The Design and Synthesis of Fluorinated Dendrimers for Sensitive ^{19}F MRI

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Supporting Information

1. General information

^{1}H, ^{19}F and ^{13}C NMR spectra were recorded on a 400 MHz or 500 MHz. Chemical shifts are in ppm and coupling constants (*J*) are in Hertz (Hz). ^{1}H NMR spectra were referenced to tetramethylsilane (d, 0.00 ppm) using $CDCl_3$ as solvent or acetone (p, 2.06 ppm) using acetone-*d*, ^{13}C NMR spectra were referenced to solvent carbons (77.16 ppm for $CDCl_3$ or 29.84 ppm for acetone-d_6). ^{19}F NMR spectra were referenced to 2% perfluorobenzene (s, -164.90 ppm) in $CDCl_3$ or acetone-d_6. The splitting patterns for ^{1}H NMR spectra are denoted as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), b (broad) and combinations thereof. ESI mass spectra were recorded on a LTQ Orbitrap spectrometer. MALDI-TOF mass spectra were recorded using the reflector mode for positive ions with α-cyano-4-hydroxylcinnamic acid as matrix. High resolution mass spectra were recorded on a 7.0 Tesla or 4.7 Tesla FTMS.

^{19}F MRI experiments of dendrimer **2** were performed on a 9.4 T microimaging system with a 10 mm inner diameter ^{19}F coil (376.4 MHz) for both radiofrequency transmission and reception. The MSME (Multi Slice Multi Echo) pulse sequence was employed for all MRI acquisitions with single average. FOV=8 x 8 mm^2, SI=40.0 mm TR=2500 ms and TE=7.6 ms were used for dendrimer **2** and FOV=8 x 8 mm^2, SI=40.0 mm TR=1000 ms and TE=7.6 ms were used for trifluoroethanol. The data collection time was 160 ms. All raw data were processed using the Matlab.

Unless otherwise indicated, all reagents were obtained from commercial supplier and used without prior purification. DMF, Et_3N and acetone were dried and freshly distilled prior to use. Flash chromatography was performed on silica gel (200-300 mesh) with either EtOAc/petroleum ether (PE, 60-90 ^{o}C) as eluents. All reactions were performed under an argon atmosphere.

2. Figure S1. ^{19}F NMR of dendrons 3, 10, 12, 14, and dendrimer 1

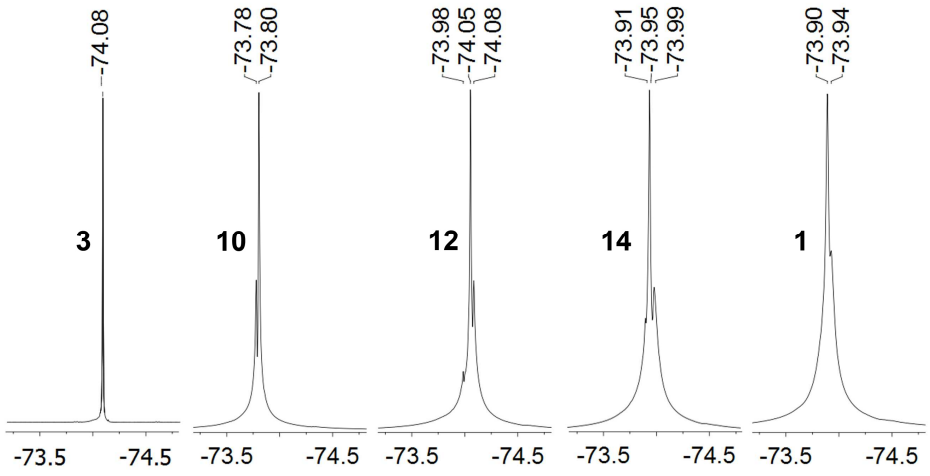
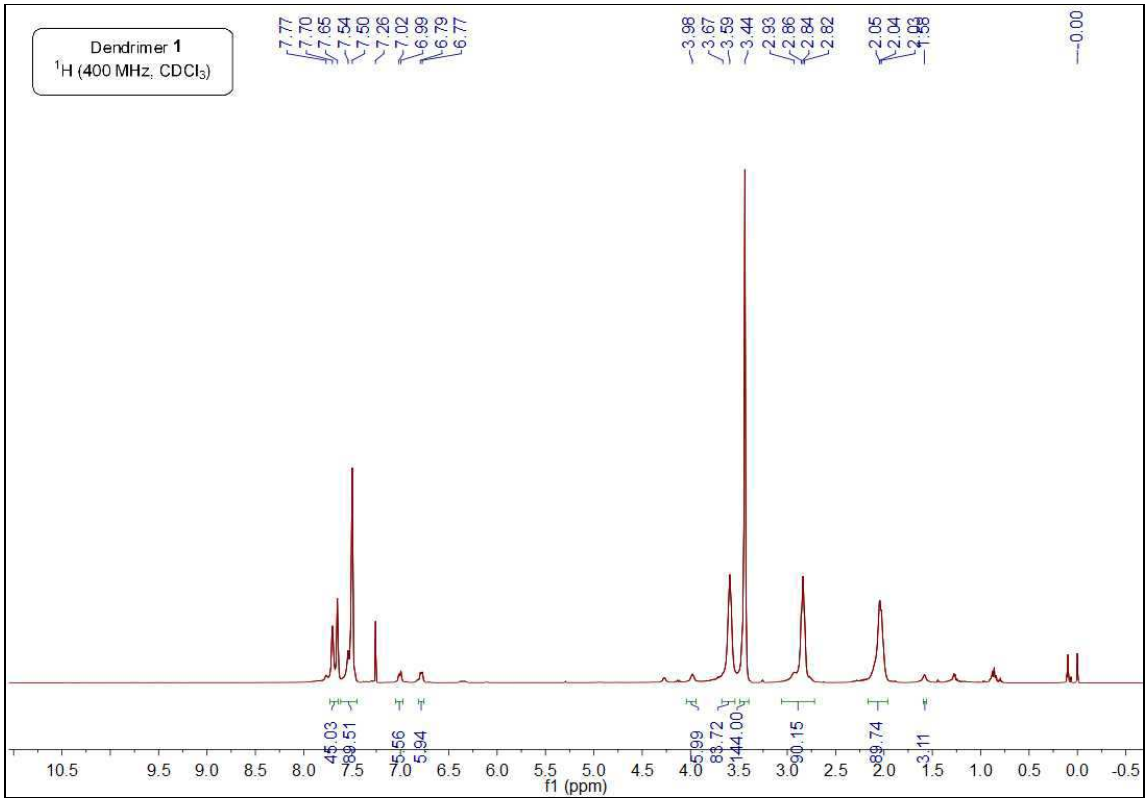
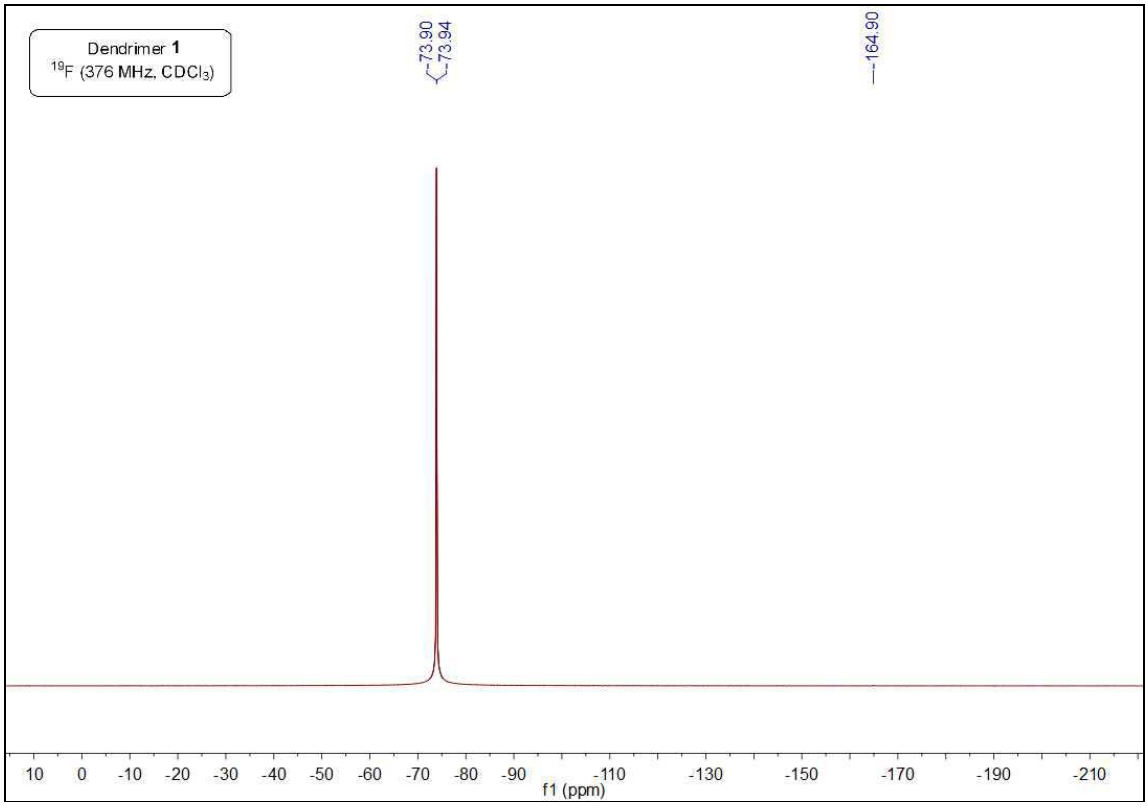
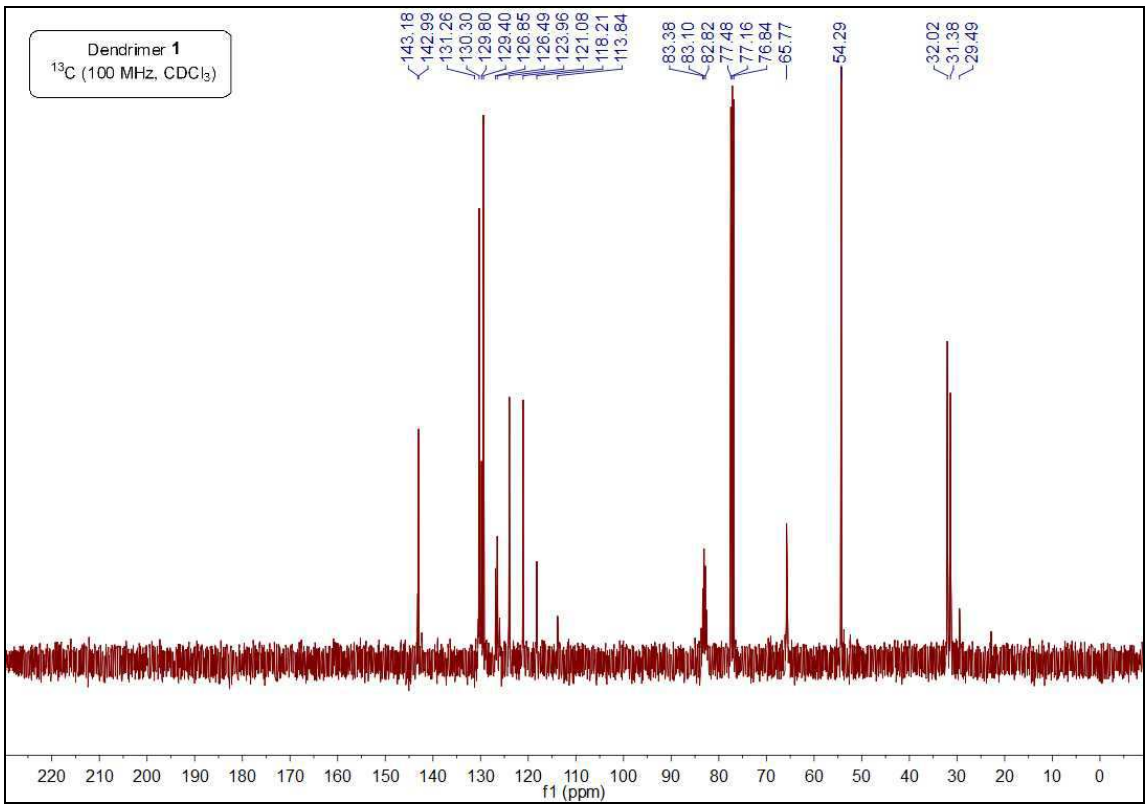


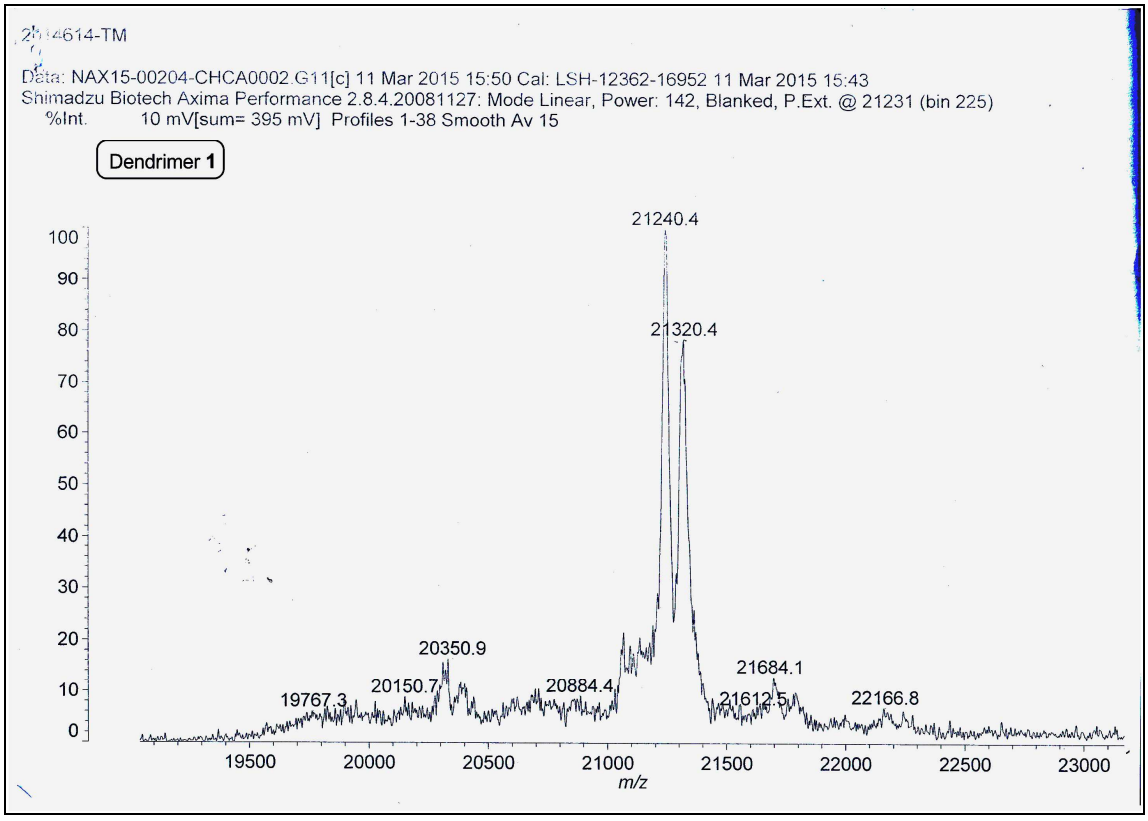
Figure S1. ^{19}F NMR of dendrons **3**, **10**, **12**, **14**, and dendrimer **1** (^{19}F NMR: 376 MHz, 25 ^{o}C, $CDCl_3$ as solvent).

3. Copies of ^{1}H NMR, ^{19}F NMR, ^{13}C NMR and MS spectra of compounds






Dendrimer 1
^{13}C (100 MHz, $CDCl_3$)
143.18
142.99
131.26
130.30
129.80
129.40
126.85
126.49
123.96
121.08
118.21
113.84
83.38
83.10
82.82
77.48
77.16
76.84
65.77
54.29
32.02
31.38
29.49
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

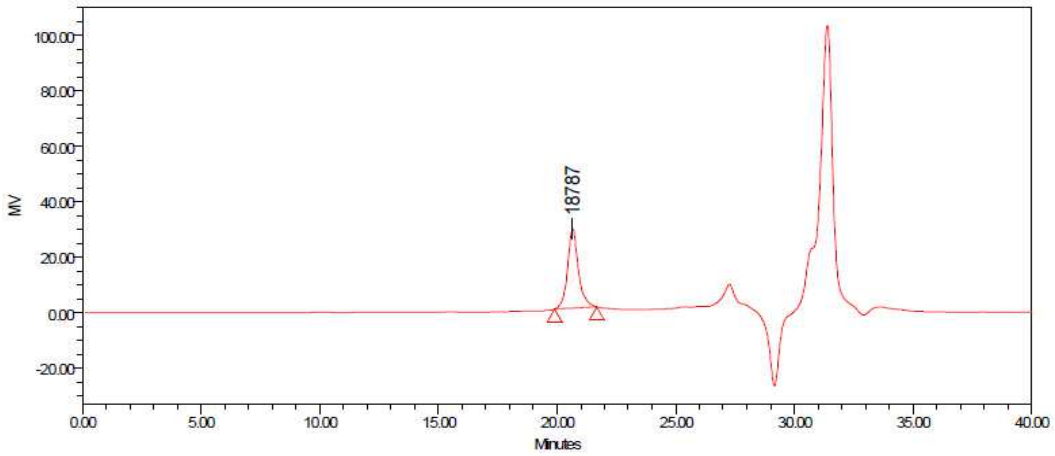

Data: NAX15-00204-CHCA0002.G11[c] 11 Mar 2015 15:50 Cal: LSH-12362-16952 11 Mar 2015 15:43
Shimadzu Biotech Axima Performance 2.8.4.20081127: Mode Linear, Power: 142, Blanked, P.Ext. @ 21231 (bin 225)
%Int. 10 mV[sum= 395 mV] Profiles 1-38 Smooth Av 15
Dendrimer 1
21240.4
21320.4
20350.9
20150.7
19767.3
20884.4
21684.1
21612.5
22166.8
100 90 80 70 60 50 40 30 20 10 0
19500 20000 20500 21000 21500 22000 22500 23000
m/z

Dendrimer **1**

SAMPLE INFORMATION

Sample Name:	1	Acquired By:	System
Sample Type:	Broad Unknown	Date Acquired:	12/20/2014 6:40:32 PM
Vial:	86	Acq. Method Set:	csj_no_2487
Injection #:	1	Date Processed:	12/20/2014 7:38:04 PM
Injection Volume:	150.00 ul	Processing Method:	lyan20141221
Run Time:	40.0 Minutes	Channel Name:	410
Sample Set Name:	20141221	Proc. Chnl. Descr.:	

Auto-Scaled Chromatogram



GPC Results

	Dist Name	Mn	Mw	MP	Mz	Mz+1	Mv	Polydispersity	MW Marker 1	MW Marker 2
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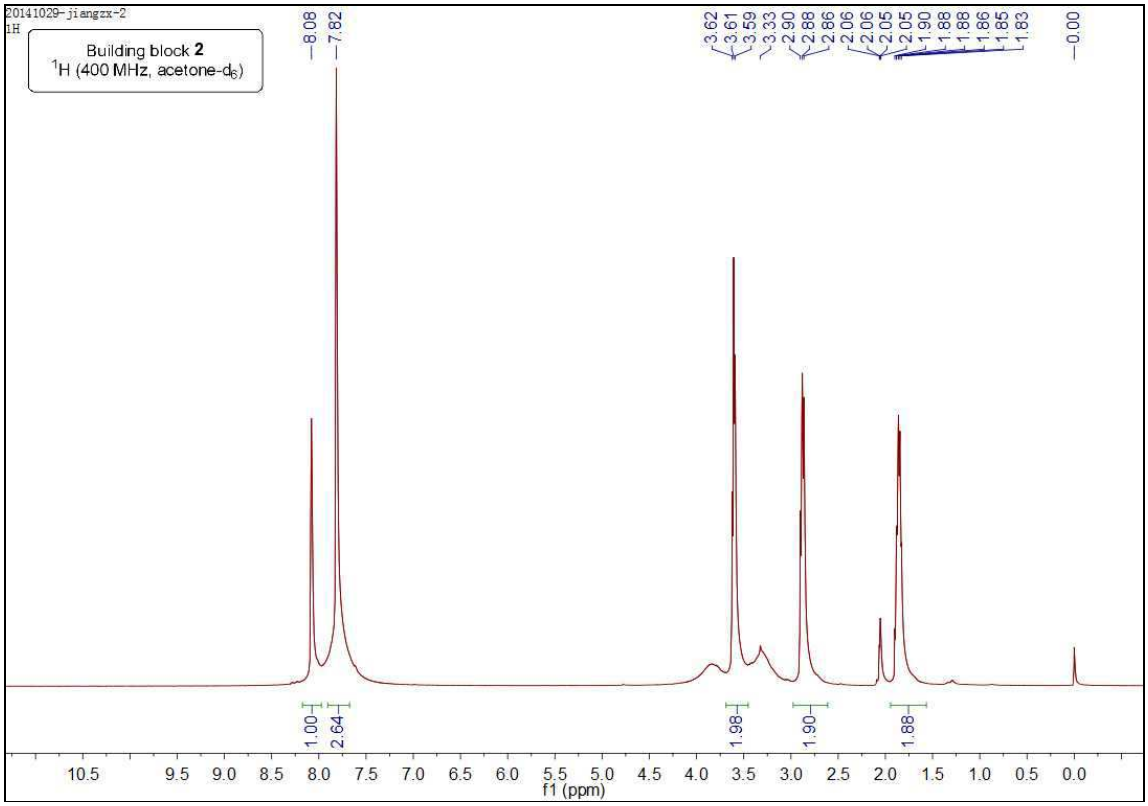

