

10.1071/CH14328_AC

©CSIRO 2015

Australian Journal of Chemistry 2015, 68 (6), 872-880

Supplementary Material

Synthesis and Bioevaluation of Novel Oxa-caged *Garcinia*

Xanthenes as anti-tumor agents

Guangqiang Miao,^A Junhai Ma,^A Kan Yang,^A Zhipeng Huang,^A Qinlan Gu,^B Yanjie Wang,^C Qinglong Guo,^{D,E} Qidong You,^{A,D} and Jinxin Wang^{A,F}

^AKey Laboratory of Drug Design and Optimization of Jiangsu Province, China Pharmaceutical University, Nanjing 210009, China.

^BHigher Vocational and Technical College, China Pharmaceutical University, Nanjing 210009, China.

^CCollege of Pharmacy, China Pharmaceutical University, Nanjing 210009, China.

^DState Key Laboratory of Natural Medicines, China Pharmaceutical University, Nanjing 210009, China.

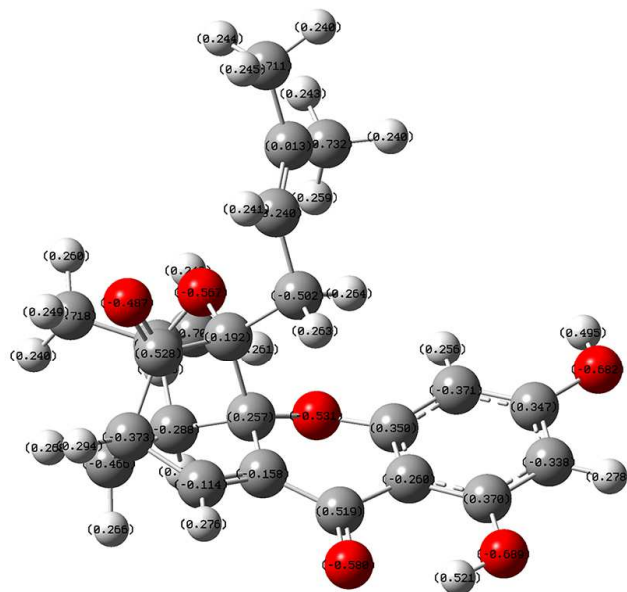
^EKey Laboratory of Carcinogenesis and Intervention of Jiangsu Province, China Pharmaceutical University, Nanjing 210009, China.

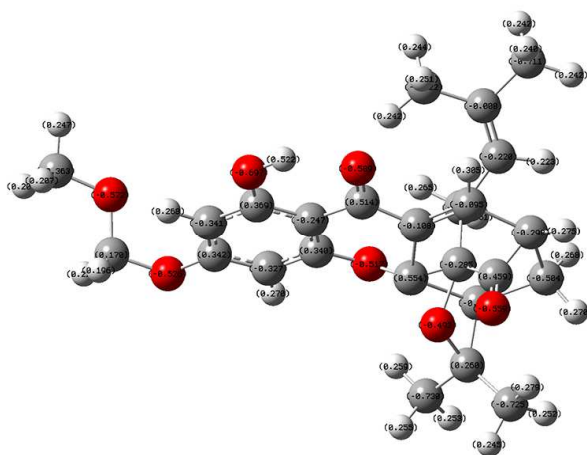
^FCorresponding author. Email: jinxinwang@163.com

p1 Cover page

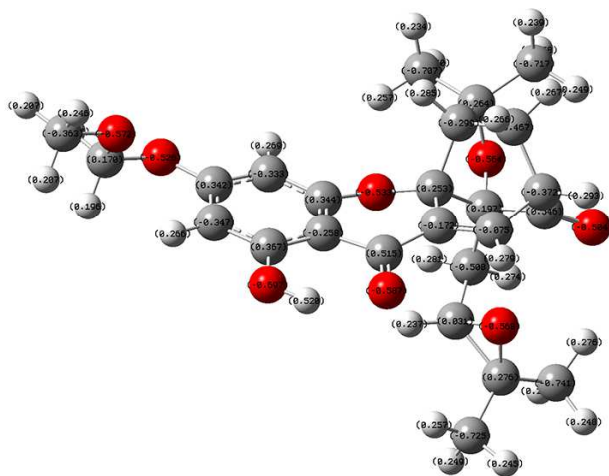
p2-10 The NBO charge distributions of compounds **1-8** and **GA**

p11-p25 Copies of ¹H-NMR, ¹³C-NMR and MS spectra for compounds **2-8**

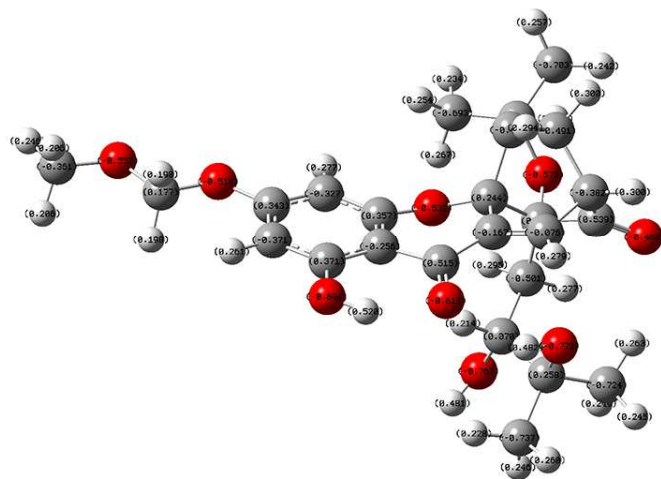




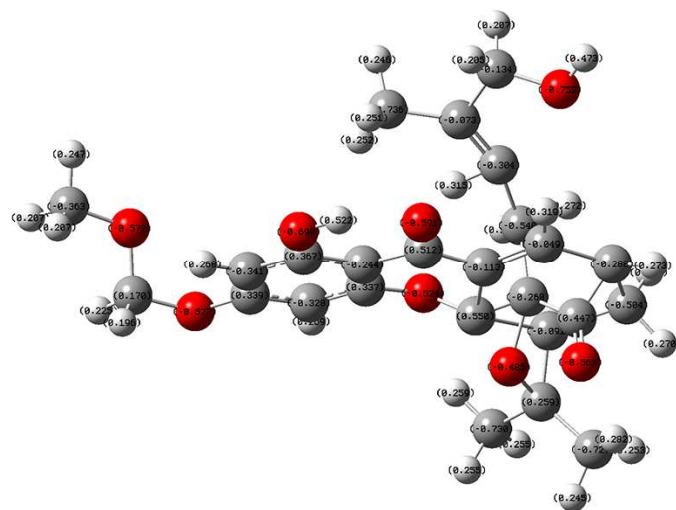
The NBO charge distributions of compounds **2**



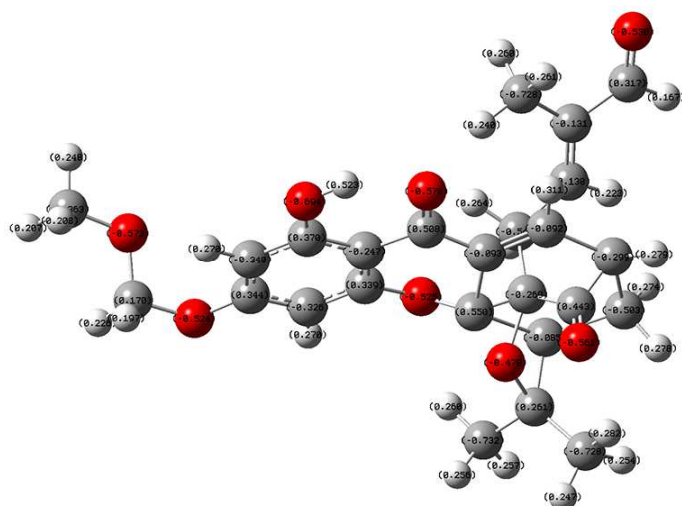
The NBO charge distributions of compounds **3**



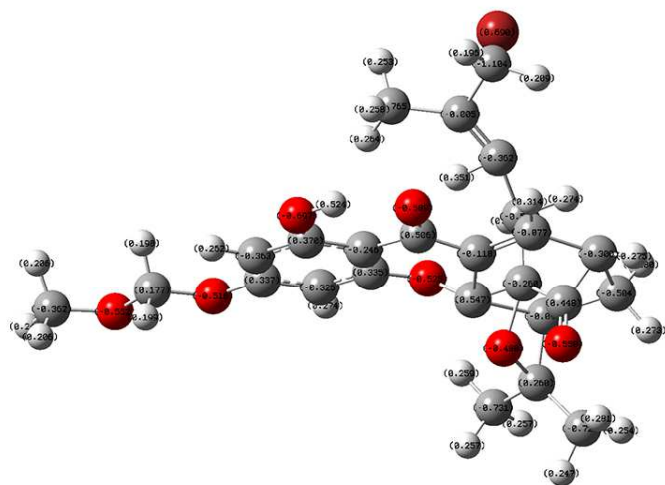
The NBO charge distributions of compounds **4a**



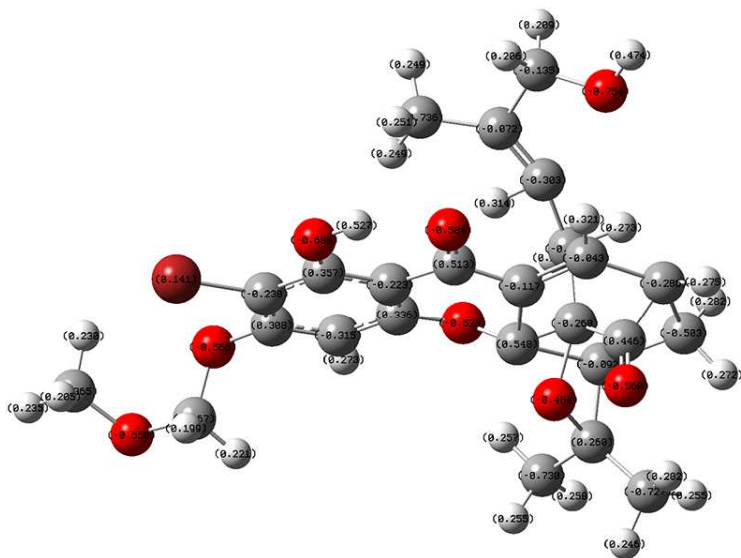
The NBO charge distributions of compounds 5



