Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information-4:

Scaffold diversity-oriented synthesis of limonoid dimers: discovery of an axially chiral agent with *in vivo* anti-breast cancer activity

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[‡] These authors contributed equally

Copies of HRESIMS, UV, IR, 1D and 2D NMR spectra of compounds 5a, and 5b

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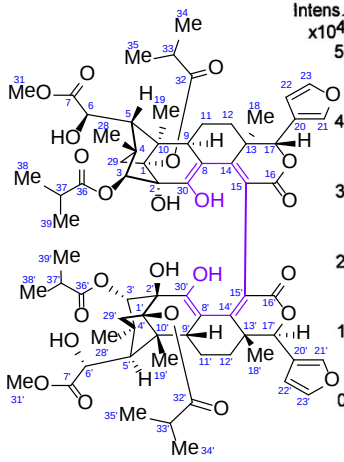
HR-ESIMS for compound **5a**



Generic Display Report

Analysis Info

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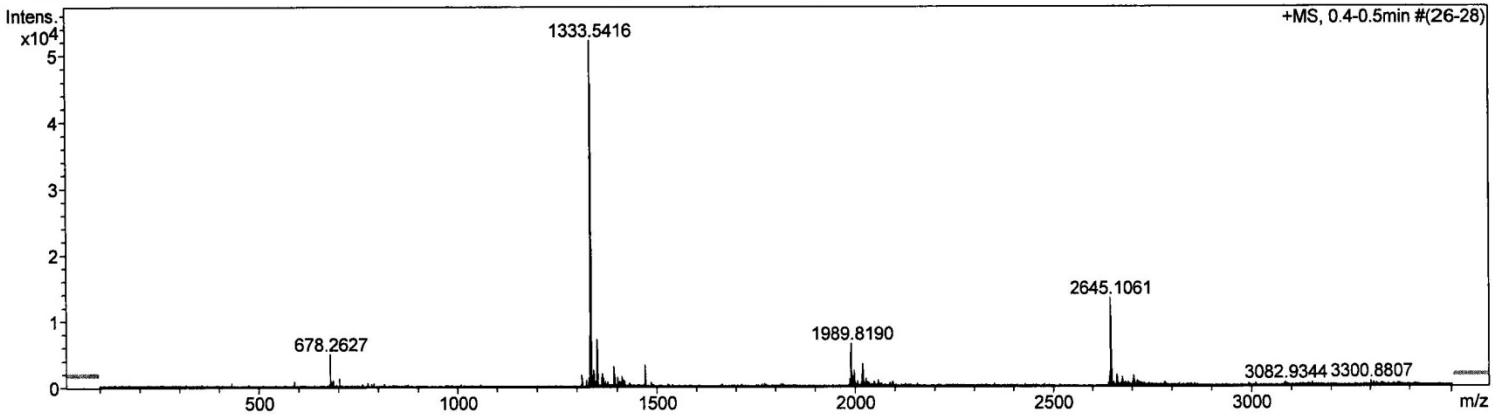
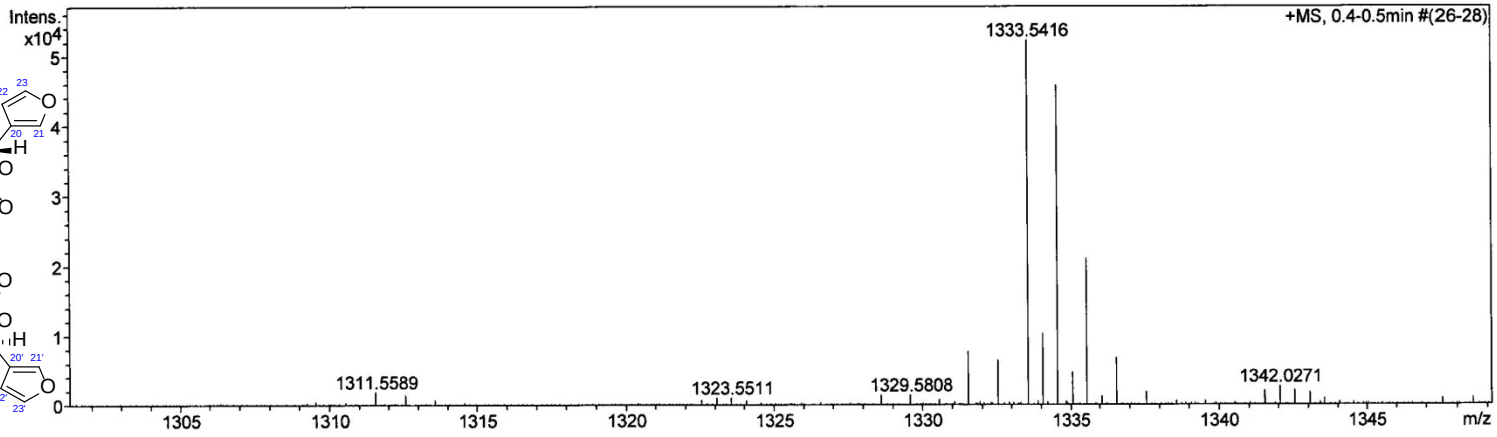
Sample Name	liwanshan_lws-42-2_pos	Instrument	maXis
Comment			





Bruker Compass DataAnalysis 4.0 printed: 9/8/2015 1:22:25 AM Page 1 of 1

HR-ESIMS for compound **5a**

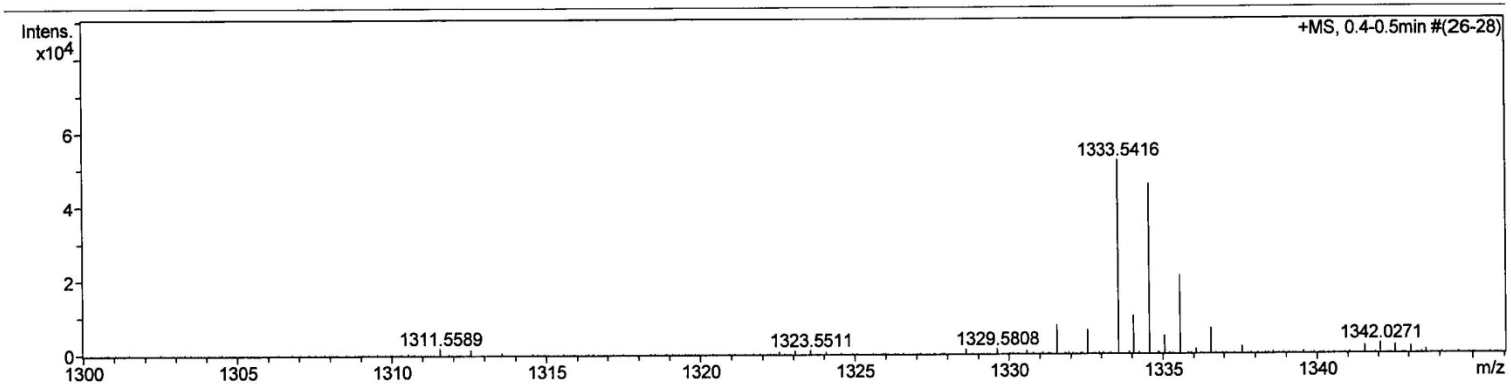
Mass Spectrum SmartFormula Report

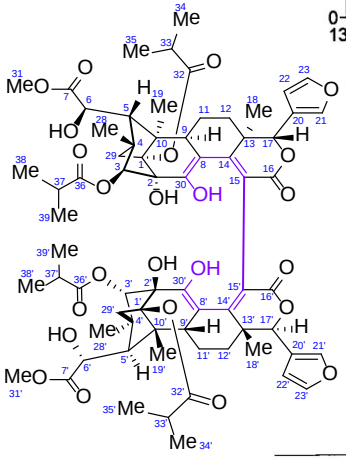
Analysis Info

Analysis Name	D:\Data\MS\data\201509\liwanshan_lws-42-2_pos_3_01_353.d	Acquisition Date	9/8/2015 1:12:33 AM
Method	LC_Direct Infusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	liwanshan_lws-42-2_pos	Instrument / Ser#	maXis 29
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3500 m/z	Set Collision Cell RF	1800.0 Vpp	Set Divert Valve	Waste





Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
1311.5589	1	C 70 H 87 O 24	100.00	1311.5582	-0.5	-0.7	22.0	27.5	even	ok
1333.5416	1	C 70 H 86 Na O 24	45.83	1333.5401	-1.1	-1.5	51.9	27.5	even	ok

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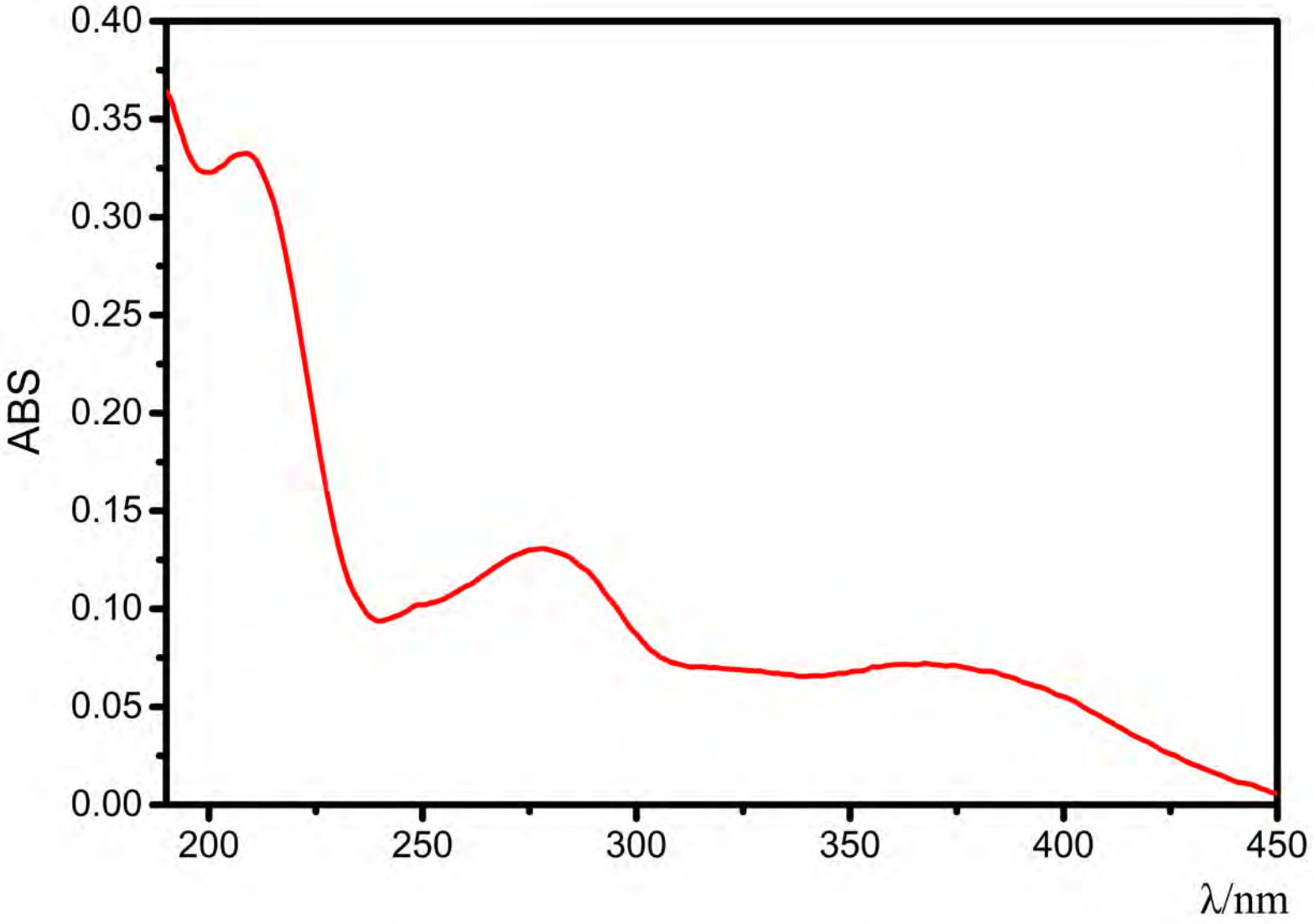
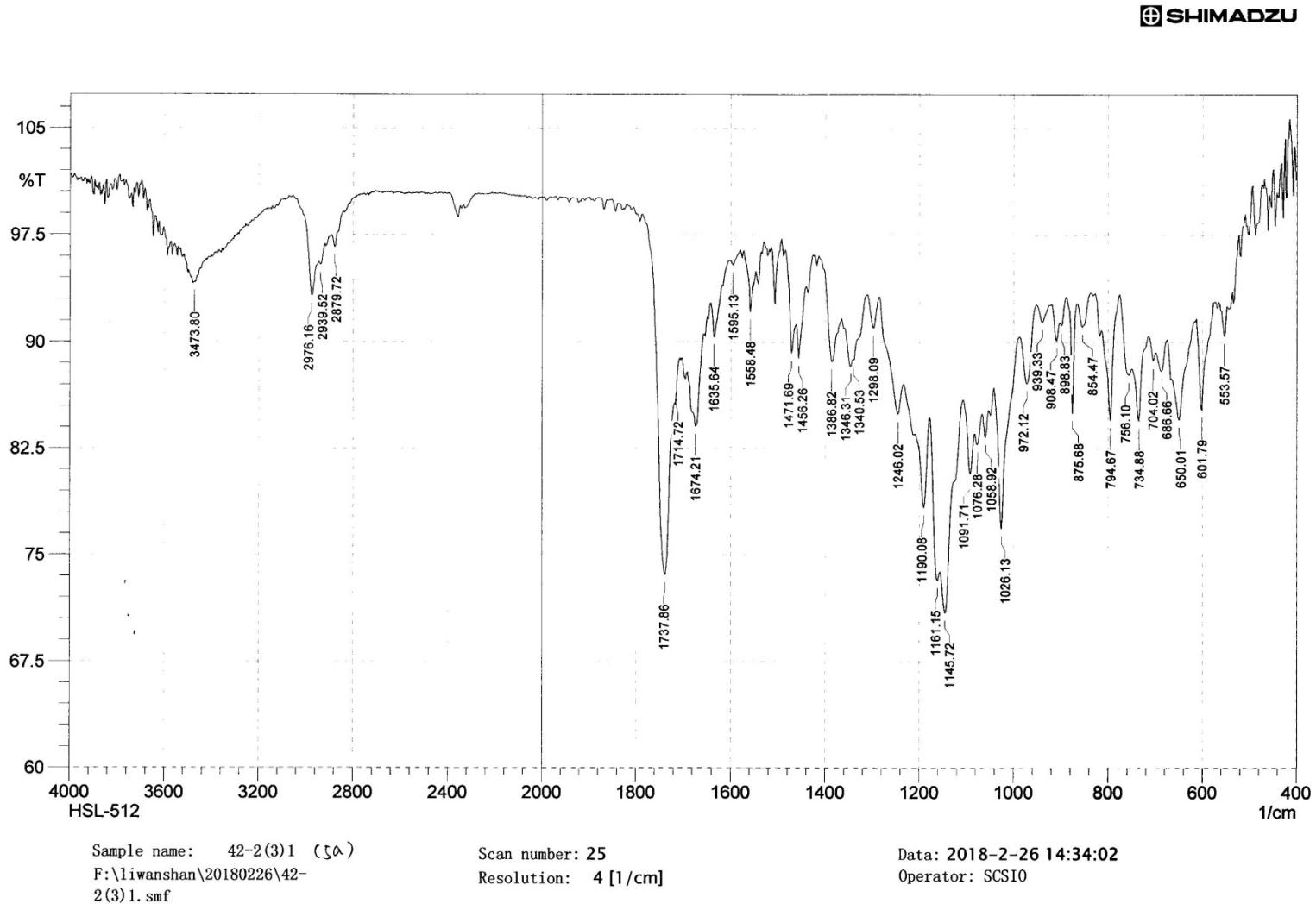
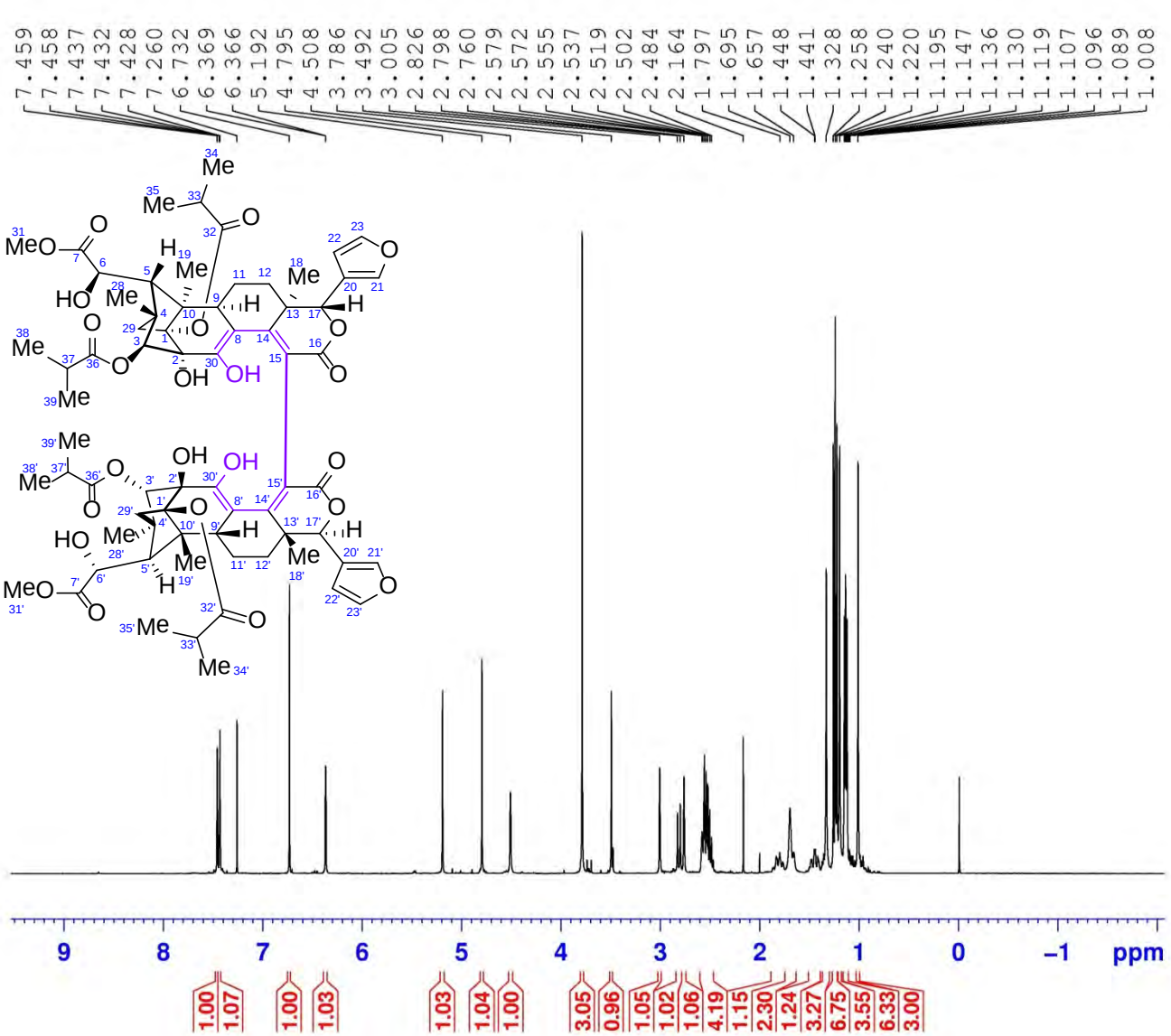
UV spectrum for compound **5a** in CH_3CN



IR spectrum for compound **5a**



^{1}H NMR (400 MHz) spectrum of compound **5a** in $CDCl_3$



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EXPNO	101
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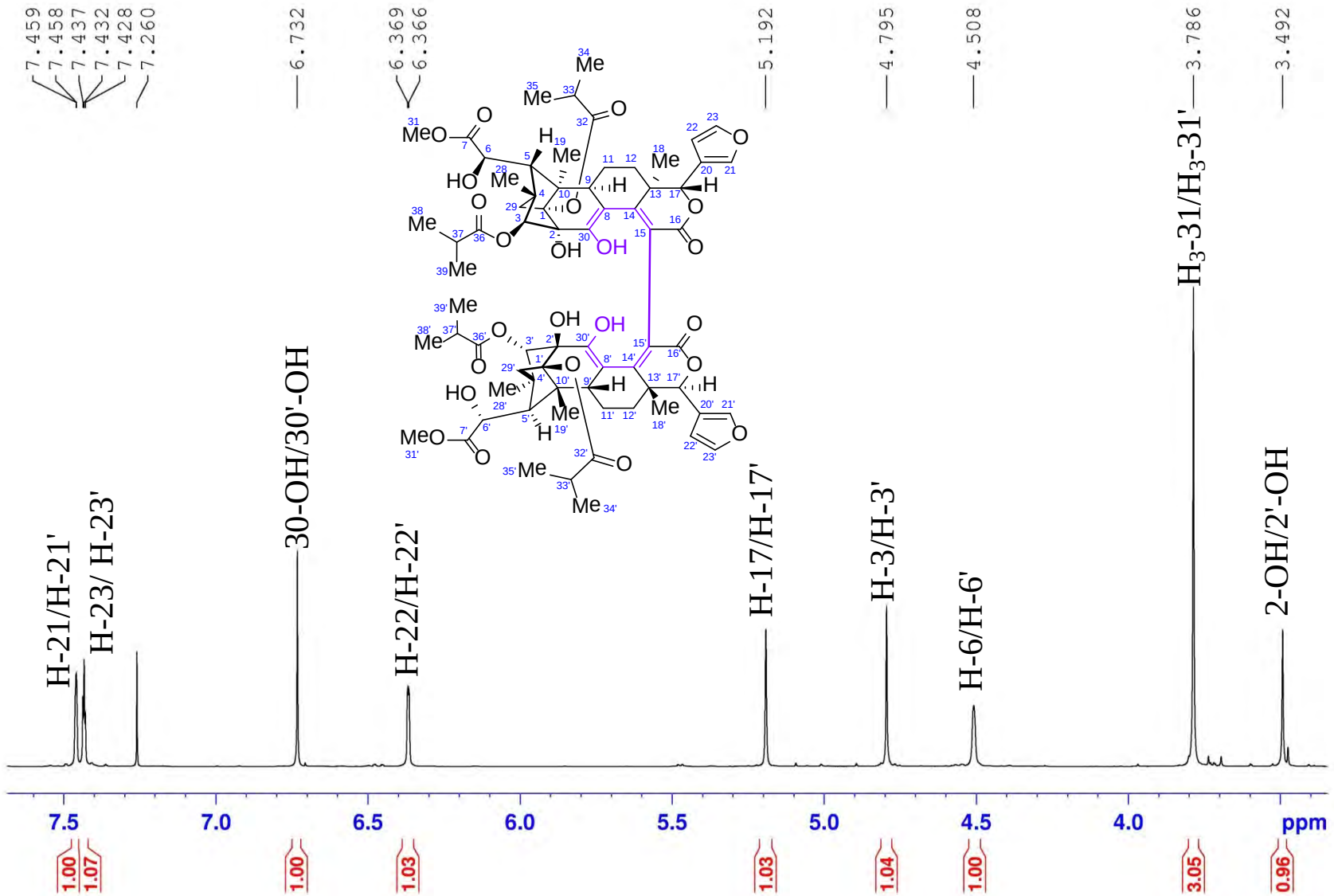
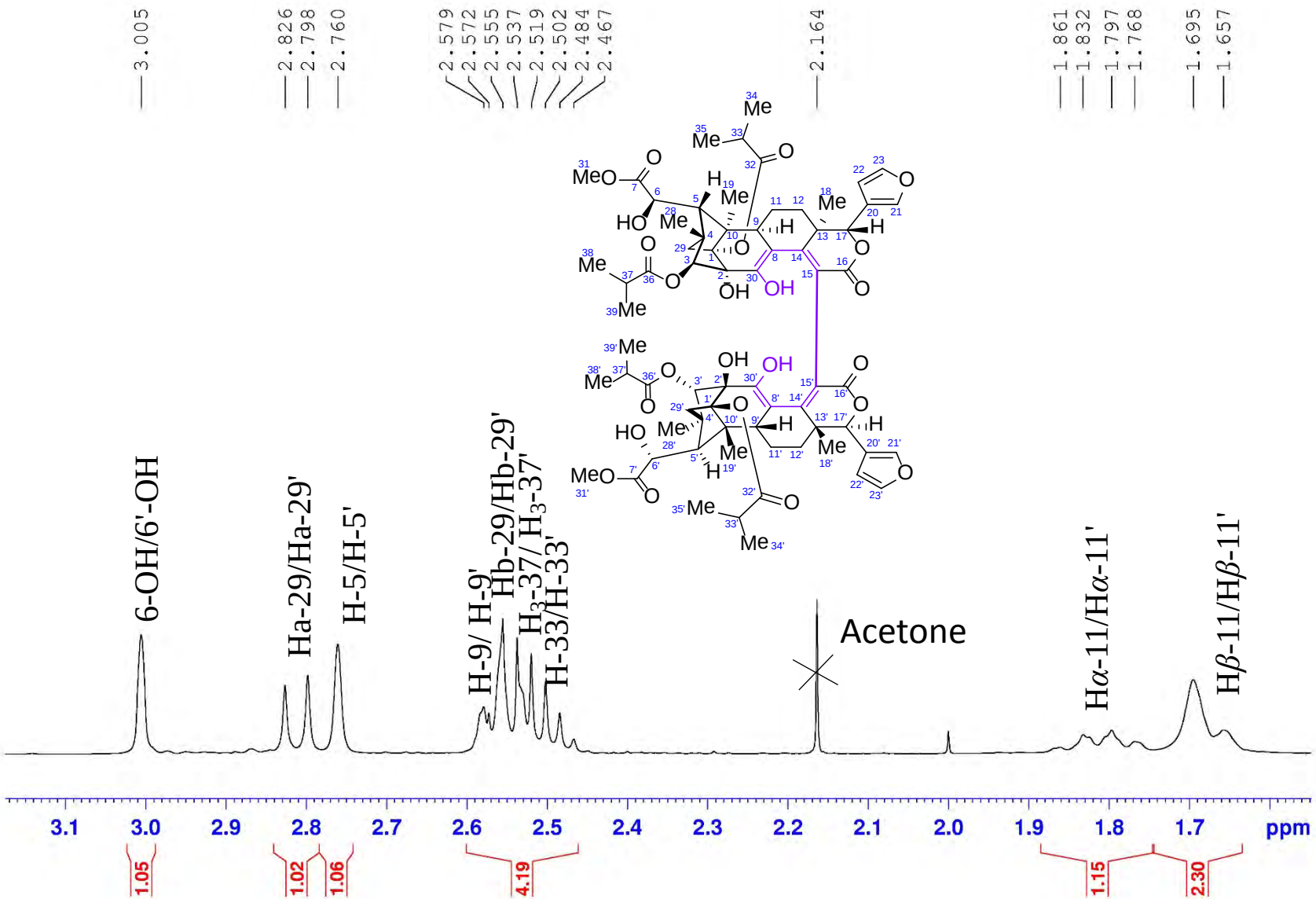
PROBHD	5 mm CPPBBO BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	51.23
DW	60.800 usec
DE	10.00 usec
TE	297.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
P1	12.00 usec
SI	65536
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

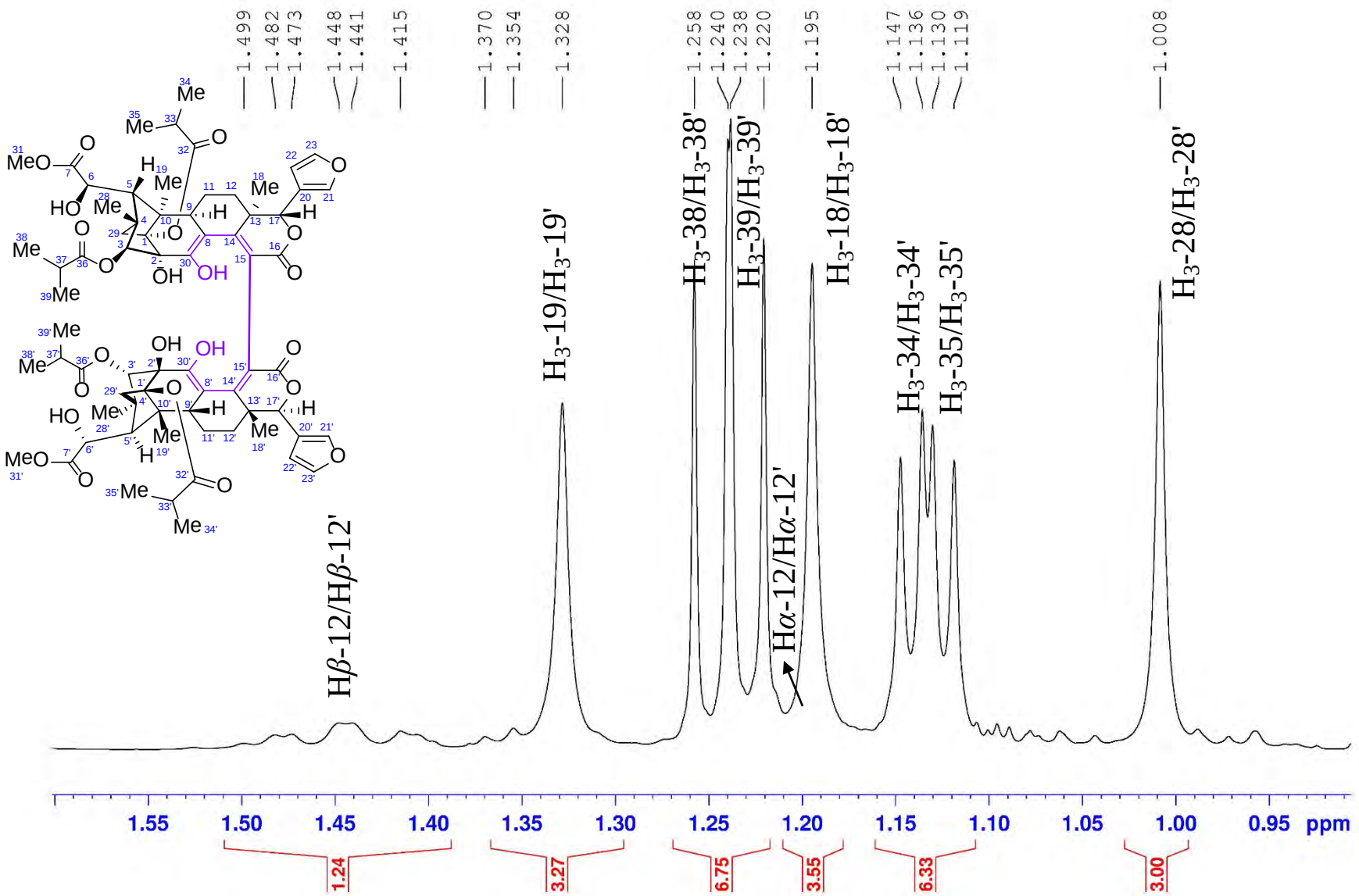
^{1}H NMR (400 MHz) spectrum of compound **5a** in $CDCl_3$



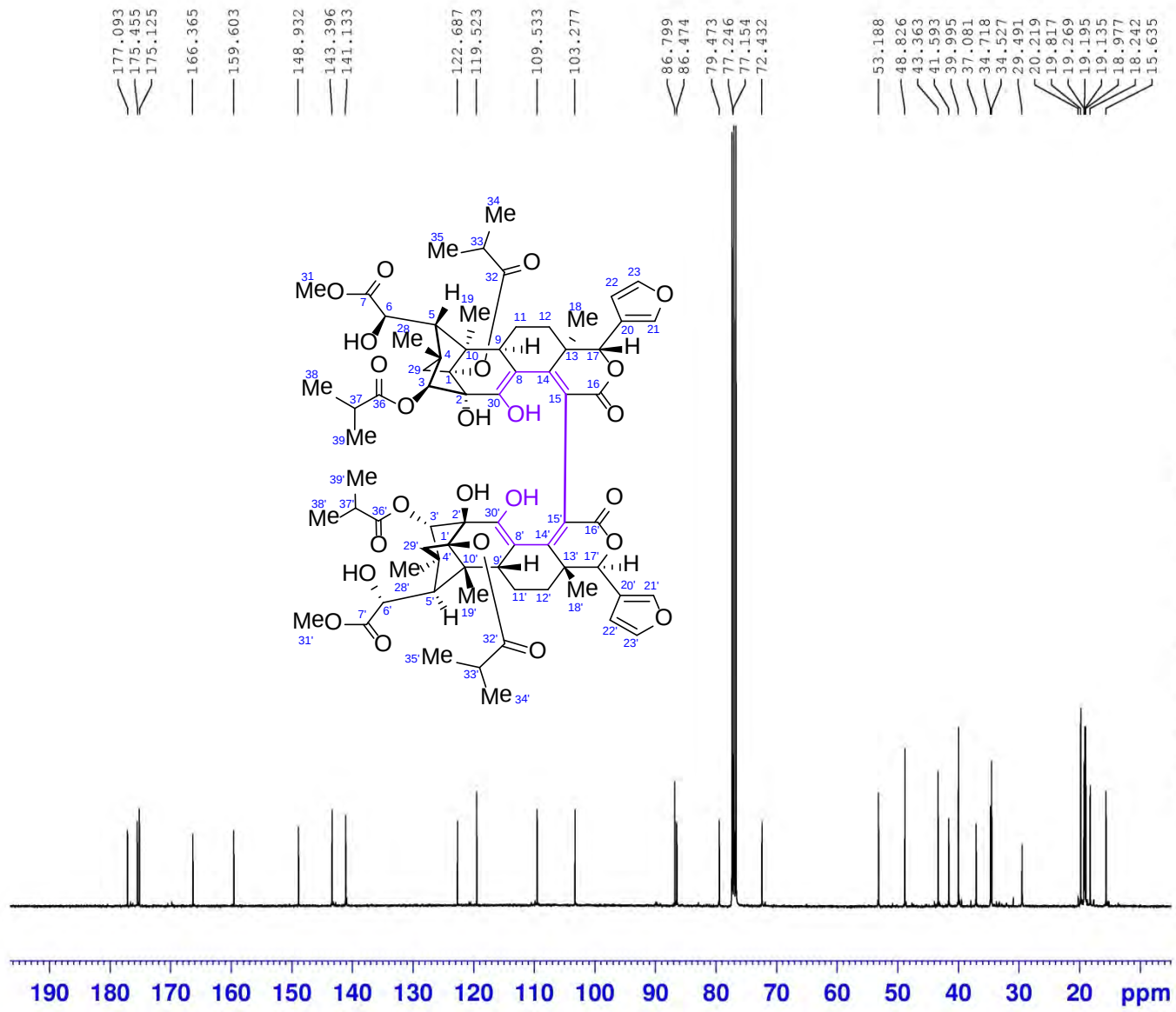
^{1}H NMR (400 MHz) spectrum of compound **5a** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **5a** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **5a** in $CDCl_3$