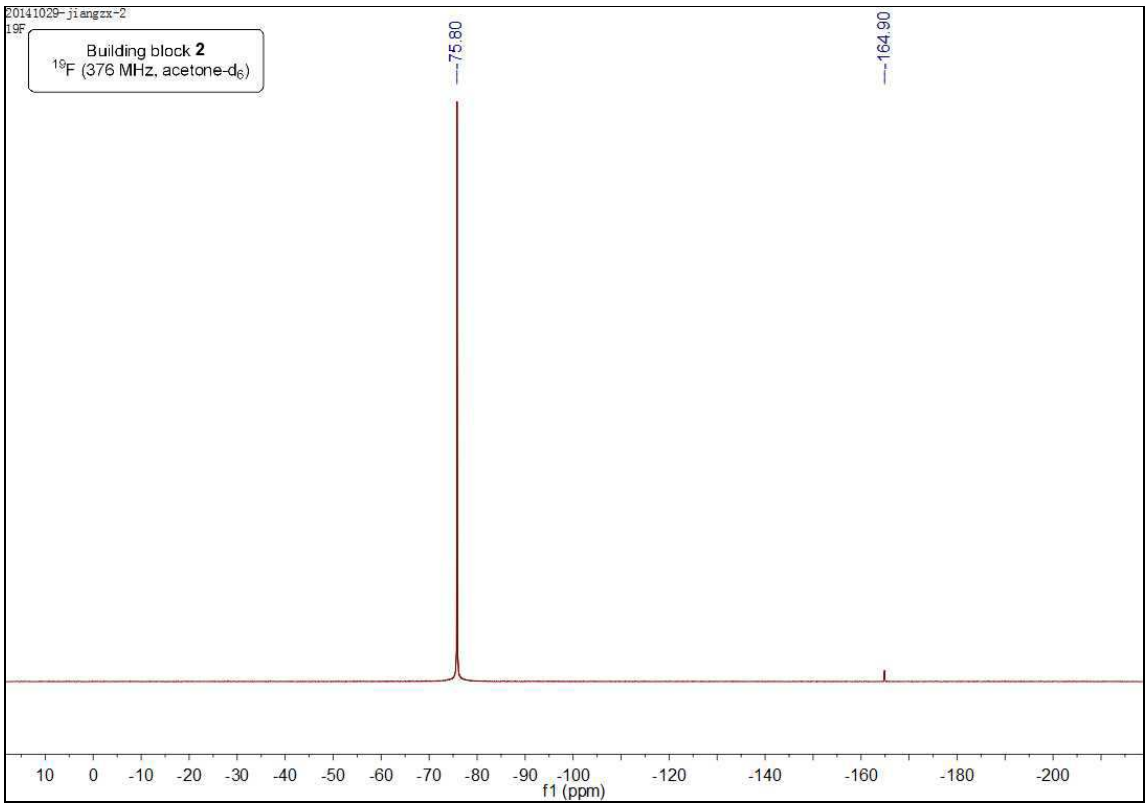
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19F
Building block 2
19F (376 MHz, acetone-d6)
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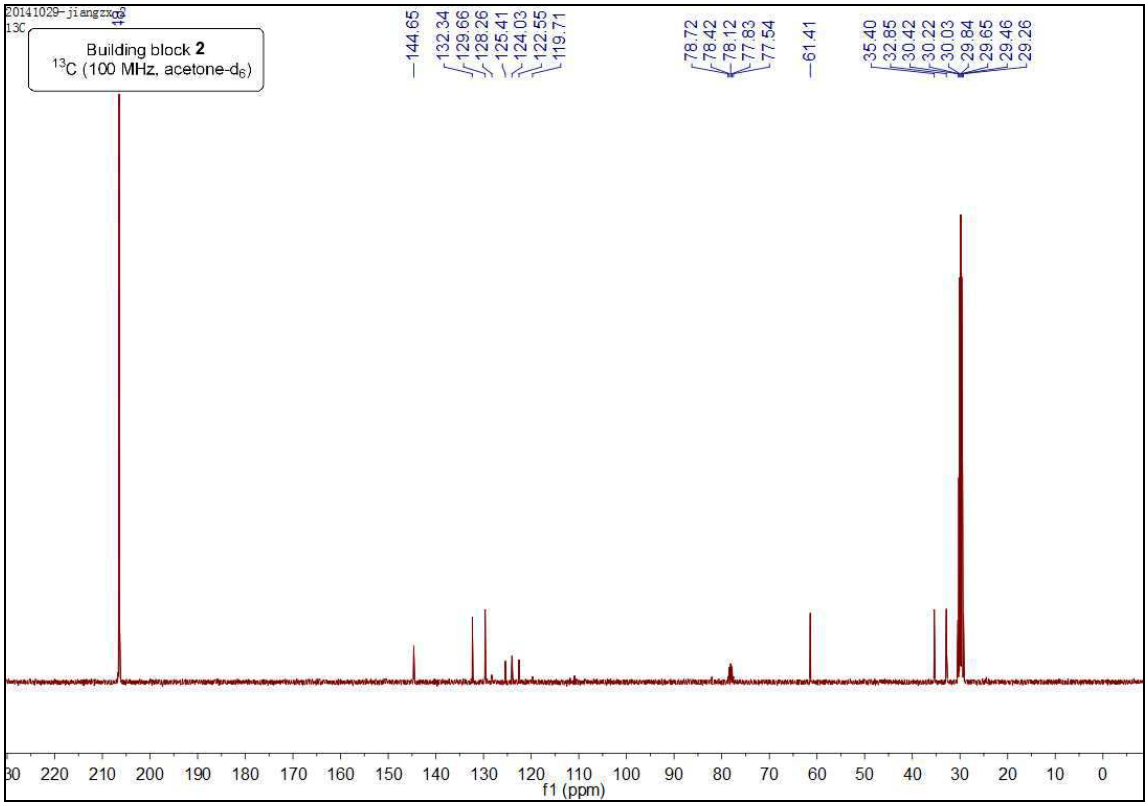
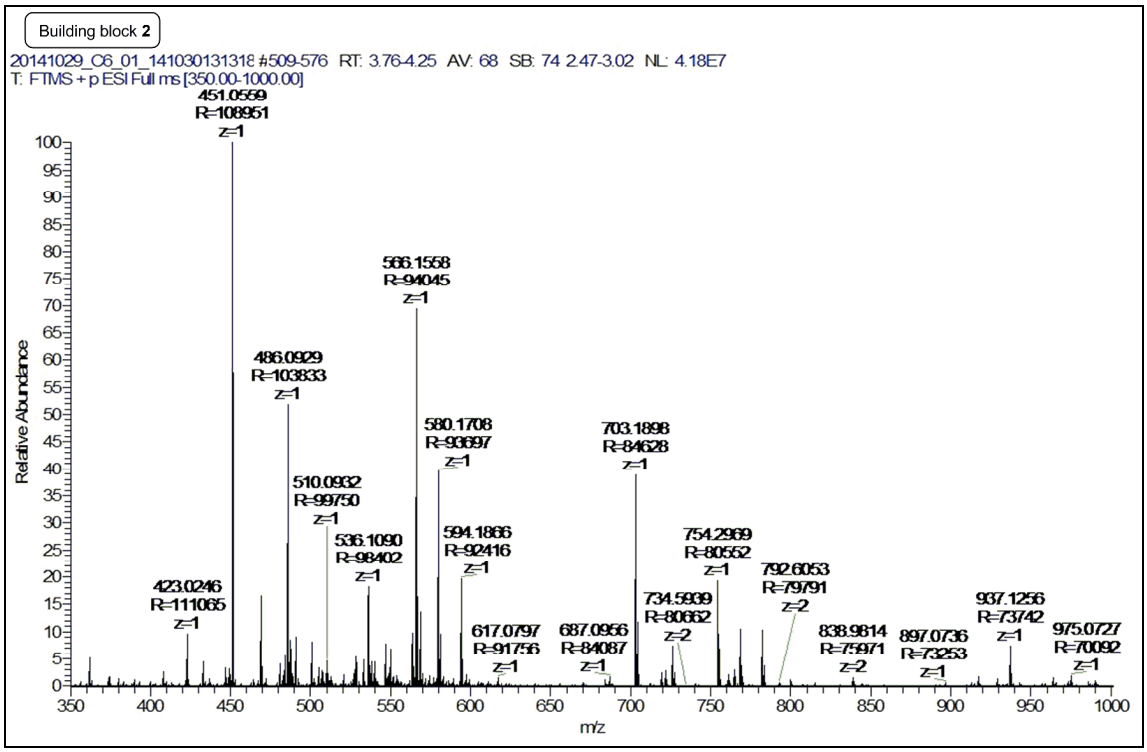
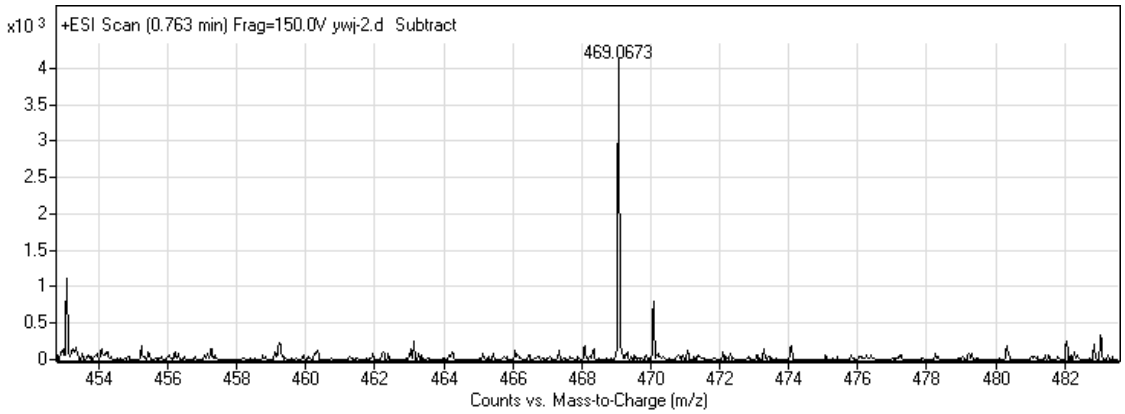
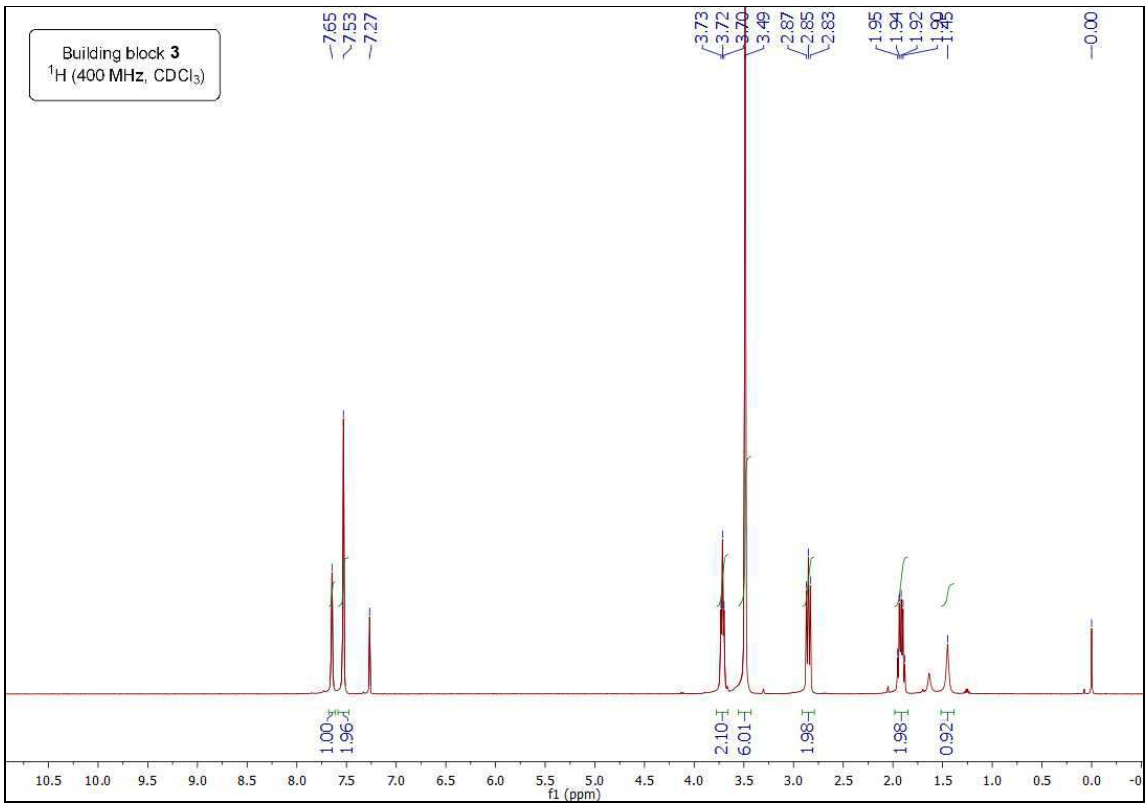
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)

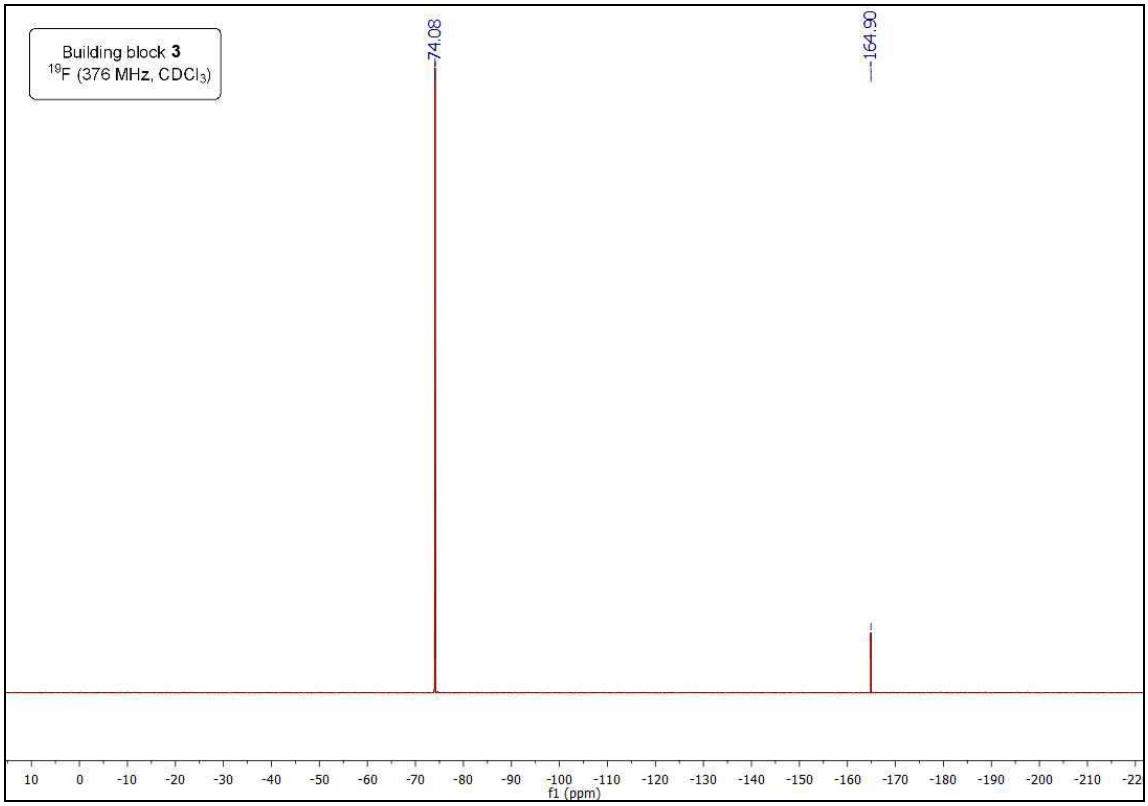

20141029-jiangzx-2
13C
Building block 2
13C (100 MHz, acetone-d6)
144.65
132.34
129.66
128.26
125.41
124.03
122.55
119.71
78.72
78.42
78.12
77.83
77.54
61.41
35.40
32.85
30.42
30.22
30.03
29.84
29.65
29.46
29.26
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



HRMS of building block **2**




Building block **3**
^{1}H (400 MHz, $CDCl_3$)
7.65
7.53
7.27
3.73
3.72
3.70
3.49
2.87
2.85
2.83
1.95
1.94
1.92
0.00
1.00
1.96
2.10
6.01
1.98
1.98
0.92
f1 (ppm)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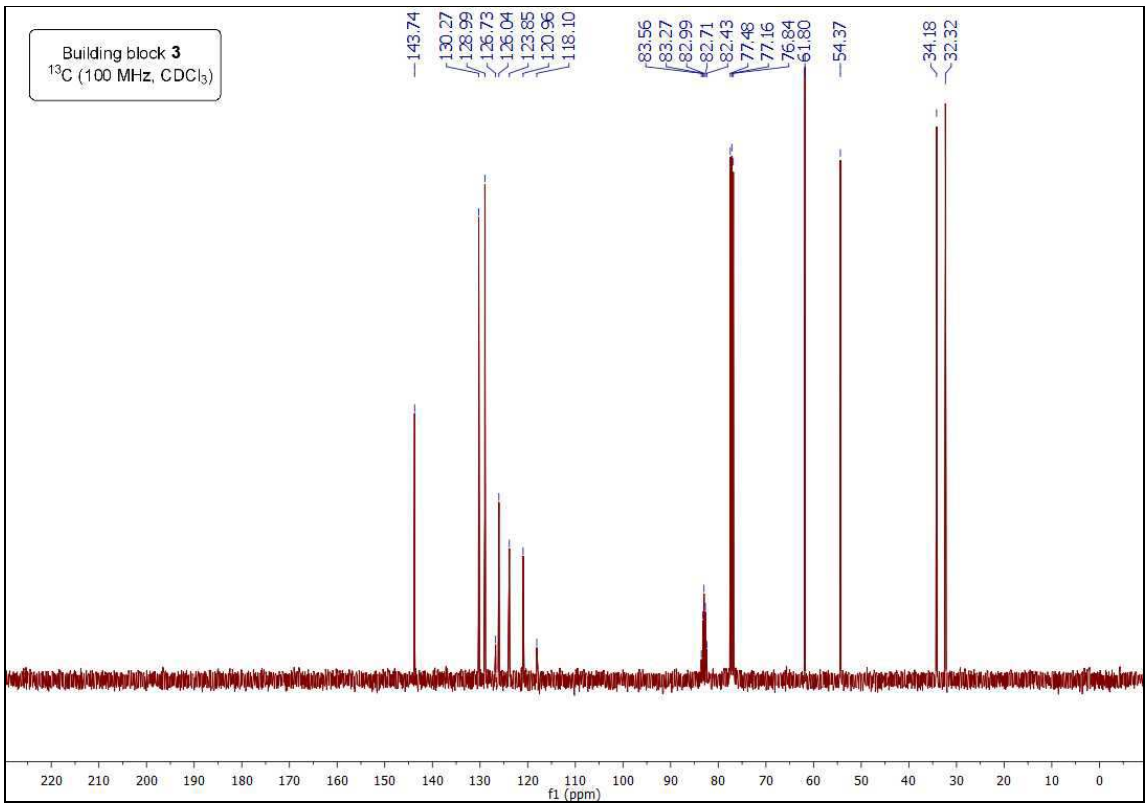
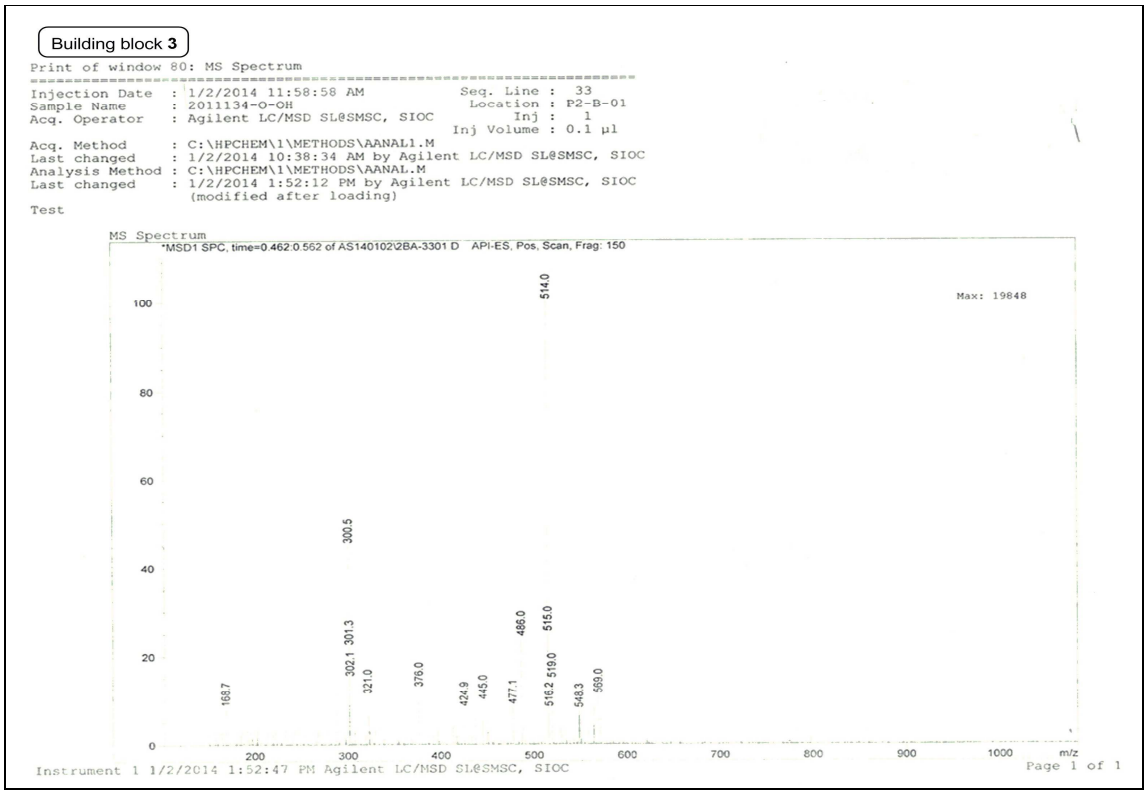
-164.90
f1 (ppm)


