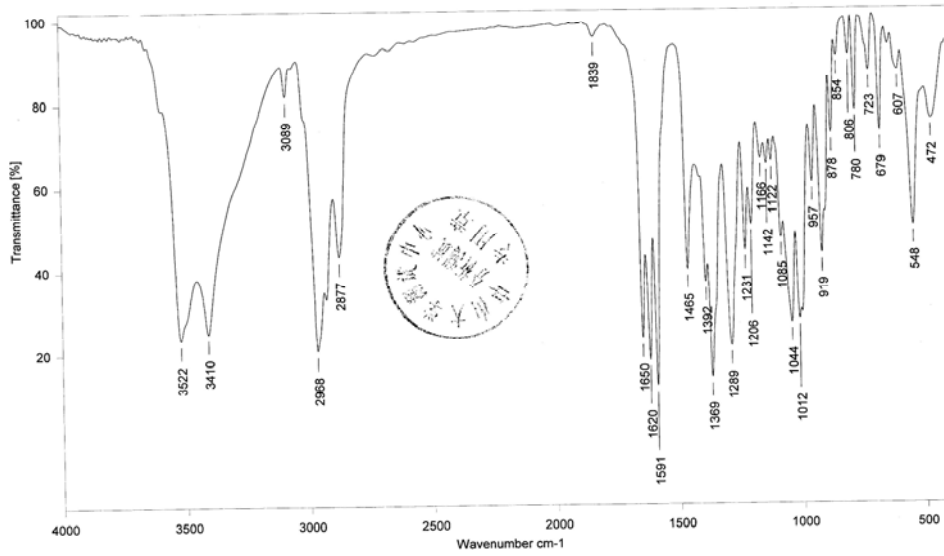


IARC SUN YAT-SEN UNIVERSITY

F
B1009043.2

2010/09/20
16:50:45 (GMT+8)

