

Supporting Information:

Granatumins A-G, Limonoids from the Seeds of a Krishna Mangrove, *Xylocarpus granatum*

Min-Yi Li,^{†,∇} Xiao-Bo Yang,[‡] Jian-Yu Pan,[§] Gang Feng,[⊥] Qiang Xiao,^{||} Jari Sinkkonen,[°] Tirumani Satyanandamurty,[¶] and Jun Wu^{*†}

[†]Key Laboratory of Marine Bio-resources Sustainable Utilization, South China Sea Institute of Oceanology, Chinese Academy of Sciences, 164 West Xingang Road, Guangzhou 510301, China

[‡]College of Chinese Medicinal Materials, Jilin Agricultural University, Changchun 130118, China

[§]Institute of Medicinal Plant Development, Chinese Academy of Medical Sciences and Peking Union Medical College, Beijing 100193, China

[⊥]Environment and Plant Protection Institute, Academy of Tropical Agriculture Sciences of China, Danzhou in Hainan Province, 571737, China

^{||}Institute of Organic Chemistry, Jiangxi Science & Technology Normal University, Nanchang 330013, China

[∇]Graduate University of Chinese Academy of Sciences, 19 Yuquan Road, Beijing 100049, China

[°]Laboratory of Organic Chemistry and Chemical Biology, Department of Chemistry, University of Turku, Turku 20014, Finland

[¶]Government Degree College at Amadala valasa, Srikakulam District, Andhra Pradesh, India

*Correspondence author. *E-mail:* wwujun2003@yahoo.com

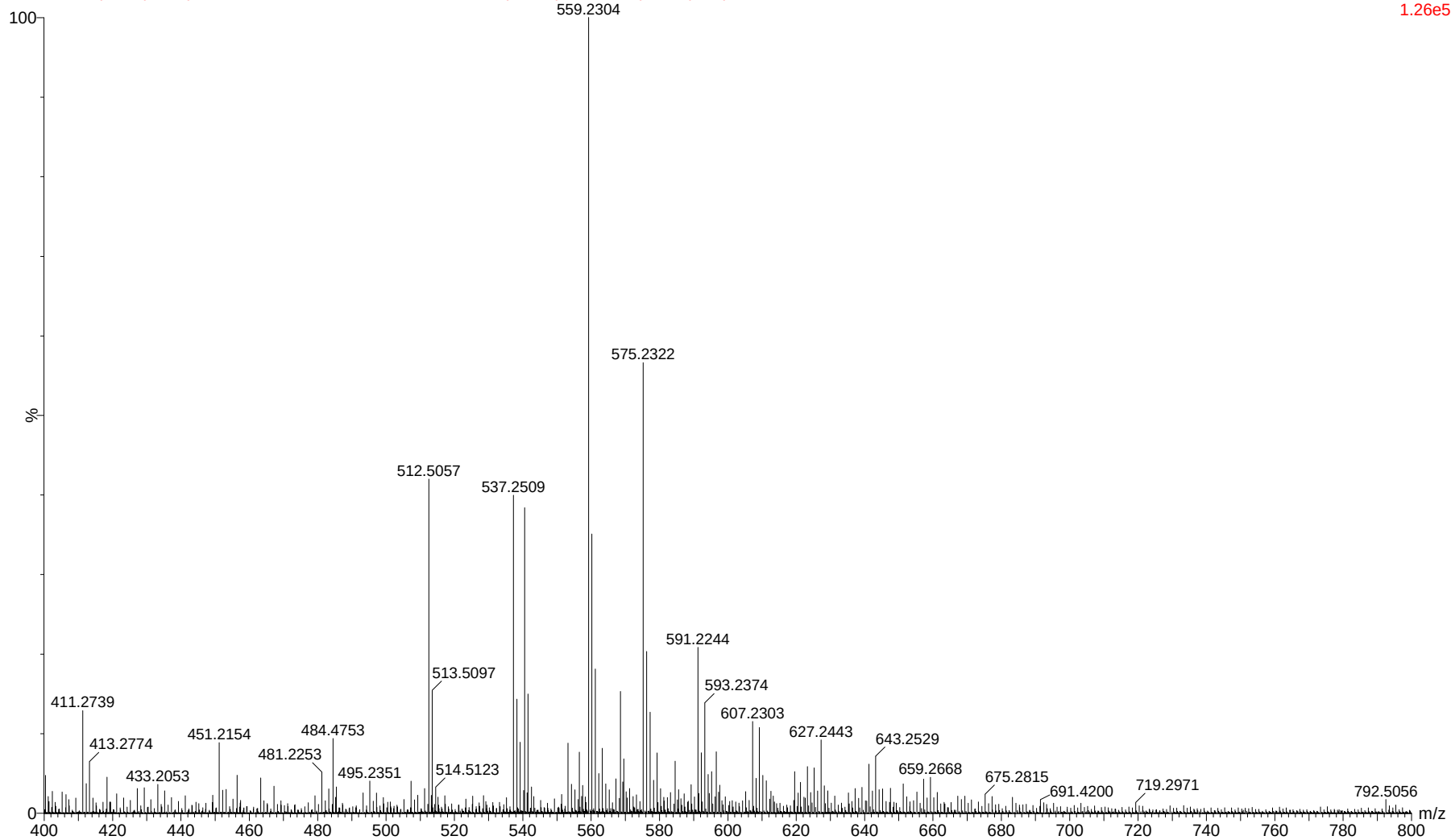
Copies of HR-TOFMS, ¹H and ¹³C NMR spectra of granatumins A-G (1-7).

HR-TOFMS of granatumin A (1)

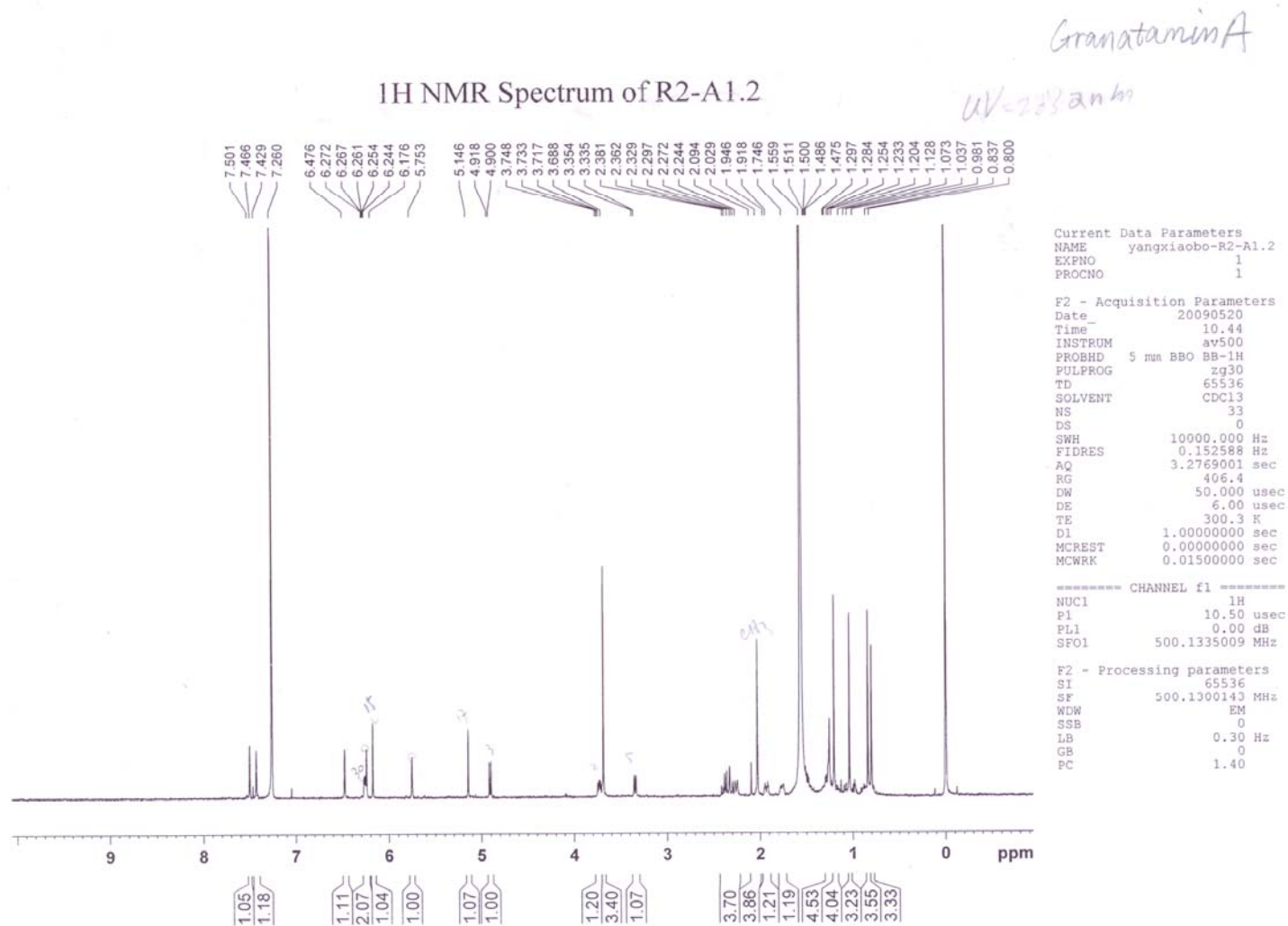
MS+

xiaobo-4 28 (1.685) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (4:36)

