Synthesis and Biological Evaluation of Conjugates of Oleanolic Acid Derivatives-Chalcones as α-Glucosidase Inhibitors

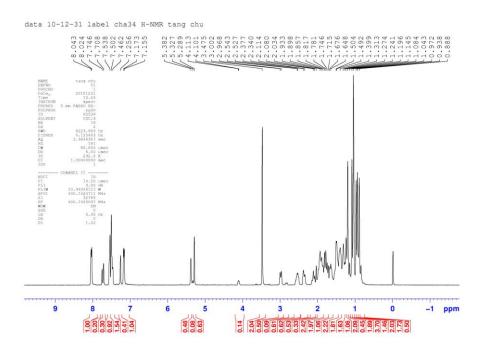
Chu Tang ^{a†}, Linhui Zhu ^{a†}, Yu Chen ^b, Rui Qin ^c, Zhi-Nan Mei ^a, Jing Xu ^{a,*}, Guangzhong Yang ^{a,*}

- ^a Laboratory for Natural Product Chemistry, College of Pharmacy, South Central University for Nationalities, 708 Minyuan Road, Wuhan 430074, P. R. China
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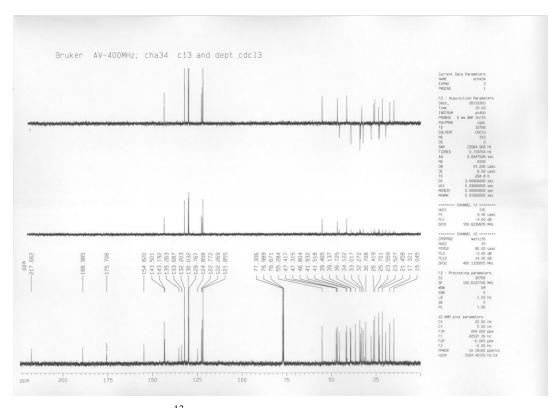
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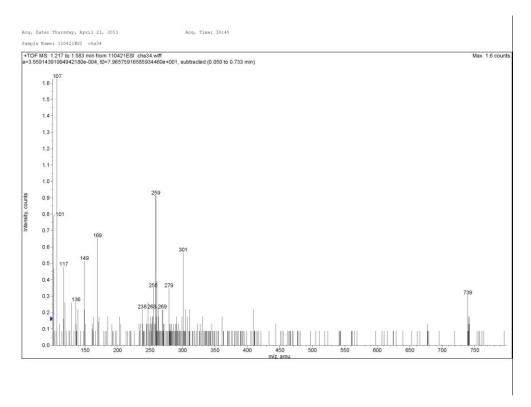
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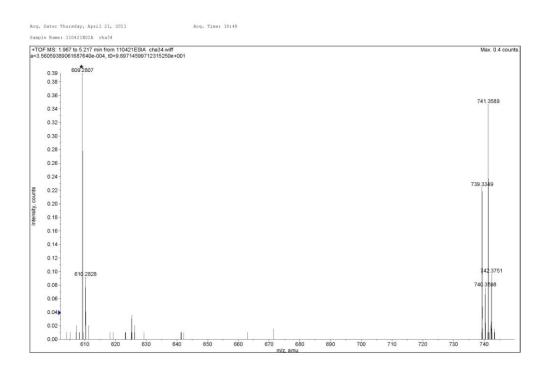
¹H NMR spectrum of compound **1a**



¹³C NMR spectrum of compound **1a**



ESI-MS spectrum of compound 1a



Acq. Date: Thursday, April 21, 2011 Sample Name: 110421ESIA cha34

Elemental composition calculator

Target m/z: +739.3349 amu
Tolerance: +10.0000 ppm
Result type: Elemental
Max num of results: 1000
Min DBE: -10.0000 Max DBE:
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: 110421ESIA cha34.wiff

+60.0000

	Elements	Min Number	Max Number
1	Br	1	1
2	С	0	200
3	C1	0	0
4	F	0	0
5	н	0	400
6	K	0	0
7	N	0	0
8	Na	0	0
9	o	2	5
10	P	0	0
11	Pt	0	0

Acq. Date: Thursday, April 21, 2011

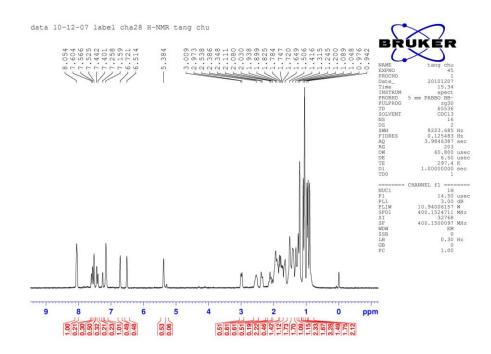
Acq. Time: 18:48

Sample Name: 110421ESIA cha34

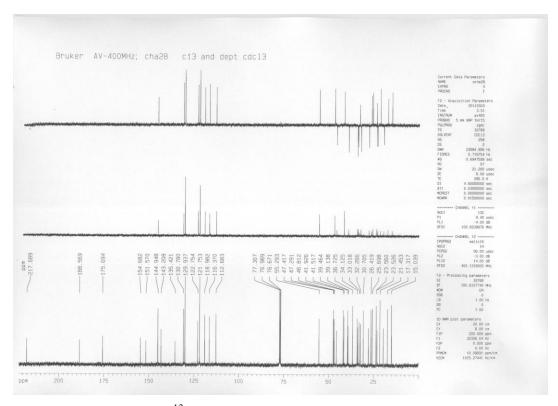
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13	si	0	0

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1	C45 H56 O4 Br	739.3361	-1.2969	-1.7541	17.5

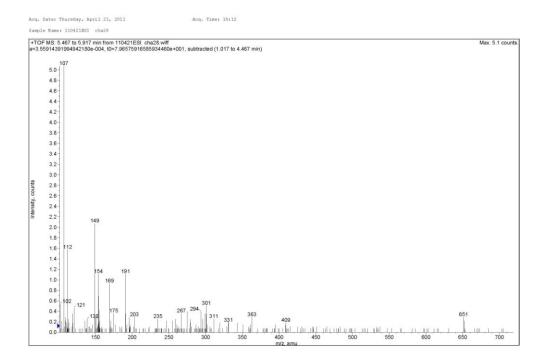
HRESIMS spectrum of compound 1a



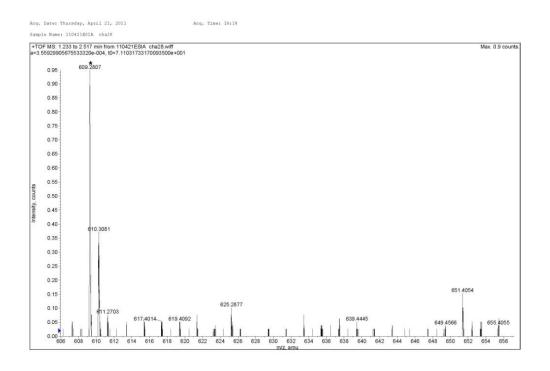
¹H NMR spectrum of compound **1b**



¹³C NMR spectrum of compound **1b**



ESI-MS spectrum of compound 1b



Acq. Date: Thursday, April 21, 2011

Elemental composition calculator

Target m/z: +651.4054 amu
Tolerance: +10.0000 ppm
Result type: Elemental
Max num of results: 1000
Min DBE: -10.0000 Max DBE: +60.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: 110421ESIA cha28.wiff

	Elements	Min Number	Max Number
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2	С	0	200
3	C1	0	0
4	F	0	0
5	Н	0	400
6	К	0	0
7	N	0	0
8	Na	0	0
9	0	2	5
10	P	0	0
11	Pt	0	0

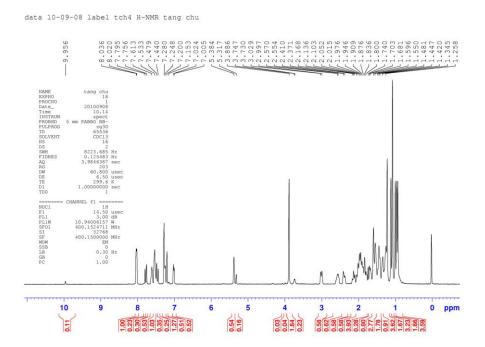
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Acq. Time: 18:18

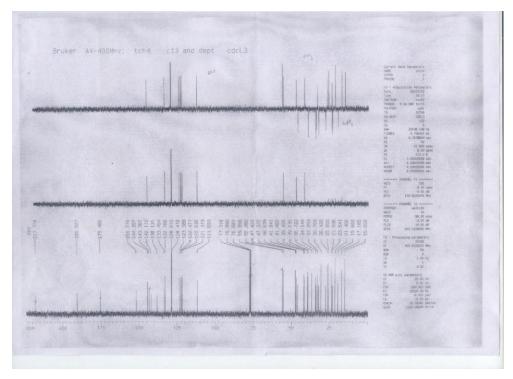
Sample Name: 110421ESIA cha28

		Elements		Min Number	×	Max Number	
	12	s		0	0		
	13	si		0	0		
		·					
	For	mula	Calc	ulated m/z (amu)	mDa Error	PPM Error	DBE
1	C43 H55 O5		651.4049		0.4496	0.6901	16.5

