Supplementary materials

Heterocycles

Semisynthesis of Triptolide Analogues Part IV: Effects of C-14 Carbamothioate Substituents on Cytotoxic Activities

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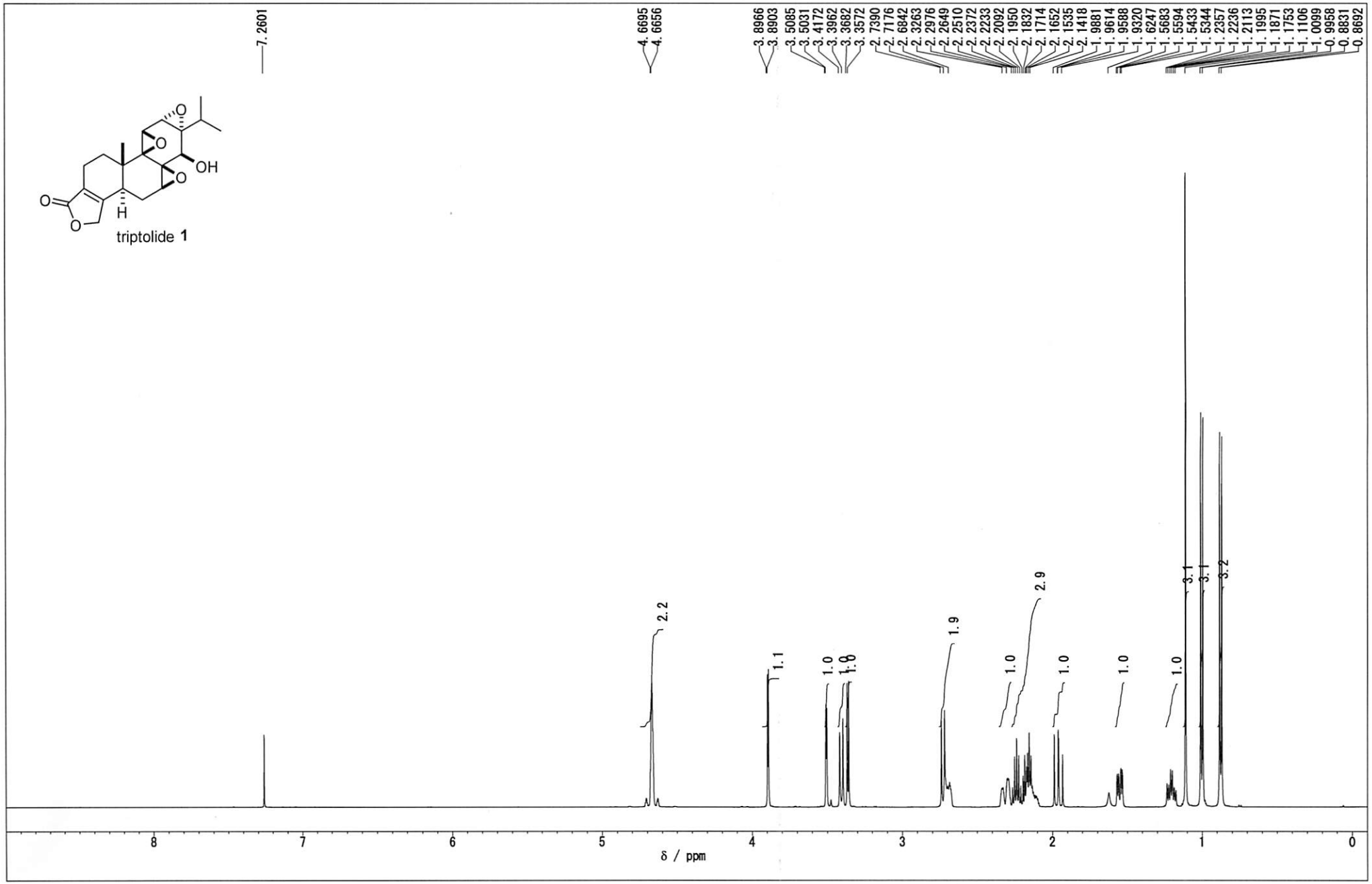
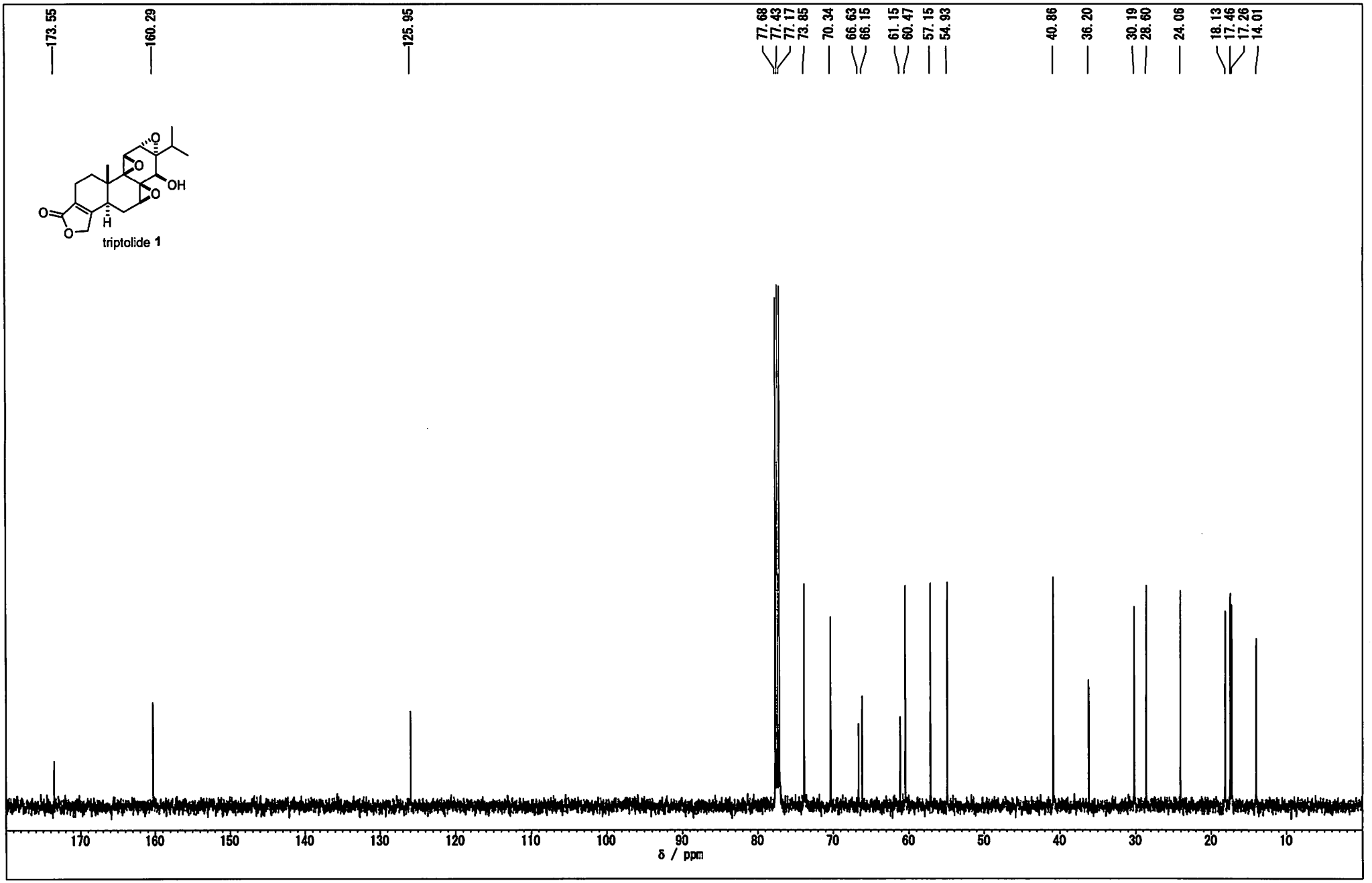
List of Figures