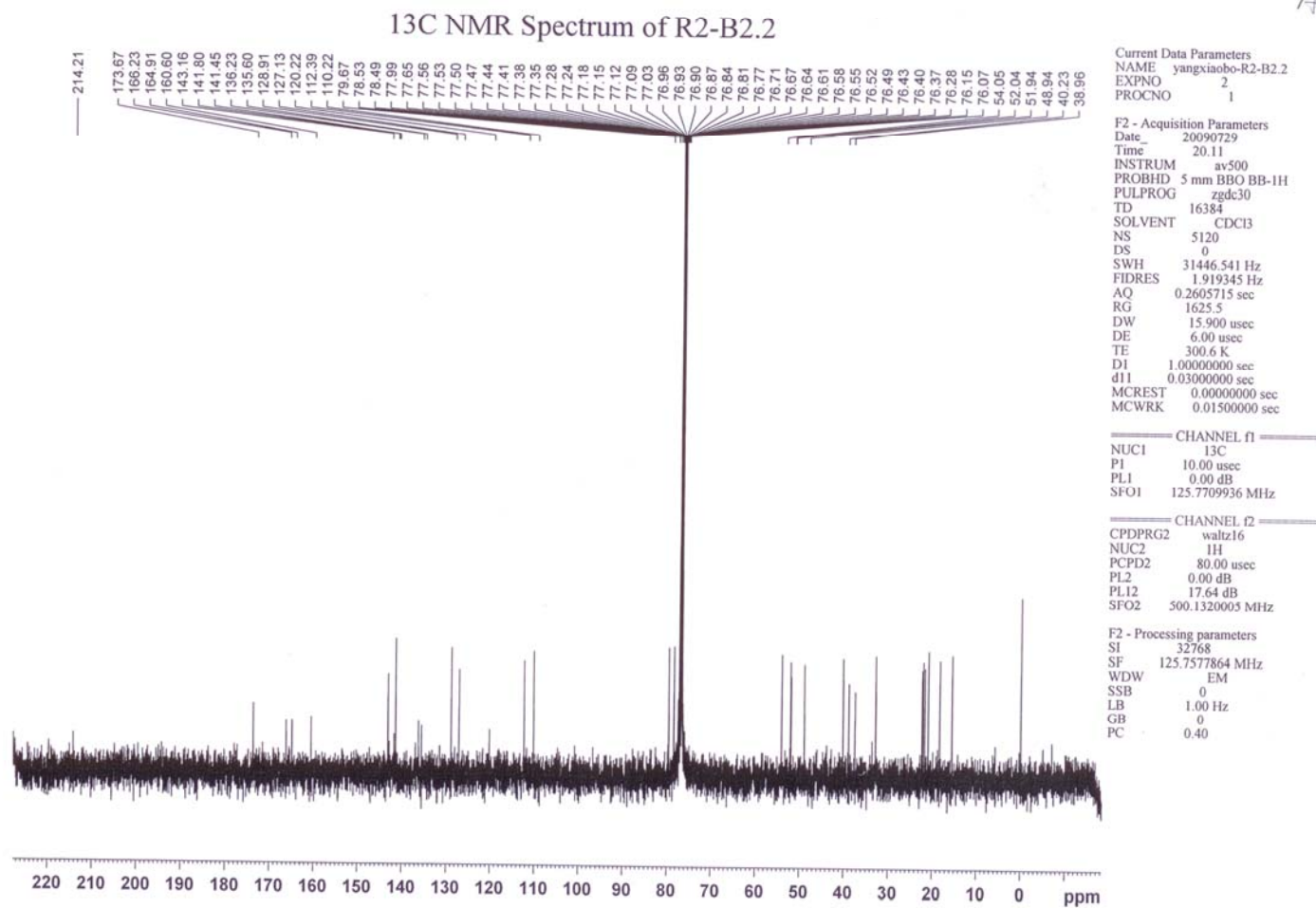
1: TOF MS ES+
1.26e5



^1H NMR (500 MHz) spectra of granatumin A (**1**) in CDCl_3

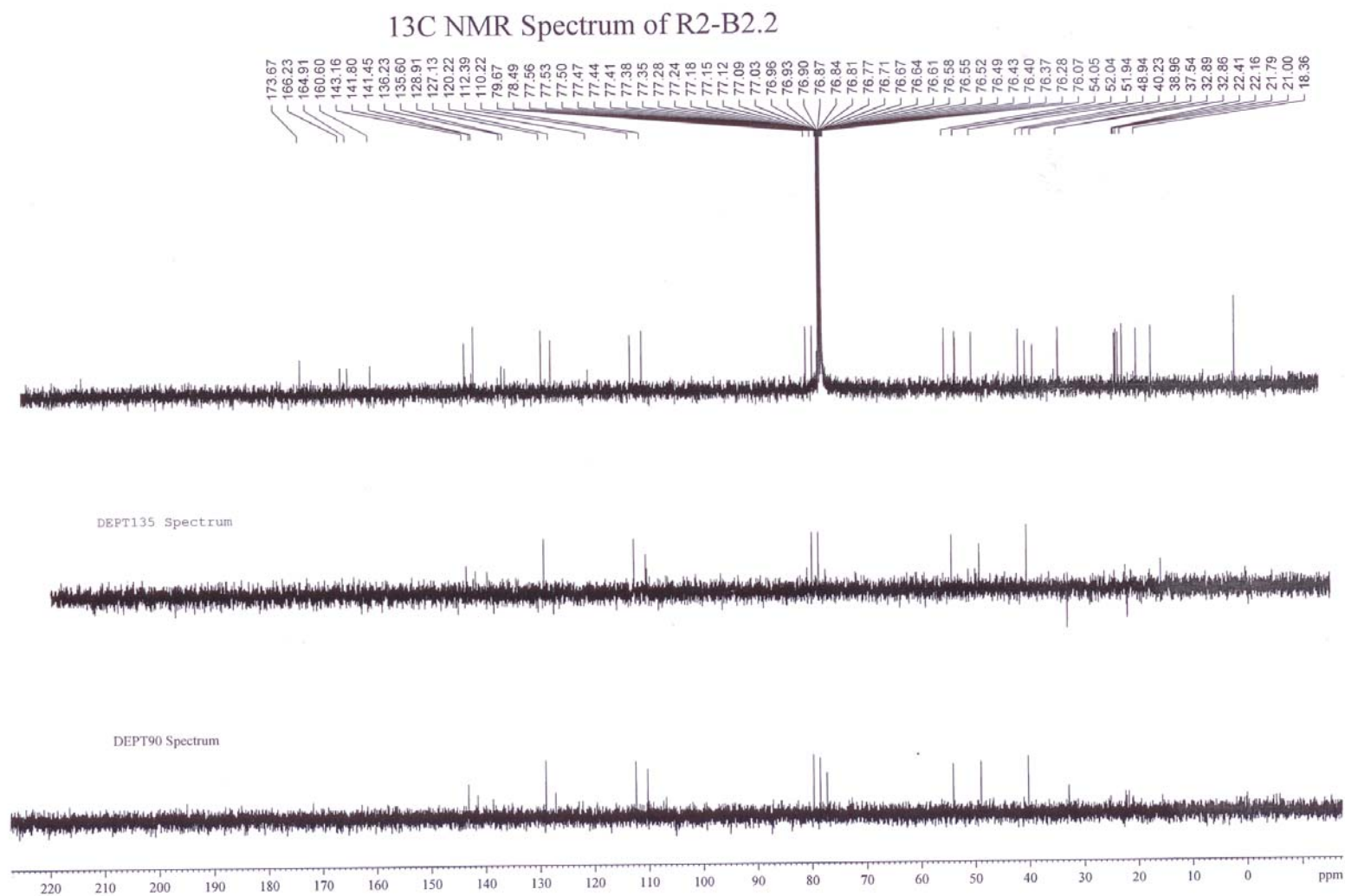


^{13}C NMR (125 MHz) spectrum of granatumin A (**1**) in CDCl_3

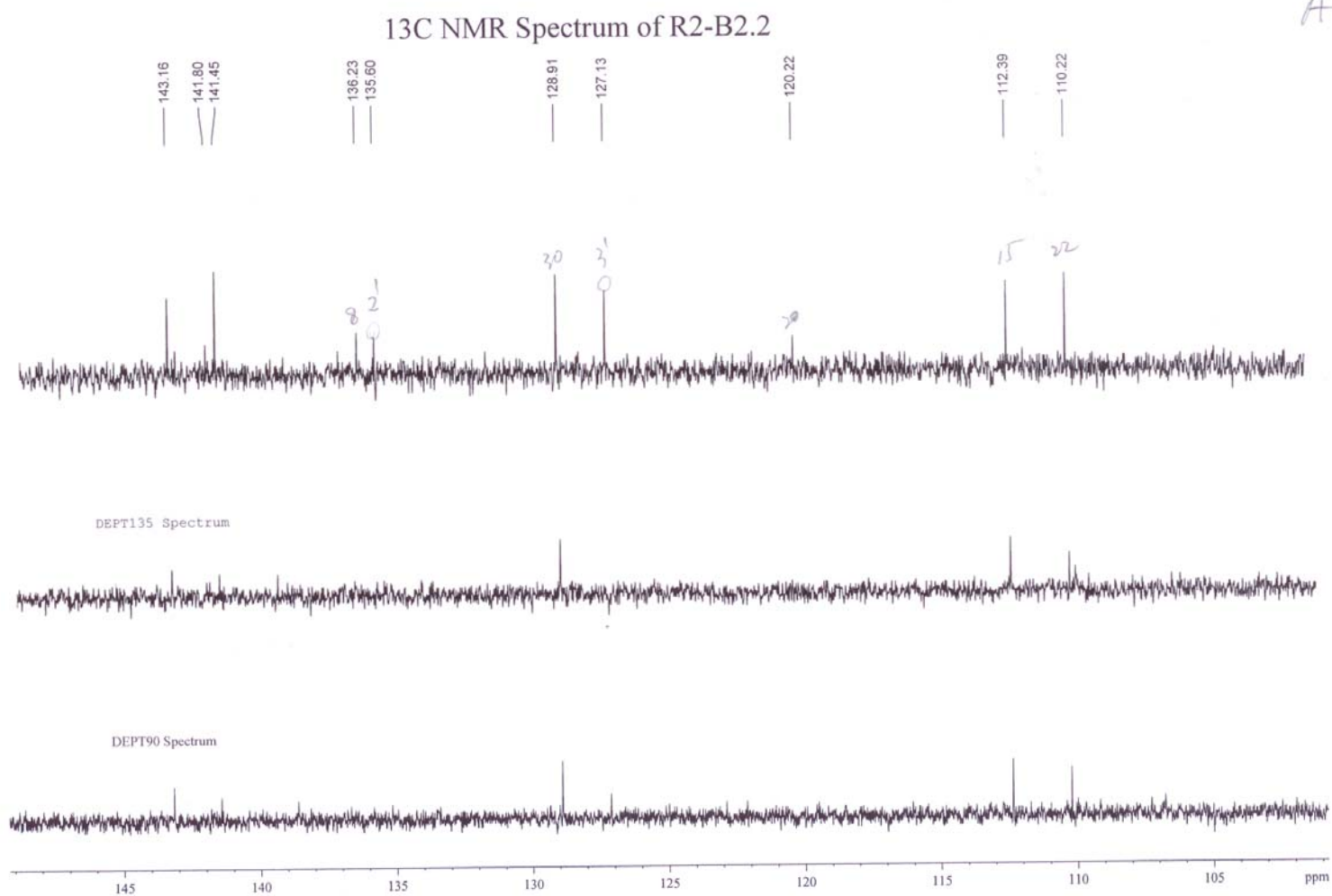


^{13}C NMR (125 MHz) and DEPT spectra of granatumin A (**1**) in CDCl_3

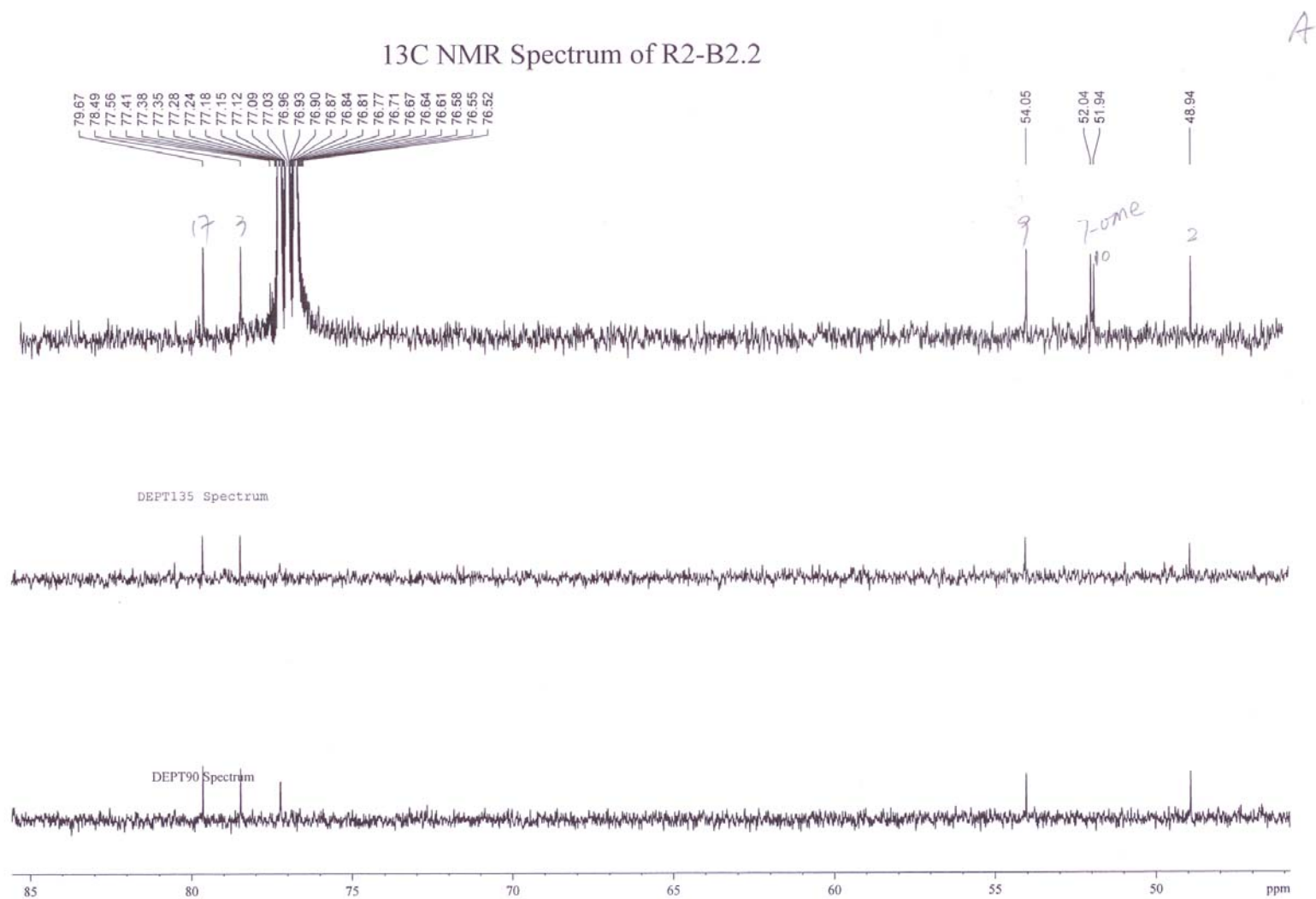
A



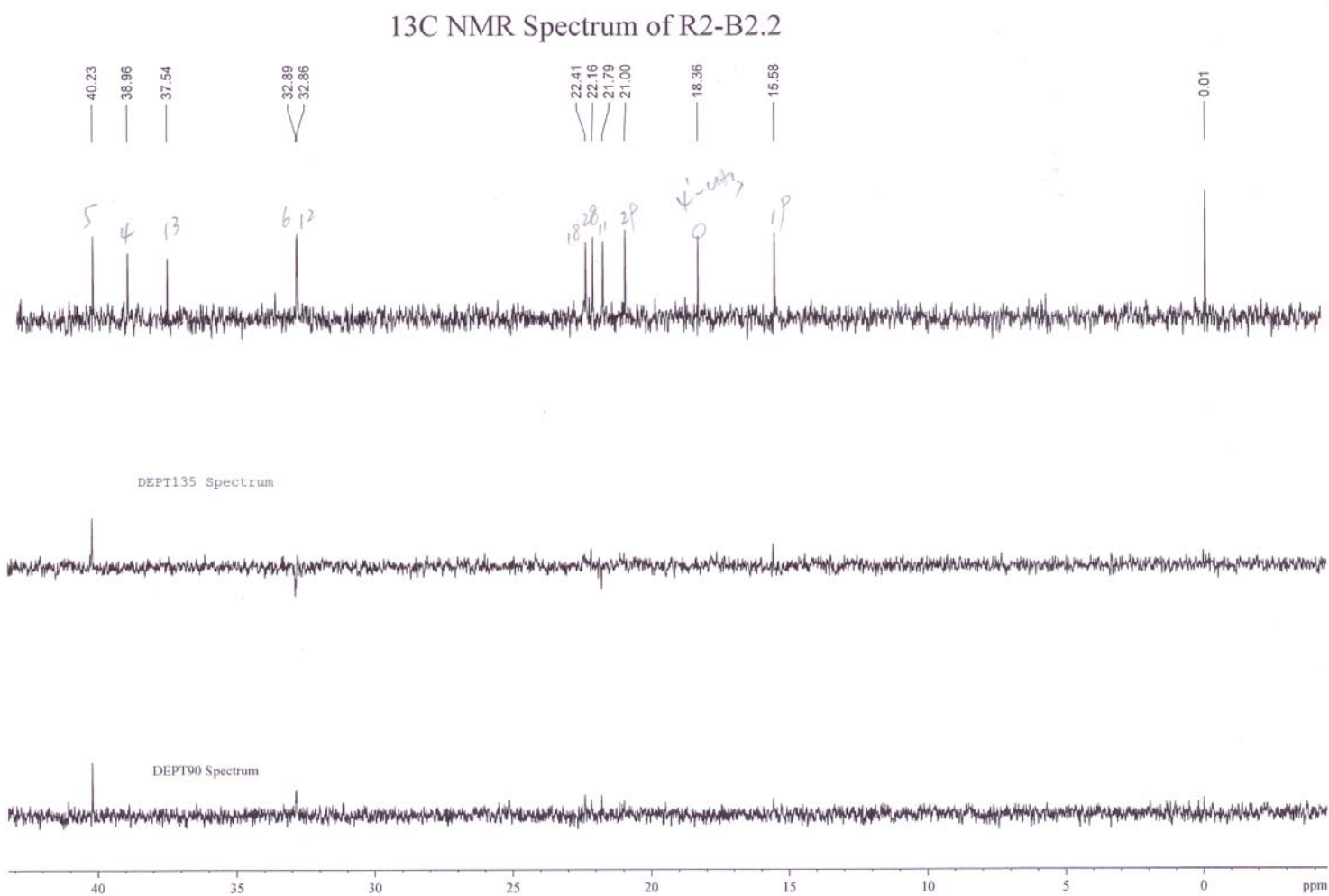
Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin A (**1**) in CDCl_3



Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin A (**1**) in CDCl_3



Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin A (**1**) in CDCl_3

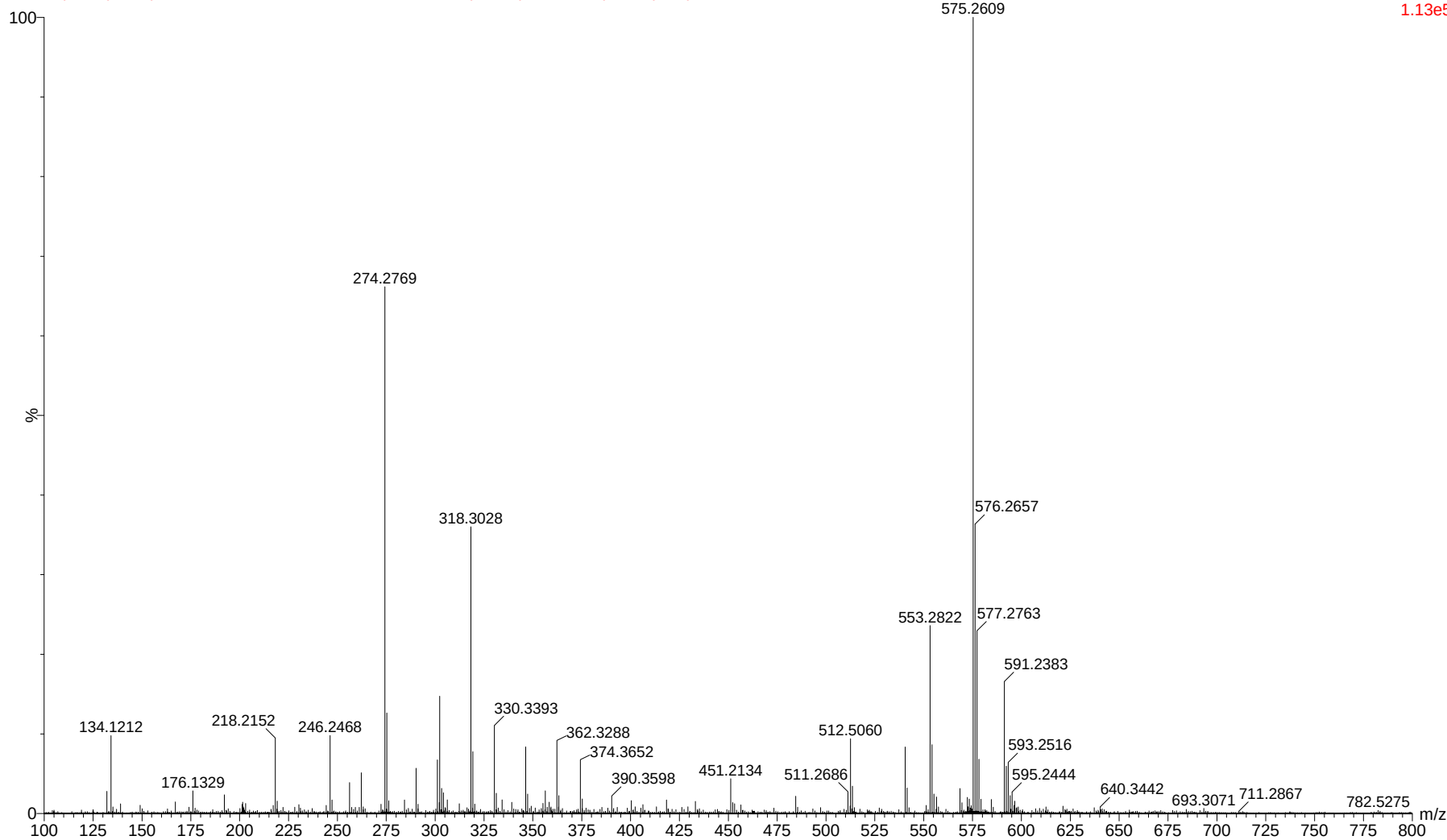


HR-TOFMS of granatumin B (2)

MS+

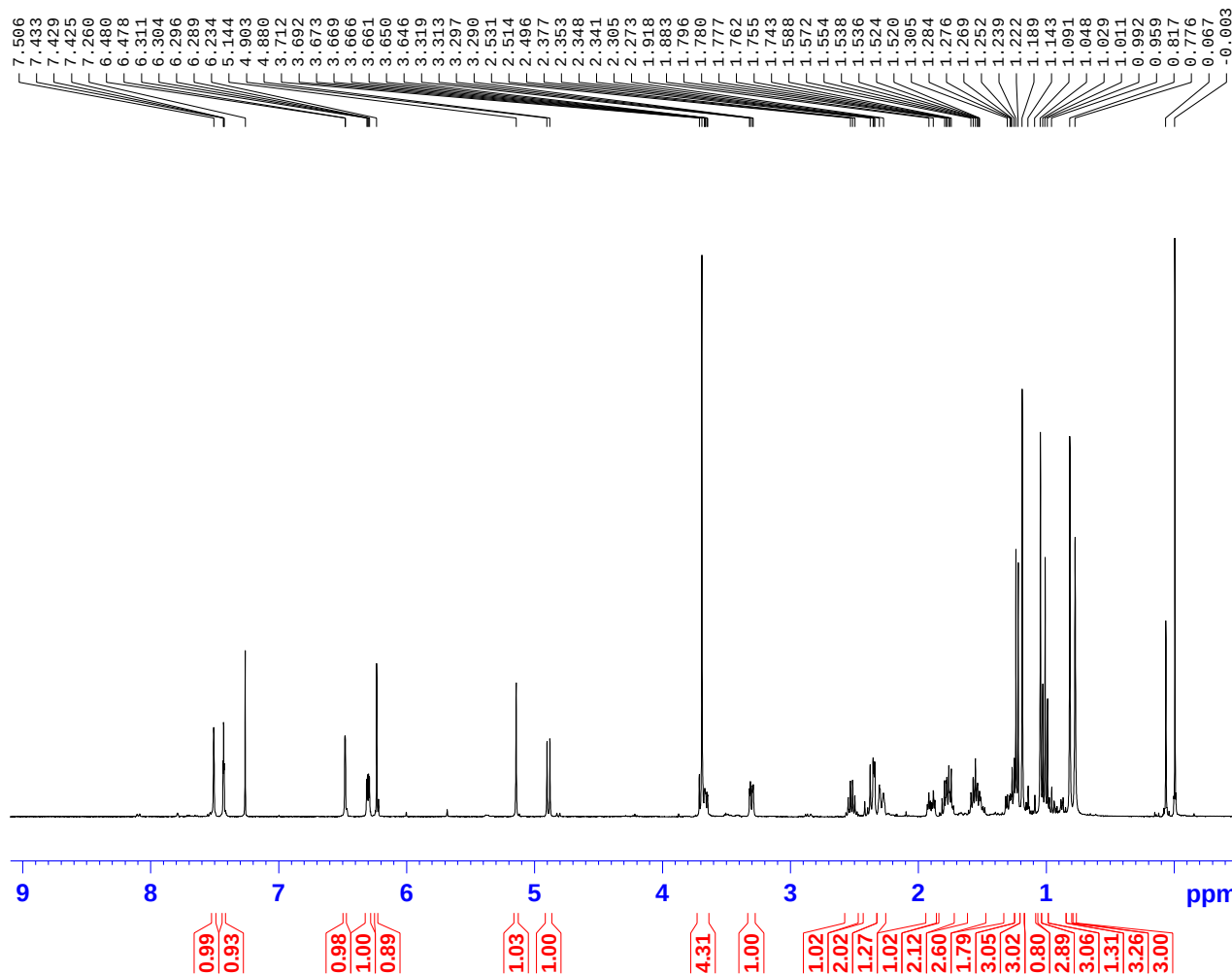
Jul-5 5 (0.286) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (2:32)

1: TOF MS ES+
1.13e5



^1H NMR (400 MHz) of granatumin B (2) in CDCl_3

^1H NMR Spectrum of Jul-5
Solvent: CDCl_3



Current Data Parameters
NAME Jul-5-400
EXPNO 1
PROCNO 1

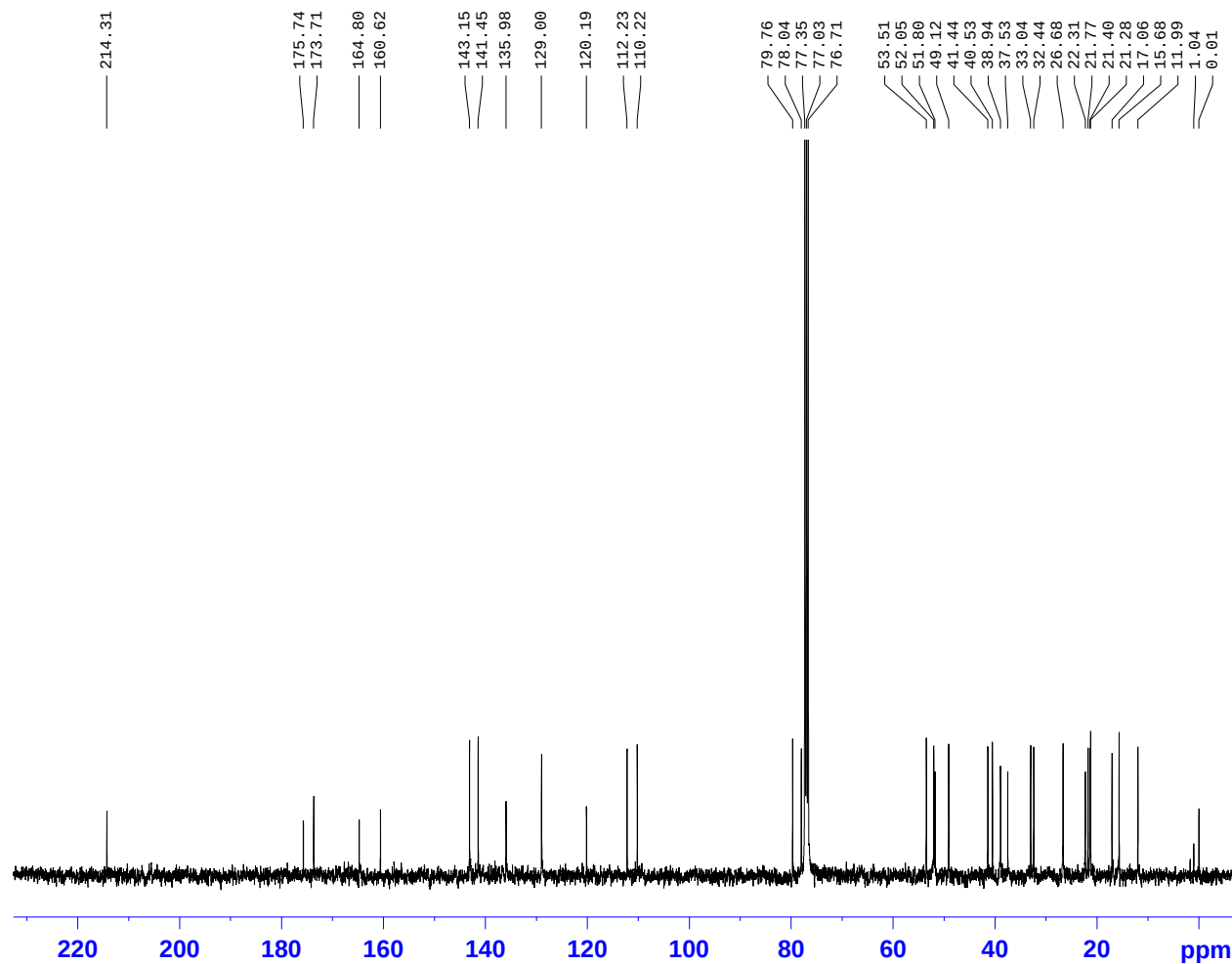
F2 - Acquisition Parameters
Date_ 20090721
Time 17.35
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl_3
NS 32
DS 0
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715922 sec
RG 362
DW 60.600 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^1H
P1 8.10 usec
PL1 -1.00 dB
SF01 399.7524686 MHz

F2 - Processing parameters
SI 32768
SF 399.7500051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (100 MHz) spectrum of granatumin B (2) in CDCl_3