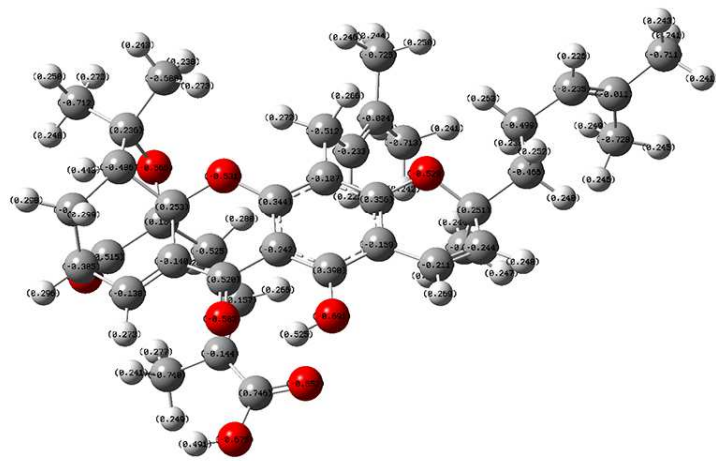
The NBO charge distributions of compounds 6



The NBO charge distributions of compounds **7**

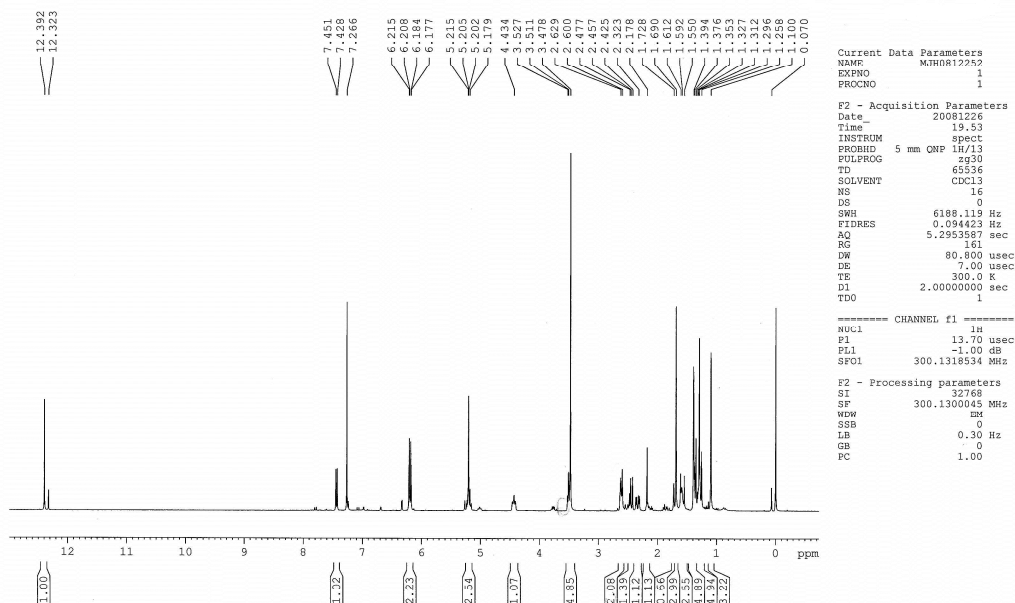


The NBO charge distributions of compounds **8**



The NBO charge distributions of GA

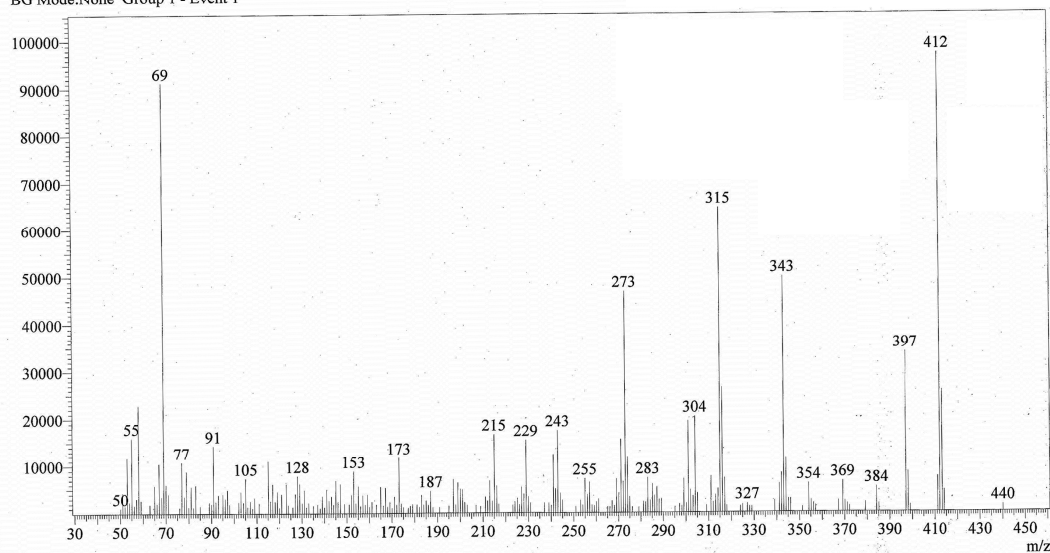
MJH0812252 H1-NMR CDCL3 303K AV-300



¹H-NMR spectrum of compound 2

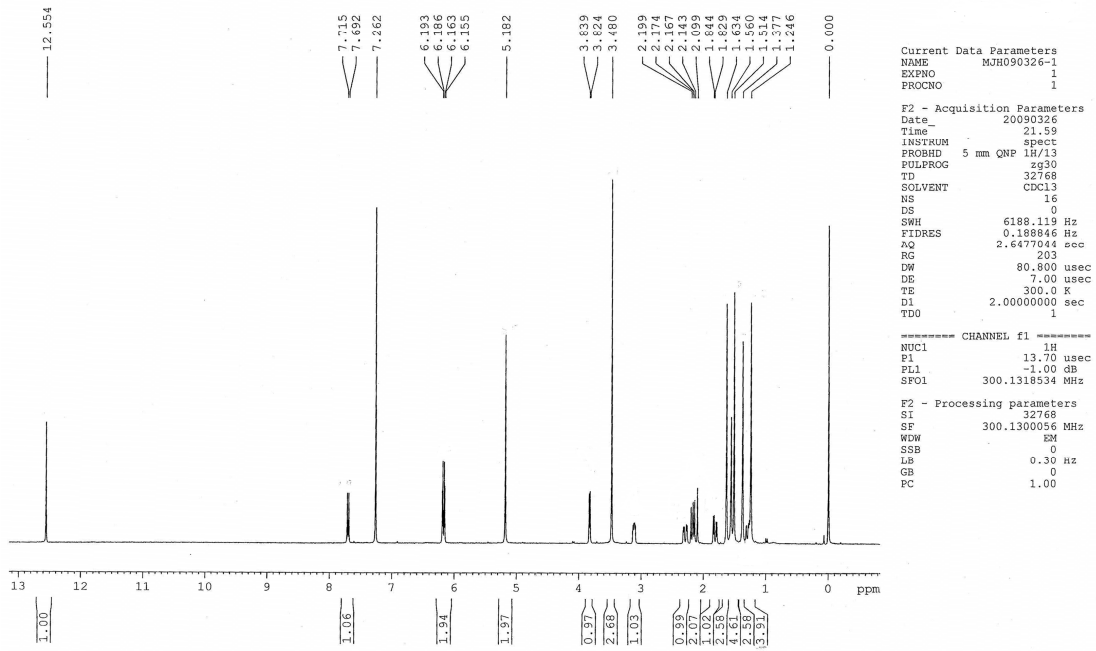
Spectrum

Line#:1 R.Time:1.933(Scan#:65)
 MassPeaks:228
 RawMode:Single 1.933(65) BasePeak:412.15(97014)
 BG Mode:None Group 1 - Event 1