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PROCNO	1
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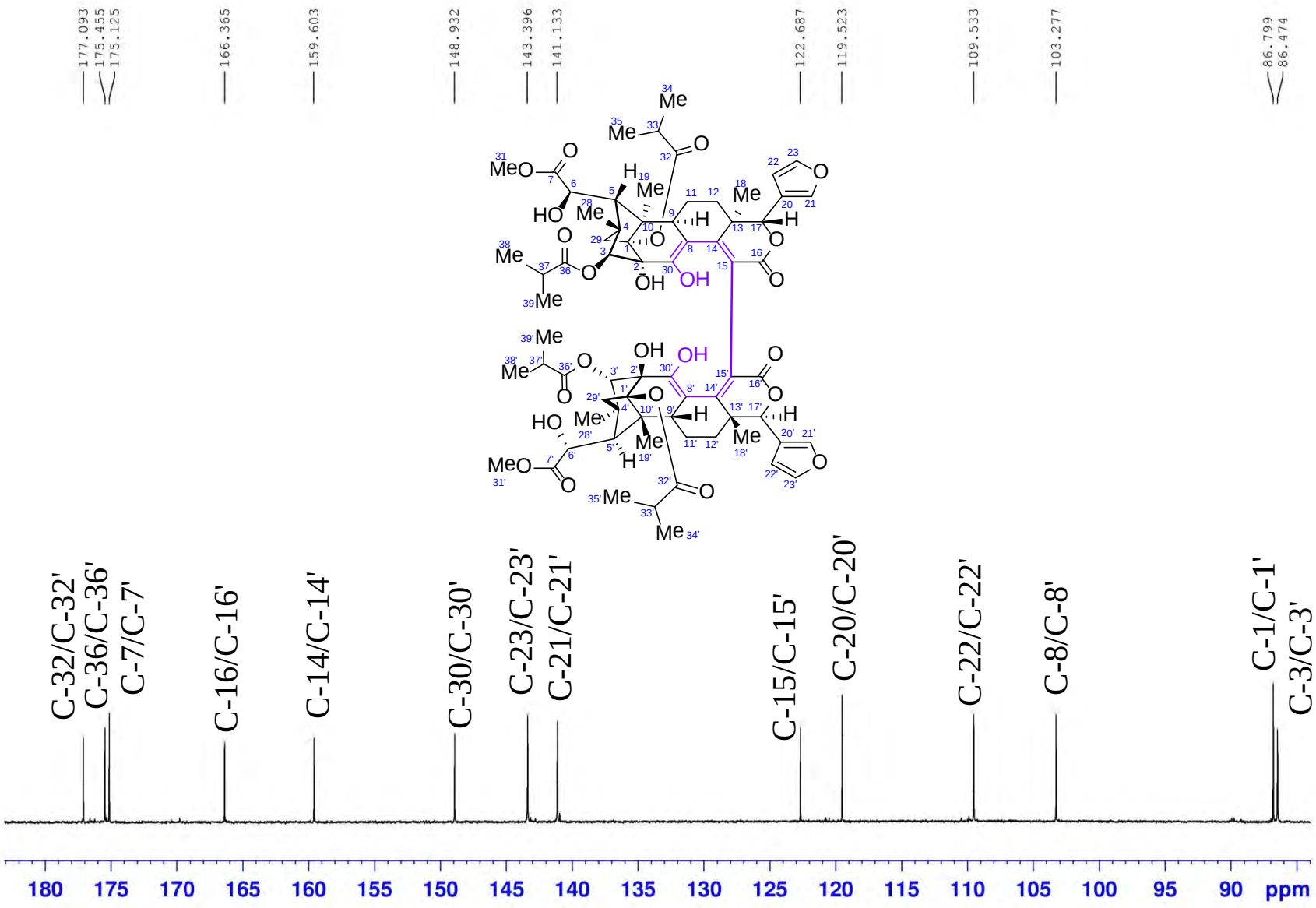
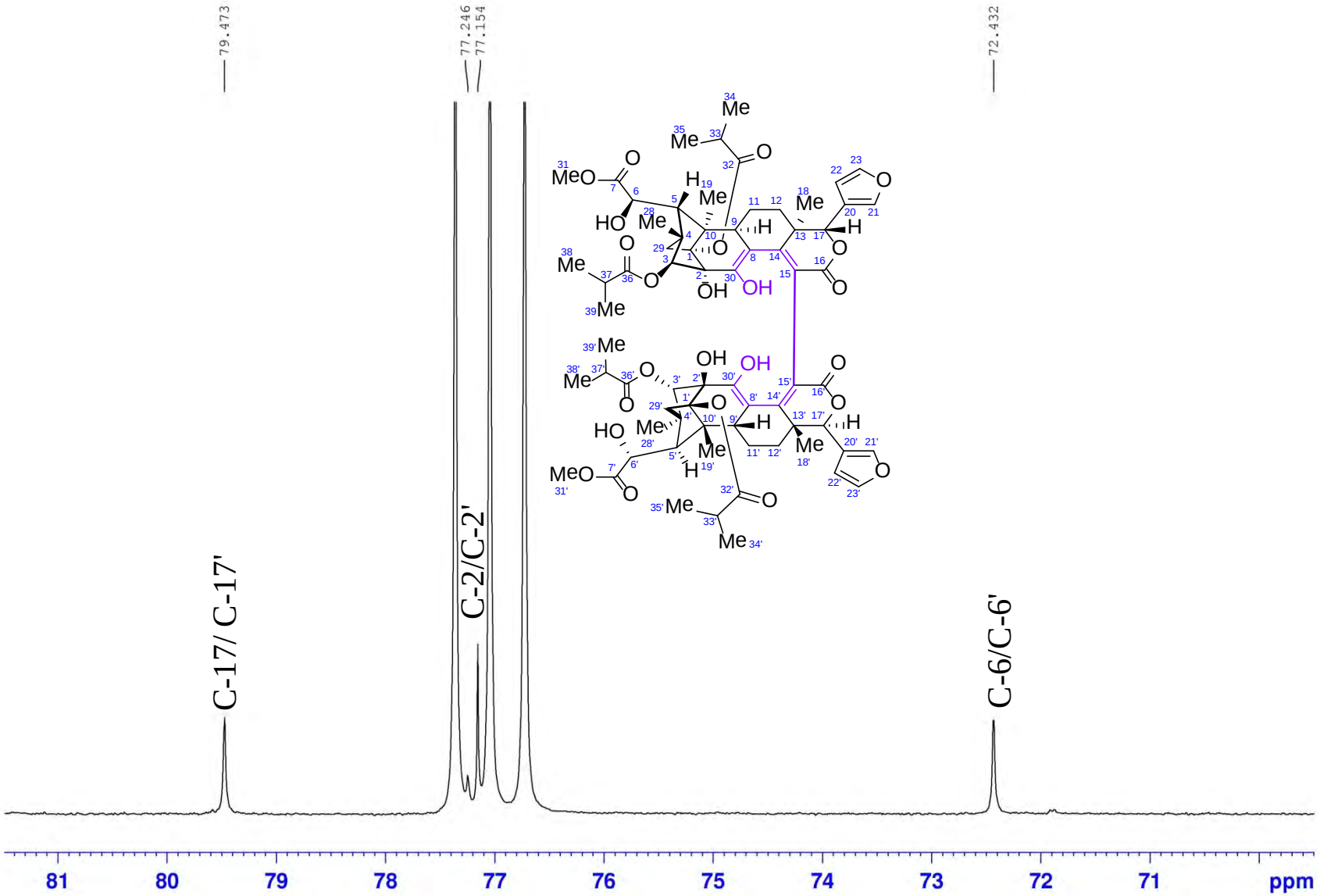
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	73.92
DW	20.800 usec
DE	18.00 usec
TE	297.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6233324 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

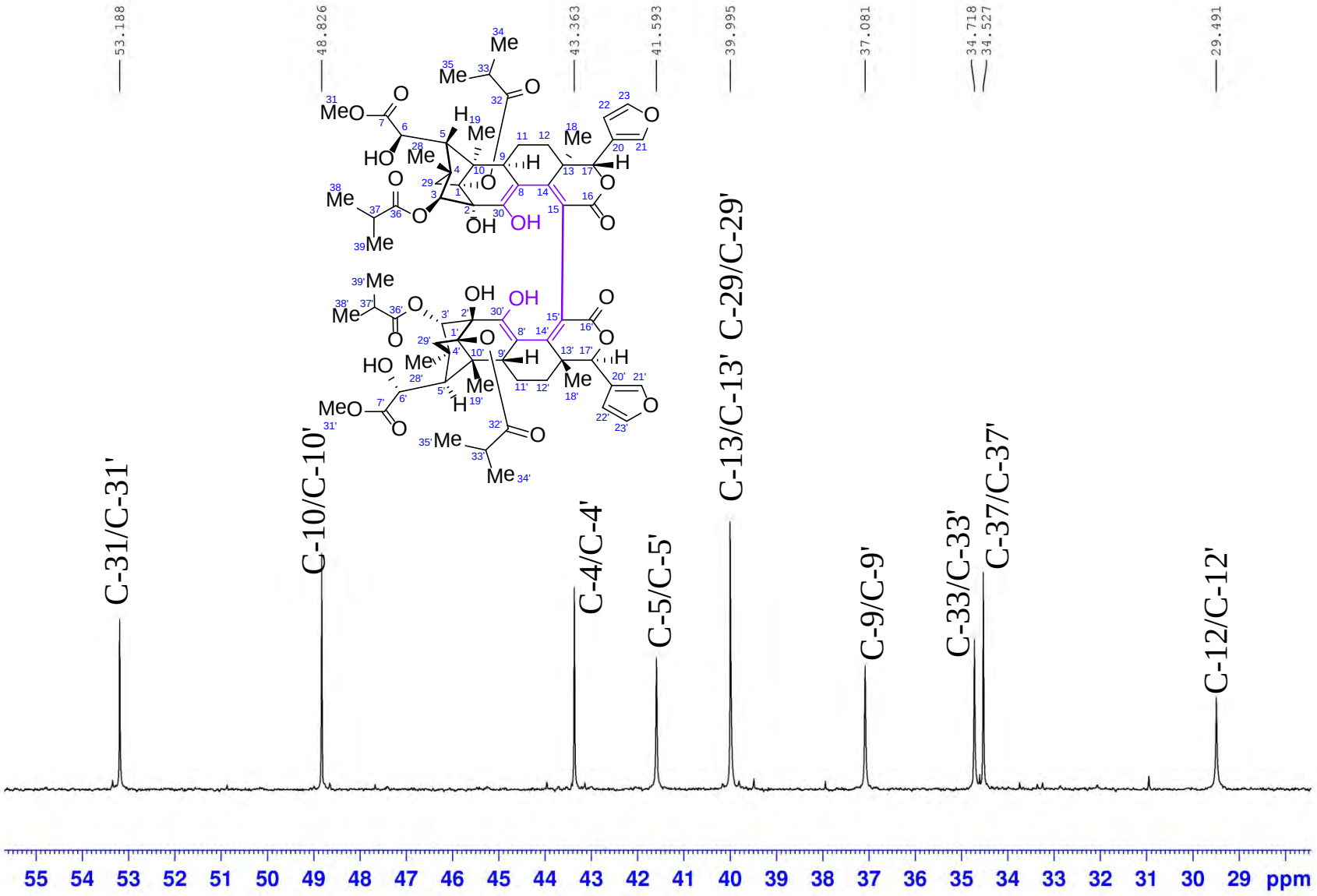
^{13}C NMR (100 MHz) spectrum of compound **5a** in $CDCl_3$



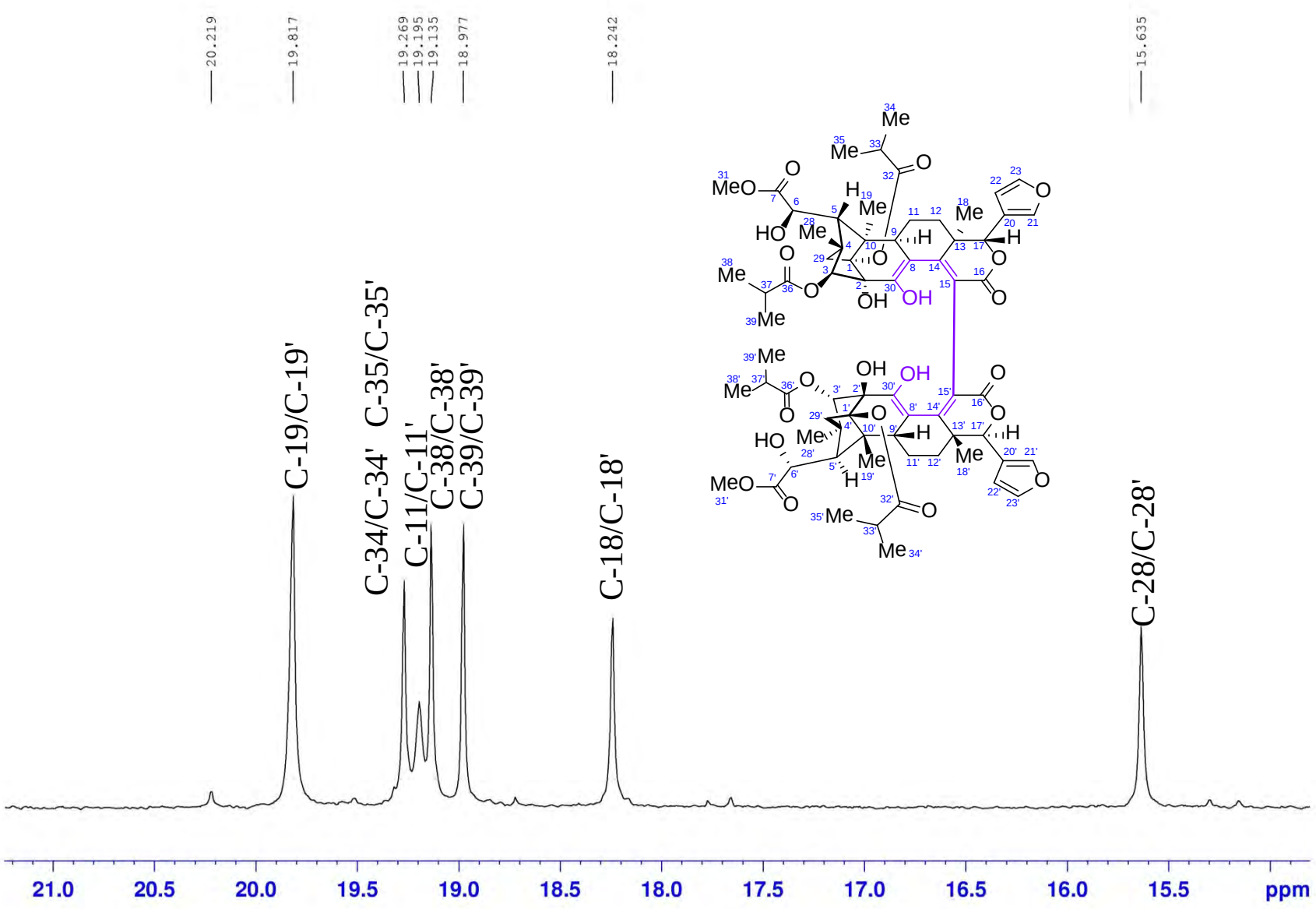
^{13}C NMR (100 MHz) spectrum of compound **5a** in $CDCl_3$



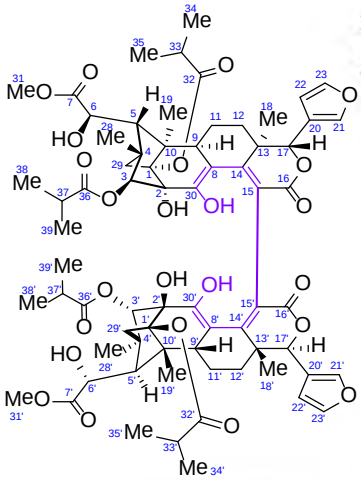
^{13}C NMR (100 MHz) spectrum of compound **5a** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **5a** in $CDCl_3$



DEPT135 (100 MHz) spectrum of compound **5a** in $CDCl_3$

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PROCNO                  1
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PULPROG       deptsp135
TD                  65536
SOLVENT             CDCl3
NS                    600
DS                      4
SWH             24038.461
FIDRES          0.366798
AQ              1.3631988
RG                 130.26
DW                 20.800
DE                  18.00
TE                  297.0
CNST2          145.0000000
D1              2.00000000
D2              0.00344828
D12             0.00002000
TD0                     1

======== CHANNEL f1 ====
SFO1           100.6233324
NUC1                   13C
P1                   10.00
P13                2000.00
SI                   32768
SF             100.6127685
WDW                    EM
SSB                     0
LB                   1.00
GB                      0
PC                   1.40
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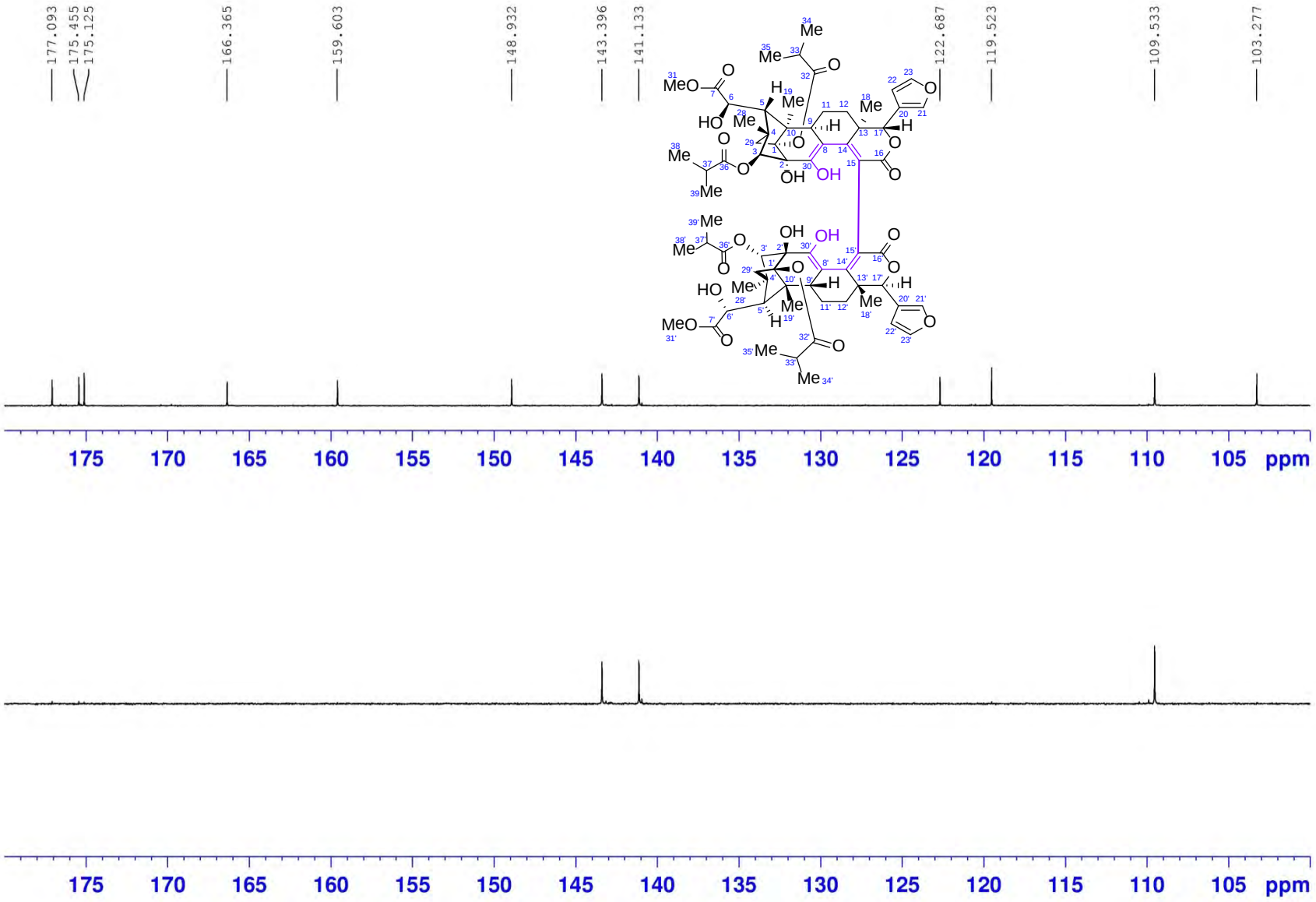
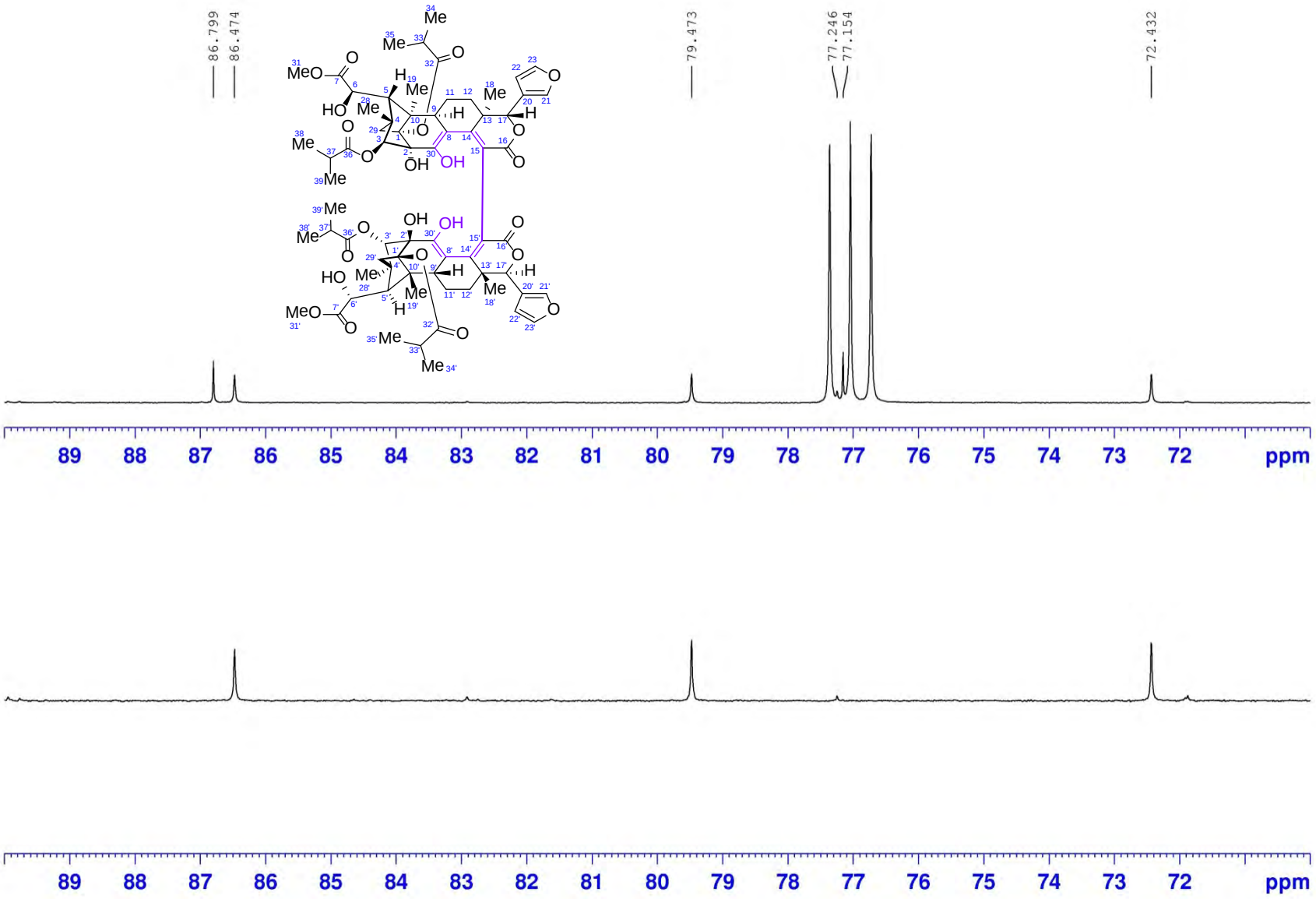
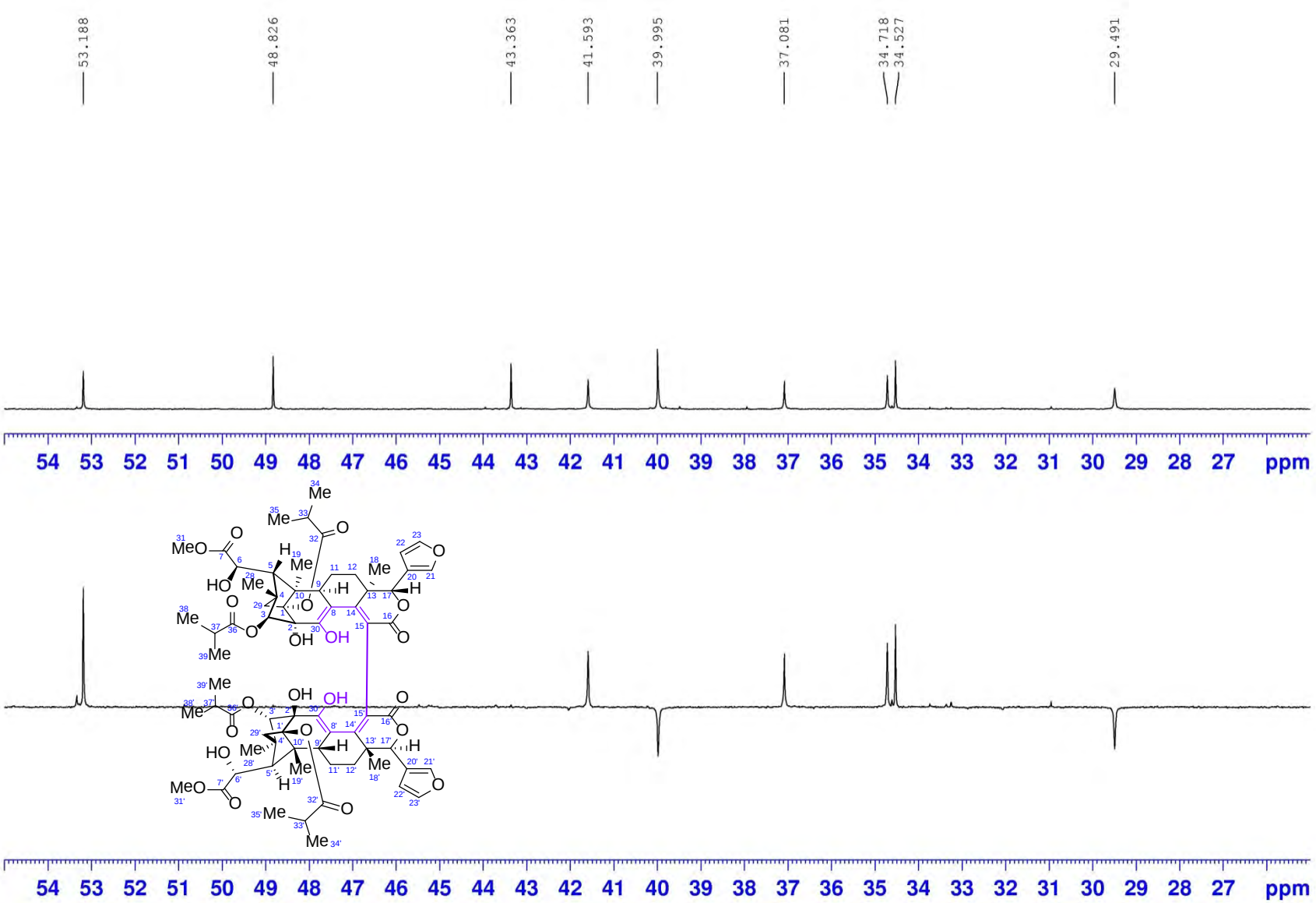
DEPT135 (100 MHz) spectrum of compound **5a** in $CDCl_3$



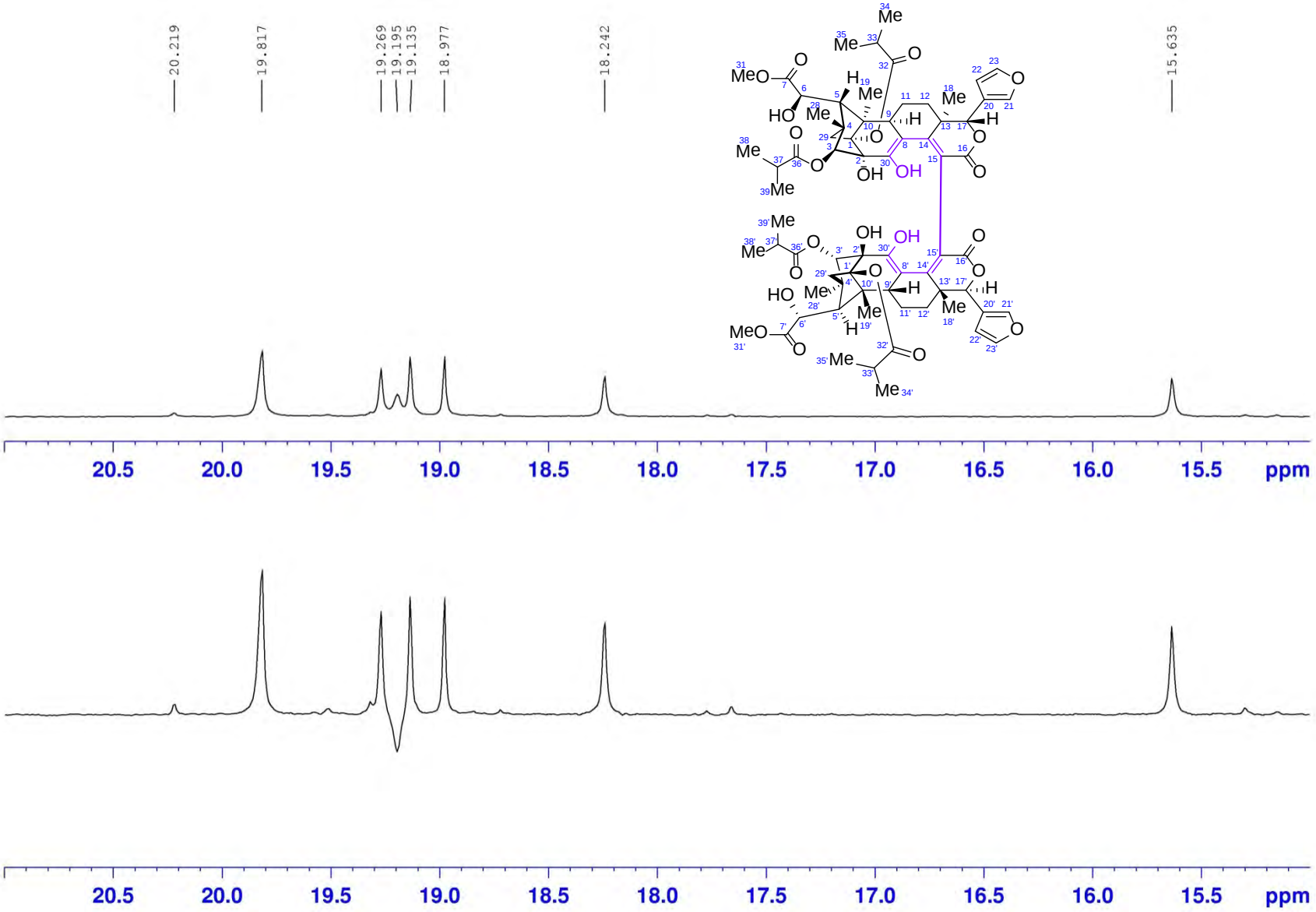
DEPT135 (100 MHz) spectrum of compound **5a** in $CDCl_3$



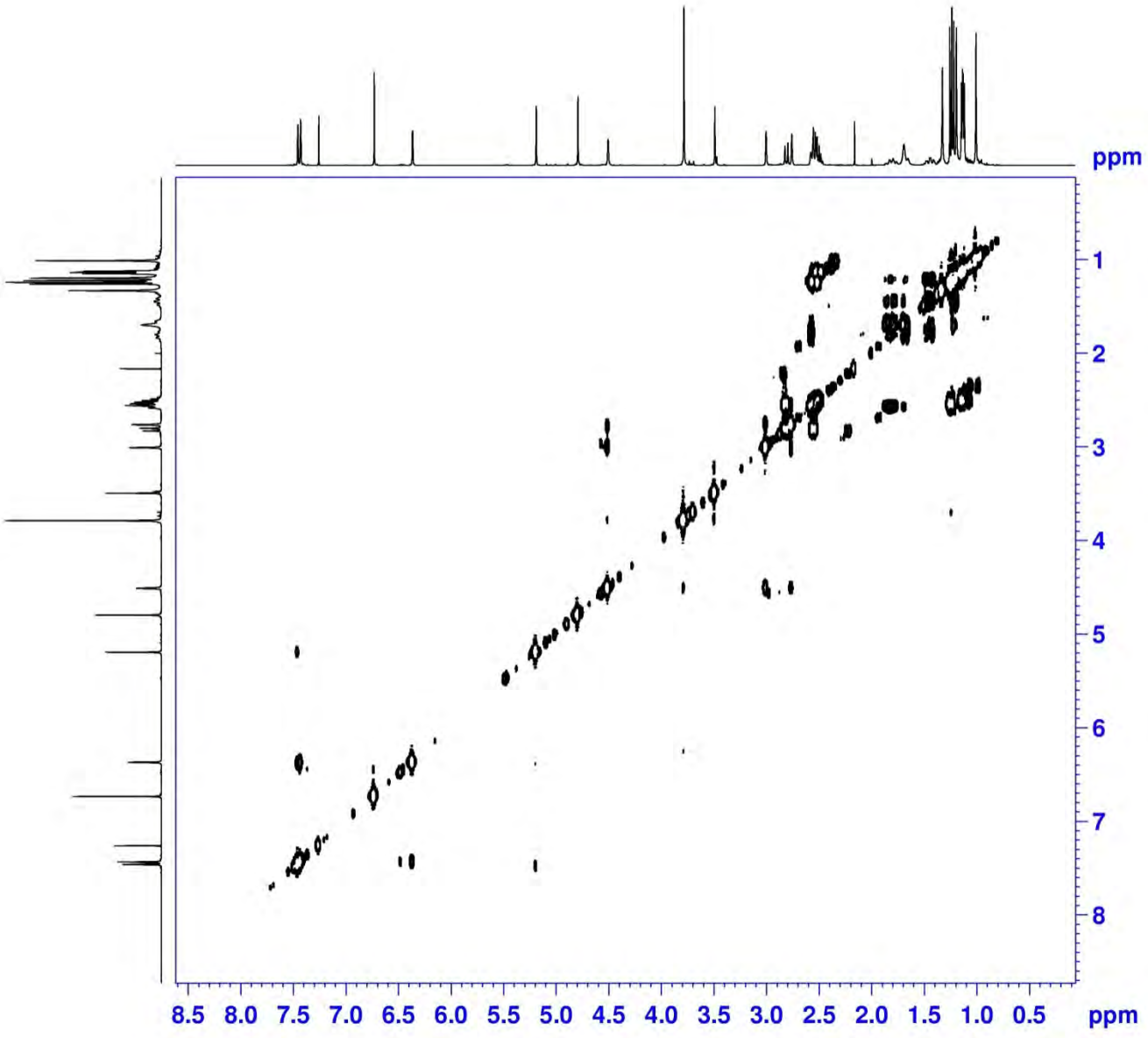
DEPT135 (100 MHz) spectrum of compound **5a** in $CDCl_3$