^{13}C NMR Spectrum of Jul-5
Solvent: CDCl_3



Current Data Parameters
NAME Jul-5-400
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090721
Time 17.47
INSTRUM av400
PROBHD 5 mm BBO BB-1H
PULPROG ph_zpgpg45
TD 65536
SOLVENT CDCl_3
NS 2732
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 14596.5
DW 20.800 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

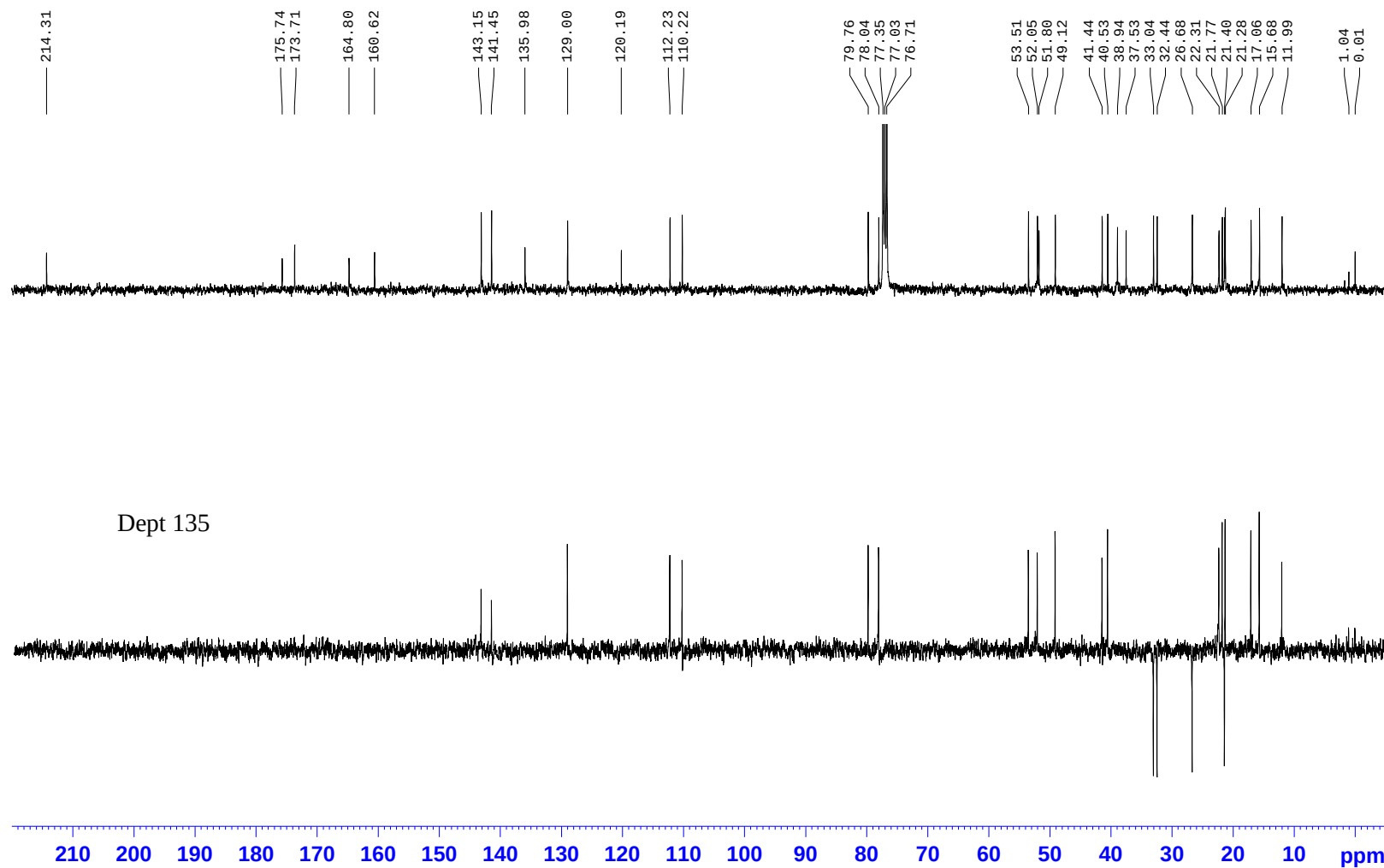
===== CHANNEL f1 =====
NUC1 ^{13}C
P1 7.60 usec
PL1 0.00 dB
SF01 100.5285754 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SF02 399.7515990 MHz

F2 - Processing parameters
SI 32768
SF 100.5172143 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

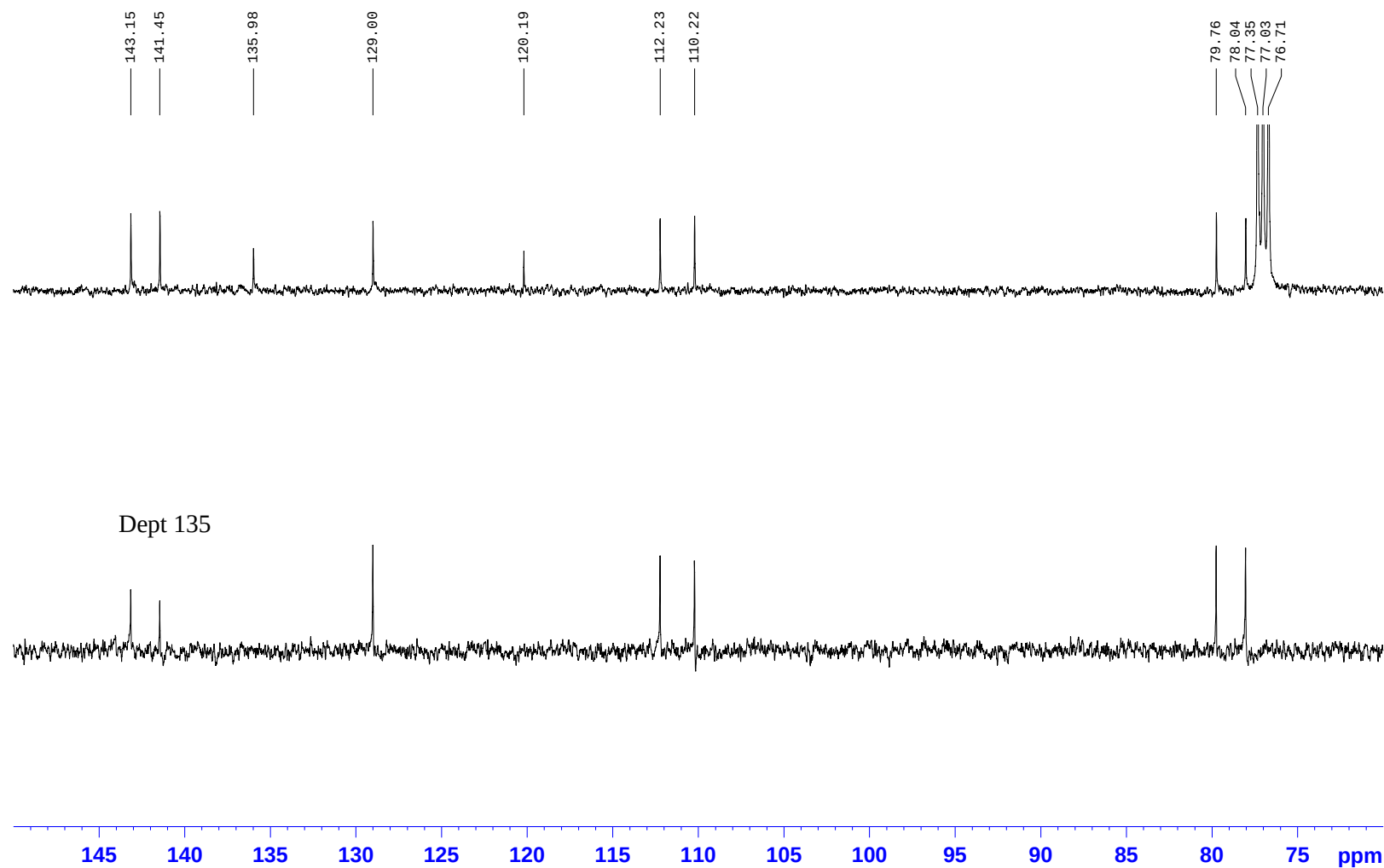
^{13}C NMR (100 MHz) and DEPT spectra of granatumin B (2) in CDCl_3

^{13}C NMR Spectrum of Jul-5
Solvent: CDCl_3



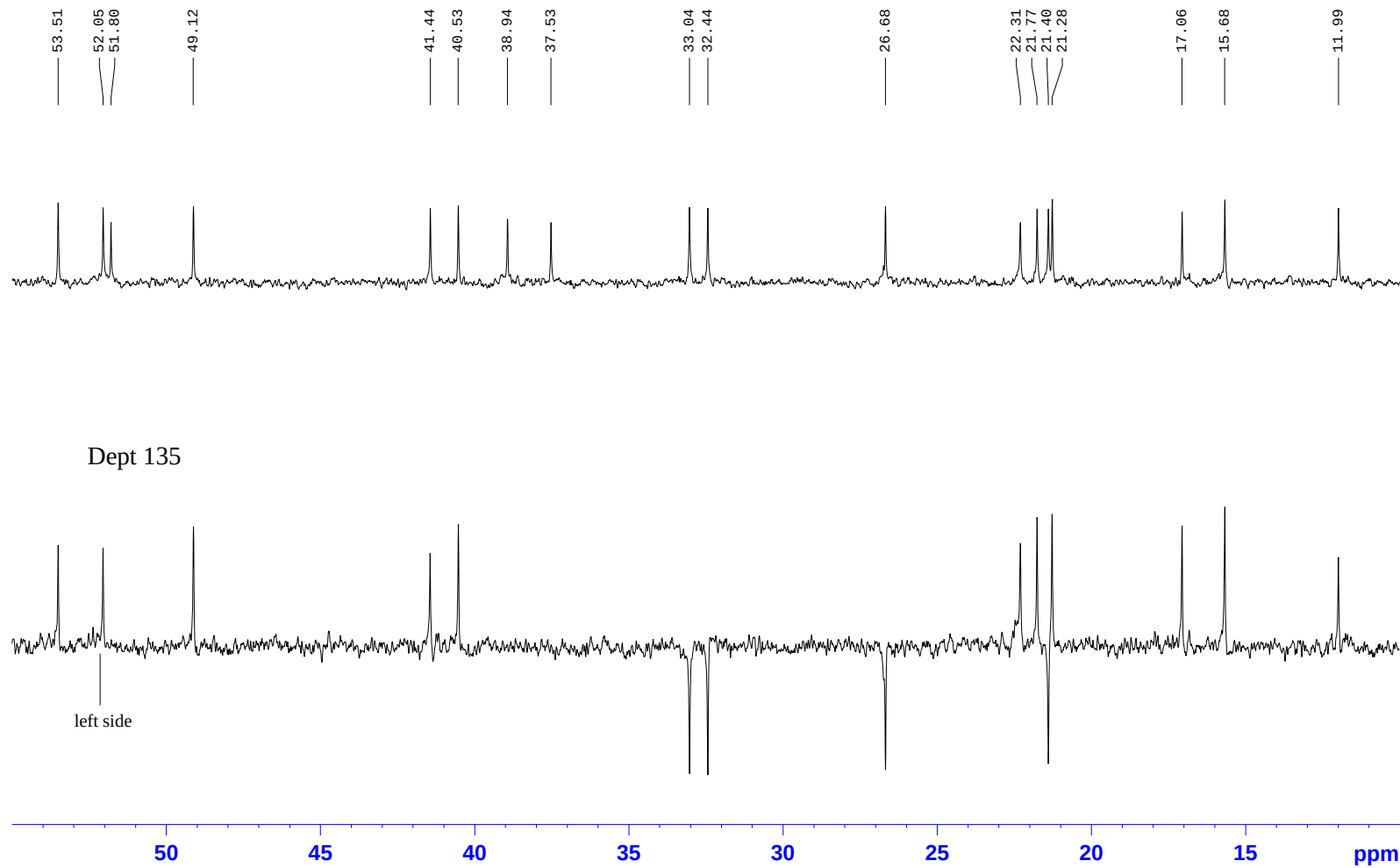
Enlarged segment of ^{13}C NMR (100 MHz) and DEPT spectra of granatumin B (2) in CDCl_3

^{13}C NMR Spectrum of Jul-5
Solvent: CDCl_3



Enlarged segment of ^{13}C NMR (100 MHz) and DEPT spectra of granatumin B (2) in CDCl_3

^{13}C NMR Spectrum of Jul-5
Solvent: CDCl_3

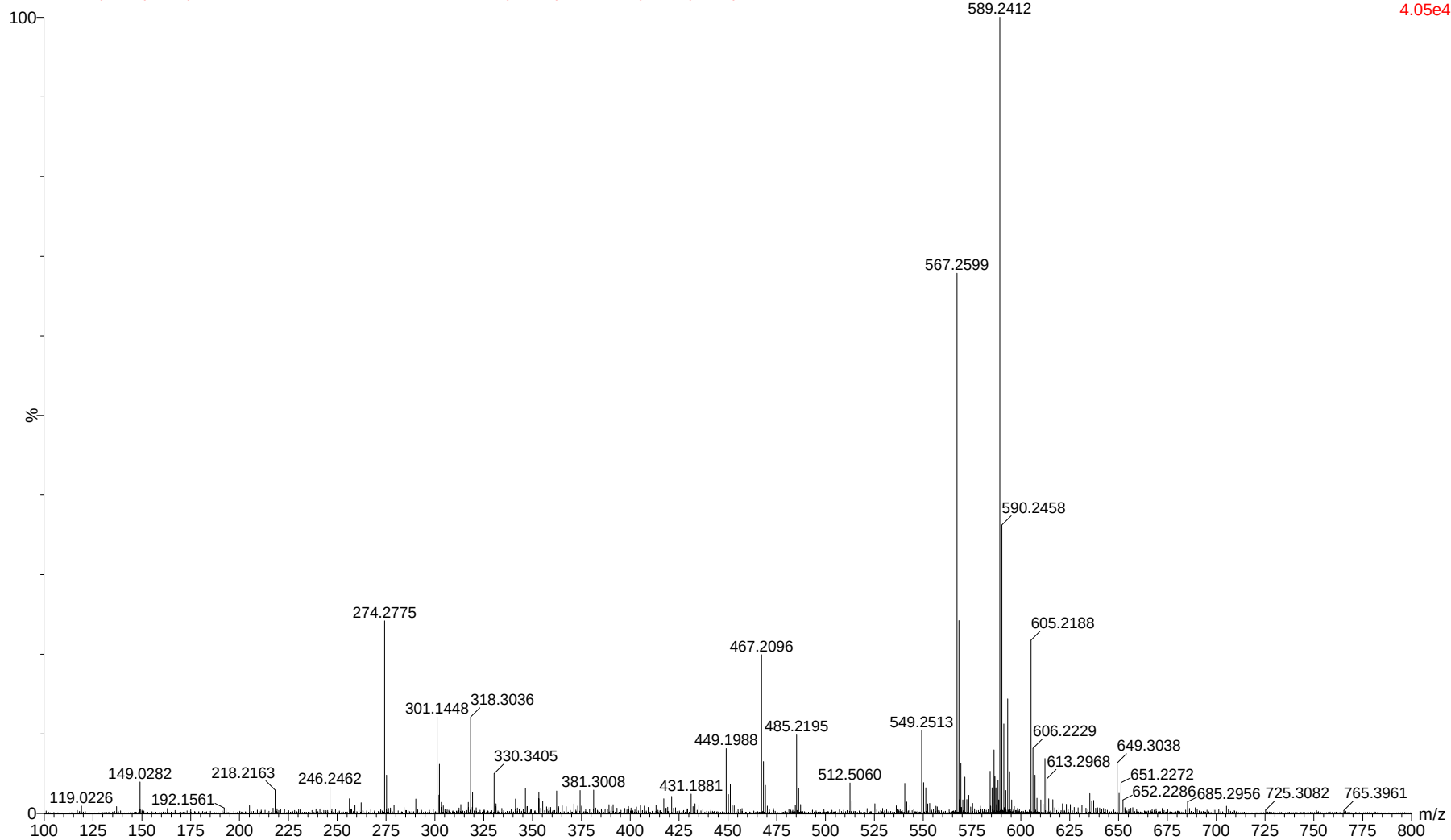


HR-TOFMS of granatumin C (3)

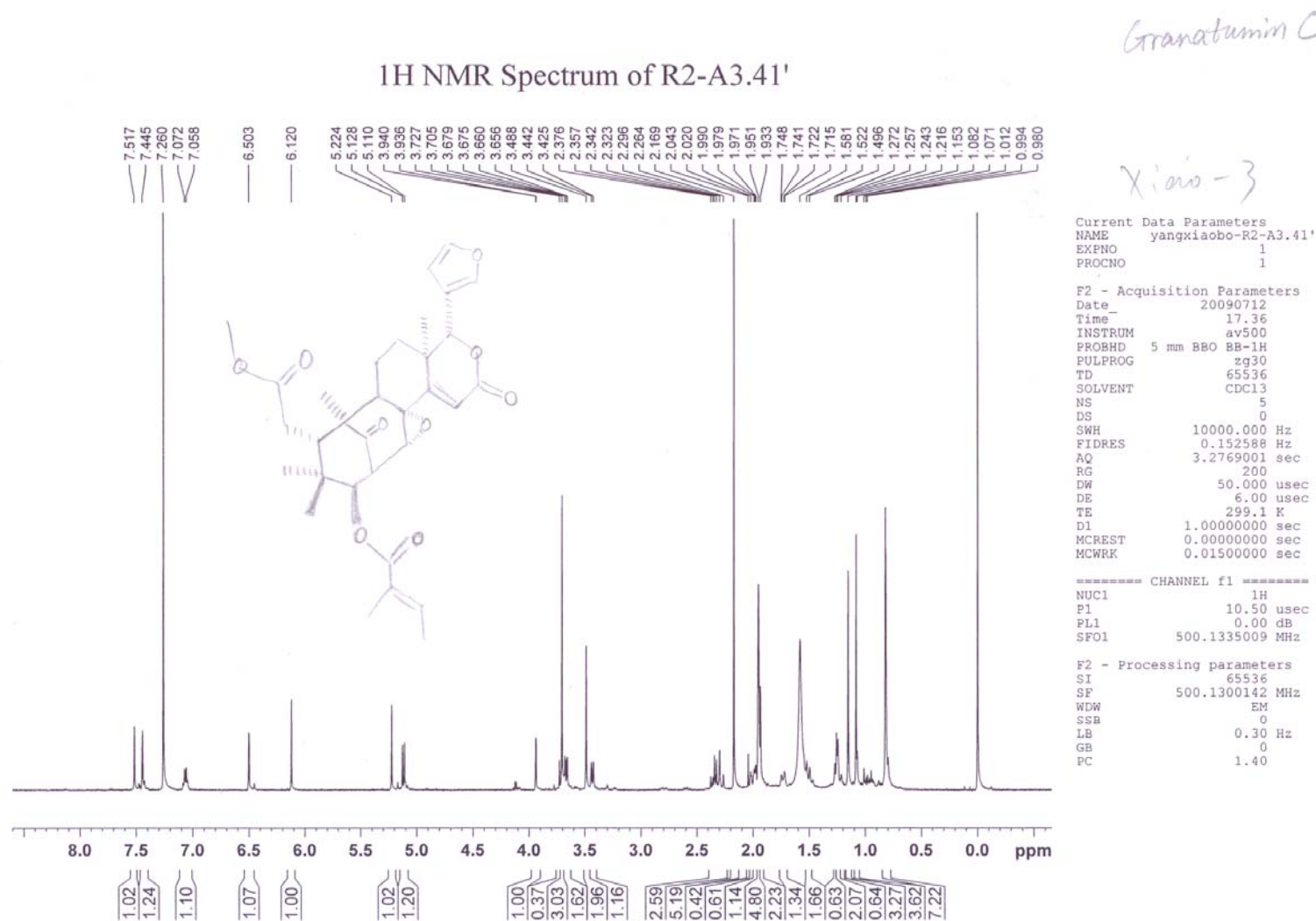
MS+

xiaobo-3 21 (1.251) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (21:32)