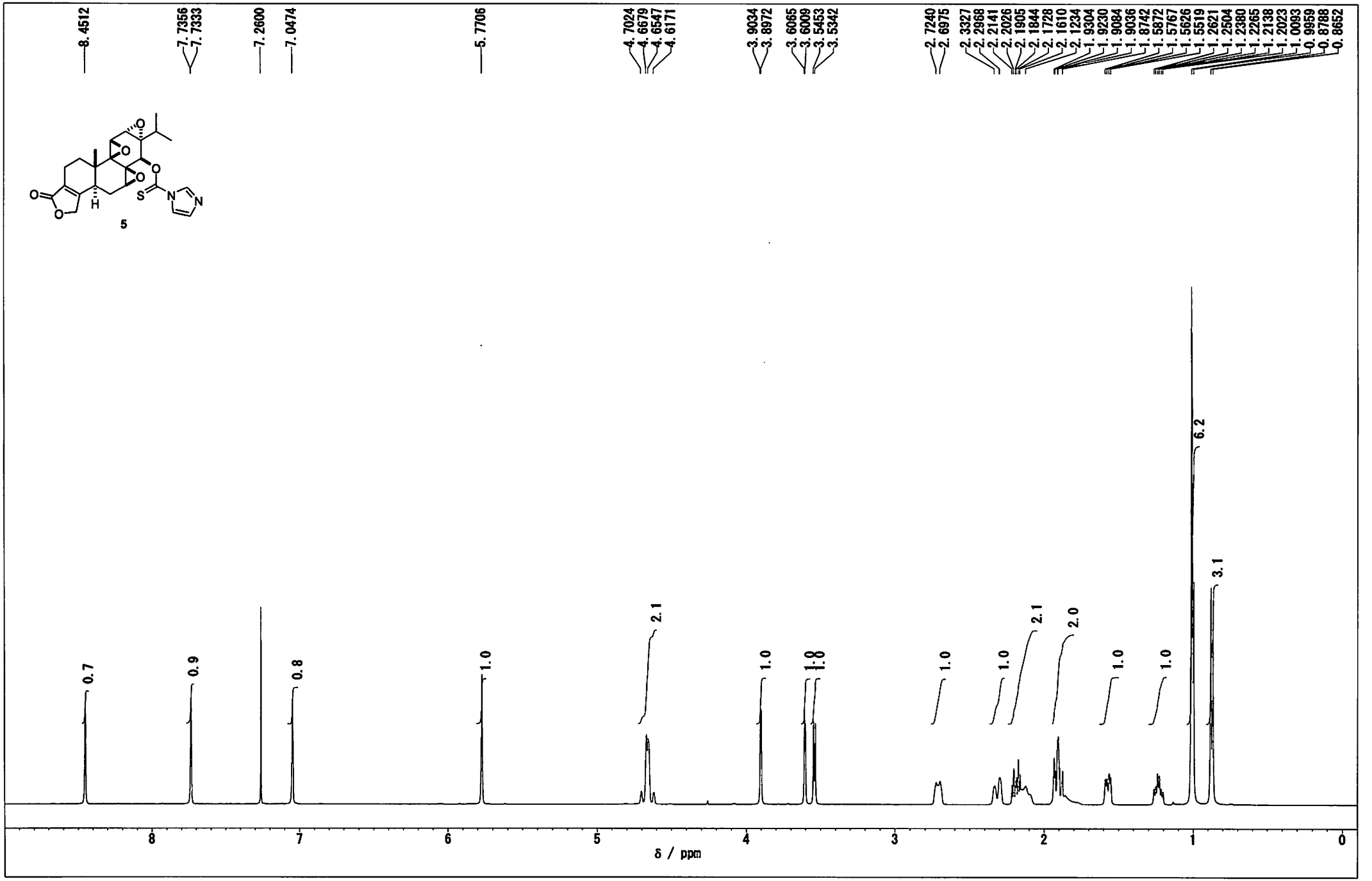
triptolide_1H/300K/DRX500/CDCl3



Triptolide_13C/300K/CDCl3/DRX500



YA01462_1H/CDCl3/300K/AM500



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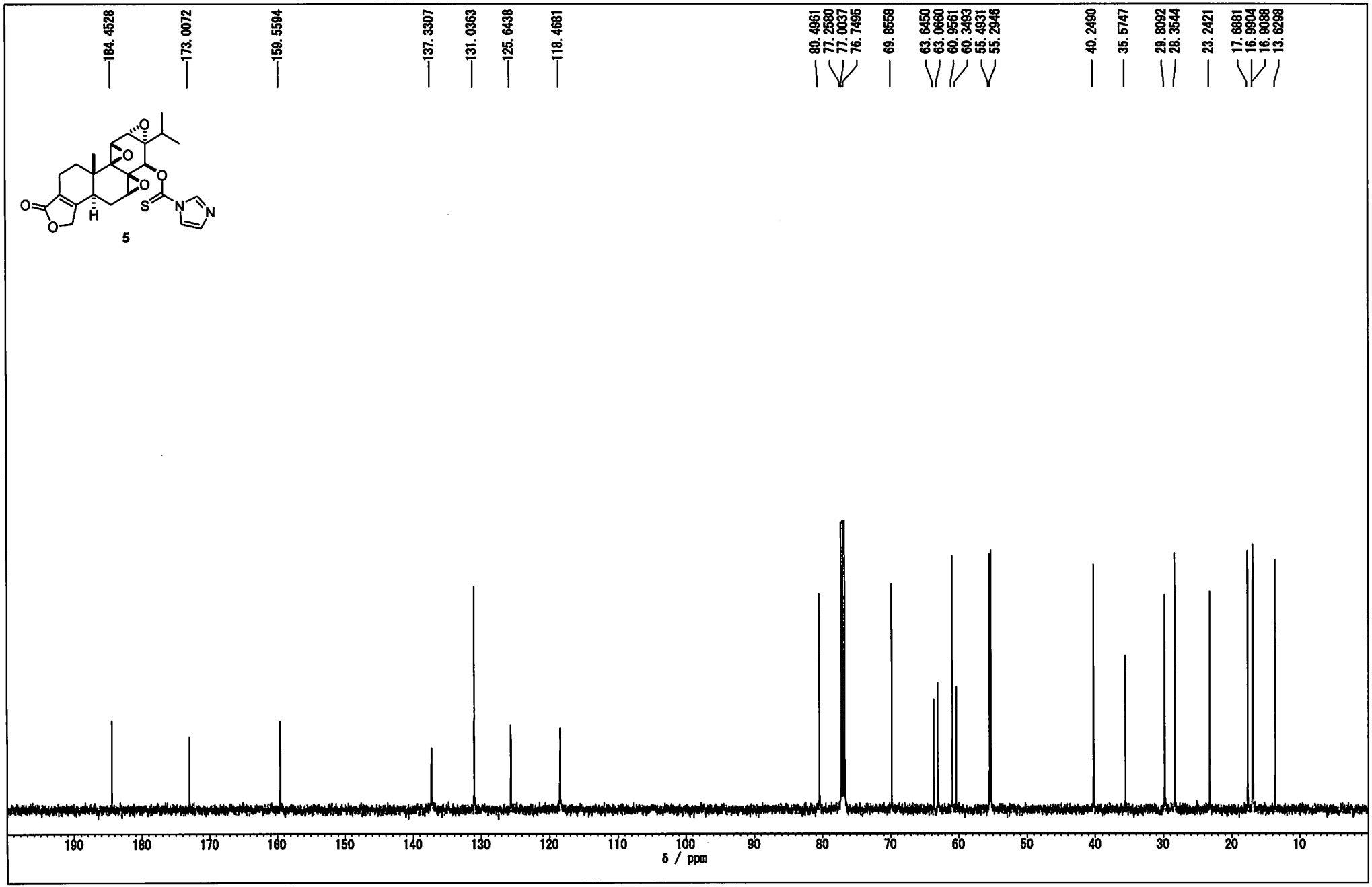

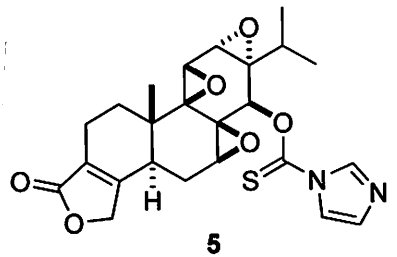

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YA01460_DEPT90/CDCl3/DRX500/300K

ppm

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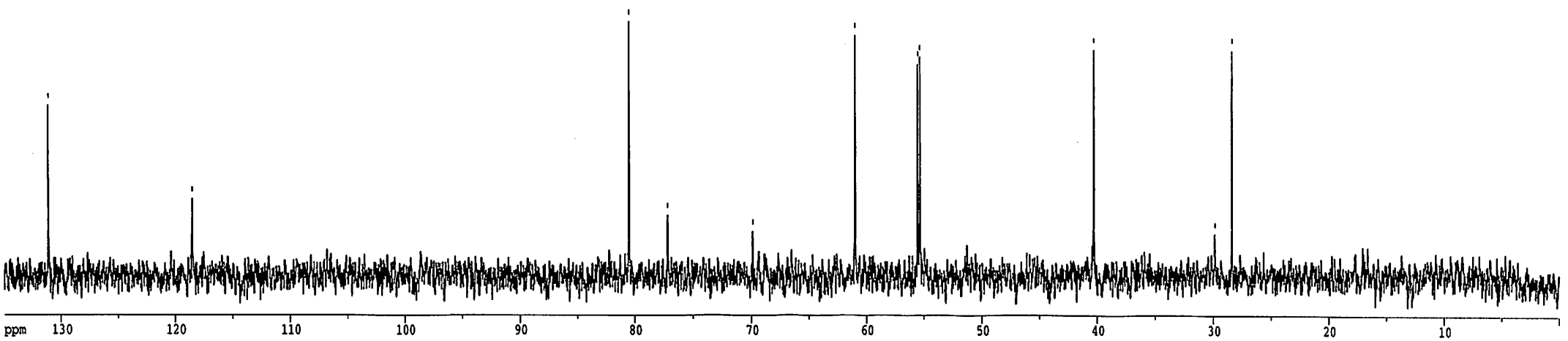
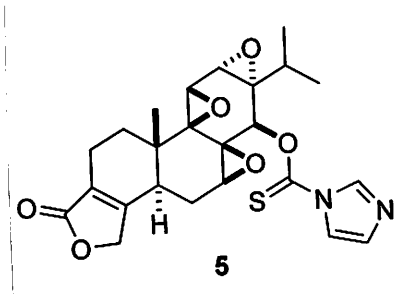
80.503
77.204
69.879
60.981
55.526
55.349
40.309
29.869
28.396