DEPT135 (100 MHz) spectrum of compound **5a** in $CDCl_3$



^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5a** in $CDCl_3$



BRUKER

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PROCNO	1
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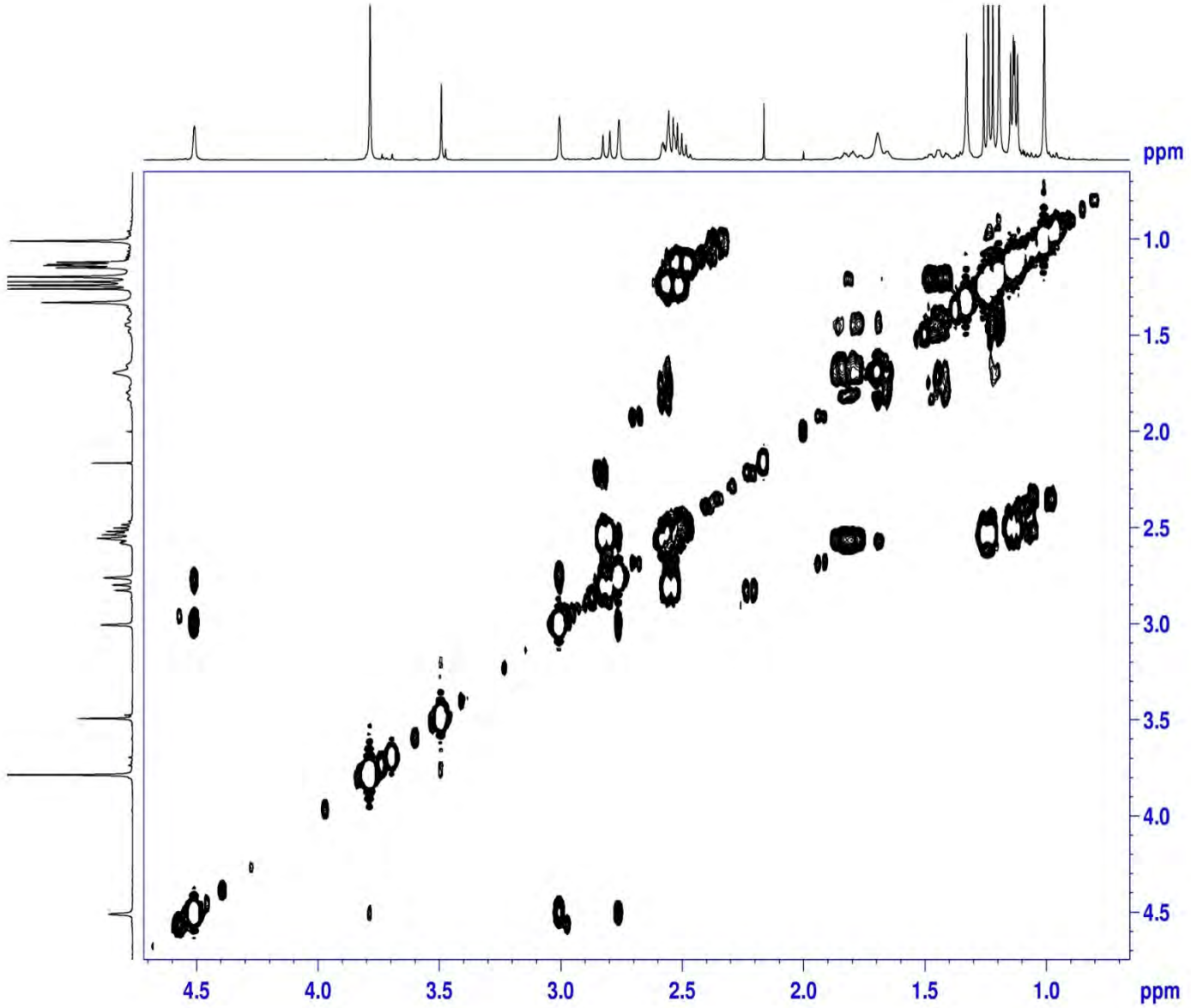
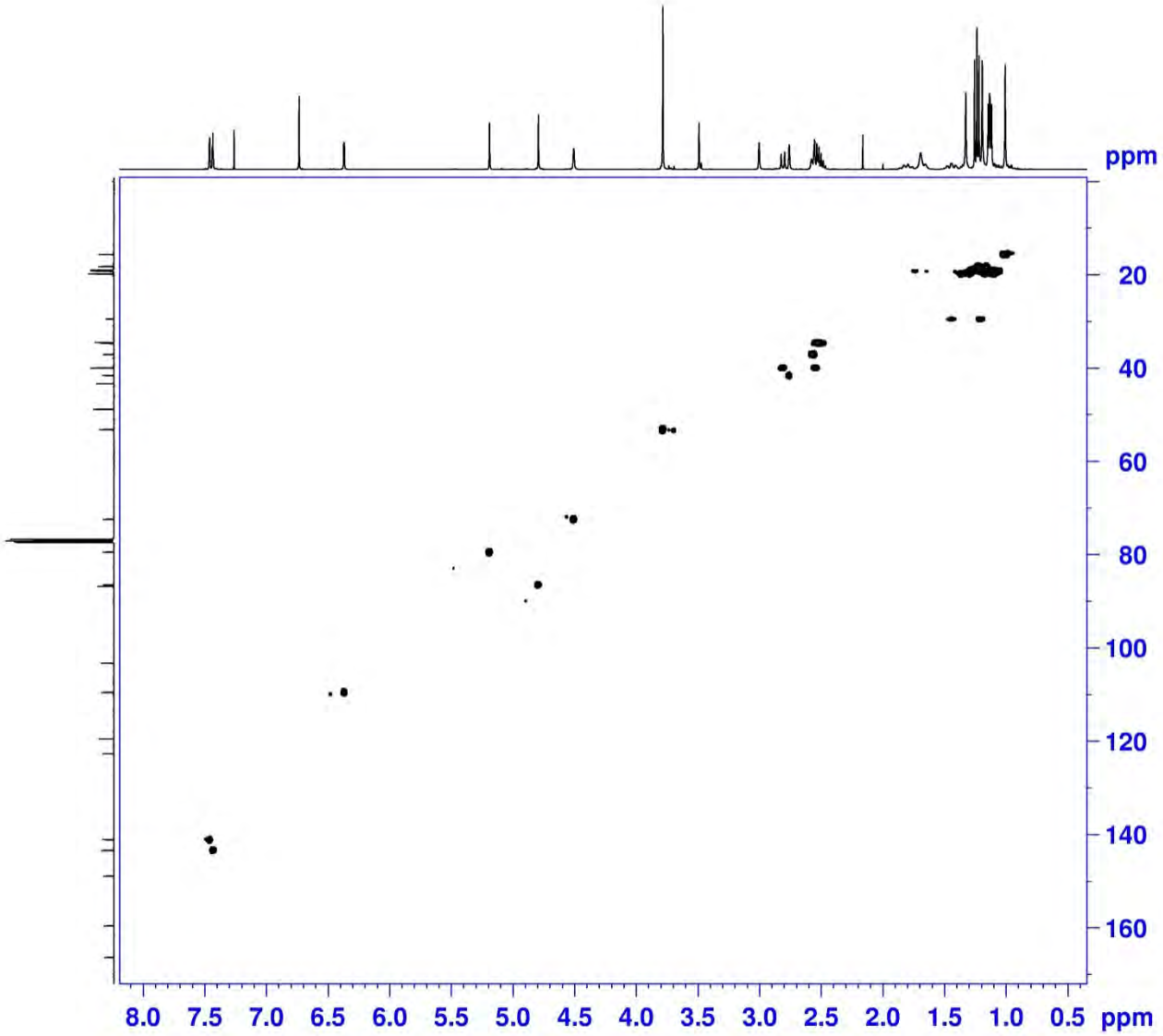
PROBHD	5 mm CPPBBO BB
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDC13
NS	8
DS	8
SWH	3906.250 Hz
FIDRES	1.907349 Hz
AQ	0.2621940 sec
RG	56.55
DW	128.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00000300 sec
D1	1.89678097 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00025600 sec
========	CHANNEL f1 ========
SFO1	400.1318006 MHz
NUC1	1H
P0	12.00 usec
P1	12.00 usec
P17	2500.00 usec
ND0	1
TD	128
SFO1	400.1318 MHz
FIDRES	30.517578 Hz
SW	9.762 ppm
FnMODE	QF
SI	1024
SF	400.1300070 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	400.1300100 MHz
WDW	QSINE
SSB	0

^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5a** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **5a** in $CDCl_3$



BRUKER

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TD	1024
SOLVENT	CDCl3
NS	16
DS	16
SWH	4302.926 Hz
FIDRES	4.202076 Hz
AQ	0.1190388 sec
RG	208.5
DW	116.200 usec
DE	10.00 usec
TE	297.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.46497905 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
D24	0.00086207 sec
IN0	0.00002080 sec
ZGOPTNS	
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SFO1	400.1320007 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
P28	0.00 usec
ND0	2
TD	256
SFO1	100.6233 MHz
FIDRES	93.900238 Hz
SW	238.896 ppm
FnMODE	Echo-Antiecho
SI	1024
SF	400.1300079 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
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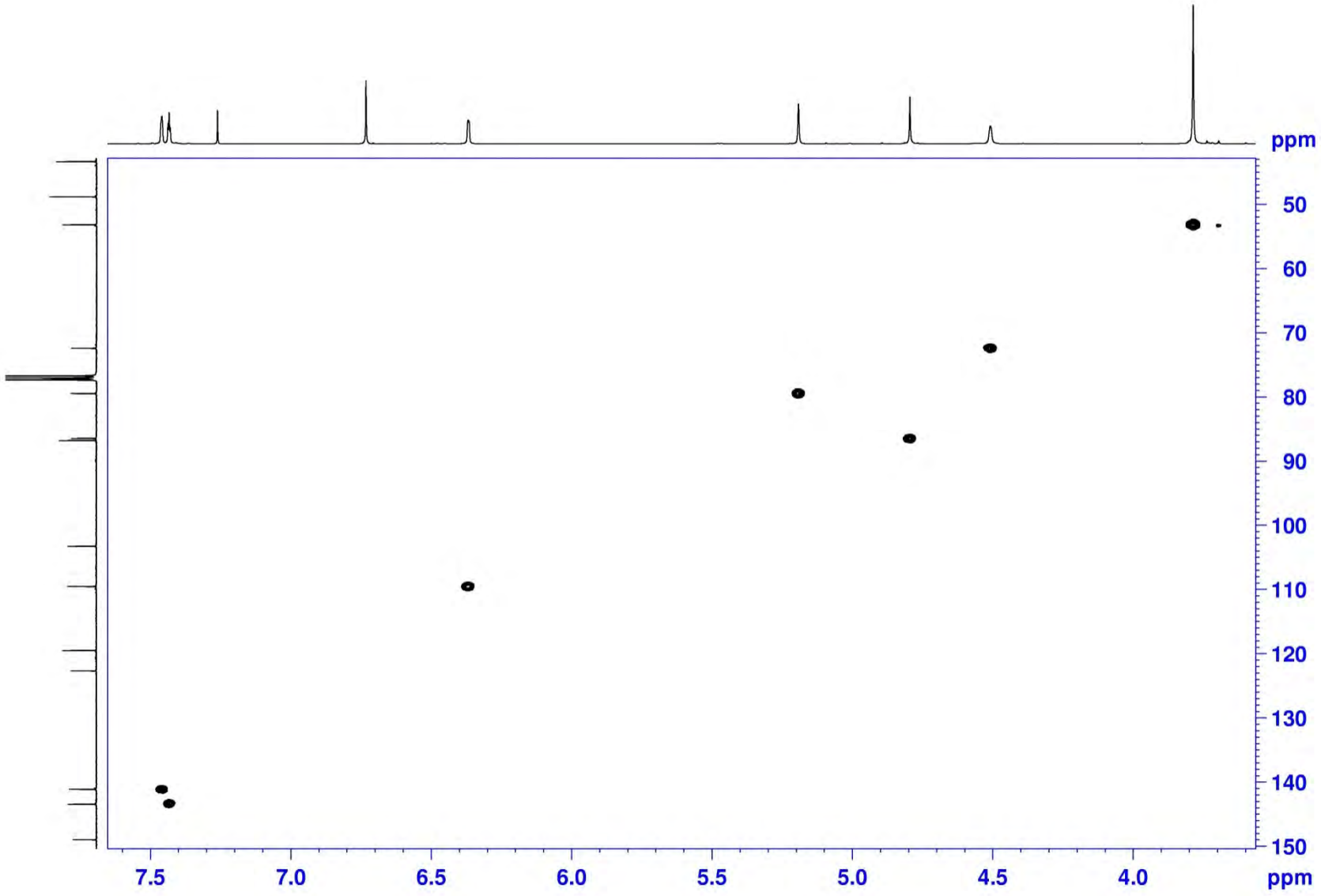
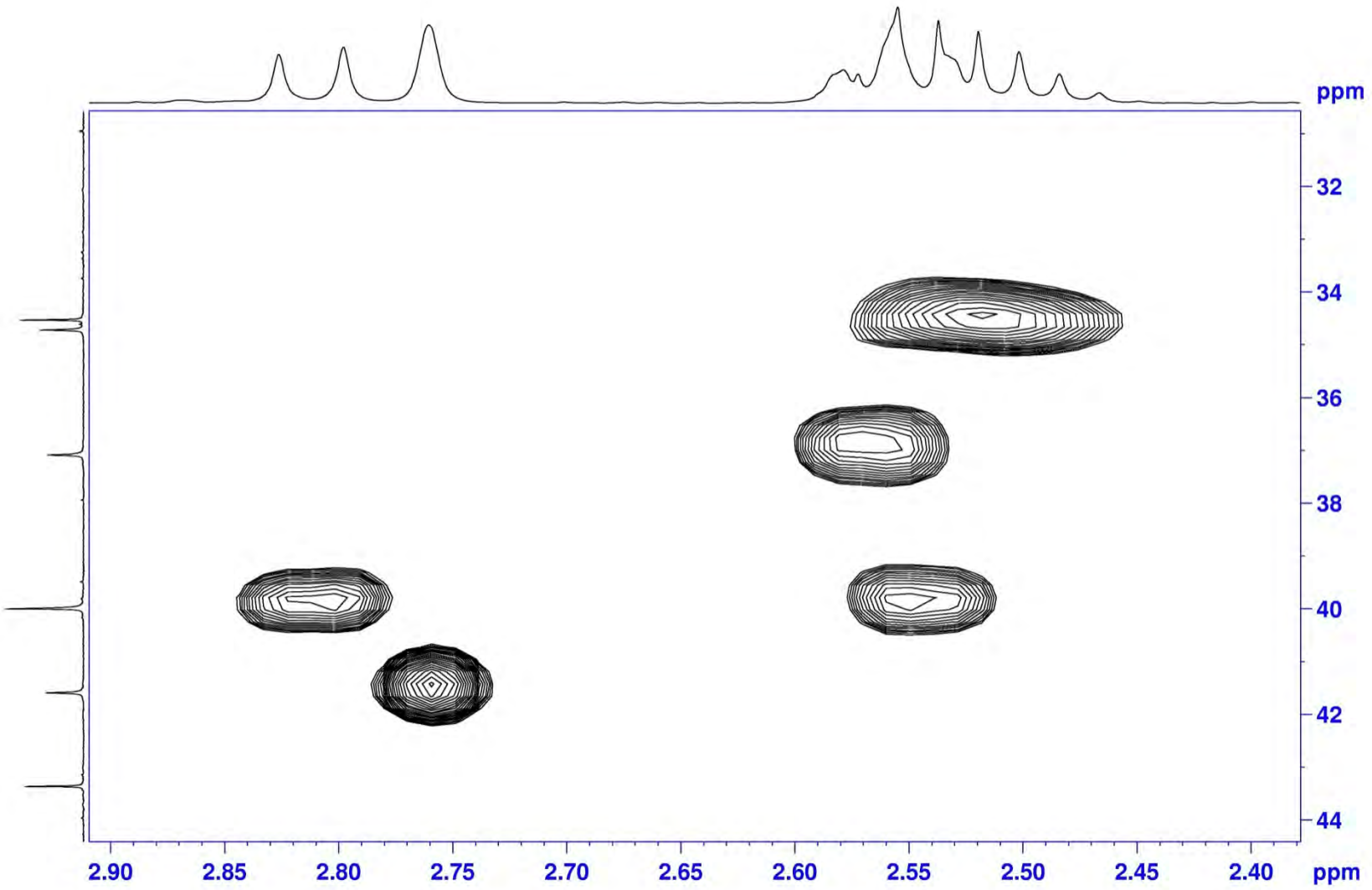
SI	1024
MC2	echo-antiecho
SF	100.6127685 MHz

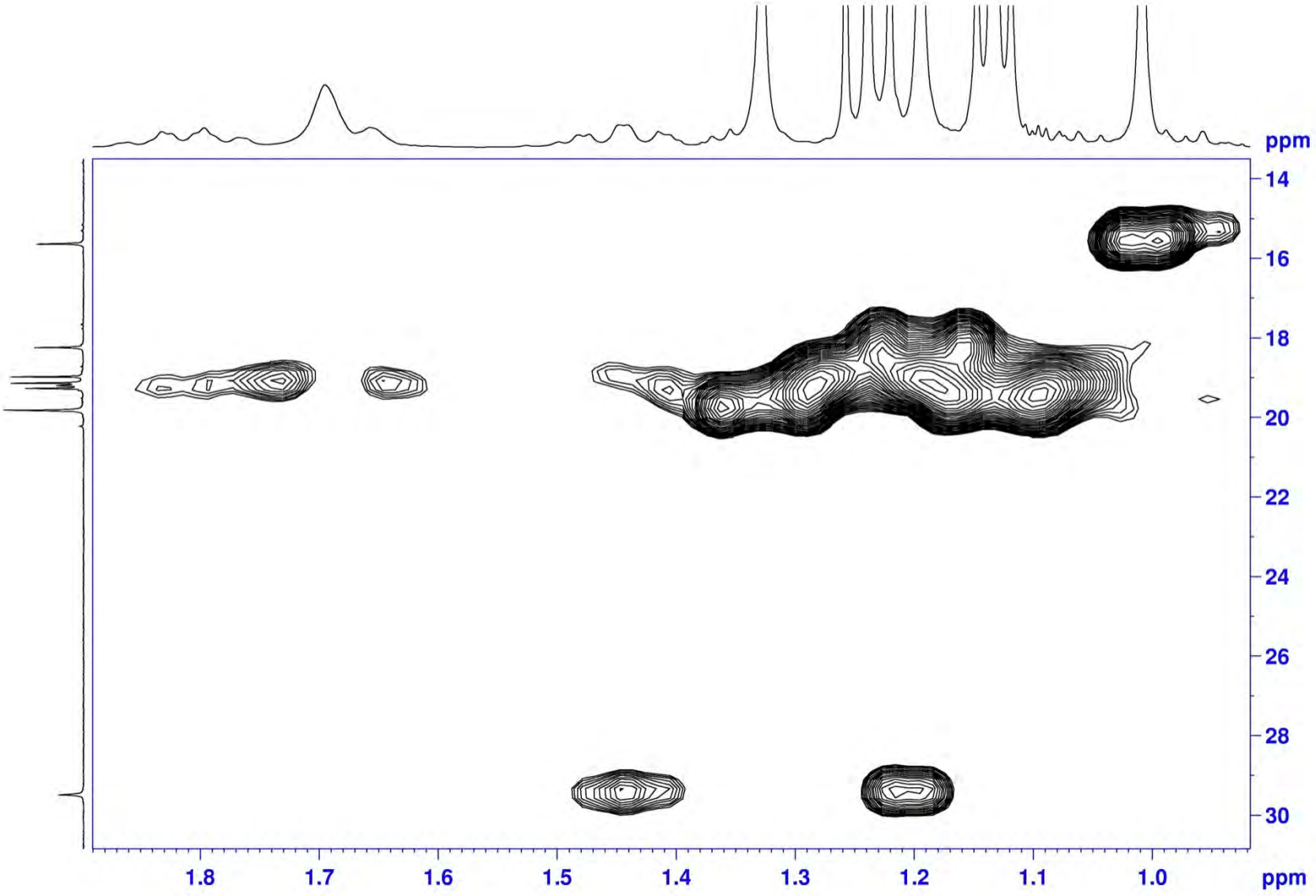
HSQC (400 MHz) spectrum of compound **5a** in $CDCl_3$



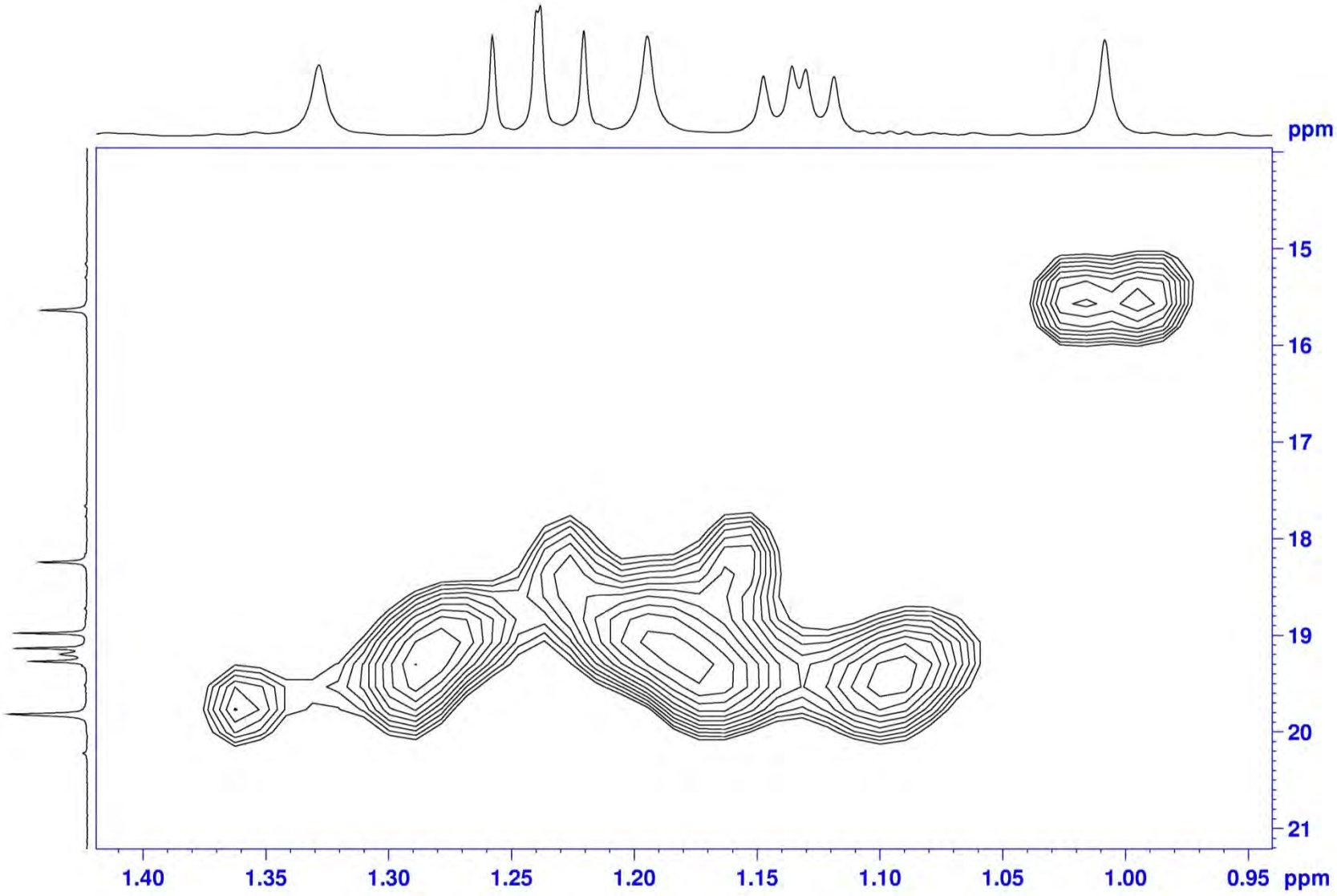
HSQC (400 MHz) spectrum of compound **5a** in $CDCl_3$



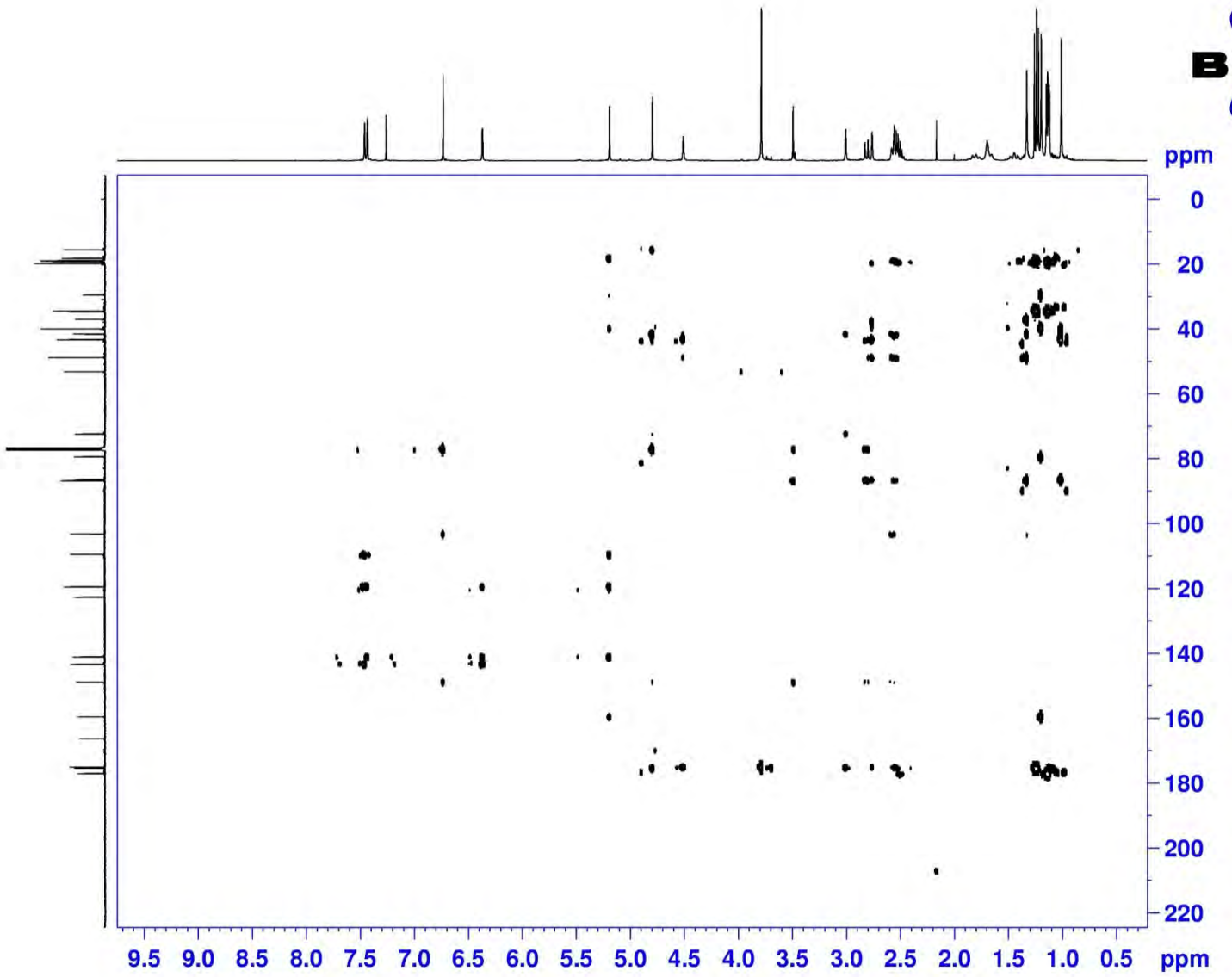
HSQC (400 MHz) spectrum of compound **5a** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **5a** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



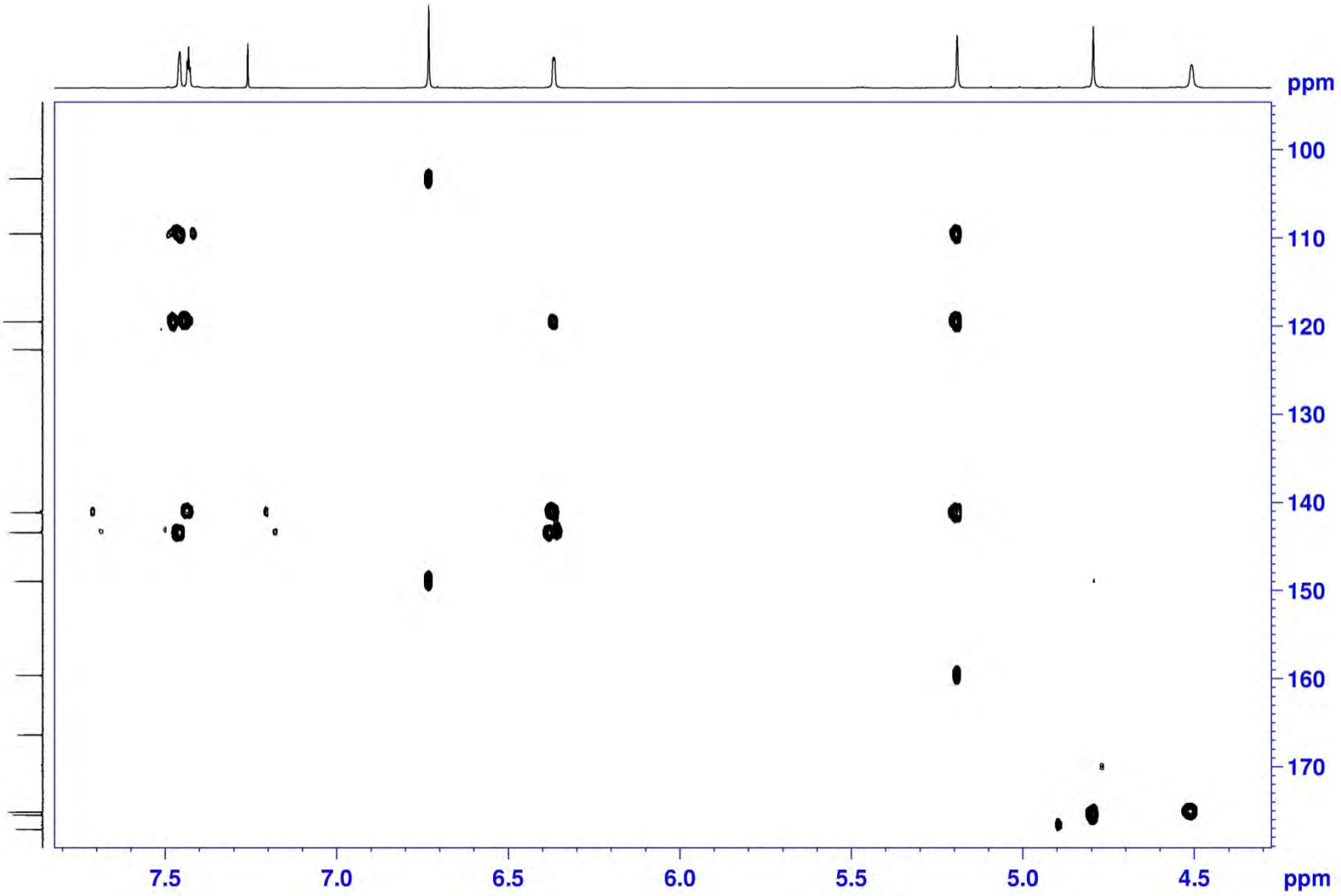
BRUKER

NAME	lws-42-2-fd
EXPNO	6
PROCNO	1
Date_	20150811
Time	23.00
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	hmbcgplpndqf
TD	4096
SOLVENT	CDCl3
NS	64
DS	16
SWH	5197.505 Hz
FIDRES	1.268922 Hz
AQ	0.3940852 sec
RG	208.5
DW	96.200 usec
DE	10.00 usec
TE	297.0 K
CNST2	145.0000000
CNST13	10.0000000
D0	0.00000300 sec
D1	1.50000000 sec
D2	0.00344828 sec
D6	0.05000000 sec
D16	0.00020000 sec
IN0	0.00002080 sec

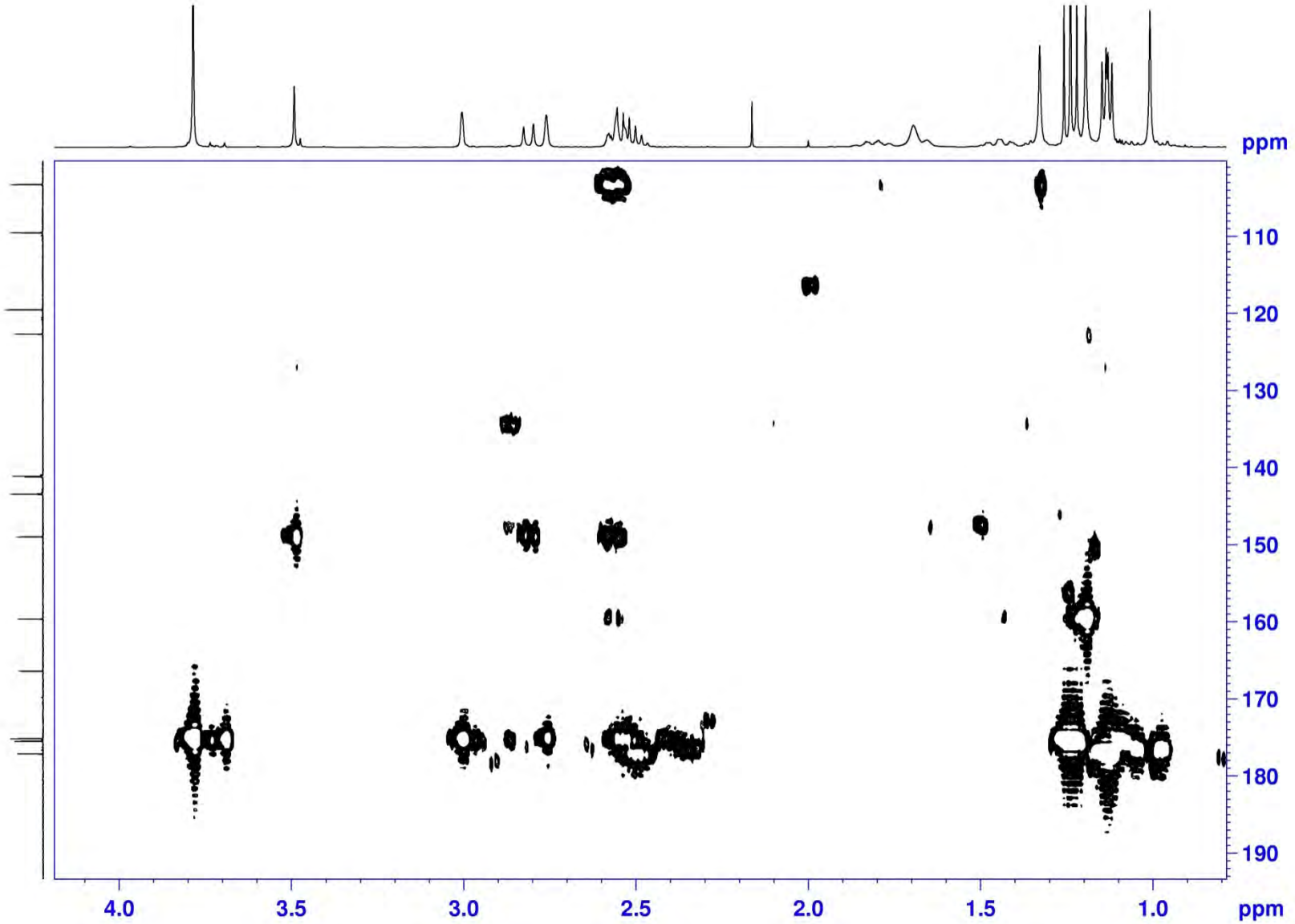
======== CHANNEL f1 ======

SFO1	400.1323208 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
ND0	2
TD	128
SFO1	100.6233 MHz
FIDRES	187.800476 Hz
SW	238.896 ppm
FnMODE	QF
SI	2048
SF	400.1300070 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	100.6127685 MHz
WDW	SINE
SSB	0
LB	0.00 Hz

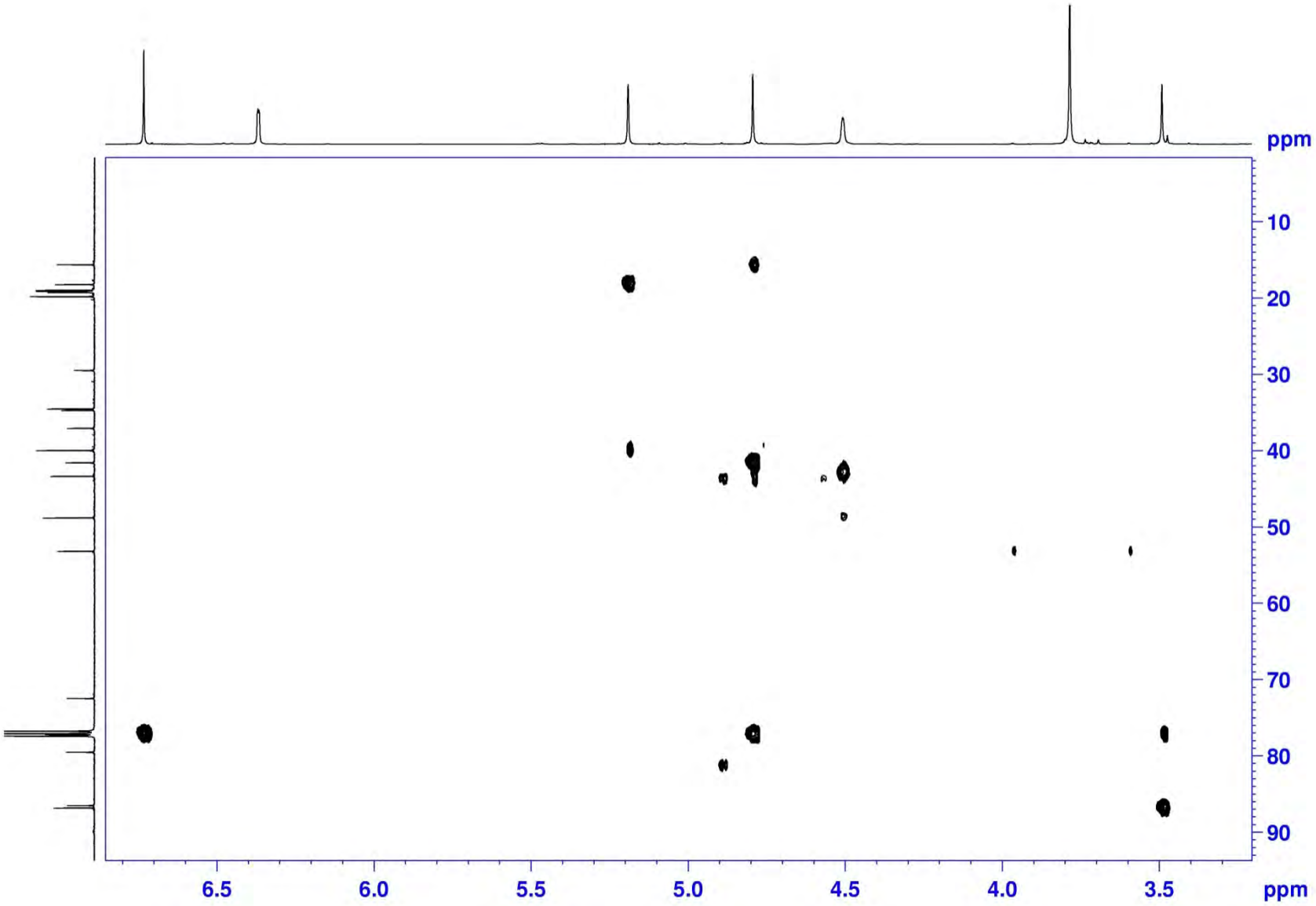
HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