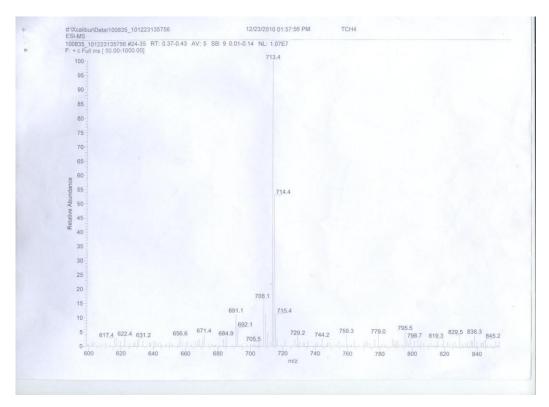
HRESIMS spectrum of compound 1b



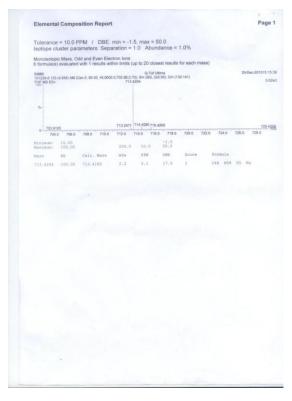
 ^{1}H NMR spectrum of compound 1c



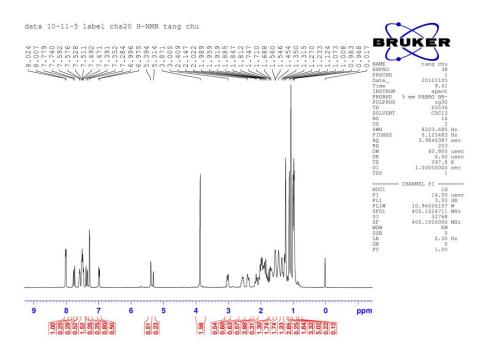
 13 C NMR spectrum of compound 1c



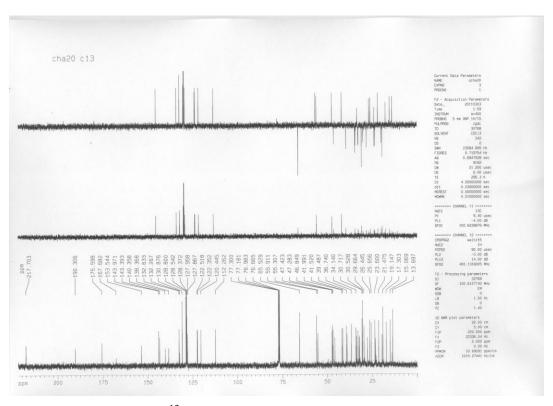
ESI-MS spectrum of compound 1c



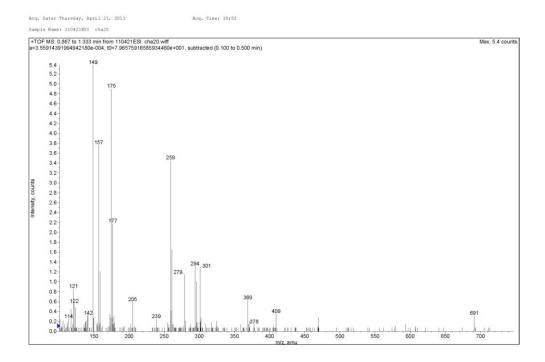
HRESIMS spectrum of compound 1c



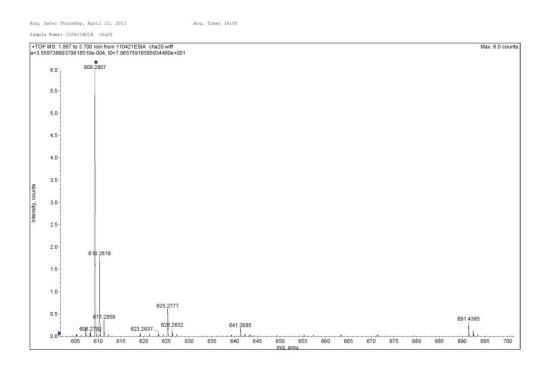
¹H NMR spectrum of compound **1d**



 13 C NMR spectrum of compound 1d



ESI-MS spectrum of compound 1d



Acq. Date: Thursday, April 21, 2011

12

Acq. Time: 18:05

Elements

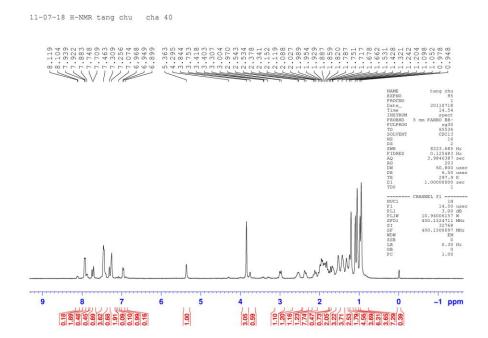
Sample Name: 110421ESIA cha20

	13 Si	0	0		
	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DB
7	C46 H59 O5	691.4362	0.2494	0.3607	17.5

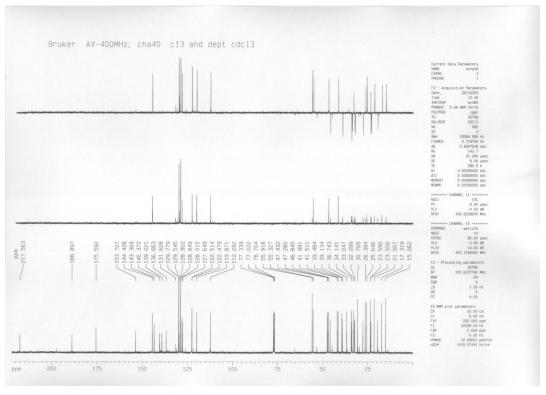
Min Number

Max Number

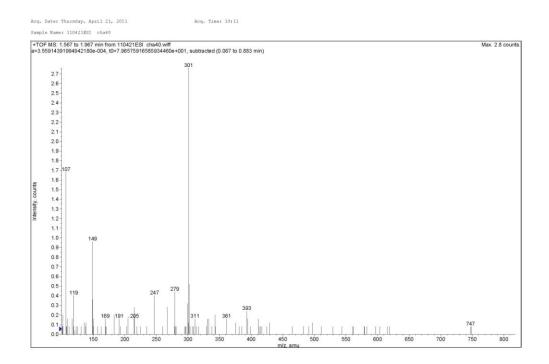
HRESIMS spectrum of compound 1d



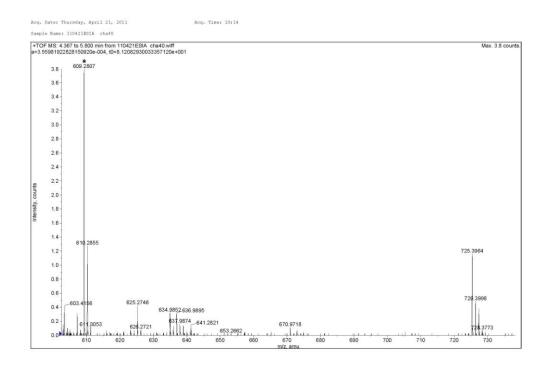
¹H NMR spectrum of compound **1e**



 13 C NMR spectrum of compound 1e



ESI-MS spectrum of compound 1e



Acq. Date: Thursday, April 21, 2011 Sample Name: 110421ESIA cha40 Acq. Time: 19:14

12 S 0 0 0 13 S1 0 0 0 0 1 13 S1 0 0 0 1 1 13 S1 1			Ele	ements	Min Numb	er	Max Number	
Formula Calculated m/z (amu) mDa Error PFM Error DB		12	s	0		0		
0.0000000000000000000000000000000000000		13	Si	0		0		
1 C46 H58 O5 C1 725.3972 -0.8782 -1.2107 17.5			Formula	Calcula	ated m/z (amu)	mDa Error	PPM Error	DB
	1	C46 H58 O5 C1		725.3972		-0.8782	-1.2107	17.5

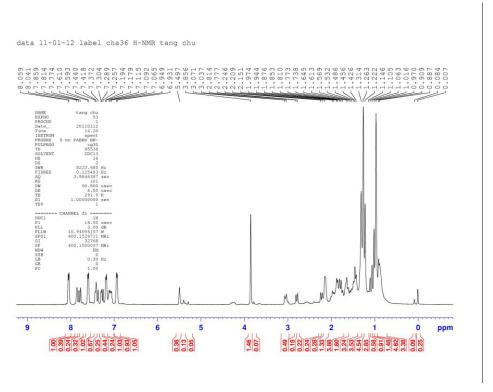
Acq. Date: Thursday, April 21, 2011

Acq. Time: 19:14

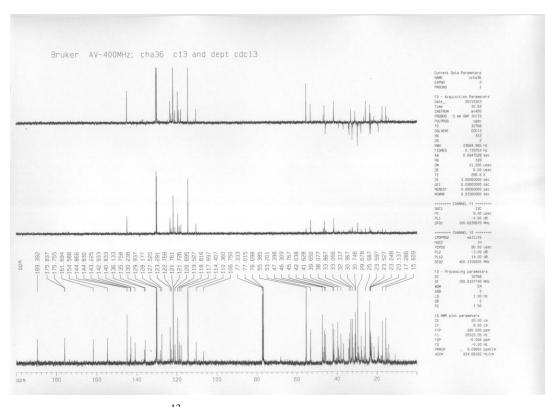
Sample Name: 110421ESIA cha40

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	Tolerance:	+10.0000 ppm	
	Result type: E	lemental	
	Max num of resul	ts: 1000	
	Min DBE:	-10.0000 Max DBE:	+60.0000
	Electron state:	OddAndEven	
	Num of charges:		
	Add water: N		
	Add proton: N	/A	
	File Name: 1	10421ESIA cha40.wiff	
	Elements	Min Number	Max Number
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2	С	0	200
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5	Н	0	400
6	К	0	0
7	N	0	o
8	Na	0	0
	10000		

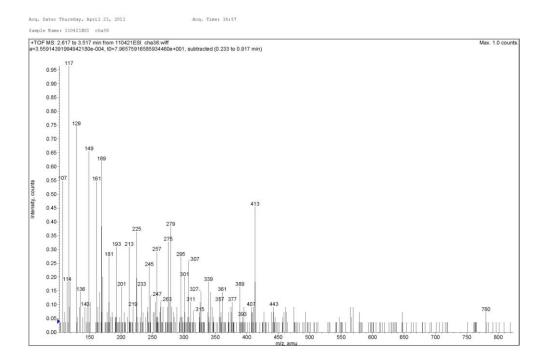
HRESIMS spectrum of compound 1e



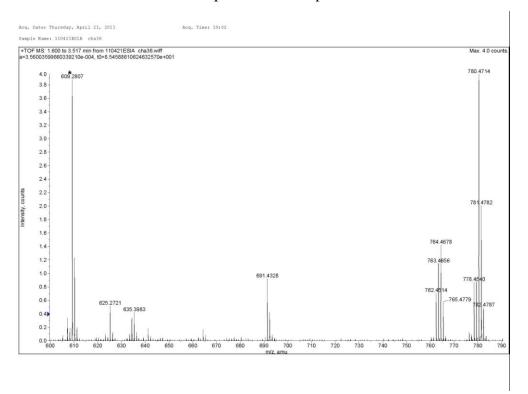
¹H NMR spectrum of compound 2a



¹³C NMR spectrum of compound **2a**



ESI-MS spectrum of compound 2a



Acq. Dates Thursday, April 21, 2011 Acq. Ti
Sample Name: 110421ESIA cha36

T
R
M
M
M
R
R
A

	Elements	Min Number	Max Number
1	Br	0	0
2	С	0	200
3	C1	0	0
4	F	0	0
5	н	0	400
6	К	0	0
7	И	1	1
8	Na	0	0
9	0	2	5
10	P	0	0
11	Pt	0	0