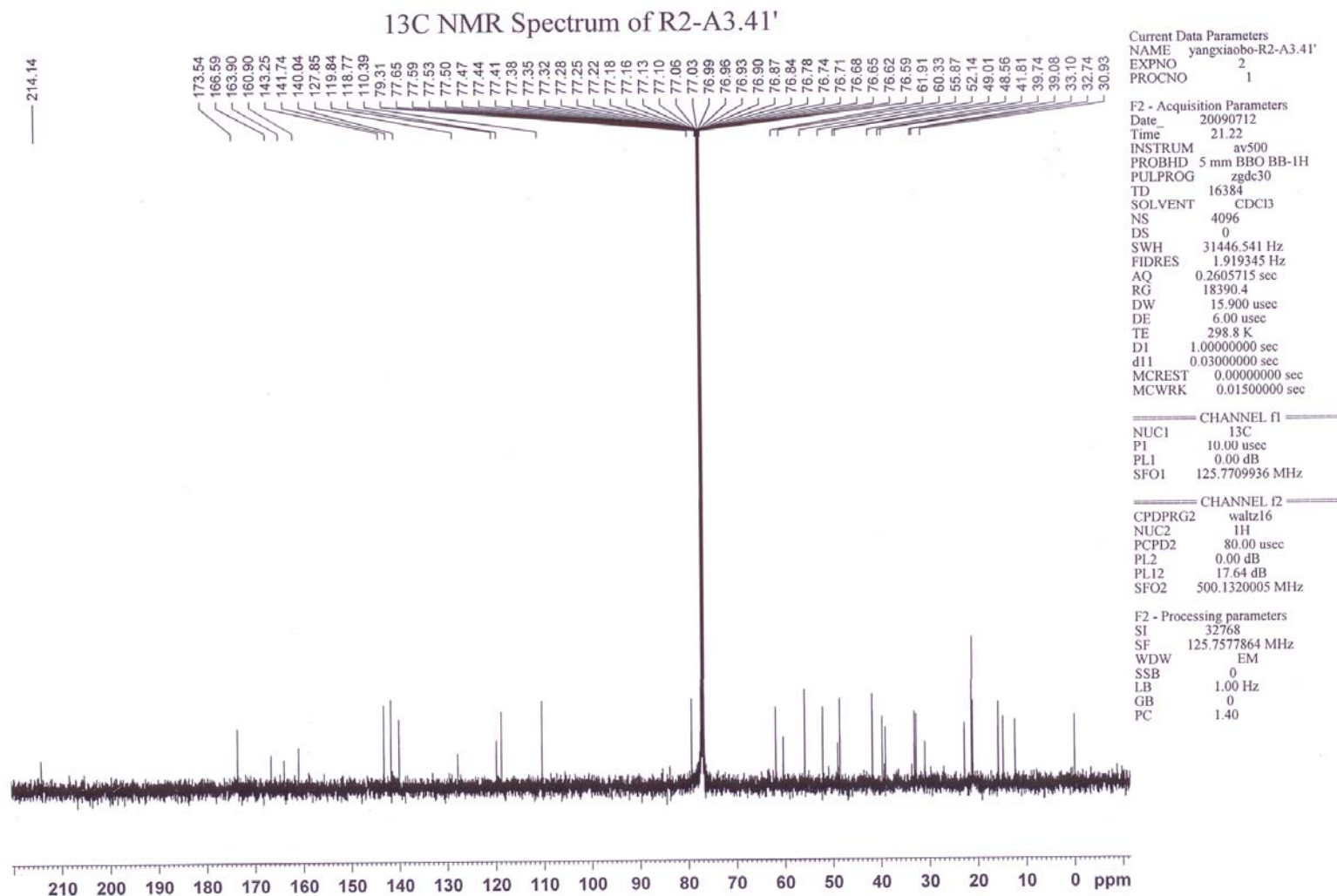
1: TOF MS ES+
4.05e4



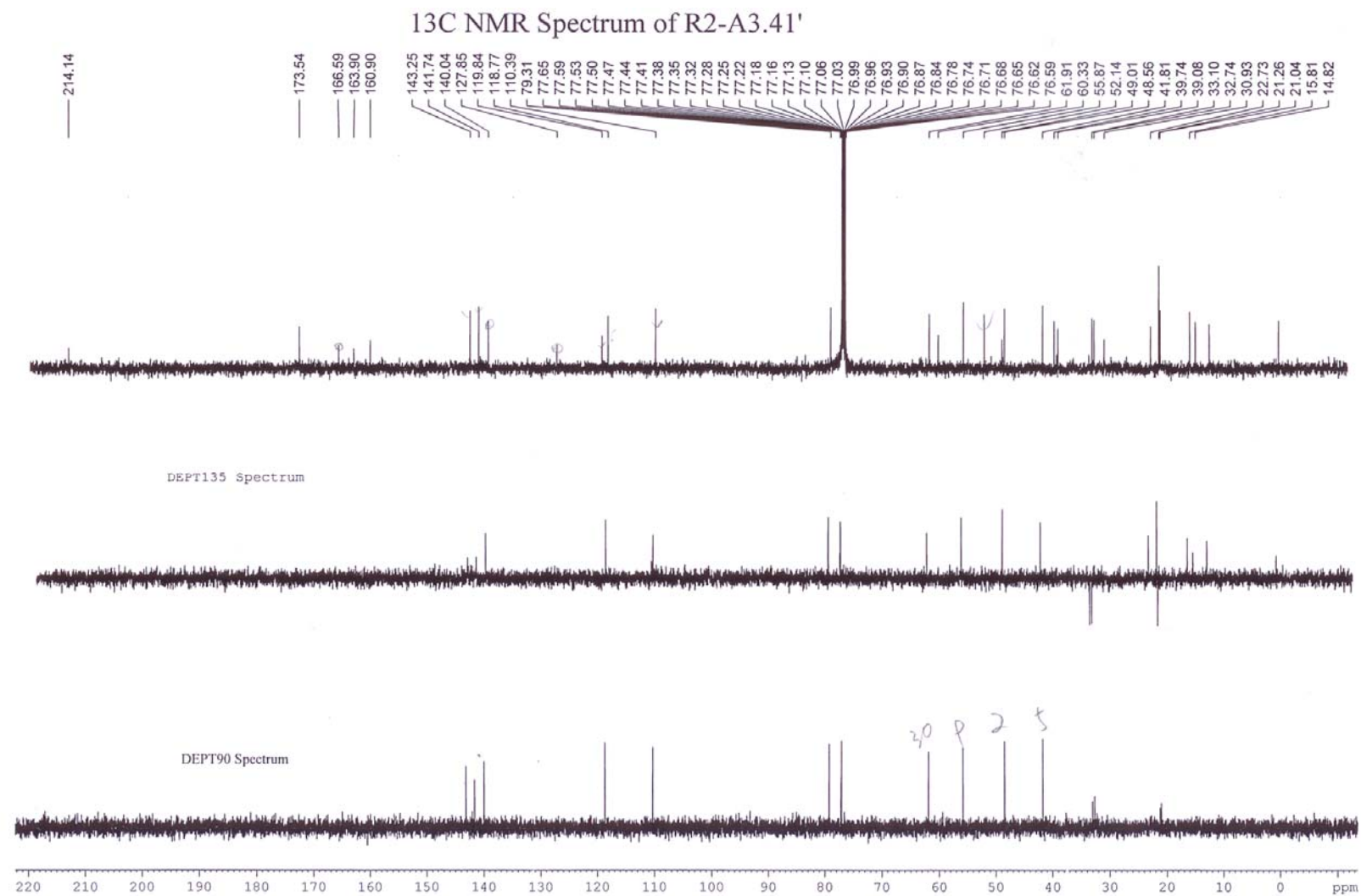
^1H NMR (500 MHz) spectrum of granatumin C (**3**) in CDCl_3



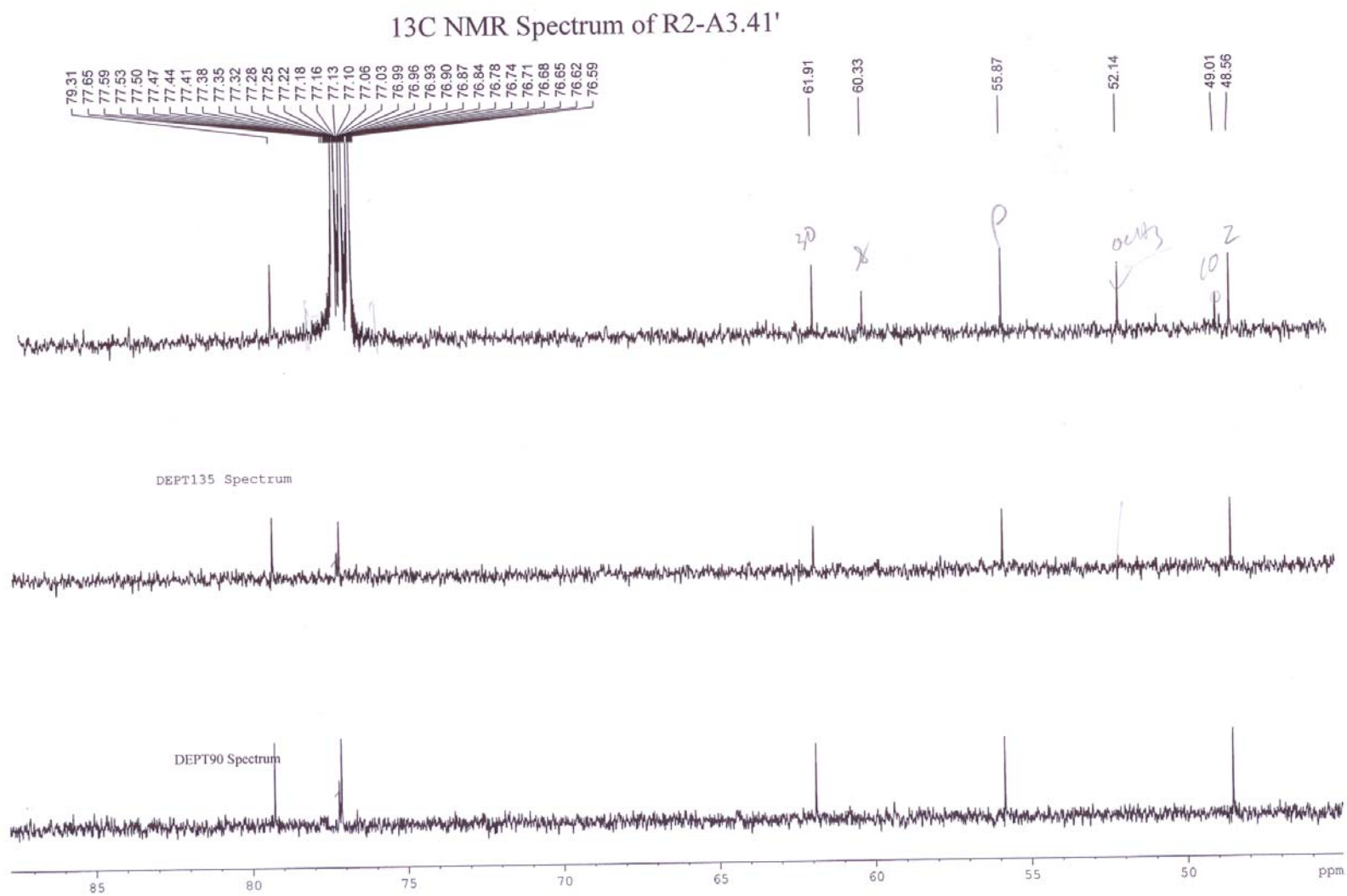
^{13}C NMR (125 MHz) spectrum of granatumin C (**3**) in CDCl_3



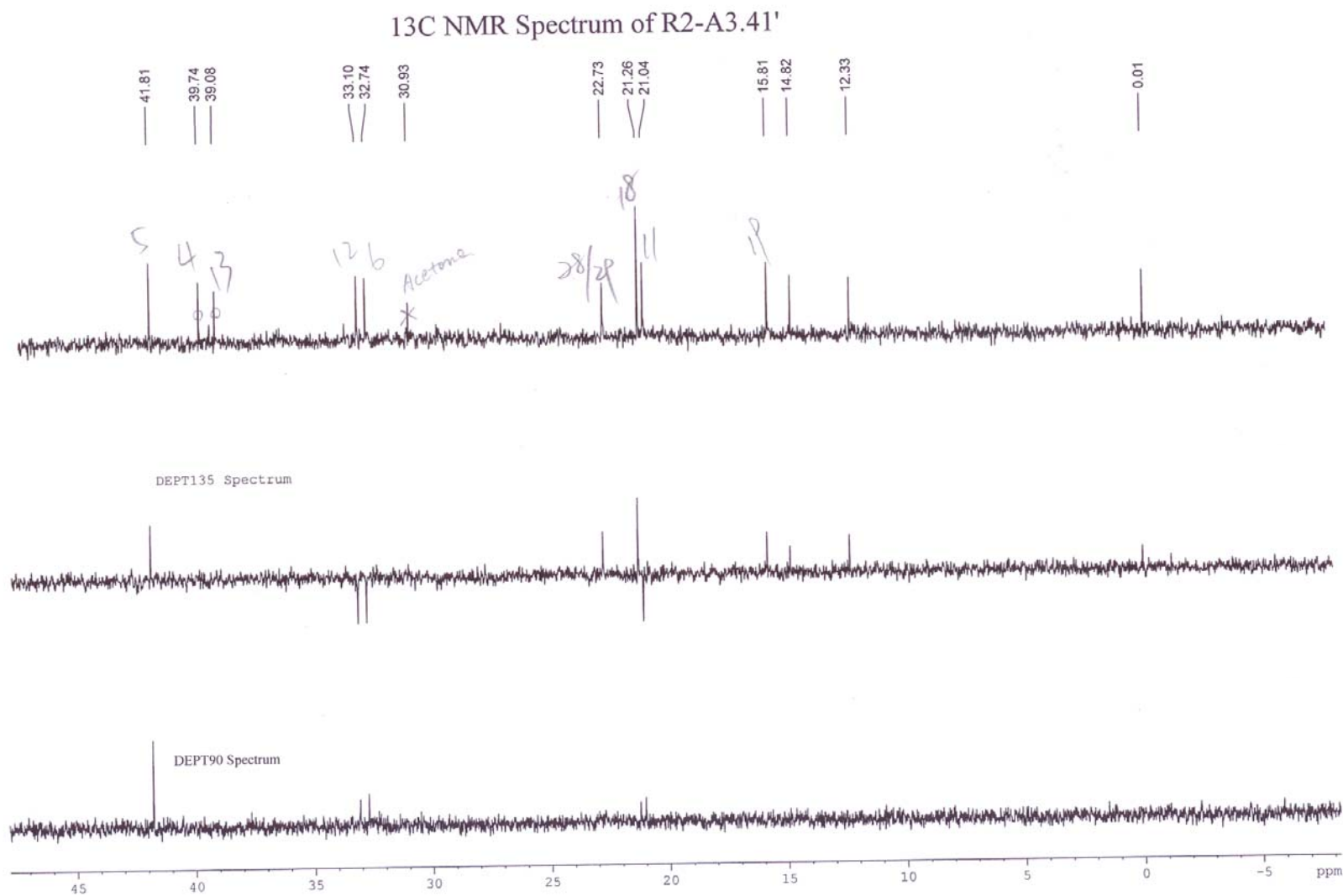
^{13}C NMR (125 MHz) and DEPT spectra of granatumin C (**3**) in CDCl_3



Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin C (**3**) in CDCl_3



Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin C (**3**) in CDCl_3

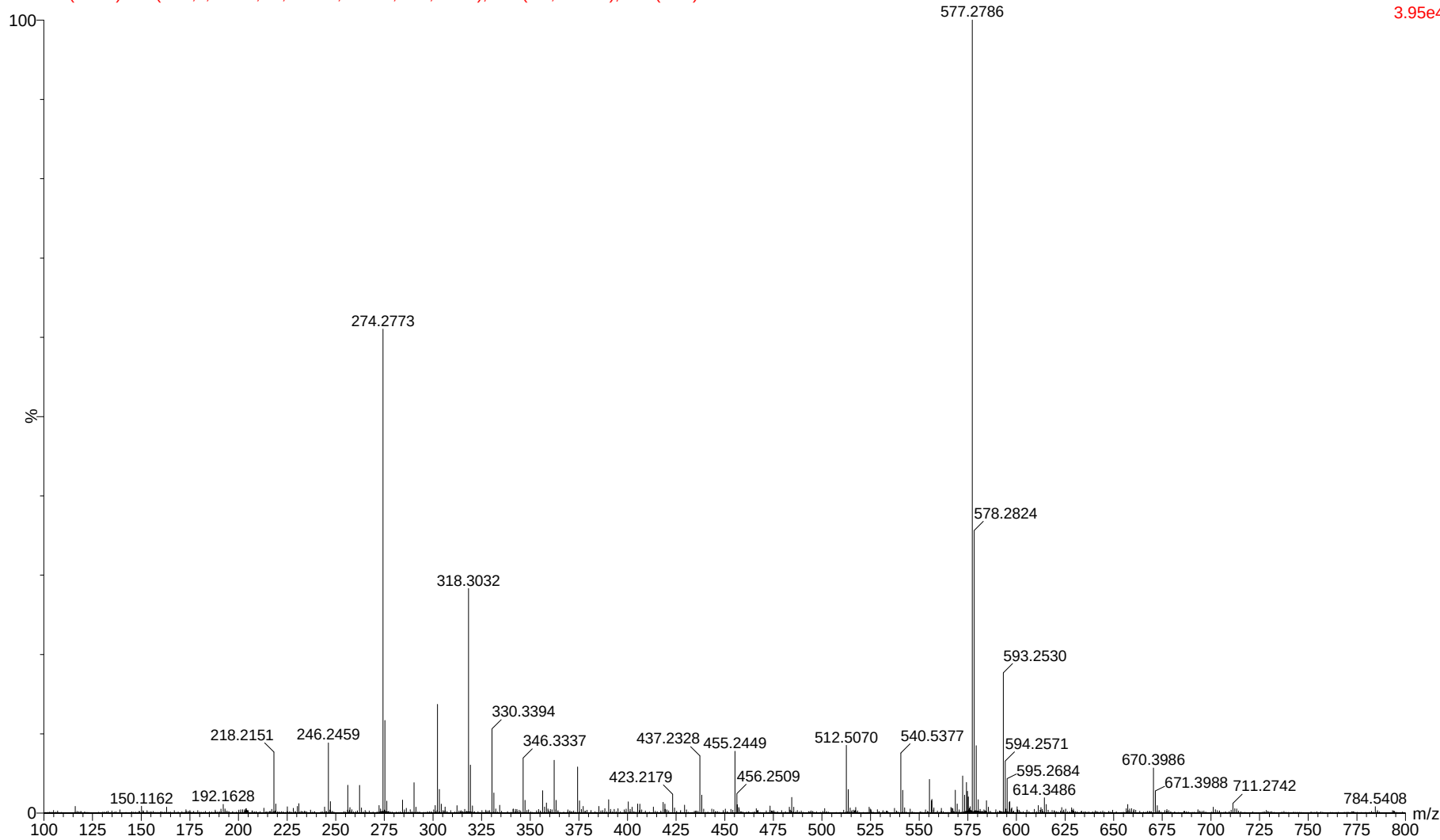


HR-TOFMS of granatumin D (4)