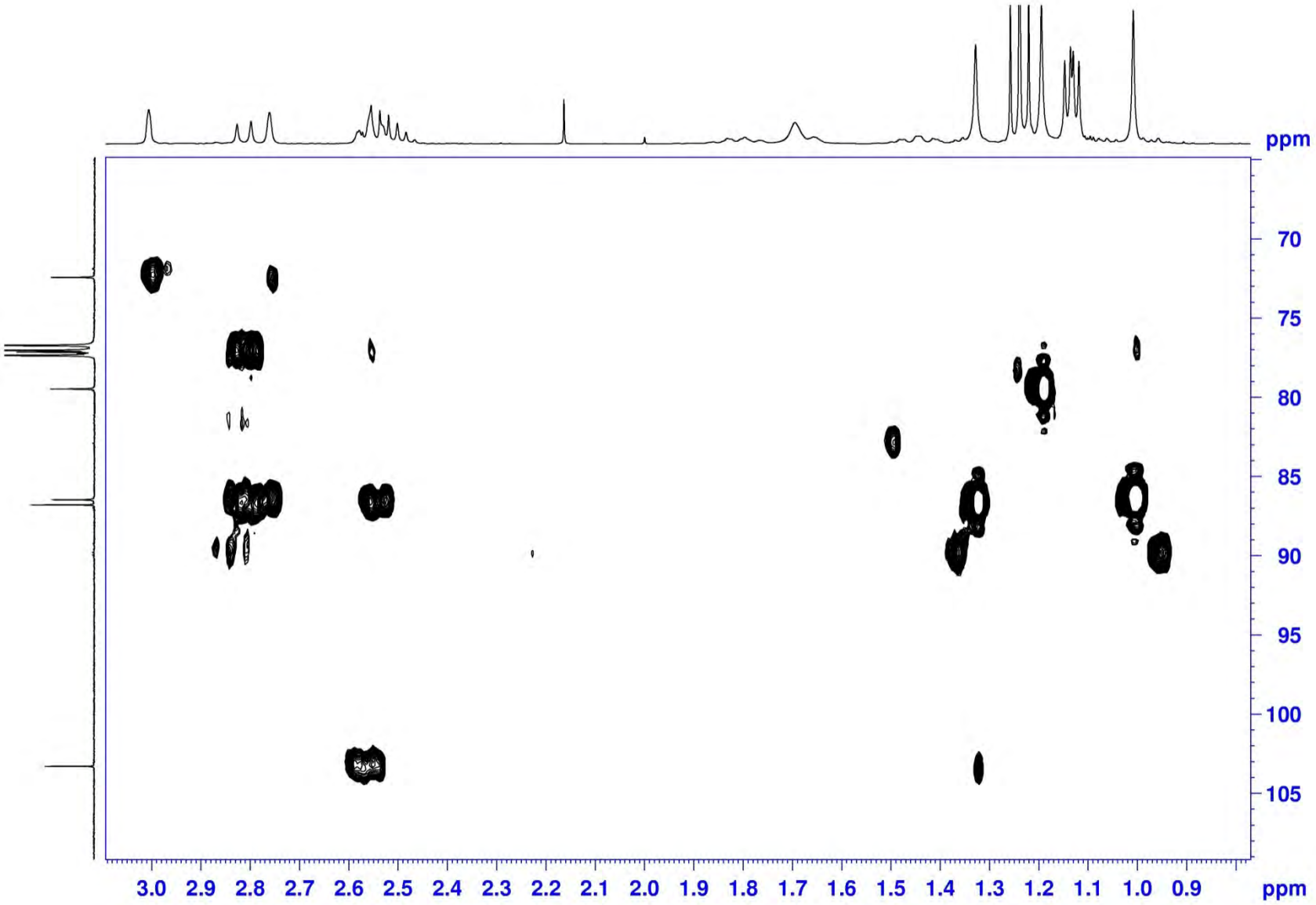
HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



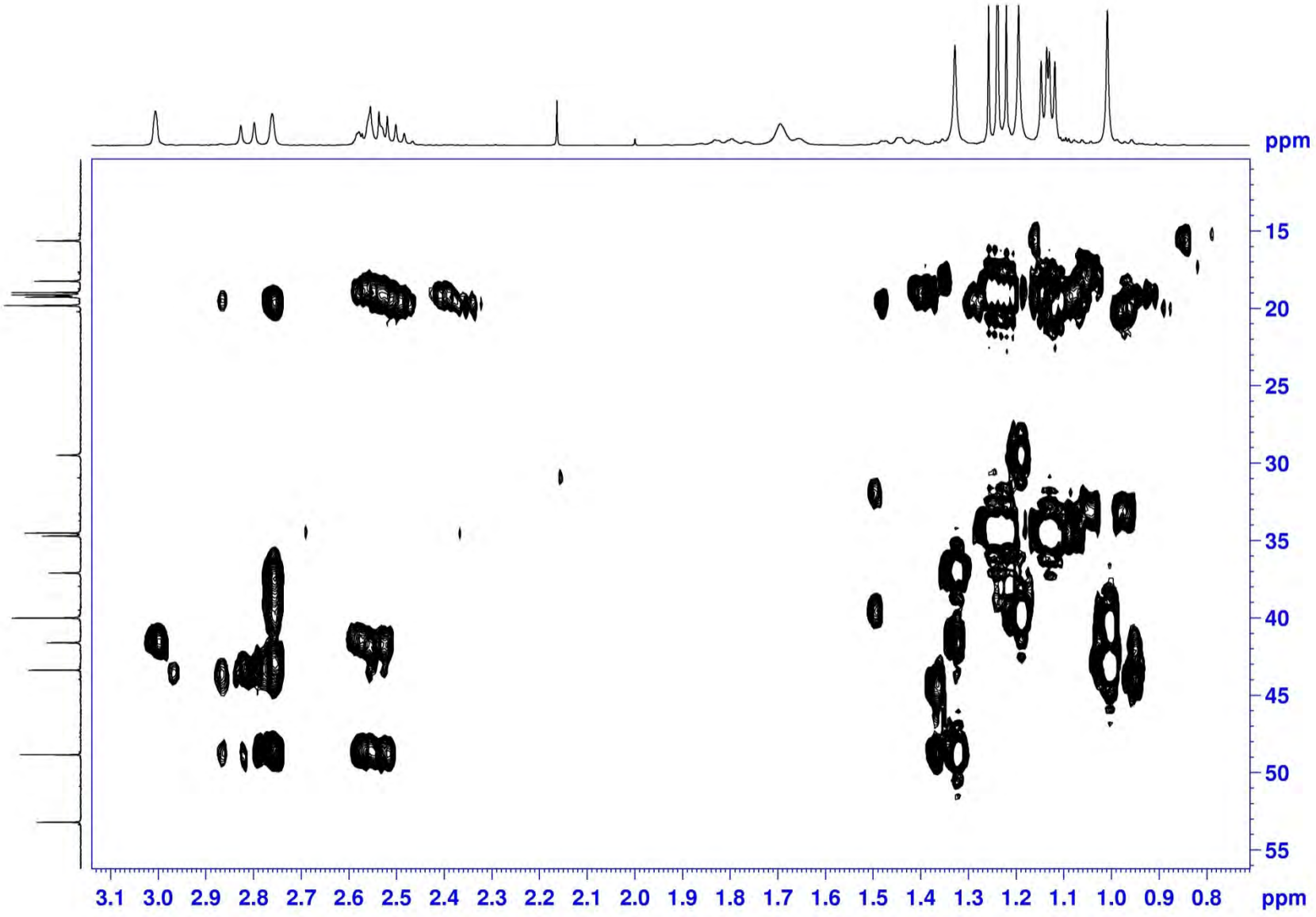
HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



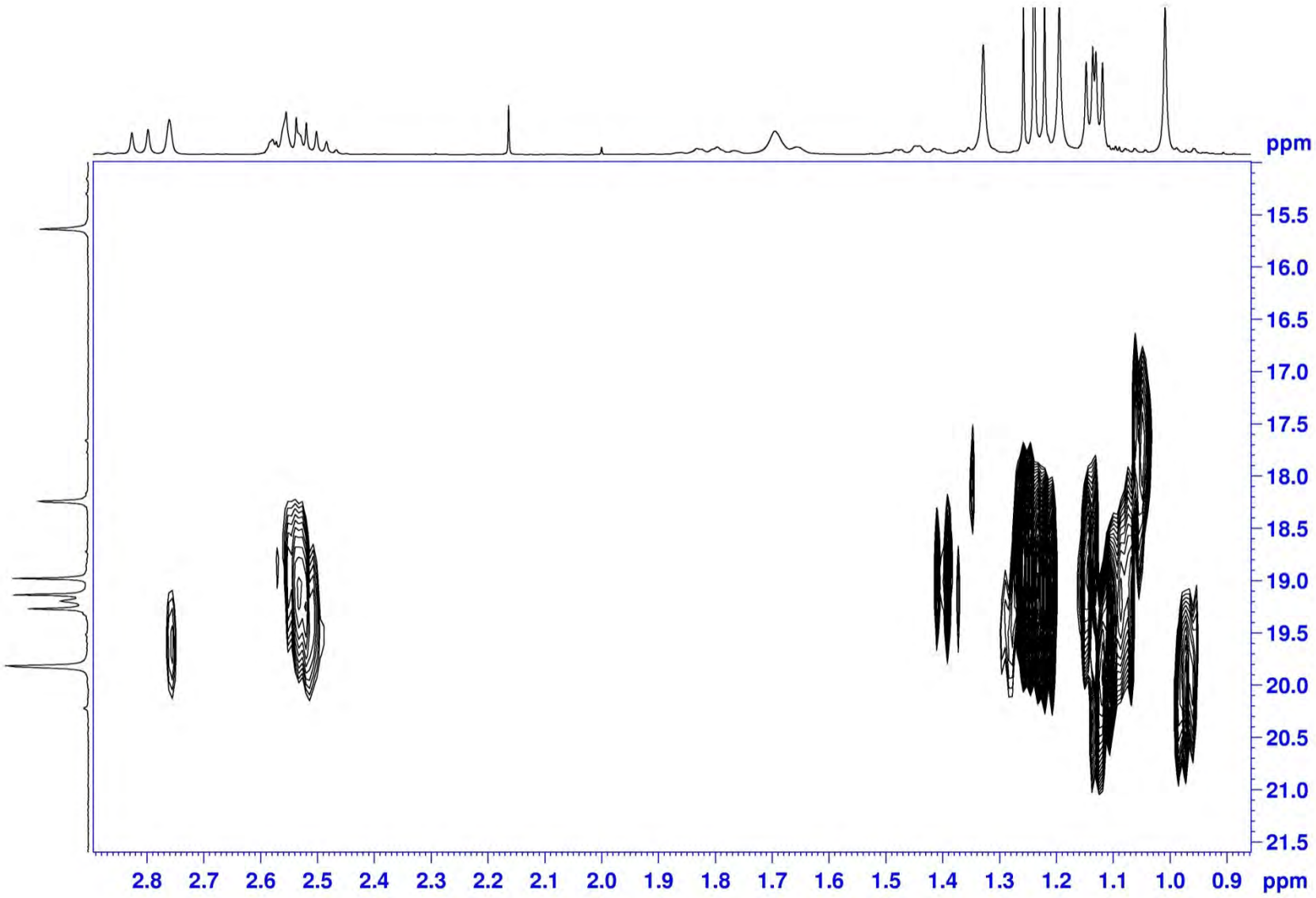
HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



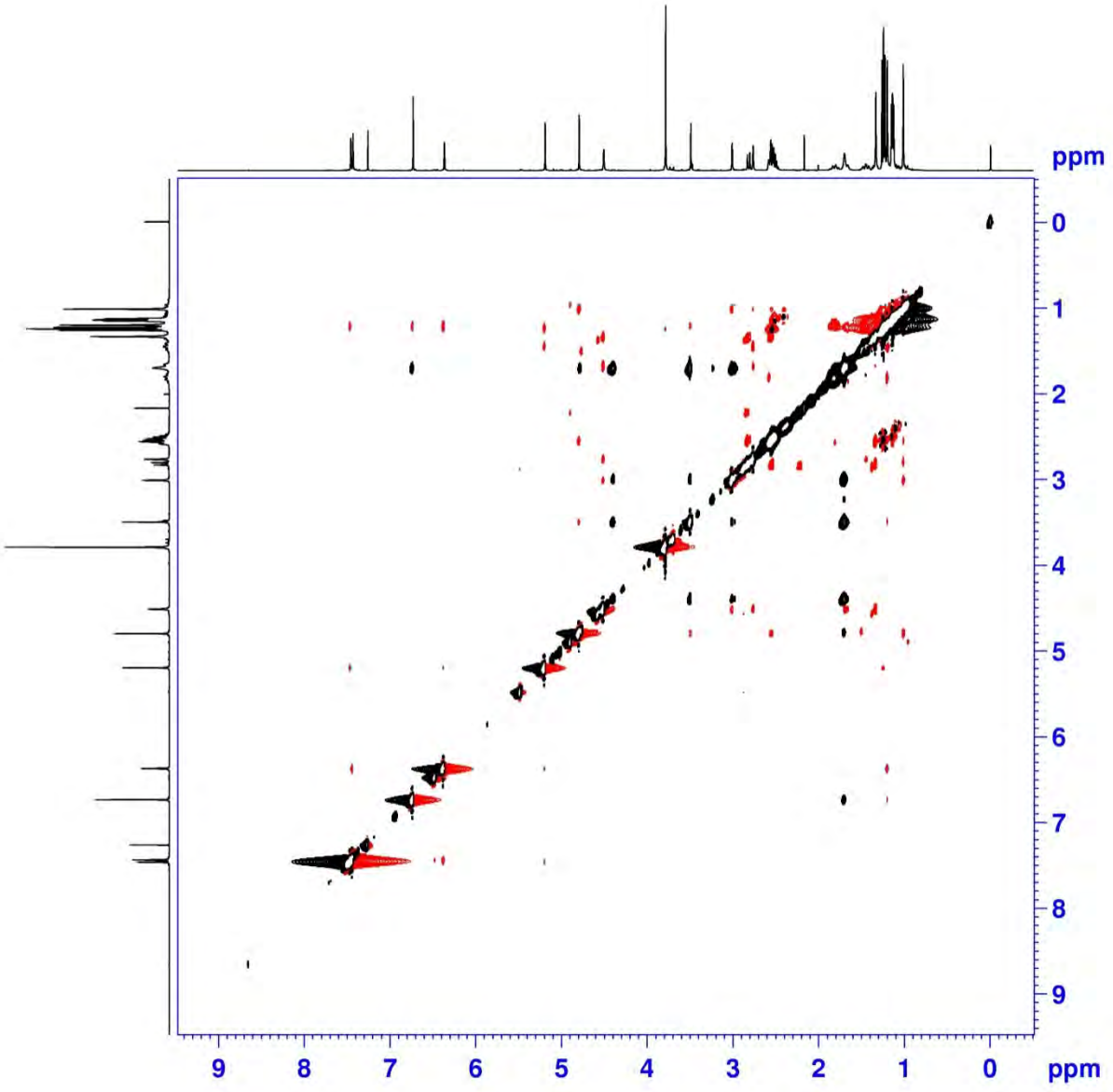
HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **5a** in $CDCl_3$



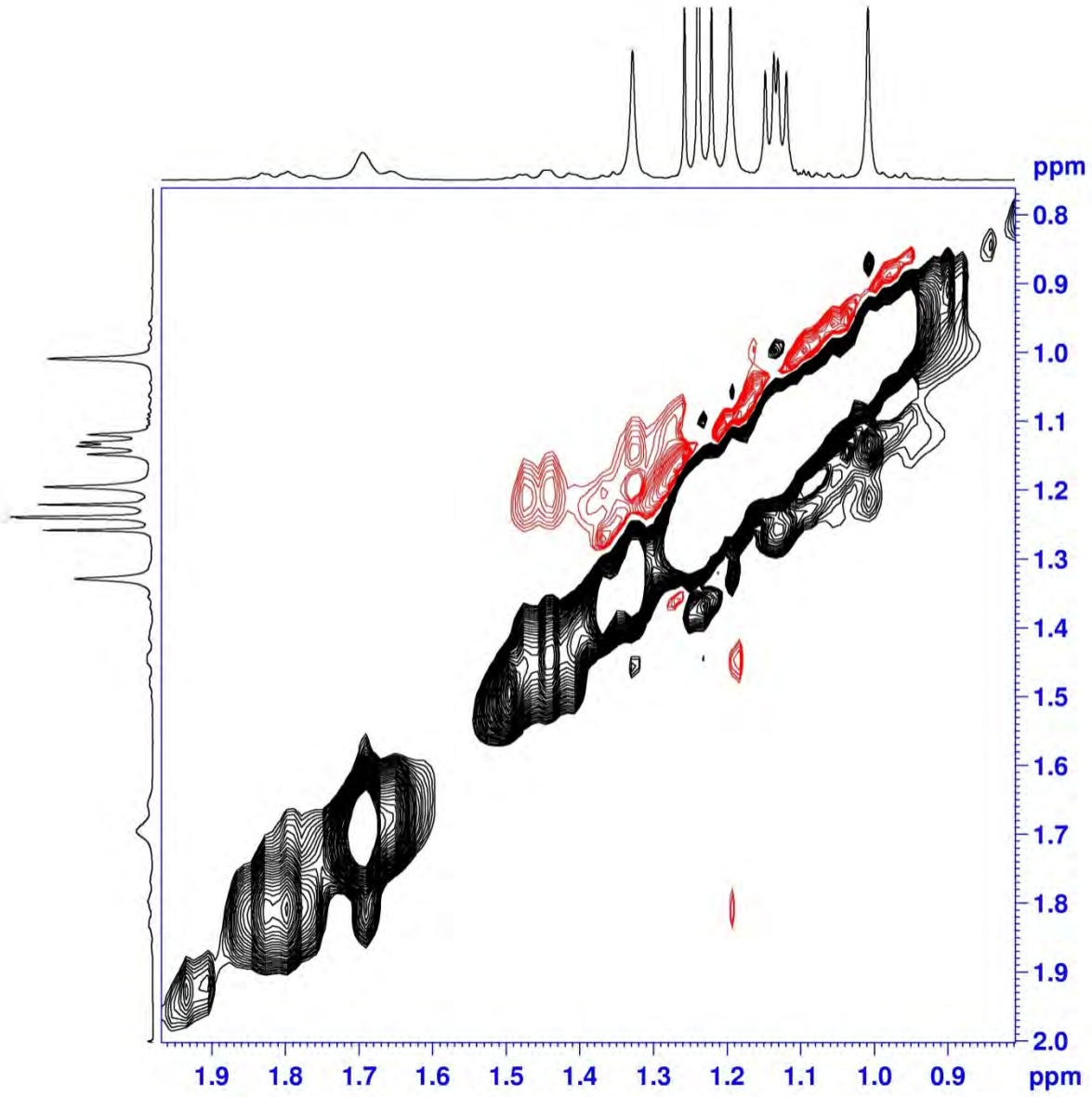
NOESY (400 MHz) spectrum of compound **5a** in $CDCl_3$



BRUKER

NAME	lws-42-2-fd	
EXPNO	7	
PROCNO	1	
Date_	20150812	
Time	3.29	
INSTRUM	spect	
PROBHD	5 mm CPPBBO BB	
PULPROG	noesygpphpp	
TD	2048	
SOLVENT	CDCl3	
NS	32	
DS	32	
SWH	4000.000	Hz
FIDRES	1.953125	Hz
AQ	0.2560500	sec
RG	117.37	
DW	125.000	usec
DE	10.00	usec
TE	297.0	K
D0	0.00010972	sec
D1	1.99385595	sec
D8	0.30000001	sec
D11	0.03000000	sec
D12	0.00002000	sec
D16	0.00020000	sec
IN0	0.00025000	sec
======== CHANNEL f1 ========		
SFO1	400.1318006	MHz
NUC1	1H	
P1	12.00	usec
P2	24.00	usec
P17	2500.00	usec
ND0	1	
TD	256	
SFO1	400.1318	MHz
FIDRES	15.625000	Hz
SW	9.997	ppm
FnMODE	States-TPPI	
SI	1024	
SF	400.1300094	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.00	
SI	1024	
MC2	States-TPPI	
SF	400.1300094	MHz
WDW	QSINE	
SSB	2	

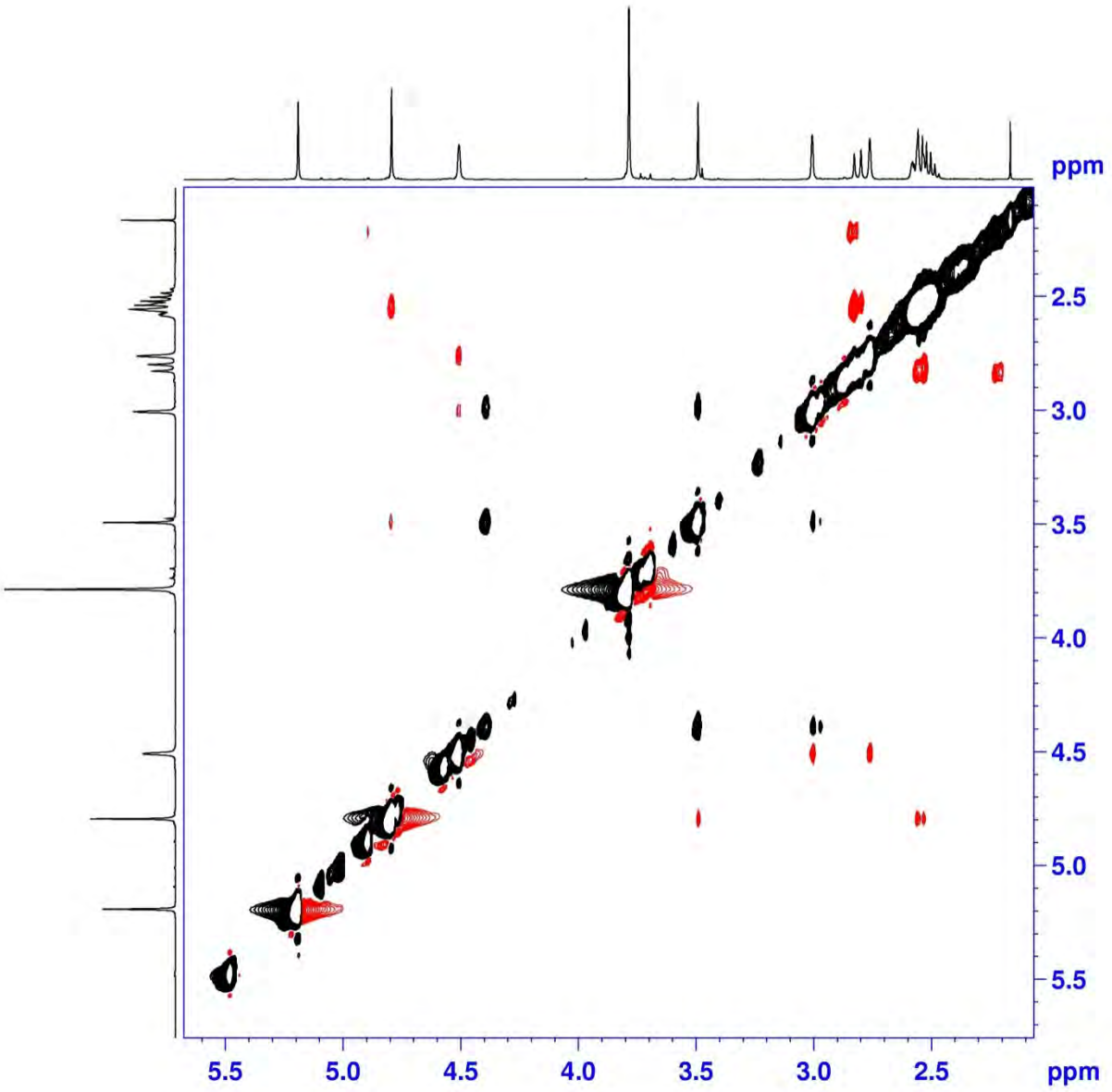
NOESY (400 MHz) spectrum of compound **5a** in $CDCl_3$



BRUKER

NAME	1ws-42-2-fd	
EXPNO	7	
PROCNO	1	
Date_	20150812	
Time	3.29	
INSTRUM	spect	
PROBHD	5 mm CPPBBO BB	
PULPROG	noesygpphpp	
TD	2048	
SOLVENT	CDCl3	
NS	32	
DS	32	
SWH	4000.000	Hz
FIDRES	1.953125	Hz
AQ	0.2560500	sec
RG	117.37	
DW	125.000	usec
DE	10.00	usec
TE	297.0	K
D0	0.00010972	sec
D1	1.99385595	sec
D8	0.30000001	sec
D11	0.03000000	sec
D12	0.00002000	sec
D16	0.00020000	sec
IN0	0.00025000	sec
======== CHANNEL f1 ========		
SFO1	400.1318006	MHz
NUC1	1H	
P1	12.00	usec
P2	24.00	usec
P17	2500.00	usec
ND0	1	
TD	256	
SFO1	400.1318	MHz
FIDRES	15.625000	Hz
SW	9.997	ppm
FnMODE	States-TPPI	
SI	1024	
SF	400.1300094	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.00	
SI	1024	
MC2	States-TPPI	
SF	400.1300094	MHz
WDW	QSINE	
SSB	2	

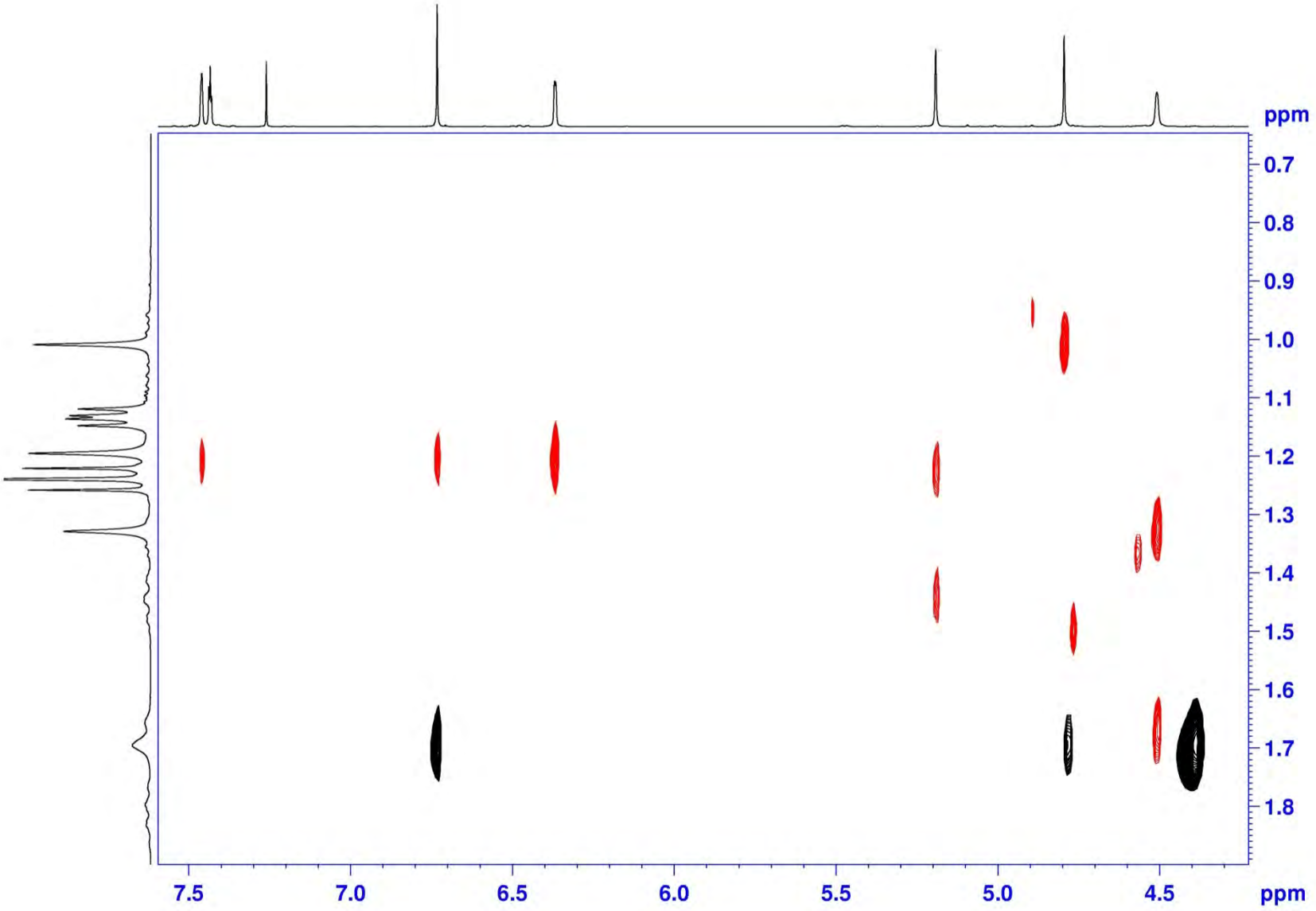
NOESY (400 MHz) spectrum of compound **5a** in $CDCl_3$



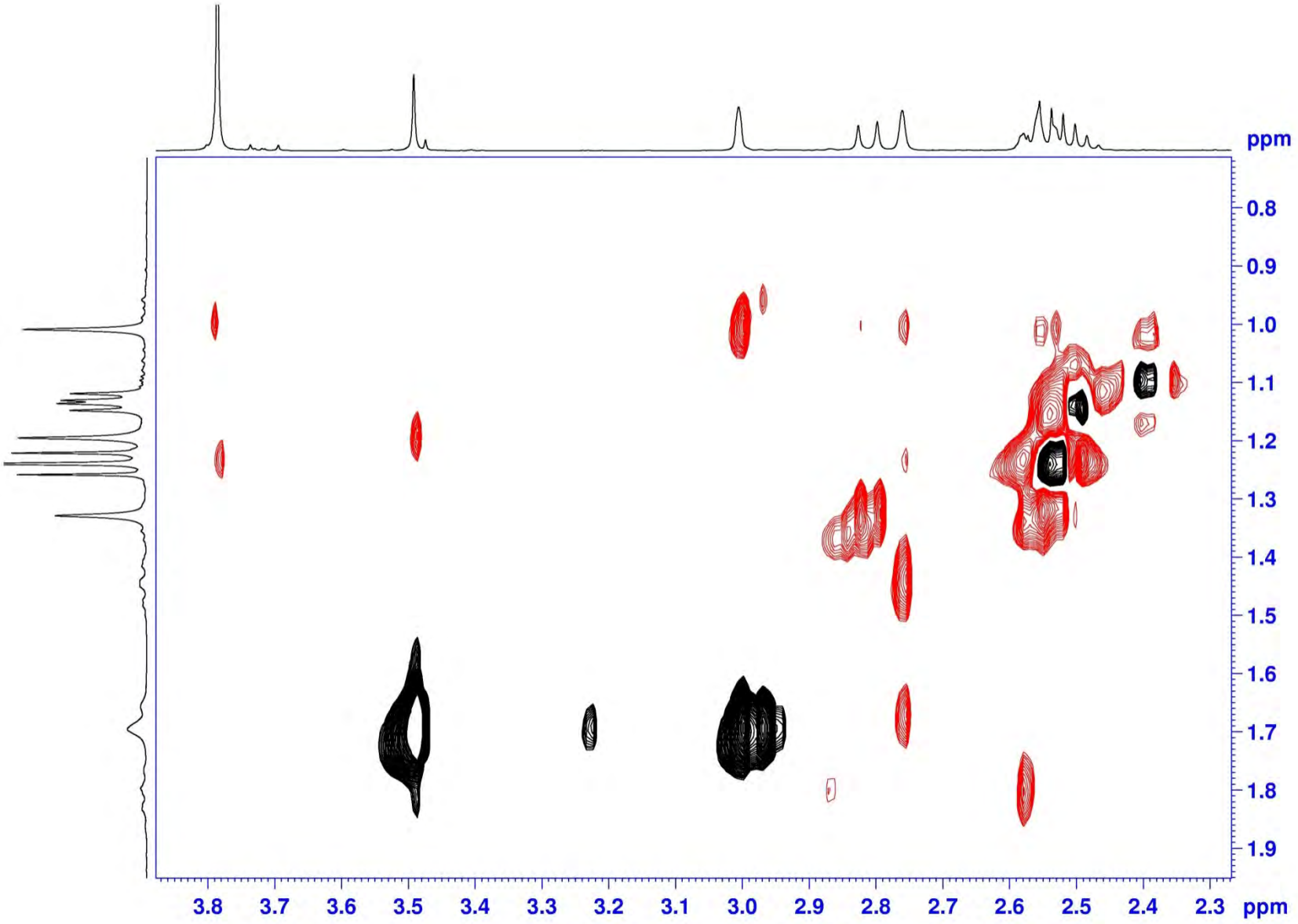
BRUKER

NAME	1ws-42-2-fd
EXPNO	7
PROCNO	1
Date_	20150812
Time	3.29
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	noesygpphpp
TD	2048
SOLVENT	CDCl3
NS	32
DS	32
SWH	4000.000 Hz
FIDRES	1.953125 Hz
AQ	0.2560500 sec
RG	117.37
DW	125.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00010972 sec
D1	1.99385595 sec
D8	0.30000001 sec
D11	0.03000000 sec
D12	0.00002000 sec
D16	0.00020000 sec
IN0	0.00025000 sec
======== CHANNEL f1 ========	
SFO1	400.1318006 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
P17	2500.00 usec
ND0	1
TD	256
SFO1	400.1318 MHz
FIDRES	15.625000 Hz
SW	9.997 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300094 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300094 MHz
WDW	QSINE
SSB	2

NOESY (400 MHz) spectrum of compound **5a** in $CDCl_3$



NOESY (400 MHz) spectrum of compound **5a** in $CDCl_3$

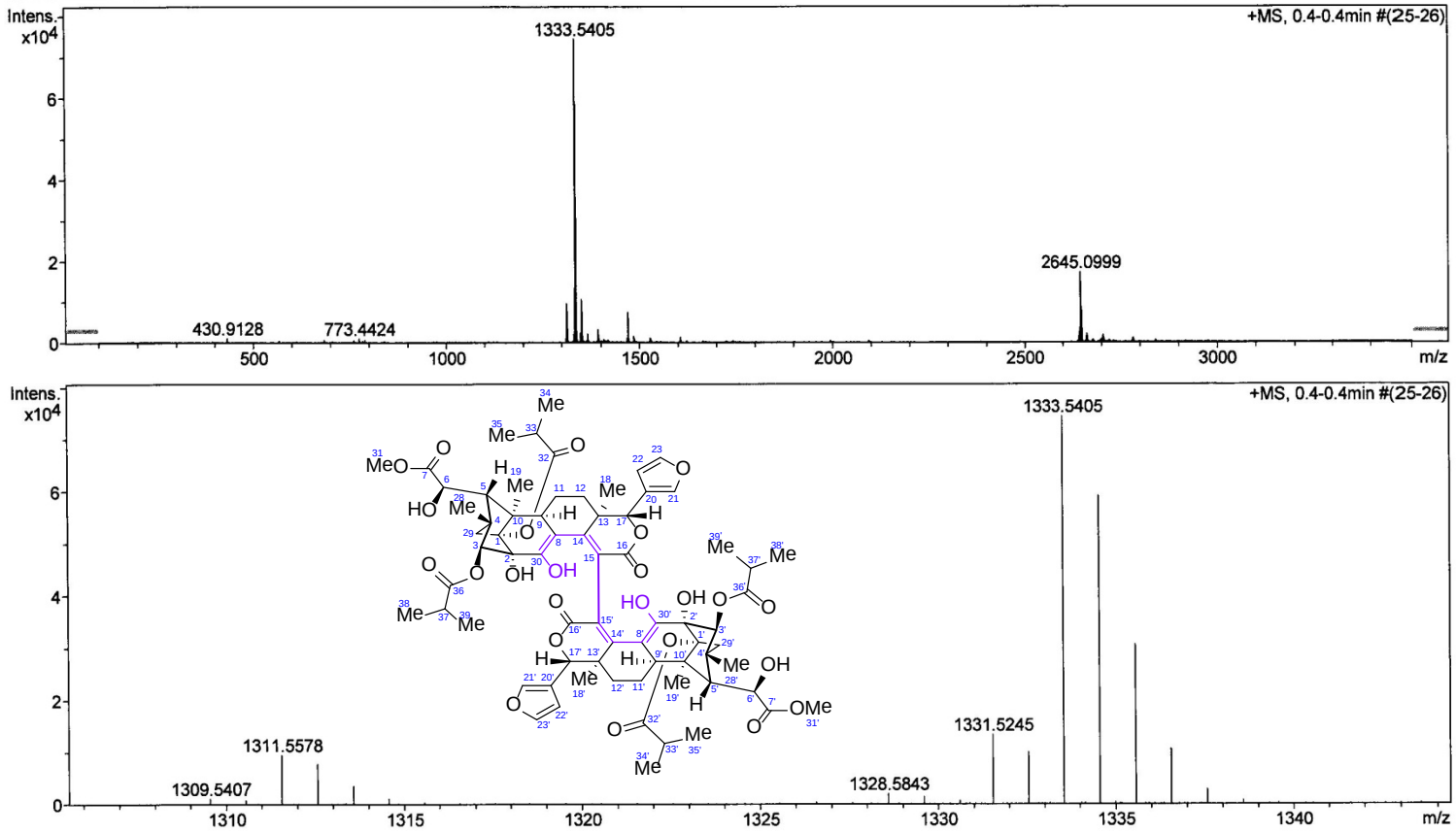


HR-ESIMS for compound **5b**

Generic Display Report

Analysis Info

Analysis Name	D:\Data\MS\data\201509\liwanshan_lws-42-7_pos_8_01_357.d	Acquisition Date	9/8/2015 1:39:55 AM
Method	LC_Direct Infusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	liwanshan_lws-42-7_pos	Instrument	maXis
Comment			