Building block 3
^{13}C (100 MHz, $CDCl_3$)
143.74
130.27
128.99
126.73
126.04
123.85
120.96
118.10
83.56
83.27
82.99
82.71
82.43
77.48
77.16
76.84
61.80
54.37
34.18
32.32
f1 (ppm)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Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC Inj : 1
Inj Volume : 0.1 µl
Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 1/2/2014 10:38:34 AM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL.M
Last changed : 1/2/2014 1:52:12 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)
Test
MS Spectrum
*MSD1 SPC, time=0.462:0.562 of AS140102\2BA-3301 D API-ES, Pos, Scan, Frag: 150
Max: 19848
514.0
300.5
302.1
301.3
321.0
376.0
486.0
515.0
516.2
519.0
168.7
424.9
445.0
477.1
548.3
569.0
m/z
Instrument 1 1/2/2014 1:52:47 PM Agilent LC/MSD SL@SMSC, SIOC
Page 1 of 1

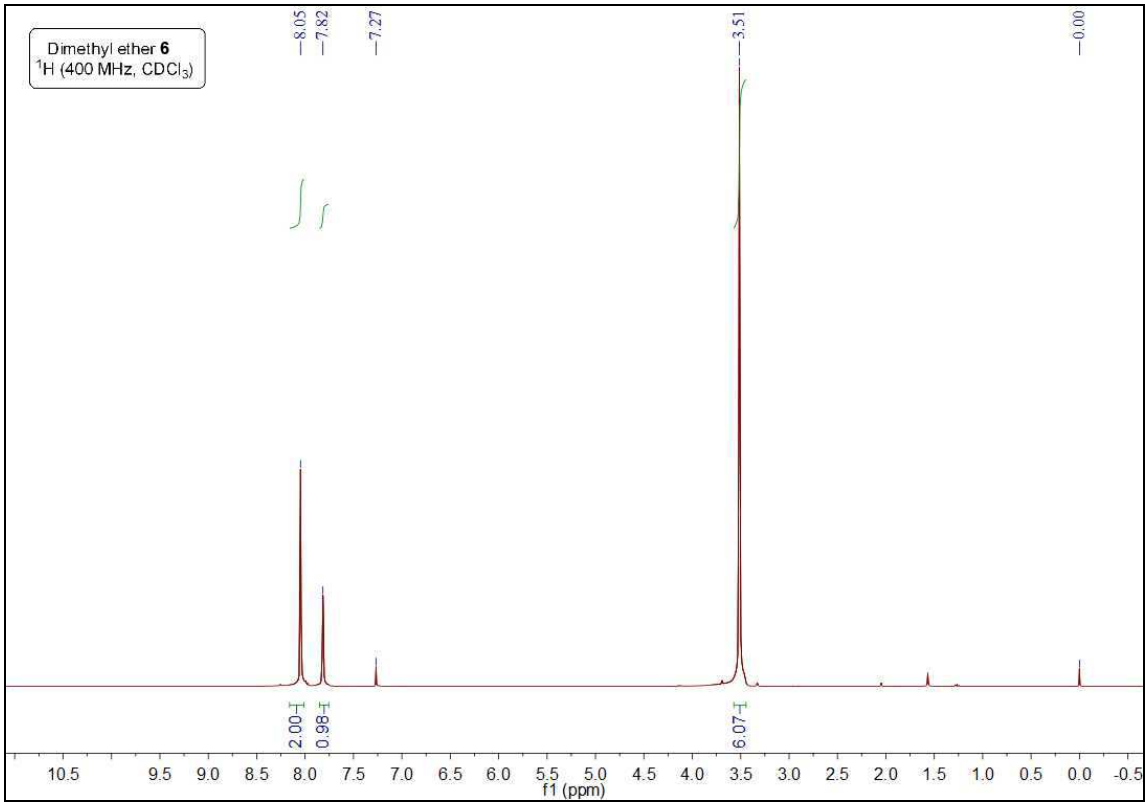
Building block **3**

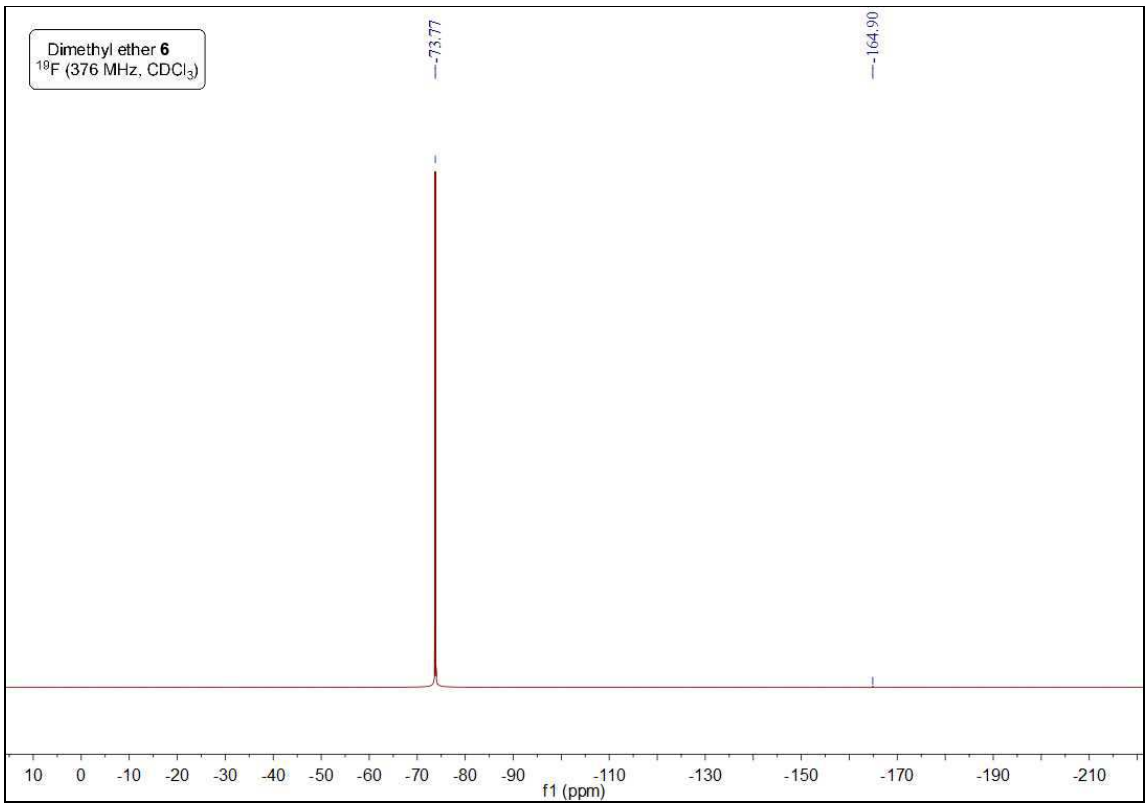
Instrument

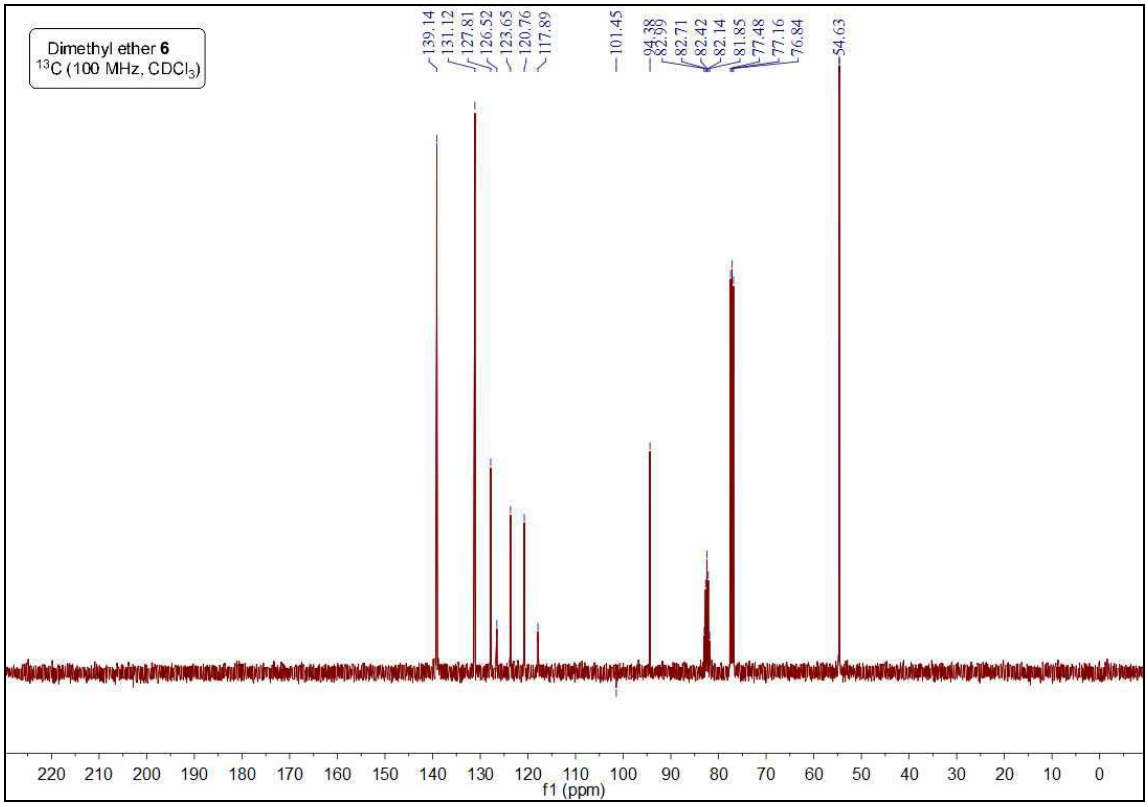
Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS

Card Serial Number	E140204
Analysis Name	D:\Data\zfj2014\20140114_000034.d
Sample Name	2011134-O-OH
Acquisition Date	5/15/2013 3:45:51 PM
Operator:	zfj
Ionization Mode	ESI-Positive
Ion Mass (Measured)	519.0785

Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 17 H 16 F 8 N 2 Na 1 O 6	0.018	519.0773	-2.25	-2.92	-1.17	6.50	ok	even
C 17 H 16 F 12 Na 1 O 3	0.013	519.0800	2.99	2.39	1.55	3.50	ok	even
C 20 H 15 F 7 N 2 Na 1 O 5	0.030	519.0761	-4.46	-5.08	-2.31	10.50	ok	even
C 20 H 15 F 11 Na 1 O 2	0.023	519.0789	0.79	0.16	0.41	7.50	ok	even




Dimethyl ether **6**
^{19}F (376 MHz, $CDCl_3$)
-73.77
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)


Dimethyl ether **6**
^{13}C (100 MHz, $CDCl_3$)
139.14
131.12
127.81
126.52
123.65
120.76
117.89
101.45
94.38
82.99
82.71
82.42
82.14
81.85
77.48
77.16
76.84
54.63
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Dimethyl ether **6**

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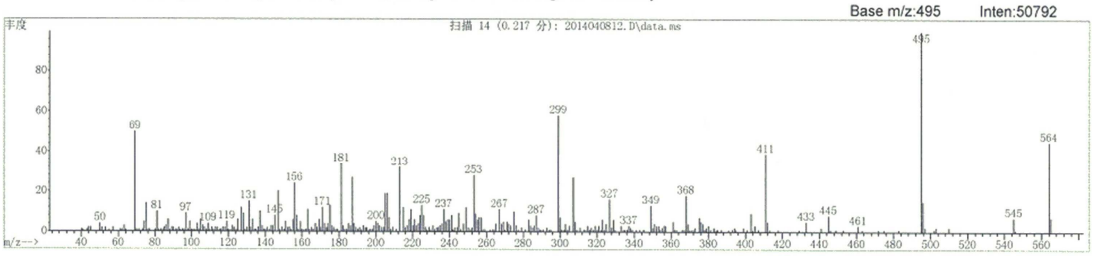
Instrument : Agilent Technologies 5973N
: Shanghai Mass spectrometry Center, Shanghai Institute of Organic Chemistry

Base m/z:495 Inten:50792



m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%
40	0.6	41	0.68	43	0.88	44	2.04	45	2.23	47	0.43	48	0.43	50	3.77	51	2.24
53	1.6	54	0.37	55	0.6	56	0.45	57	1.55	59	0.46	60	0.34	61	1.18	62	1.32
63	2.82	64	0.91	65	0.48	69	50.02	70	1.23	71	0.55	73	1.1	74	4.56	75	13.9
76	1.31	77	1.35	78	0.42	79	0.41	80	1.01	81	10.18	82	0.55	83	0.82	84	0.56
85	2.08	86	2.54	87	5.6	88	1.41	89	1.77	90	1.89	91	1.09	92	0.91	93	1.91
94	1.27	95	1.24	96	0.8	97	9.24	98	1.19	99	4.58	100	0.57	101	1.08	102	0.65
103	3.85	104	1.43	105	6.21	106	3.25	107	1.5	108	0.9	109	4.08	110	0.88	111	1.68
112	1.27	113	1.54	114	1.71	115	0.59	116	0.5	117	1.64	118	2.49	119	4.52	120	0.66
121	0.78	122	2.81	123	2.48	124	1	125	6.03	127	11.98	128	8.7	129	0.85	130	1.83
131	15.23	132	1.54	133	6.06	134	1.08	135	1.25	136	2.45	137	10.14	138	2.67	139	1.44
140	1.26	141	1.64	142	0.82	143	3.48	144	3.23	145	8.02	147	19.8	148	1.14	149	1.86
150	1.34	151	5.25	152	2.33	153	1.86	154	0.96	155	5.95	156	23.89	157	7.97	158	1.35
159	4.86	160	0.77	161	1.46	162	1.27	163	11.31	164	1.03	165	2.26	166	0.54	167	3.95
168	2.37	169	6.38	170	1.91	171	12.46	172	3.78	173	1.54	174	4.29	175	13.19	176	2.91
177	1.95	178	0.54	179	1.15	181	33.53	182	2.55	183	1.09	184	1.19	185	4.31	186	3.34
187	26.61	188	4.37	189	2.08	190	1.16	191	2.42	192	0.8	193	2.58	194	2.16	195	2.33
196	0.51	197	0.54	198	1.11	199	3.03	200	5.35	201	4.26	202	3.24	203	2.27	204	1.62
205	19.34	206	19.25	207	6.79	208	1.47	209	2.37	210	0.4	211	1.3	213	32.17	215	12.4
216	1.91	217	3.32	218	6.45	219	10.94	220	2.56	221	6.35	222	3.46	223	4.15	224	8.49
225	12.72	226	7.9	227	1.78	228	0.63	229	1.78	230	1.38	231	3.34	232	0.94	233	2.02
234	1.52	235	3.35	236	3.85	237	11.36	238	4.67	239	5.85	240	5.55	241	8.41	242	1
243	1.88	244	2.44	245	9.35	246	1.48	248	3.68	249	11.54	250	1.88	251	1.02	252	2.15
253	28.05	254	9.1	255	5.76	256	7.08	257	7.44	258	1.17	259	1.47	260	0.31	261	0.46

Dimethyl ether **6**

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T*14-04*-1365

Sample Serial Number: 2012185-YWJ-1-3

Operator: Li

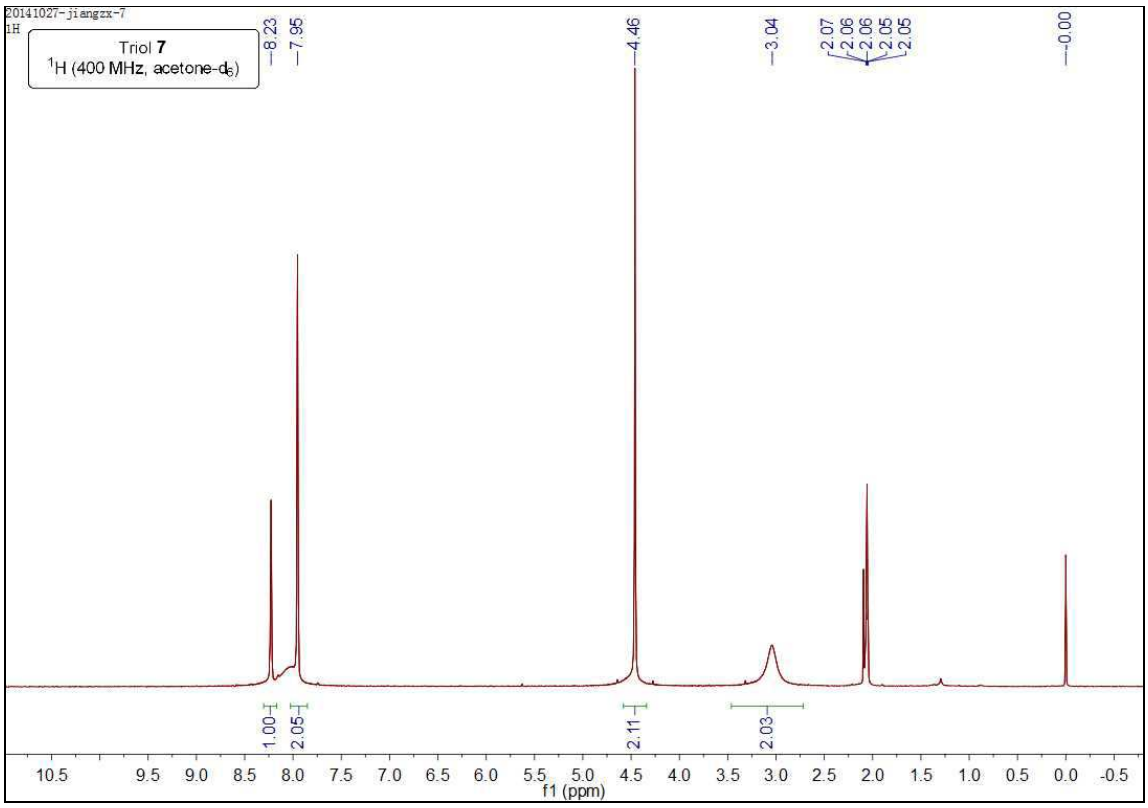
Date: 2014/05/05

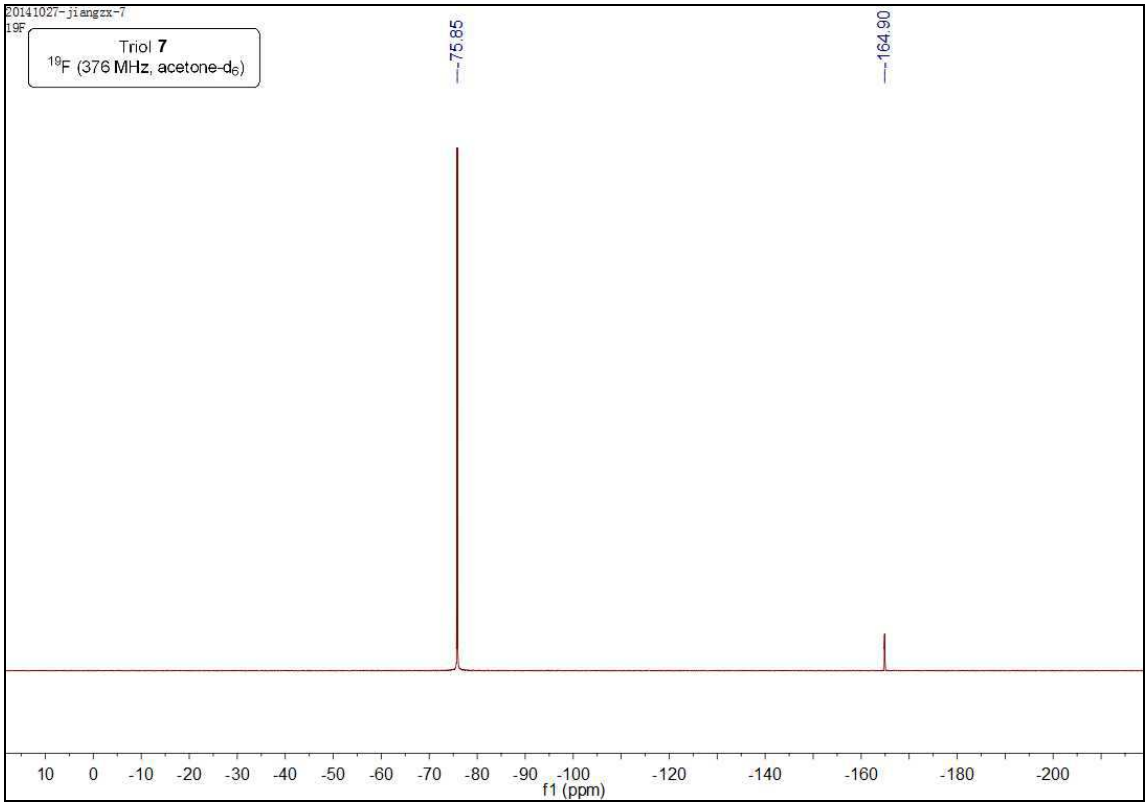
Elemental Composition Report

Single Mass Analysis
Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
398 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 O: 0-2 F: 0-12 I: 0-1