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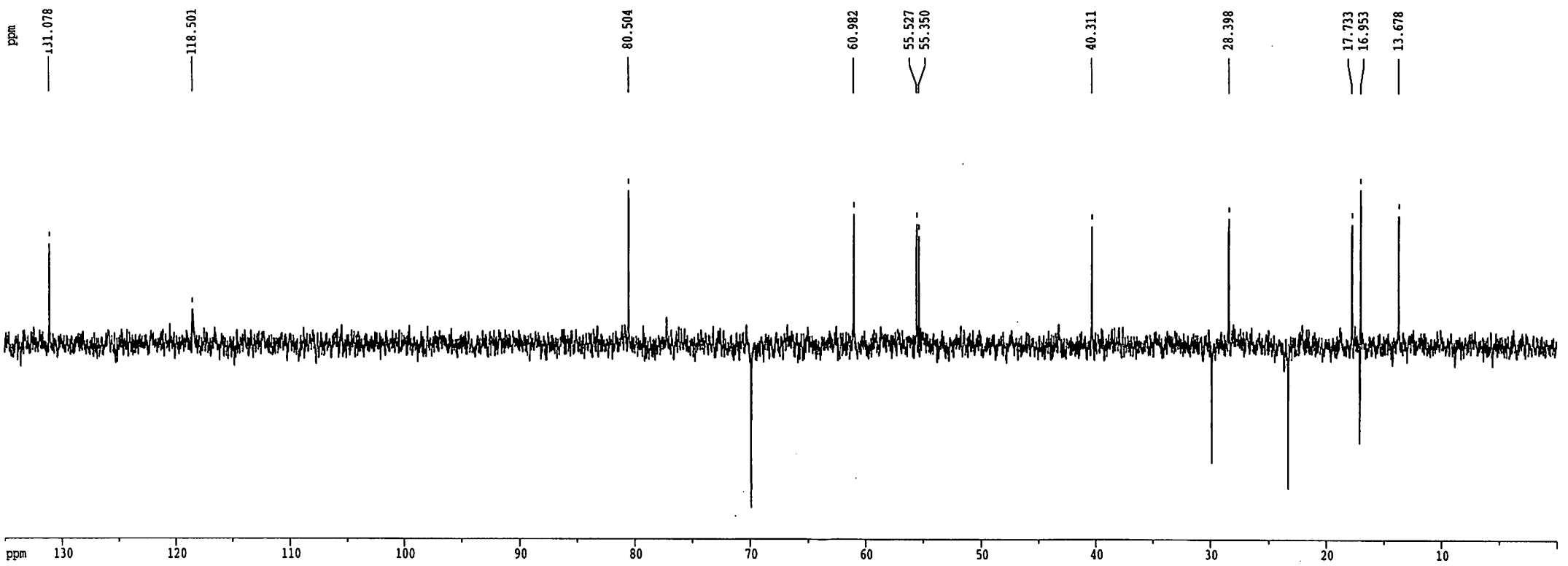
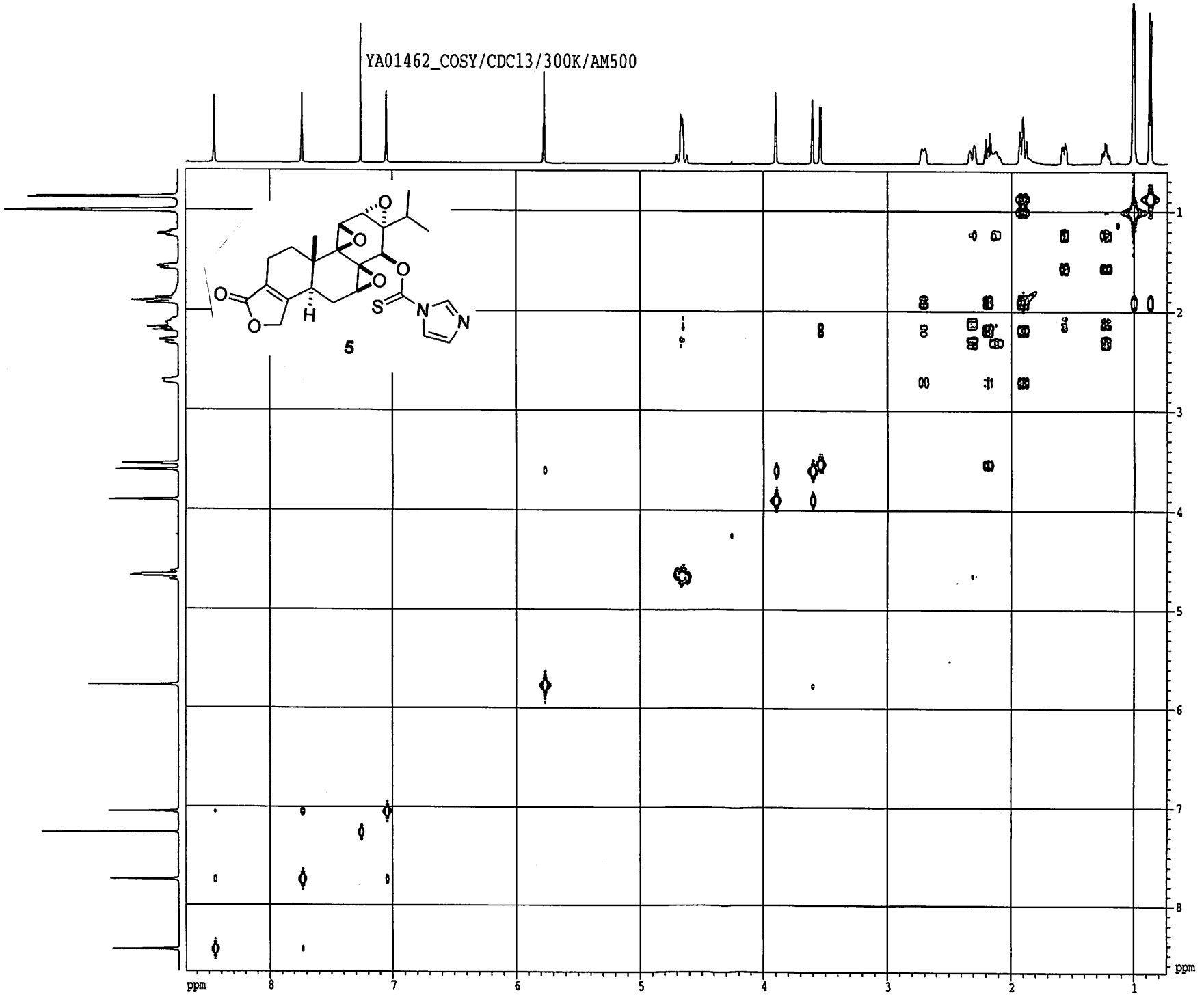
YA01460_DEPT135/CDCl3/DRX500/300K





Current Data Parameters

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EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20040715
Time	12.40
INSTRUM	drx500
PROBHD	5 mm Multinu
PULPROG	cosygs
TD	2048
SOLVENT	CDCl3
NS	1
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	512
DW	111.200 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	2.00000000 sec
d13	0.00000300 sec
D16	0.00010000 sec
IN0	0.00022221 sec

========== CHANNEL f1 ==========

NUC1	1H
P0	11.60 usec
P1	11.60 usec
PL1	-4.00 dB
SFO1	500.0325001 MHz

========== GRADIENT CHANNEL ========

P16	1000.00 usec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	500.0325 MHz
FIDRES	17.578894 Hz
SW	9.000 ppm

F2 - Processing parameters

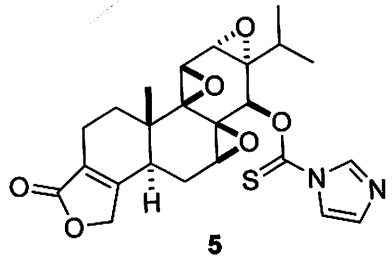
SI	1024
SF	500.0300119 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	1024
MC2	QF
SF	500.0300102 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	27.00 cm
CX1	22.00 cm
F2PLO	8.691 ppm
F2LO	4345.62 Hz
F2PHI	0.735 ppm
F2HI	367.35 Hz
F1PLO	8.680 ppm
F1LO	4340.15 Hz
F1PHI	0.603 ppm
F1HI	301.40 Hz
F2PPMCM	0.29467 ppm/cm
F2HZCM	147.34306 Hz/cm
F1PPMCM	0.36714 ppm/cm
F1HZCM	183.57957 Hz/cm



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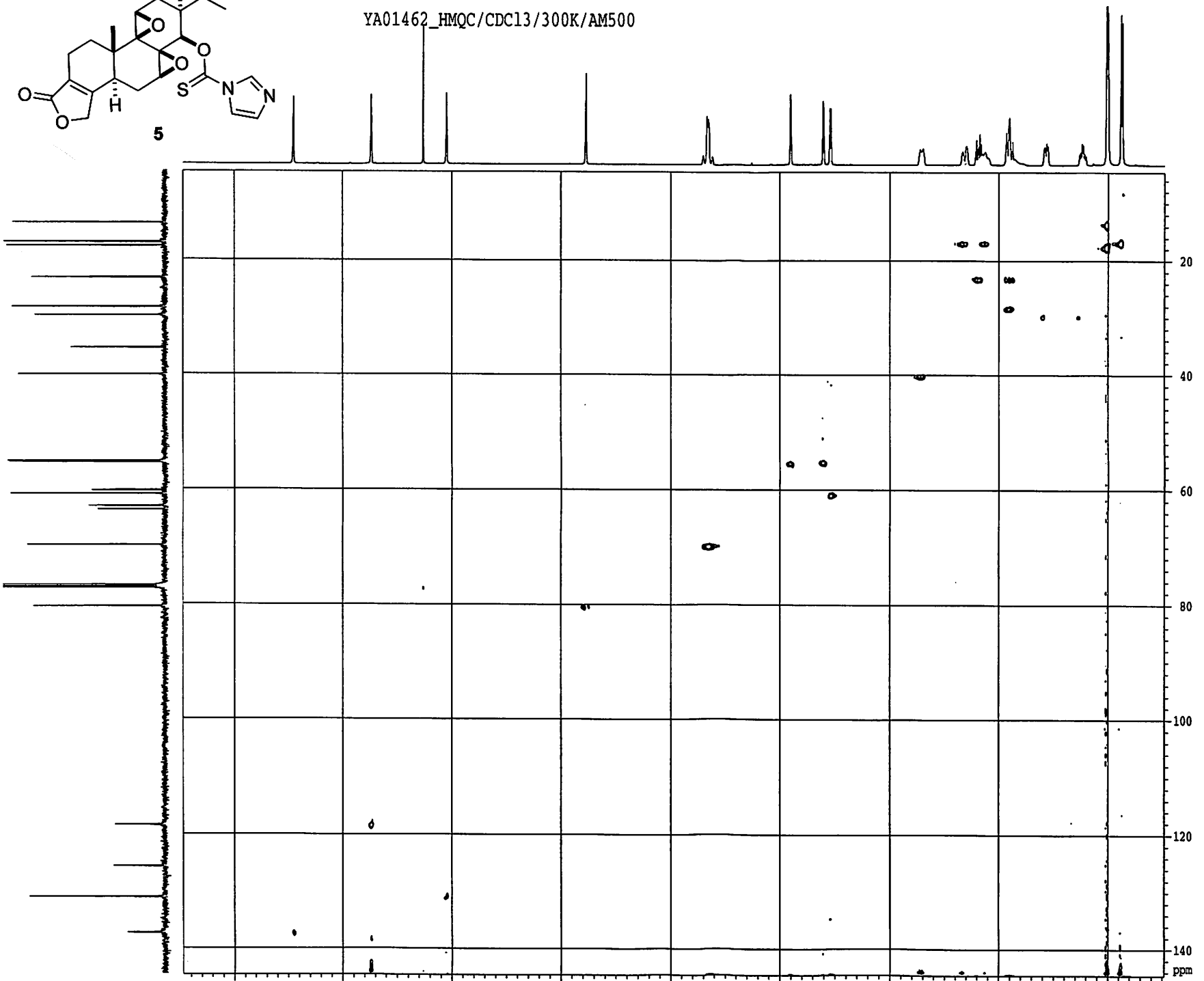


Current Data Parameters

NAME	YA01462
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20040715
Time	12.51
INSTRUM	drx500
PROBHD	5 mm Multinu
PULPROG	inv4gstp
TD	2048
SOLVENT	CDCl3
NS	2
DS	32
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	32768
DW	111.200 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	2.00000000 sec
d2	0.00344828 sec
d4	0.00172414 sec
d11	0.03000000 sec
d13	0.00000300 sec
D16	0.00010000 sec
d20	0.00062414 sec
d21	0.00234428 sec
IN0	0.00001420 sec