Acq. Date: Thursday, April 21, 2011

Acq. Time: 19:02

Elements

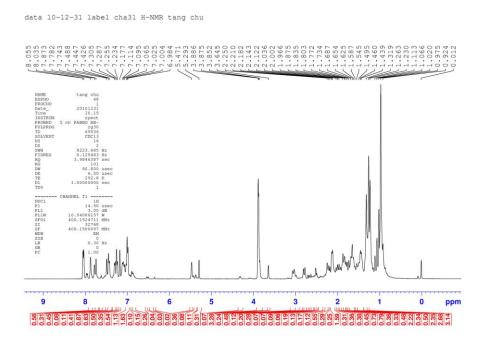
Sample Name: 110421ESIA cha36

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13	Si	0	0		
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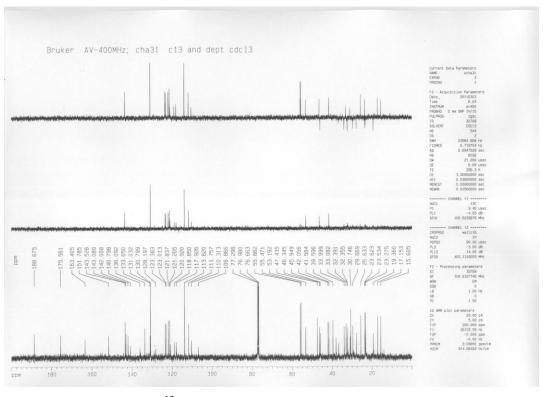
Min Number

Max Number

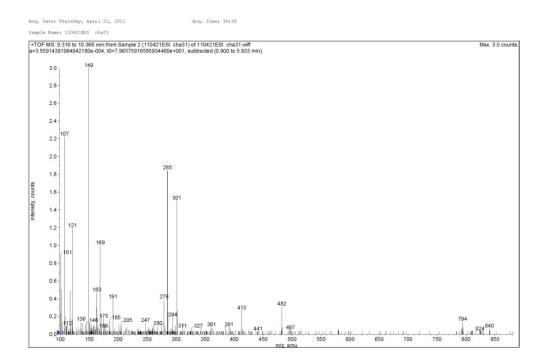
HRESIMS spectrum of compound 2a



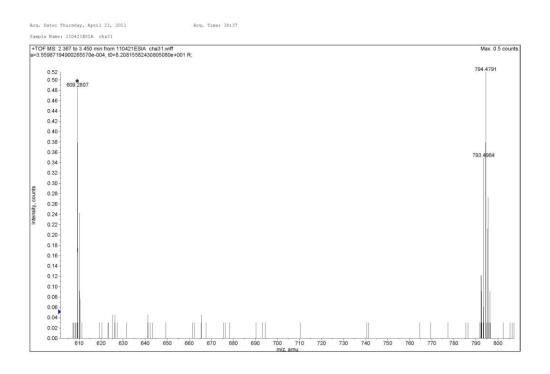
¹H NMR spectrum of compound **2b**



 13 C NMR spectrum of compound **2b**



ESI-MS spectrum of compound 2b



Acq. Date: Thursday, April 21, 2011

Acq. Time: 18:37

Elements

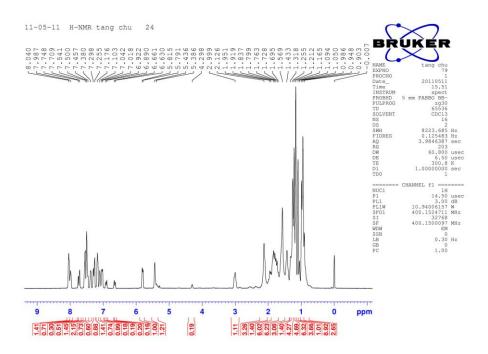
Sample Name: 110421ESIA cha31

Formula Calculated m/z (amu) mDa Error PPM Error D	Formula Calculated m/z (amu) mDa Error PPM Error D		12	Si		0		0	
			10	J.		9		9	
1 C53 H64 N O5 794.4784 0.6502 0.8184 22.	1 C53 H64 N O5 794.4784 0.6502 0.8184 22.		For	mula	Calc	ulated m/z (amu)	mDa Eri	or PPM Error	DBI
		1	C53 H64 N O5		794.4784		0.6502	0.8184	22.5
								1 555 55	