IR of scoparane C

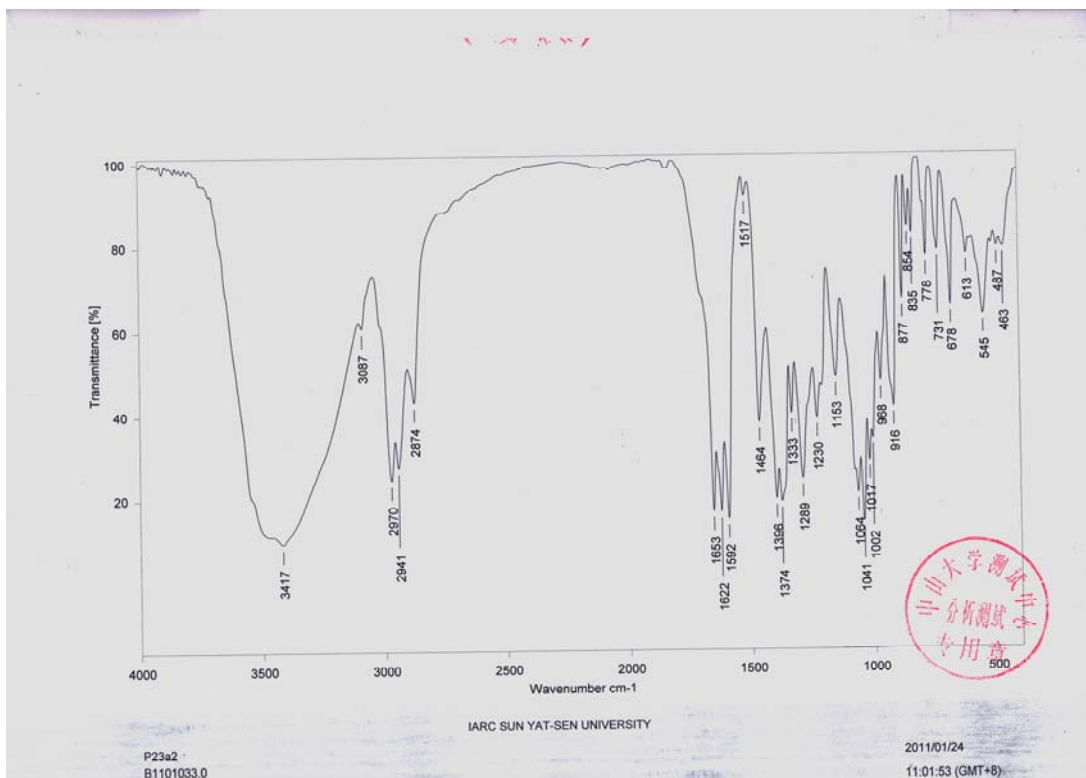


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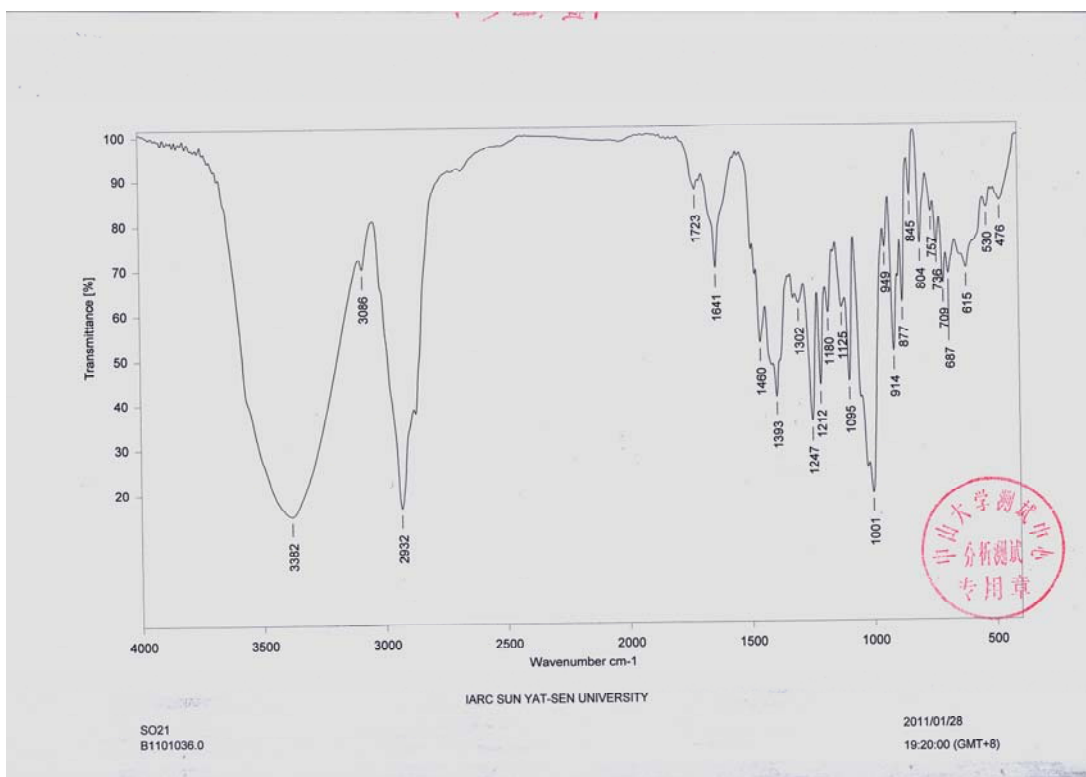
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2010/09/20
17:12:27 (GMT+8)