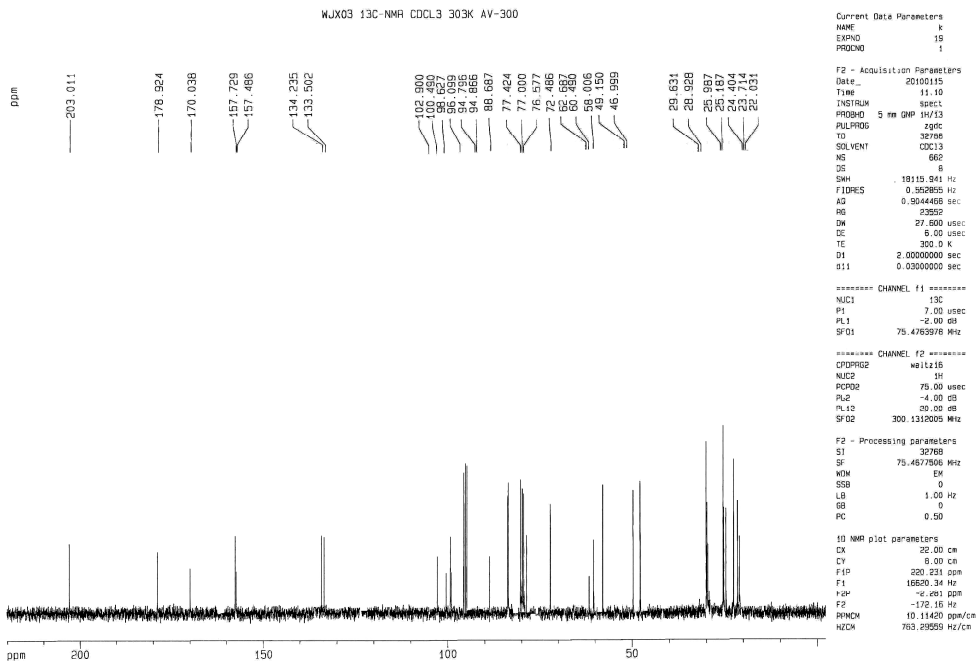


MS spectrum of compound 2

MJH090326-1 CDCl3 303K AV-300



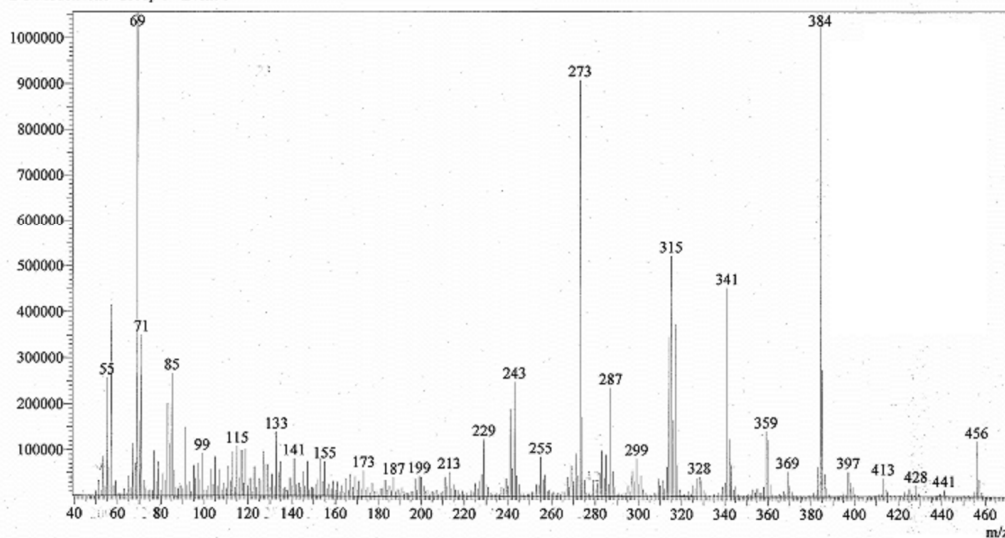
¹H-NMR spectrum of compound 3



¹³C-NMR spectrum of compound 3

Spectrum

Line#:1 R.Time:3.325(Scan#:232)
 MassPeaks:405
 RawMode:Single 3.325(232) BasePeak:69.05(2034519)
 BG Mode:None Group 1 - Event 1



MS spectrum of compound 3

Monoisotopic Mass, Even Electron Ions
 1571 formula(e) evaluated with 11 results within limits (all results (up to 1000) for each mass)

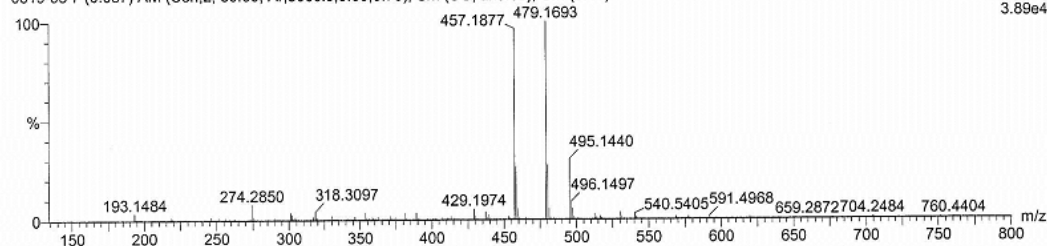
Elements Used:

C: 0-50 H: 0-100 N: 0-5 O: 0-12 Na: 0-1

MJH0903261

0319-05 7 (0.087) AM (Cen,2, 80.00, Ar,5000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (3:41)

TOF MS ES+
 3.89e4

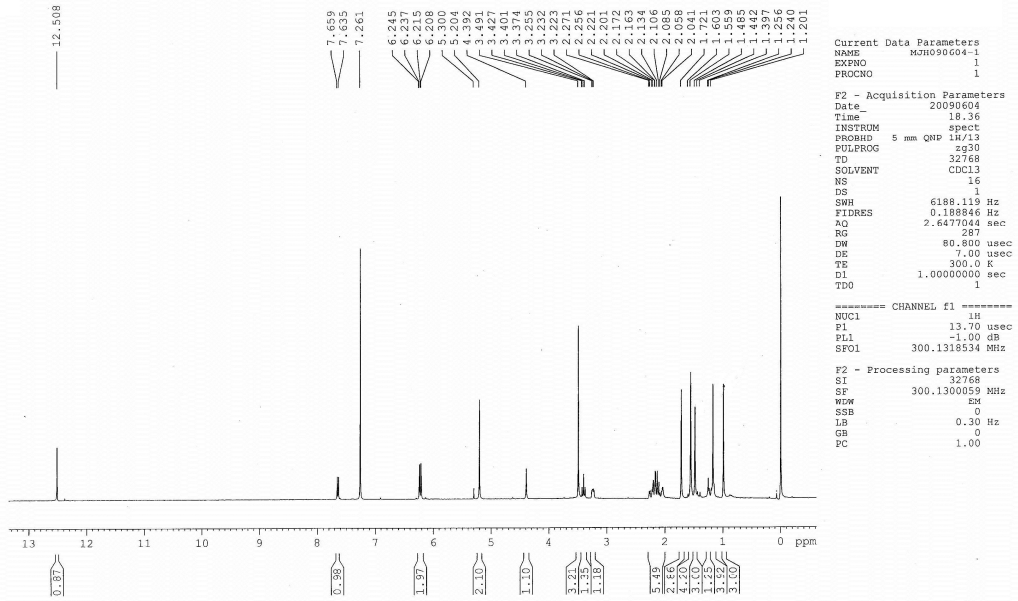


Minimum: 80.00
 Maximum: 100.00

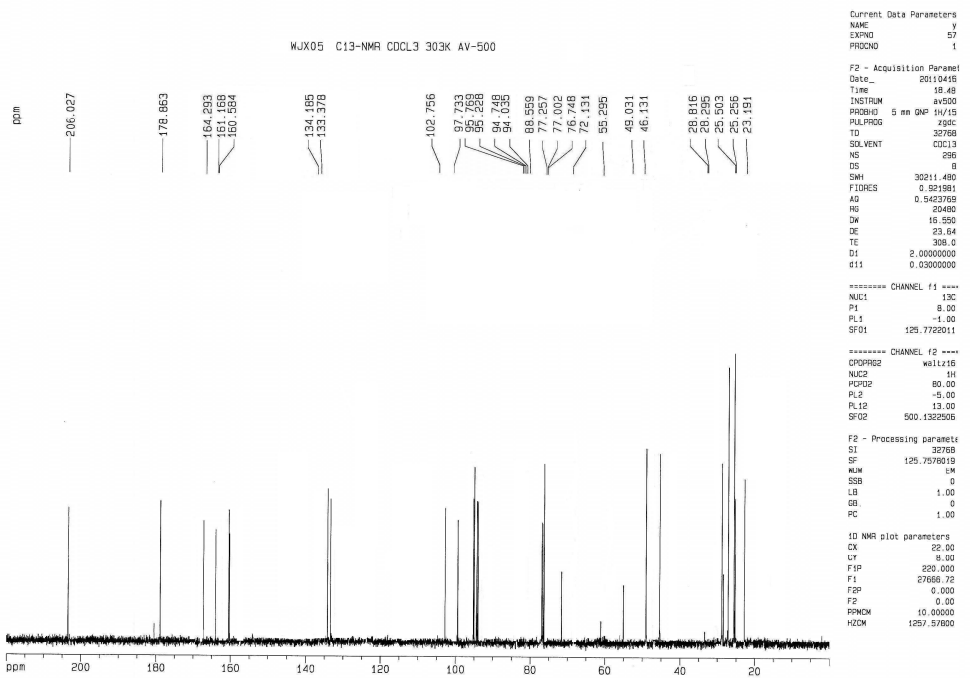
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
457.1877	96.19	457.1876	0.1	0.2	16.5	68.8	C26 H25 N4 O4
		457.1862	1.5	3.3	11.5	11.8	C25 H29 O8
		457.1892	-1.5	-3.3	17.5	188.2	C29 H26 N2 O2 Na
		457.1852	2.5	5.5	13.5	31.2	C24 H26 N4 O4 Na
		457.1910	-3.3	-7.2	4.5	278.7	C17 H30 N4 O9 Na
		457.1838	3.9	8.5	8.5	22.4	C23 H30 O8 Na
479.1693	100.00	479.1695	-0.2	-0.4	16.5	78.1	C26 H24 N4 O4 Na
		479.1682	1.1	2.3	11.5	13.4	C25 H28 O8 Na
		479.1706	-1.3	-2.7	14.5	79.0	C27 H27 O8
		479.1719	-2.6	-5.4	19.5	189.4	C28 H23 N4 O4
		479.1666	2.7	5.6	10.5	16.5	C22 H27 N2 O10

HRMS spectrum of compound 3

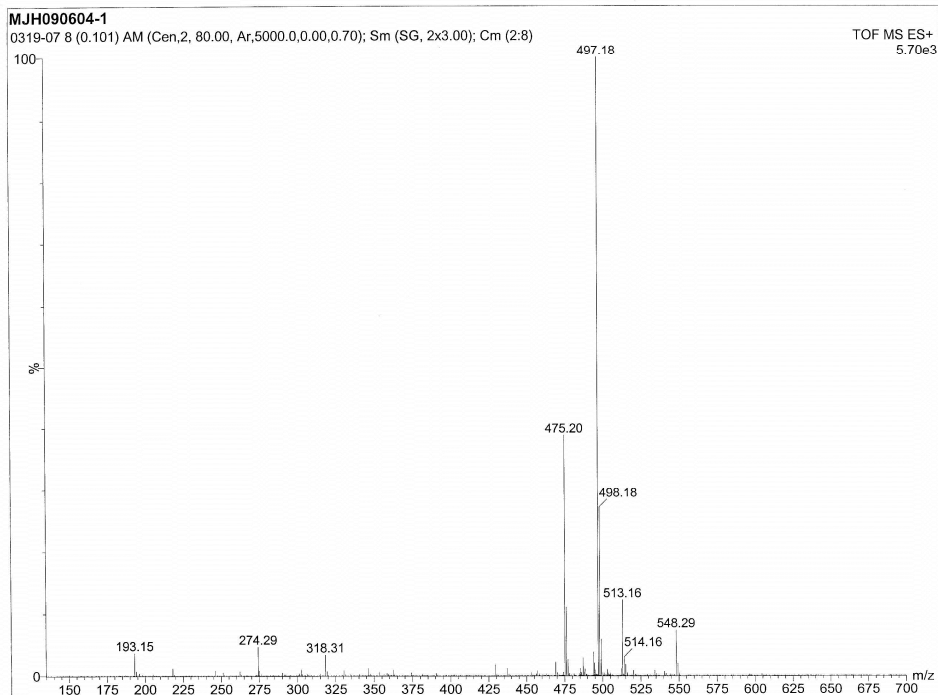
MJH090604-1 CDCL3 303K AV-300



¹H-NMR spectrum of compound 4a



¹³C-NMR spectrum of compound 4a



MS spectrum of compound 4a

Monoisotopic Mass, Even Electron Ions

839 formula(e) evaluated with 7 results within limits (all results (up to 1000) for each mass)