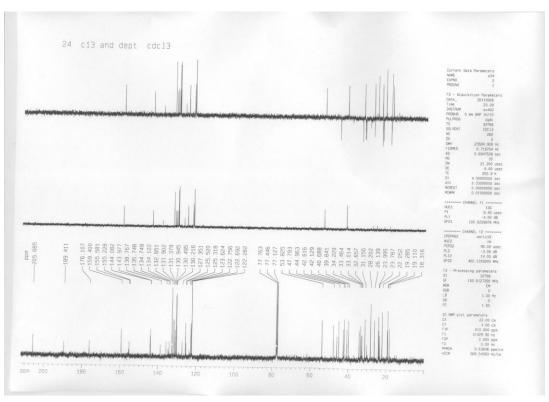
Min Number

Max Number

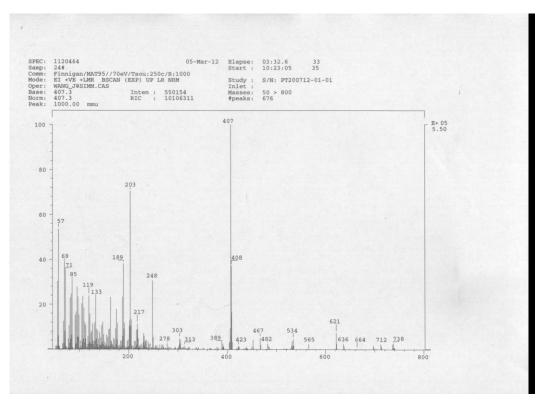
HRESIMS spectrum of compound 2b



 ^{1}H NMR spectrum of compound 3a

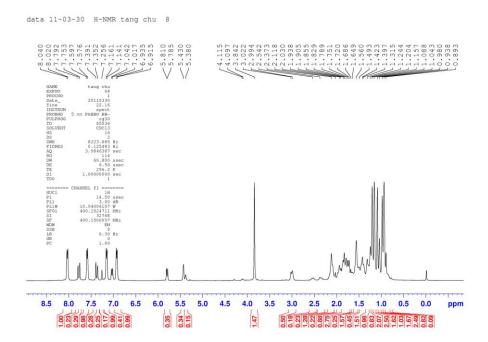


¹³C NMR spectrum of compound **3a**

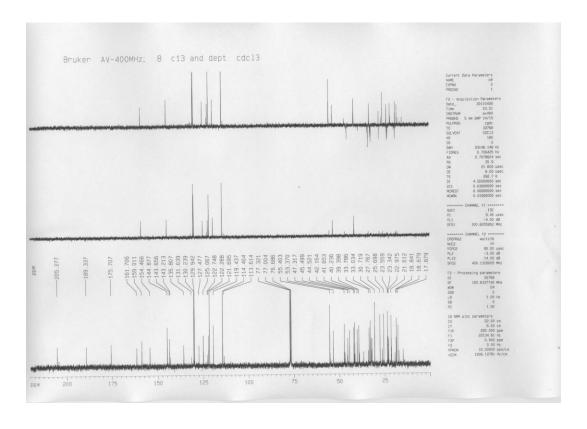


EI-MS spectrum of compound 3a

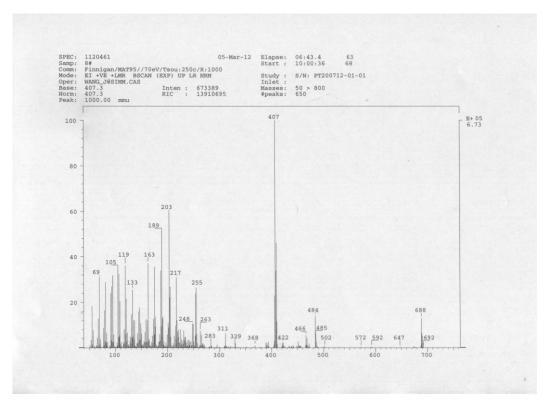
HREIMS spectrum of compound 3a



 1 H NMR spectrum of compound 3b

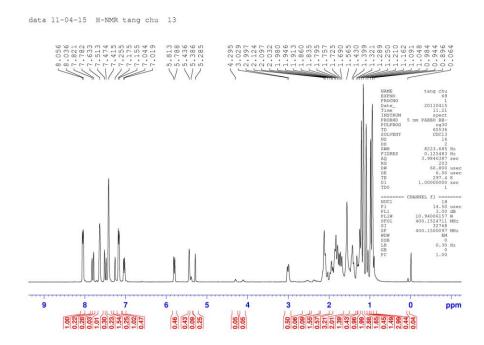


¹³C NMR spectrum of compound **3b**

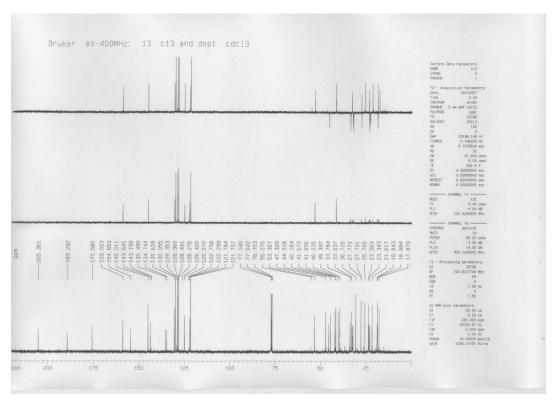


EI-MS spectrum of compound 3b

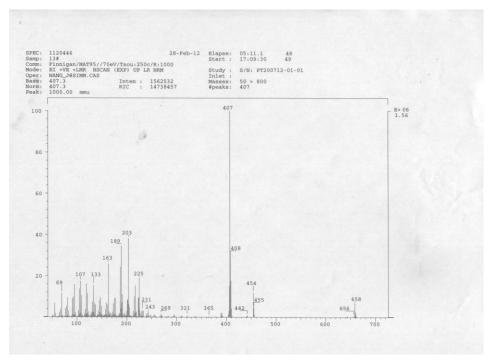
HREIMS spectrum of compound 3b



¹H NMR spectrum of compound **3c**

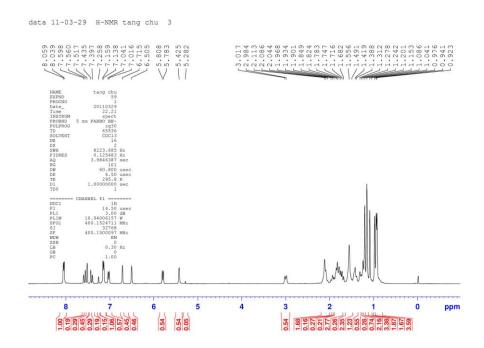


 13 C NMR spectrum of compound 3c

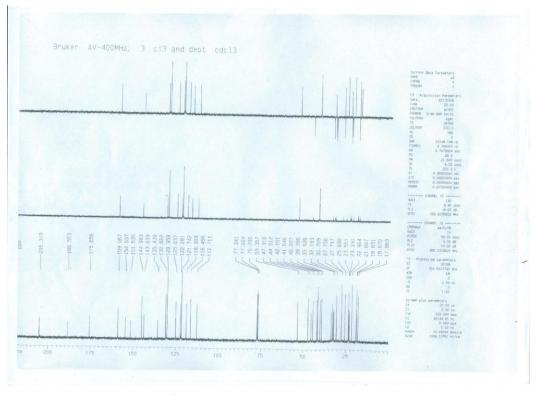


EI-MS spectrum of compound 3c

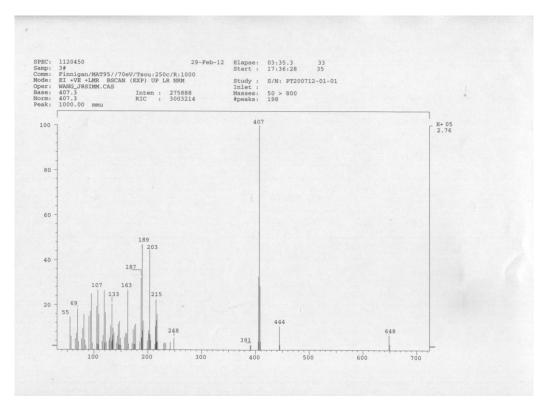
HREIMS spectrum of compound 3c



¹H NMR spectrum of compound **3d**

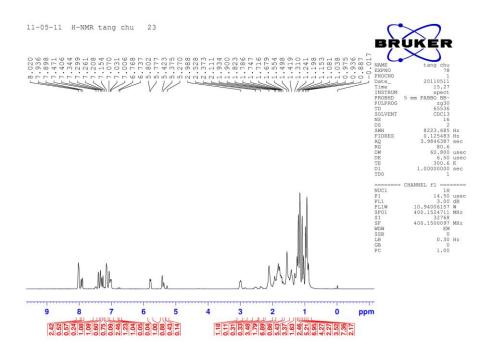


 13 C NMR spectrum of compound **3d**

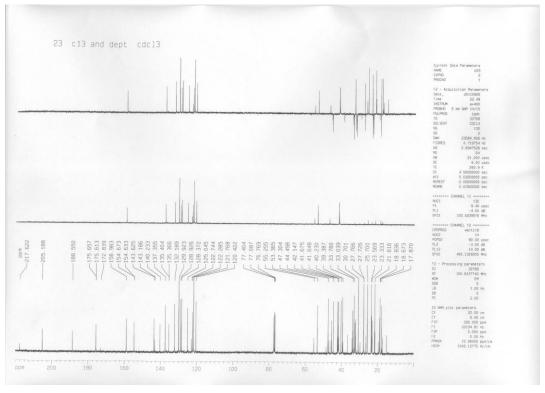


EI-MS spectrum of compound 3d

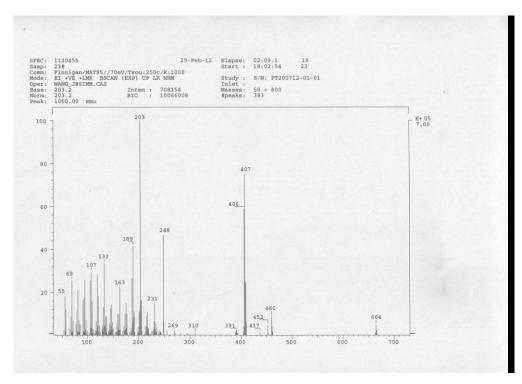
HREIMS spectrum of compound 3d



¹H NMR spectrum of compound **3e**



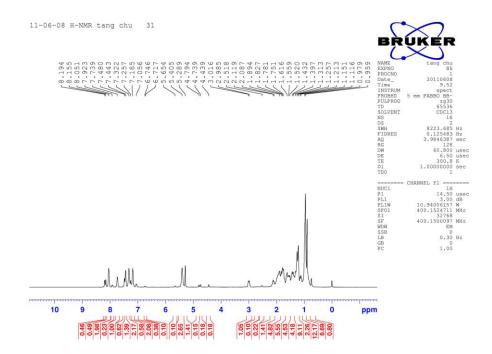
¹³C NMR spectrum of compound **3e**



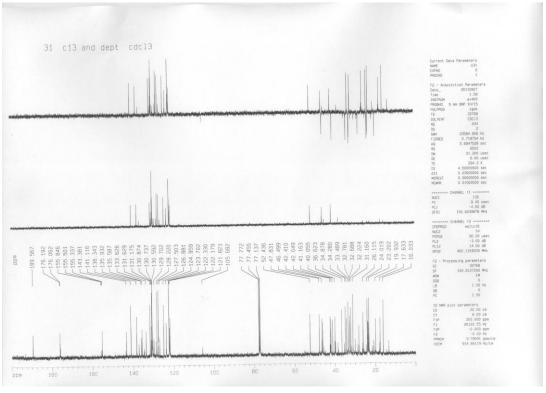
EI-MS spectrum of compound 3e

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Samp: 131migam/MATPS)/TOMO/TROW:220c/R:10000
Mode: EI +VE +12R BSCAN (EXP) UP HR NRW
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Lint: ( 0)
Lint: (
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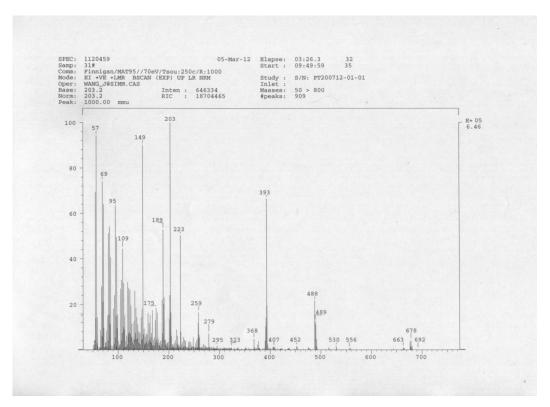
HREIMS spectrum of compound 3e



¹H NMR spectrum of compound **4a**



¹³C NMR spectrum of compound **4a**

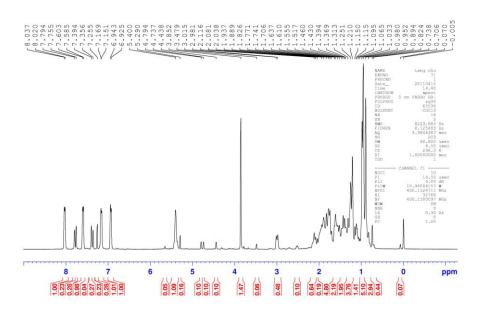


EI-MS spectrum of compound 4a

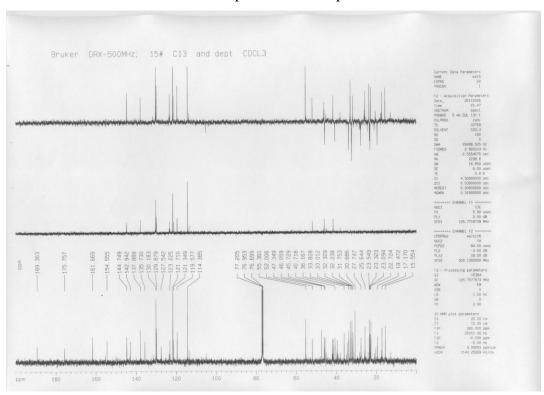
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```

HREIMS spectrum of compound 4a

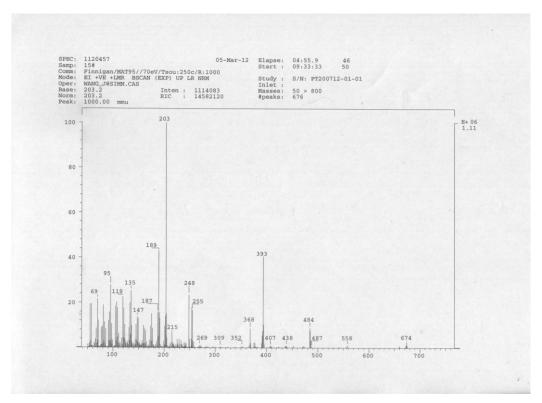
data 11-04-15 H-NMR tang chu 15



¹H NMR spectrum of compound **4b**



¹³C NMR spectrum of compound **4b**



EI-MS spectrum of compound 4b

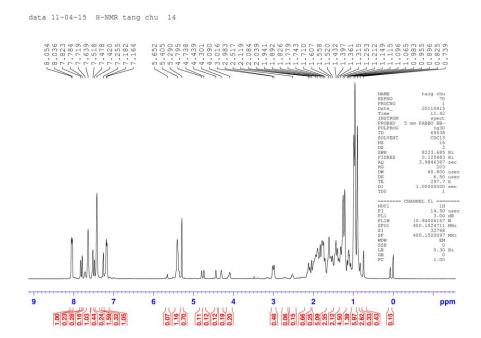
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Sams: 15 migan/MAT95/70eV/Thou:220c/R:10000
Mode: E1 -VE -LARE BSCAN (EXP) UP HR NRM

Oper: NAME_UNSIMM.CAS
Limit: (741)
Peak: 1000.00 mmu R:0: -2.0 > 60.0
Data: CHASS: cornwerted

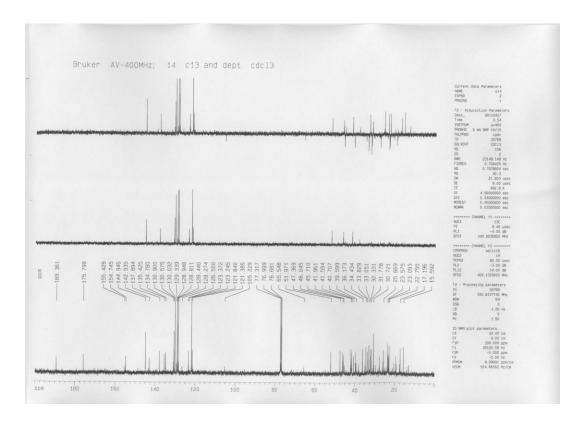
121695

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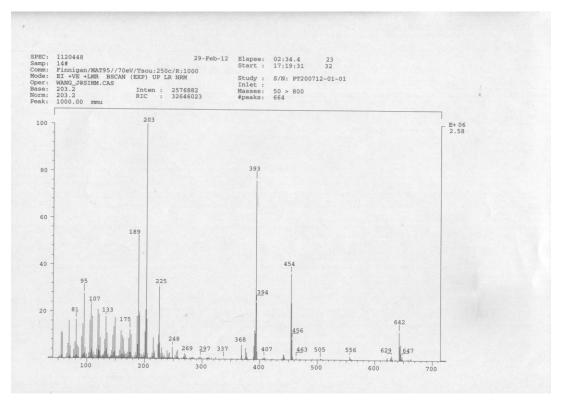
HREIMS spectrum of compound 4b