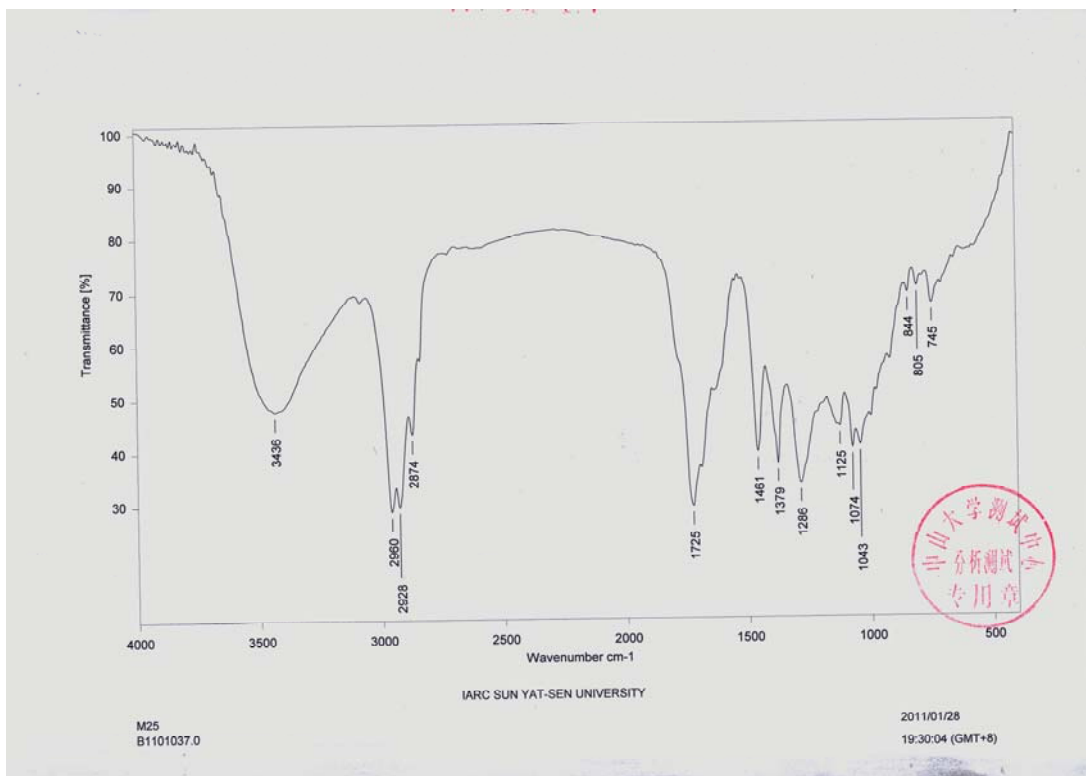
IR of scoparane D



IR of scoparane E



IR of scoparane F

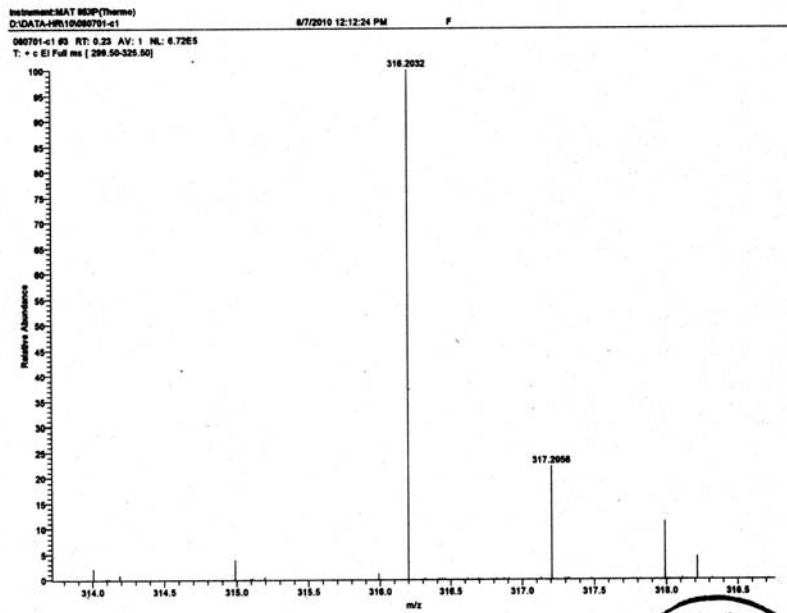


IR of scoparane G

SPECTRUM - MS
File: D:\DATA-HR\10\080701-c1.RAW
Full ms [299.500 - 325.500] - Range: 316.000 - 316.800
Scan No. 3 of 7
Scan #: 3
RT: 0.23