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
563.9458	563.9459	-0.1	-0.2	26.0	2773048.3	C30 H7 O2 F2 I
	563.9457	0.1	0.2	15.0	2773031.5	C22 H8 O2 F7 I
	563.9456	0.2	0.4	4.0	2773017.3	C14 H9 O2 F12 I
	563.9447	1.1	2.0	30.0	2773053.3	C33 H6 O F I
	563.9446	1.2	2.1	19.0	2773037.3	C25 H7 O F6 I
	563.9444	1.4	2.5	8.0	2773021.3	C17 H8 O F11 I


20141027-jiangzx-7
1H
Triol 7
1H (400 MHz, acetone-d6)
8.23
7.95
4.46
3.04
2.07
2.06
2.06
2.05
2.05
0.00
1.00
2.05
2.11
2.03
f1 (ppm)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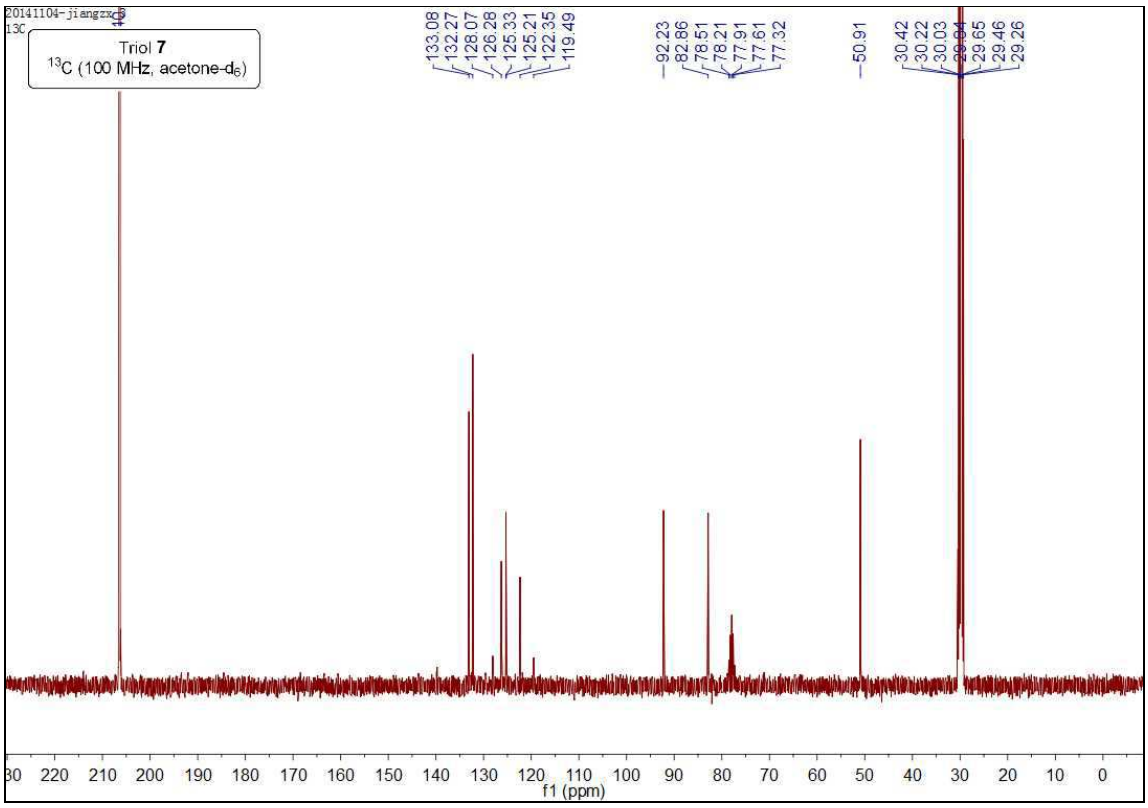
19F
Triol 7
19F (376 MHz, acetone-d6)
-75.85
-164.90
f1 (ppm)



Triol **7**

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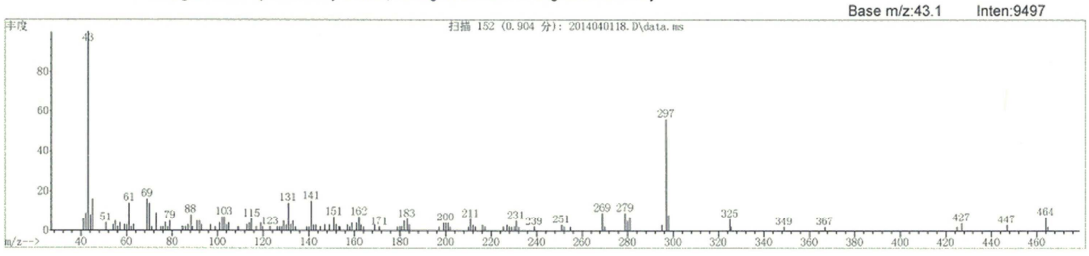
Sample Name : 2012185-YWJ-1-3MW.464
Instrument : Agilent Technologies 5973N
: Shanghai Mass spectrometry Center, Shanghai Institute of Organic Chemistry

Base m/z:43.1 Inten:9497



m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%
41	5.55	42	8.52	43	100	44	8.09	45	15.82	51	4.04	54	2.99	55	5.02	56	2.1
57	4.32	59	2.67	60	2.86	61	14.48	62	2.01	63	2.52	69	15.62	70	14.23	71	1.9
73	8.75	75	2.43	76	2.45	77	4.32	78	2.06	79	4.74	84	2.19	85	1.72	86	1.63
87	2.59	88	7.84	89	1.57	91	5.4	92	5.27	93	2.84	97	2.83	99	1.69	101	3.56
102	7.02	103	7.48	104	2.83	105	4.49	109	1.91	113	2.99	114	3.95	115	6.11	117	1.68
119	4.26	120	1.77	123	1.58	126	1.64	127	1.64	128	2.19	129	4.54	130	3.23	131	14.08
132	3.02	133	4.64	134	1.57	139	1.84	140	1.71	141	15.45	142	3.08	143	2.84	145	2.12
147	2.88	149	2.69	151	6.63	152	2.61	153	2.01	154	2.34	157	3.06	158	1.88	159	4.16
161	3.64	162	7.14	163	2.81	164	2.45	169	3.4	171	1.76	179	1.81	180	2.39	181	2.11
182	4.93	183	5.97	184	3.46	197	2.13	199	3.86	200	3.64	201	4.48	202	1.57	210	1.76
211	5.77	212	2.51	213	2.28	216	3.24	217	1.97	225	1.73	227	3.46	228	2.37	229	2.45
230	1.61	231	4.99	232	2.44	239	1.63	251	2.67	252	1.68	255	1.74	269	8.62	270	1.85
279	8.59	280	4.5	281	7.43	295	2.6	297	56.04	298	8.49	325	5.66	326	1.74	349	1.58
367	2.06	425	1.7	427	4.11	447	3.39	464	7	465	1.67						

8 Peak Value:

43	100	297	56.04	45	15.82	69	15.62	141	15.45	61	14.48	70	14.23	131	14.08

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Triol **7**

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T*14-04*-1366

Sample Serial Number: 2012185-YWJ-1-3a

Operator: Li

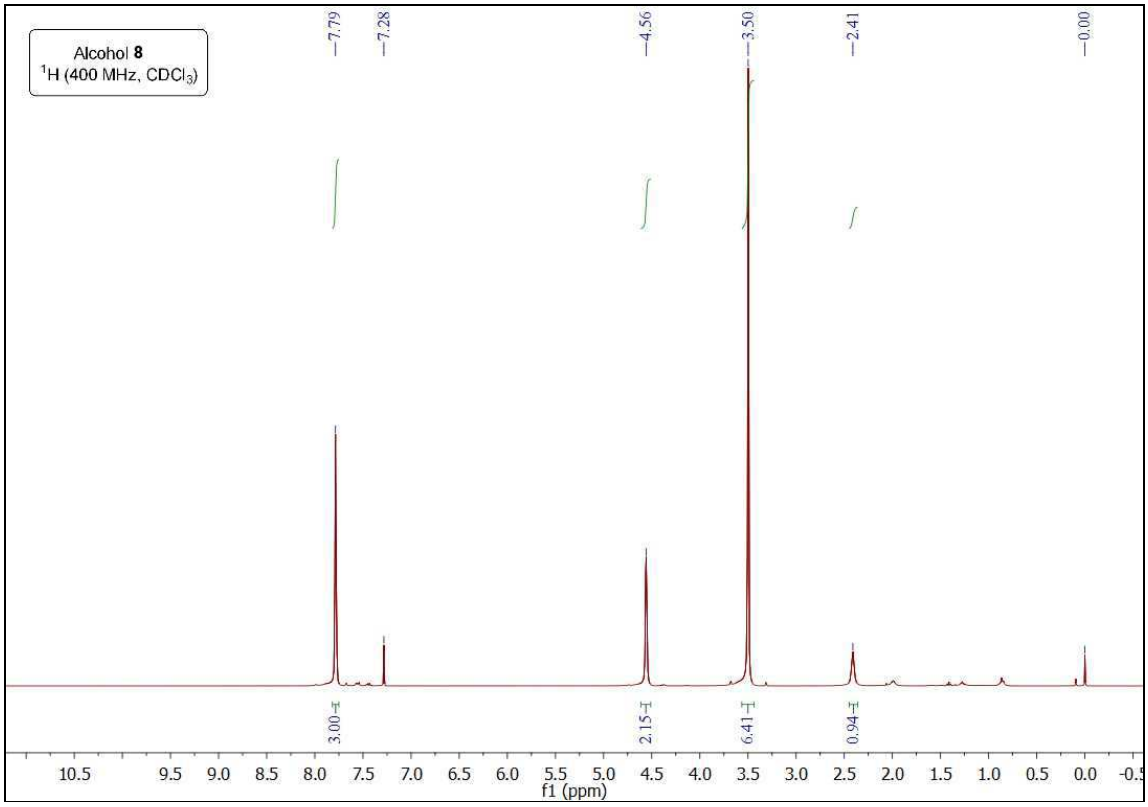
Date: 2014/05/05

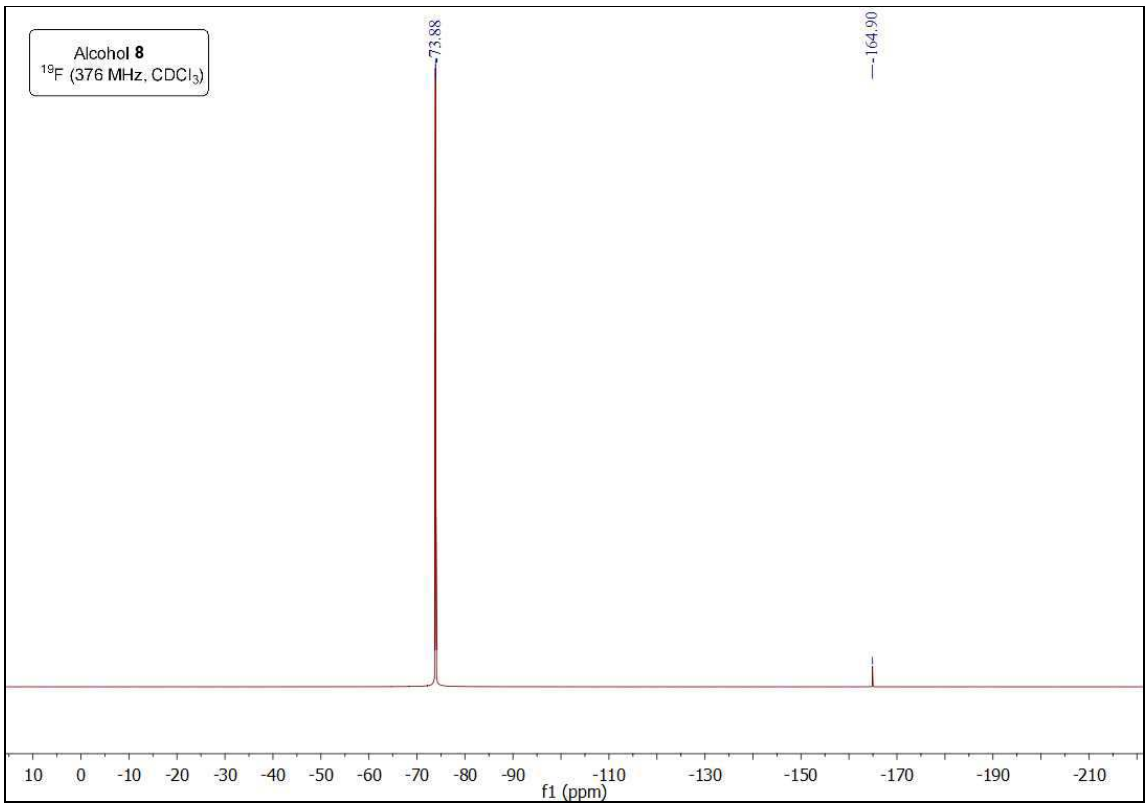
Elemental Composition Report

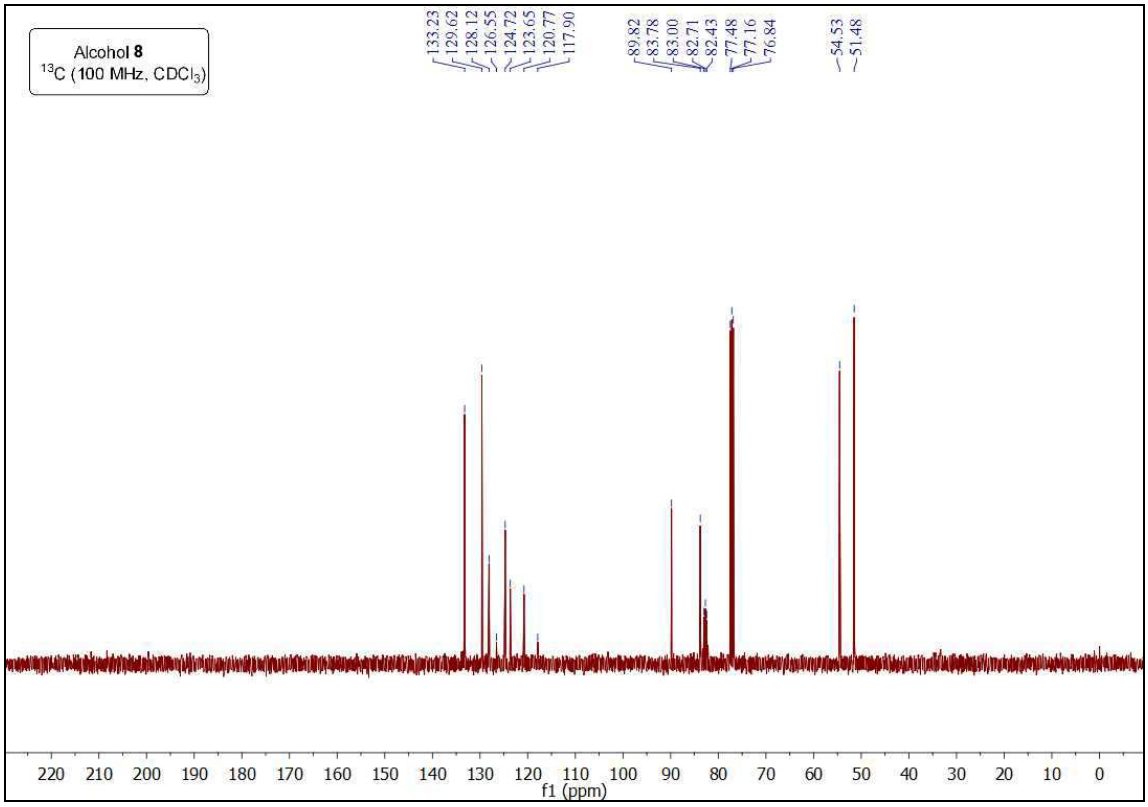
Single Mass Analysis
Tolerance = 2.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off

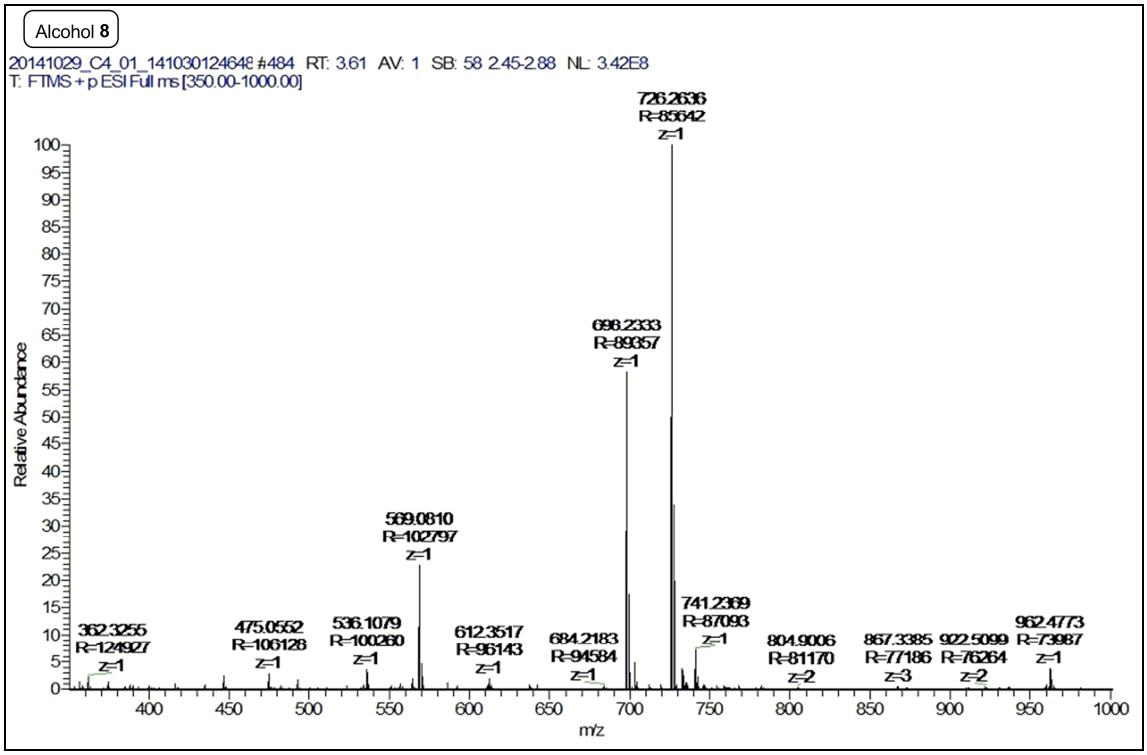
Monoisotopic Mass, Odd and Even Electron Ions
241 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 O: 0-3 F: 0-12