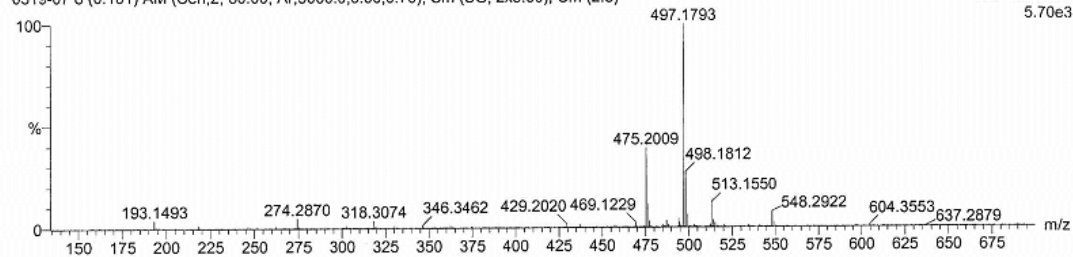
Elements Used:

C: 0-50 H: 0-100 N: 0-5 O: 0-12 Na: 0-1

MJH090604-1

0319-07 8 (0.101) AM (Cen,2, 80.00, Ar,5000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:8)

TOF MS ES+
5.70e3

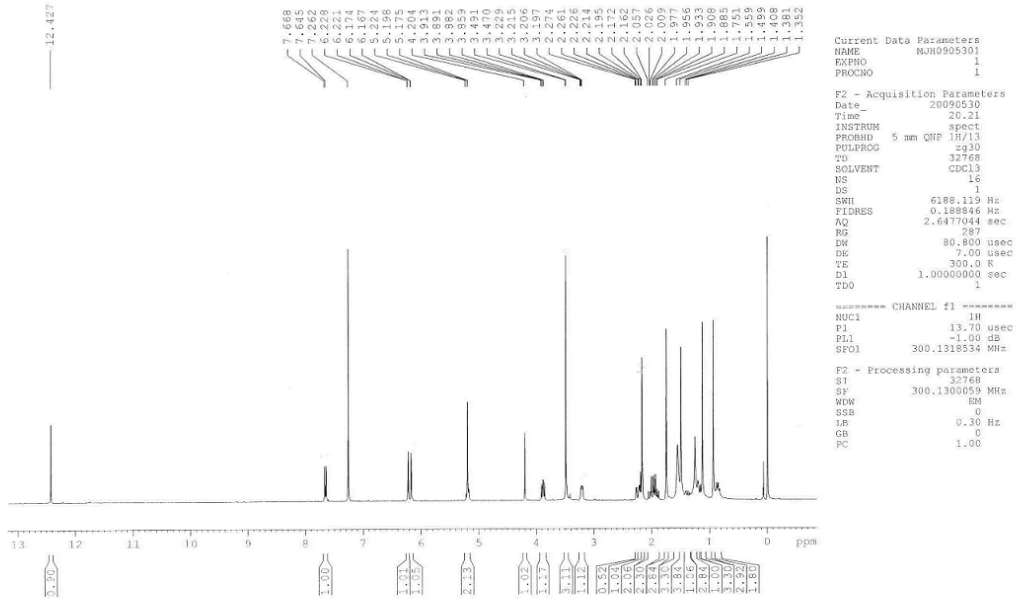


Minimum: 80.00
Maximum: 100.00

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
497.1793	100.00	497.1788	0.5	1.0	10.5	2.4	C25 H30 O9 Na
		497.1801	-0.8	-1.6	15.5	13.1	C26 H26 N4 O5 Na
		497.1812	-1.9	-3.8	13.5	11.4	C27 H29 O9
		497.1771	2.2	4.4	9.5	2.4	C22 H29 N2 O11
		497.1825	-3.2	-6.4	18.5	28.7	C28 H25 N4 O5
		497.1747	4.6	9.3	6.5	15.1	C20 H30 N2 O11 Na
		497.1841	-4.8	-9.7	19.5	55.8	C31 H26 N2 O3 Na

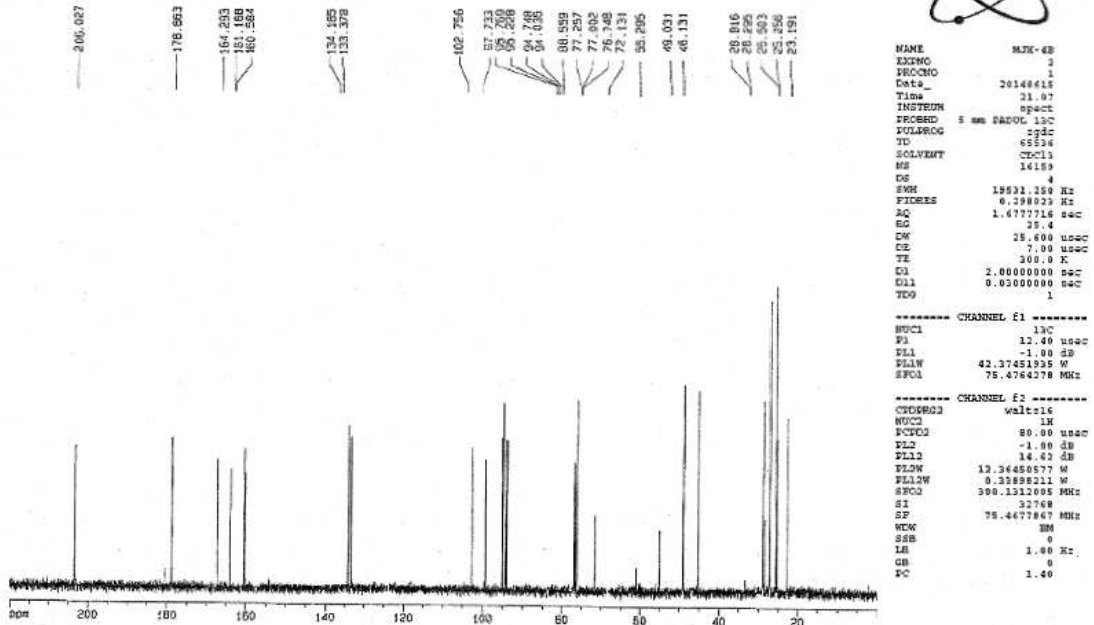
HRMS spectrum of compound 4a

MJH0905301 H1-NMR CDCL3 303K AV-300



¹H-NMR spectrum of compound **4b**

MJH-4B C13-NMR cdcl3 303K AV-300



¹³C-NMR spectrum of compound **4b**

Monoisotopic Mass, Even Electron Ions

839 formula(e) evaluated with 7 results within limits (all results (up to 1000) for each mass)

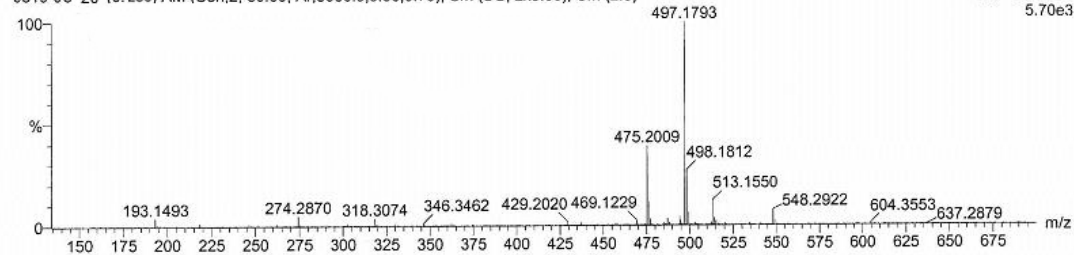
Elements Used:

C: 0-50 H: 0-100 N: 0-5 O: 0-12 Na: 0-1

MJH090604-1

0319-08 20 (0.250) AM (Cen,2, 80.00, Ar,5000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:8)