Data points: 1

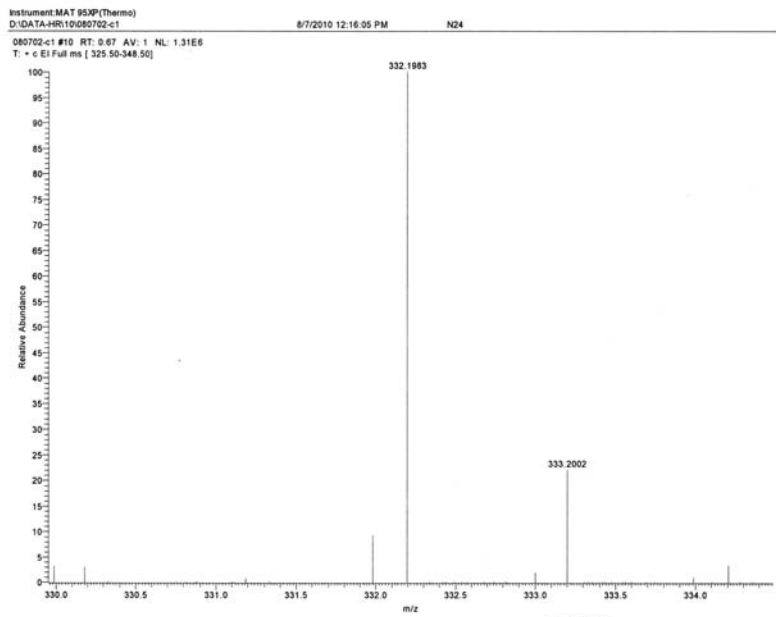
Mass	Relative Intensity	Theoretical Mass	Delta [ppm]	Delta [mmu]	RDB	Composition
316.2032	92.7	316.2033	-0.2	-0.1	7.0	C ₂₀ H ₂₈ O ₃



MS of scopararane C

SPECTRUM - MS
File : D:\DATA-HR\10\080702-c1.RAW
Full ms [325.500 - 348.500] - Range: 332.000 - 332.800
Scan No. 10 of 12
Scan #: 10
RT: 0.67
Data points: 1

Mass	Relative Intensity	Theoretical Mass	Delta [ppm]	Delta [mmu]	RDB	Composition
332.1983	79.4	332.1982	0.2	0.1	7.0	C ₂₀ H ₂₈ O ₄



MS of scopararane D

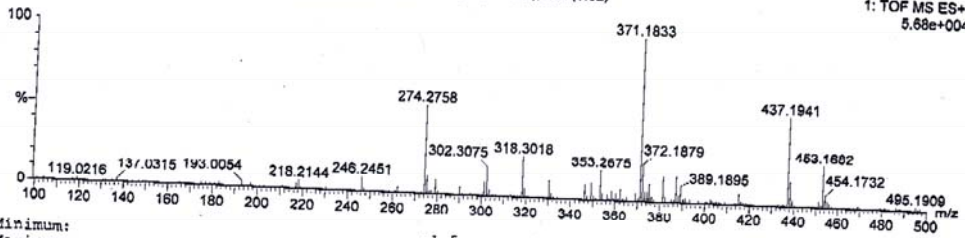
Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
 148 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
 Elements Used:
 C: 0-500 H: 0-1000 O: 0-200 Na: 0-1
 27-Oct-2010 04:13
 P23a2 6 (0.345) AM (Cen,3, 80.00, Ar,5000.0,345.00,0.70,LS 10); Sm (Mn, 2x1.00); Cm (6:32)

1: TOF MS ES+
5.68e+004



Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
371.1833	371.1858	-2.5	6.7	9.5	279.3	C22 H27 O5
200	371.1834	-0.1	-0.3	6.5	318.3	C20 H28 O5 Na ✓
	371.1800	3.3	8.9	18.5	943.1	C29 H23