Minimum:				-1.5						
Maximum:		2.0	5.0	50.0						
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula				
464.0286	464.0285	0.1	0.2	28.0	86.8	C31	H6	O3	F2	
	464.0283	0.3	0.6	17.0	27.4	C23	H7	O3	F7	
	464.0282	0.4	0.9	6.0	0.8	C15	H8	O3	F12	
	464.0274	1.2	2.6	32.0	110.0	C34	H5	O2	F	
	464.0272	1.4	3.0	21.0	44.4	C26	H6	O2	F6	
	464.0270	1.6	3.4	10.0	4.6	C18	H7	O2	F11	

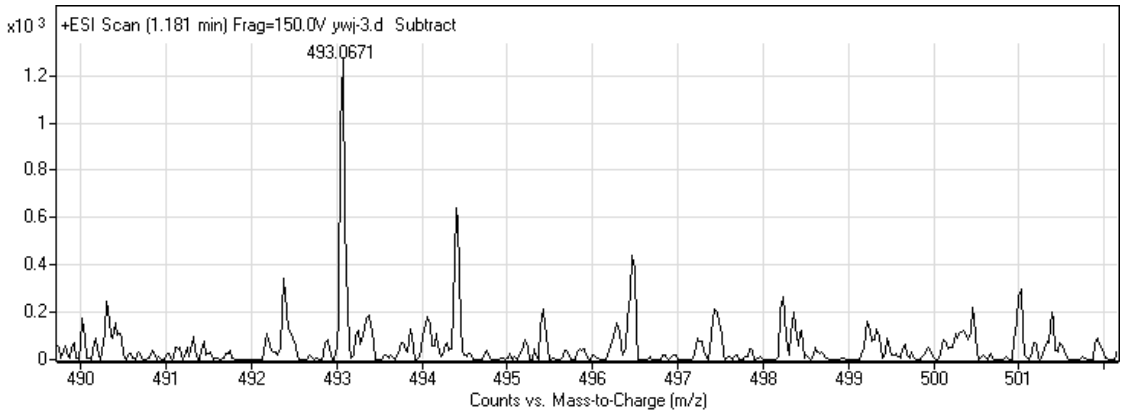


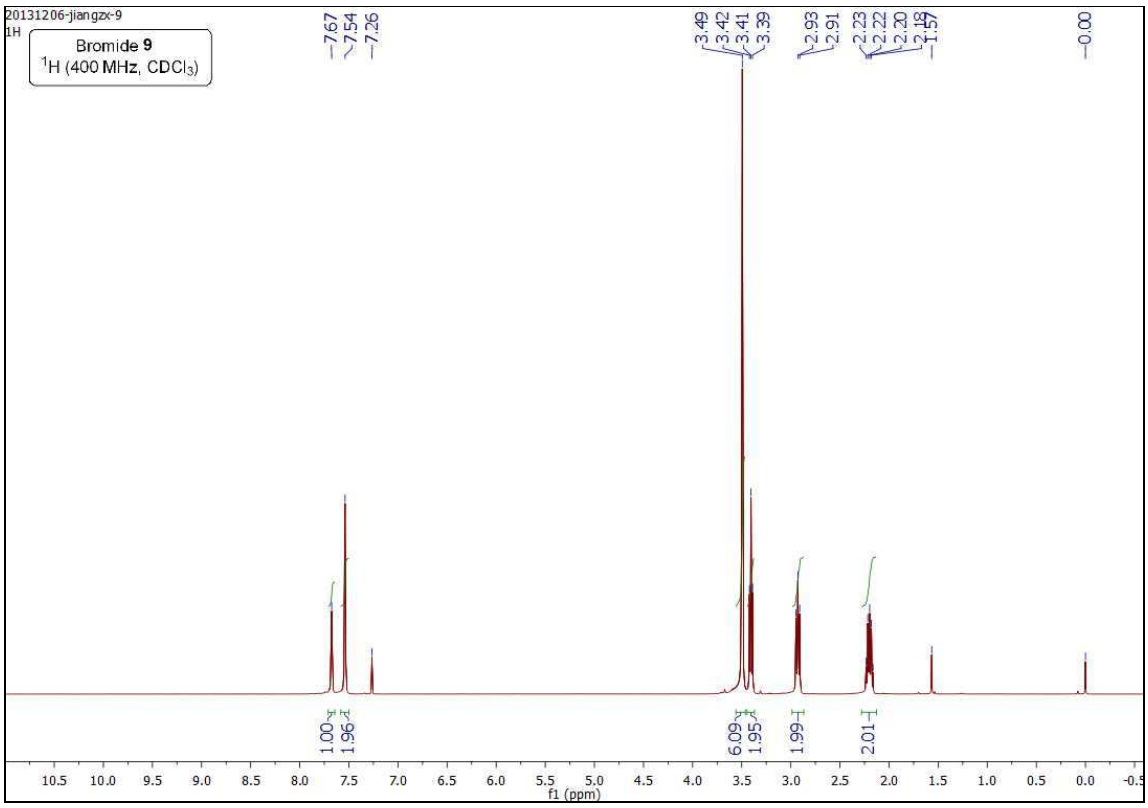

Alcohol 8
19F (376 MHz, CDCl3)
-73.88
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

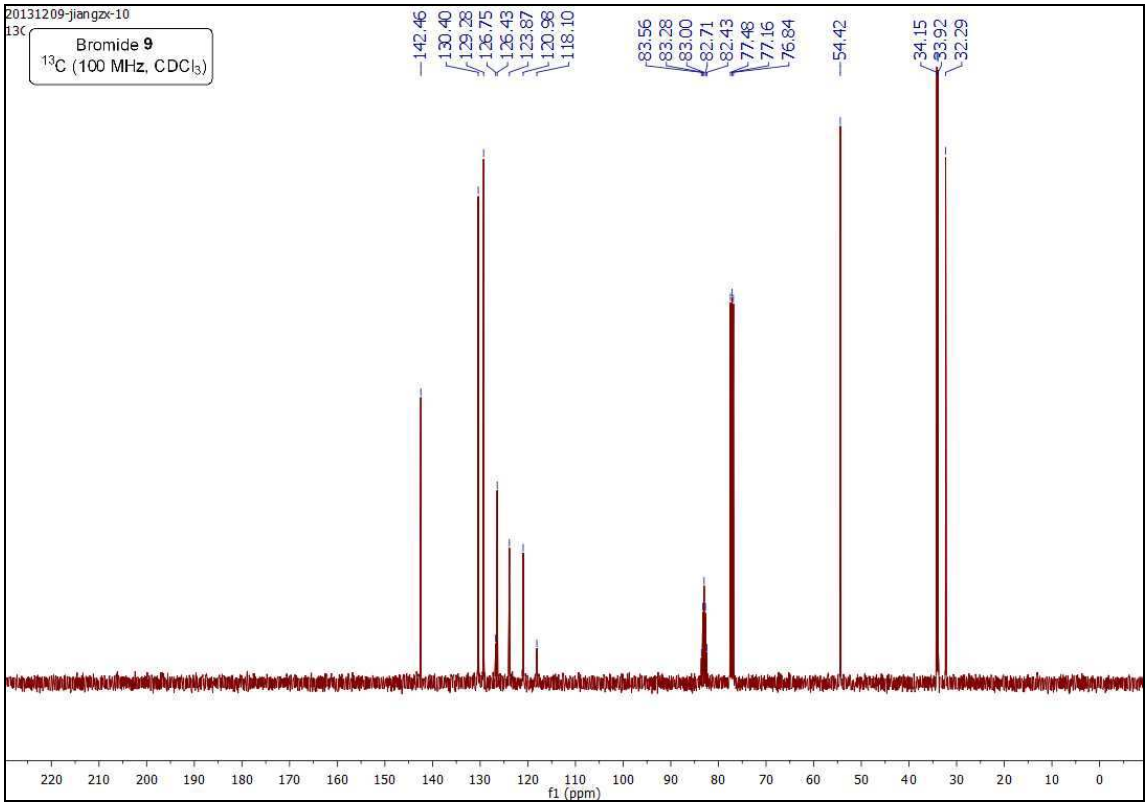

Alcohol 8
13C (100 MHz, CDCl3)
133.23
129.62
128.12
126.55
124.72
123.65
120.77
117.90
89.82
83.78
83.00
82.71
82.43
77.48
77.16
76.84
54.53
51.48
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

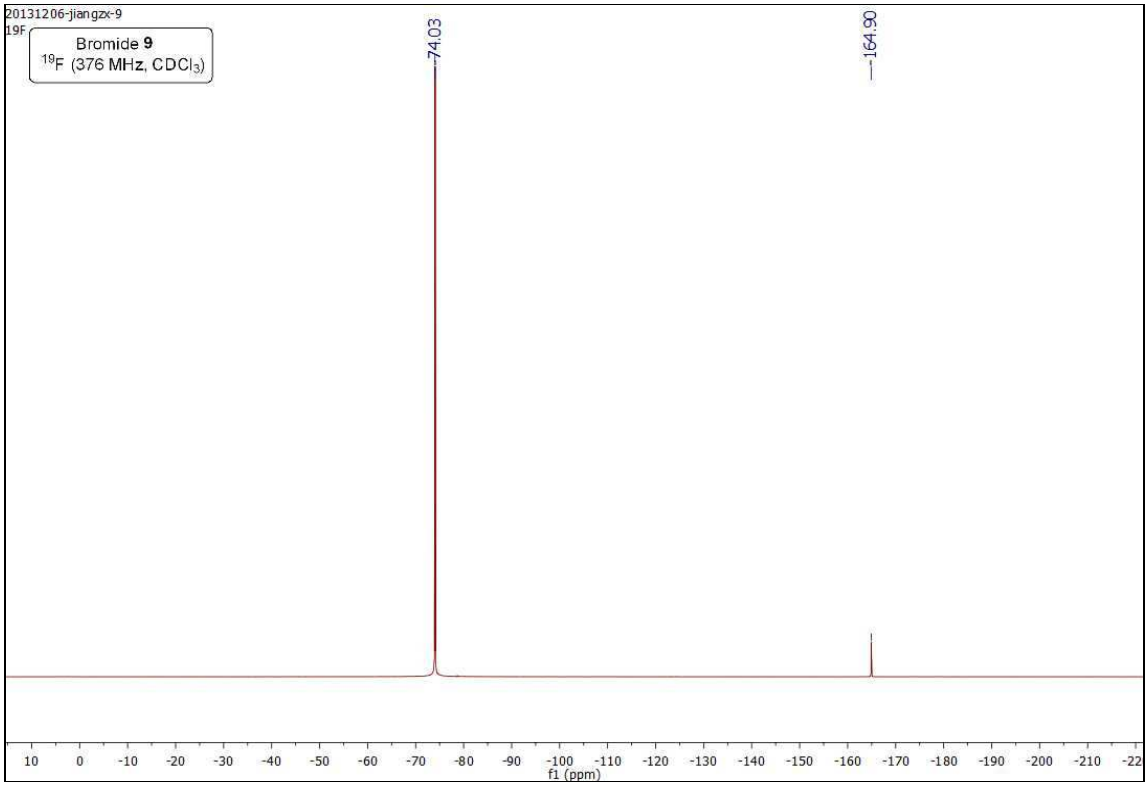


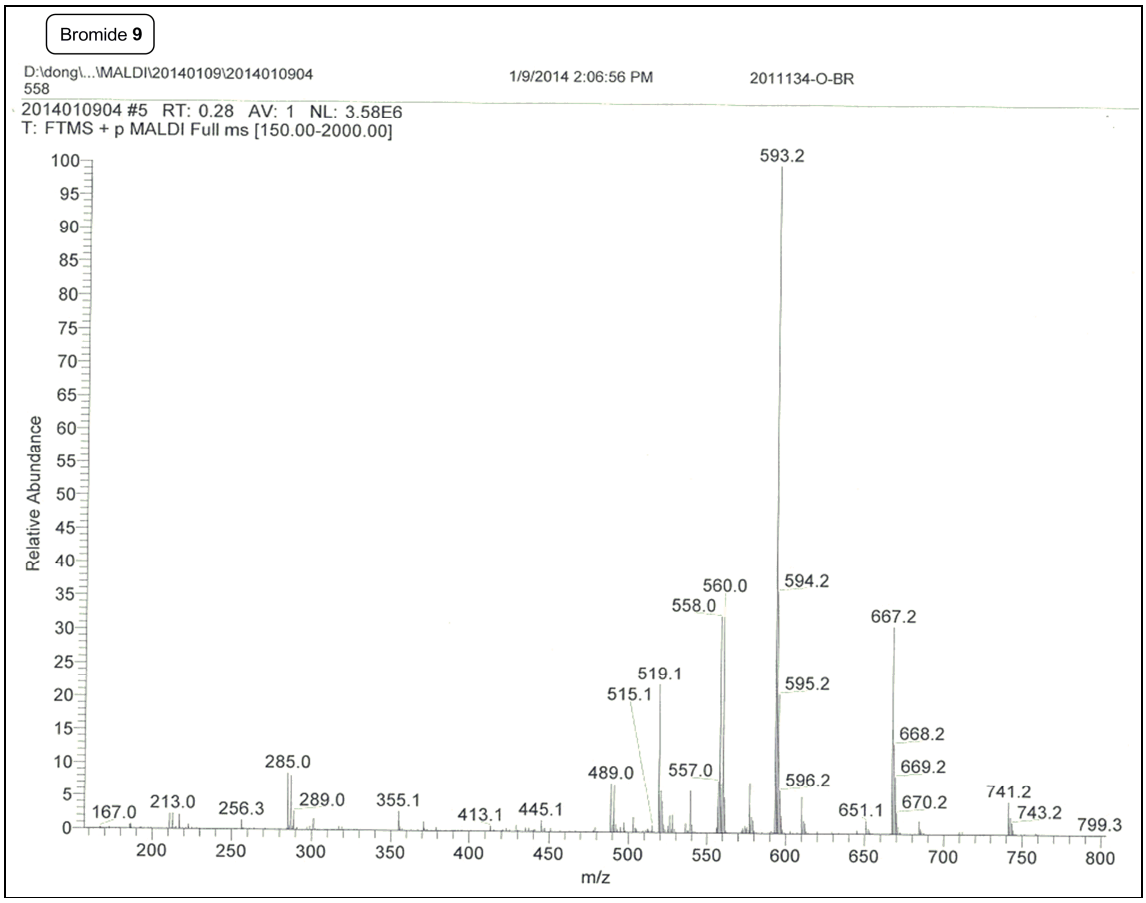
HRMS of alcohol **8**




20131206-jiangzx-9
1H
Bromide 9
1H (400 MHz, CDCl3)
7.67
7.54
7.26
3.49
3.42
3.41
3.39
2.93
2.91
2.23
2.22
2.20
2.18
1.57
0.00
1.00
1.96
6.09
1.95
1.99
2.01
f1 (ppm)


20131209-jiangzx-10
13C
Bromide 9
13C (100 MHz, CDCl3)
142.46
130.40
129.28
126.75
126.43
123.87
120.98
118.10
83.56
83.28
83.00
82.71
82.43
77.48
77.16
76.84
54.42
34.15
33.92
32.29
f1 (ppm)


20131206-jiangzx-9
19F
Bromide 9
19F (376 MHz, CDCl3)
-74.03
-164.90
f1 (ppm)


Bromide 9
D:\dong\...\MALDI\20140109\2014010904
558
1/9/2014 2:06:56 PM
2011134-O-BR
2014010904 #5 RT: 0.28 AV: 1 NL: 3.58E6
T: FTMS + p MALDI Full ms [150.00-2000.00]
Relative Abundance
m/z
593.2
594.2
560.0
558.0
667.2
519.1
515.1
595.2
668.2
669.2
489.0
557.0
596.2
741.2
743.2
670.2
651.1
285.0
289.0
355.1
413.1
445.1
167.0
213.0
256.3
799.3

Bromide **9**

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: Thermo Fisher Scientific LTQ FT Ultra