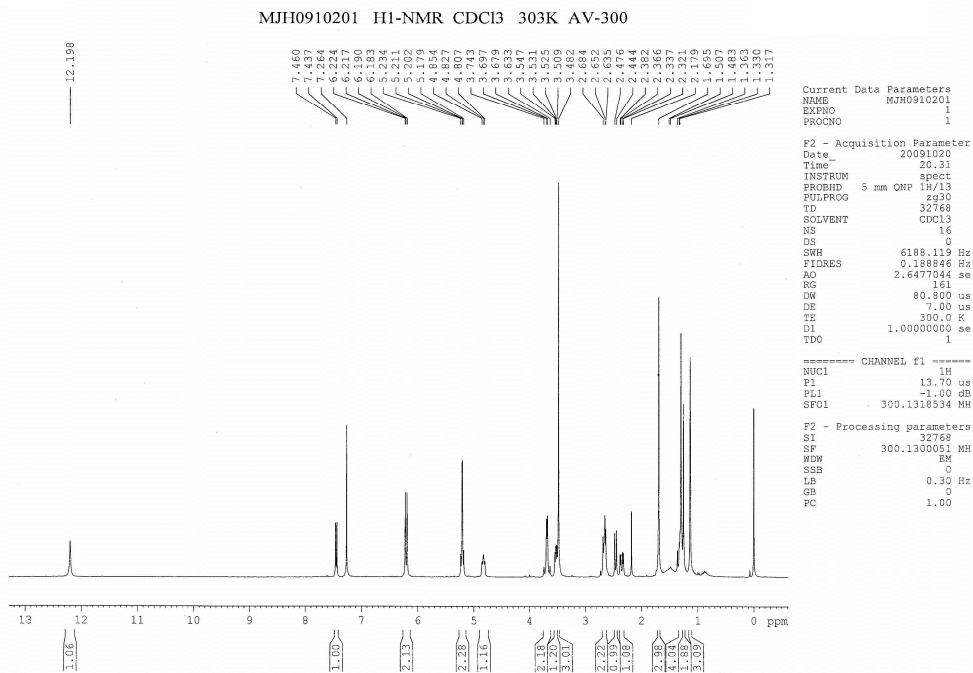
TOF MS ES+
5.70e3



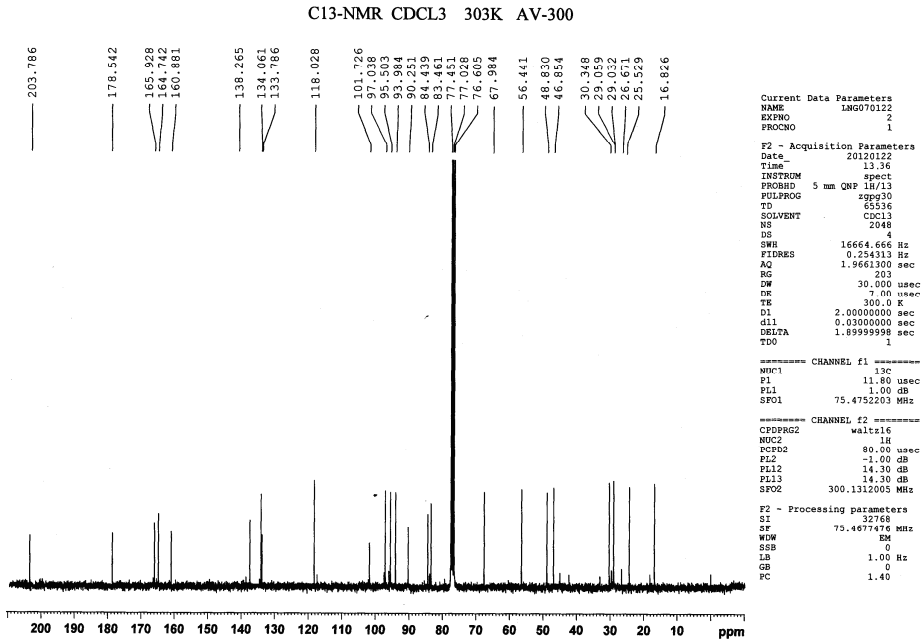
Minimum: 80.00
Maximum: 100.00

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
497.1793	100.00	497.1788	0.5	1.0	10.5	2.4	C25 H30 O9 Na
		497.1801	-0.8	-1.6	15.5	13.1	C26 H26 N4 O5 Na
		497.1812	-1.9	-3.8	13.5	11.4	C27 H29 O9
		497.1771	2.2	4.4	9.5	2.4	C22 H29 N2 O11
		497.1825	-3.2	-6.4	18.5	28.7	C28 H25 N4 O5
		497.1747	4.6	9.3	6.5	15.1	C20 H30 N2 O11 Na
		497.1841	-4.8	-9.7	19.5	55.8	C31 H26 N2 O3 Na