¹H NMR spectrum of compound **4c**

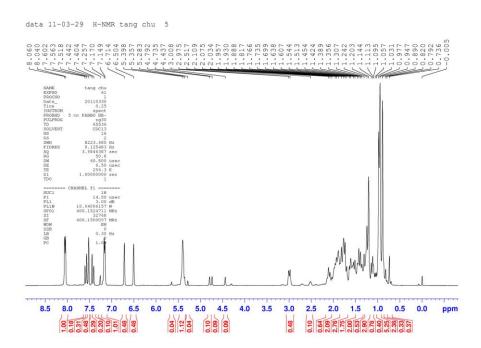


 13 C NMR spectrum of compound 4c

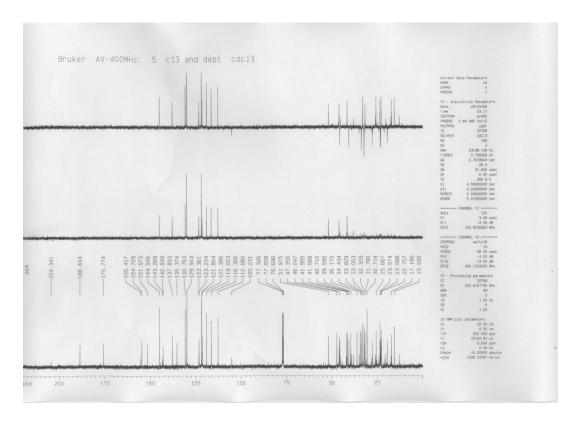


EI-MS spectrum of compound 4c

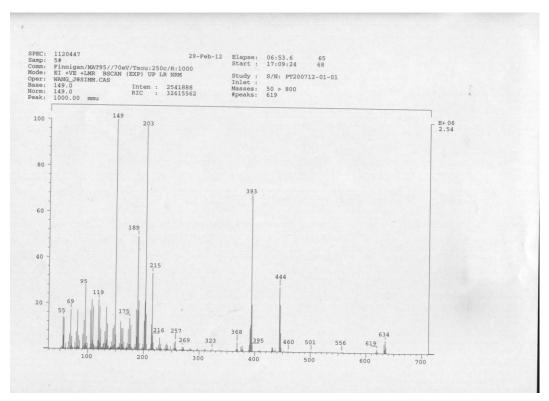
HREIMS spectrum of compound 4c



¹H NMR spectrum of compound **4d**



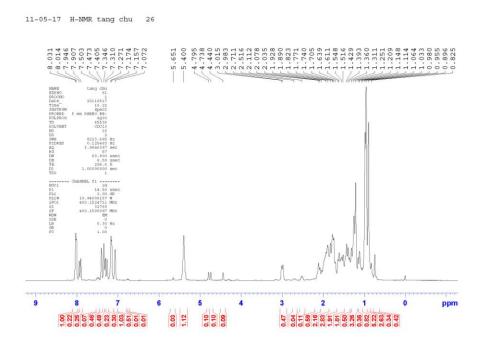
¹³C NMR spectrum of compound **4d**



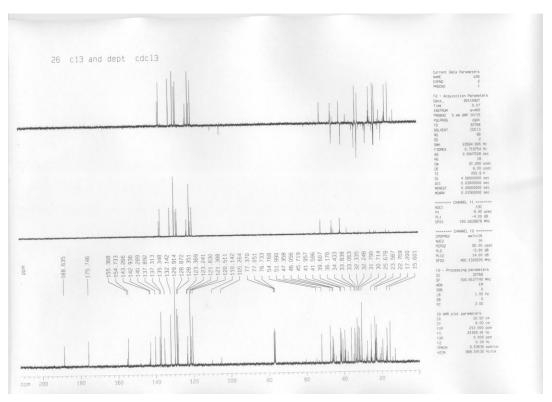
EI-MS spectrum of compound 4d

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Samp: 3 migrar/MACPS//TOWY/Town.220c/R.10000
Mode: EI -WE -LBR BSCAN (EXP) UP HR NRM
Oper: WANA_D85IMM.CAS
Comparison of the Comparison of
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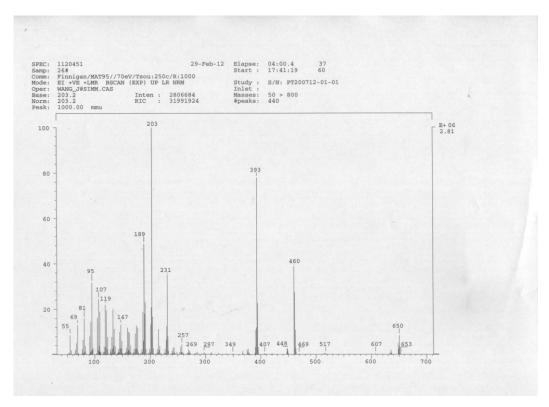
HREIMS spectrum of compound $\mathbf{4d}$



¹H NMR spectrum of compound **4e**



 13 C NMR spectrum of compound **4e**



EI-MS spectrum of compound 4e

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h120256-cl
26#
Finnigan/MA
EI +VE +LME
WANG JESTMA
                                                                                                           20-Mar-12 Elapse: 05:22.4 50
Start: 17:55:06 55
                                                                                                                                      Study : S/N: PT200712-01-01
Inlet :
                                                                                            Delta R+D Composition
-1.0 -0.5 C5.H13.S
-3.2 -1.5 C5.H15.S
  121.1018 * 606434 19.09
                                                                            1.30 -0.1
                                                                                                                     C9.8H3
-1.5 C6.8H7.S
C9.H15
-1.5 C7.H15
-1.5 C7.H17.S
C10.H13
-0.5 C7.H17.S
C11.H13
0.5 C8.H17.S
C11.H13
0.5 C8.H17.S
C12.H15
C12.H15
C12.H15
C12.H15
C12.H15
C12.H17
0.5 C9.H21.S
C12.H9
C13.H17
0.5 C10.H21.S
C13.H19
C13.H21
-1.5 C10.H25.S
                                                      8.09
7.54
19.81
                                                                           0.55 -0.5
0.51 0.5
1.35 -0.5
135.1175 * 387335
                                                      12.19
                                                                            0.83 -0.2
                               299482
                                                       9.43
                                                                           0.88 1.1
0.82 0.7
0.67 -0.3
163.1483 * 275296
173.1330 * 279742
                                                      8.66
8.80
                                                                           0.59 0.4
0.60 0.0
175.1483 * 417568
177.1642 * 362437
                                                     13.14
11.41
                                                                           0.90 0.4
0.78 0.1
                                                                                                                      C13. H.9

C13. H21

-1.5 C10. H25. S

C14. H19

0.5 C11. H23. S

C14. H21

-0.5 C11. H25. S

C14. H23

-1.5 C11. H27. S

C15. H21

0.5 C12. H25. S

C15. H21

0.5 C12. H26. S

C15. H21

0.5 C12. H26. S

C15. H21

0.5 C12. H27. S

C15. H21

-0.5 C12. H27. S

C15. H21

-0.5 C12. H27. S
187.1500 * 590428 18.58
189.1658 * 1510394 47.54
                                                                           1.27 -1.3
3.24 -1.4
190.1716 * 562152 17.69 1.21 0.5
191.1805 * 703890 22.16 1.51 -0.5
201.1656 * 429305 13.51
202.1726 * 658007 20.71
                                                                            0.92 -1.3
                                                                           1.41 -0.5
 203.1806 * 3177110 100.00
204.1850 * 487637 15.35 1.05 2.8 4.0
215.1791 * 354968 11.17 0.76 0.9 5.2
230.0400 * 417568 13.14 0.90 0.2 9.0
231.0482 * 1149201 36.17 2.47 -0.3 8.5
                                                                                                                     13.5 C16.H7.02
C16.H8.02
C26.H47.S
8.5 C29.H43
C29.H44
3.0 C26.H48.S
C29.H45
2.5 C26.H49.S
                                                                          -3,6