Card Serial Number : 2014010904

Sample Serial Number: 2011134-O-BR

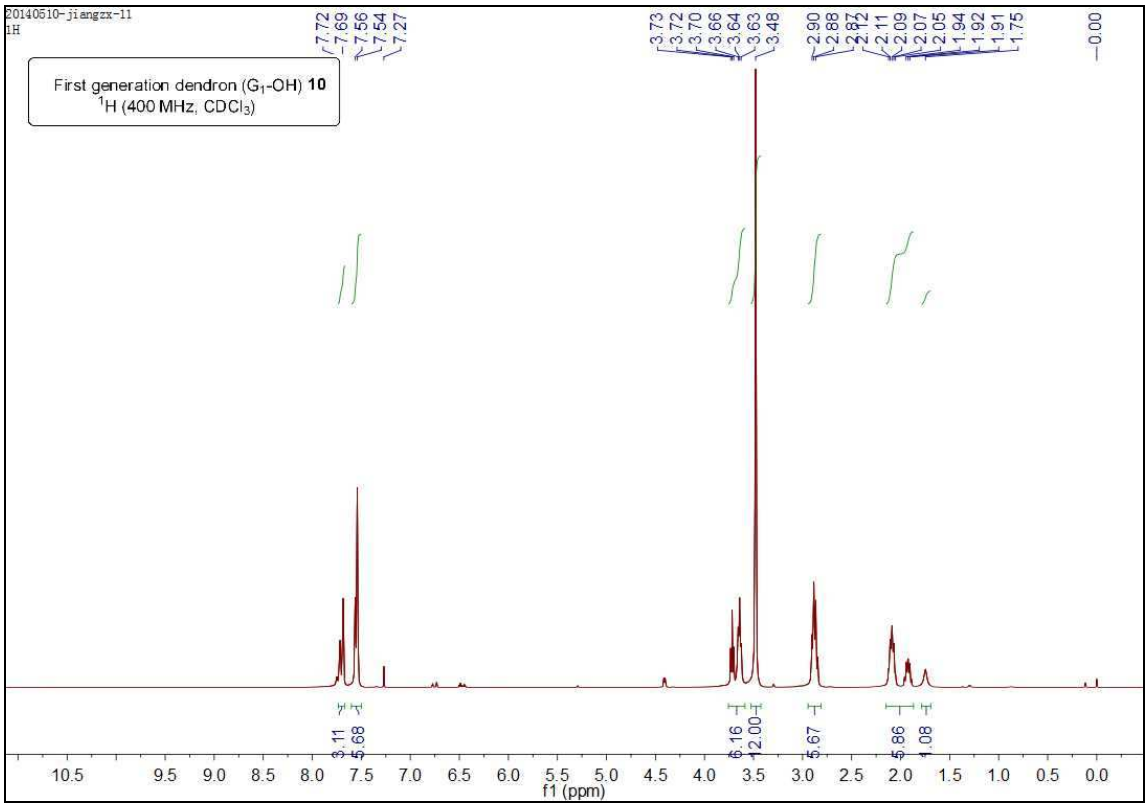
Operator : Dong Date: 2014/01/09

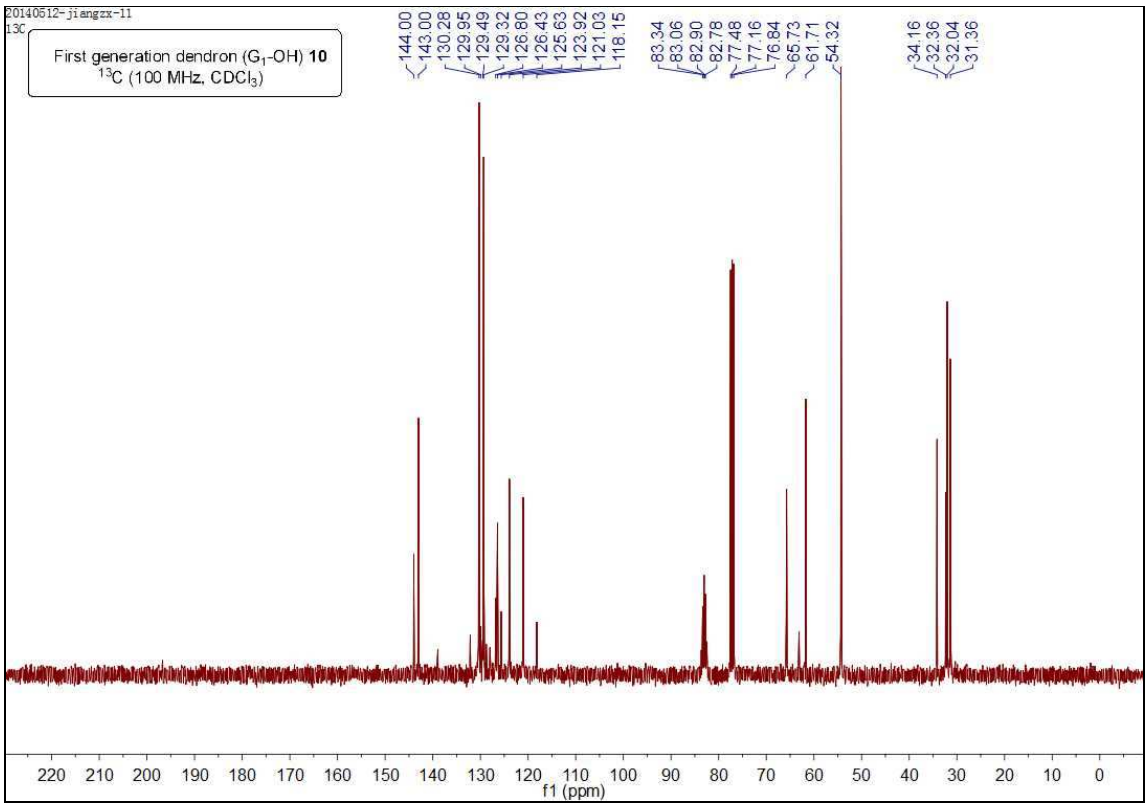
Operation Mode: MALDI

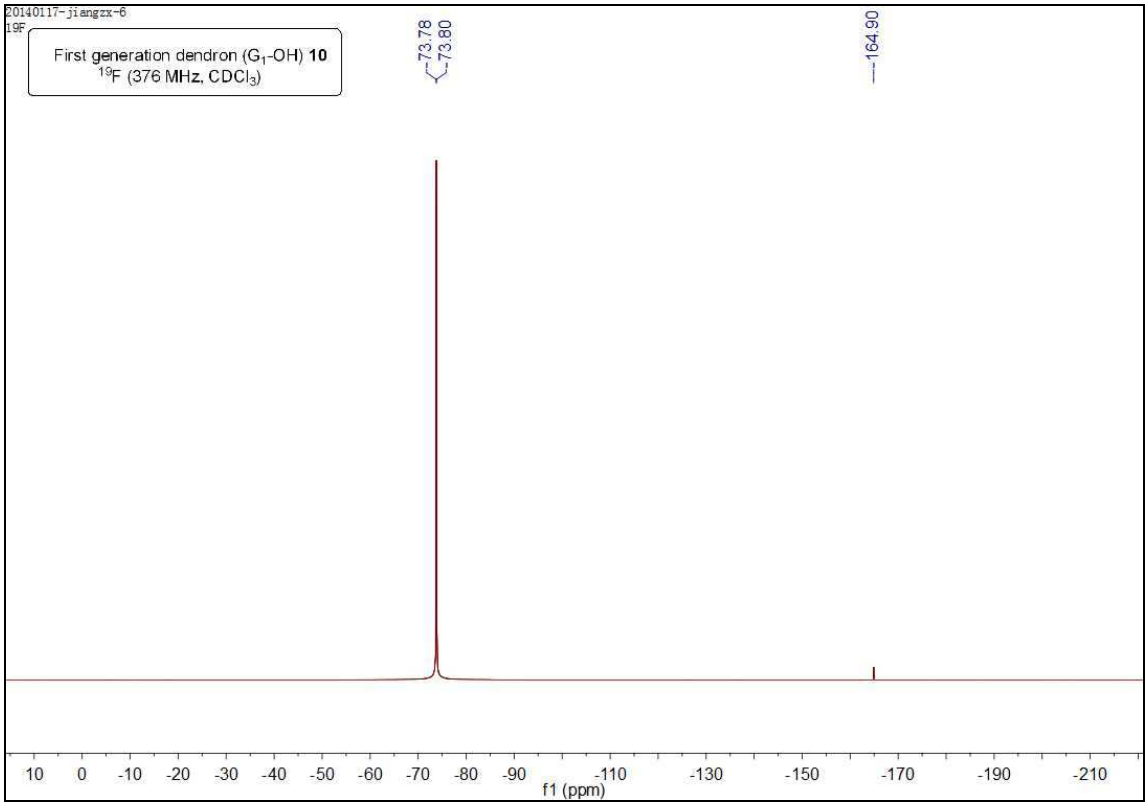
Elemental composition search on mass 558.0061

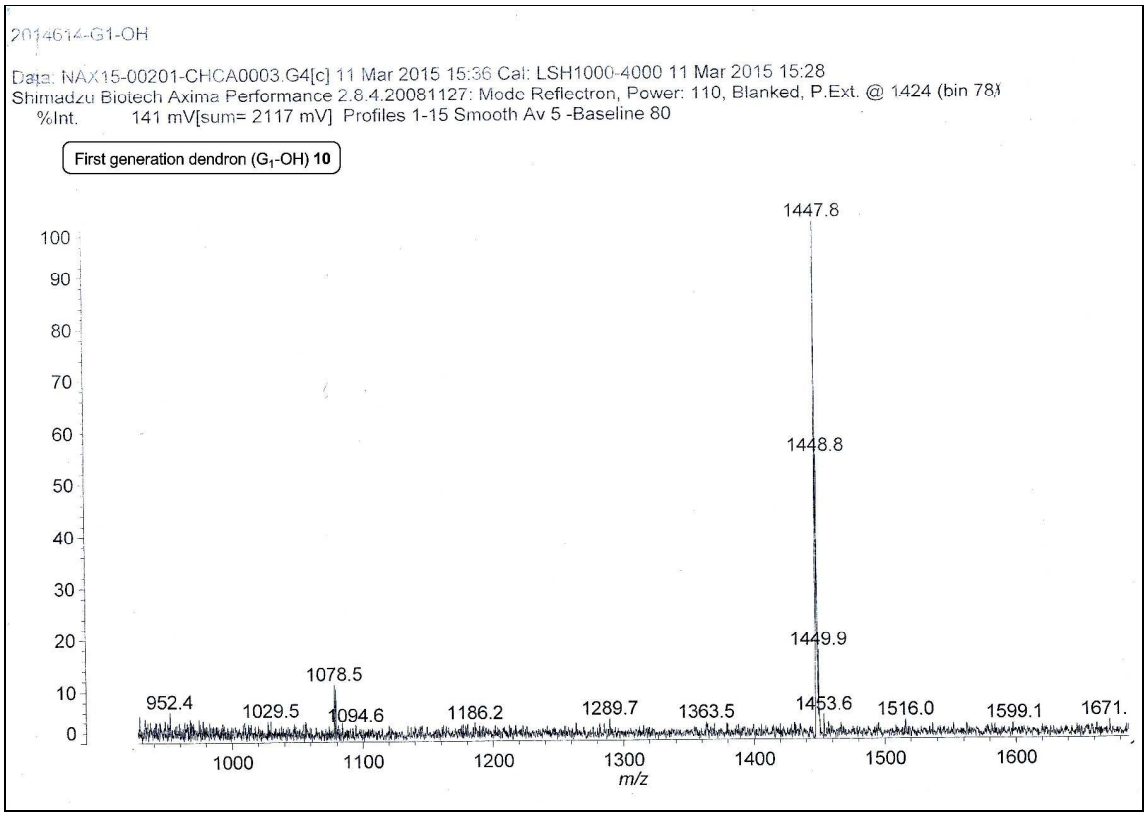
m/z= 553.0061-563.0061

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
558.0060	558.0058	0.39	4.0	$C_{17}H_{15}O_2BrF_{12}$
	558.0070	-1.66	0.0	$C_{14}H_{16}O_3BrF_{13}$
	558.0047	2.44	8.0	$C_{20}H_{14}OBrF_{11}$
	558.0083	-3.99	7.0	$C_{19}H_{16}O_4BrF_9$
	558.0034	4.77	1.0	$C_{15}H_{14}BrF_{15}$

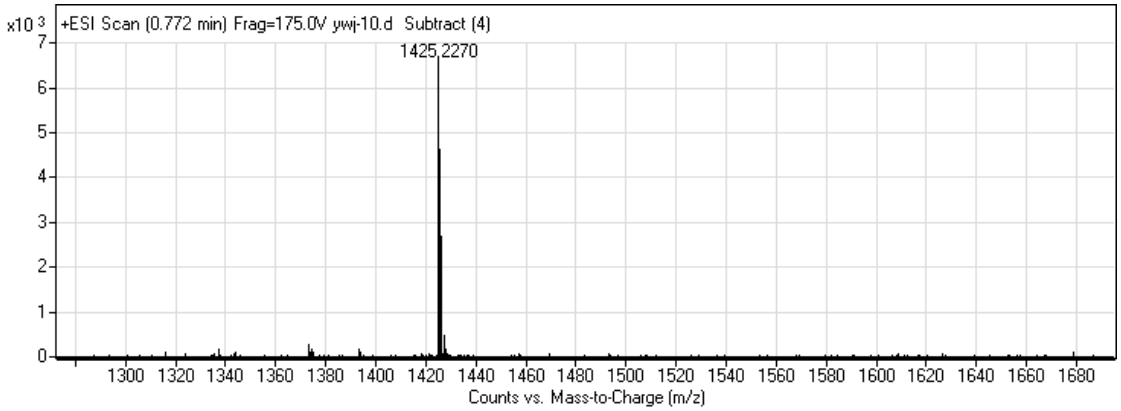


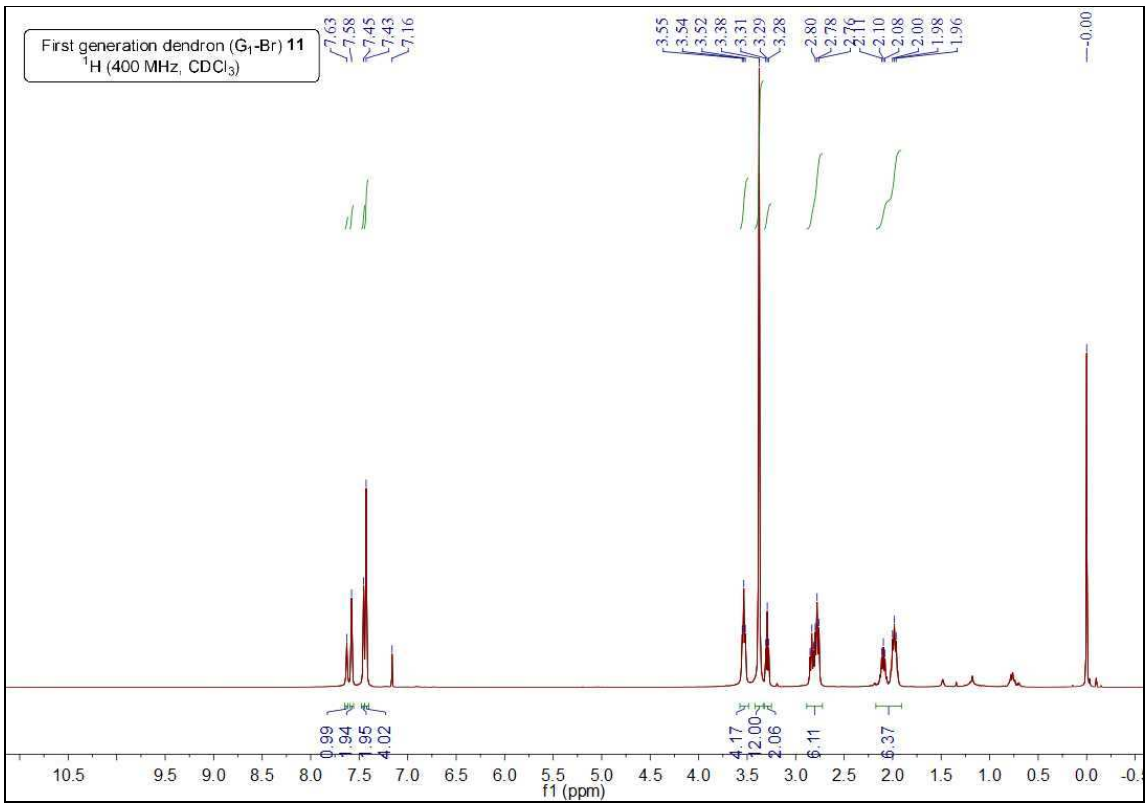

20140512-jiangzx-11
13C
First generation dendron (G_1-OH) **10**
^{13}C (100 MHz, $CDCl_3$)
144.00
143.00
130.28
129.95
129.49
129.32
126.80
126.43
125.63
123.92
121.03
118.15
83.34
83.06
82.90
82.78
77.48
77.16
76.84
65.73
61.71
54.32
34.16
32.36
32.04
31.36
f1 (ppm)

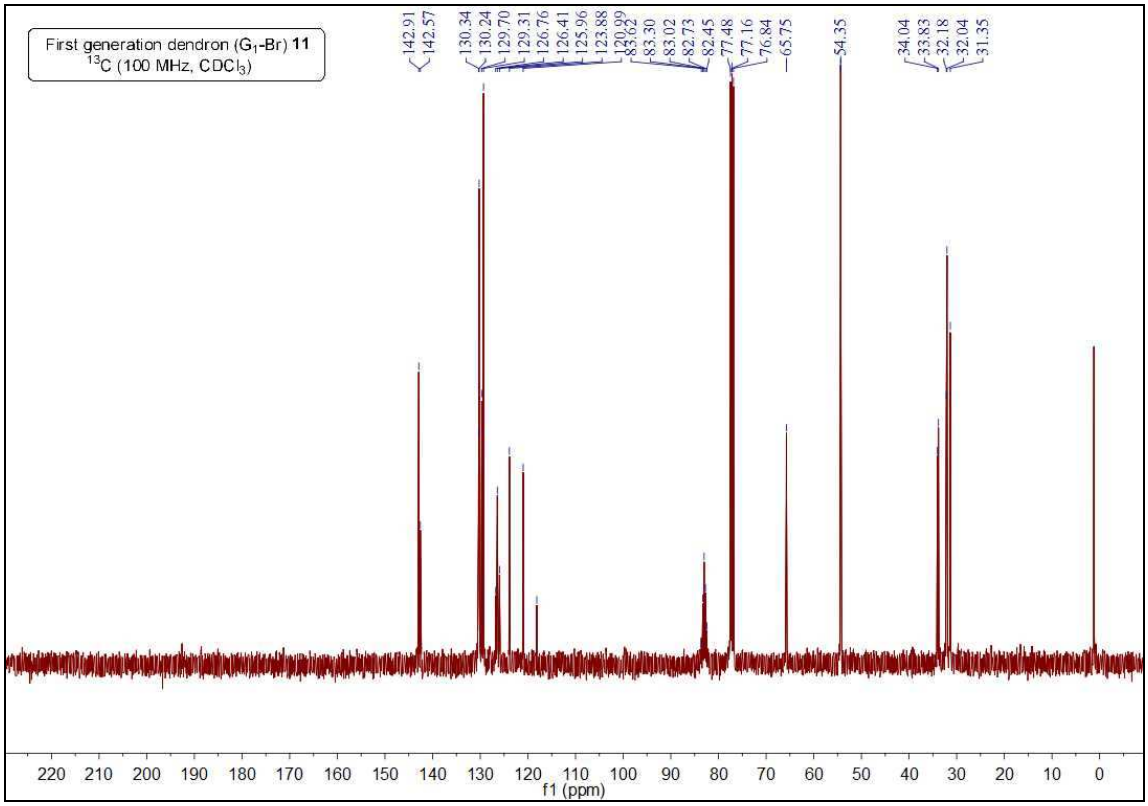

20140117-jiangzx-6
19F
First generation dendron (G_1-OH) **10**
^{19}F (376 MHz, $CDCl_3$)
-73.78
-73.80
-164.90
f1 (ppm)

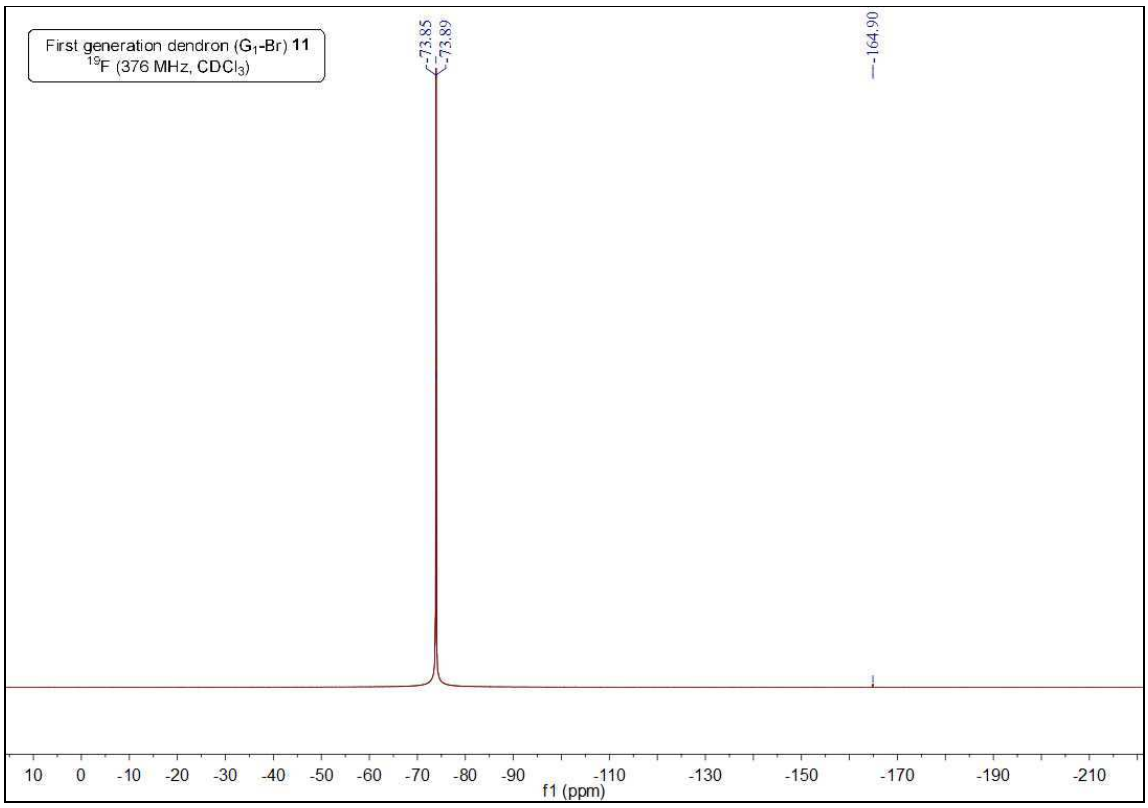


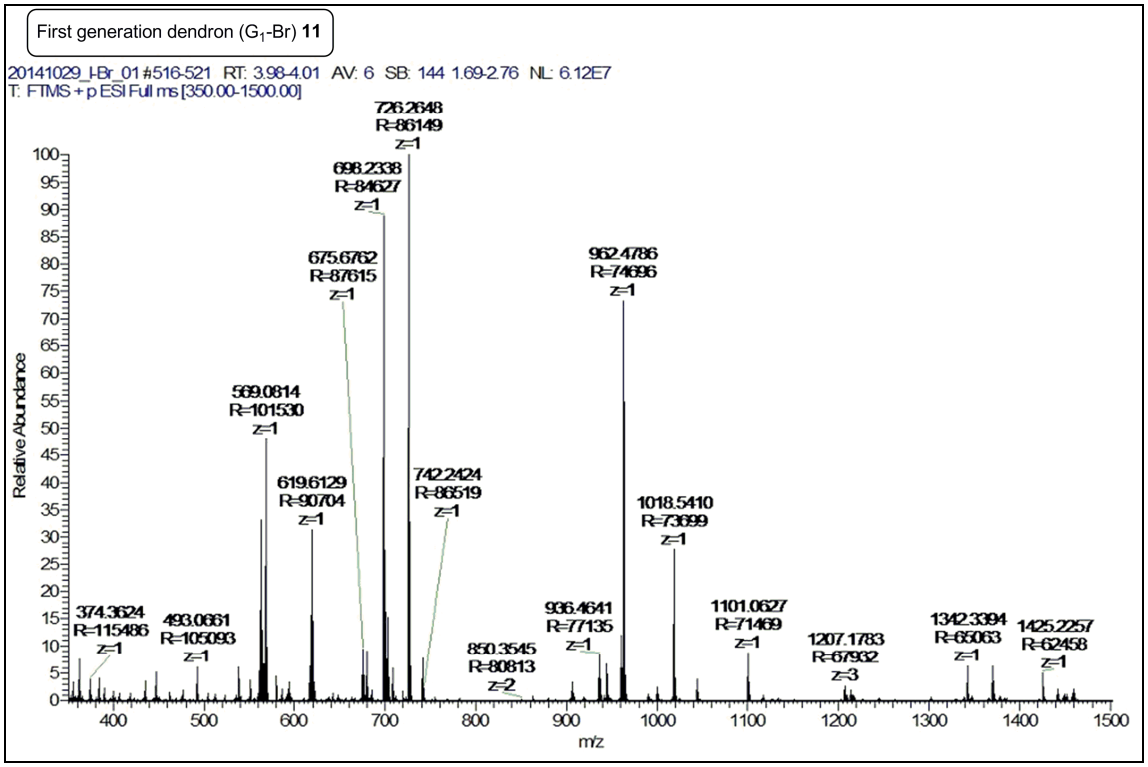
HRMS of first generation dendron (G_1-OH) **10**