Bruker Compass DataAnalysis 4.0 printed: 9/8/2015 2:48:29 AM Page 1 of 1

HR-ESIMS for compound **5b**

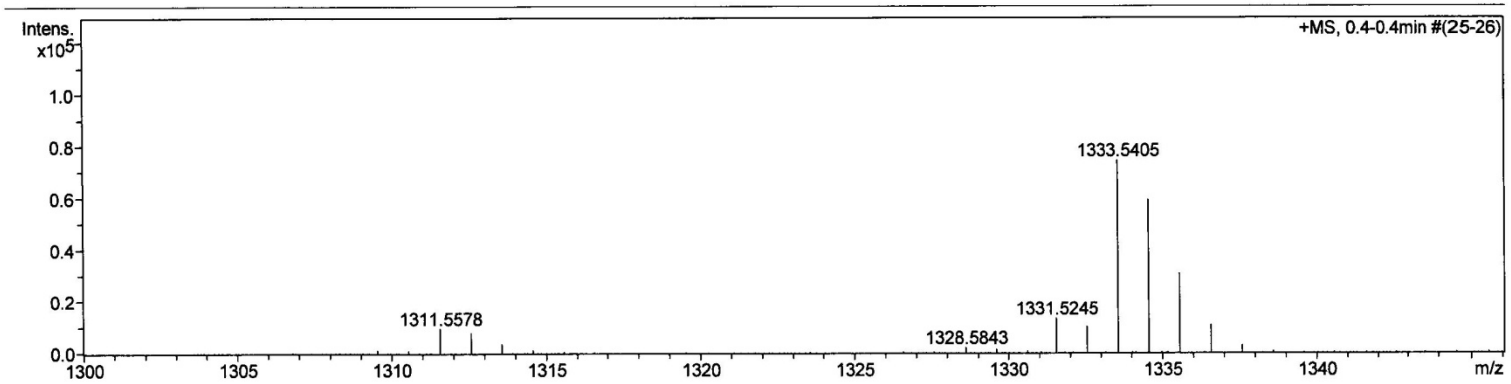
Mass Spectrum SmartFormula Report

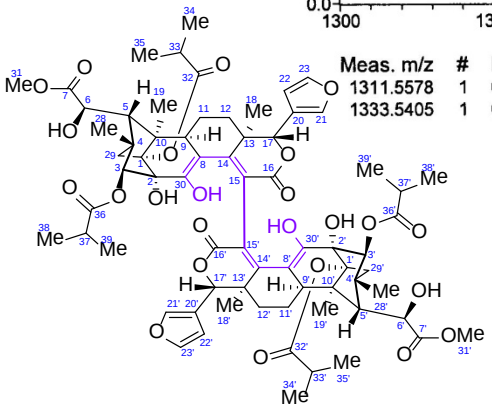
Analysis Info

Analysis Name	D:\Data\MS\data\201509\liwanshan_lws-42-7_pos_8_01_357.d	Acquisition Date	9/8/2015 1:39:55 AM
Method	LC_Direct Infusion_pos_100-3000mz.m	Operator	SCSIO
Sample Name	liwanshan_lws-42-7_pos	Instrument / Ser#	maXis 29
Comment			

Acquisition Parameter

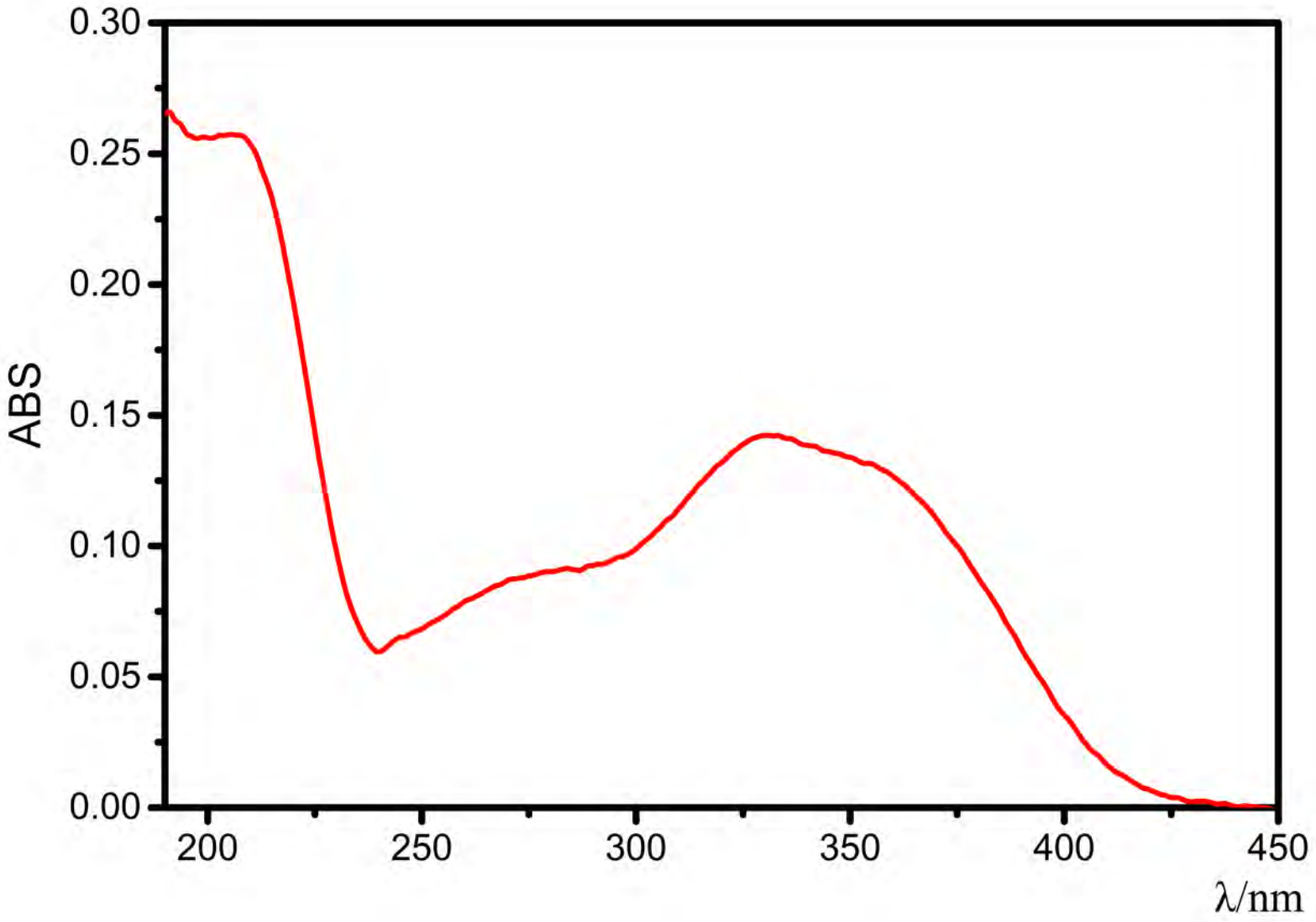
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3500 m/z	Set Collision Cell RF	1800.0 Vpp	Set Divert Valve	Waste



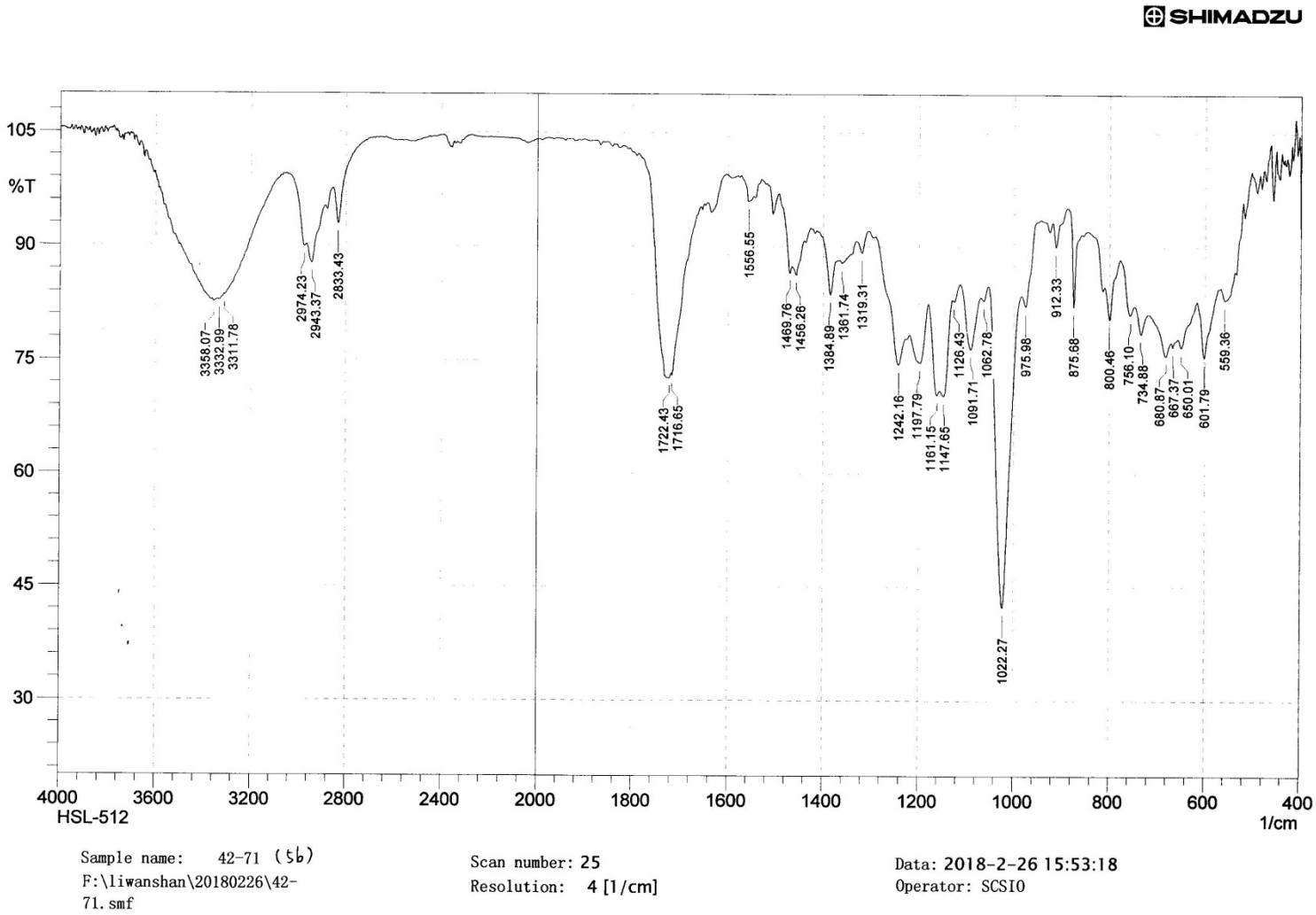


Meas. m/z	#	Formula	Score	m/z	err [ppm]	err [mDa]	mSigma	rdb	e⁻ Conf	N-Rule
1311.5578	1	C 70 H 87 O 24	100.00	1311.5582	0.3	0.4	16.1	27.5	even	ok
1333.5405	1	C 70 H 86 Na O 24	100.00	1333.5401	-0.3	-0.4	33.5	27.5	even	ok

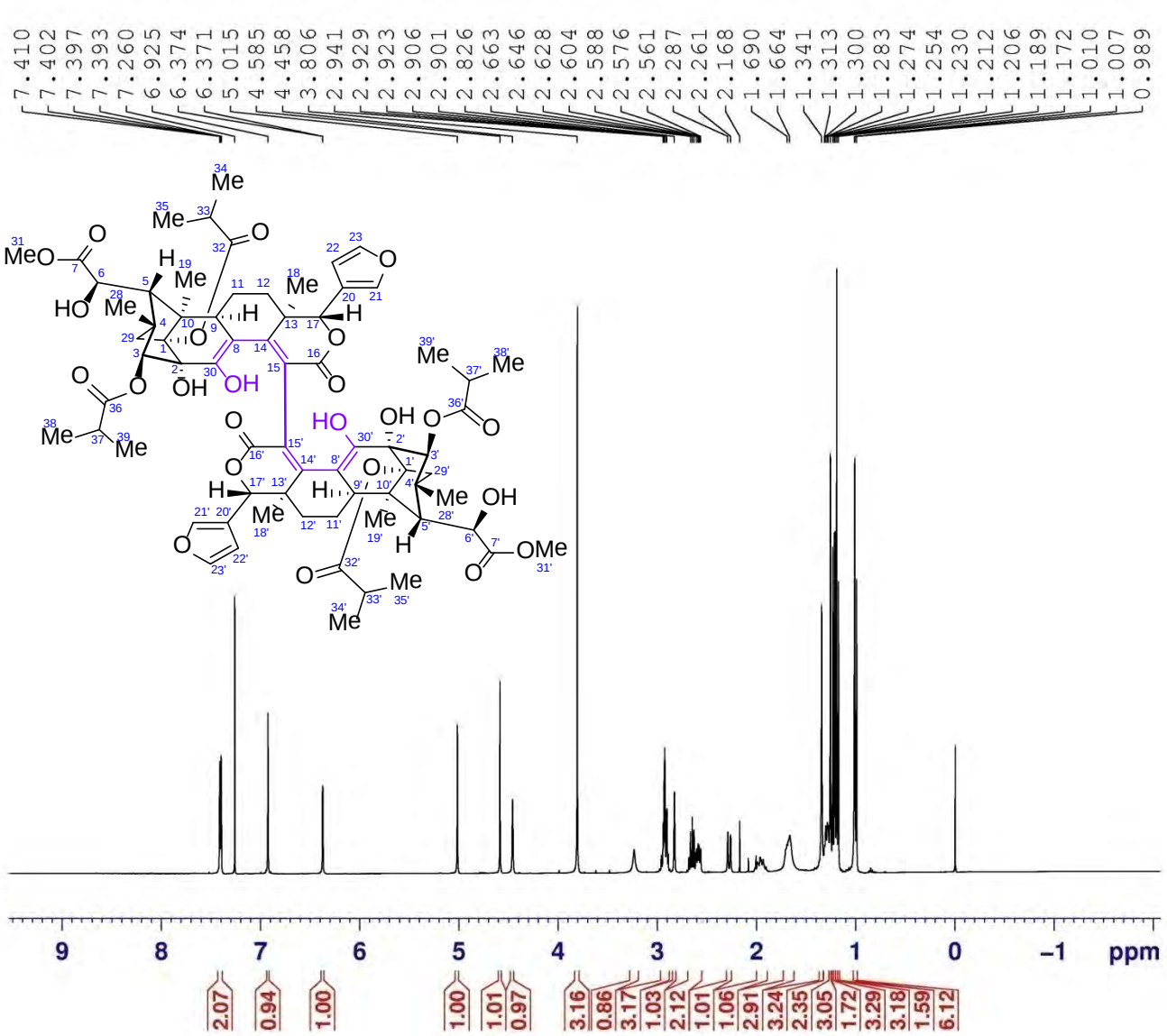
UV spectrum for compound **5b** in CH_3CN