0.58 0.7 13.0

0.88 -0.3 3.5

-3.7

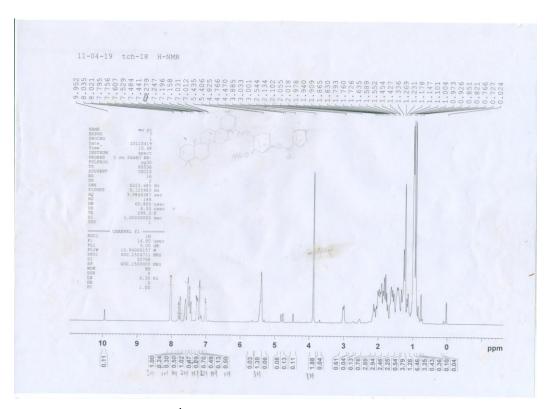
0.81 -0.6 8.0

2.8

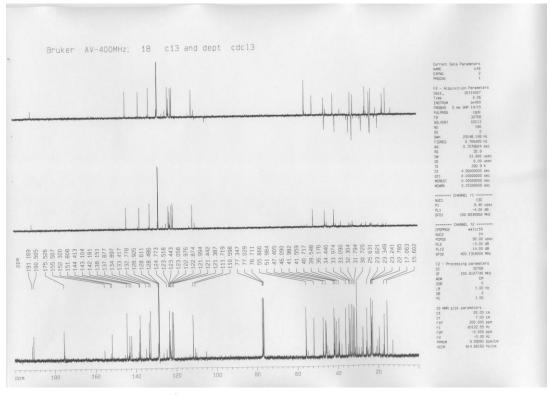
5.24 -1.3 7.5

2.0
232.0517 * 271917 8.56
391.3401 * 410276 12.91
392.3449 * 377020
                                                      11.87
 393.3535 * 2443520
```

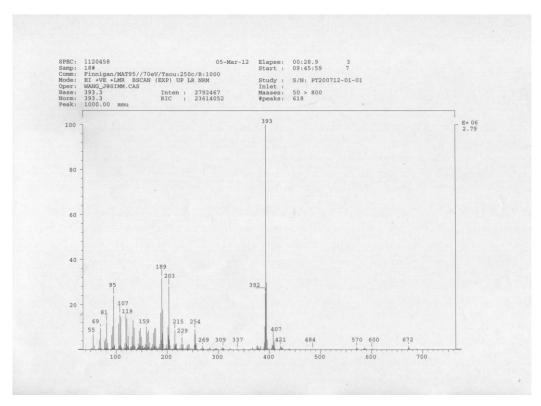
HREIMS spectrum of compound 4e



¹H NMR spectrum of compound **4f**

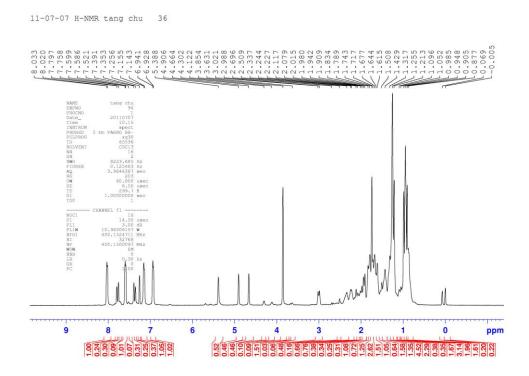


¹³C NMR spectrum of compound **4f**

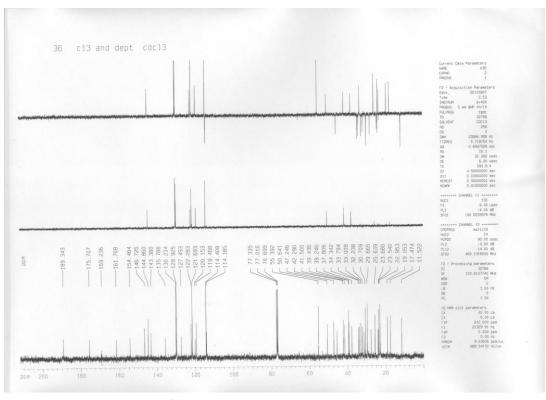


EI-MS spectrum of compound 4f

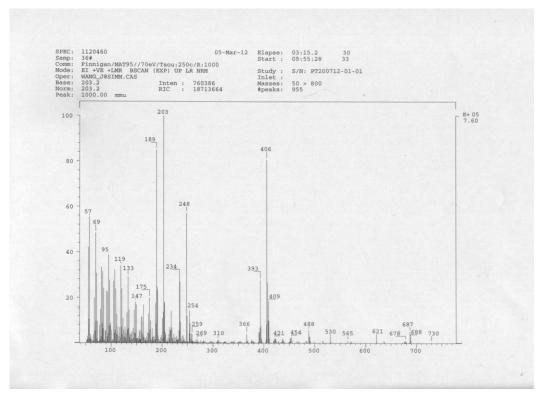
HREIMS spectrum of compound 4f



¹H NMR spectrum of compound **5a**

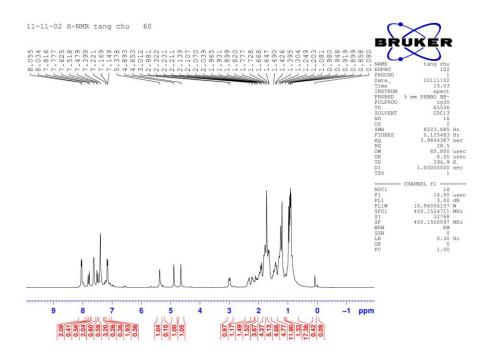


¹³C NMR spectrum of compound **5a**

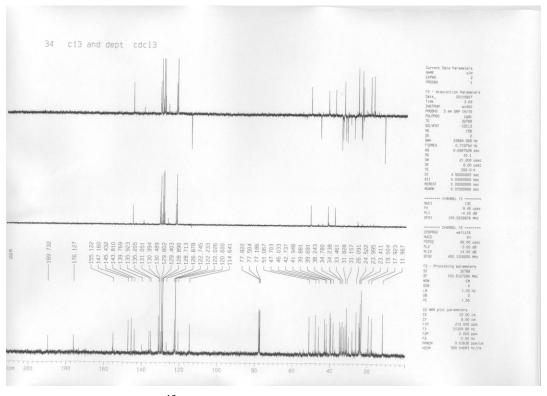


EI-MS spectrum of compound 5a

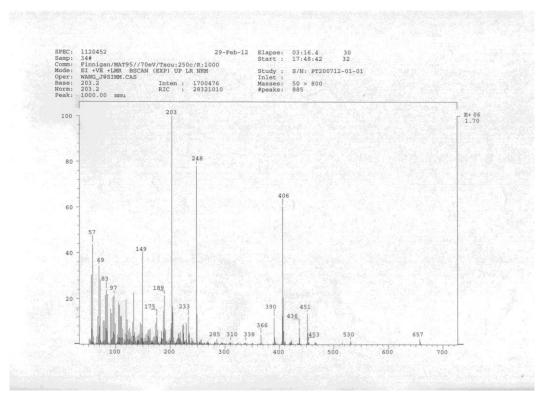
HREIMS spectrum of compound 5a



¹H NMR spectrum of compound **5b**



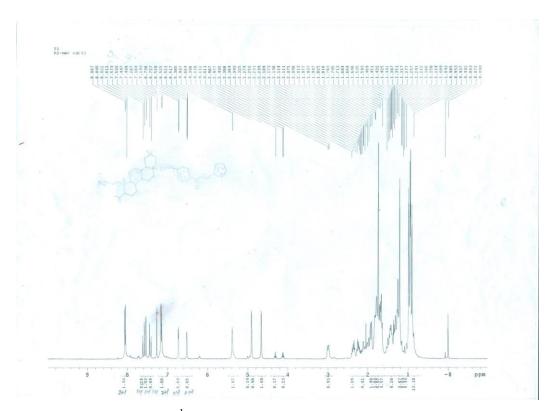
 13 C NMR spectrum of compound **5b**



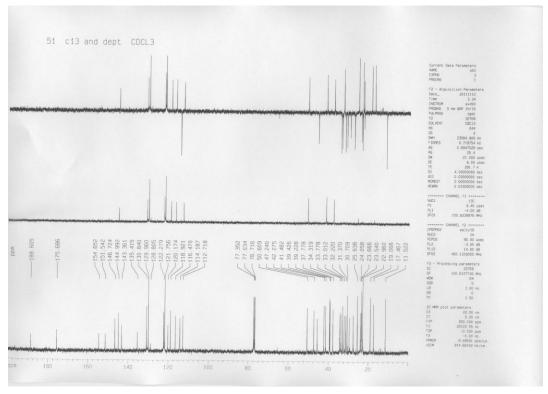
EI-MS spectrum of compound 5b

12.000	Substantia V								
LIST: h	120257-c1				20-1	far-12		03:22.9	31
Samp: 3	40						Start :	18:10:42	39
Comm: F	innigan/MATS I +VE +LMR	95//70eV	Tsou:22	Uc/R:10	0000			S/N: PT200	
Oper: W	ANG JØSIMM.	BSCAN (EXP) UP	HR NRM			Inlet :	S/N: PT200	712-01-01
Limt: (O I	LAD					Turec :		
1 (715) C46.H	00.N.03							
Peak: 1	000.00 mmu	R+D:	-2.0 >	60.0					
Data: C	MASS : conve	erted							
				9-24					
Mann	13521: Intensity * 370618 * 417034 * 618349 * 547747 * 546502 * 314776	ans.	9070	(mmu)	D.D	Common	Inter		
91 05434	* 370618	0.50	0.76	Delta	A 5	CT UT	reron		
93.07042	* 417034	10.79	0.86	0.0	3.5	C7. H9			
95.08550	* 618349	16.01	1.27	0.6	2.5	C7.H11			
105.0678	* 547747	14.18	1.13	2.6	4.5	C8.H9			
107.0832	* 546502	14.15	1.13	2.9	3.5	C8.H11			
109.0990	* 314776	8.15	0.65	2.7	2.5	C8.H13			
119.0855	* 661742	17.13	1.36	0.6	4.5	C9.H11			
121 1016	* 222205	9.50	0.31	0.1	2 =	00 1112			
131 0858	* 291123	7.54	0.68	0.1	5.5	C10 W1	1		
133 1019	* 690018	17.86	1 42	-0.2	4.5	C10 H1	3		
134,1040	* 150630 * 332205 * 291123 * 690018 * 168414	4.36	0.35						
135,1174	* 170014	4.40	0.35	-0.1	3.5	C10.H1	5		
145.1011	* 297526	7.70	0.61	0.6	5.5	C11.H1	3		
147.1161	* 294325	7.62	0.61	1.3	4.5	C11.H1	5		
149.1308	* 690018 168414 170014 * 297526 294325 173216 225678 190288 255200 298059 146362 430372 212874	4.48	0.36	2.3	3.5	C11.H1	7		
159.1156	225678	5.84	0.47	1.8	5.5	C12.H1	5		
160,1150	* 154187	3.99	0.32	-2.4	5.5	C11.H1	4.N		
162 1401	* 255200	6.63	0.39	0.6	3.5	C12.H1	0		
173 1320	* 298059	7 72	0.53	1.0	5 5	C13 H1	7		
174 1350	* 146362	3 79	0.01	4.0	3.3	CTS'HT	,		
175.1482	* 430372	11.14	0.89	0.5	4.5	C13.H1	9		
177.1637	* 212874	5,51	0.44	0.6	3.5	C13.H2	1		
187.1483	* 476788	12.34	0.98	0.4	5.5	C14.H1	9		
188.1512	* 223900	5.80	0.46	4500	125.55				
189.1641	* 712248	18.44	1.47	0.2	4.5	C14.H2	1		
190.1693	240795	6.23	0.50	2.9	4.0	C14.H2	2		
201 1626	* 222122	5.75	0.45	1.7	5 5	C15 N2	1		
202,1694	* 352656	9.13	0.73	2.8	5.0	C15.H2	2		
203.1796	* 3041063	78.72	6.27	0.4	4.5	C15.H2	3		
204.1840	* 521782	13.51	1.08						
205.1591	* 355146	9.19	0.73	0.1	4.5	C14.H2	1.0		
215.1784	* 212874 * 476788 * 223900 * 712248 * 240795 * 210384 * 222122 * 352656 * 3041063 * 521782 * 355146 * 171259 * 250754	4.43	0.35	1.6	5.5	C16.H2	3		
216.1761	* 171259 * 250754 * 174105 * 188866 * 182641 * 326158 * 313176 * 143161	6.49	0.52	-0.8	5.5	C15.H2	2.N		
219.1383	174105	4.51	0.36	0.2	5.5	C14.H1	9.02		
224 0824	* 192641	4.89	0.39	1.6	10.5	C15 H1	1.02		
225 0000	* 326150	9.13	0.38	1.0	0.5	C15 H1	2.02		
233.1529	* 313176	8 11	0.65	1.3	5 5	C15 H2	1 02		
234,1601	* 143161	3.71	0.30	1.9	5.0	C15.H2	2.02		
235.1677	* 183175	4.74	0.38	2.1	4.5	C15.H2	3.02		
248.1777	* 2170003	56.17	4.47	0.0	5.0	C16.H24	1.02		
249.1795	* 408142	10.56	0.84						
390.3175	* 294147 * 135691 * 439620	7.61	0.61	-1.5	9.5	C28.H4	0.N		
404.3259	* 135691	3.51	0.28	200	20020	250071001			
405.3397	439620	11.38	0.91	-0.2	9.0	C29.H4	5.N		
406 3400	* 3863217	100.00	7 00	-2 5	0.5	020 111	26.H45.03		
400.3499	* 1264264	22 72	2.95	0.7	3.5	C26 H4	7 07		
408.3507	* 308730	100.00 32.73 7.99	0.64	-3.0	3.5	C25 H4	5 N 03		
436.3201	* 147784			1.5	9.5	C29 H43	2.N.O2		
451.3452	* 283832			-0.1	9.0	C30.H45	5.N.O2		
452.3411	* 154187	3.99	0.32						
657.4190	* 176239	4.56	0.36	-0.8	19.0	C45.H55	5.N.O3		

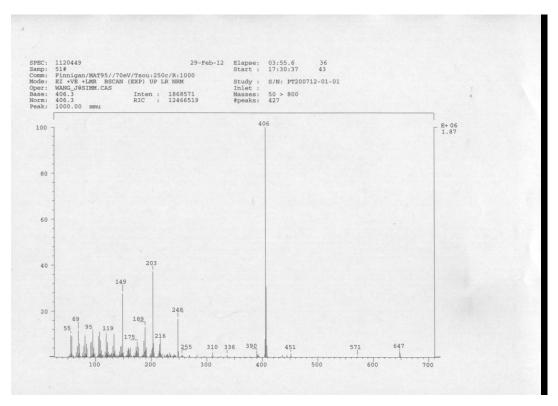
HREIMS spectrum of compound 5b



 ^{1}H NMR spectrum of compound 5c

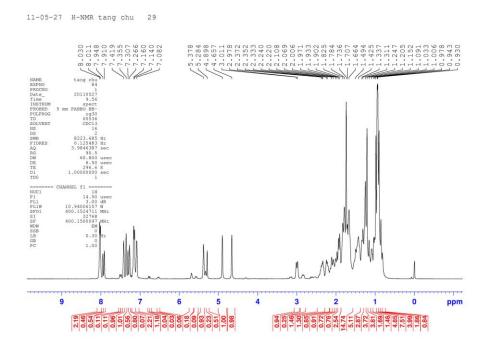


¹³C NMR spectrum of compound **5c**

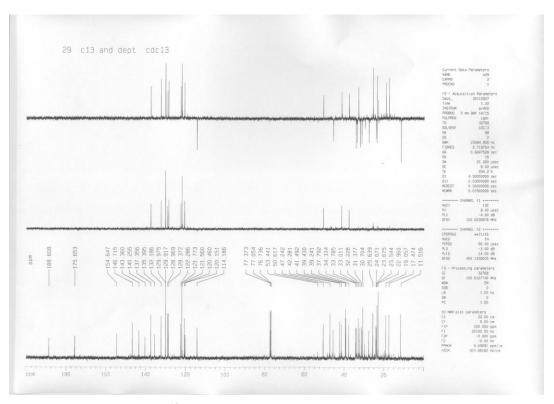


EI-MS spectrum of compound 5c

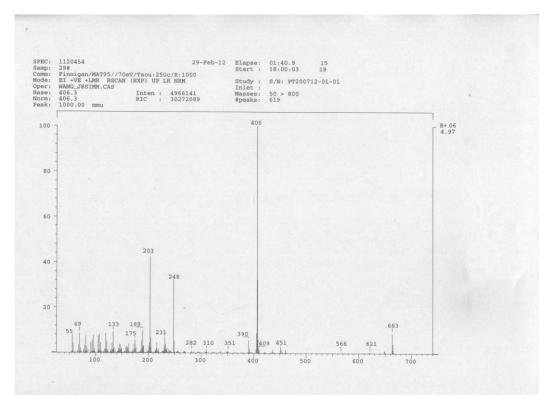
HREIMS spectrum of compound 5c



¹H NMR spectrum of compound **5d**



 13 C NMR spectrum of compound **5d**



EI-MS spectrum of compound 5d