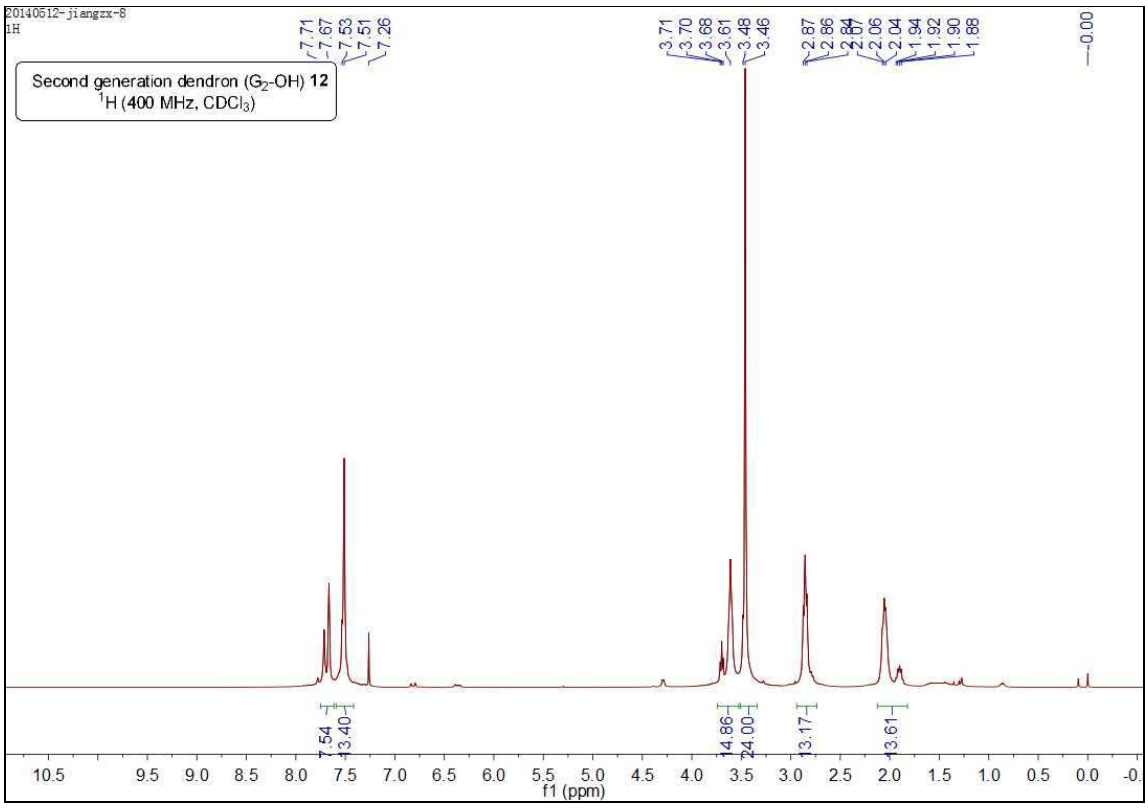


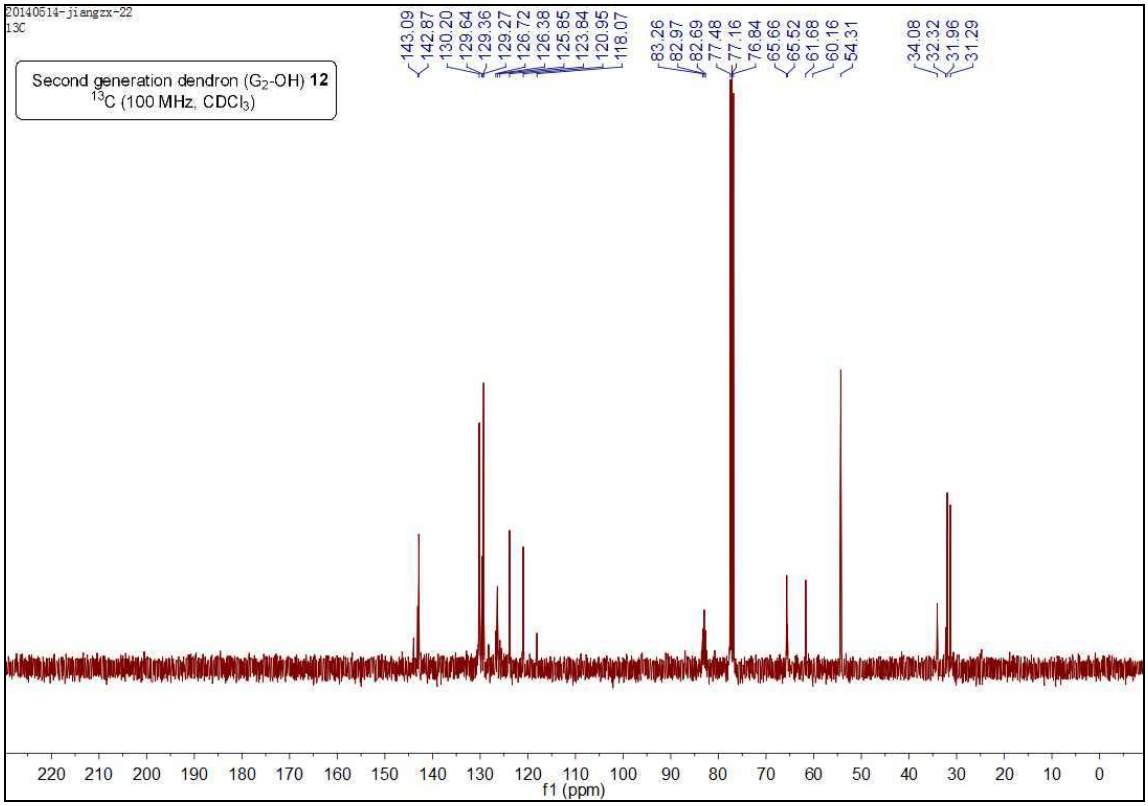

First generation dendron (G_1-Br) **11**
^{1}H (400 MHz, $CDCl_3$)
7.63
7.58
7.45
7.43
7.16
3.55
3.54
3.52
3.38
3.31
3.29
3.28
2.80
2.78
2.76
2.10
2.08
2.00
1.98
1.96
0.00
0.99
1.94
1.95
4.02
4.17
12.00
2.06
6.11
6.37
f1 (ppm)

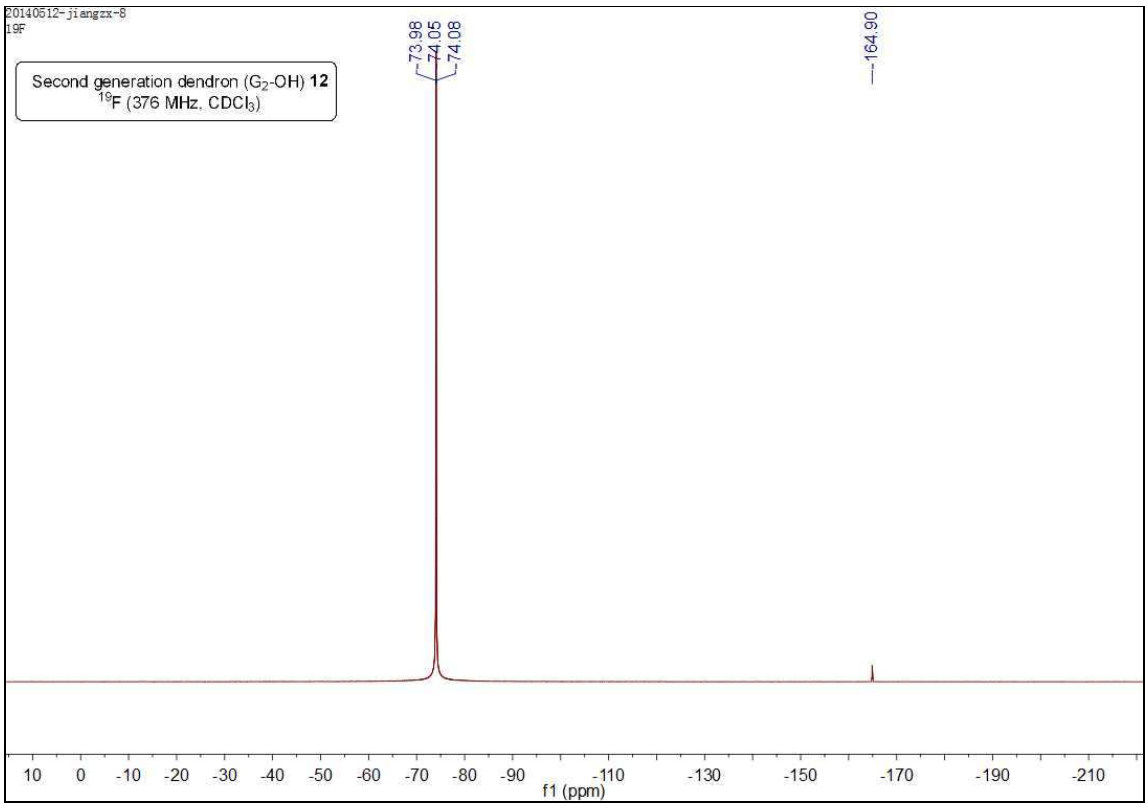

First generation dendron (G_1-Br) **11**
^{13}C (100 MHz, $CDCl_3$)
142.91
142.57
130.34
130.24
129.70
129.31
126.76
126.41
125.96
123.88
120.99
83.62
83.30
83.02
82.73
82.45
77.48
77.16
76.84
65.75
54.35
34.04
33.83
32.18
32.04
31.35
f1 (ppm)

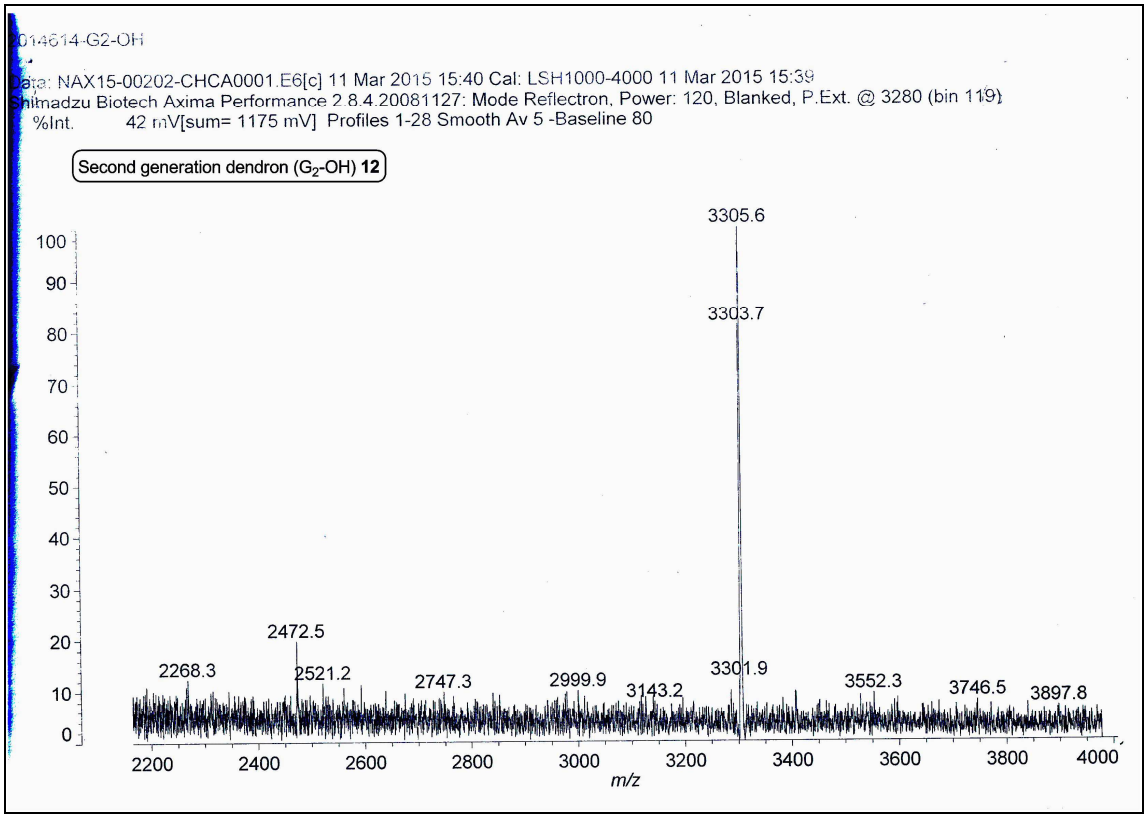

First generation dendron (G_1-Br) **11**
^{19}F (376 MHz, $CDCl_3$)
-73.85
-73.89
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

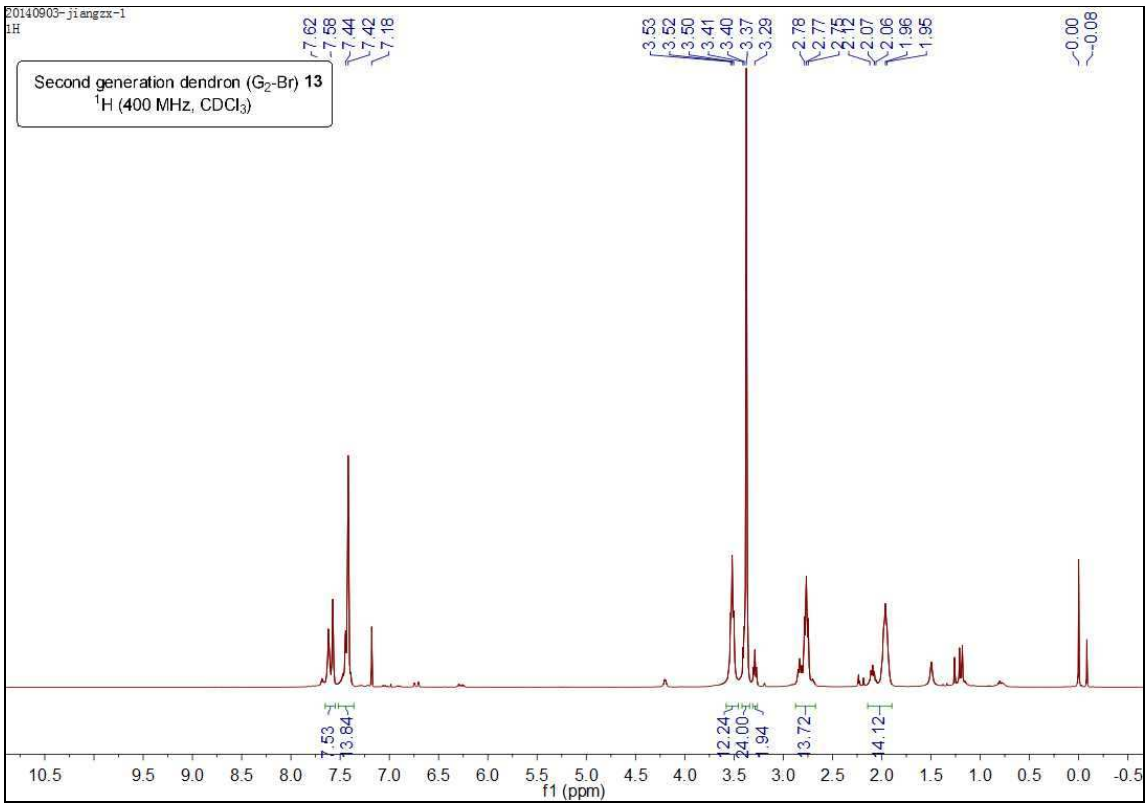

First generation dendron (G_1-Br) **11**
20141029_I-Br_01 #516-521 RT: 3.98-4.01 AV: 6 SB: 144 1.69-2.76 NL: 6.12E7
T: FTMS + p ESI Full ms [350.00-1500.00]
726.2648 R=86149 z=1
698.2338 R=84627 z=1
675.6762 R=87615 z=1
962.4786 R=74696 z=1
569.0814 R=101530 z=1
619.6129 R=90704 z=1
742.2424 R=86519 z=1
1018.5410 R=73699 z=1
374.3624 R=115486 z=1
493.0661 R=105093 z=1
850.3545 R=80813 z=2
936.4641 R=77135 z=1
1101.0627 R=71469 z=1
1207.1783 R=67932 z=3
1342.3394 R=65063 z=1
1425.2257 R=62458 z=1
Relative Abundance
m/z

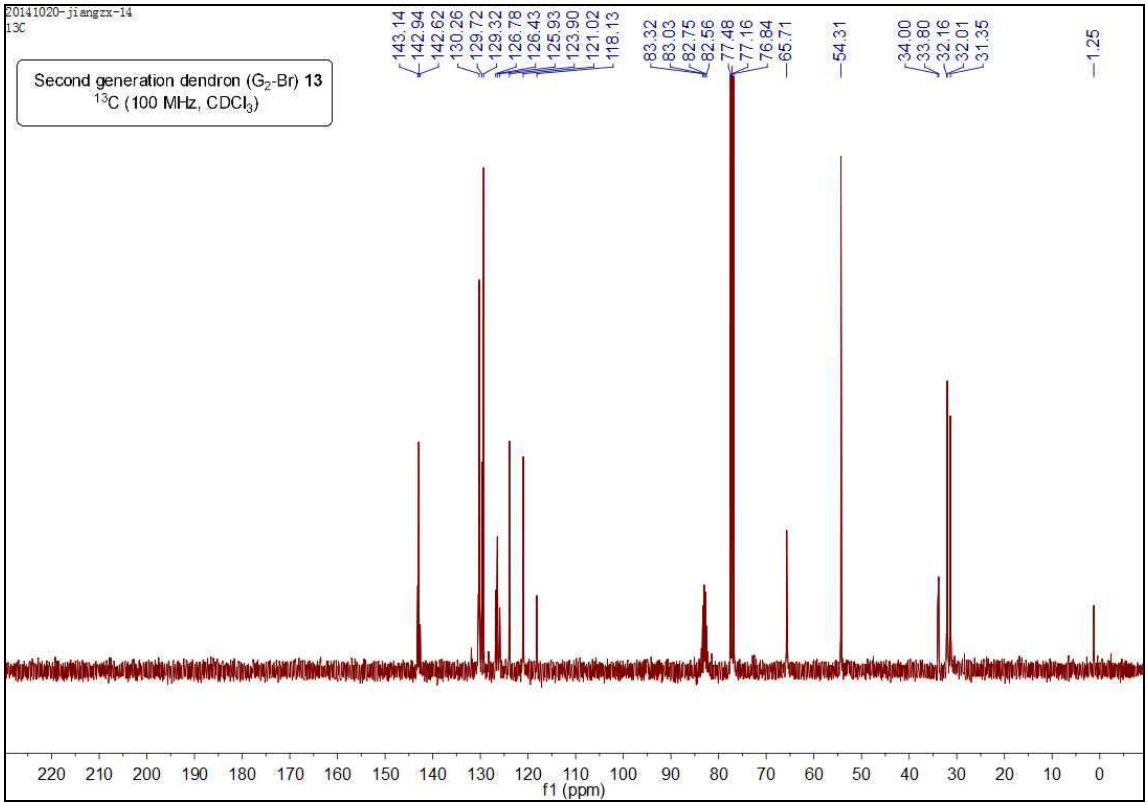

20140512-jiangzx-8
1H
Second generation dendron (G2-OH) 12
1H (400 MHz, CDCl3)
7.71
7.67
7.53
7.51
7.26
3.71
3.70
3.68
3.61
3.48
3.46
2.87
2.86
2.84
2.07
2.06
2.04
1.94
1.92
1.90
1.88
0.00
7.54
13.40
14.86
24.00
13.17
13.61
f1 (ppm)

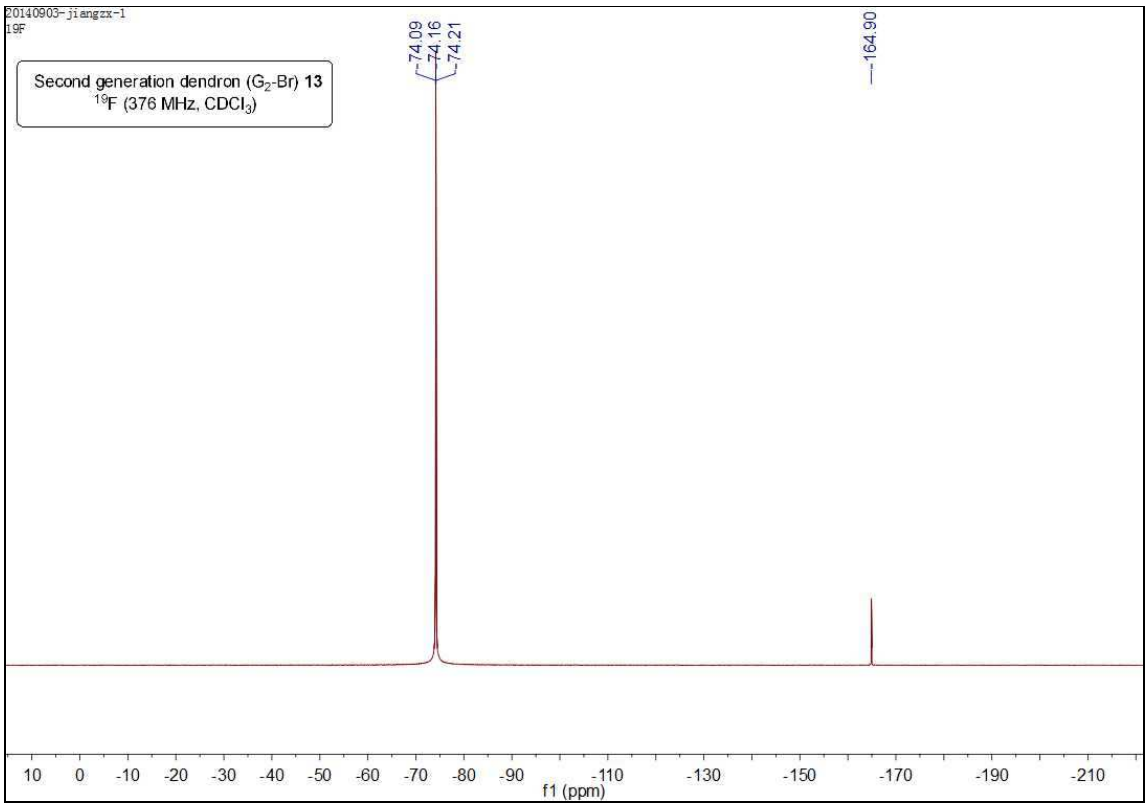

20140514-jiangzx-22
13C
Second generation dendron (G2-OH) 12
13C (100 MHz, CDCl3)
143.09
142.87
130.20
129.64
129.36
129.27
126.72
126.38
125.85
123.84
120.95
118.07
83.26
82.97
82.69
77.48
77.16
76.84
65.66
65.52
61.68
60.16
54.31
34.08
32.32
31.96
31.29
f1 (ppm)