IR spectrum for compound **5b**



^{1}H NMR (400 MHz) spectrum of compound **5b** in $CDCl_3$

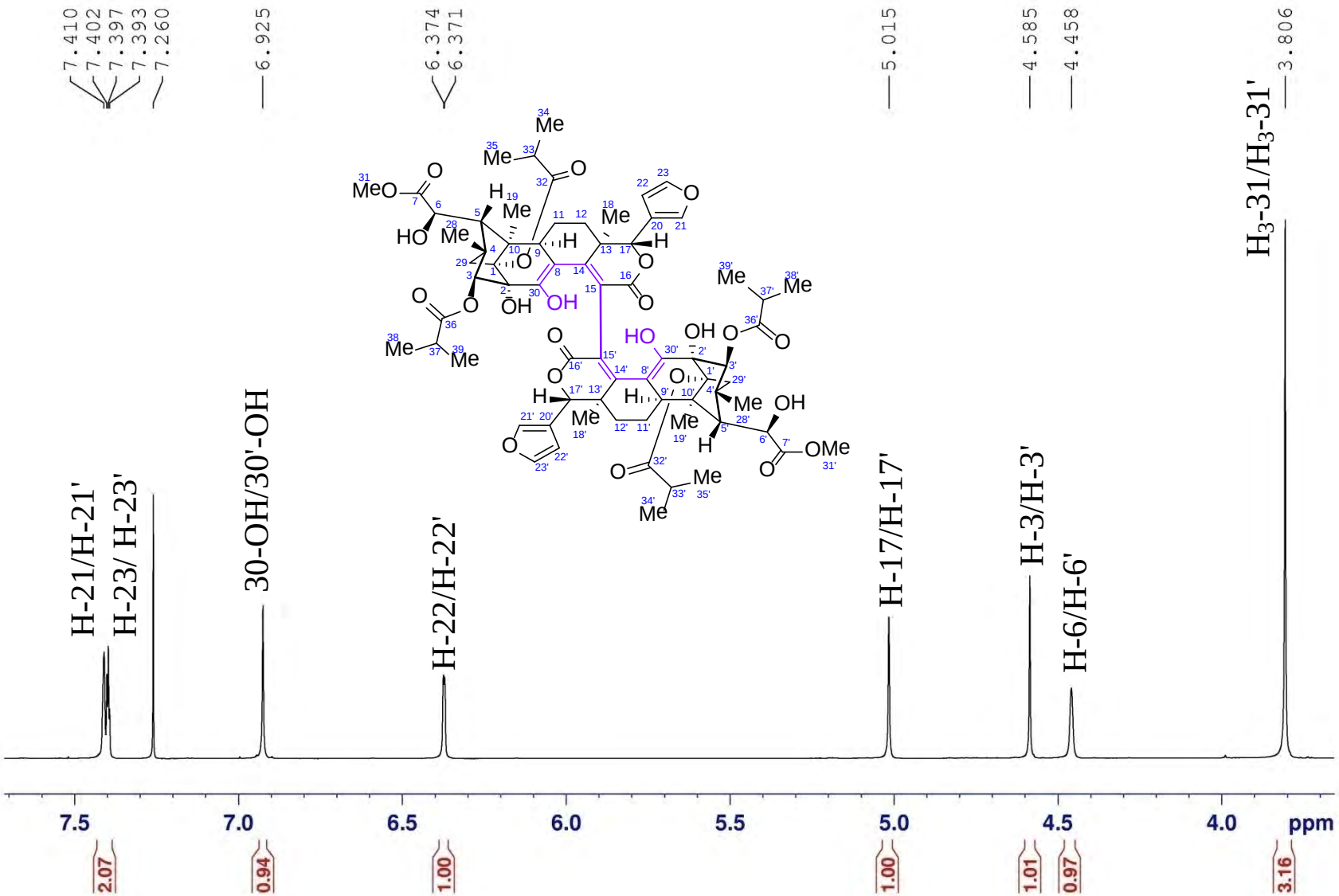


BRUKER

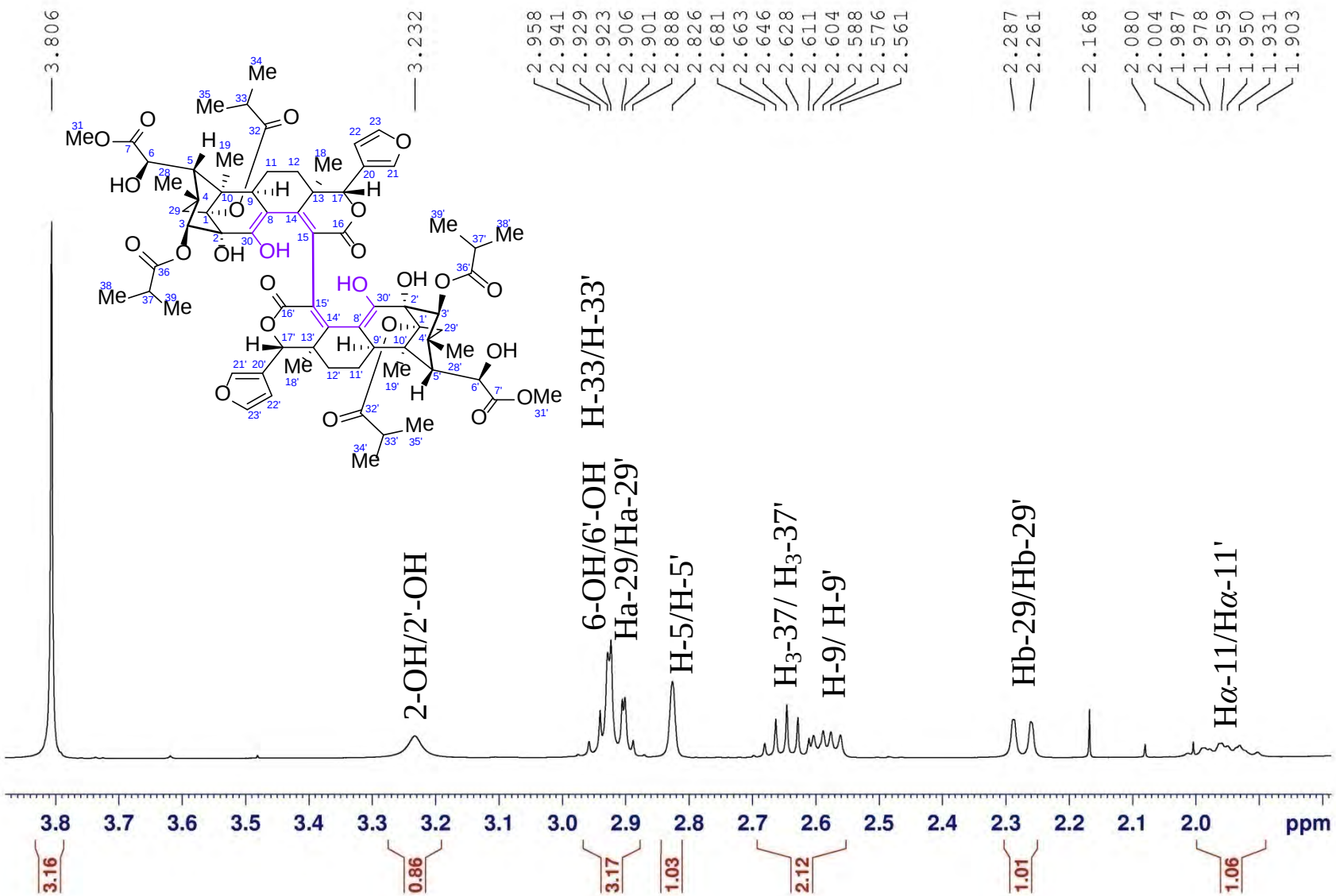
```
NAME          lws-42-7-fd
EXPNO                   1
PROCNO                  1
Date_            20150821
Time                23.33
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                  85.34
DW                  60.800 usec
DE                  10.00 usec
TE                  297.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  12.00 usec
SI                  65536
SF           400.1300094 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

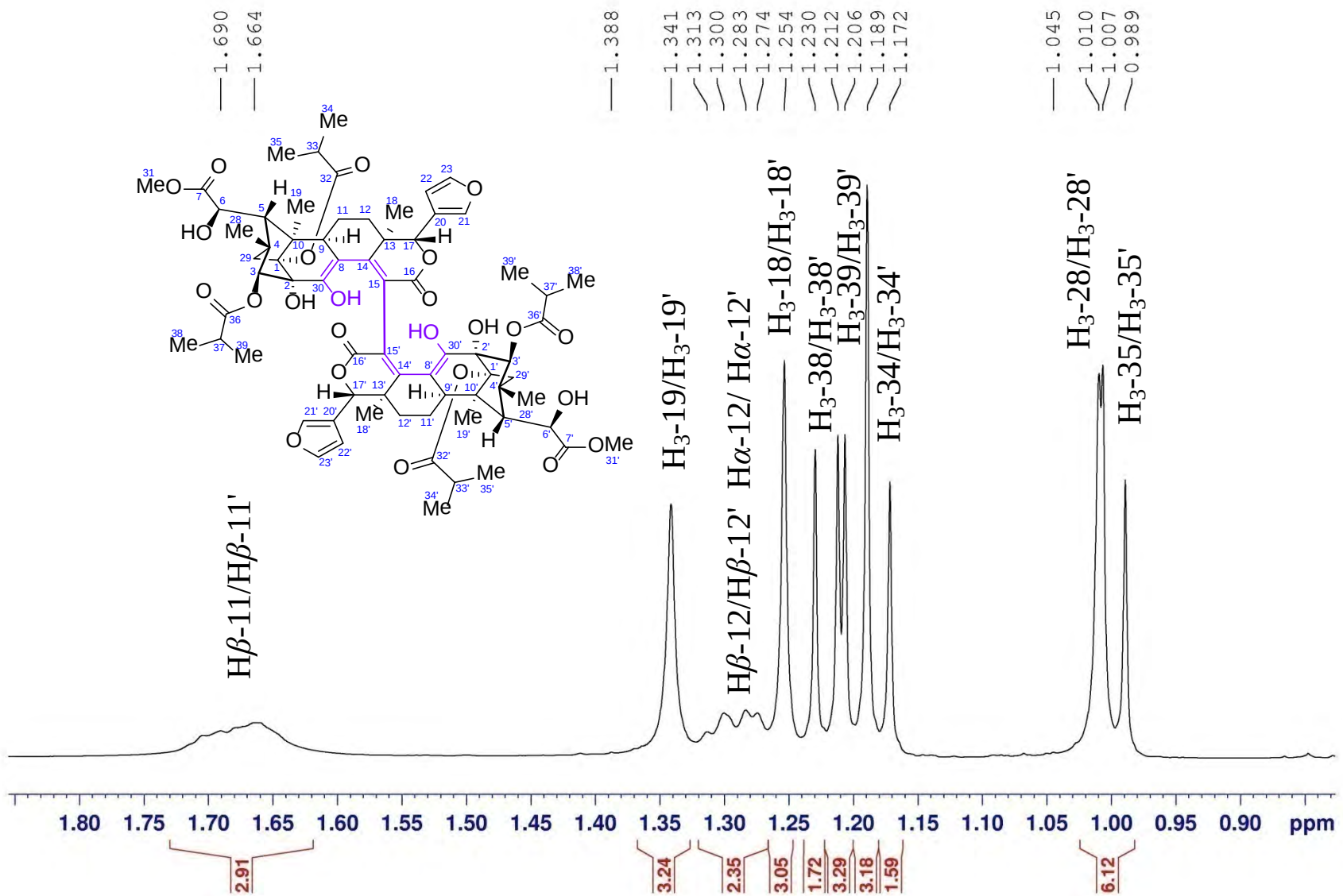
^{1}H NMR (400 MHz) spectrum of compound **5b** in $CDCl_3$



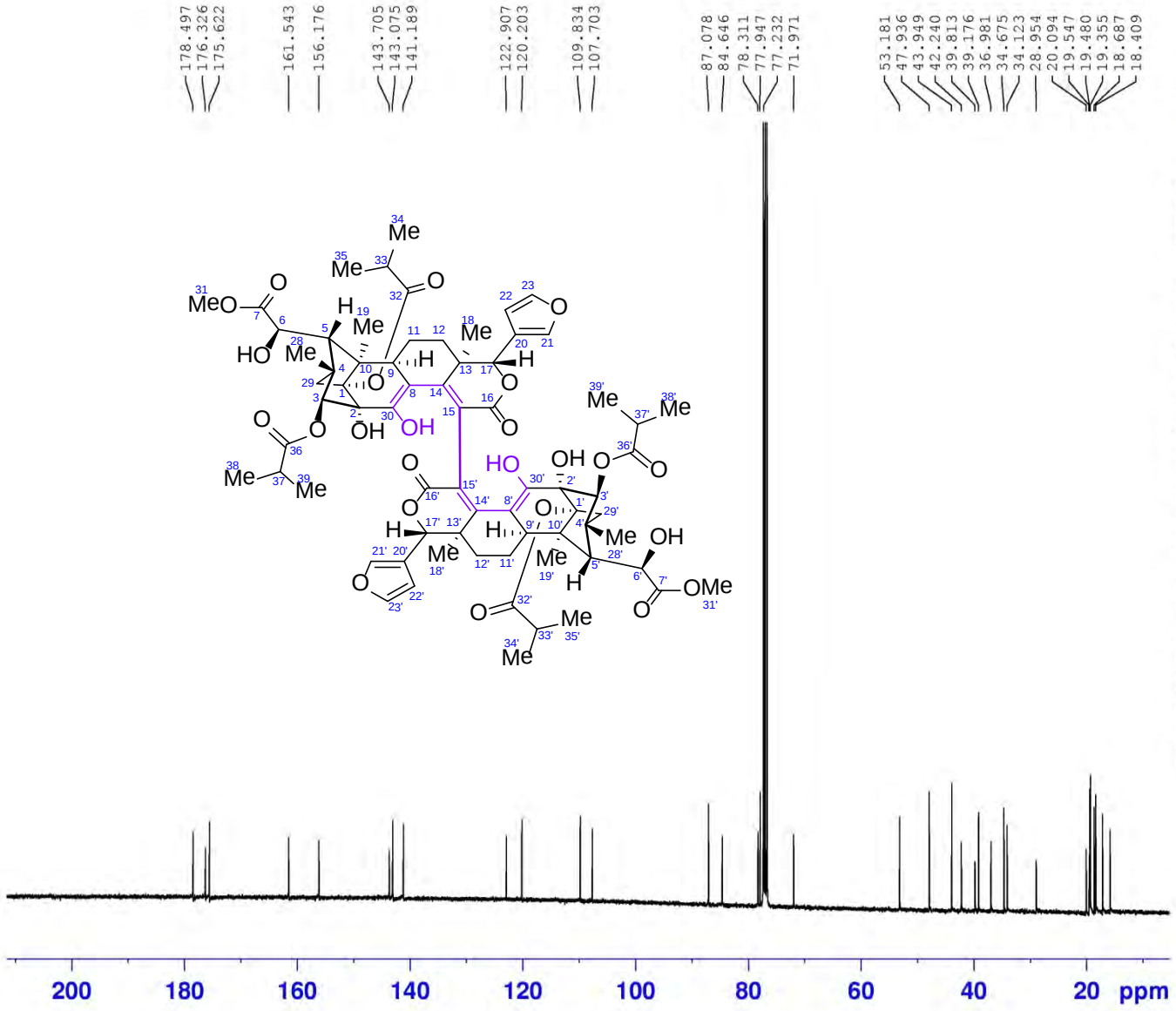
^{1}H NMR (400 MHz) spectrum of compound **5b** in $CDCl_3$



^{1}H NMR (400 MHz) spectrum of compound **5b** in $CDCl_3$

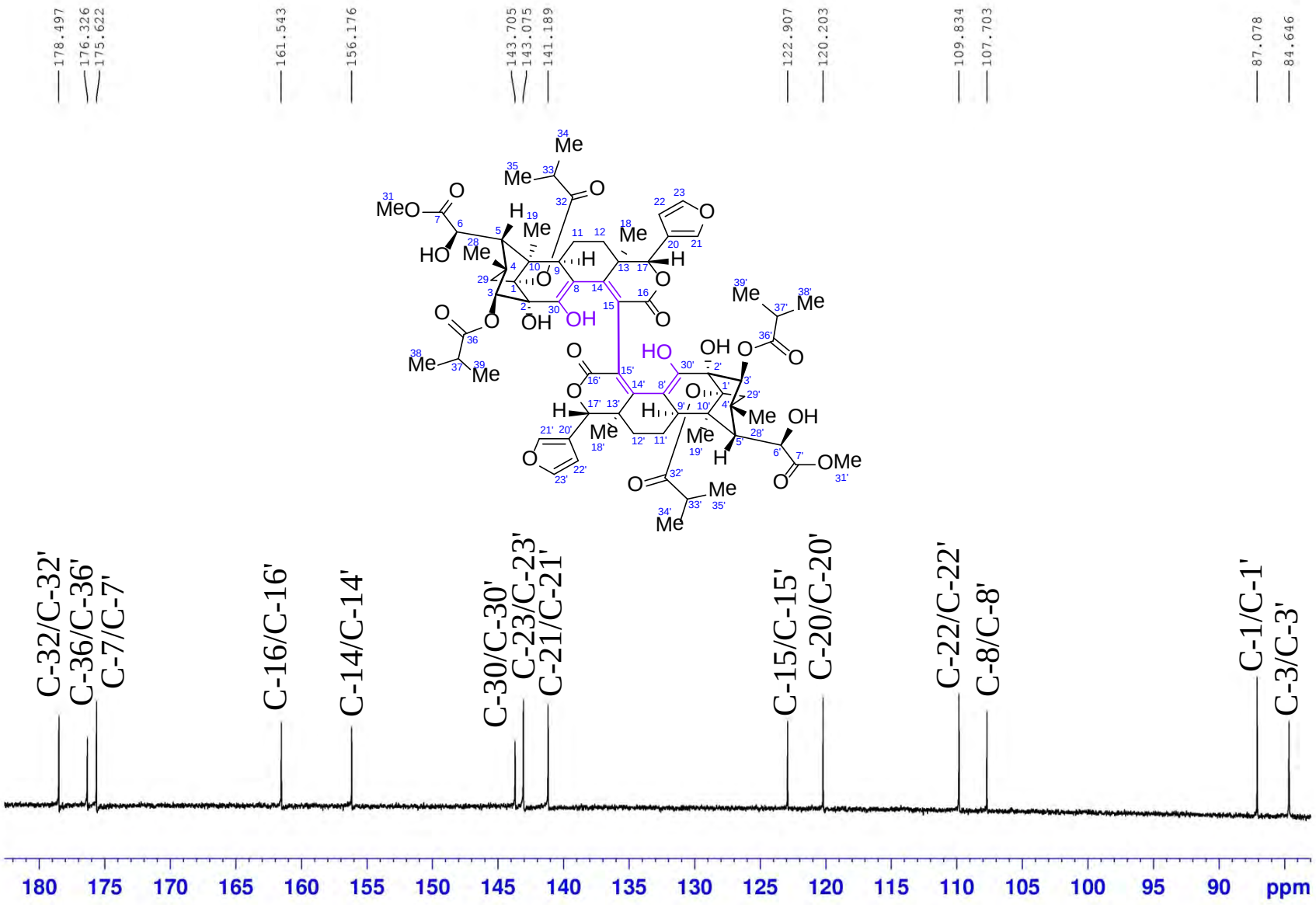


^{13}C NMR (100 MHz) spectrum of compound **5b** in $CDCl_3$

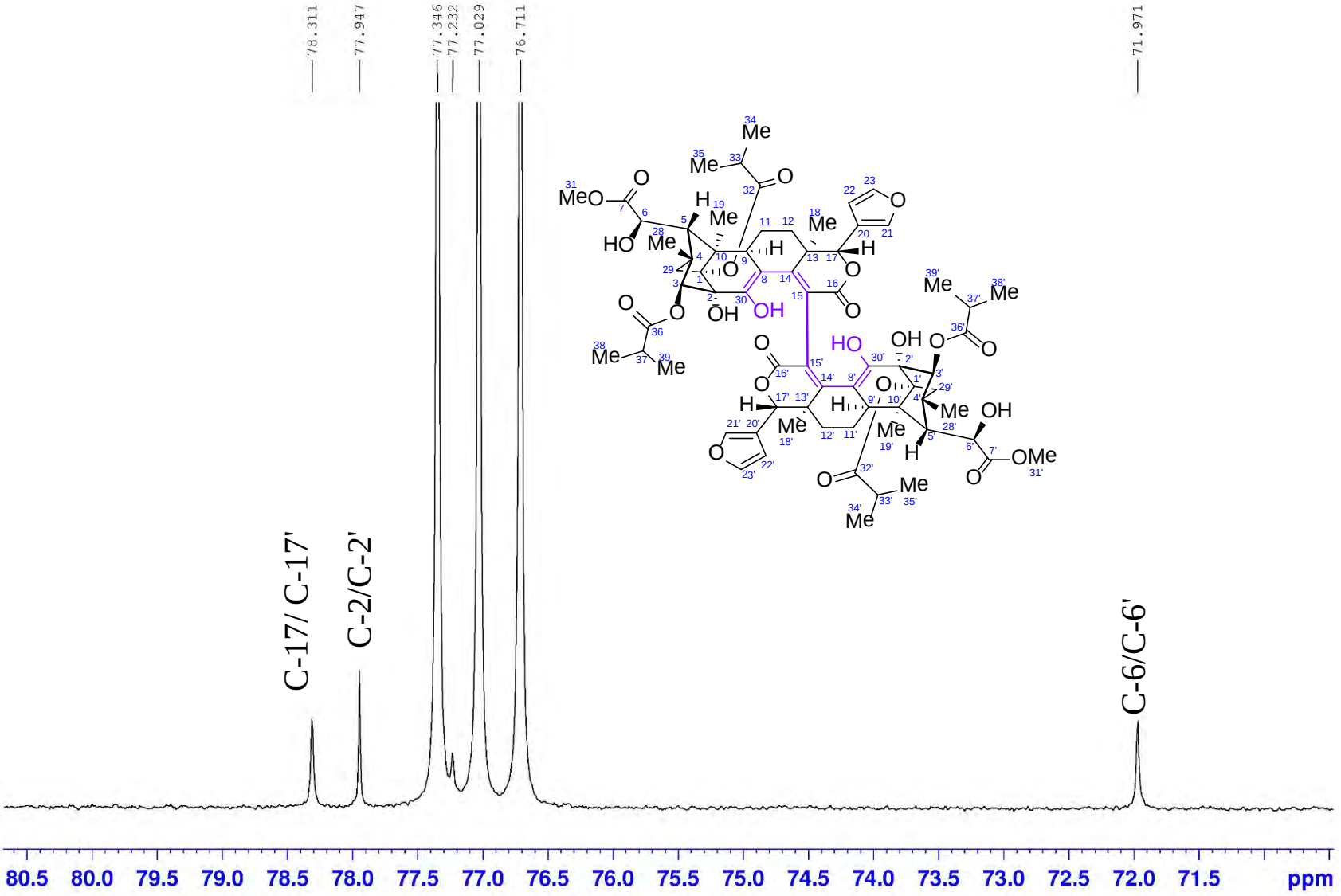


NAME	lws-42-7-fd
EXPNO	2
PROCNO	1
Date_	20150822
Time	0.34
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	102.3
DW	20.800 usec
DE	18.00 usec
TE	297.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6233324 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

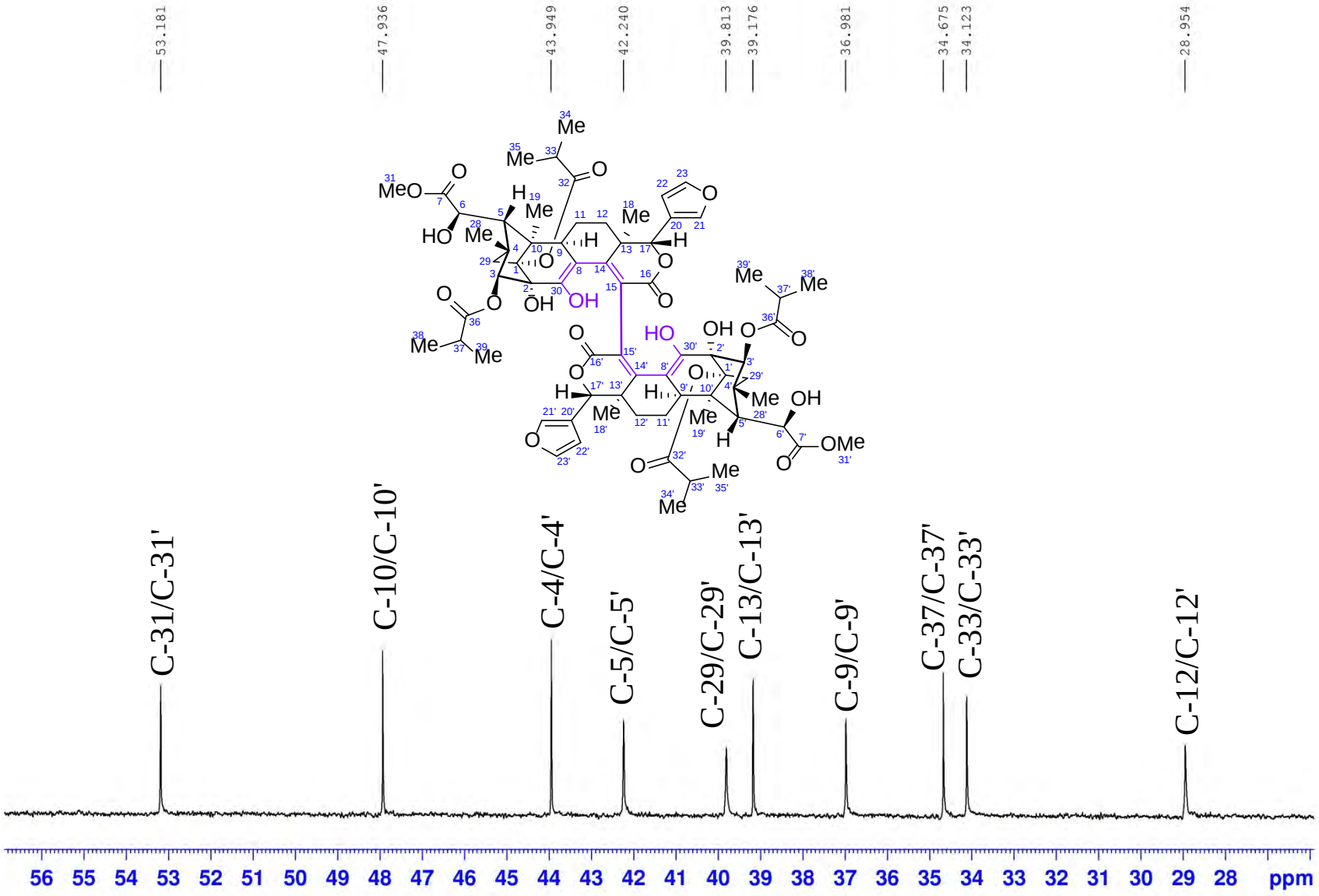
^{13}C NMR (100 MHz) spectrum of compound **5b** in $CDCl_3$



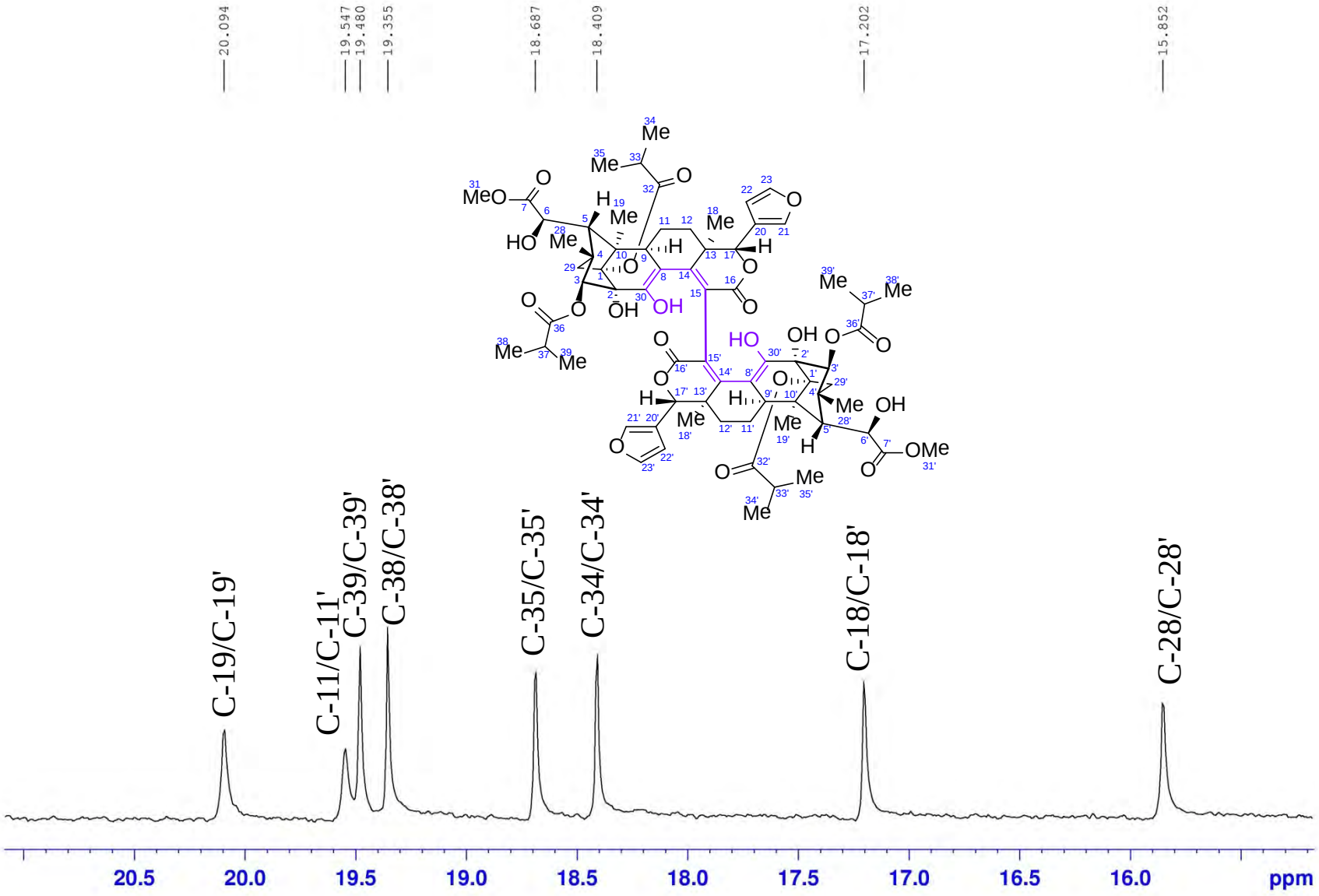
^{13}C NMR (100 MHz) spectrum of compound **5b** in $CDCl_3$



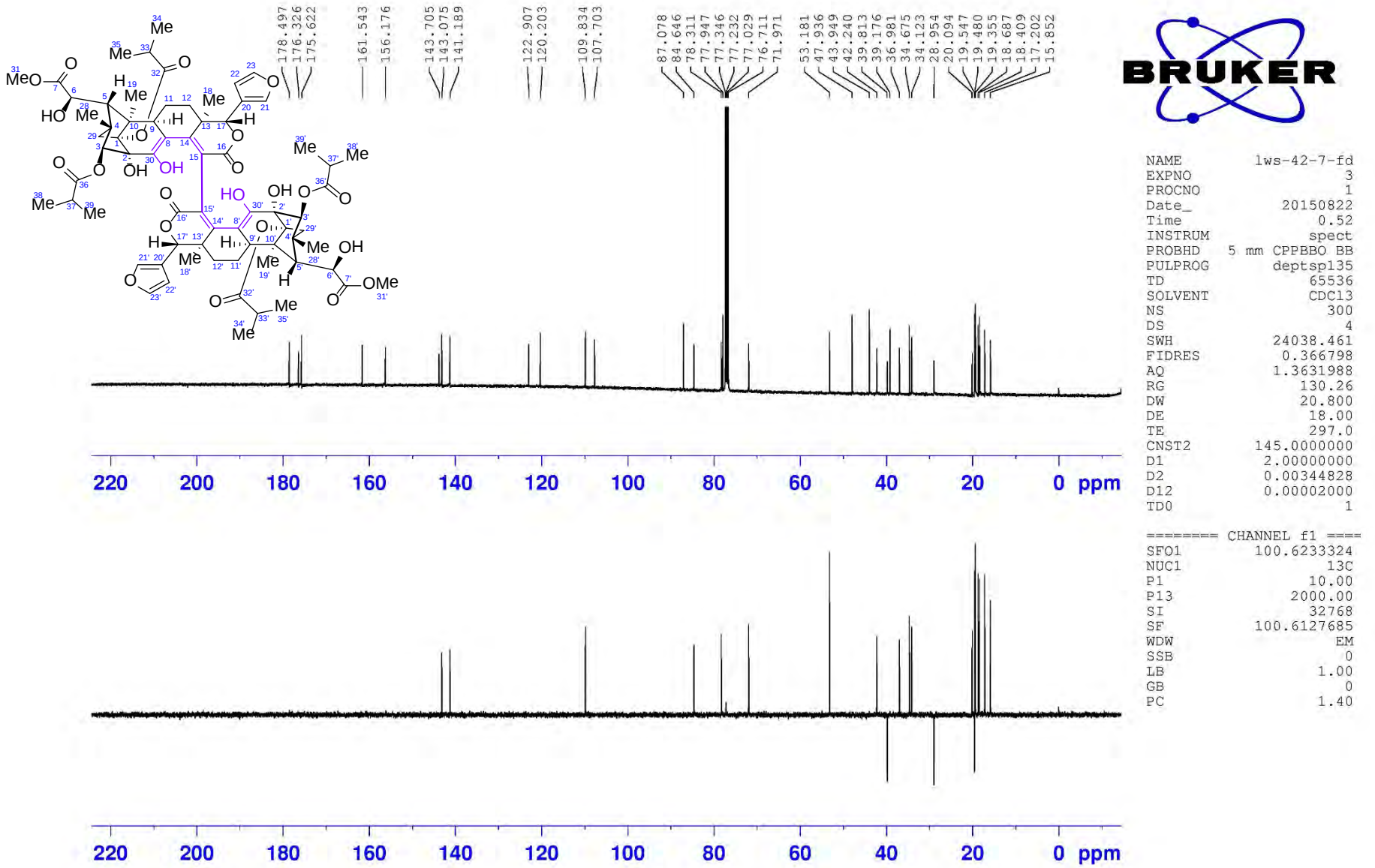
^{13}C NMR (100 MHz) spectrum of compound **5b** in $CDCl_3$



^{13}C NMR (100 MHz) spectrum of compound **5b** in $CDCl_3$



DEPT135 (100 MHz) spectrum of compound **5b** in $CDCl_3$

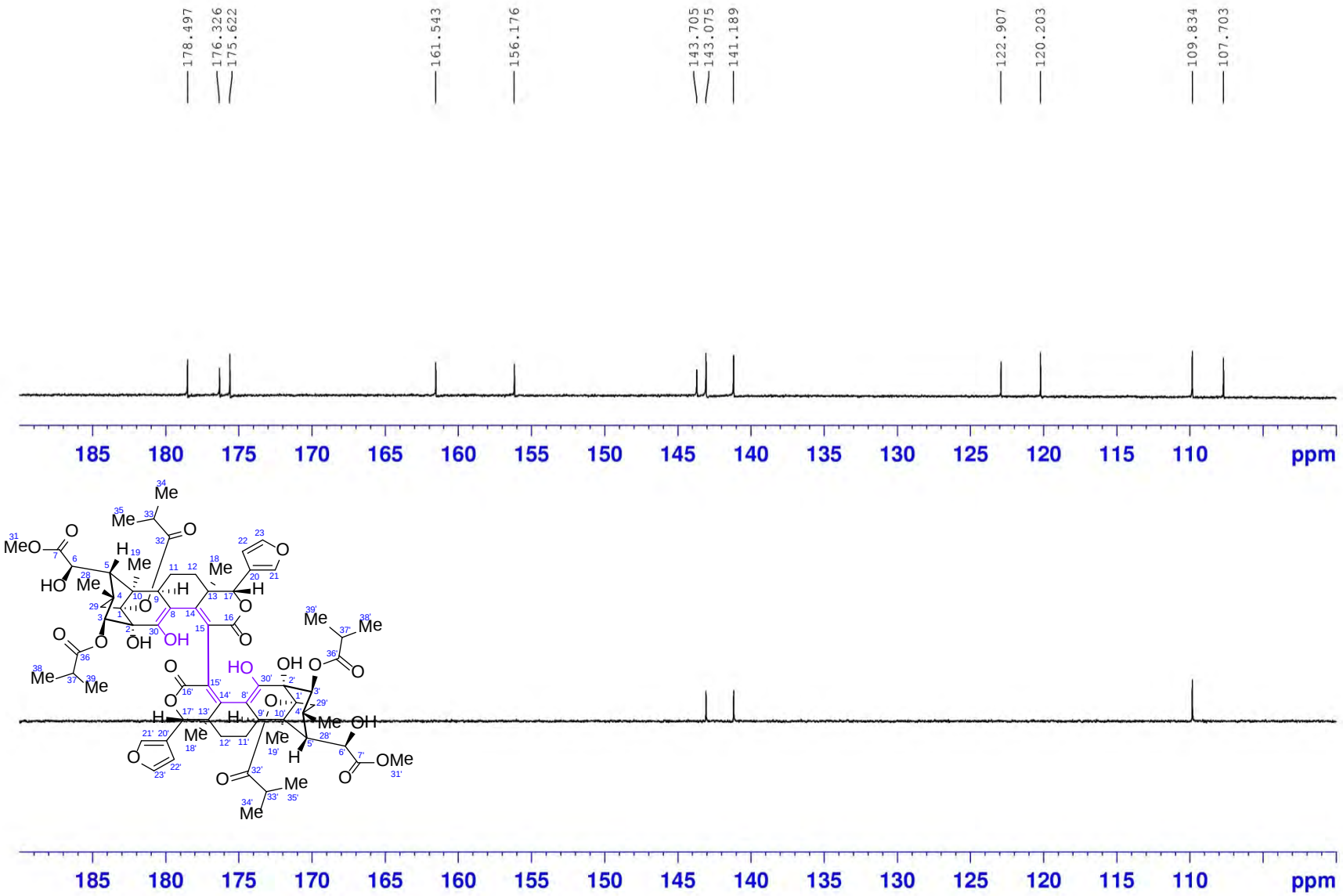


BRUKER

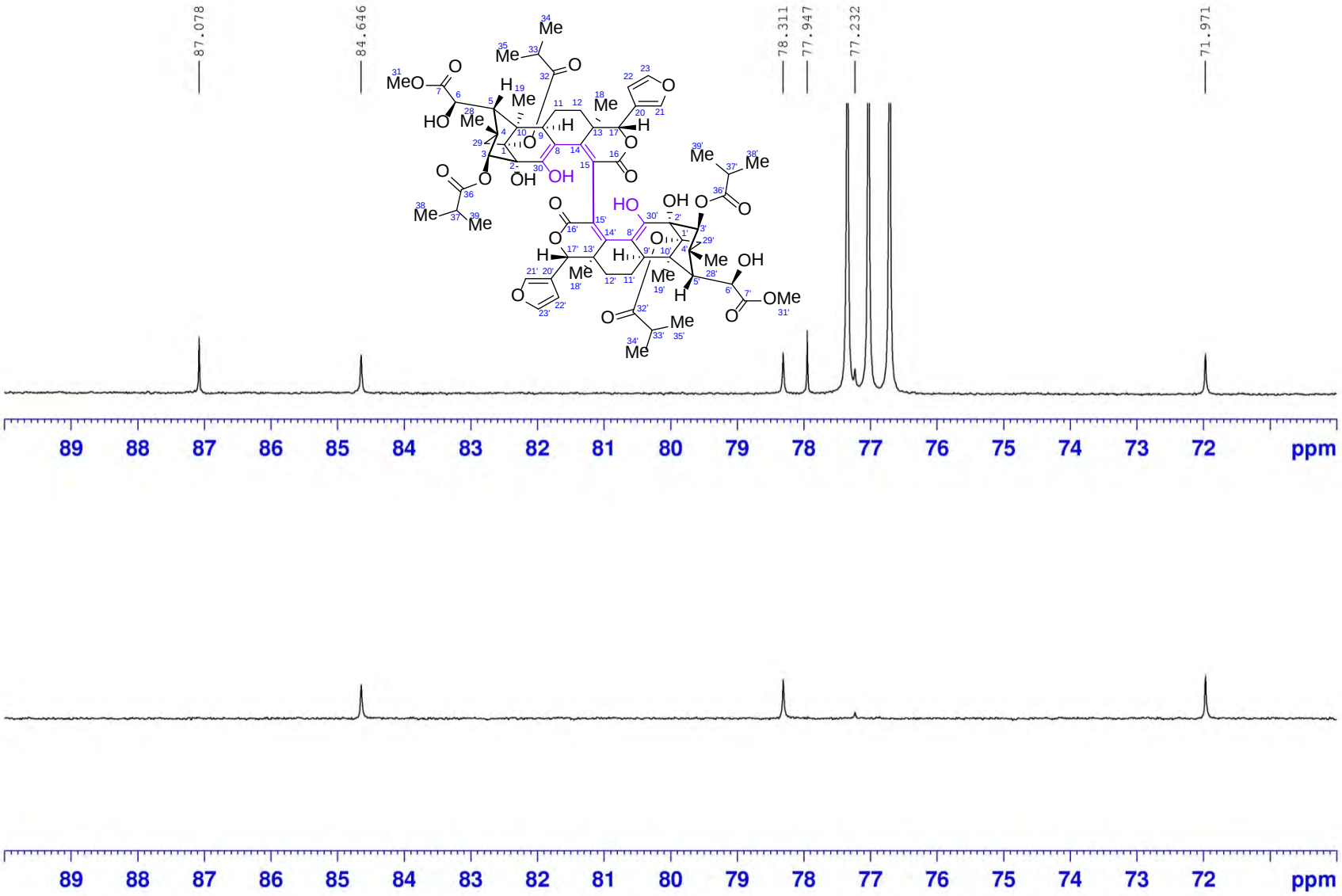
NAME	lws-42-7-fd
EXPNO	3
PROCNO	1
Date_	20150822
Time	0.52
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	deptsp135
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	24038.461
FIDRES	0.366798
AQ	1.3631988
RG	130.26
DW	20.800
DE	18.00
TE	297.0
CNST2	145.0000000
D1	2.00000000
D2	0.00344828
D12	0.00002000
TD0	1

======== CHANNEL f1 ====	
SFO1	100.6233324
NUC1	13C
P1	10.00
P13	2000.00
SI	32768
SF	100.6127685
WDW	EM
SSB	0
LB	1.00
GB	0
PC	1.40

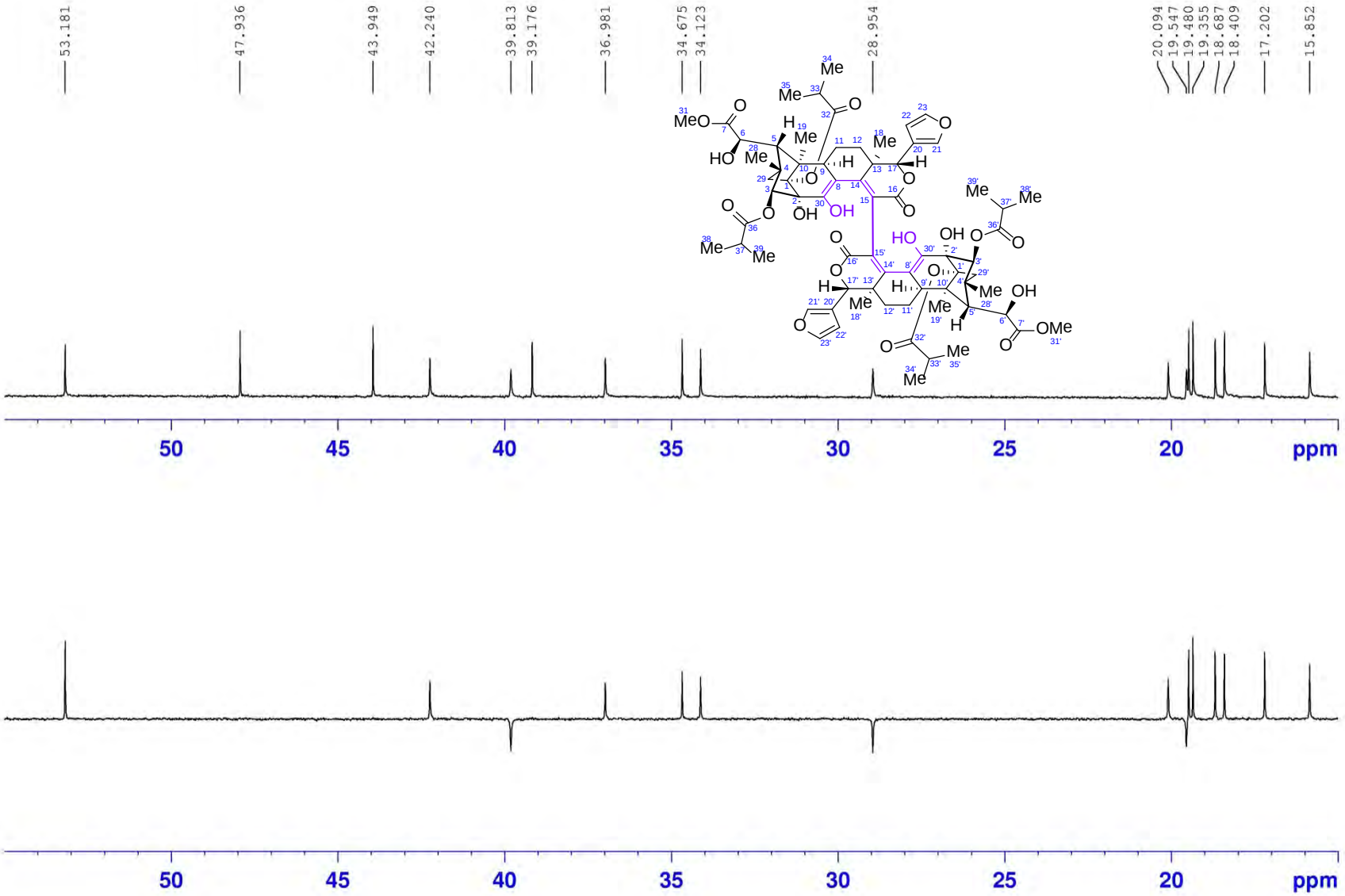
DEPT135 (100 MHz) spectrum of compound **5b** in $CDCl_3$



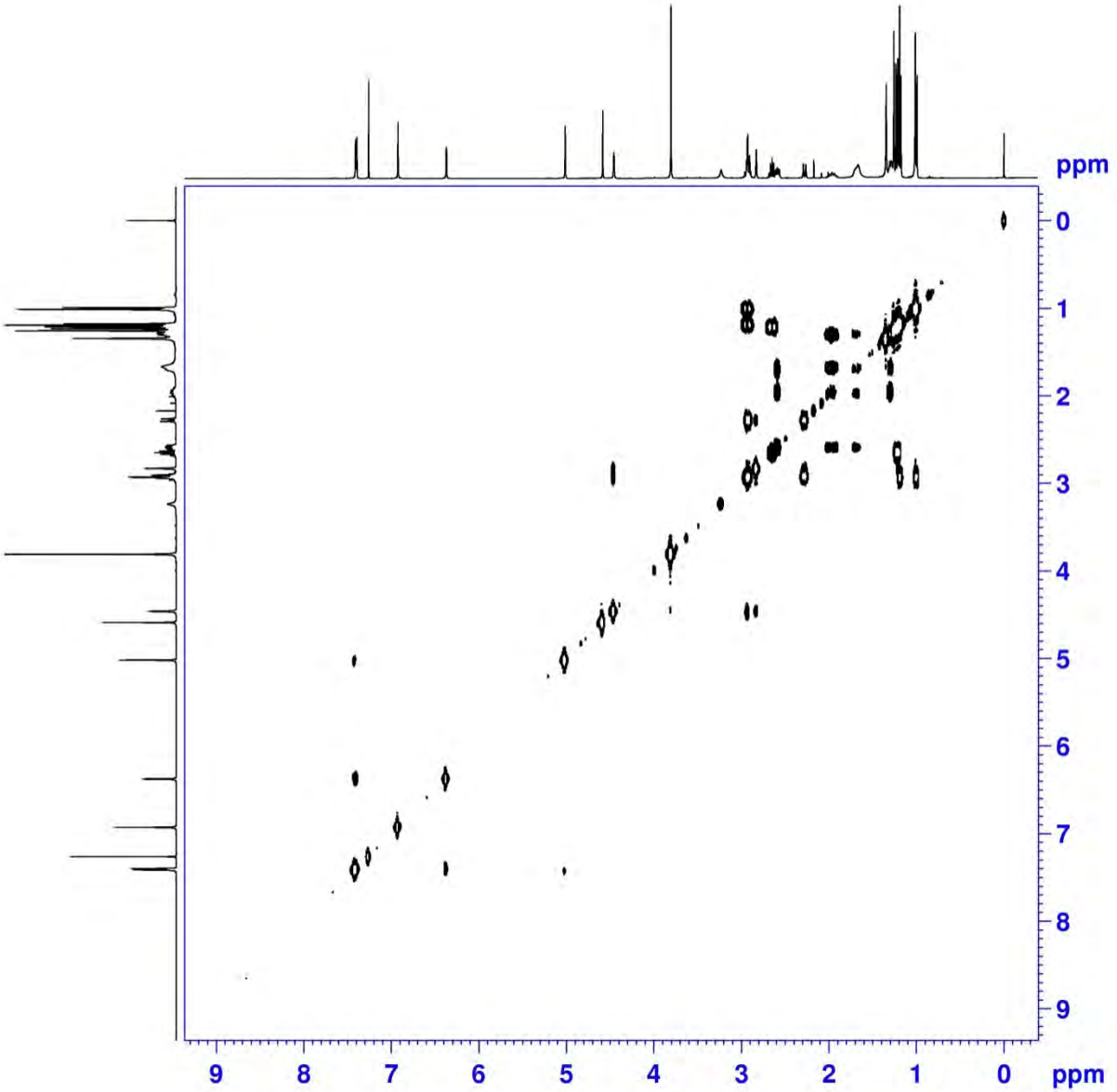
DEPT135 (100 MHz) spectrum of compound **5b** in $CDCl_3$



DEPT135 (100 MHz) spectrum of compound **5b** in $CDCl_3$



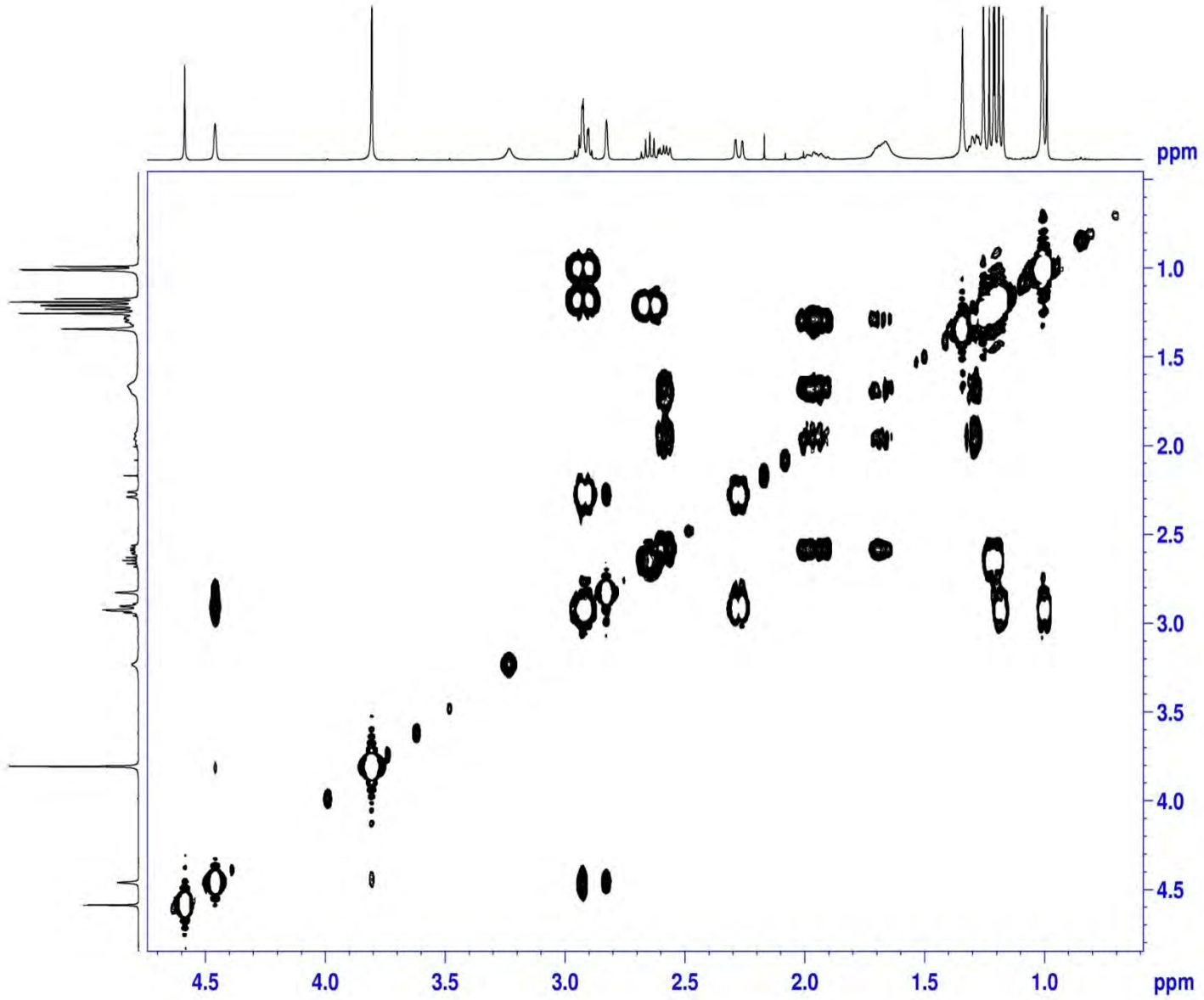
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5b** in $CDCl_3$



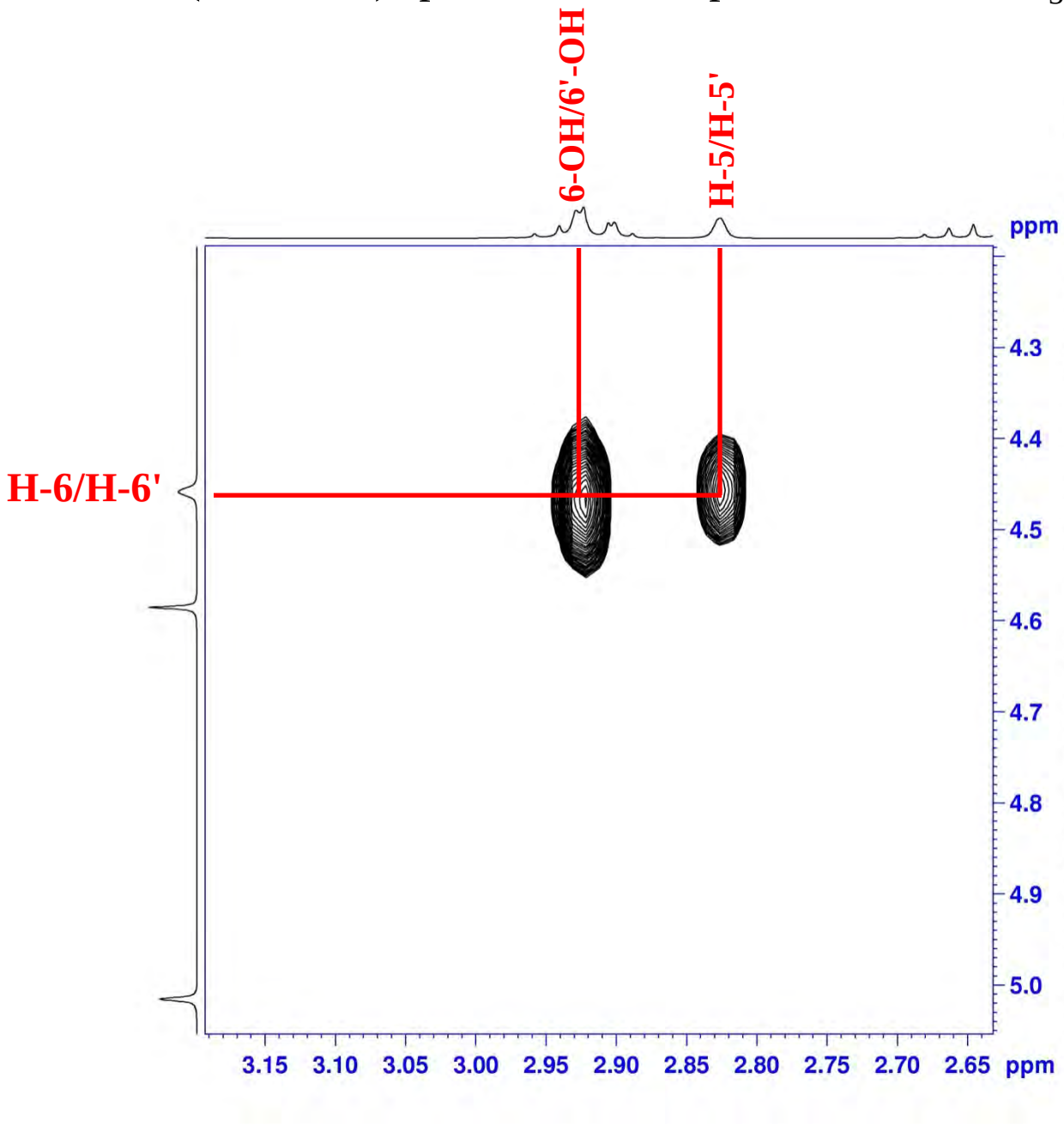
BRUKER

NAME	lws-42-7-fd
EXPNO	4
PROCNO	1
Date_	20150822
Time	0.54
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	8
DS	8
SWH	3906.250 Hz
FIDRES	1.907349 Hz
AQ	0.2621940 sec
RG	208.5
DW	128.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00000300 sec
D1	1.89678097 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00025600 sec
========	CHANNEL f1 ========
SFO1	400.1318006 MHz
NUC1	1H
P0	12.00 usec
P1	12.00 usec
P17	2500.00 usec
ND0	1
TD	128