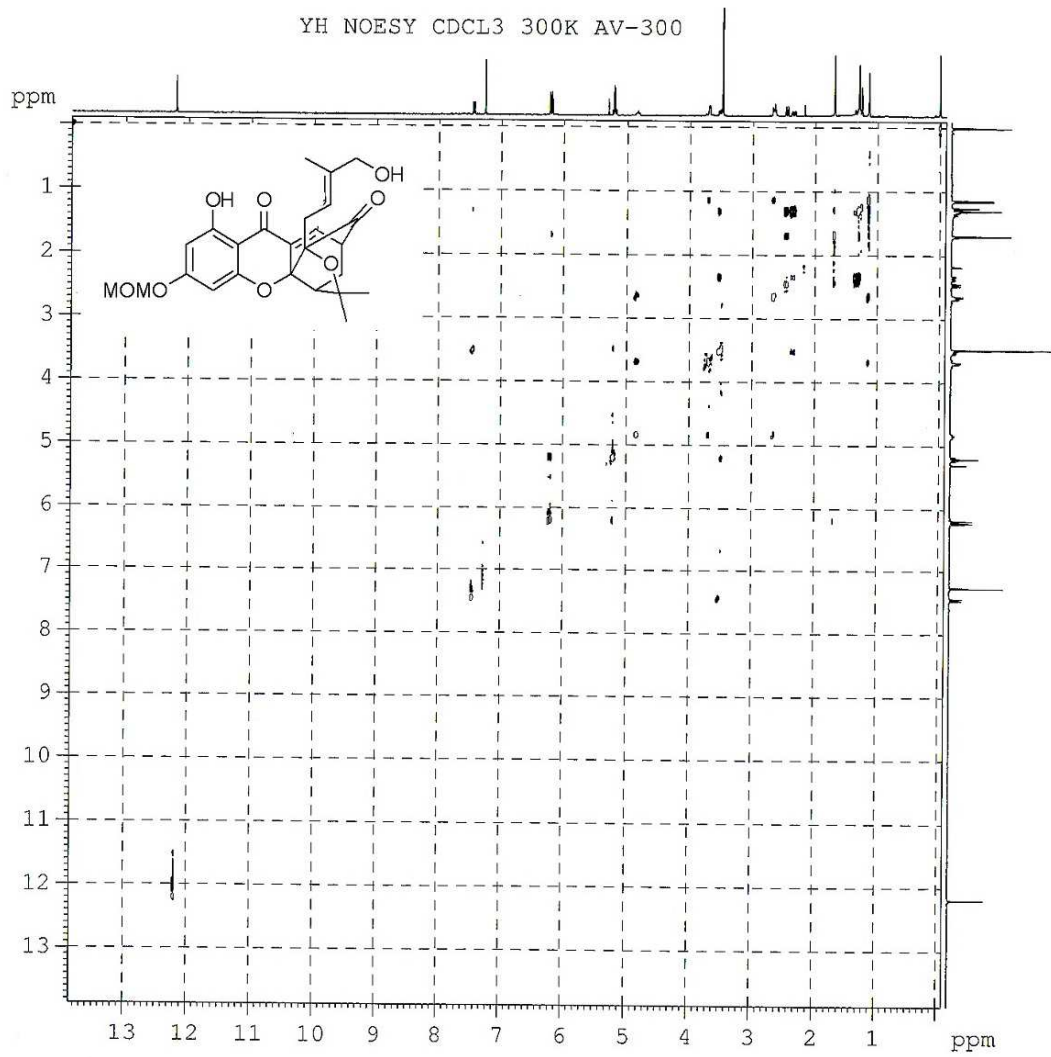
HRMS spectrum of compound **4b**



¹H-NMR spectrum of compound 5

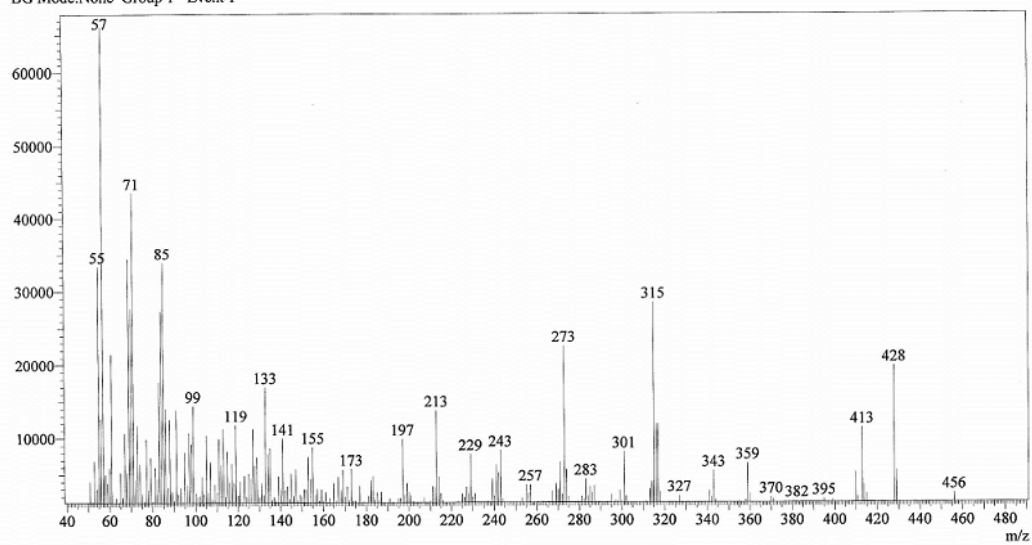


¹³C-NMR spectrum of compound 5

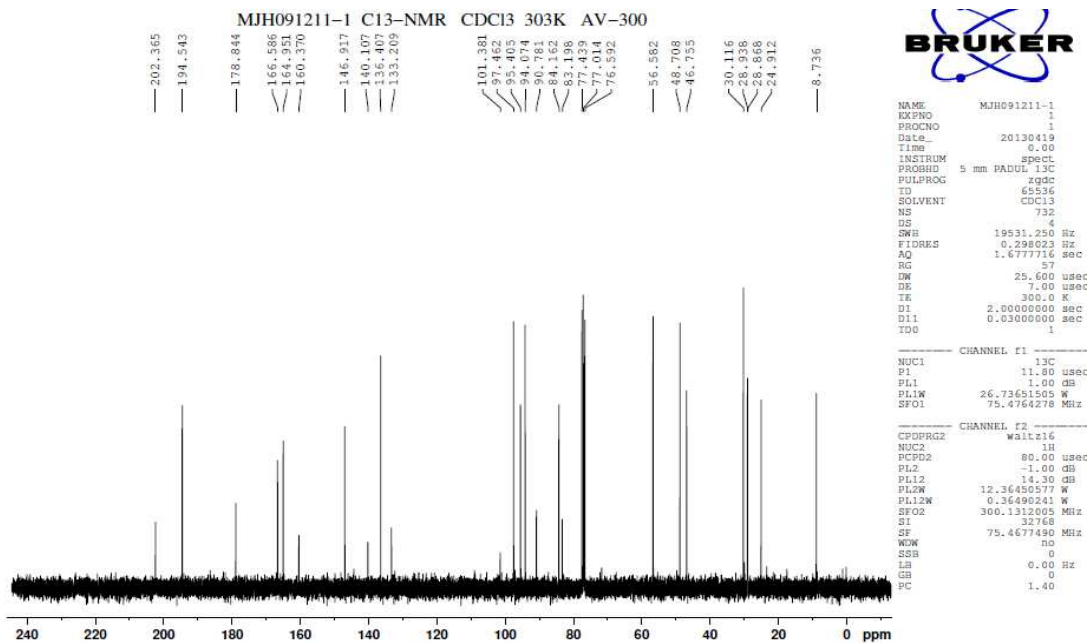
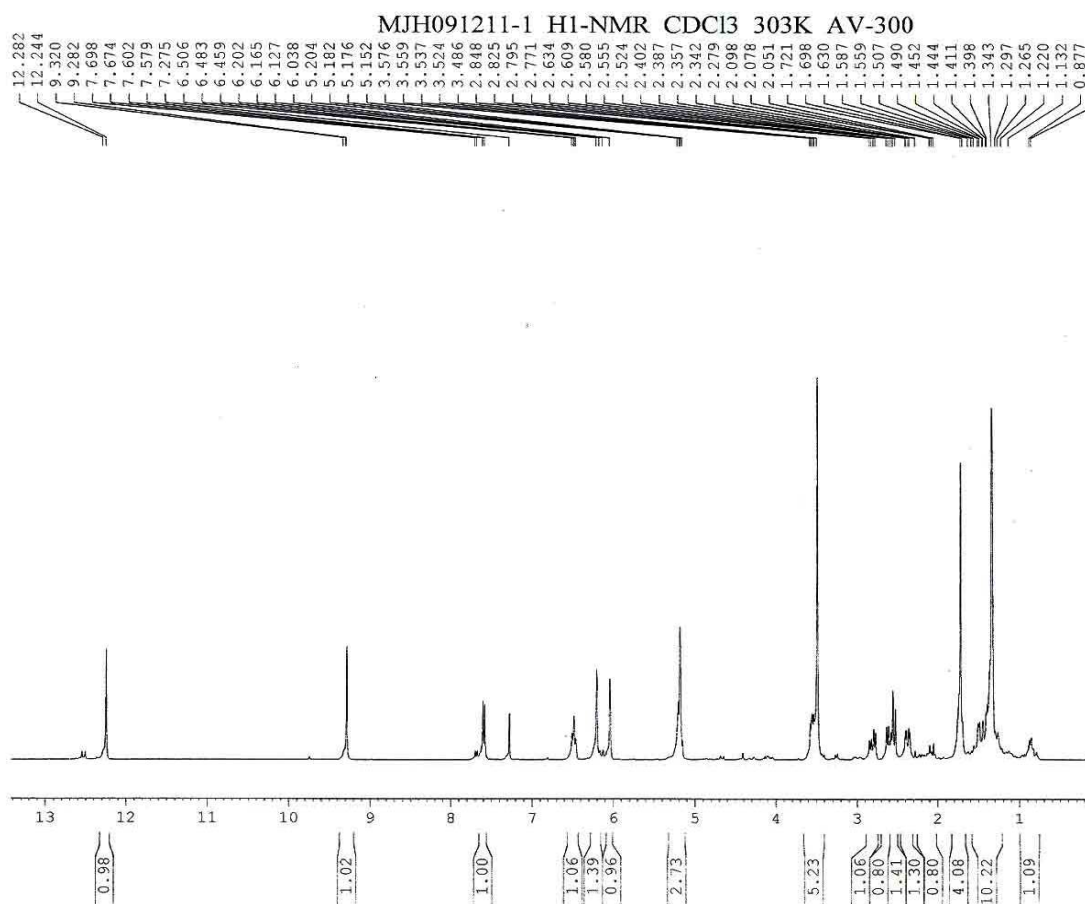


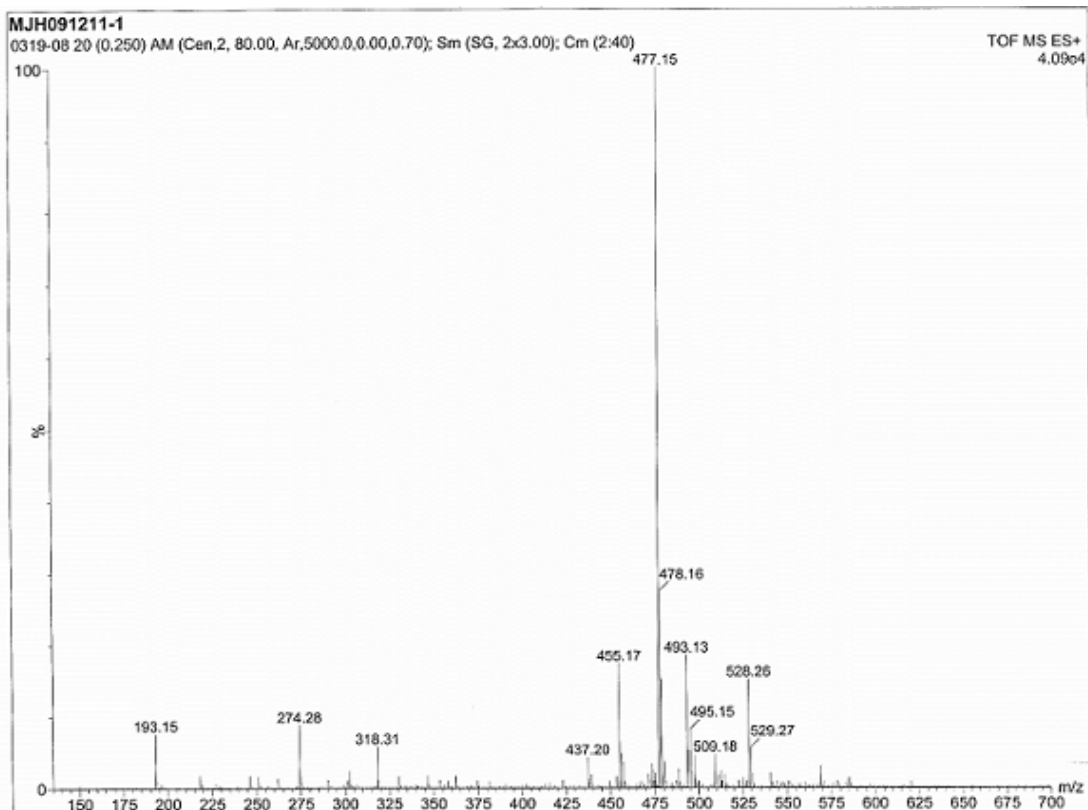
NOESY spectrum of compound 5

Line#:1 R.Time:2.617(Scan#:2)
 MassPeaks:245
 RawMode:Single 2.617(2) BasePeak:57.05(66127)
 BG Mode:None Group 1 - Event 1



MS spectrum of compound 5





MS spectrum of compound 6

Monoisotopic Mass, Even Electron Ions

803 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)

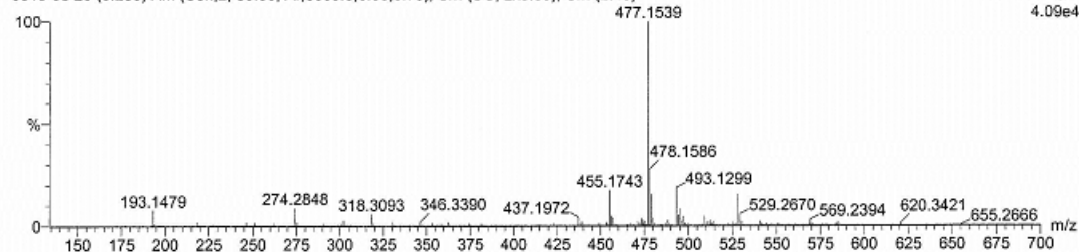
Elements Used:

C: 0-50 H: 0-100 N: 0-5 O: 0-12 Na: 0-1

MJH091211-1

0319-08 20 (0.250) AM (Cen,2, 80.00, Ar,5000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:40)