MS+

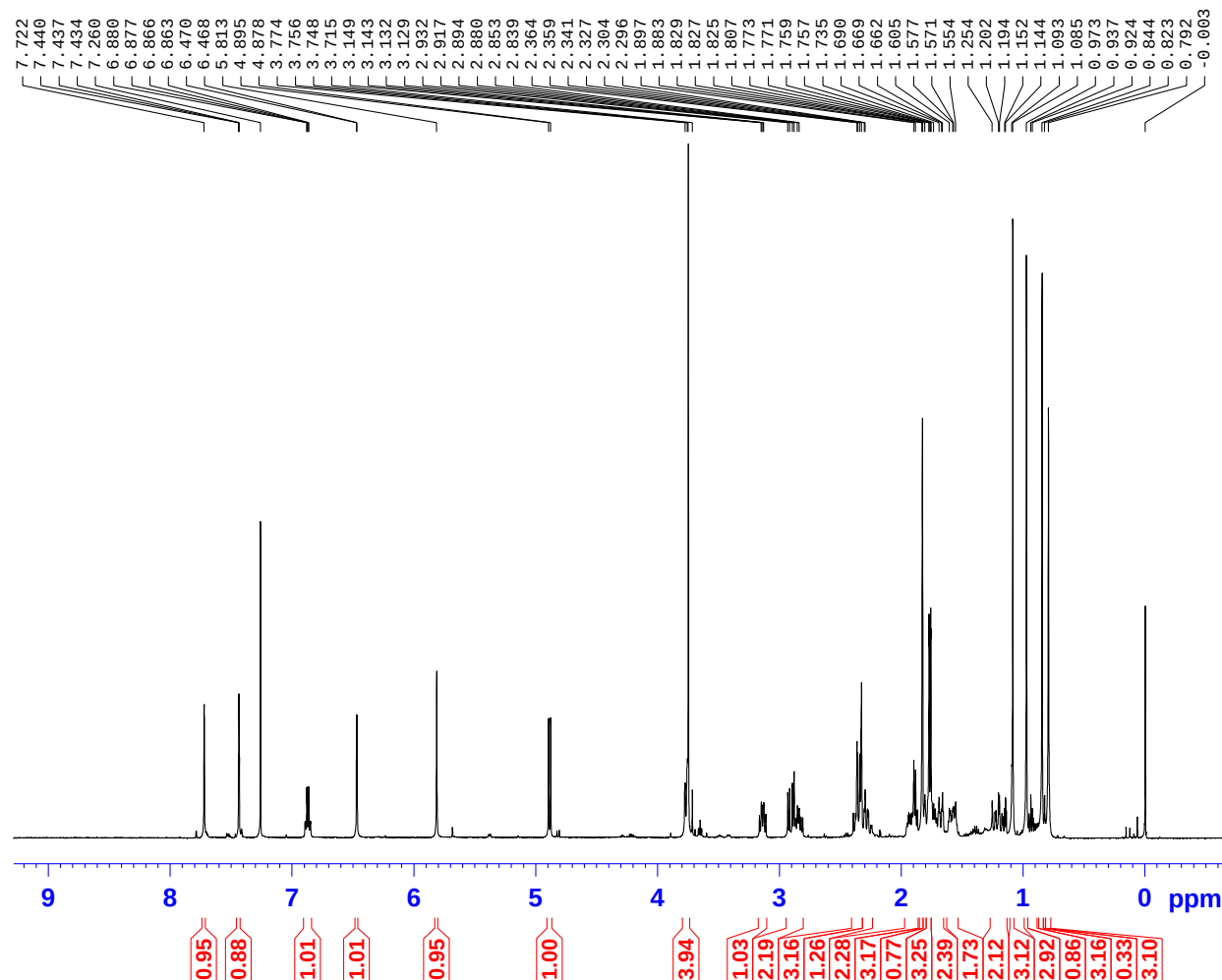
Jul-9 31 (1.866) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (9:33)

1: TOF MS ES+
3.95e4



¹H NMR (500 MHz) spectrum of granatumin D (**4**) in CDCl₃

1H NMR Spectrum of Jul-9
Solvent: CDCl₃



Current Data Parameters
NAME Jul-9
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090723
Time 18.28
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 456.1
DW 62.400 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.10 usec
PL1 -1.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (125 MHz) spectrum of granatumin D (4) in CDCl_3

^{13}C NMR Spectrum of Jul-9
Solvent: CDCl_3



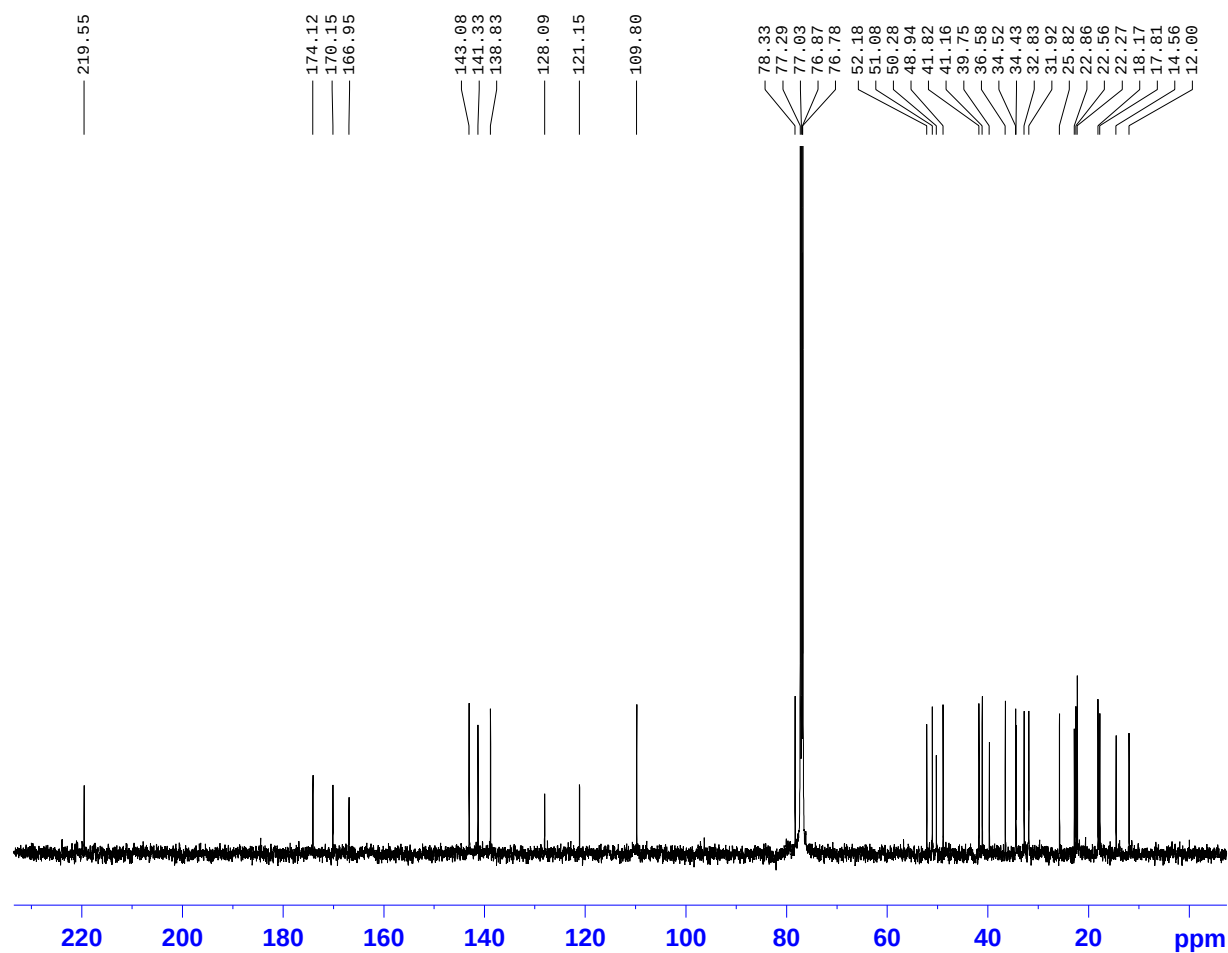
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NAME Jul-9
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090723
Time 18.30
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PROBHD 5 mm PABBO BB-
PULPROG ph_zpgpg45
TD 65536
SOLVENT CDCl_3
NS 3609
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 6502
DW 16.500 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^{13}C
P1 6.85 usec
PL1 -1.00 dB
SF01 125.7719996 MHz

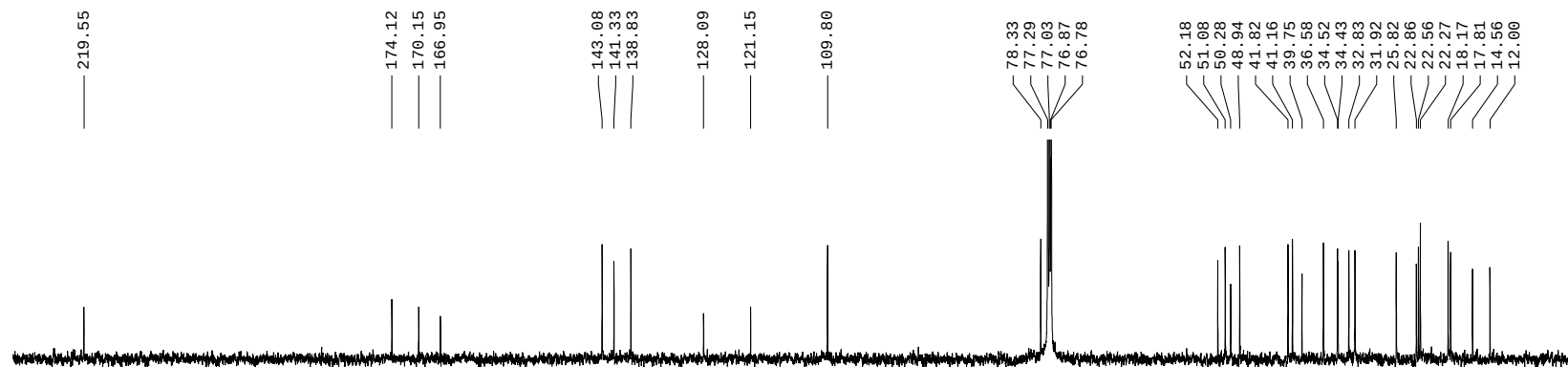
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 86.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577871 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

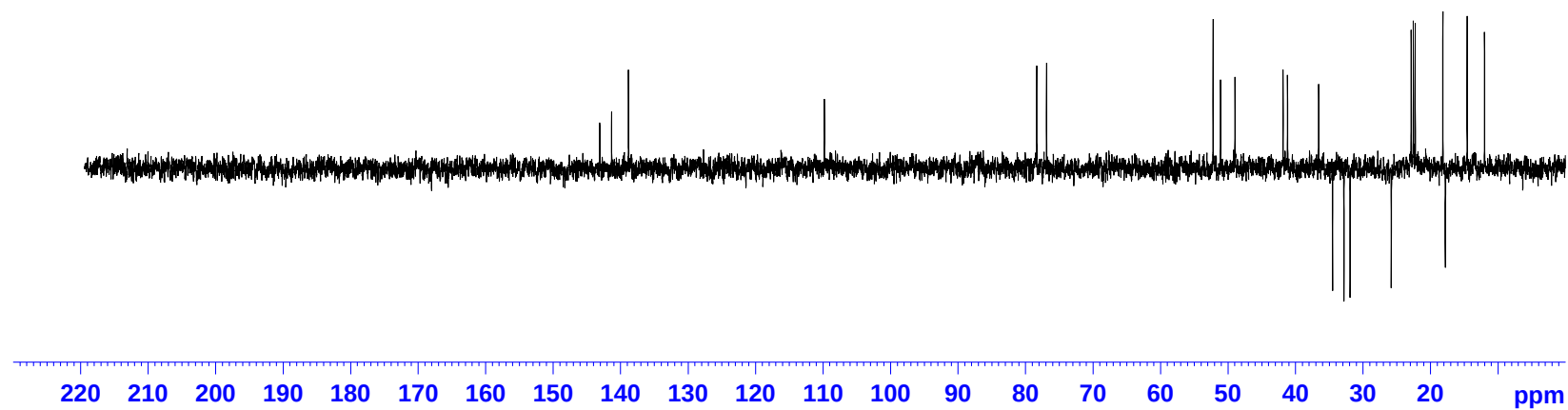


^{13}C NMR (125 MHz) and DEPT spectra of granatumin D (**4**) in CDCl_3

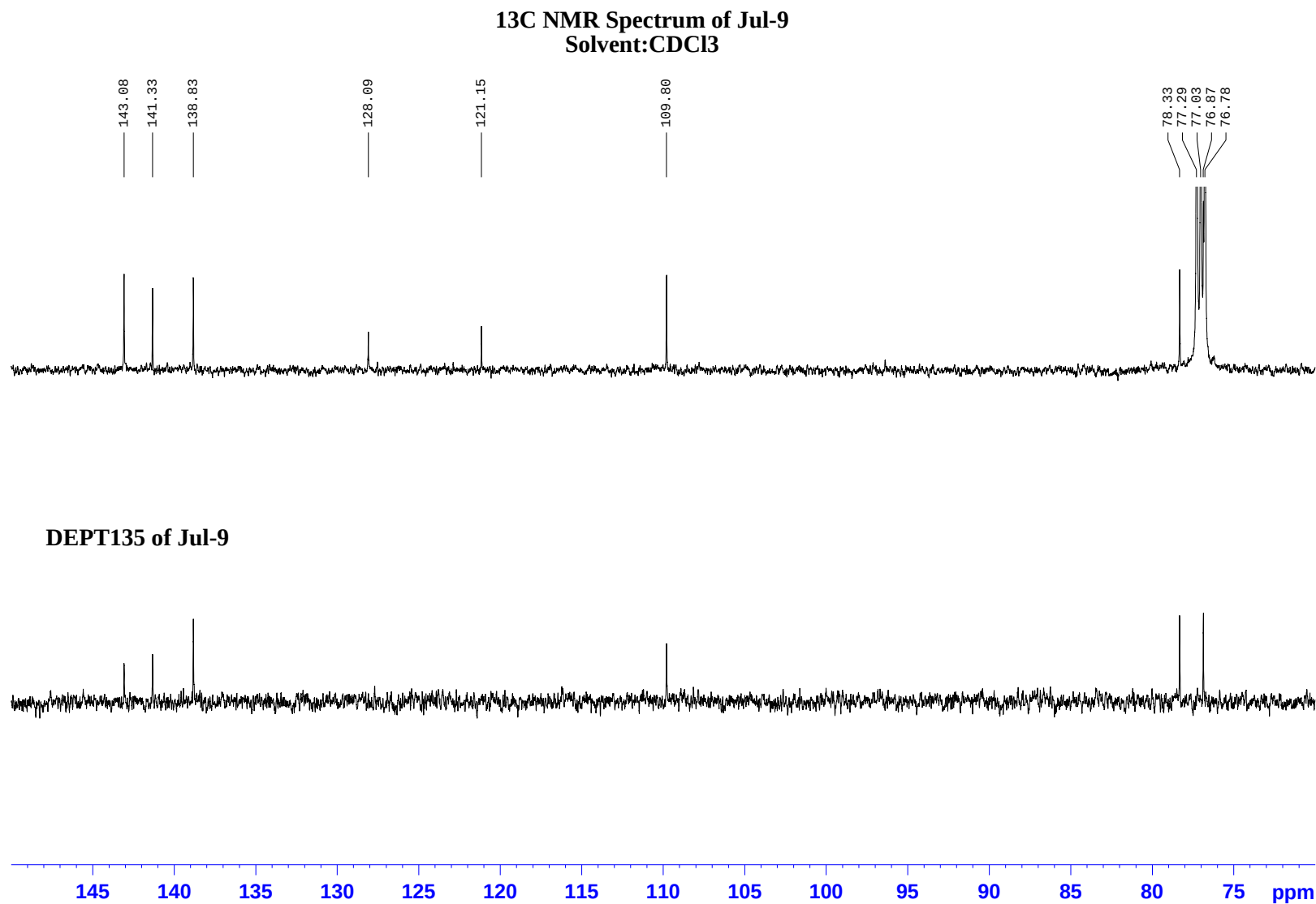
^{13}C NMR Spectrum of Jul-9
Solvent: CDCl_3



DEPT135 of Jul-9

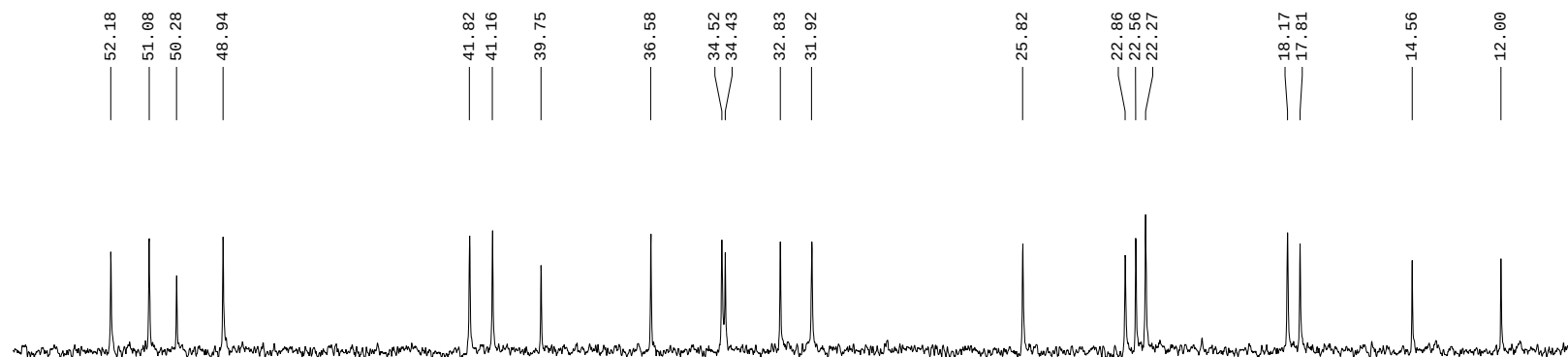


Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin D (**4**) in CDCl_3

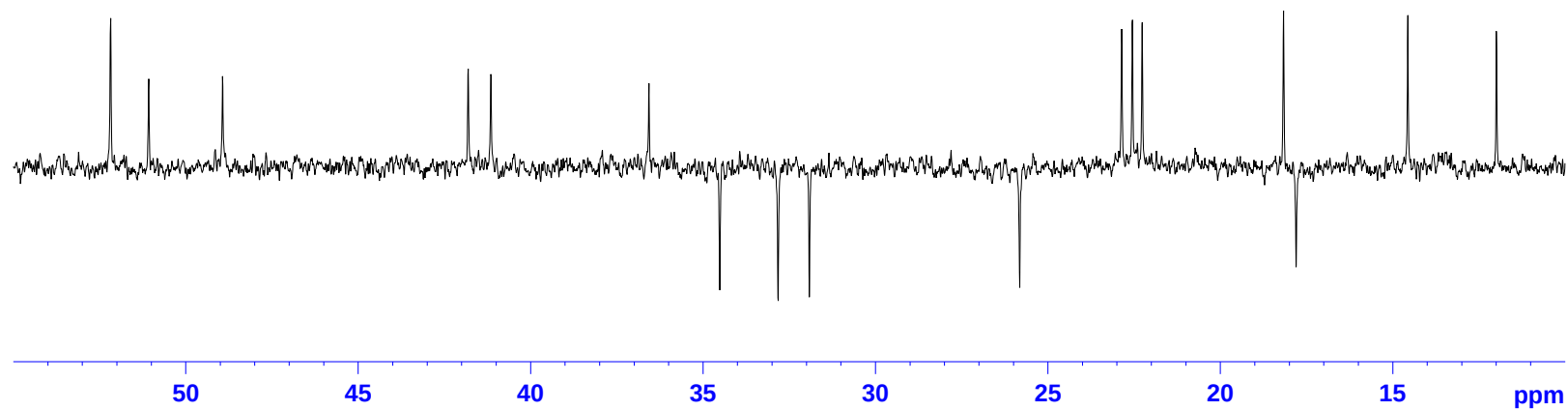


Enlarged segment of ^{13}C NMR (125 MHz) and DEPT spectra of granatumin D (**4**) in CDCl_3