MS of scopararane E

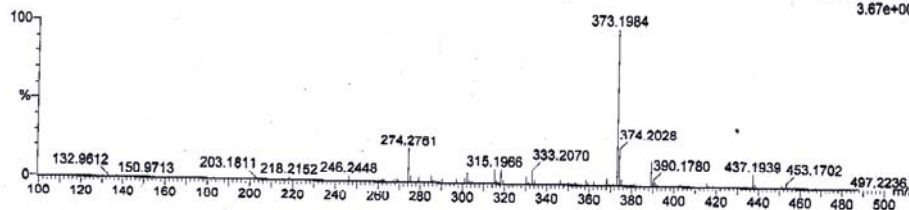
Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
 142 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
 Elements Used:
 C: 0-500 H: 0-1000 O: 0-200 Na: 0-1
 27-Oct-2010 09:55:01
 5021 19 (1.224) AM (Cen,3, 80.00, Ar,5000.0,345.00,0.70,LS 10); Sm (Mn, 2x1.00); Cm (5:28)

1: TOF MS ES+
3.67e+004



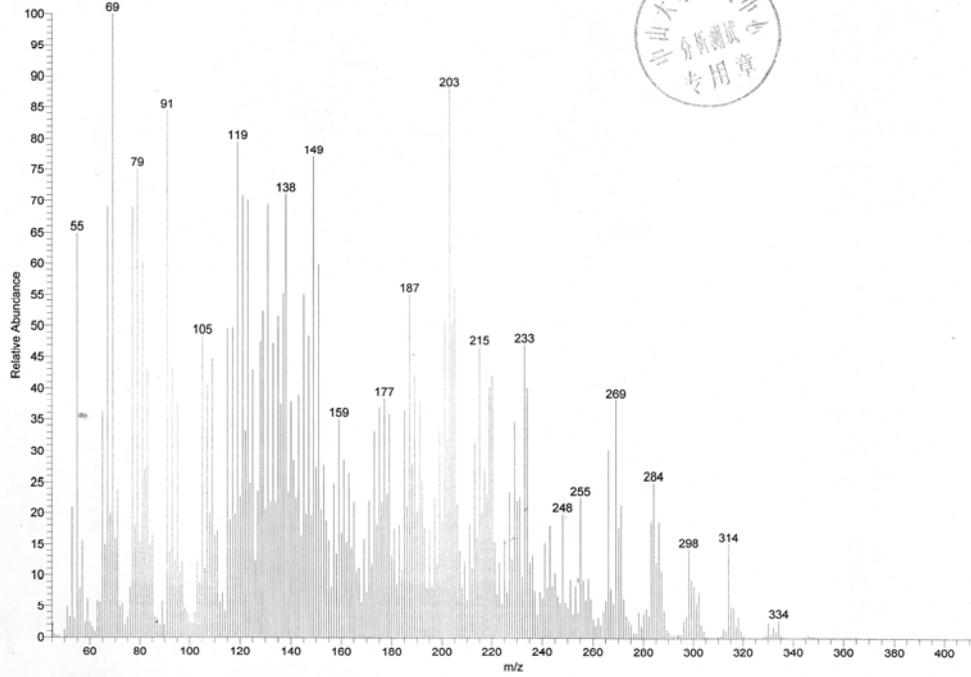
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
373.1984	373.1991	-0.7	-1.9	5.5	19.8	C20 H30 O5 Na ✓
250	373.2015	-3.1	-8.3	8.5	44.3	C22 H29 O5
	373.1956	2.8	7.5	17.5	550.4	C29 H25

MS of scopararane F

Instrument: DSQ(Thermo)
Ionization Method: EI
D:\DSQ\DATA\LR\10\080604
080604 #72 RT: 1.19 AV: 1 NL: 7.53E5
T: +c Full ms [45.00-800.00]

8/6/2010 9:48:31 AM

M25



MS of scoparane G