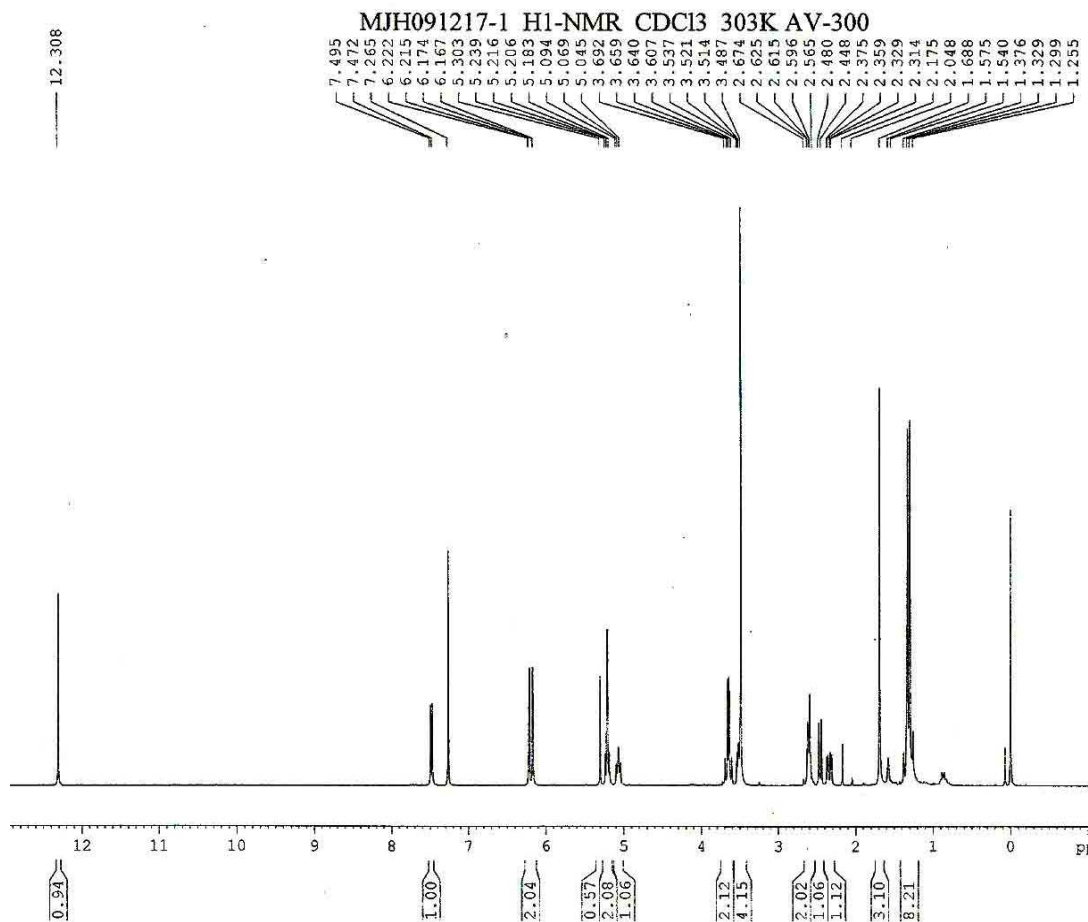
TOF MS ES+
4.09e4



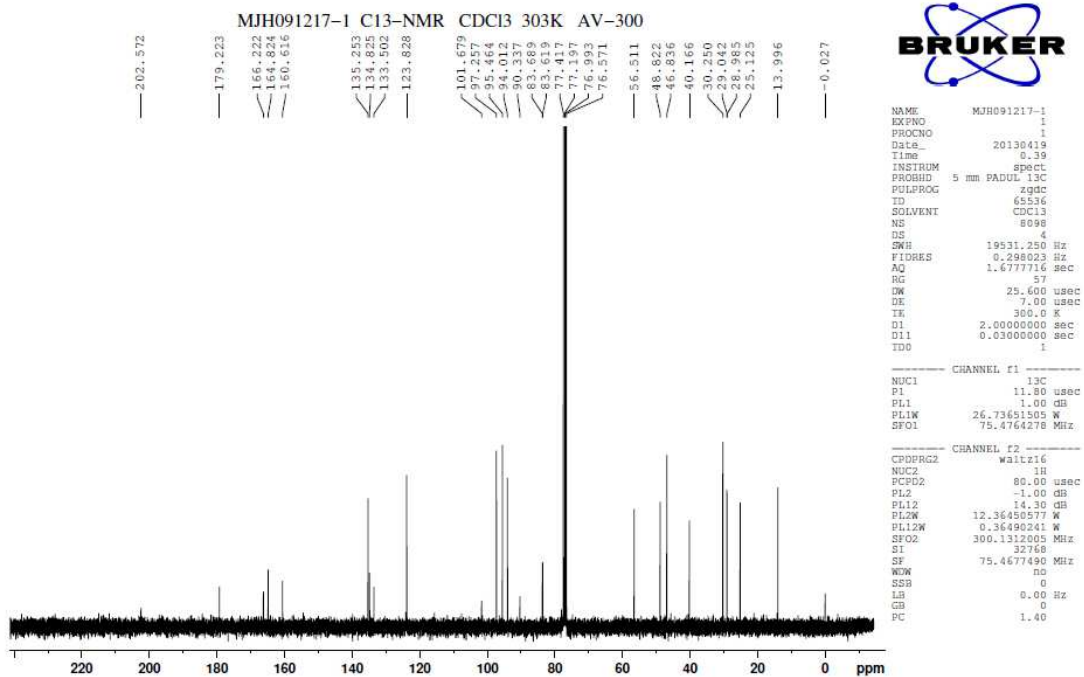
Minimum: 80.00
Maximum: 100.00

Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
477.1539	100.00	477.1539	0.0	0.0	17.5	1367.7	C26 H22 N4 O4 Na
		477.1549	-1.0	-2.1	15.5	1169.2	C27 H25 O8
		477.1525	1.4	2.9	12.5	1256.3	C25 H26 O8 Na
		477.1509	3.0	6.3	11.5	1318.2	C22 H25 N2 O10