^{13}C NMR Spectrum of Jul-9
Solvent: CDCl_3



DEPT135 of Jul-9

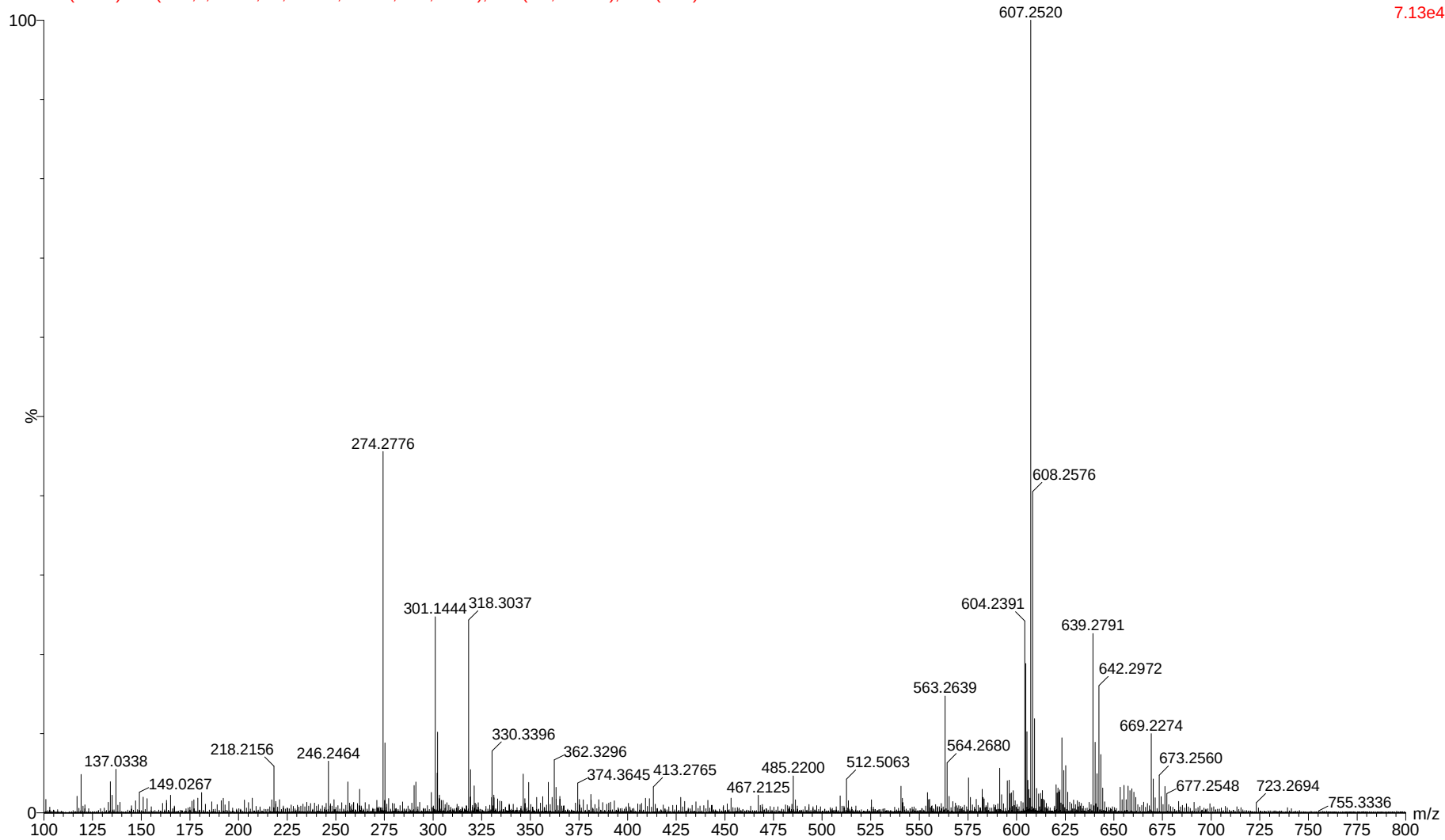


HR-TOFMS of granatumin E (5)

MS+

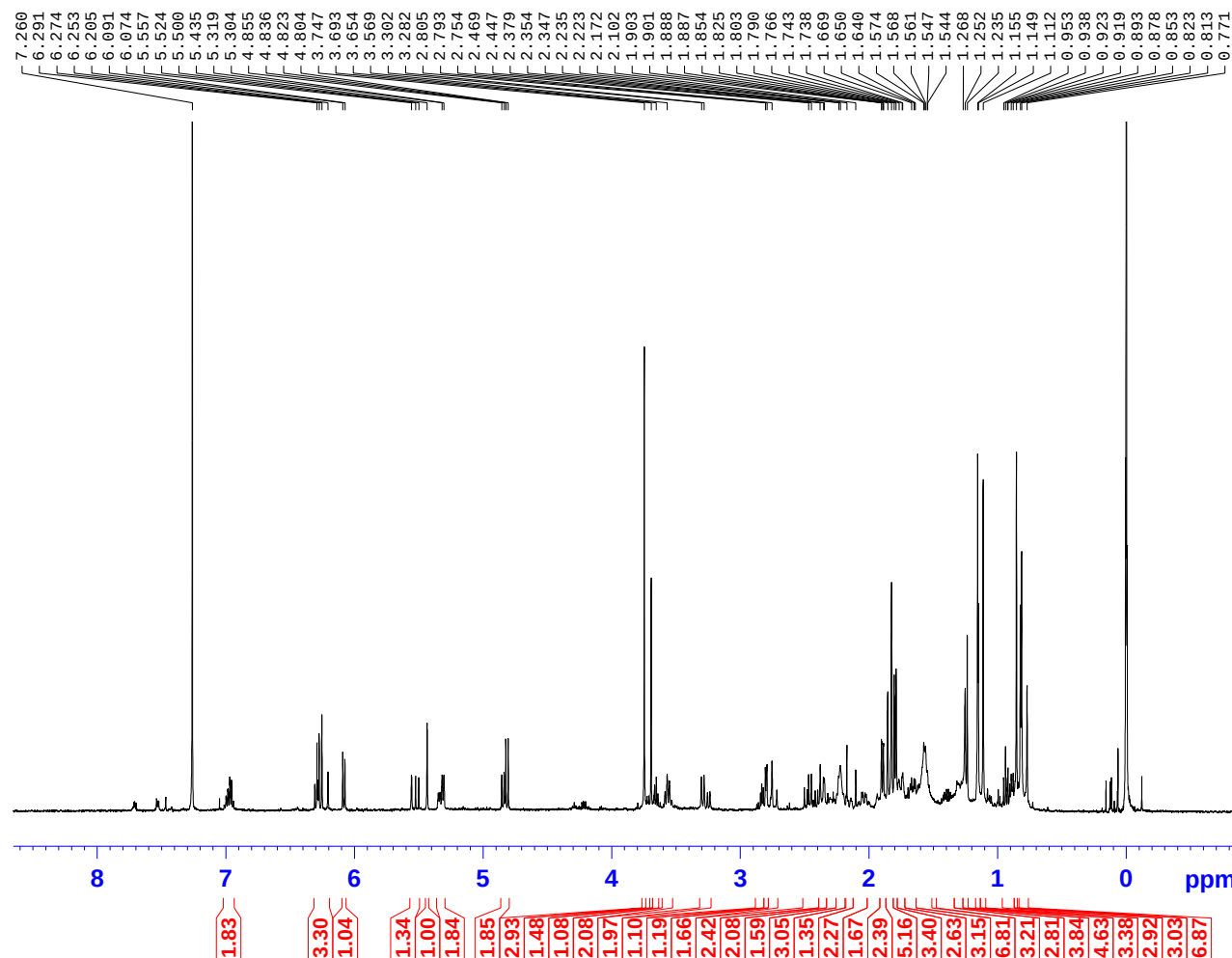
Jul-8 26 (1.559) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (7:32)

1: TOF MS ES+
7.13e4



^1H NMR (500 MHz) spectrum of granatumin E (5) in CDCl_3

1H NMR Spectrum of Jul-8
Solvent: CDCl_3



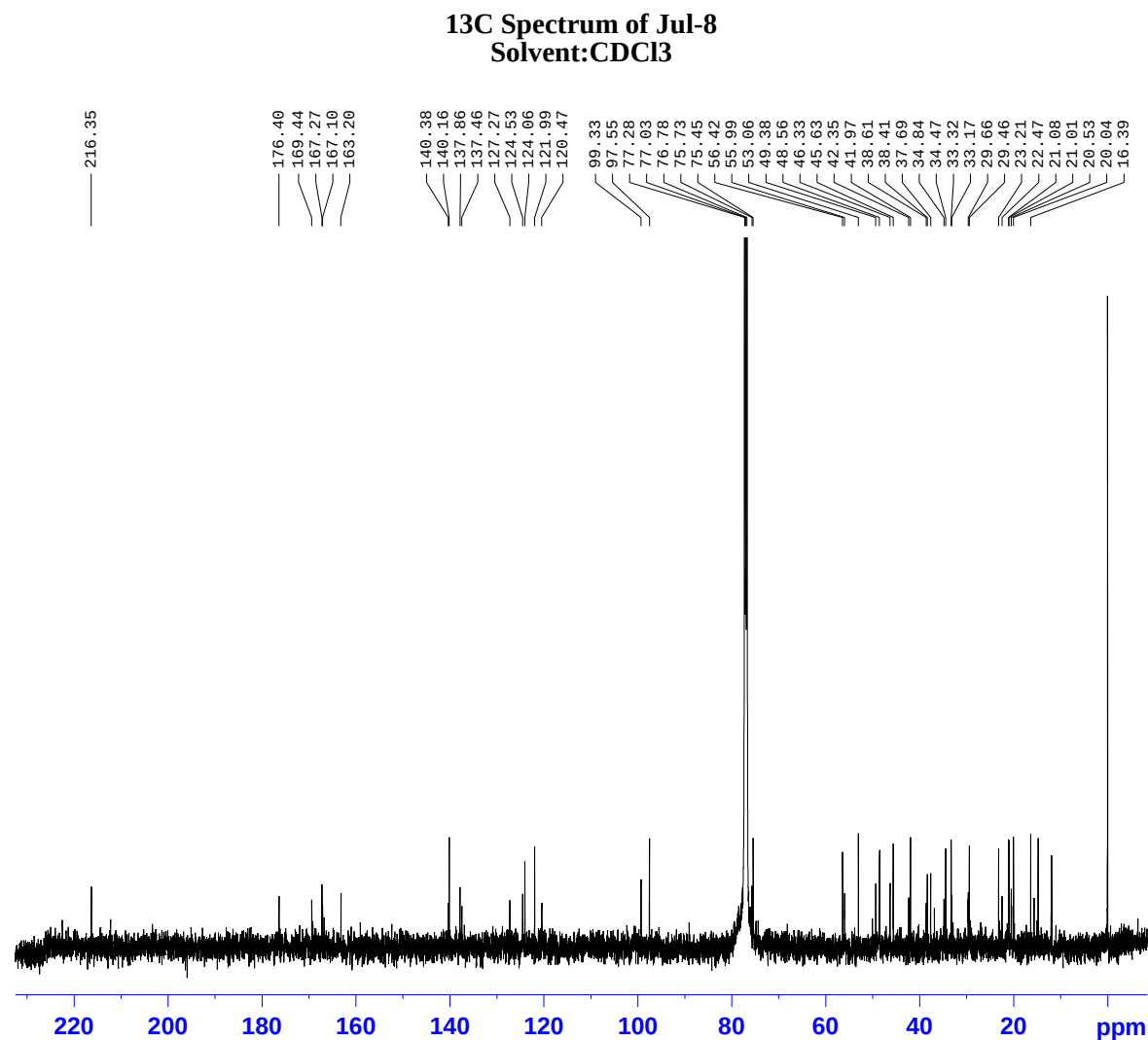
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EXPNO 1
PROCNO 1

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Time 10.47
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PULPROG zg30
TD 65536
SOLVENT CDCl_3
NS 32
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 574.7
DW 62.400 usec
DE 6.00 usec
TE 298.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^1H
P1 9.10 usec
PL1 -1.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 0.20

^{13}C NMR (125 MHz) spectrum of granatumin E (5) in CDCl_3



Current Data Parameters
NAME Jul-8
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090720
Time 9.52
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG ph_zpgpg45
TD 65536
SOLVENT CDCl_3
NS 26624
DS 2
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 6502
DW 16.500 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

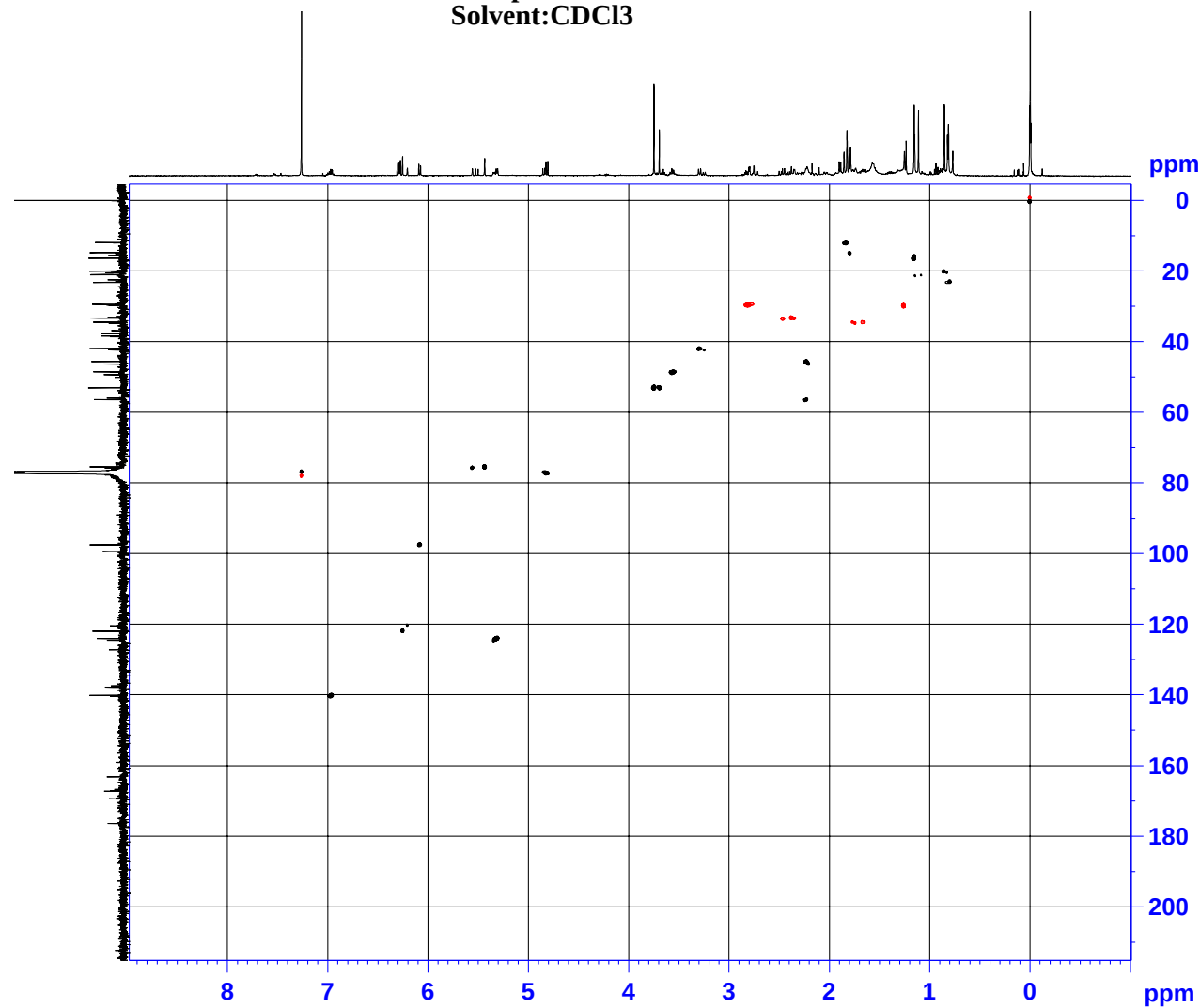
===== CHANNEL f1 =====
NUC1 ^{13}C
P1 6.85 usec
PL1 -1.00 dB
SF01 125.7718739 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 86.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577869 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

HSQCED spectra of granatumin E (5) in CDCl₃

HESCED Spectrum of Jul-8
Solvent:CDCl₃



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Current Data Parameters
NAME      Jul-8
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20090719
Time      10.58
INSTRUM   AV500
PROBHD    5 mm PABBO BB-
PULPROG   hsqcedetgpsi2
TD        1024
SOLVENT   CDCl3
NS        24
DS        16
SWH        5000.000 Hz
FIDRES     4.882813 Hz
AQ         0.1025500 sec
RG         25000
DW         100.000 usec
DE         6.00 usec
TE         298.1 K
CST2      145.0000000
d0         0.0000300 sec
D1         1.50000000 sec
d4         0.00172414 sec
d11        0.03000000 sec
D16        0.00020000 sec
D21        0.00344828 sec
D24        0.00086207 sec
DELTA      0.00222488 sec
DELTA1     0.00120800 sec
DELTA2     0.00147414 sec
DELTA3     0.00000207 sec
DELTA4     0.00027414 sec
IN0        0.00001807 sec
MCREST     0.00000000 sec
MCWRK      0.25000051 sec
STICNT     128

===== CHANNEL f1 =====
NUC1       1H
P1         9.10 usec
P2         18.20 usec
P2B        0.00 usec
PL1        -1.00 dB
SFO1       500.1320005 MHz

===== CHANNEL f2 =====
CPDPRG2    garp
NUC2       13C
P3         7.00 usec
P4         14.00 usec
P14        500.00 usec
PCPD2      66.00 usec
PL0        120.00 dB
PL2        -1.00 dB
PL12       18.40 dB
SFO2       125.7709030 MHz
SP3        10.26 dB
SPHMA3     Crp60,0.5,20.1
SPOFF3     0.00 Hz

F1 - Acquisition parameters
ND0        2
TD         256
SFO1       125.7711 MHz
FIDRES     108.056709 Hz
SW         219.944 ppm
FMODE      Echo-Antiecho