============ CHANNEL f1 ============

NUC1	1H
P1	11.60 usec
p2	23.20 usec
PL1	-4.00 dB
SFO1	500.0325001 MHz

============ CHANNEL f2 ============

CPDPRG2	garp
NUC2	13C
P3	9.00 usec
p4	18.00 usec
PCPD2	80.00 usec
PL2	1.00 dB
PL12	14.00 dB
SFO2	125.7420239 MHz

============ GRADIENT CHANNEL ========

P16	1000.00 usec

F1 - Acquisition parameters

ND0	4
TD	512
SFO1	125.742 MHz
FIDRES	34.386005 Hz
SW	140.014 ppm

F2 - Processing parameters

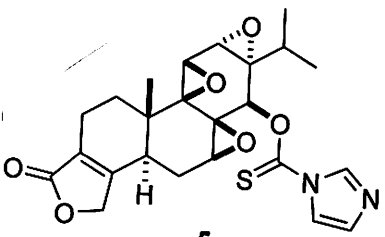
SI	1024
SF	500.0300098 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	512
MC2	TPPI
SF	125.7326462 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	27.00 cm
CX1	22.00 cm
F2PLO	9.477 ppm
F2LO	4738.59 Hz
F2PHI	0.484 ppm
F2HI	242.19 Hz
F1PLO	144.597 ppm
F1LO	18180.55 Hz
F1PHI	4.572 ppm
F1HI	574.91 Hz
F2PPMCM	0.33305 ppm/cm
F2HZCM	166.53343 Hz/cm
F1PPMCM	6.36475 ppm/cm
F1HZCM	800.25641 Hz/cm



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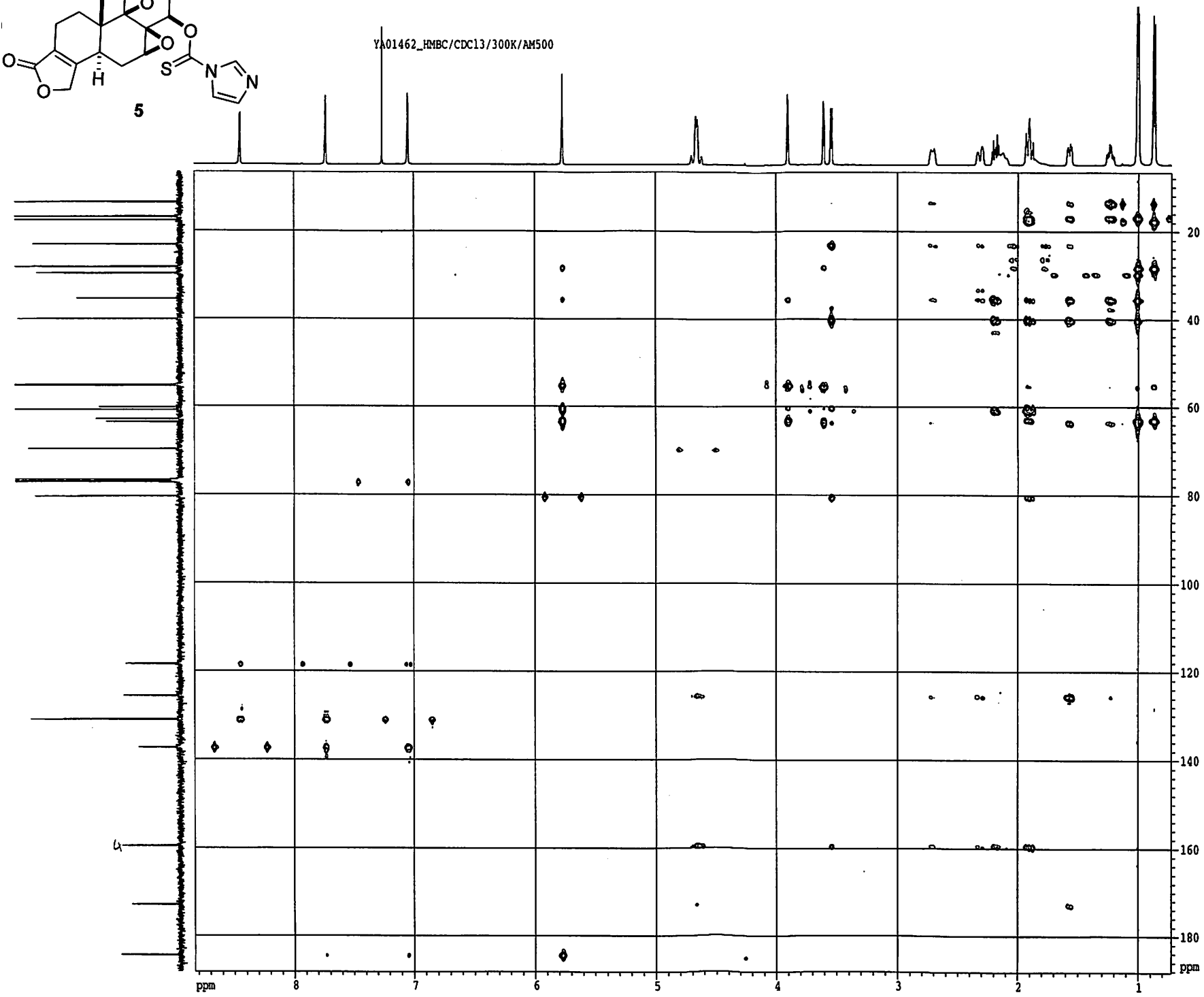


Current Data Parameters

NAME	YA01462
EXPNO	7
PROCNO	1

F2 - Acquisition Parameters

Date_	20040715
Time	13.31
INSTRUM	drx500
PROBHD	5 mm Multinu
PULPROG	inv4gslplrnd
TD	2048
SOLVENT	CDCl3
NS	8
DS	32
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	16384
DW	111.200 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	2.00000000 sec
d2	0.00344828 sec
D6	0.06500000 sec
d13	0.00000300 sec
D16	0.00010000 sec
IN0	0.00001989 sec

============ CHANNEL f1 ============

NUC1	1H
P1	11.60 usec
p2	23.20 usec
PL1	-4.00 dB
SFO1	500.0325001 MHz

============ CHANNEL f2 ============

NUC2	13C
P3	9.00 usec
PL2	1.00 dB
SFO2	125.7451673 MHz

============ GRADIENT CHANNEL ========

P16	1000.00 usec

F1 - Acquisition parameters

ND0	2
TD	512
SFO1	125.7452 MHz
FIDRES	49.104336 Hz
SW	199.939 ppm

F2 - Processing parameters

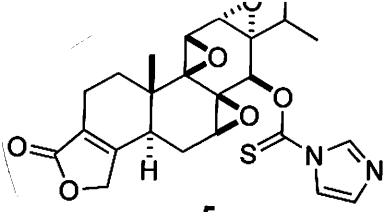
SI	1024
SF	500.0300111 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	512
MC2	QF
SF	125.7326430 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	27.00 cm
CX1	22.00 cm
F2PLO	8.833 ppm
F2LO	4416.74 Hz
F2PHI	0.728 ppm
F2HI	363.83 Hz
F1PLO	188.264 ppm
F1LO	23670.93 Hz
F1PHI	6.660 ppm
F1HI	837.41 Hz
F2PPMCM	0.30020 ppm/cm
F2HZCM	150.10777 Hz/cm
F1PPMCM	8.25471 ppm/cm
F1HZCM	1037.88708 Hz/cm



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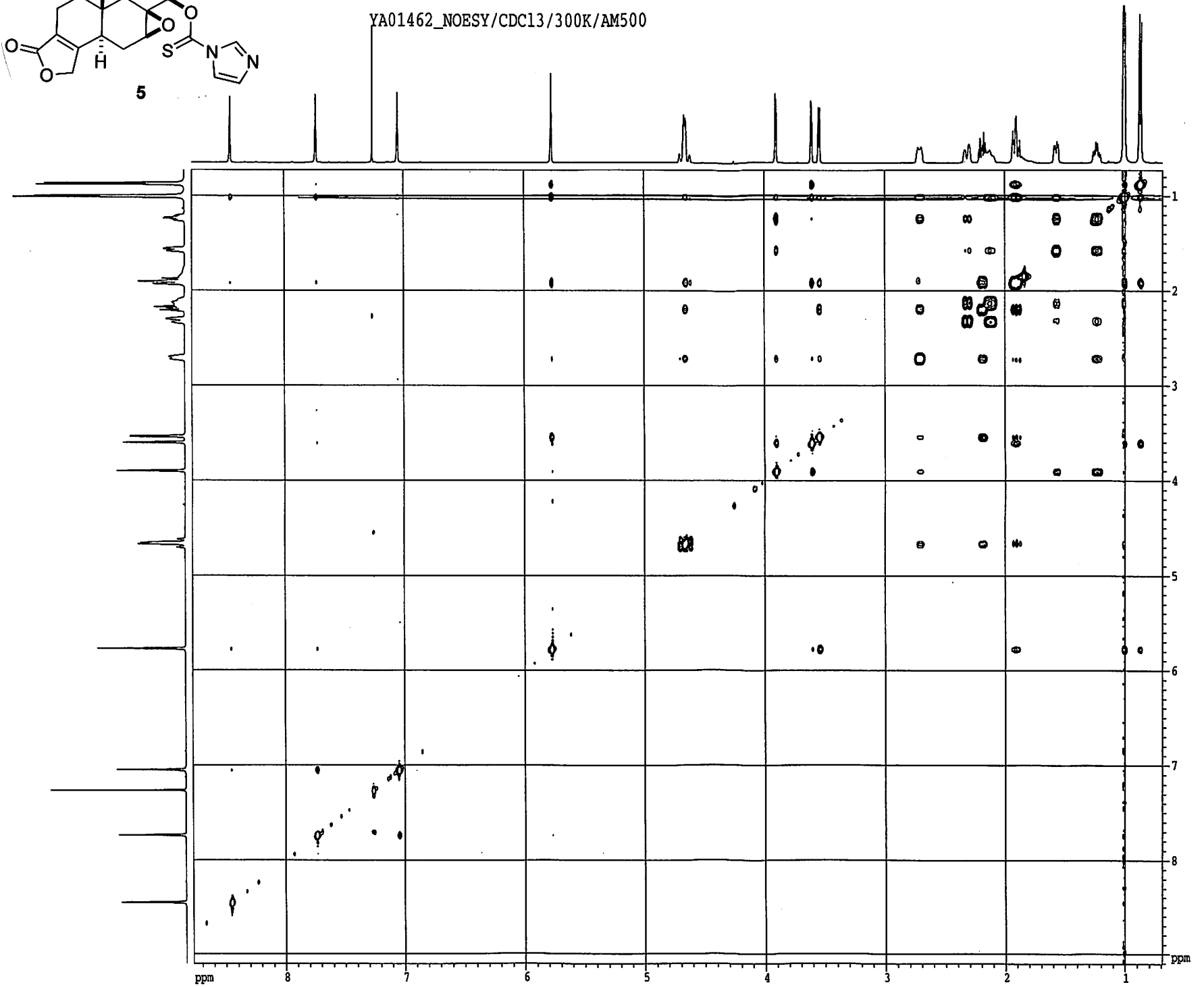
YA01462_NOESY/CDCl3/300K/AM500