```
LIST: h120259-c1

CAMBER 124 CORNEL 249

CAMBER 244 CORNEL 245 CORNEL 220C/R:10000

Mode: E1+WE +MER SECAN (EKP) UP HR NRM

Oper: MANN_JSENDCAS

Limit: (M/1) C46.H100.N.S.O3

Peak: 1000.00 mmu R-D1 -2.0 > 60.0

Data: CAMSS: converted

92049

Mass Intensity %RA %RIC Delta R-D Composition
91.05533 * 160233 6.96 0.48 -0.6 4.3 CT.NT

93.07105 * 183708 7.98 0.55 -0.6 3.5 CT.N4 (.111.5)

54.08552 * 277628 1.18.5 0.82 0.2 2.5 CT.N1

107.0839 * 277628 1.18.5 0.82 0.2 2.5 CT.N1

110.0996 * 145295 6.31 0.44 2.2 2.5 CR.N1

110.0996 * 145295 6.31 0.44 2.2 2.5 CR.N1

111.0058 * 307663 13.37 0.93 0.35 1.3 CC.N1

113.1008 * 359408 15.4 0.84 2.1 3.5 CC.N1

113.1008 * 359408 15.4 0.84 2.1 3.5 CC.N1

113.10899 * 148140 6.44 0.45 0.2 5.5 Cl.N1

113.11089 * 148140 6.44 0.45 0.2 5.5 Cl.N1

113.11089 * 97456 4.23 0.29 2.1 4.5 CL.N1

115.1157 * 93188 4.05 0.28 1.7 3.5 Cl.NH3

115.1158 * 12037 5.23 0.36 1.8 5.5 CL.NH3

117.1468 * 179440 7.80 0.36 1.3 T.S. CL.NH3

117.1468 * 179440 7.80 0.36 1.8 5.5 CL.NH3

117.1468 * 179440 7.80 0.5 1.8 4.5 CL.NH3

119.1476 * 11209 4.87 0.3 0.3 0.4 5.5 CL.NH3

119.1476 * 11209 4.87 0.3 0.3 0.7 5.5 CL.NH3

119.1476 * 11209 4.87 0.3 0.3 0.7 5.5 CL.NH3

120.1598 * 107959 4.8 5.6 0.3 0.3 0.7 5.5 CL.NH3

121.1476 * 11209 4.87 0.3 0.3 0.7 5.5 CL.NH3

121.1476 * 11209 4.87 0.3 0.3 0.7 5.5 CL.NH3

121.1476 * 11209 4.87 0.3 0.3 0.7 5.5 CL.NH3

122.1480 * 15954 * 15.5 1.1 0.7 0.3 4.5 CL.NH3

123.1598 * 107959 4.8 6.8 4.7 0.3 0.4 5.5 CL.NH3

123.1598 * 107959 4.8 6.8 4.7 0.3 0.4 5.5 CL.NH3

124.1766 * 113106 4.91 0.3 4 4 5.5 CL.NH3

125.1876 * 113106 4.91 0.3 4 5.5 CL.NH3

126.1876 * 113106 4.91 0.3 4 5.5 CL.NH3

127.1688 * 179440 7.8 0.5 0.5 0.5 0.5 CL.NH3

128.1641 * 35954 * 15.9 1.1 0.7 0.3 4.5 CL.NH3

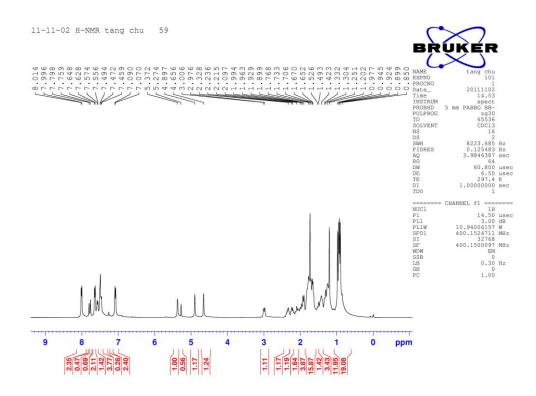
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120.1898 * 107959 * 1.8 6.8 4 0.3 8 -1.5 CL.NH3

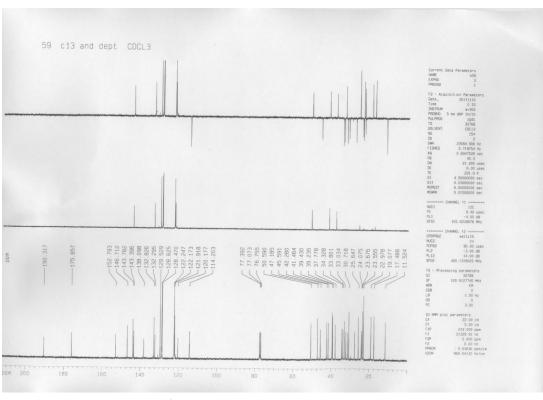
120.1898 * 107959 * 1.8 6.8 4 0.3 8 -1.5 CL.NH3

120.1898 * 107959 * 1.8 6.8 4 0.3 8 -1.5
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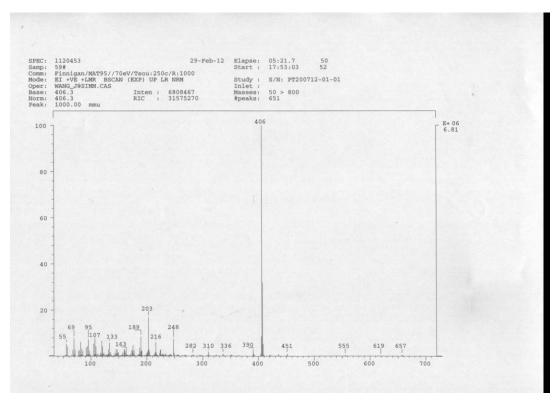
HREIMS spectrum of compound 5d