F2 - Processing parameters
SI         1024
SF         500.1300102 MHz
WDW        COSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        echo-antiecho
SF         125.7577672 MHz
WDW        COSINE
SSB        2
LB         0.00 Hz
GB         0
```

HMBC spectra of granatumin E (5) in CDCl₃

HMBC Spectrum of Jul-8
Solvent:CDCl₃



Current Data Parameters
NAME Jul-8
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090724
Time 22.15
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG hmbcgp1pndqf
TD 1024
SOLVENT CDCl₃
NS 192
DS 16
SWH 5000.000 Hz
FIDRES 4.892813 Hz
AQ 0.1025500 sec
RG 29193
DW 100.000 usec
DE 6.00 usec
TE 298.1 K
CNST2 145.0000000
CNST13 10.0000000
d0 0.00000300 sec
D1 1.50000000 sec
d2 0.00344628 sec
d6 0.05000000 sec
D16 0.00020000 sec
IN0 0.00001656 sec
MCRCST 0.00000000 sec
MCWRK 1.50000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.10 usec
p2 18.20 usec
PL1 -1.00 dB
SF01 500.1320005 MHz

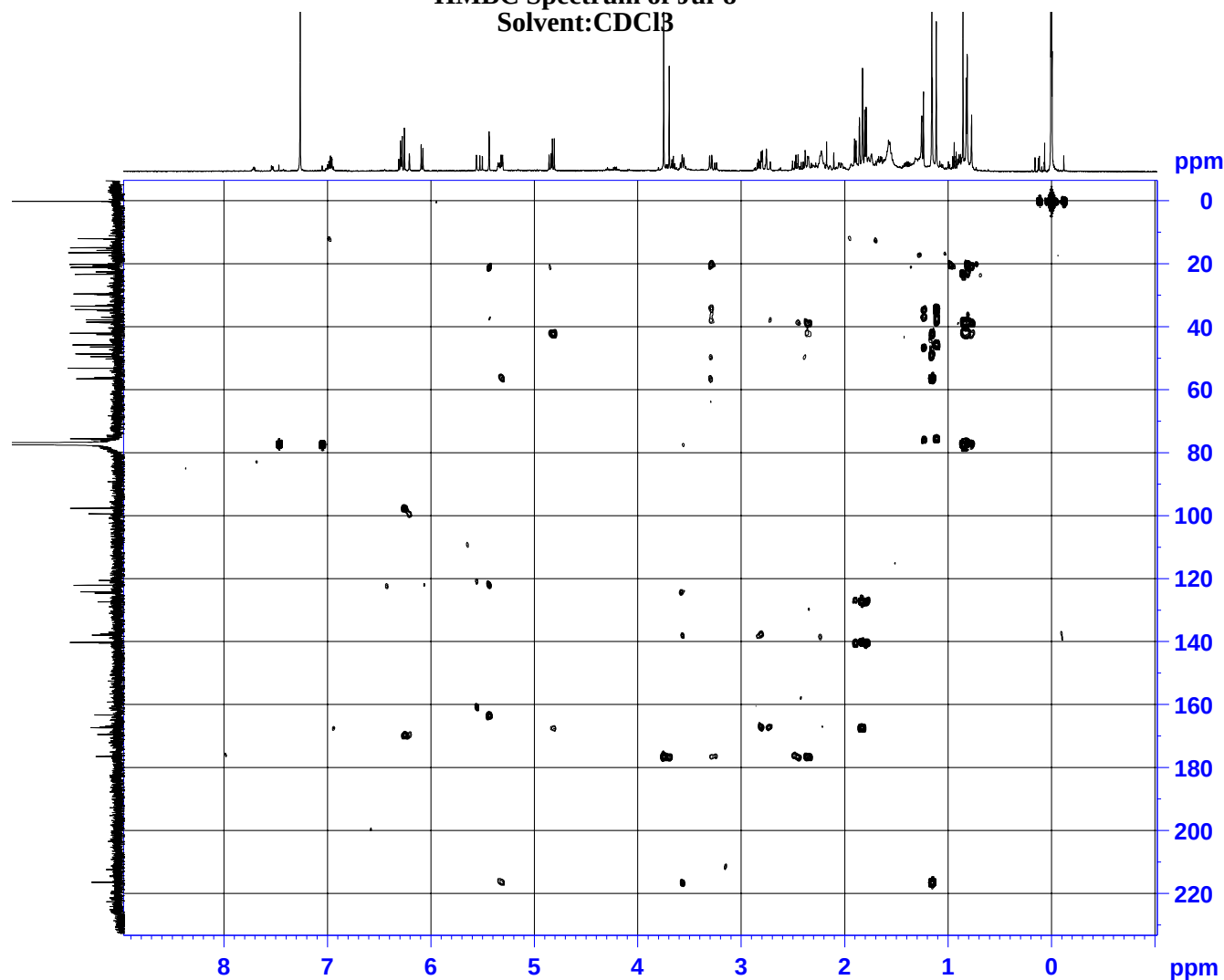
===== CHANNEL f2 =====
NUC2 13C
P3 7.00 usec
PL2 -1.00 dB
SF02 125.7719996 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 128
SF01 125.772 MHz
FIDRES 235.849060 Hz
SW 240.027 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 500.1300130 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 125.7577538 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

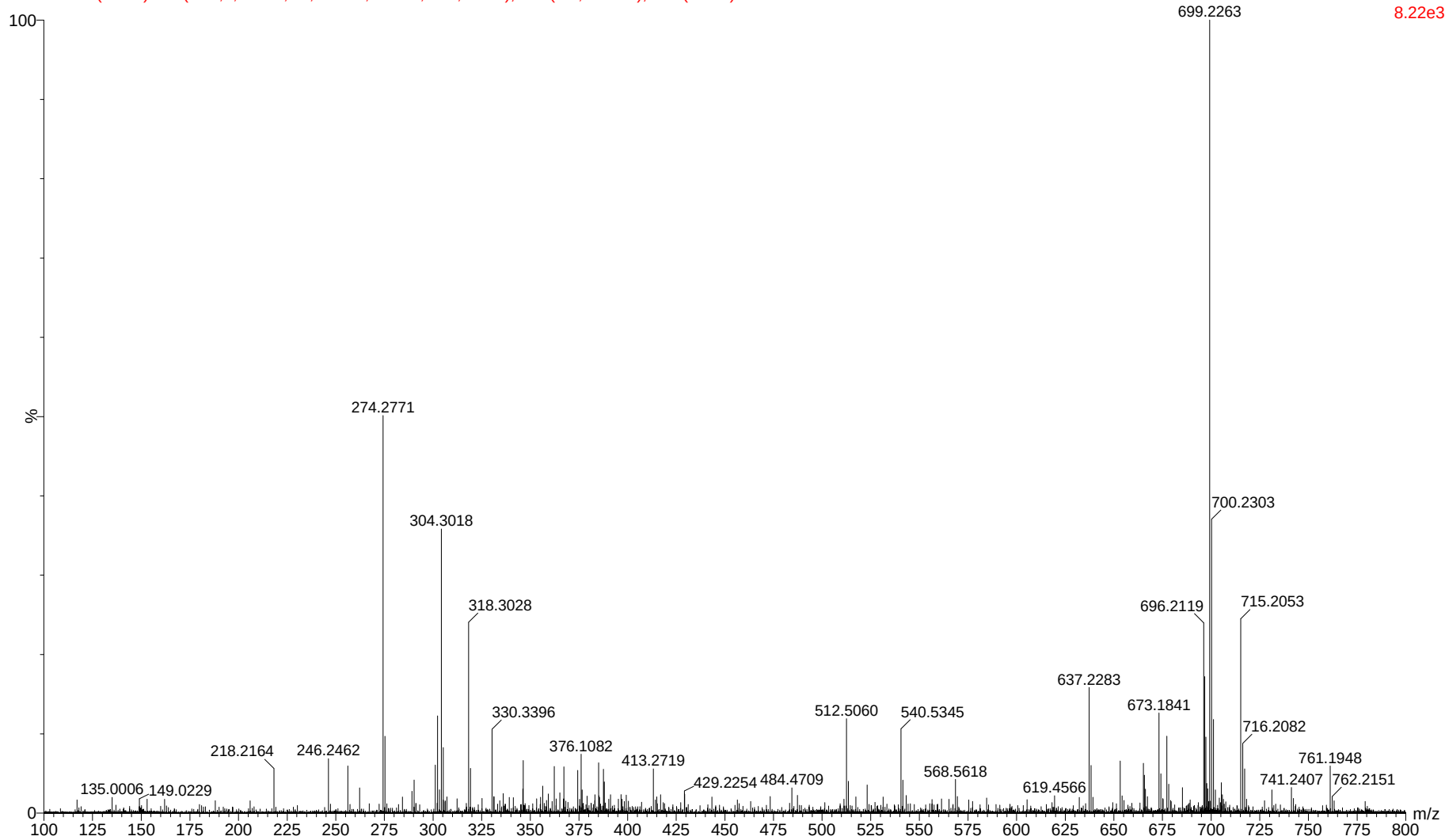


HR-TOFMS of granatumin F (6)

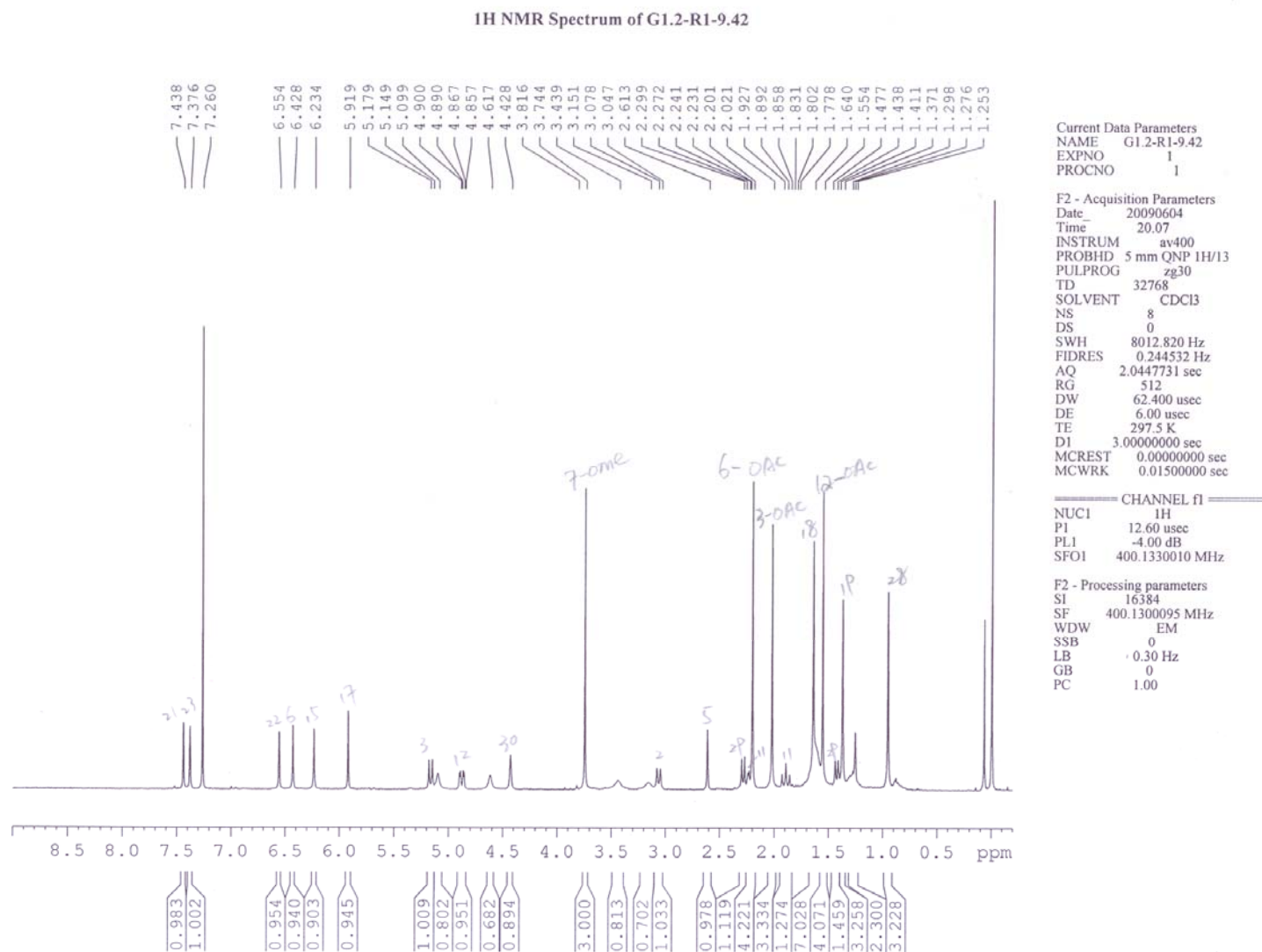
MS+

xiaobo-6 33 (1.769) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (29:36)

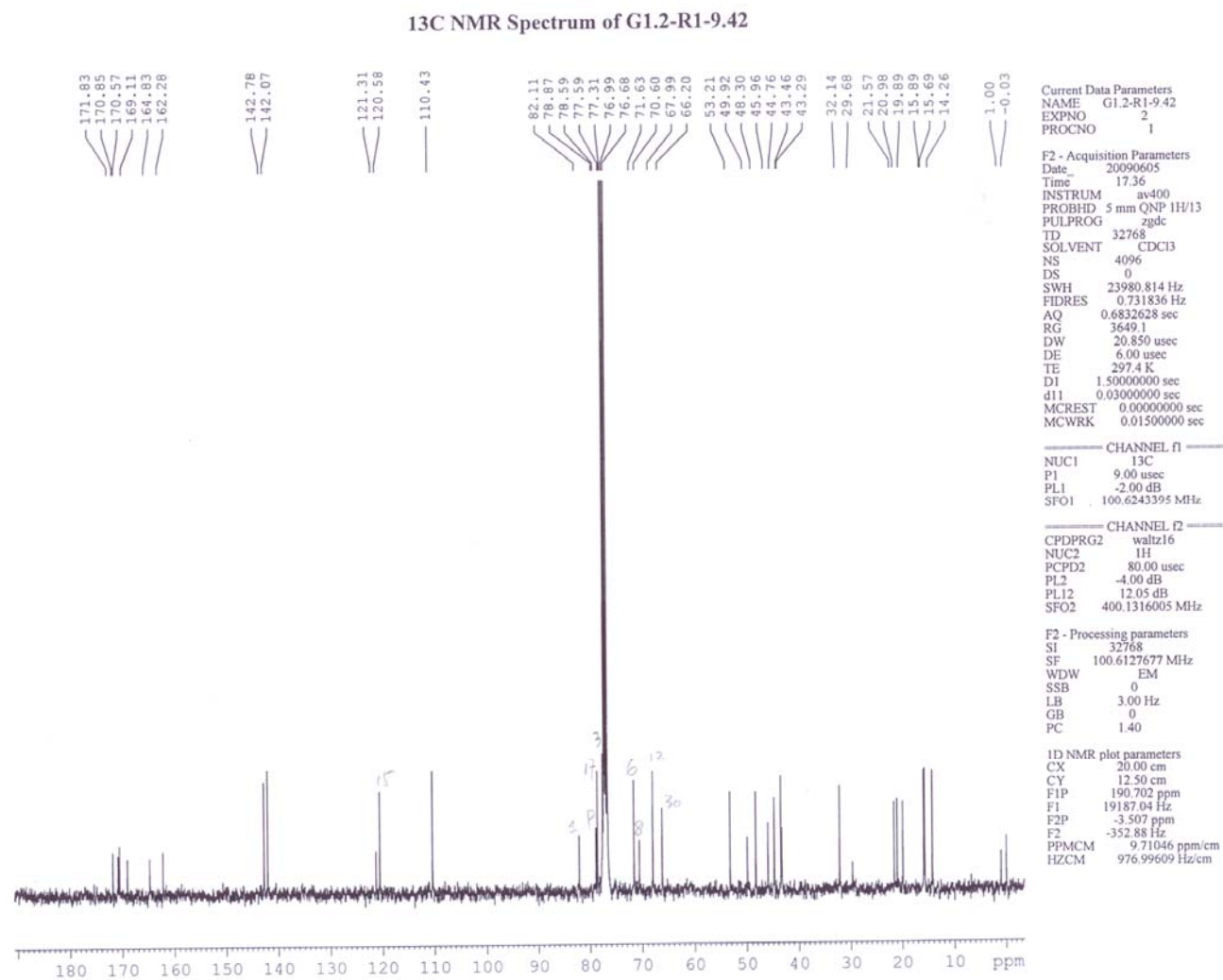
1: TOF MS ES+
8.22e3



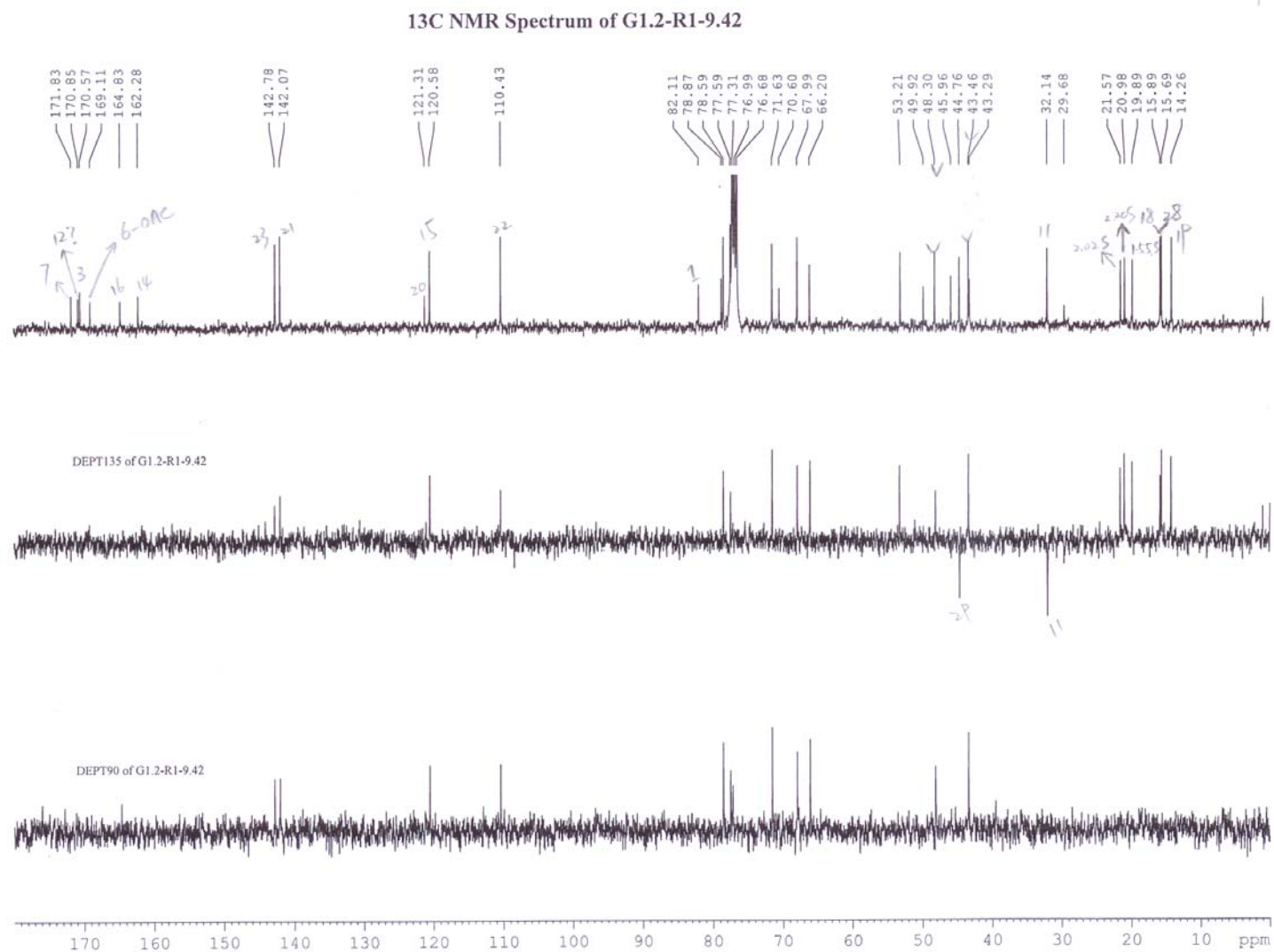
^1H NMR (400 MHz) spectrum of granatumin F (**6**) in CDCl_3



^{13}C NMR (100 MHz) spectrum of granatumin F (**6**) in CDCl_3



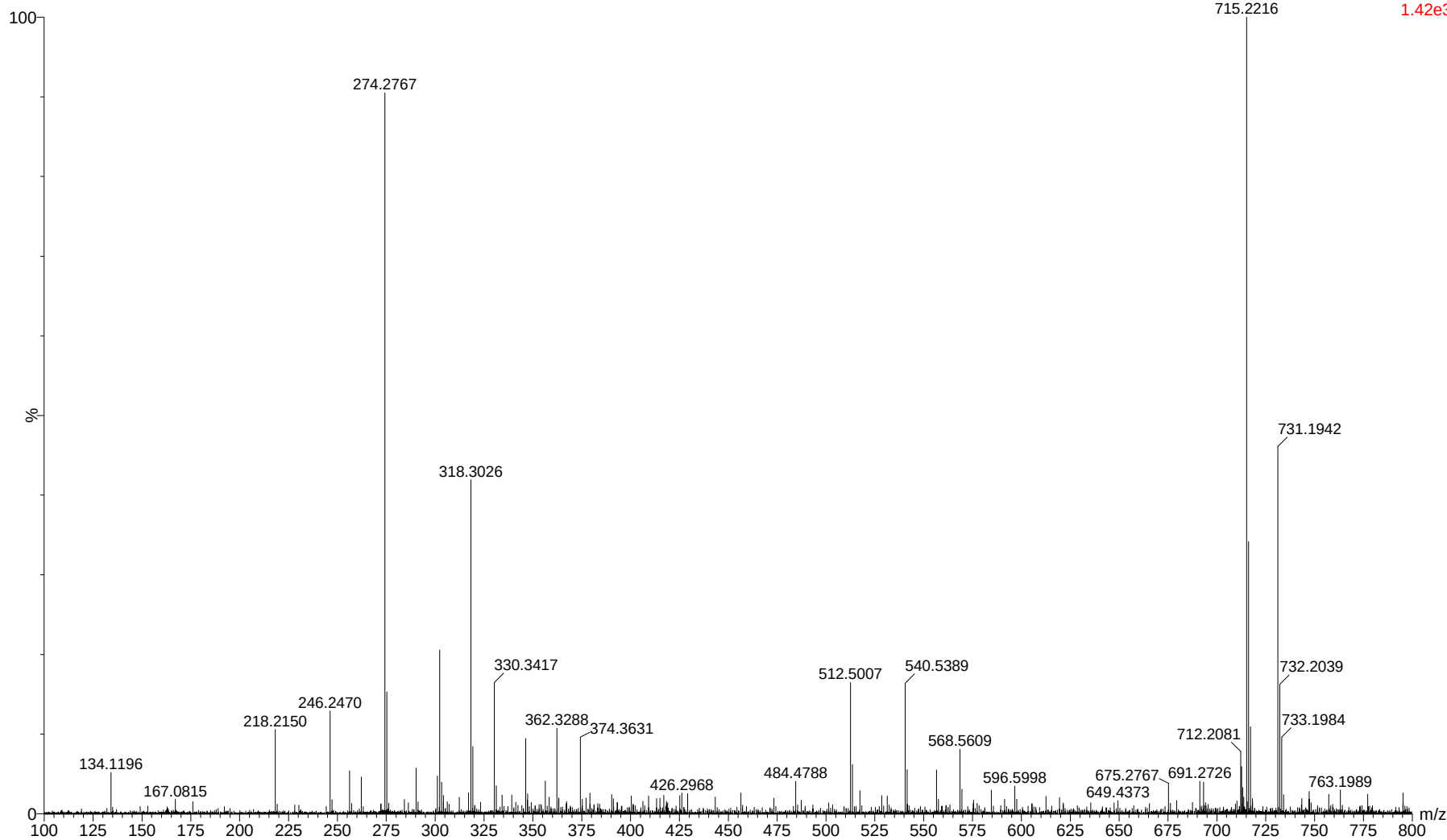
^{13}C NMR (100 MHz) and DEPT spectra of granatumin F (**6**) in CDCl_3



HR-TOFMS of granatumin G (7)

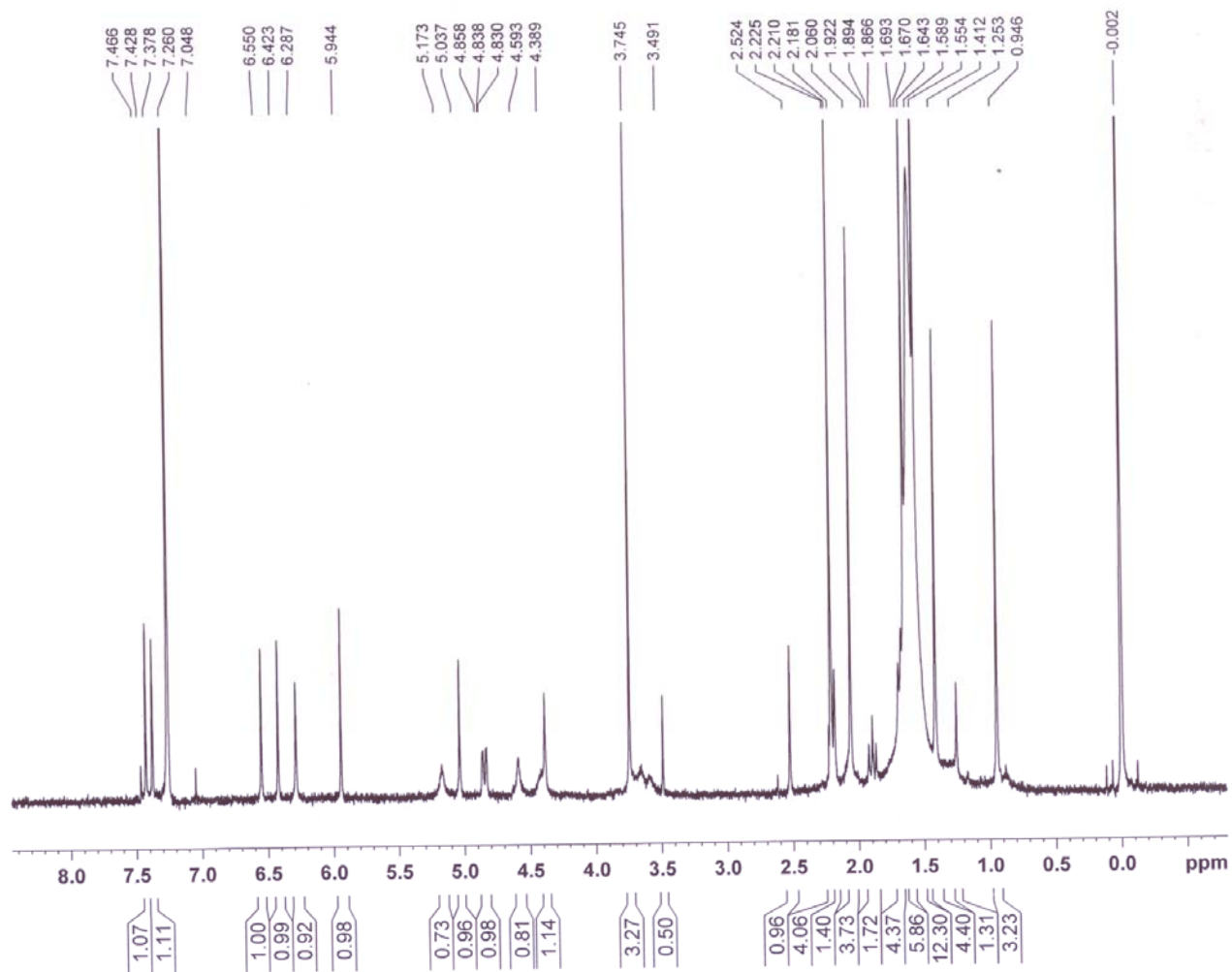
MS+

Jul-4 28 (1.683) AM (Cen,2, 80.00, Ar,6000.0,556.28,0.70,LS 10); Sm (Mn, 2x1.00); Cm (27:32)



^1H NMR (400 MHz) spectrum of granatumin G (**7**) in CDCl_3

^1H NMR Spectrum of G1.2-R1-9.41



Current Data Parameters
NAME yangxiaobo-G1.2-R1-9.41
EXPNO 1
PROCNO 1

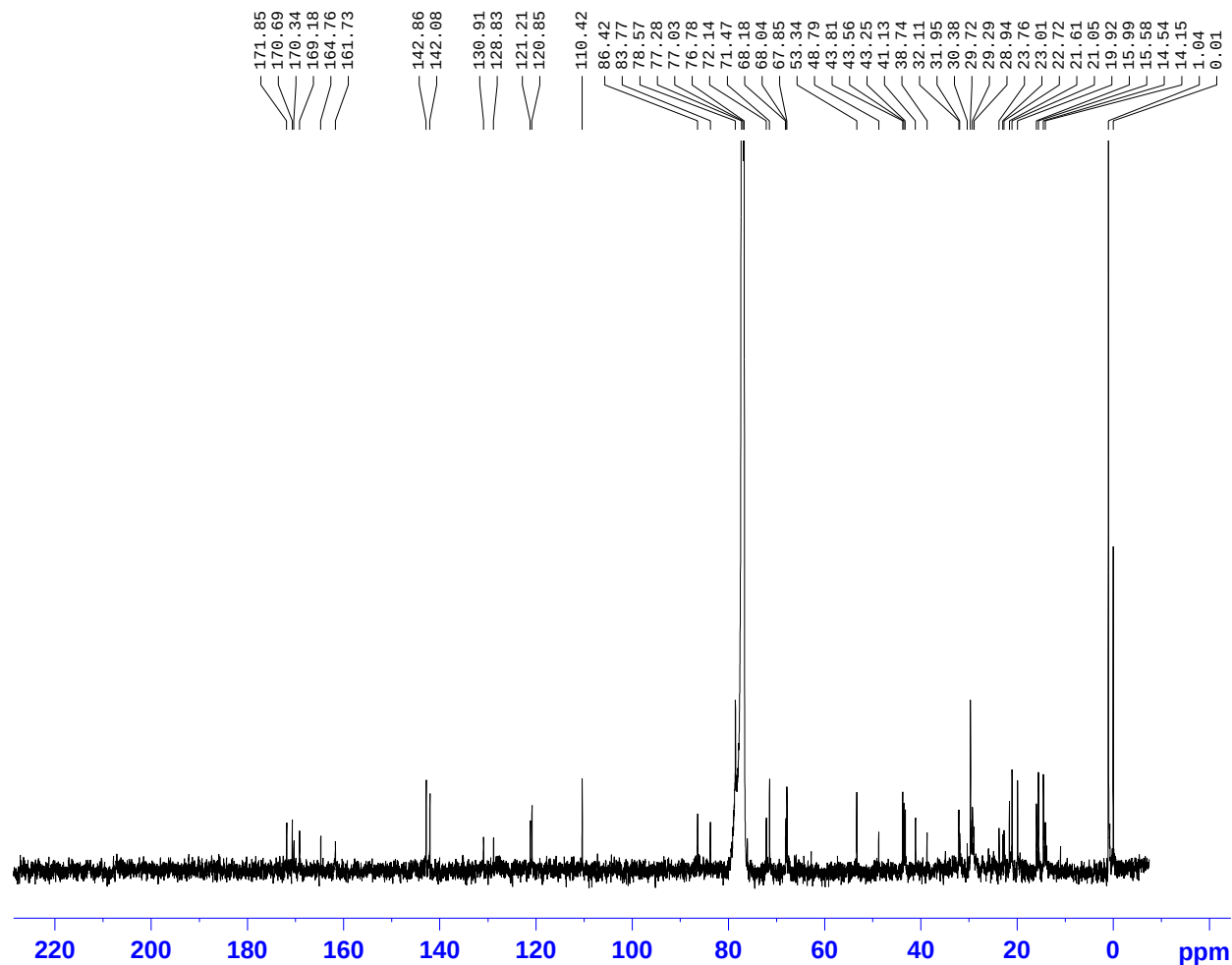
F2 - Acquisition Parameters
Date_ 20090401
Time_ 16.40
INSTRUM av500
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl_3
NS 17
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 200
DW 50.000 usec
DE 6.00 usec
TE 297.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 ^1H
P1 10.50 usec
PL1 0.00 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 65536
SF 500.1300143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

^{13}C NMR (400 MHz) spectrum of granatumin G (7) in CDCl_3

^{13}C NMR Spectrum of Jul-4
Solvent: CDCl_3



Current Data Parameters

NAME Jul-4
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20090725