Current Data Parameters

NAME	YA01462
EXPNO	8
PROCNO	1

F2 - Acquisition Parameters

Date_	20040715
Time	16.10
INSTRUM	drx500
PROBHD	5 mm Multinu
PULPROG	noesytp
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	5000.000 Hz
FIDRES	2.441406 Hz
AQ	0.2048500 sec
RG	256
DW	100.000 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	2.00000000 sec
D8	1.00000000 sec
IN0	0.00009999 sec

============ CHANNEL f1 ============

NUC1	1H
P1	11.60 usec
PL1	-4.00 dB
SFO1	500.0325001 MHz

F1 - Acquisition parameters

ND0	2
TD	500
SFO1	500.0325 MHz
FIDRES	10.001250 Hz
SW	10.001 ppm

F2 - Processing parameters

SI	1024
SF	500.0300108 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40

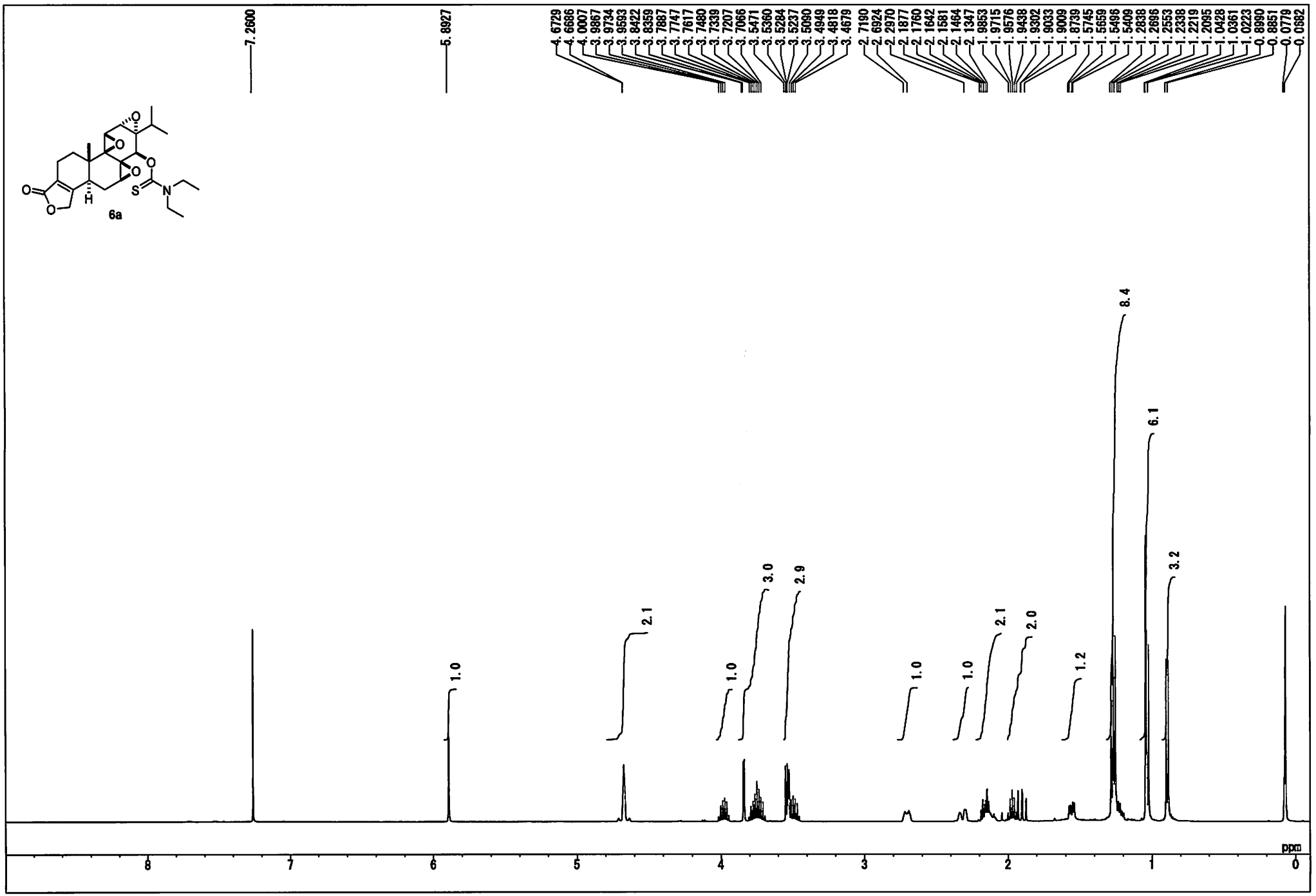
F1 - Processing parameters

SI	512
MC2	TPPI
SF	500.0300070 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

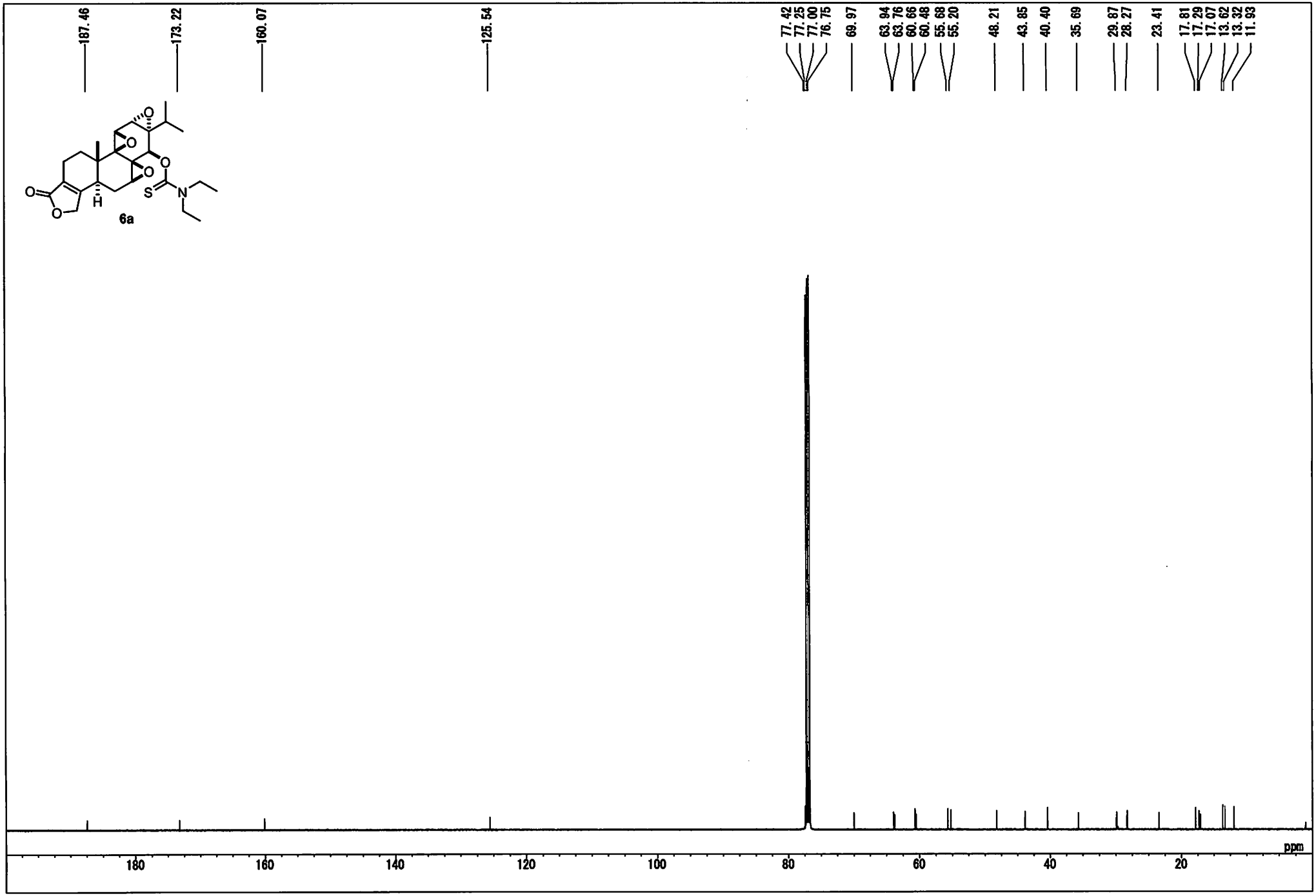
2D NMR plot parameters

CX2	27.00 cm
CX1	22.00 cm
F2PLO	8.777 ppm
F2LO	4388.80 Hz
F2PHI	0.692 ppm
F2HI	345.83 Hz
F1PLO	9.107 ppm
F1LO	4553.99 Hz
F1PHI	0.728 ppm
F1HI	364.02 Hz
F2PPMCM	0.29946 ppm/cm
F2HZCM	149.73958 Hz/cm
F1PPMCM	0.38088 ppm/cm
F1HZCM	190.45349 Hz/cm