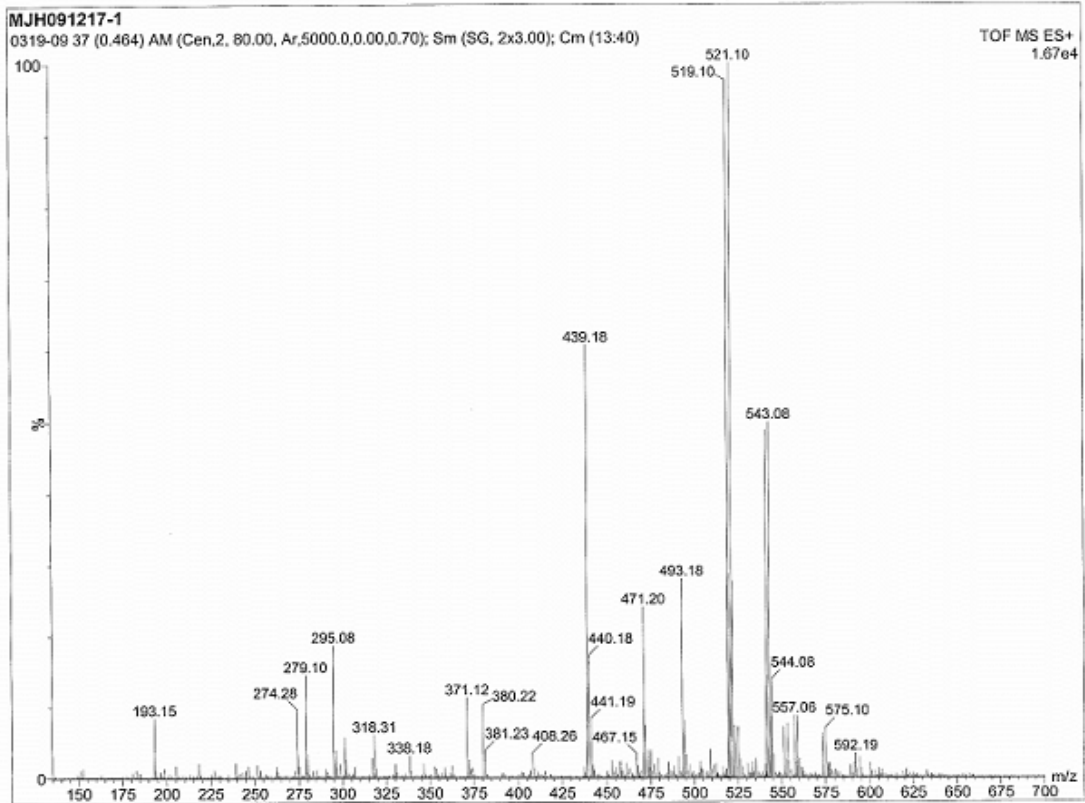
HRMS spectrum of compound 6



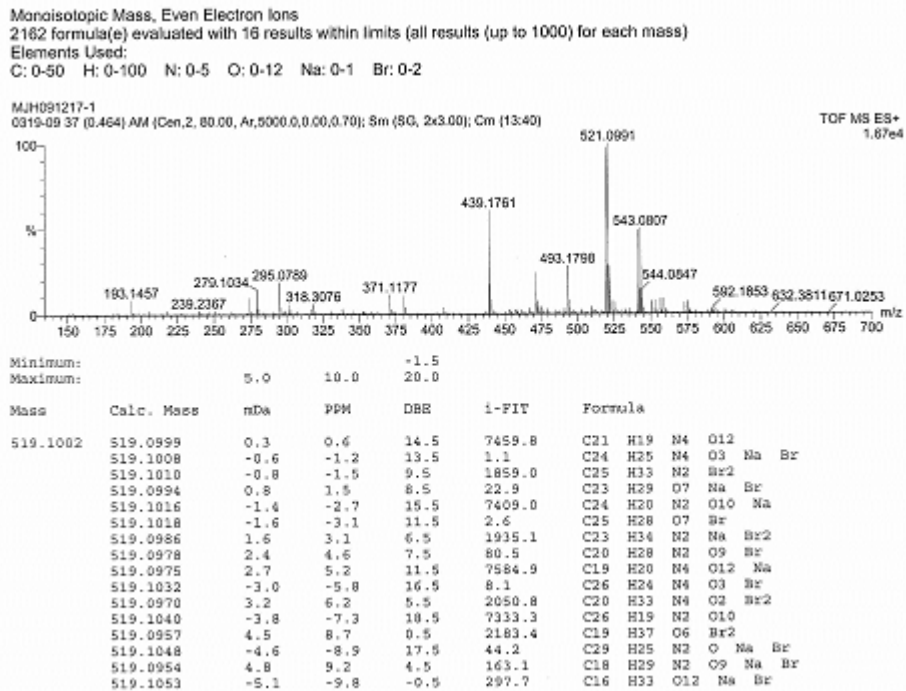
¹H-NMR spectrum of compound 7



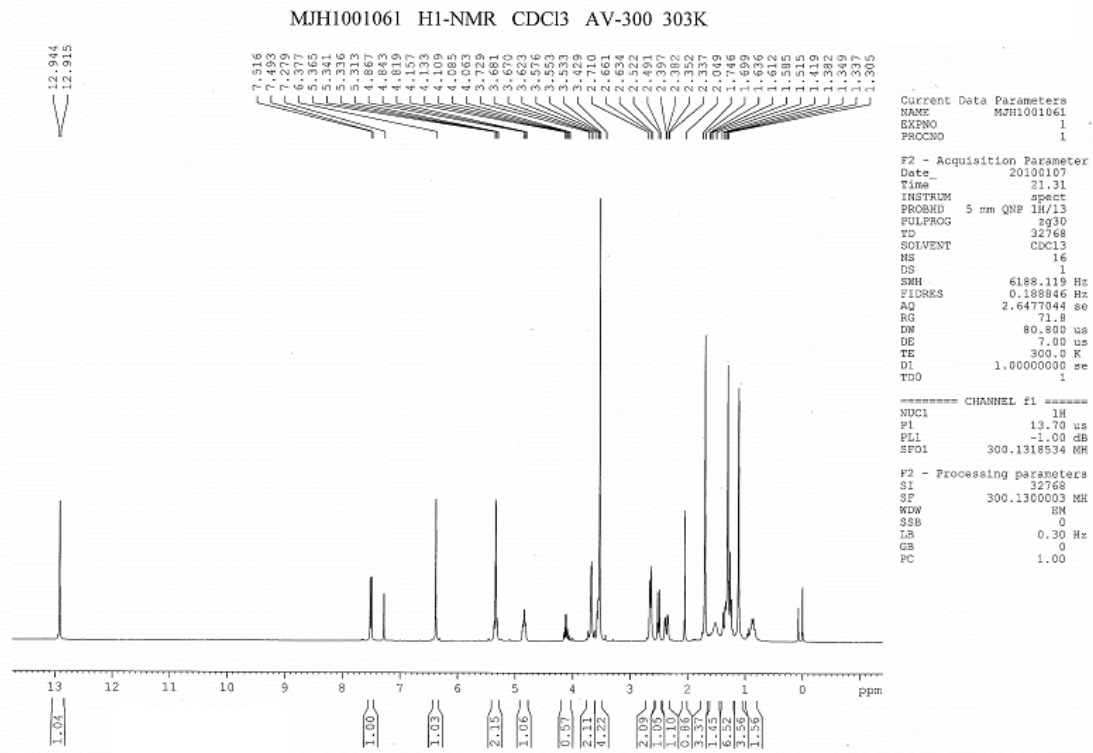
¹³C-NMR spectrum of compound 7



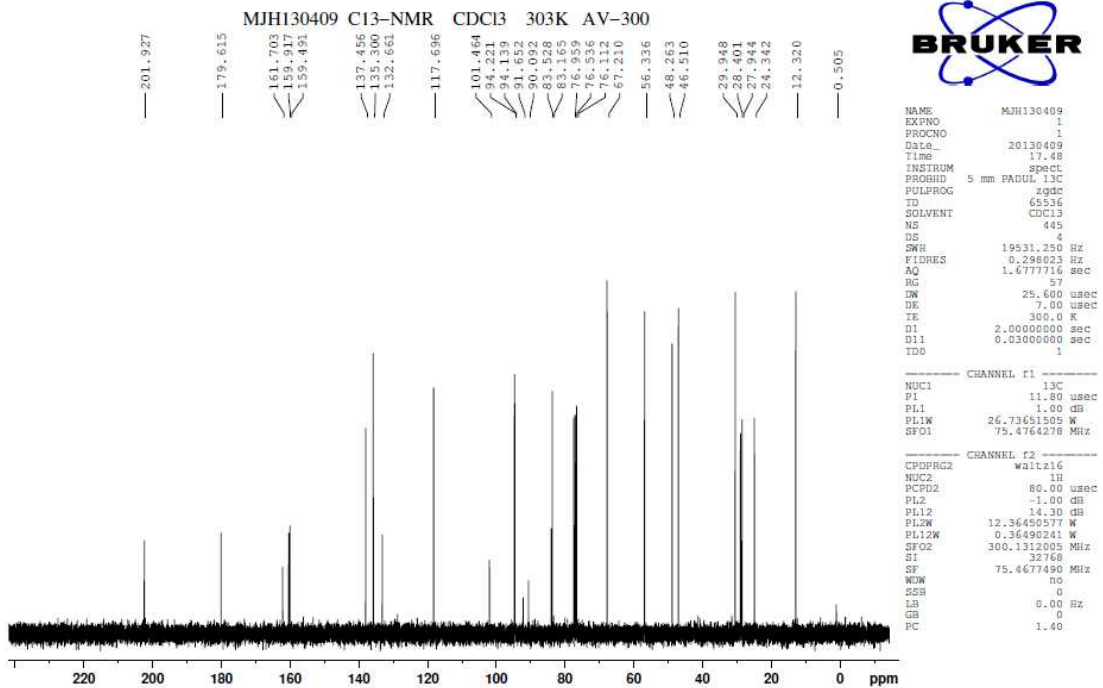
MS spectrum of compound 7



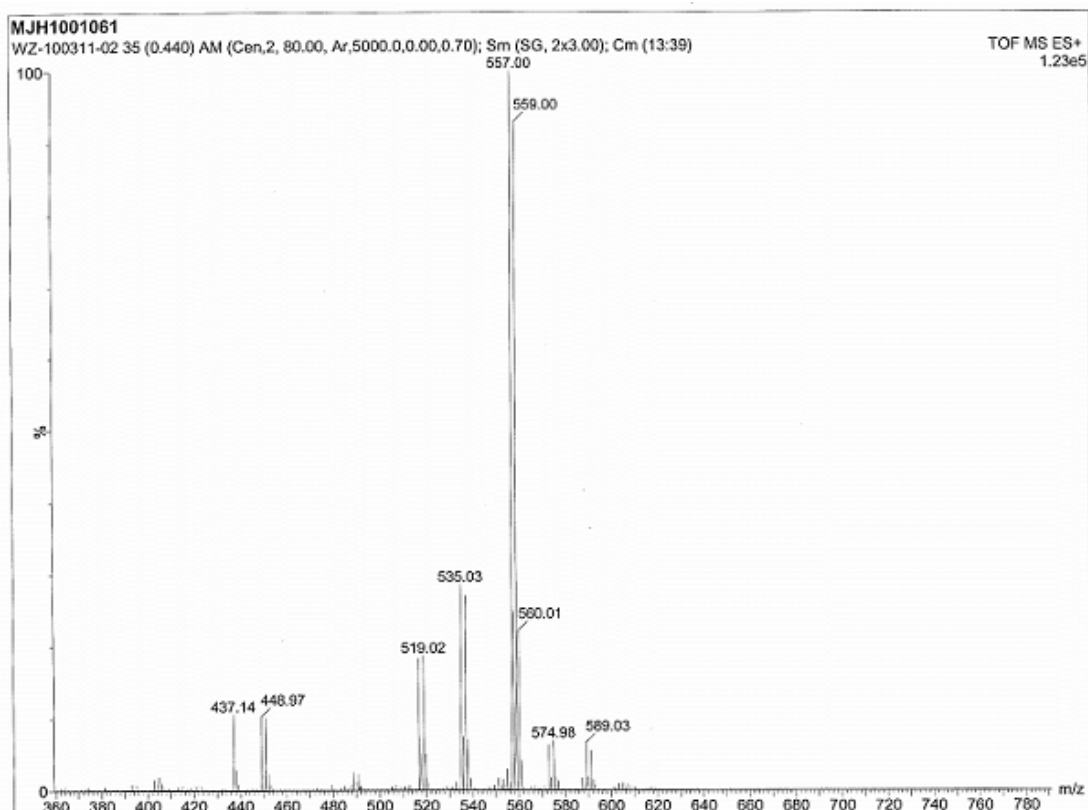
HRMS spectrum of compound 7



¹H-NMR spectrum of compound 8

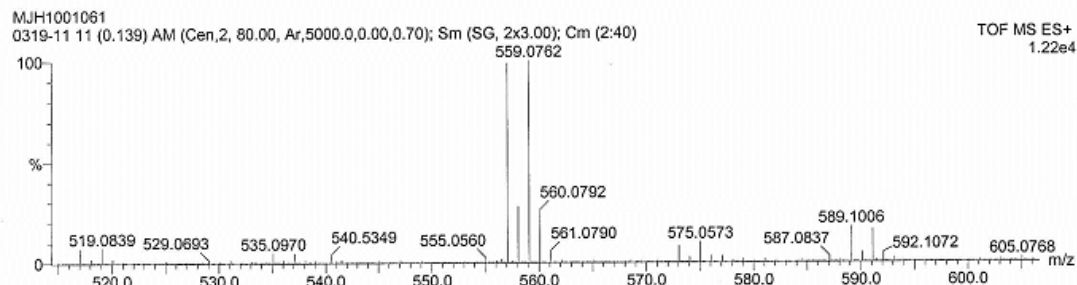


¹³C-NMR spectrum of compound 8



MS spectrum of compound 8

Monoisotopic Mass, Even Electron Ions
2375 formula(e) evaluated with 15 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-50 H: 0-100 N: 0-5 O: 0-12 Na: 0-1 Br: 0-2



Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
557.0778	557.0779	-0.1	-0.2	9.5	1378.1	C25 H32 N2 O Na Br2
	557.0771	0.7	1.3	10.5	11.8	C22 H26 N2 O10 Br
	557.0787	-0.9	-1.6	11.5	0.8	C25 H27 O8 Na Br
	557.0763	1.5	2.7	8.5	1446.9	C22 H31 N4 O3 Br2
	557.0800	-2.2	-3.9	16.5	12.6	C26 H23 N4 O4 Na Br
	557.0803	-2.5	-4.5	12.5	1336.3	C27 H31 N2 O Br2
	557.0750	2.8	5.0	3.5	1529.8	C21 H35 O7 Br2
	557.0808	-3.0	-5.4	18.5	5354.3	C26 H18 N2 O11 Na
	557.0747	3.1	5.6	7.5	42.9	C20 H27 N2 O10 Na Br
	557.0811	-3.3	-5.9	14.5	12.7	C27 H26 O8 Br
	557.0739	3.9	7.0	5.5	1513.1	C20 H32 N4 O3 Na Br2
	557.0822	-4.4	-7.9	-0.5	1721.3	C15 H35 N4 O8 Br2
	557.0824	-4.6	-8.3	19.5	43.2	C28 H22 N4 O4 Br
	557.0731	4.7	8.4	6.5	104.6	C17 H26 N4 O12 Br
	557.0725	5.3	9.5	0.5	1607.7	C19 H36 O7 Na Br2

HRMS spectrum of compound 8