¹H NMR spectrum of compound **5e**



 13 C NMR spectrum of compound **5e**



EI-MS spectrum of compound 5e

```
Elapse: 05:28.3
Start: 09:20:10
                                                                                                                                      Study : S/N: PT200712-01-01
Inlet :
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1.57
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0.88
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1.49
0.57
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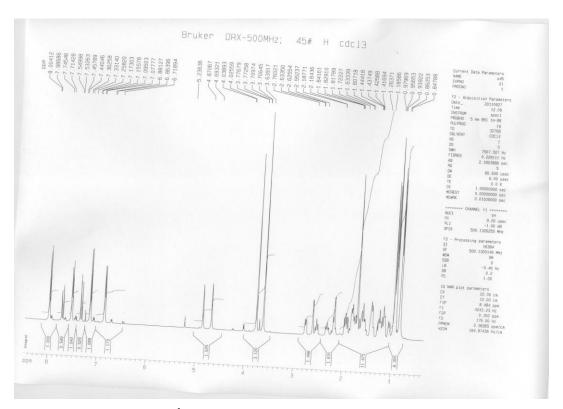
0.35

0.35

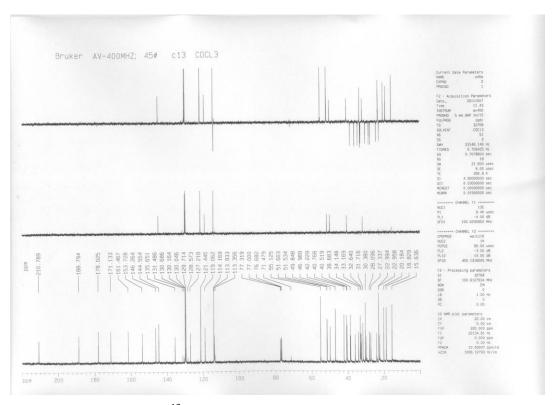
0.35

                                          2.5 C7.H11
                                                                                                                                                                                                                                                                                                                                                                                                                                                                            177.1634
185.1314
185.1314
187.1481
187.1481
188.1493
199.1679
191.1804
199.1480
200.1489
200.1489
202.1679
204.1820
204.1820
214.163
214.163
214.163
214.163
217.1830
218.1895
     0.2 1.5 C7.H13
0.3 5.5 C7.H5.C
0.6 4.5 C8.H9
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       0.6 5.5 C14.H19
0.4 4.5 C14.H21
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       -0.4 3.5 C14.H23
0.7 6.5 C15.H19
0.3 5.5 C9.H9
1.4 5.0 C9.H10
-0.3 4.5 C9.H11
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       2.4 5.5 C15.H21
0.1 4.5 C15.H23
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               0.8 6.5 C16.H21
-2.7 6.5 C15.H20.N
2.0 5.5 C16.H22
-0.4 5.5 C15.H22.N
0.0 5.0 C15.H22.N
1.3 4.5 C15.H22.N
1.3 4.5 C15.H23.N
1.3 4.5 C15.H24.N
1.3 4.5 C15.H24.N
0.6 10.5 C15.H24.0
0.8 10.0 C15.H22.0
0.1 9.5 C15.H13.02
                                          3.5 C10.H15
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  0.36
0.66
0.85
0.16
0.19
0.16
0.14
0.15
0.13
0.11
0.11
0.12
0.12
  0.3 6.5 Cll.Hll
1.0 5.5 Cll.Hl3
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 -2.6 6.5 C16.H22.N
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              6.5 C17.H24.N
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              5.5 C17.H26.N
                                                                                                                                                                                                                                                                                                                                                                                                                                                                               336.2691 *
390.3149 *
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       0.89
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           357525
1.9 5.5 C13.H17
-2.2 5.5 C12.H16.N
2.1 4.5 C13.H19
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    0.67
1.81
8.65
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               0.14
0.37
1.78
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               -1.5
0.5 5.0
0.1 9.0
-2.6
-1.0 4.0
1.7
1.6 3.5
  1.0 3.5 C13.H21
1.7 6.5 C14.H17
                                                                                                                                                                                                                                                                                                                                                                                                                                                                               406.3457 * 16650496
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            100.00
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            20.59
                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 30.11
5.06
0.53
0.50
0.6 5.5 C14.H19
0.4 4.5 C14.H21
```

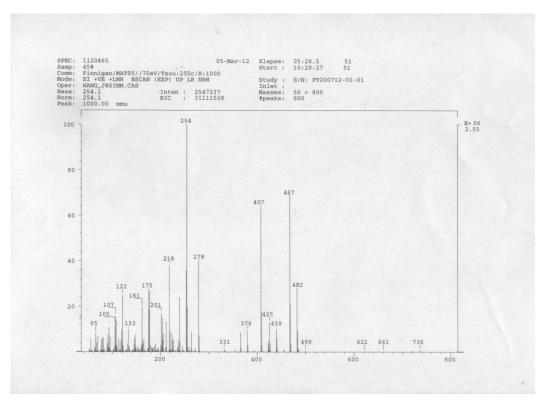
HREIMS spectrum of compound 5e



¹H NMR spectrum of compound **6a**



¹³C NMR spectrum of compound **6a**



EI-MS spectrum of compound 6a

```
Liff: Nilo270-c1

Semp: 458
Comm: Finniqan/RAT95//ToeV/Tsou; 220-/Rij0000
Mode: EI - VE - Lifk: BSCAN (EXP) UP NR NNM

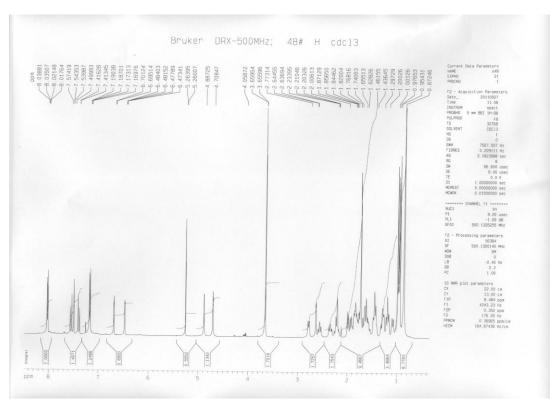
Comm: Finniqan/RAT95//ToeV/Tsou; 220-/Rij0000
Mode: EI - VE - Lifk: BSCAN (EXP) UP NR NNM

Study: 1 (7.89) C48 (100.07)
Peak: 1006.00 mm. RD; -2.0 > 60.0

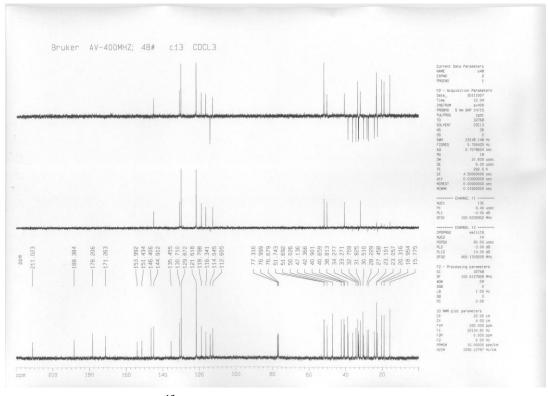
Data: CMMS: 1 (7.89) C48 (100.07)
Peak: 1006.00 mm. RD; -2.0 > 60.0

Mans: Inter: CMMS: 1 (7.60) C6 (1.78) C7 (1
```

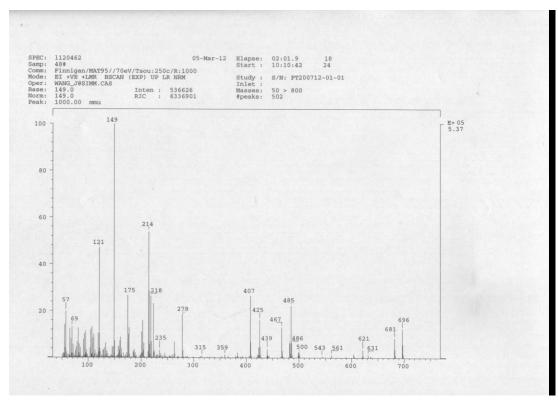
HREIMS spectrum of compound 6a



¹H NMR spectrum of compound **6b**

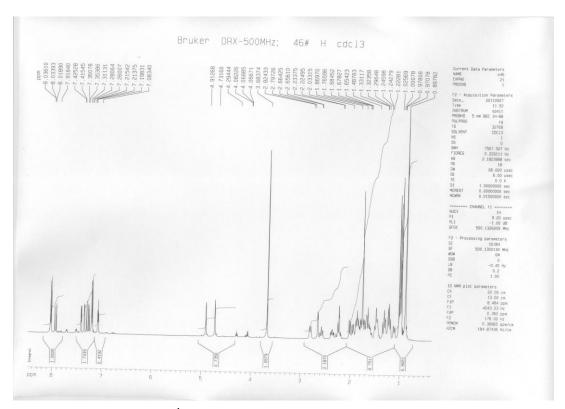


¹³C NMR spectrum of compound **6b**

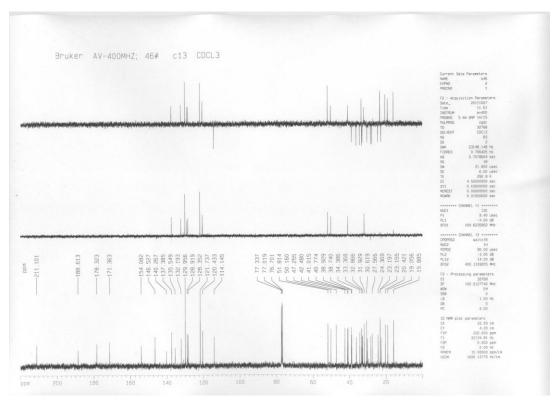


EI-MS spectrum of compound 6b

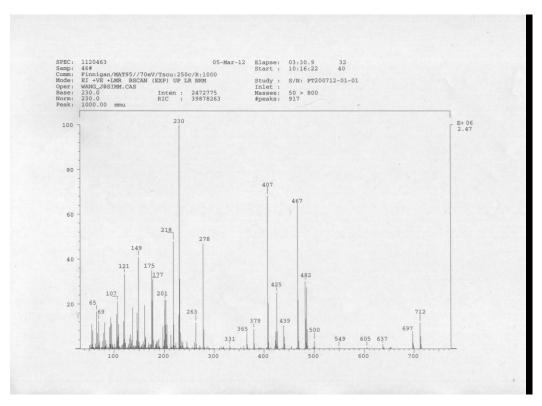
HREIMS spectrum of compound 6b



 1 H NMR spectrum of compound 6c



¹³C NMR spectrum of compound **6c**



EI-MS spectrum of compound 6c

```
Elapse: 06:26.1
Start: 10:46:39
                                                                                                            Study : S/N: PT200712-01-01
Inlet :
                                           %RA
8.96
21.51
                       349643
839741
145.1017 * 238515
146.0372 * 771644
                                           6.11
                                                             0.30 0.0
0.96 -0.4
201.1649 *
                        935104
                                           23.96
                                                             1.17 -0.5
202.0442 * 527021
203.1444 * 1014775
                                           26.00
                                                             1.26 -0.8
                                                            0.39 1.6
0.62 -0.1
0.32 -1.0
204.1498 * 316482
205.1594 * 496843
213.0384 * 253924
                                          8.11
12.73
6.50
218.1682 * 2171496
                                           55.63
                                                            2.71 -1.2
219.1740 * 527376
229.0319 * 632397
                                           13.51
16.20
                                                             0.66
                                                                        1.9 5.5 C16.H23.O3
-0.2 5.0 C17.H26.O3
                                                           1.04 0.8 -0.5 C22.H87.S.05 -2.6 4.5 C25.H83.05 -2.6 4.5 C25.H83.05 -1.2 8.5 C28.H81.03 -1.2 8.5 C28.H81.03 -1.2 8.5 C28.H84.03 -1.3 0.30 -0.5 8.0 C29.H84.03 -1.3 0.30 -0.5 8.0 C29.H84.03 -1.3 0.3 0.0 9.5 C30.H83.08 -1.0 0.0 9.5 C30.H83.08 -1.0 0.0 9.5 C30.H83.08 -1.0 0.0 9.5 C30.H83.08
                                           19.11
                                           10.74 6.10
439.3214 * 419373
440.3297 * 238231
                                                                          2.1 9.0 C31.H46.O4
```

HREIMS spectrum of compound 6c