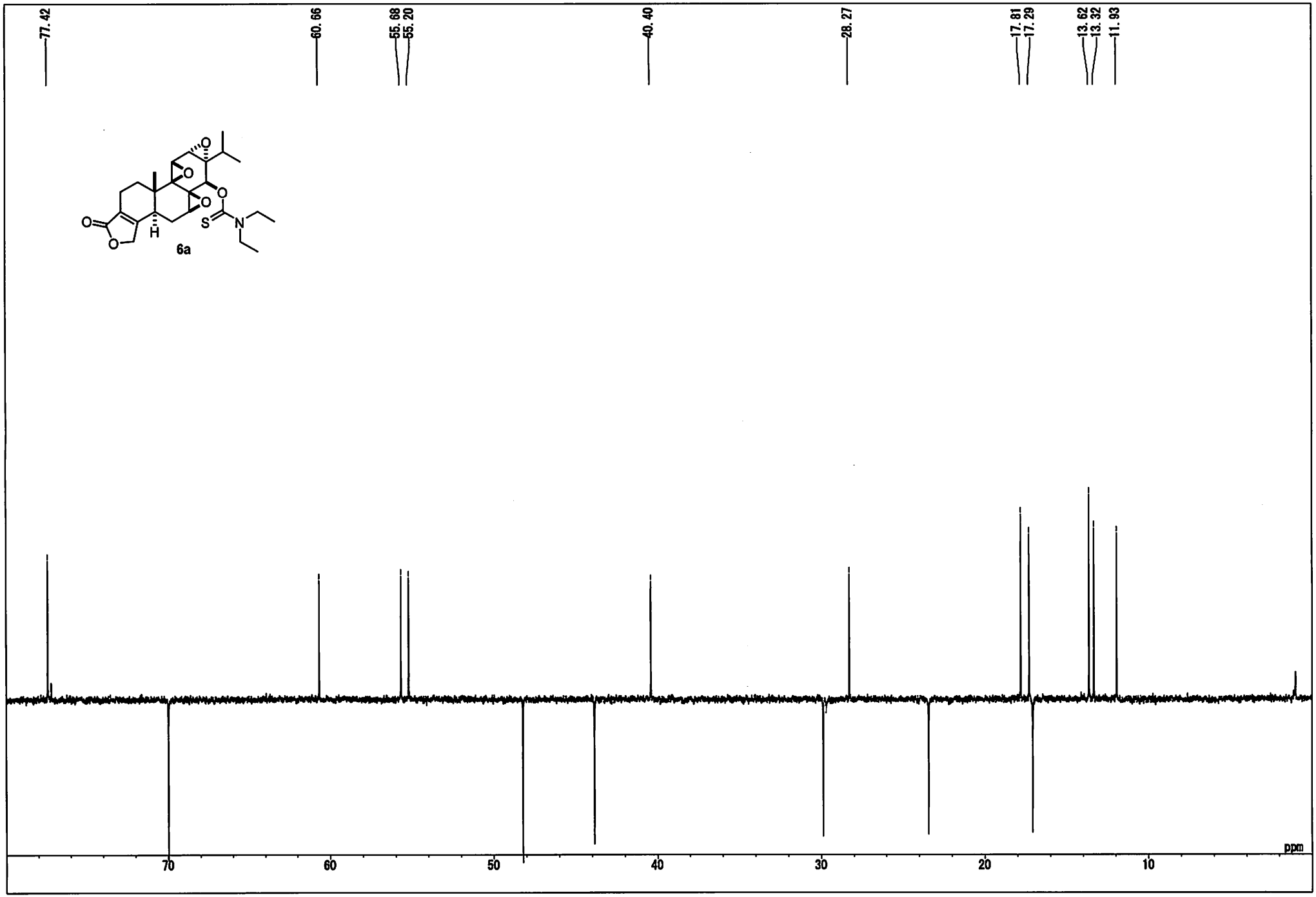
CK20-1, 500MHz, CDCl3, take-2, 2014/12/27