SFO1	400.1318 MHz
FIDRES	30.517578 Hz
SW	9.762 ppm
FnMODE	QF
SI	1024
SF	400.1300080 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	400.1300080 MHz
WDW	OSINE

^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5b** in $CDCl_3$



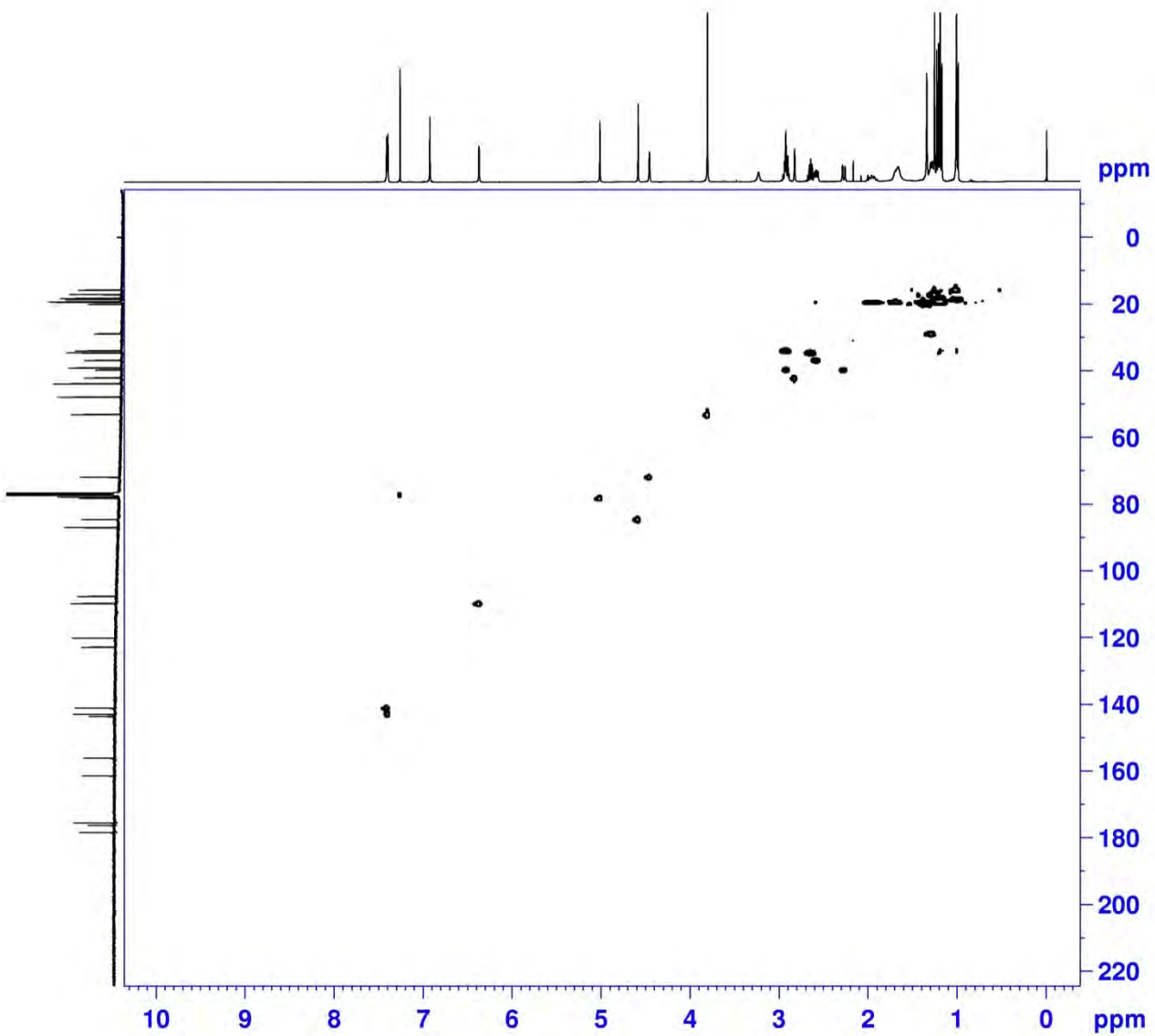
^{1}H-^{1}H COSY (400 MHz) spectrum of compound **5b** in $CDCl_3$



BRUKER

NAME	lws-42-7-fd
EXPNO	4
PROCNO	1
Date_	20150822
Time	0.54
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	cosygpppqf
TD	2048
SOLVENT	CDCl3
NS	8
DS	8
SWH	3906.250 Hz
FIDRES	1.907349 Hz
AQ	0.2621940 sec
RG	208.5
DW	128.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00000300 sec
D1	1.89678097 sec
D11	0.03000000 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00025600 sec
======== CHANNEL f1 ========	
SFO1	400.1318006 MHz
NUC1	1H
P0	12.00 usec
P1	12.00 usec
P17	2500.00 usec
ND0	1
TD	128
SFO1	400.1318 MHz
FIDRES	30.517578 Hz
SW	9.762 ppm
FnMODE	QF
SI	1024
SF	400.1300080 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	400.1300080 MHz
WDW	QSINE
SSB	0
LB	0.00 Hz
GB	0

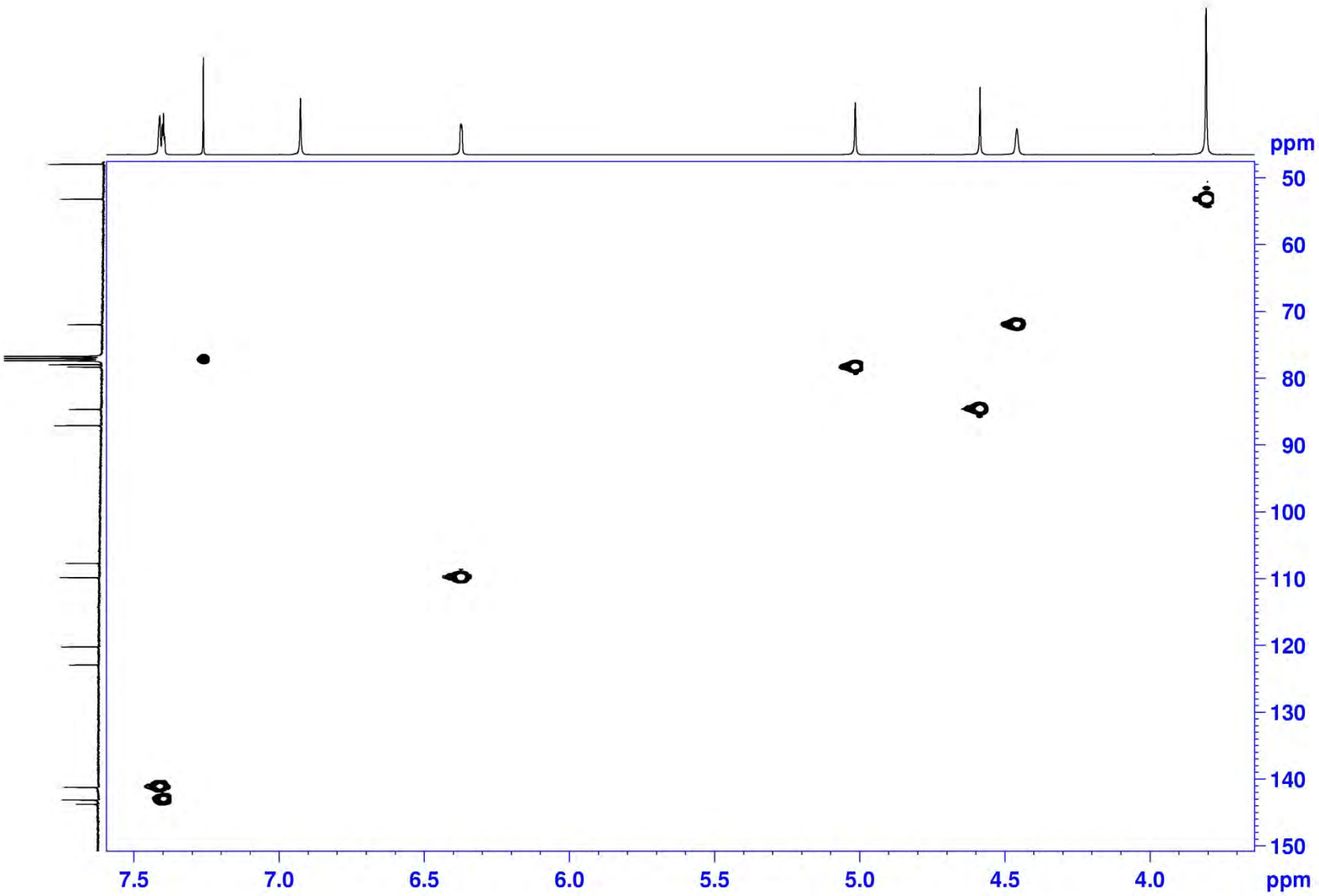
HSQC (400 MHz) spectrum of compound **5b** in $CDCl_3$



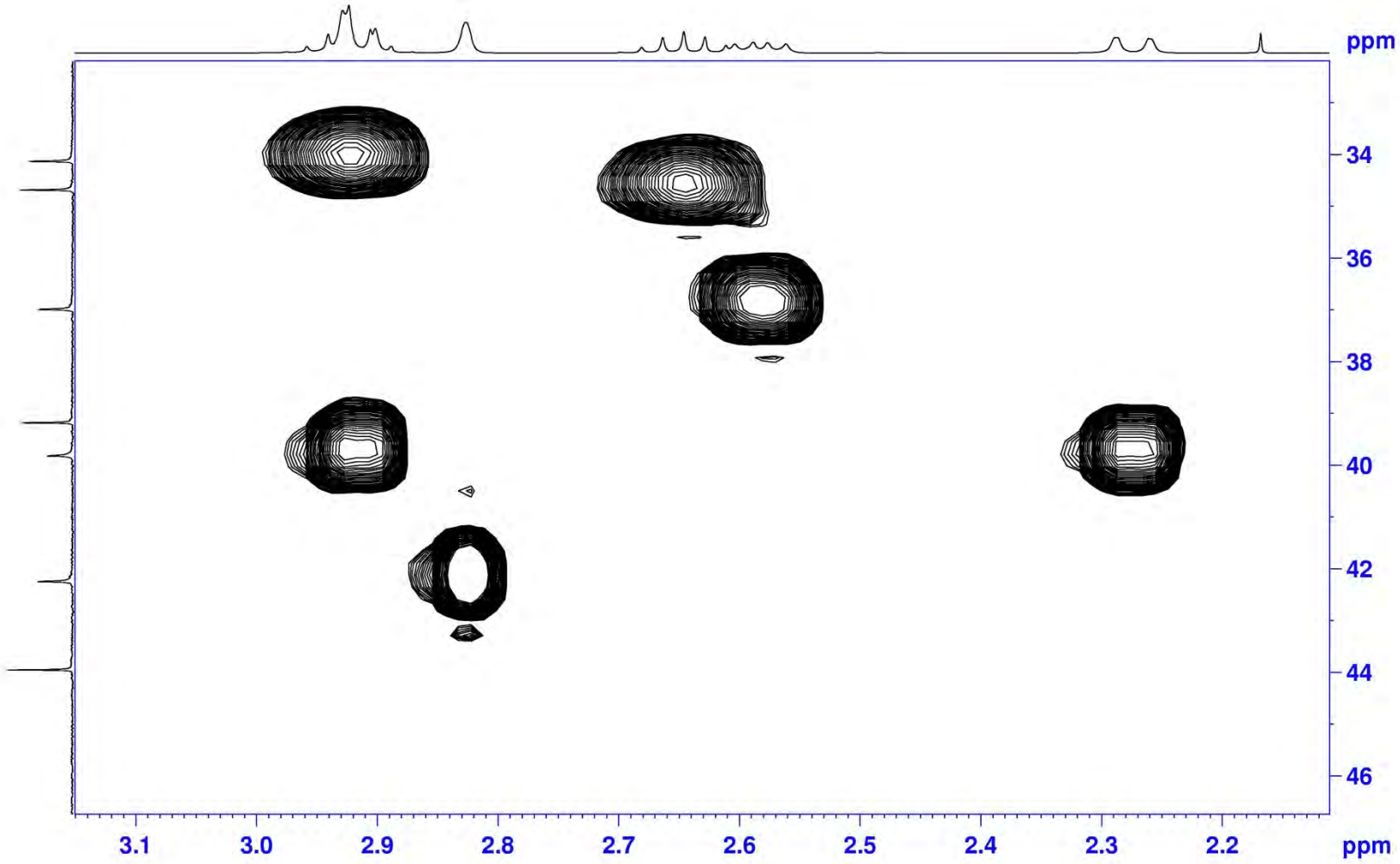
BRUKER

NAME	lws-42-7-fd
EXPNO	5
PROCNO	1
Date_	20150822
Time	1.33
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	hsqcetgpsi2
TD	1024
SOLVENT	CDCl3
NS	16
DS	16
SWH	4302.926 Hz
FIDRES	4.202076 Hz
AQ	0.1190388 sec
RG	208.5
DW	116.200 usec
DE	10.00 usec
TE	297.0 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.46497905 sec
D4	0.00172414 sec
D11	0.03000000 sec
D16	0.00020000 sec
D24	0.00086207 sec
IN0	0.00002080 sec
ZGOPTNS	
========	CHANNEL f1 ========
SFO1	400.1320007 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
P28	0.00 usec
ND0	2
TD	256
SFO1	100.6233 MHz
FIDRES	93.900238 Hz
SW	238.896 ppm
FnMODE	Echo-Antiecho
SI	1024
SF	400.1300079 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	echo-antiecho
SF	100.6127685 MHz

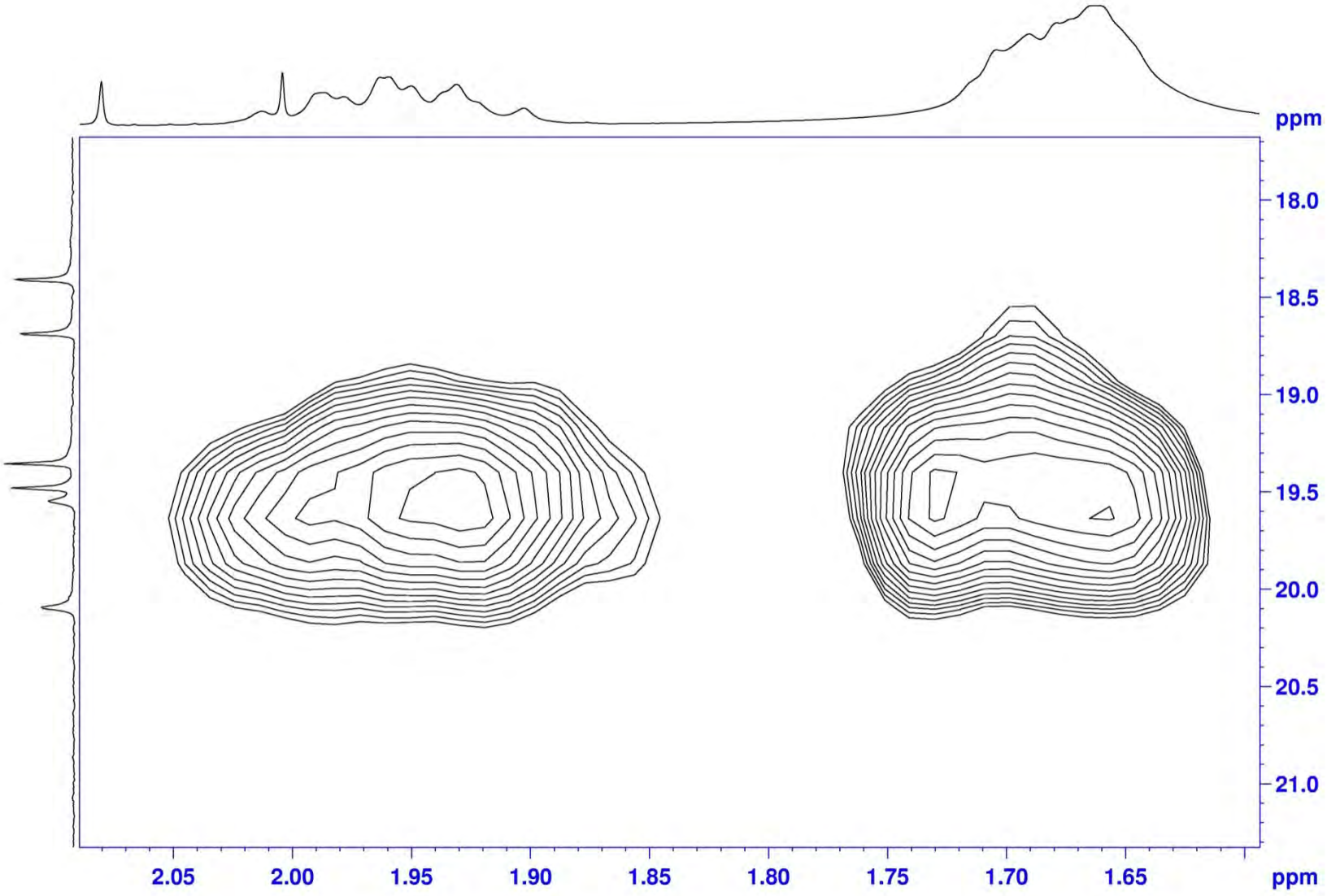
HSQC (400 MHz) spectrum of compound **5b** in $CDCl_3$



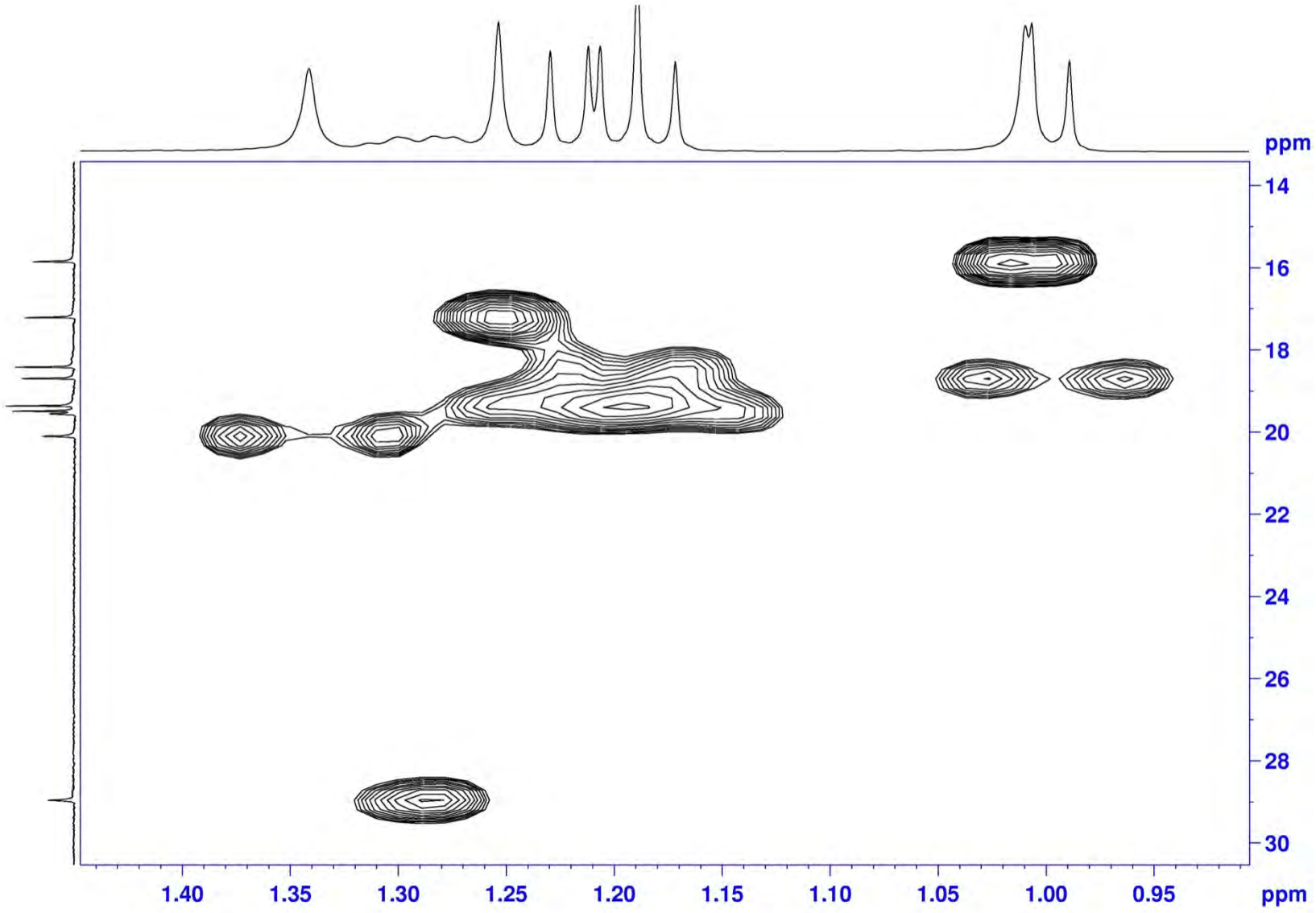
HSQC (400 MHz) spectrum of compound **5b** in $CDCl_3$



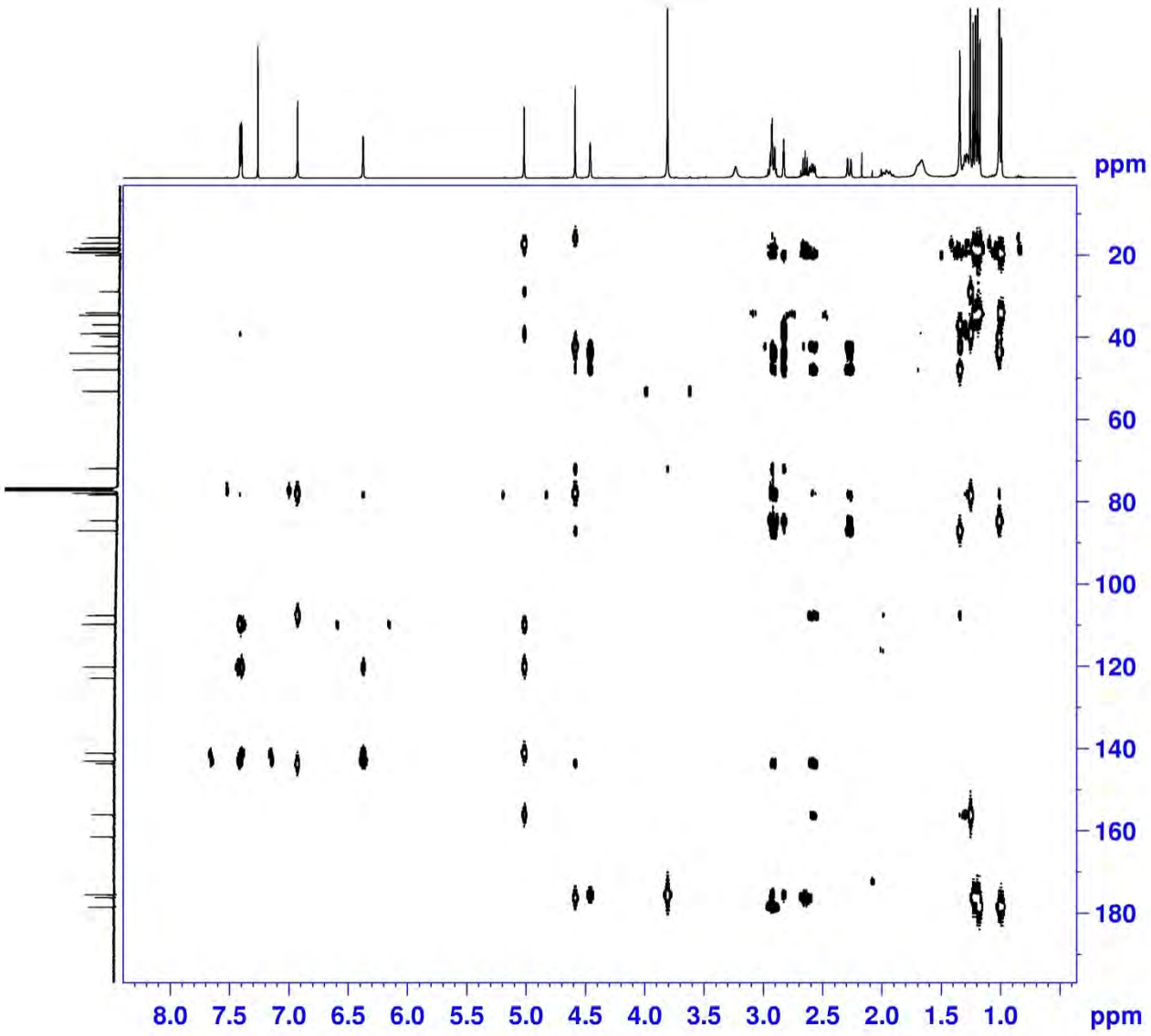
HSQC (400 MHz) spectrum of compound **5b** in $CDCl_3$



HSQC (400 MHz) spectrum of compound **5b** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$

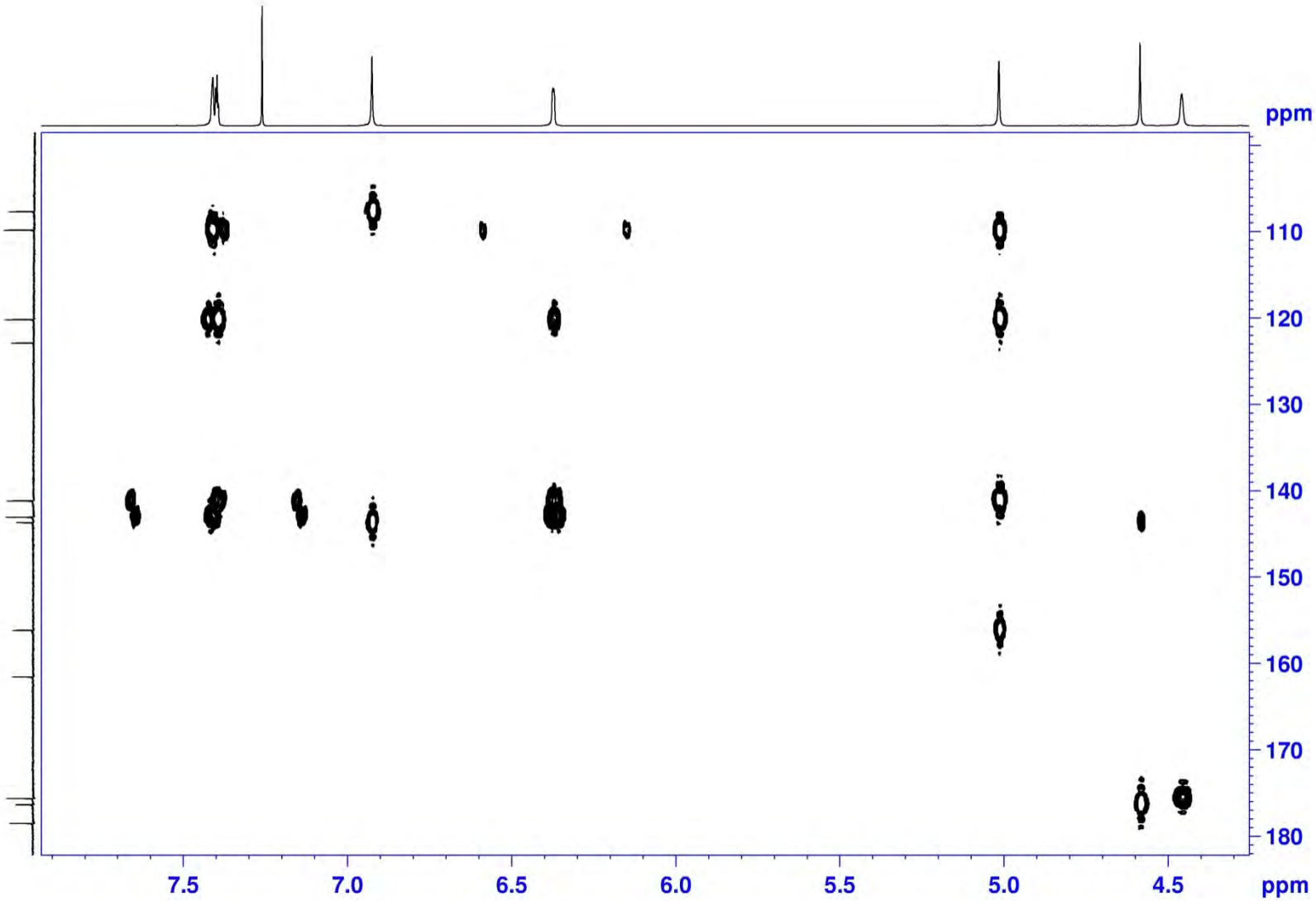


BRUKER

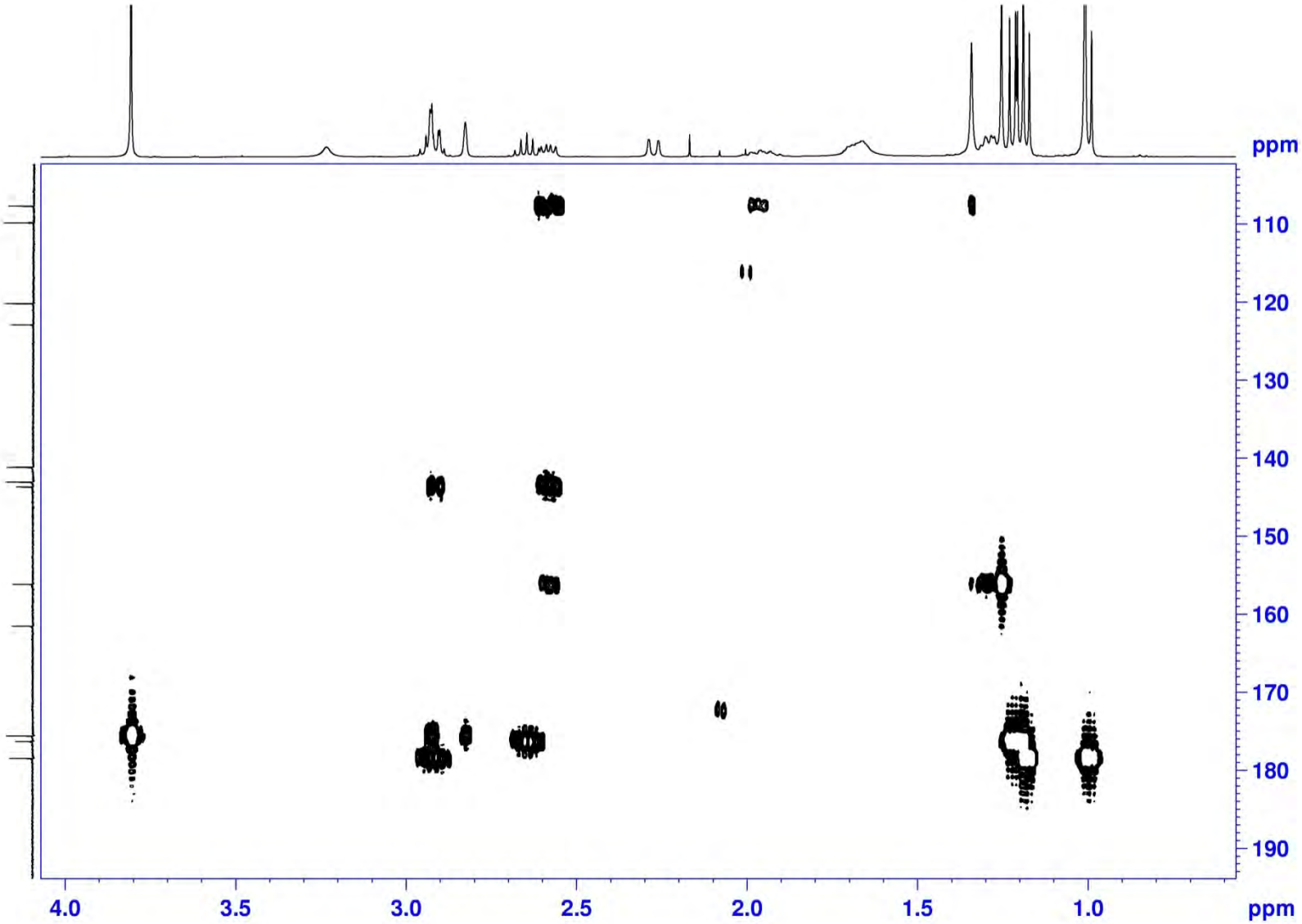
NAME	lws-42-7-fd
EXPNO	6
PROCNO	1
Date_	20150822
Time	3.25
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	hmbcgplpndqf
TD	4096
SOLVENT	CDCl3
NS	32
DS	16
SWH	5197.505 Hz
FIDRES	1.268922 Hz
AQ	0.3940852 sec
RG	208.5
DW	96.200 usec
DE	10.00 usec
TE	297.0 K
CNST2	145.0000000
CNST13	10.0000000
D0	0.00000300 sec
D1	1.50000000 sec
D2	0.00344828 sec
D6	0.05000000 sec
D16	0.00020000 sec
IN0	0.00002080 sec

======== CHANNEL f1 ========	
SFO1	400.1323208 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
ND0	2
TD	128
SFO1	100.6233 MHz
FIDRES	187.800476 Hz
SW	238.896 ppm
FnMODE	QF
SI	2048
SF	400.1300094 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	100.6127685 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

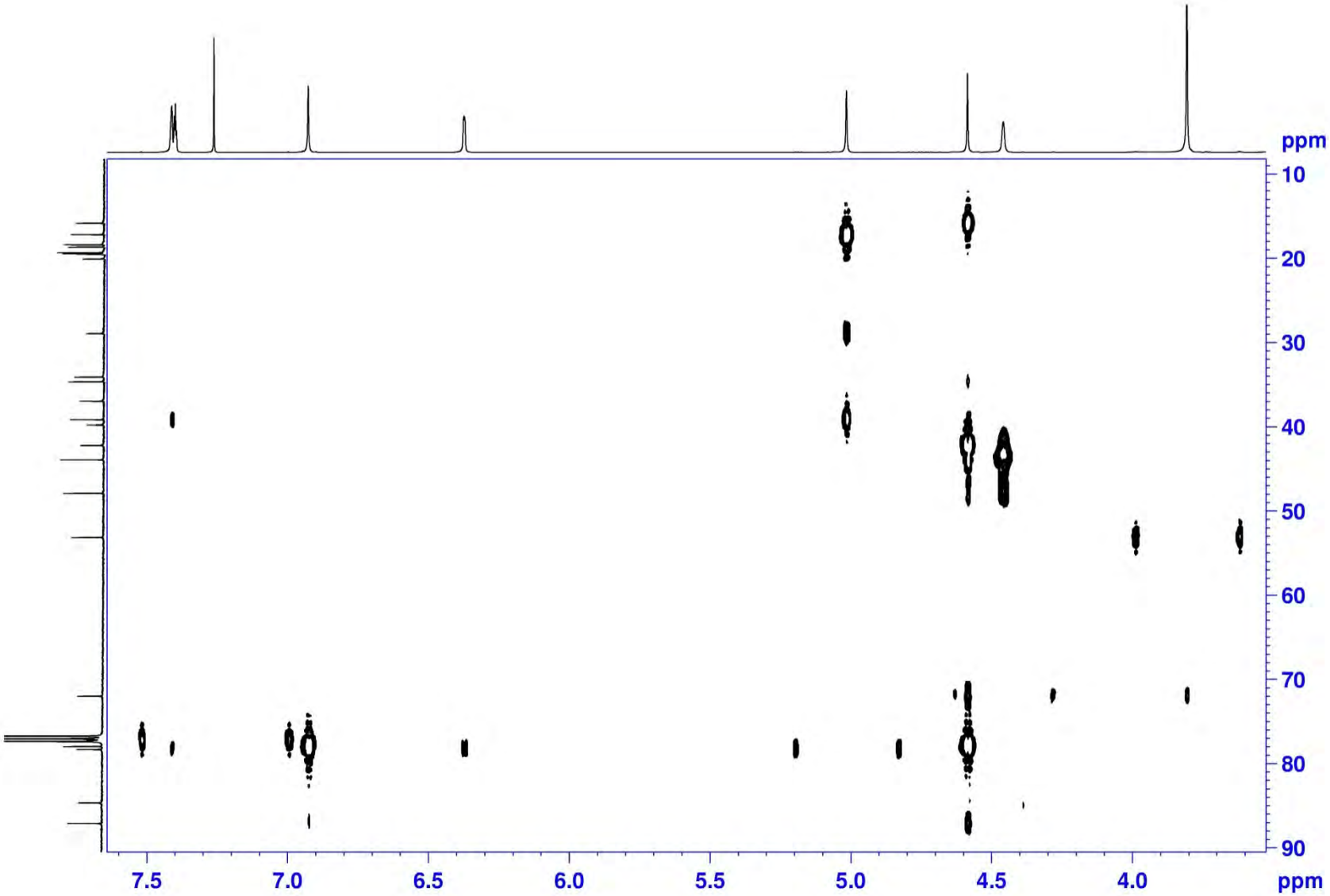
HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



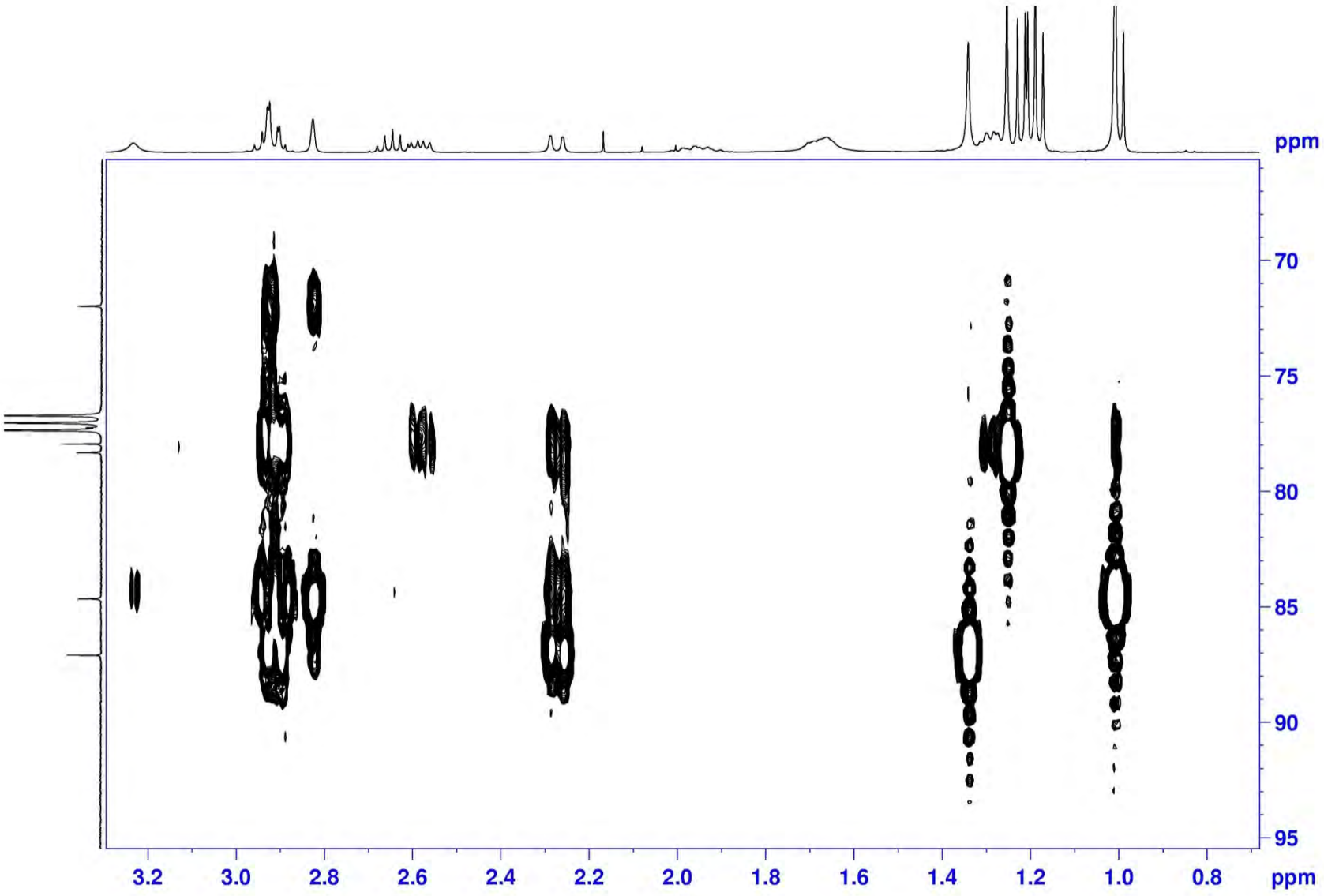
HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



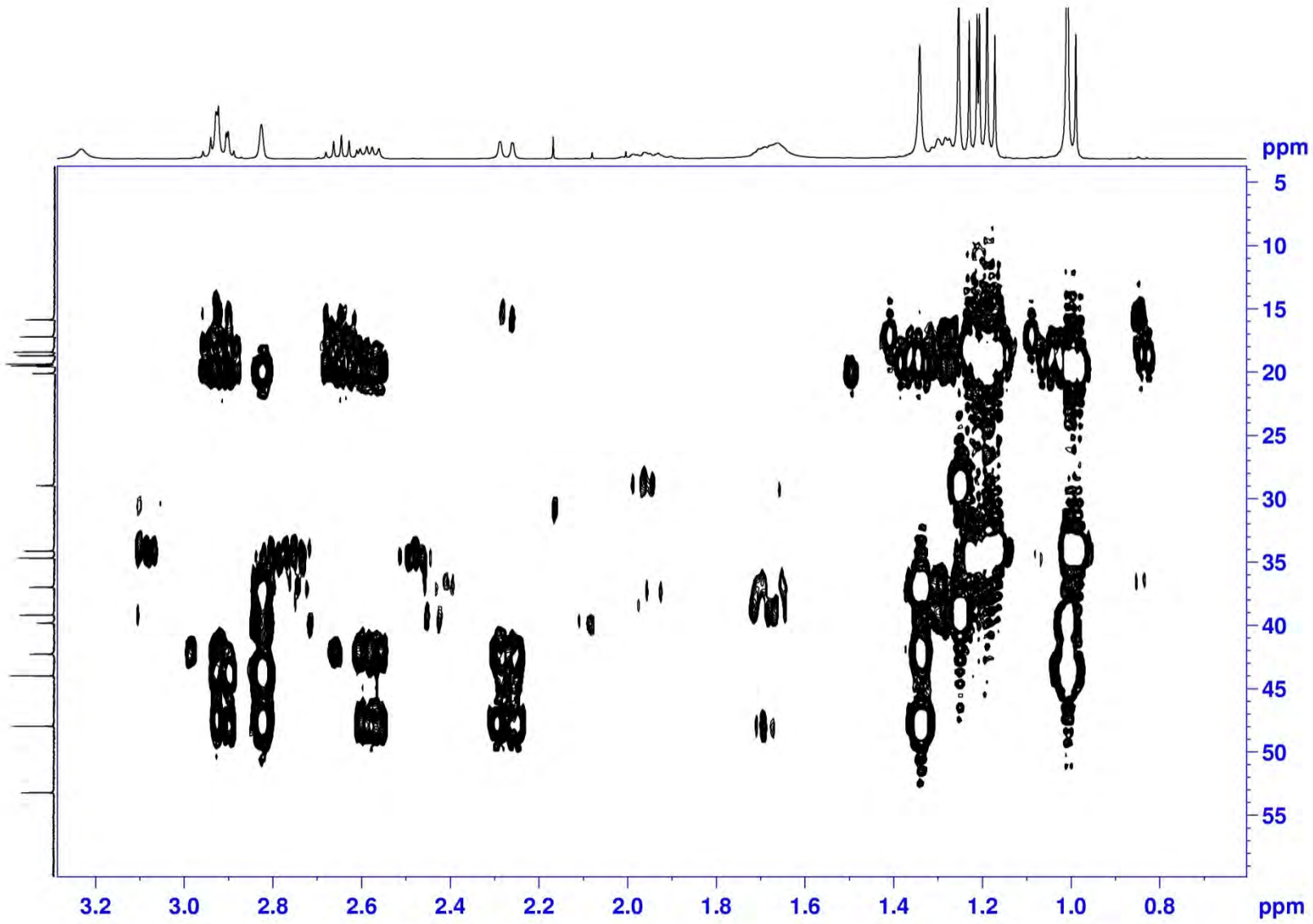
HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



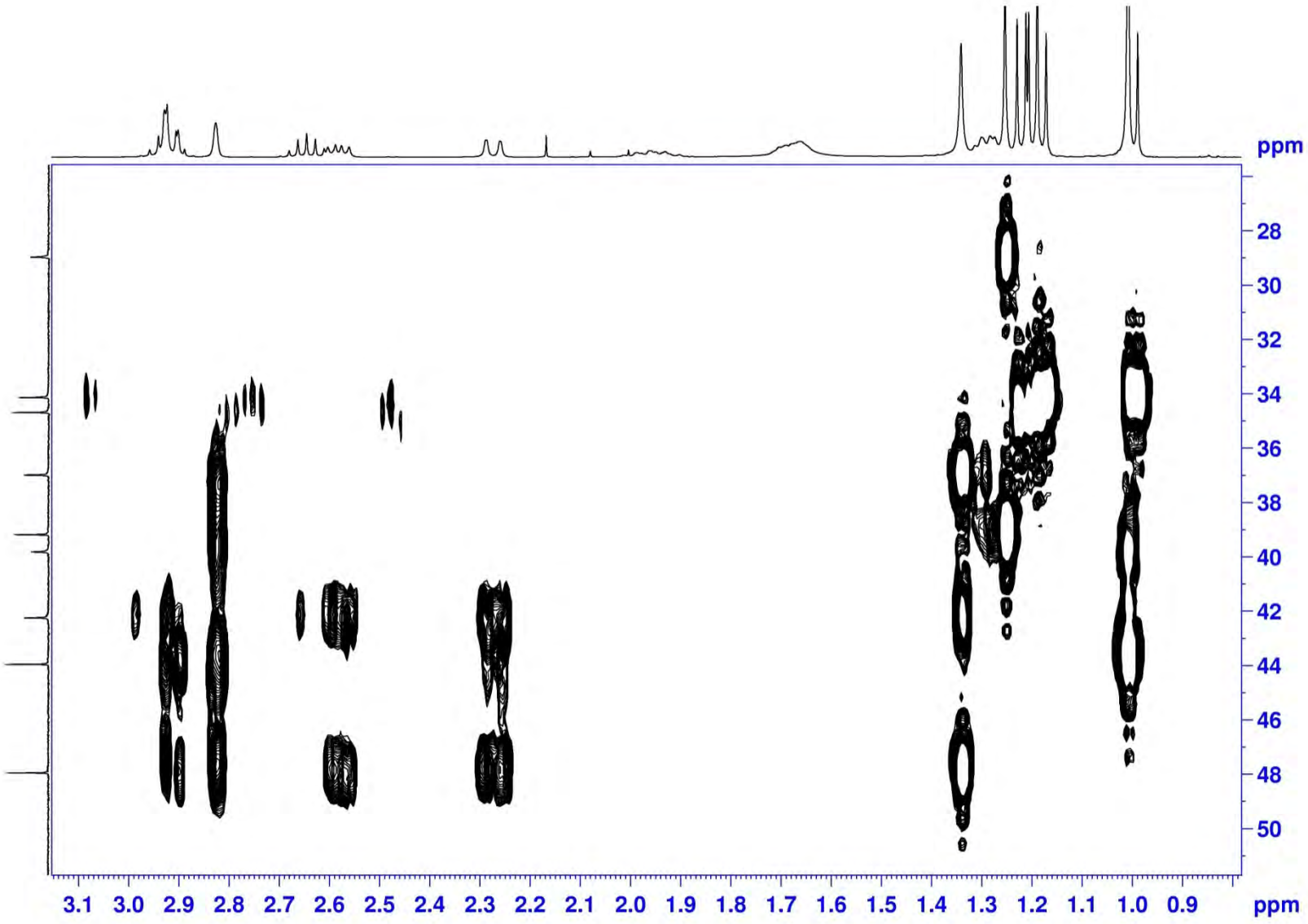
HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



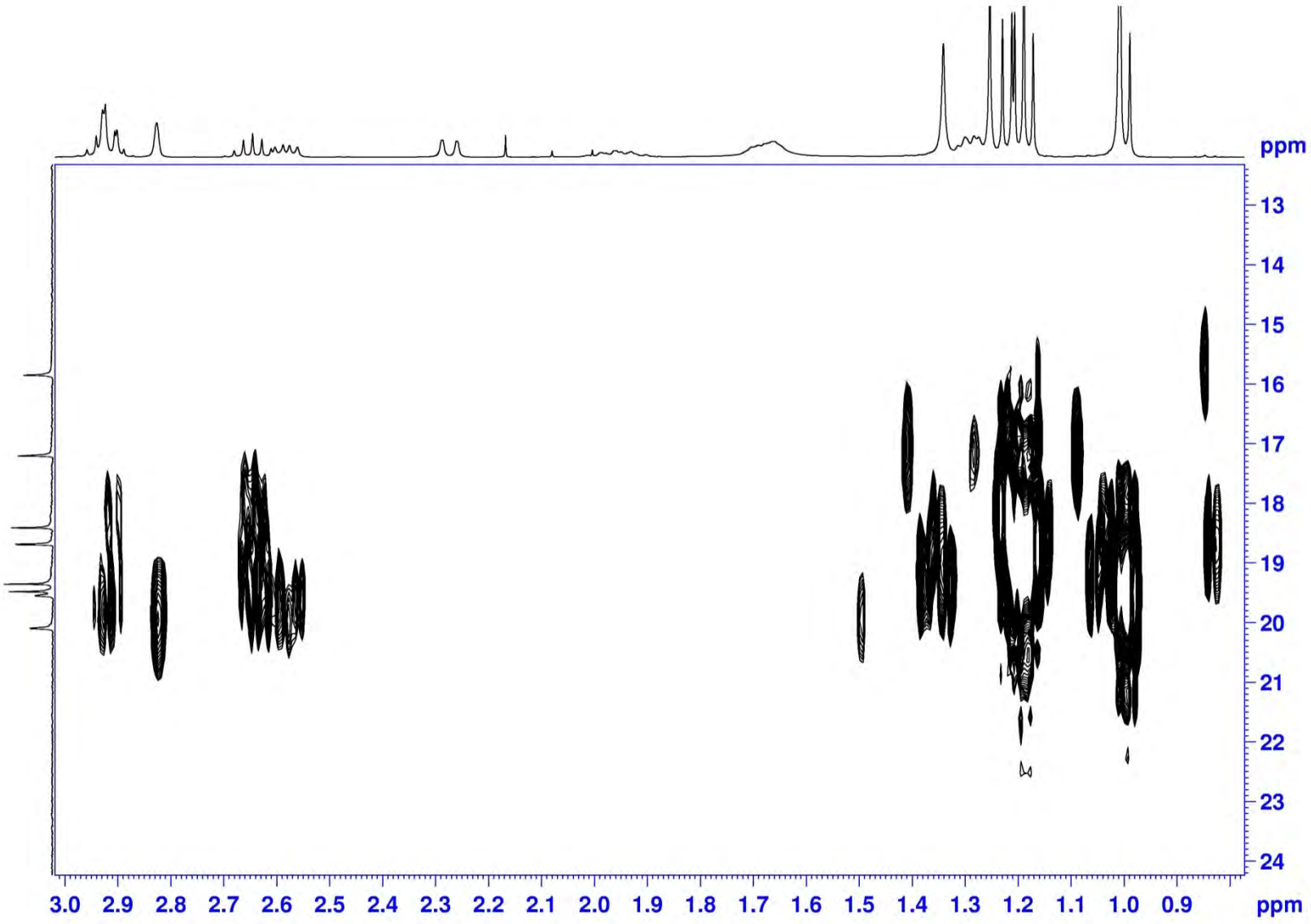
HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



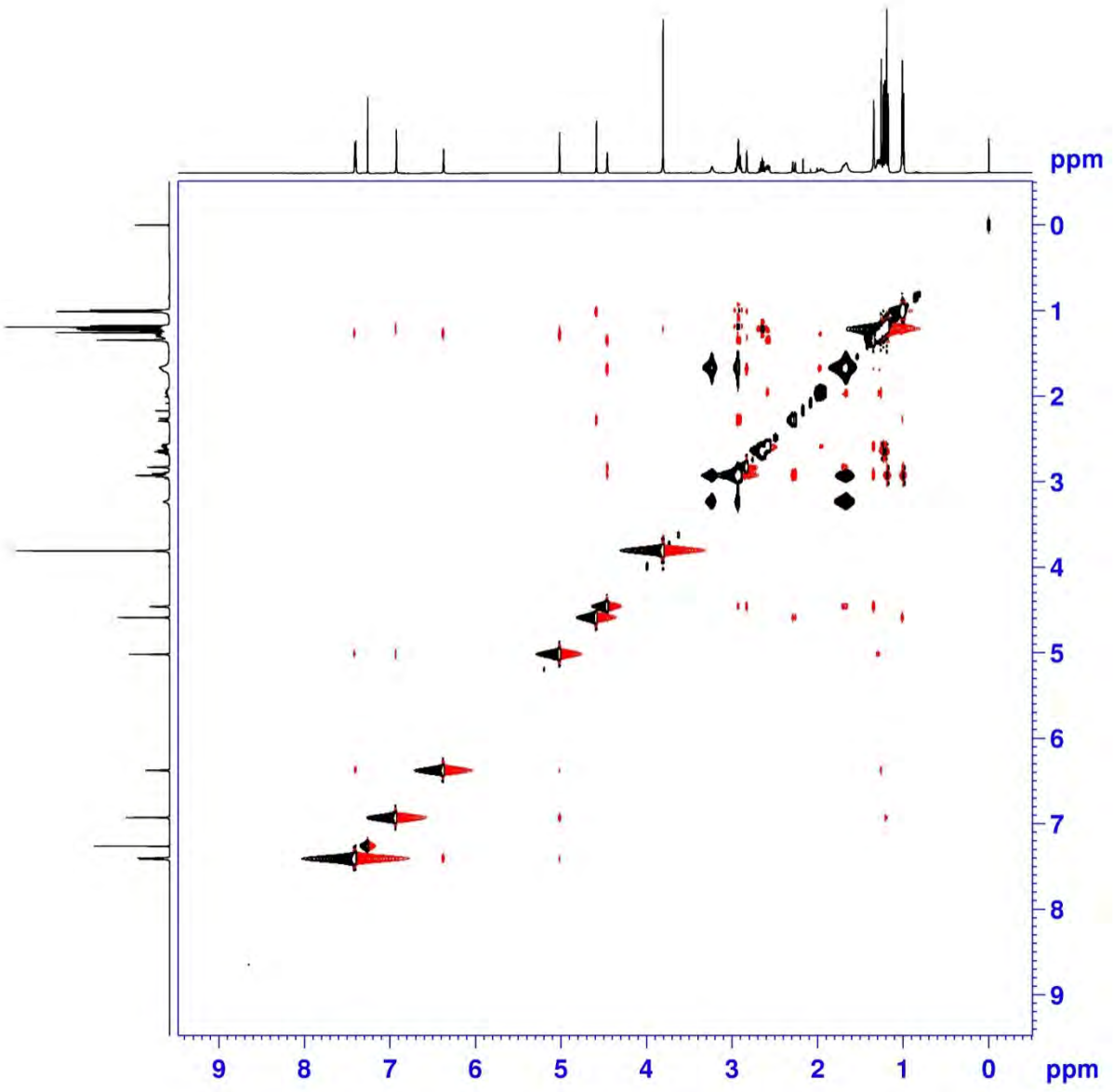
HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



HMBC (400 MHz) spectrum of compound **5b** in $CDCl_3$



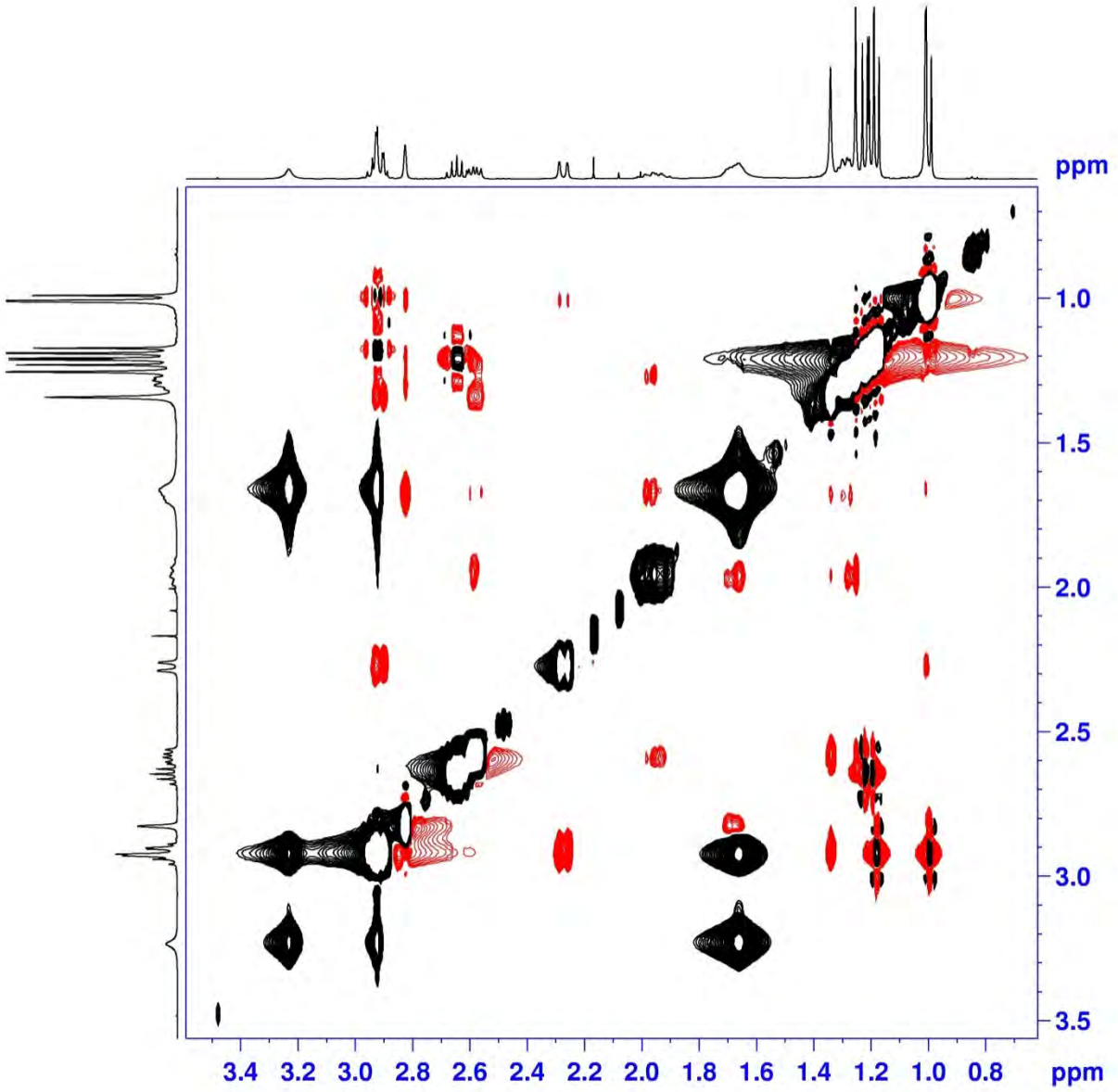
NOESY (400 MHz) spectrum of compound **5b** in $CDCl_3$



BRUKER

NAME	lws-42-7-fd
EXPNO	7
PROCNO	1
Date_	20150822
Time	5.40
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	noesygpphpp
TD	2048
SOLVENT	CDCl3
NS	16
DS	32
SWH	4000.000 Hz
FIDRES	1.953125 Hz
AQ	0.2560500 sec
RG	208.5
DW	125.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00010972 sec
D1	1.99385595 sec
D8	0.30000001 sec
D11	0.03000000 sec
D12	0.00002000 sec
D16	0.00020000 sec
IN0	0.00025000 sec
========	CHANNEL f1 ========
SFO1	400.1318006 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
P17	2500.00 usec
ND0	1
TD	256
SFO1	400.1318 MHz
FIDRES	15.625000 Hz
SW	9.997 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300094 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300094 MHz
WDW	QSINE

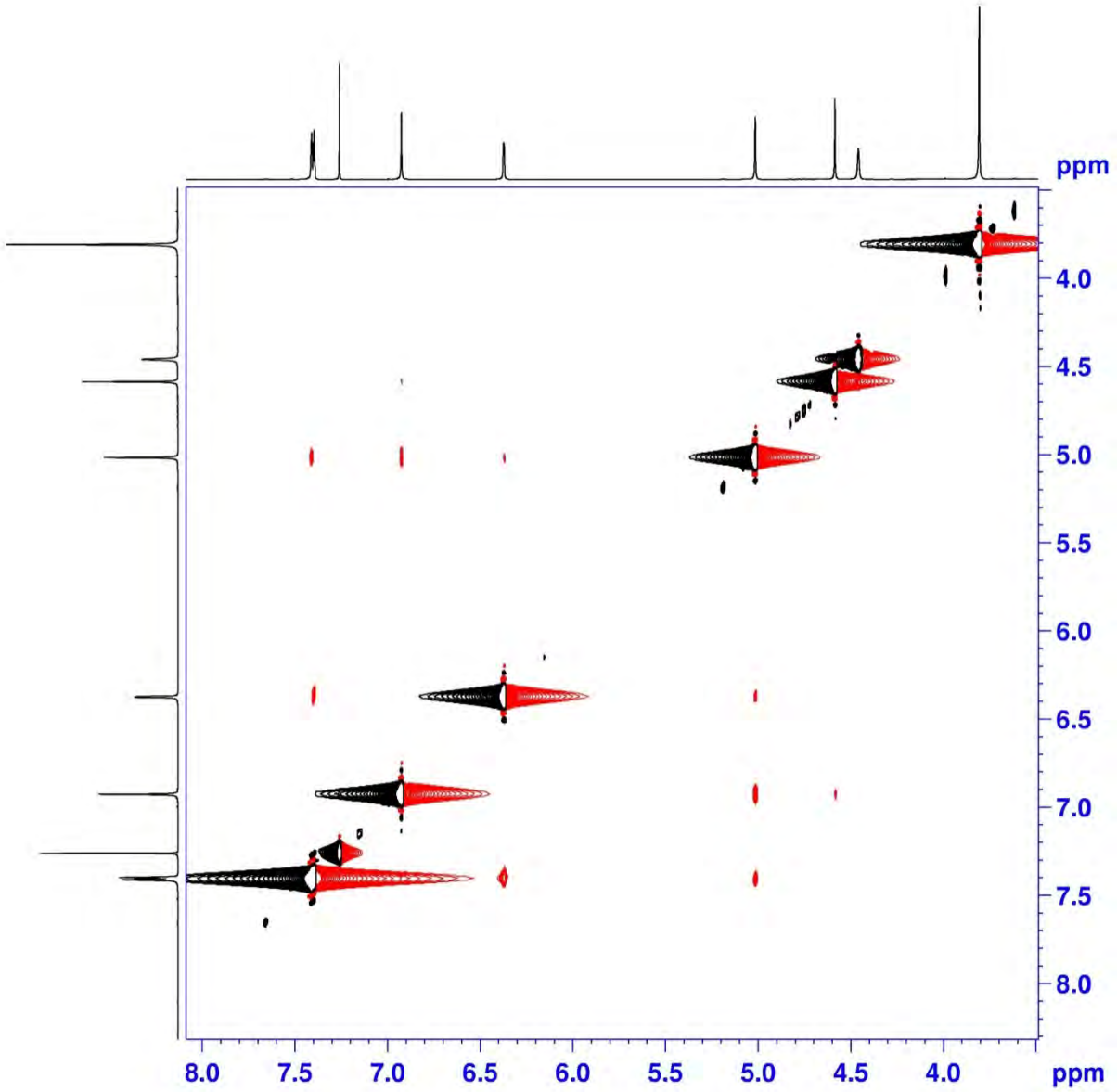
NOESY (400 MHz) spectrum of compound **5b** in $CDCl_3$



BRUKER

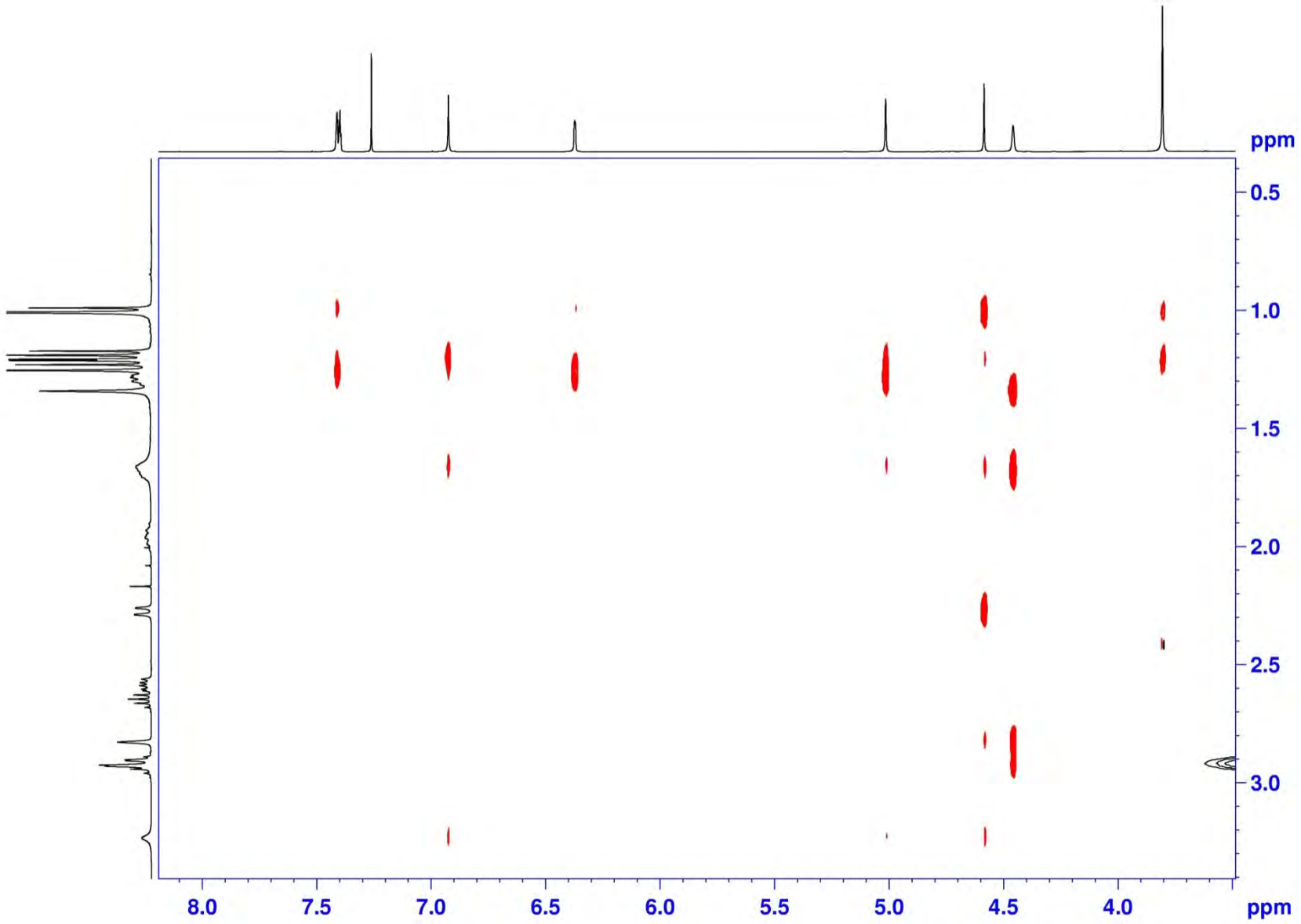
NAME	lws-42-7-fd
EXPNO	7
PROCNO	1
Date_	20150822
Time	5.40
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	noesygpphpp
TD	2048
SOLVENT	CDCl3
NS	16
DS	32
SWH	4000.000 Hz
FIDRES	1.953125 Hz
AQ	0.2560500 sec
RG	208.5
DW	125.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00010972 sec
D1	1.99385595 sec
D8	0.30000001 sec
D11	0.03000000 sec
D12	0.00002000 sec
D16	0.00020000 sec
IN0	0.00025000 sec
========	CHANNEL f1 ========
SFO1	400.1318006 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
P17	2500.00 usec
ND0	1
TD	256
SFO1	400.1318 MHz
FIDRES	15.625000 Hz
SW	9.997 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300094 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300094 MHz
WDW	QSINE

NOESY (400 MHz) spectrum of compound **5b** in $CDCl_3$

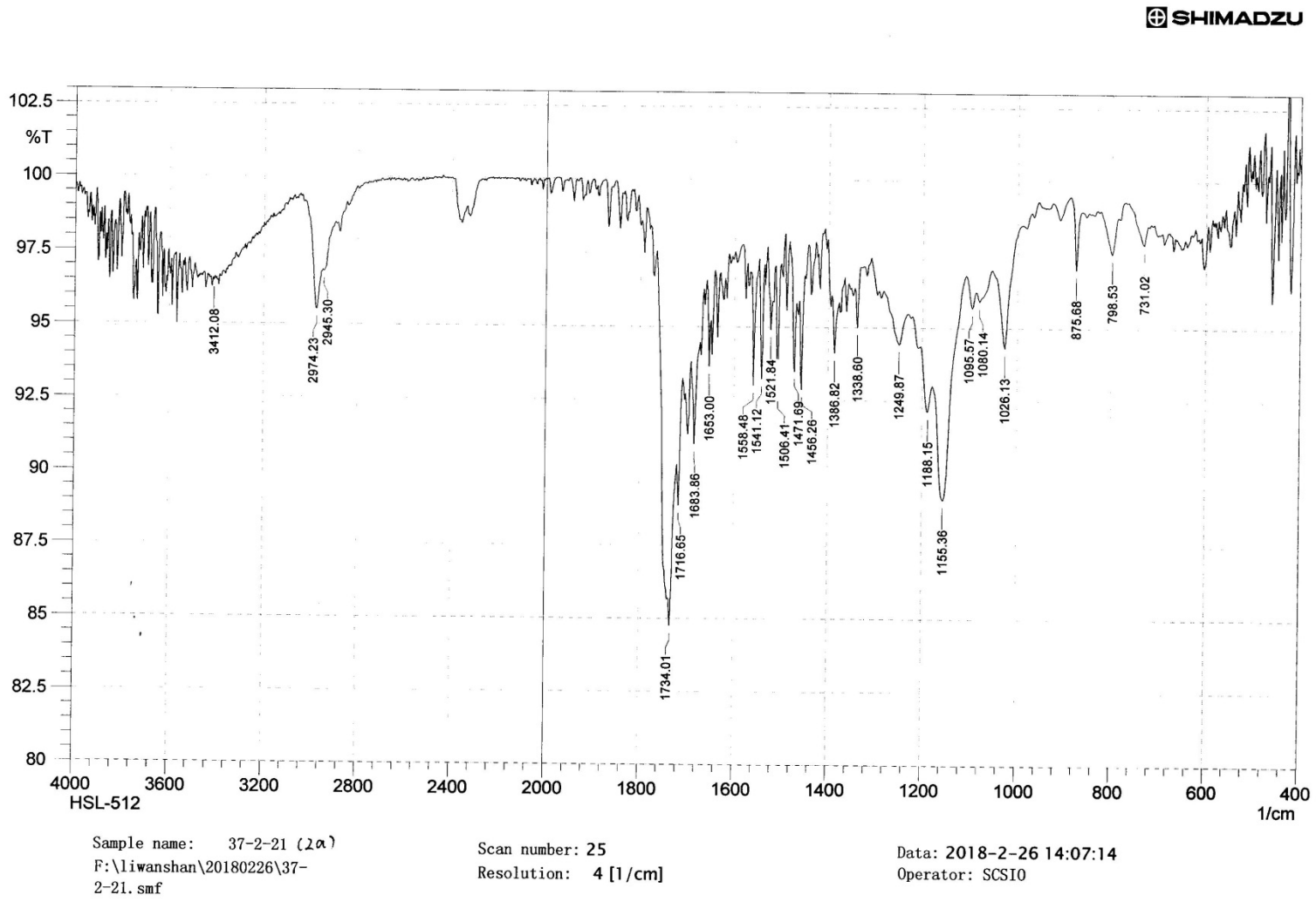


NAME	lws-42-7-fd
EXPNO	7
PROCNO	1
Date_	20150822
Time	5.40
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	noesygpphpp
TD	2048
SOLVENT	CDCl3
NS	16
DS	32
SWH	4000.000 Hz
FIDRES	1.953125 Hz
AQ	0.2560500 sec
RG	208.5
DW	125.000 usec
DE	10.00 usec
TE	297.0 K
D0	0.00010972 sec
D1	1.99385595 sec
D8	0.30000001 sec
D11	0.03000000 sec
D12	0.00002000 sec
D16	0.00020000 sec
IN0	0.00025000 sec
======== CHANNEL f1 ========	
SFO1	400.1318006 MHz
NUC1	1H
P1	12.00 usec
P2	24.00 usec
P17	2500.00 usec
ND0	1
TD	256
SFO1	400.1318 MHz
FIDRES	15.625000 Hz
SW	9.997 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300094 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300094 MHz
WDW	QSINE

NOESY (400 MHz) spectrum of compound **5b** in $CDCl_3$



IR spectrum for compound **2a**



IR spectrum for compound **2b**