Time 17.09
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG ph_zgpg45
TD 65536
SOLVENT CDCl_3
NS 61440
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 1824.6
DW 16.500 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

NUC1 ^{13}C
P1 6.85 usec
PL1 -1.00 dB
SF01 125.7719996 MHz

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

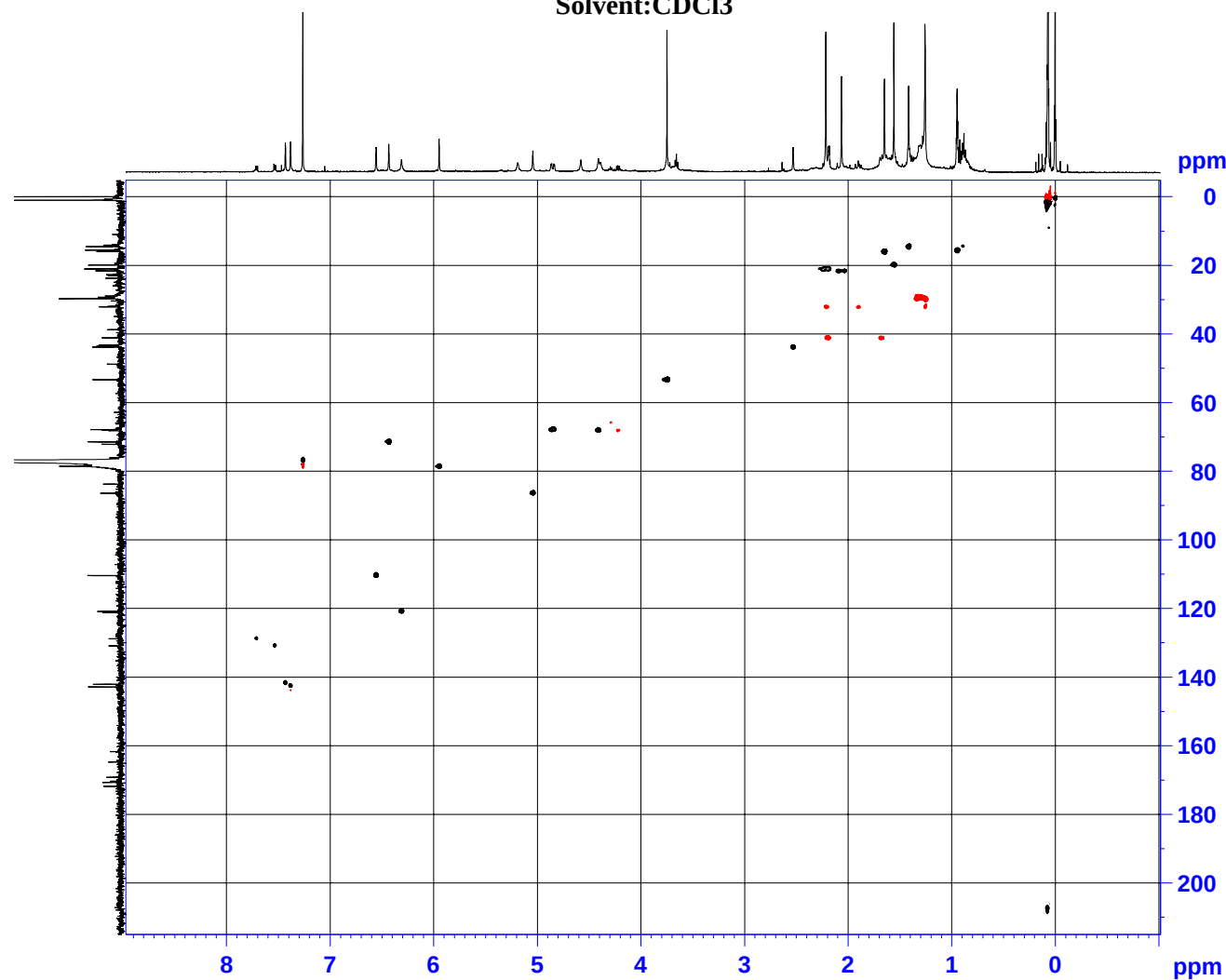
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 86.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 21.00 dB
SF02 500.1320005 MHz

F2 - Processing parameters

SI 32768
SF 125.7577879 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

HSQCED spectrum of granatumin G (7) in CDCl₃

HSQCED Spectrum of Jul-4
Solvent:CDCl₃



Current Data Parameters
NAME Jul-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090722
Time 17.12
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG hsqcedetgps15p2
TD 1024
SOLVENT DMSO
NS 48
DS 16
SWH 5000.000 Hz
FIDRES 4.862613 Hz
AQ 0.1025500 sec
RG 26000
DW 100.000 usec
DE 6.00 usec
TE 298.1 K
CHST2 145.000000
d0 0.0000300 sec
d1 1.5000000 sec
d4 0.00172414 sec
d11 0.03000000 sec
d16 0.00020000 sec
d21 0.00344020 sec
D24 0.00080207 sec
DELTA 0.00222400 sec
DELTA1 0.00120000 sec
DELTA2 0.00147414 sec
DELTA3 0.00080207 sec
DELTA4 0.00027414 sec
IN0 0.00018007 sec
MCREST 0.00000000 sec
MOMK 0.25000001 sec
ST1CNT 128

===== CHANNEL f1 =====
NUC1 1H
P1 9.10 usec
p2 18.20 usec
P28 0.00 usec
PL1 -1.00 dB
SFO1 500.1320005 MHz

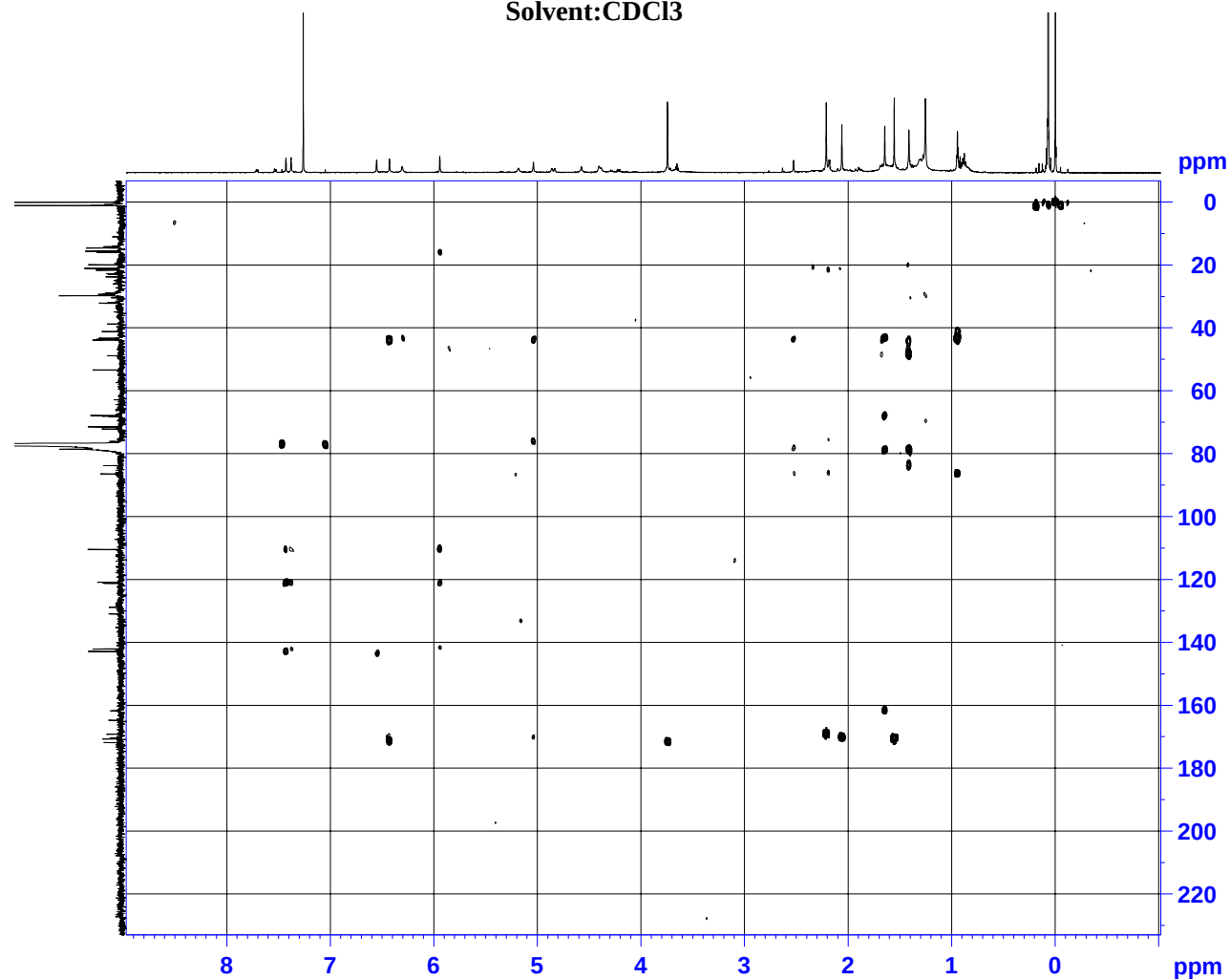
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 7.00 usec
p4 14.00 usec
P14 500.00 usec
PCPD2 66.00 usec
PL0 120.00 dB
PL2 -1.00 dB
PL12 18.40 dB
SFO2 125.7709936 MHz
SP3 10.26 dB
SPNAM3 Crp60, 0.5, 20.1
SPOFF3 0.00 Hz

F2 - Processing parameters
SI 1024
SF 500.1308135 MHz
WDW QSINE
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
WC2 echo-antilecho
SF 125.7577879 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

HMBC spectrum of granatumin G (7) in CDCl₃

HMBC Spectrum of Jul-4
Solvent:CDCl₃



Current Data Parameters
NAME Jul-4
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090722
Time 22.47
INSTRUM AV500
PROBHD 5 mm PABBO BB-
PULPROG hmbcggp1ndqf
TD 1024
SOLVENT DMSO
NS 128
DS 16
SWH 5000.000 Hz
FIDRES 4.882813 Hz
AQ 0.1025500 sec
RG 29193
DW 100.000 usec
DE 6.00 usec
TE 298.1 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 0.00001656 sec
MCREST 0.00000000 sec
MCWRK 1.50000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.10 usec
P2 19.20 usec
PL1 -1.00 dB
SF01 500.1320005 MHz

===== CHANNEL f2 =====
NUC2 13C
P3 7.00 usec
PL2 -1.00 dB
SF02 125.7719996 MHz

===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 128
SF01 125.772 MHz
FIDRES 235.849060 Hz
SW 240.027 ppm
FIRMODE QF

F2 - Processing parameters
SI 2048
SF 500.1300135 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 125.7577879 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0