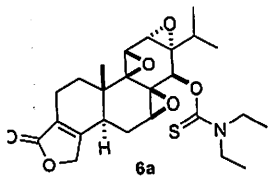


CK20-1, 125MHz, CDCl3, 2014/12/27



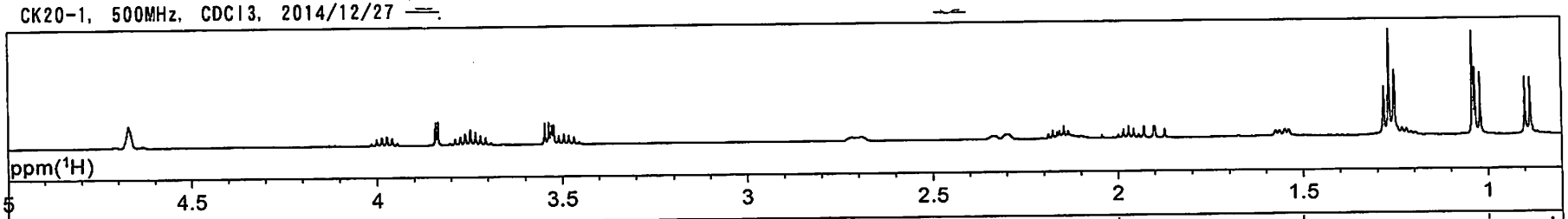
CK20-1, dept135, CDCl3, 2014/12/27

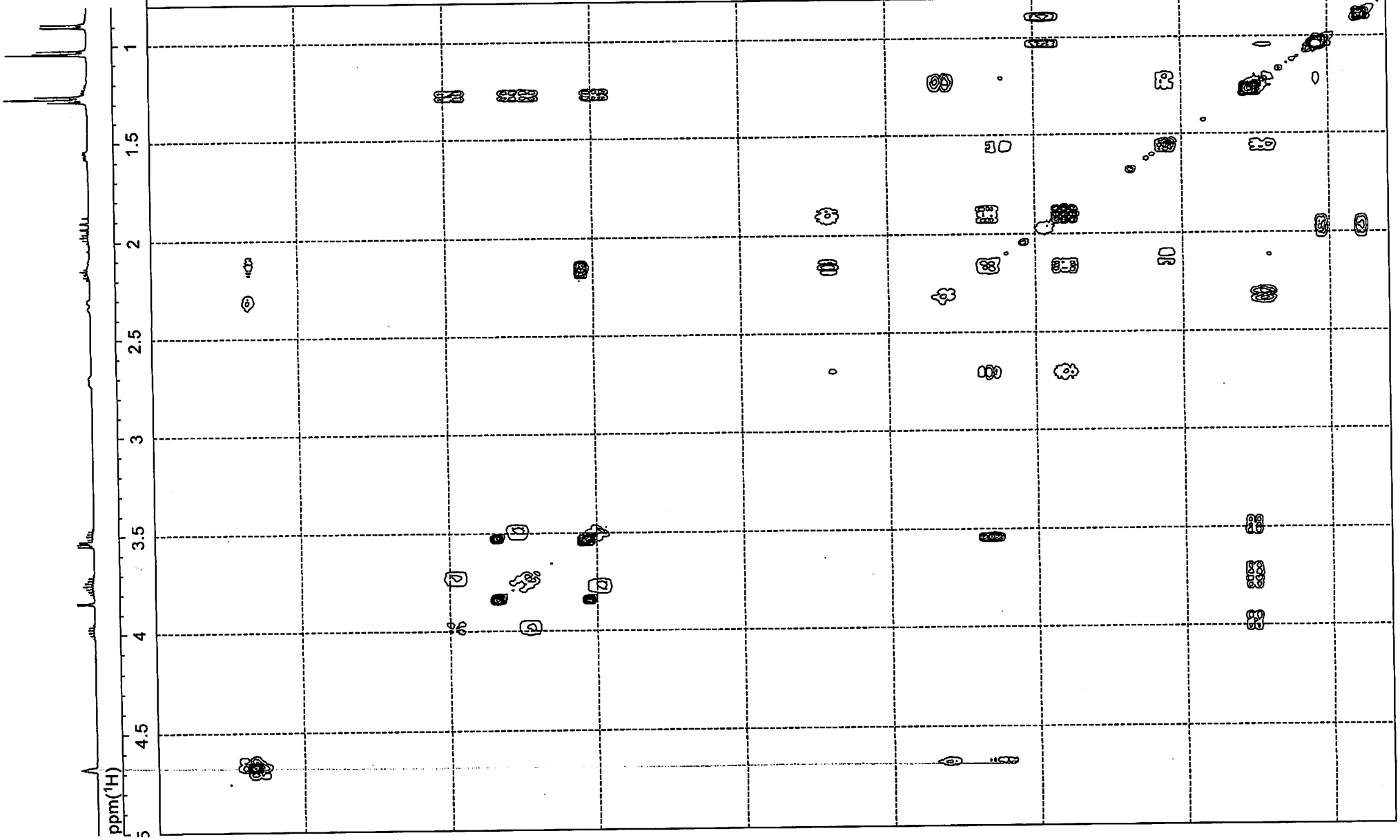


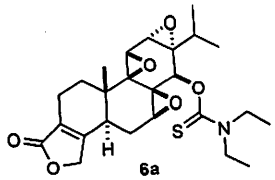


COSY

CK20-1, 500MHz, CDCl3, 2014/12/27

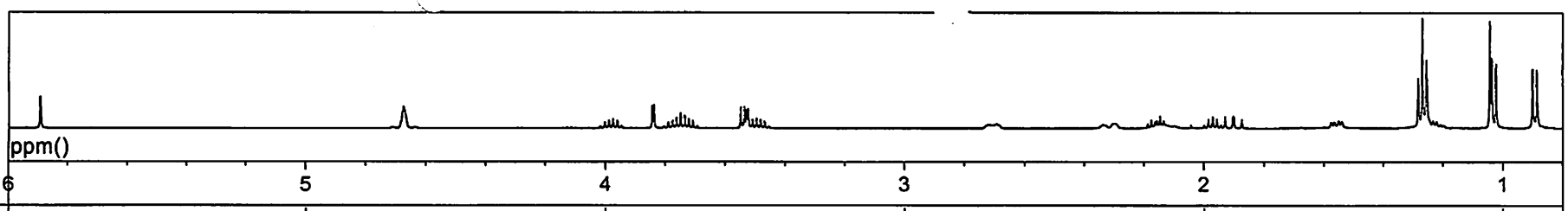


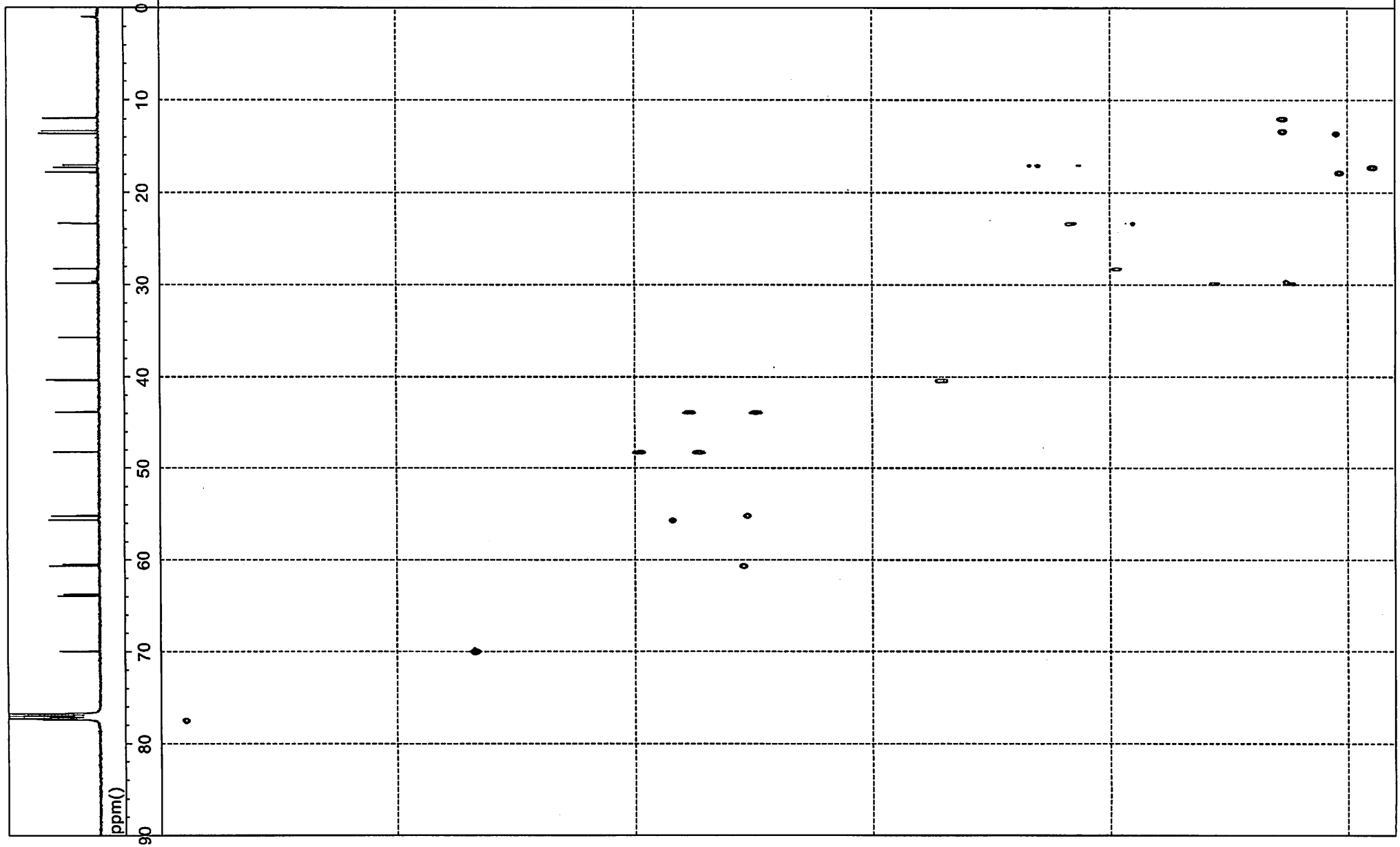


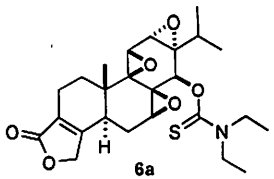


6a

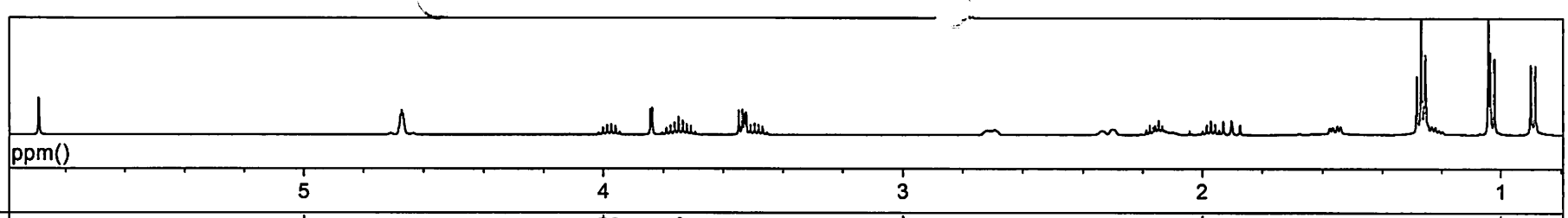
CK20-1, hsqc, CDCl3, 2014/12/27

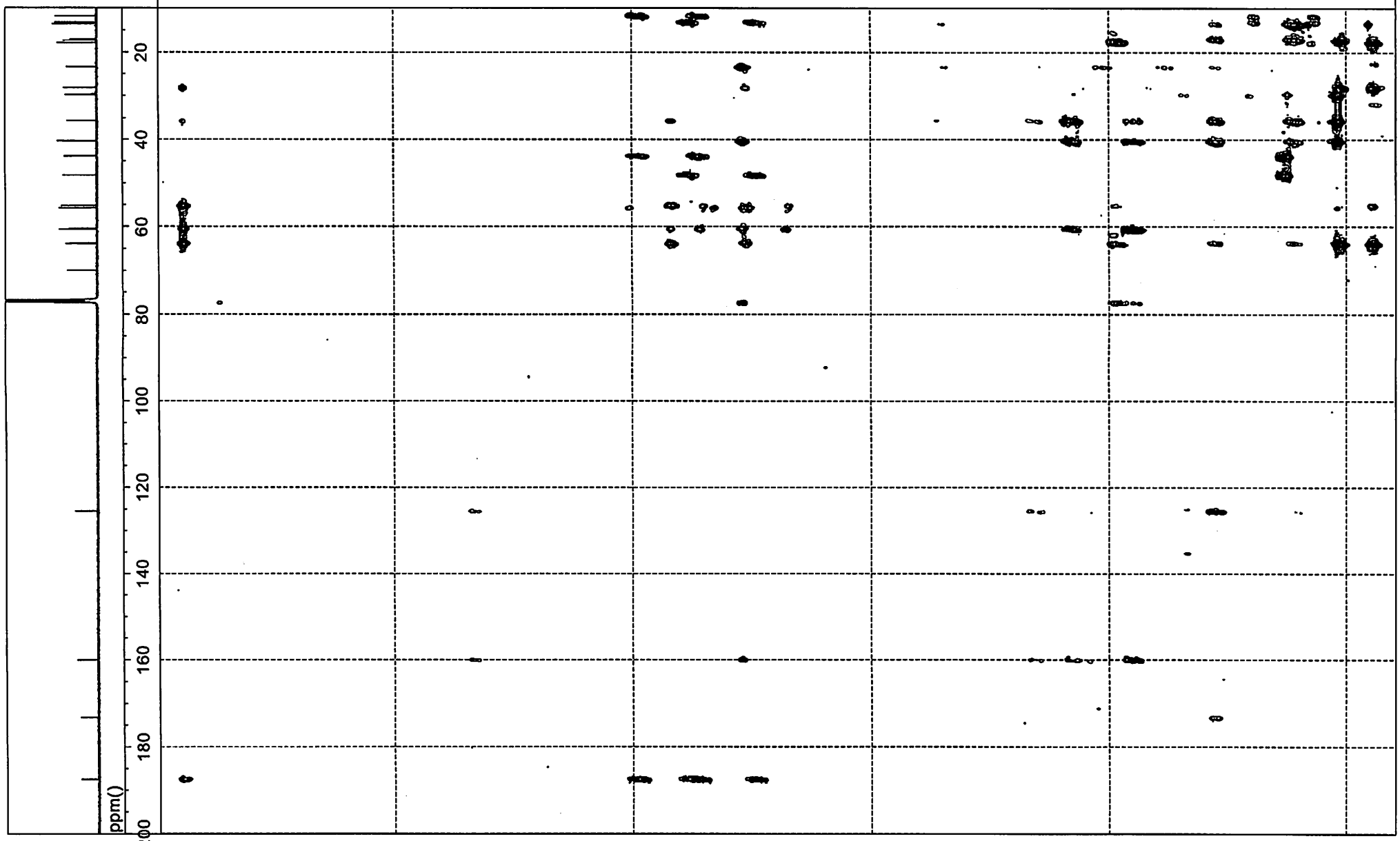


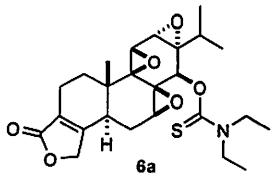




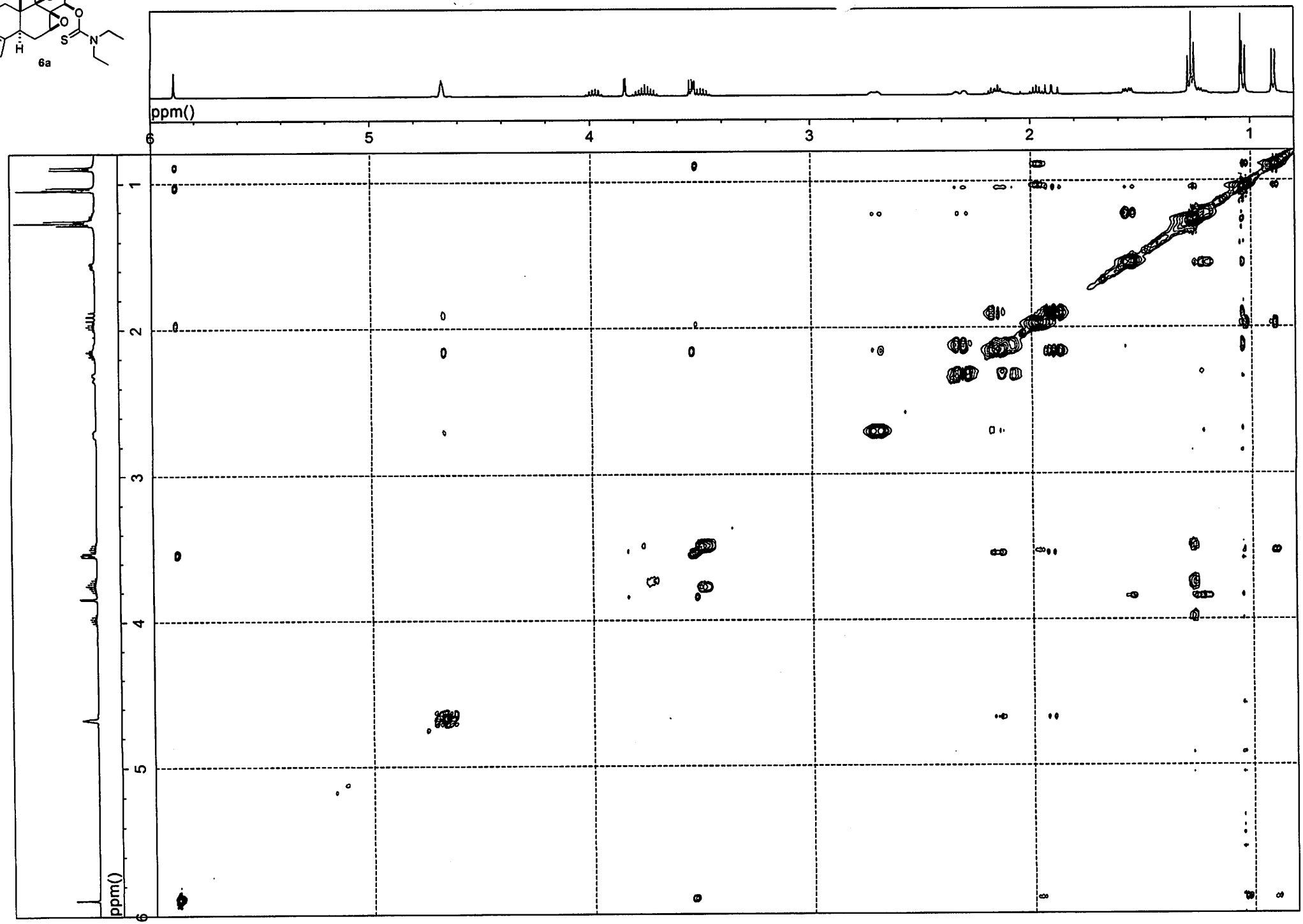
CK20-1, hmbc, CDCl3, 2014/12/27





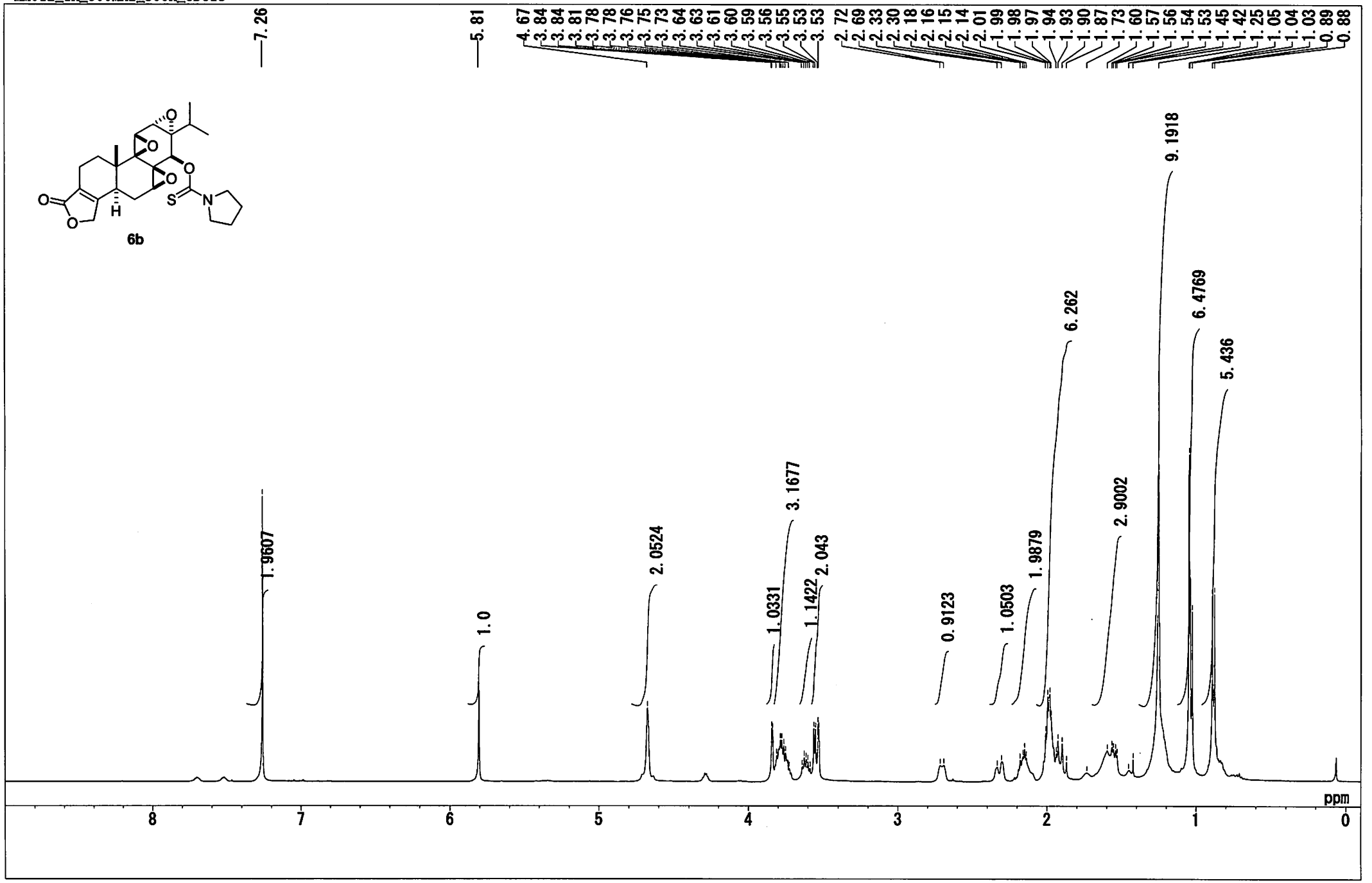


CK20-1, noesy, CDCl3, 2014/12/27

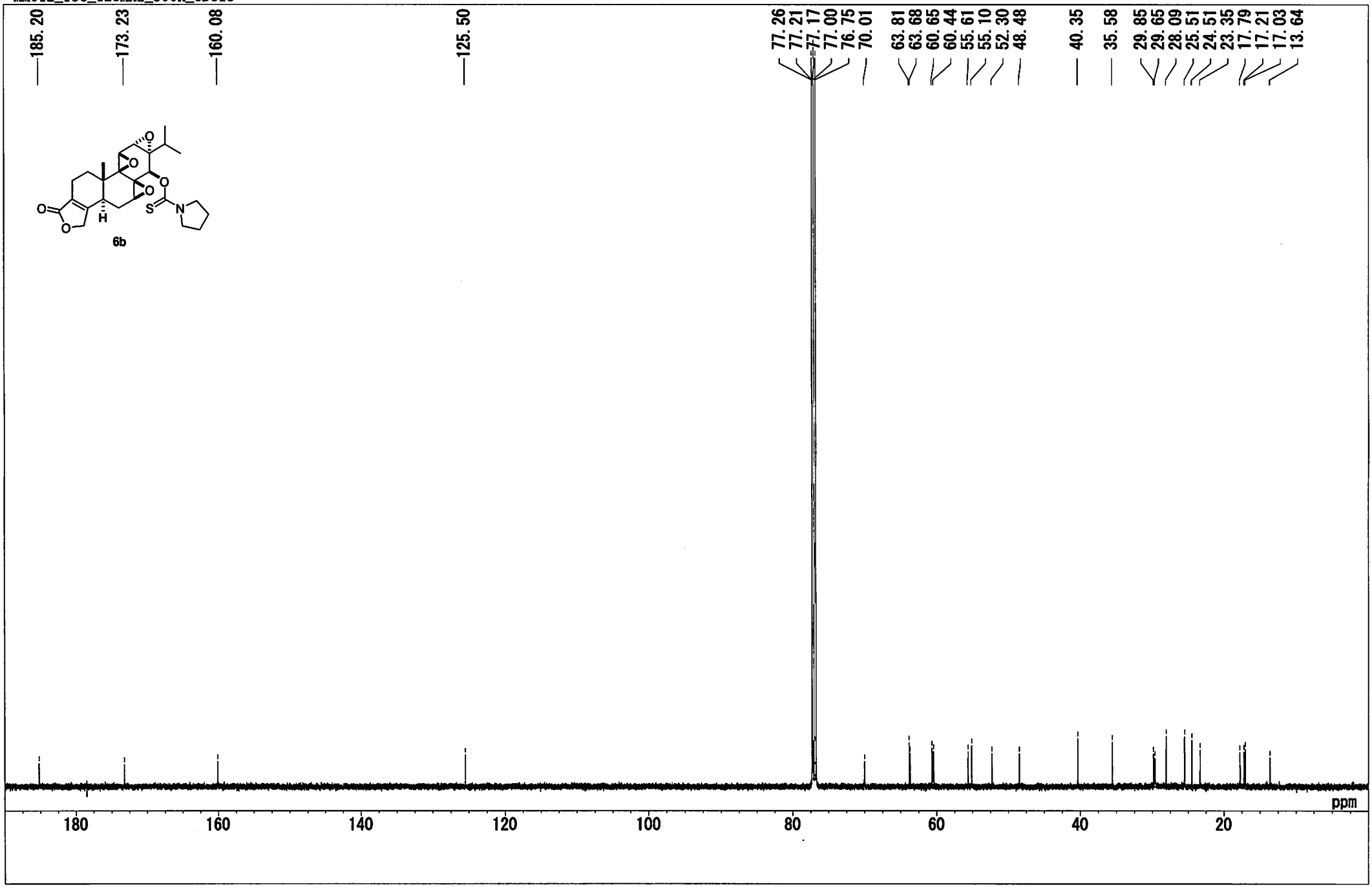


MM012_1H_500MHz_300K_CDCl3

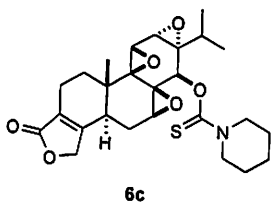
6b



MM012_13C_125MHz_300K_CDC13



MM014_1H_500MHz_300K_CDCl3



6c

MM014_13C_125MHz_300K_CDC13

186.98
178.50
173.21
160.07
125.48
77.93
77.25
77.00
76.74
76.64
70.01
63.78
63.75
60.65
60.53
55.68
55.10
51.79
47.32
40.33
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29.64
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26.00
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23.35
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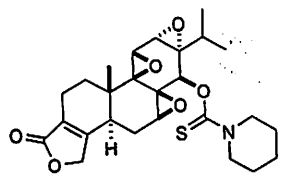
17.17
17.02
13.56



6c

200 180 160 140 120 100 80 60 40 20 0

δ/ppm (13C)