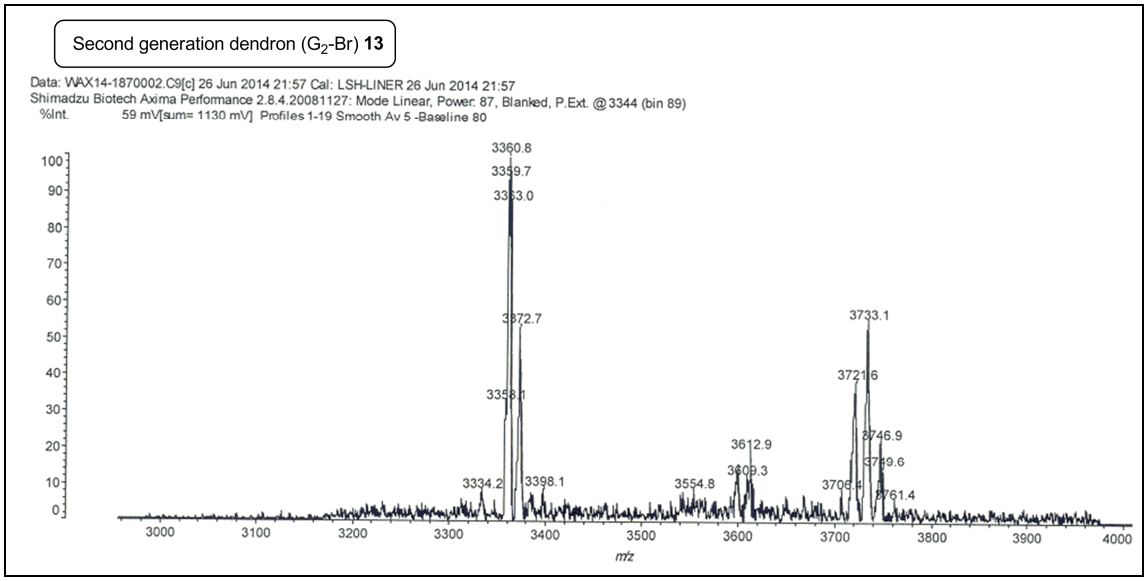

20140512-jiangzx-8
19F
Second generation dendron (G2-OH) 12
19F (376 MHz, CDCl3)
-73.98
-74.05
-74.08
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

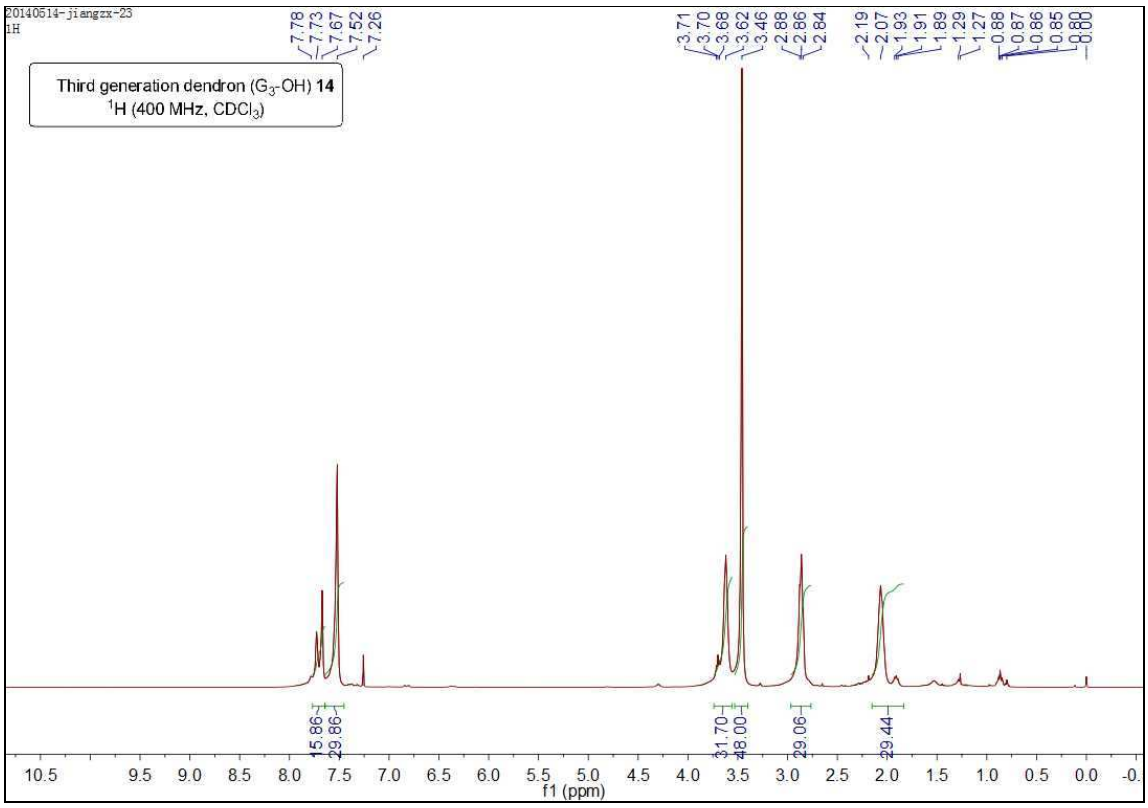

20140614-G2-OH
Data: NAX15-00202-CHCA0001.E6[c] 11 Mar 2015 15:40 Cal: LSH1000-4000 11 Mar 2015 15:39
Shimadzu Biotech Axima Performance 2.8.4.20081127: Mode Reflectron, Power: 120, Blanked, P.Ext. @ 3280 (bin 119)
%Int. 42 mV[sum= 1175 mV] Profiles 1-28 Smooth Av 5 -Baseline 80
Second generation dendron (G2-OH) 12
3305.6
3303.7
2472.5
2268.3
2521.2
2747.3
2999.9
3143.2
3301.9
3552.3
3746.5
3897.8
100 90 80 70 60 50 40 30 20 10 0
2200 2400 2600 2800 3000 3200 3400 3600 3800 4000
m/z

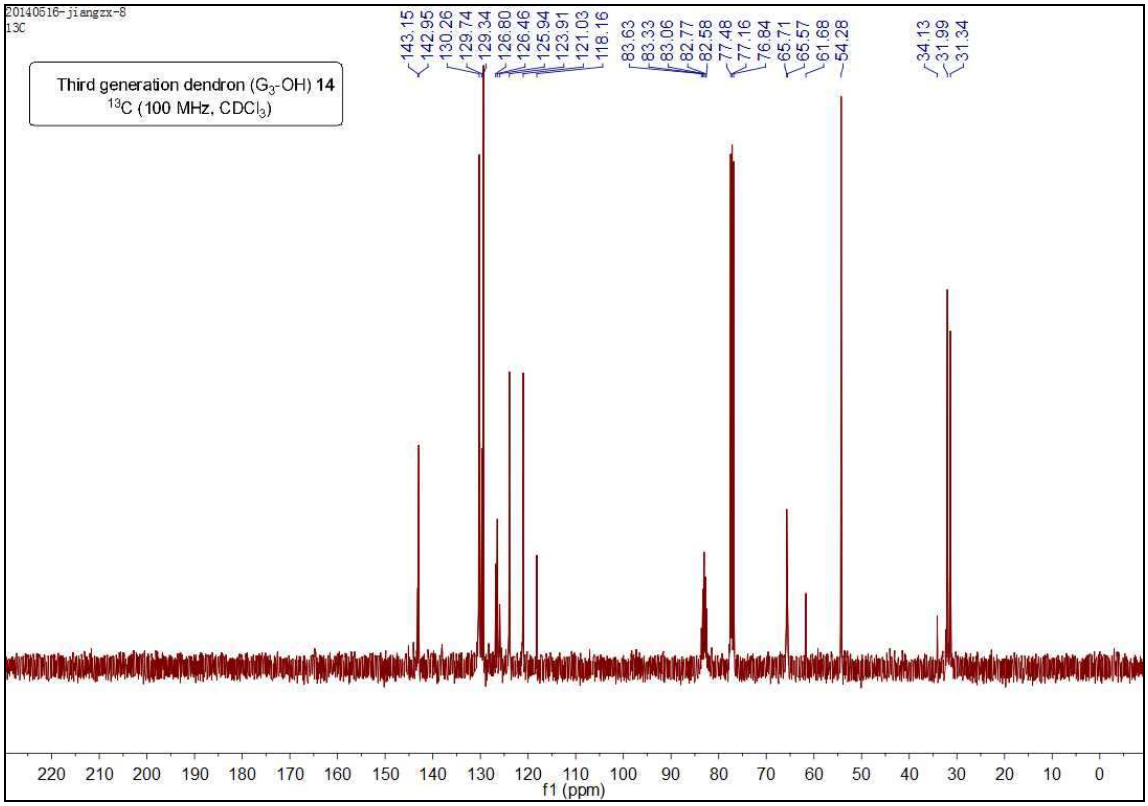

20140903-jiangzx-1
1H
Second generation dendron (G2-Br) 13
1H (400 MHz, CDCl3)
7.62
7.58
7.44
7.42
7.18
3.53
3.52
3.50
3.41
3.40
3.37
3.29
2.78
2.77
2.75
2.12
2.07
2.06
1.96
1.95
0.00
-0.08
7.53
13.84
12.24
24.00
1.94
13.72
14.12
f1 (ppm)

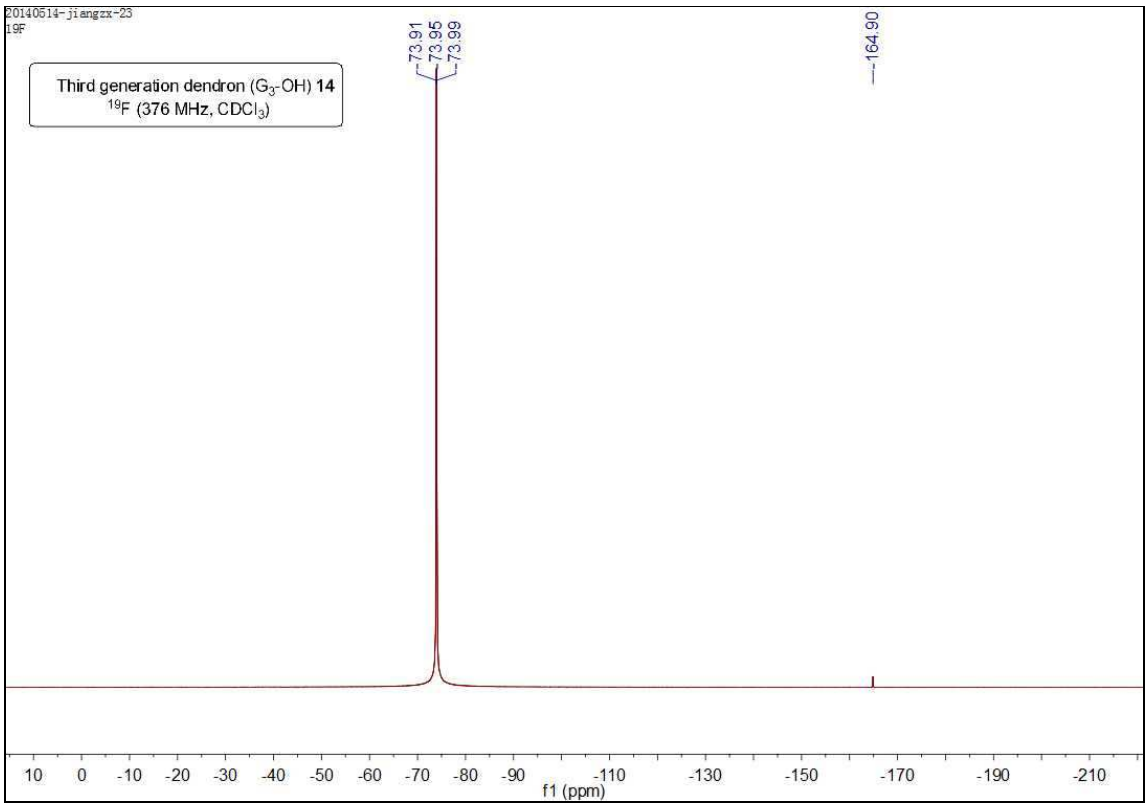

20141020-jiangzx-14
13C
Second generation dendron (G2-Br) 13
13C (100 MHz, CDCl3)
143.14
142.94
142.62
130.26
129.72
129.32
126.78
126.43
125.93
123.90
121.02
118.13
83.32
83.03
82.75
82.56
77.48
77.16
76.84
65.71
54.31
34.00
33.80
32.16
32.01
31.35
1.25
f1 (ppm)

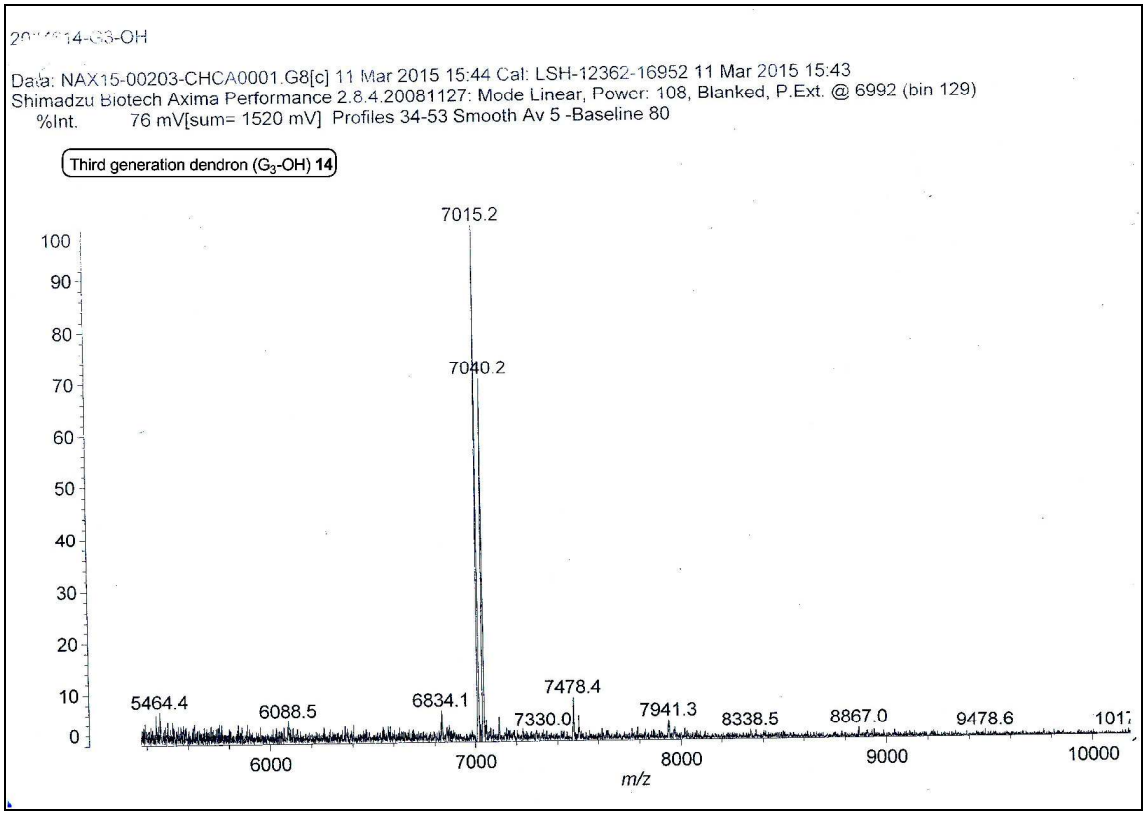

20140903-jiangzx-1
19F
Second generation dendron (G_2-Br) **13**
^{19}F (376 MHz, $CDCl_3$)
-74.09
-74.16
-74.21
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

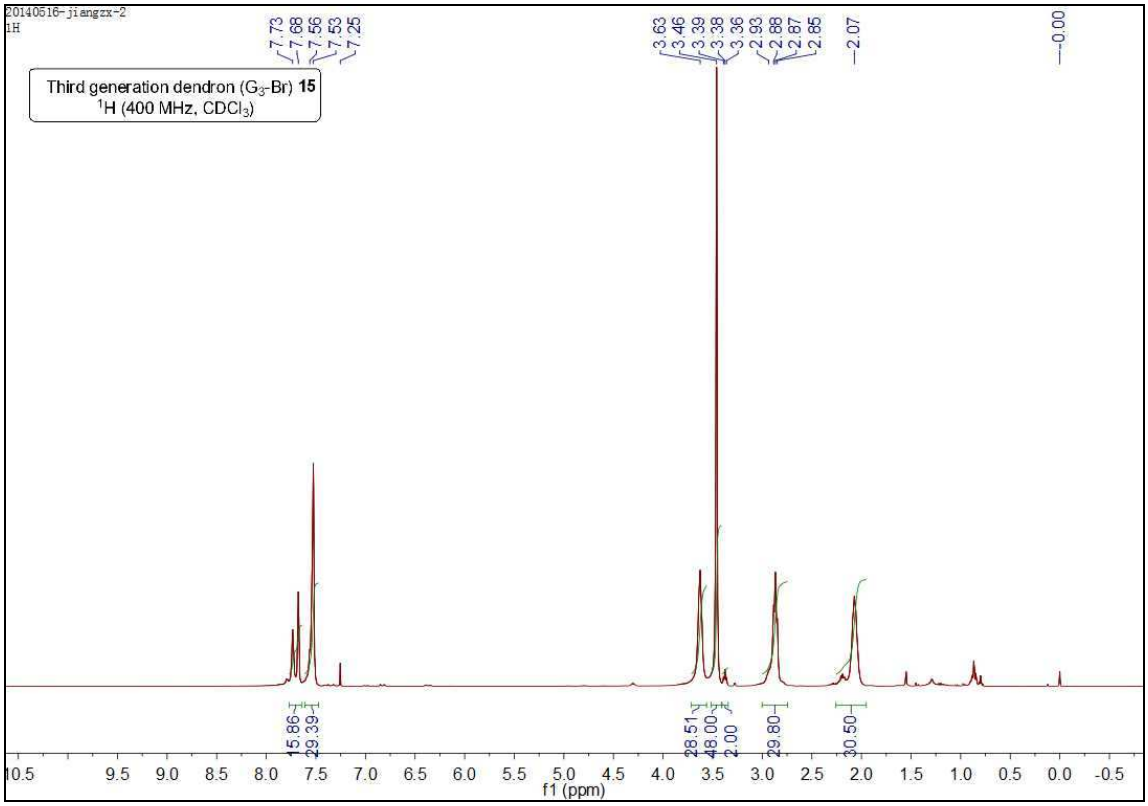

Second generation dendron (G_2-Br) **13**
Data: WAX14-1870002.C9[c] 26 Jun 2014 21:57 Cal: LSH-LINER 26 Jun 2014 21:57
Shimadzu Biotech Axima Performance 2.8.4.20081127: Mode Linear, Power: 87, Blanked, P.Ext. @3344 (bin 89)
%Int. 59 mV[sum= 1130 mV] Profiles 1-19 Smooth Av 5 -Baseline 80
3360.8
3359.7
3363.0
3372.7
3358.1
3334.2
3398.1
3554.8
3612.9
3609.3
3706.4
3733.1
3721.6
3746.9
3749.6
3761.4
100 90 80 70 60 50 40 30 20 10 0
3000 3100 3200 3300 3400 3500 3600 3700 3800 3900 4000
m/z

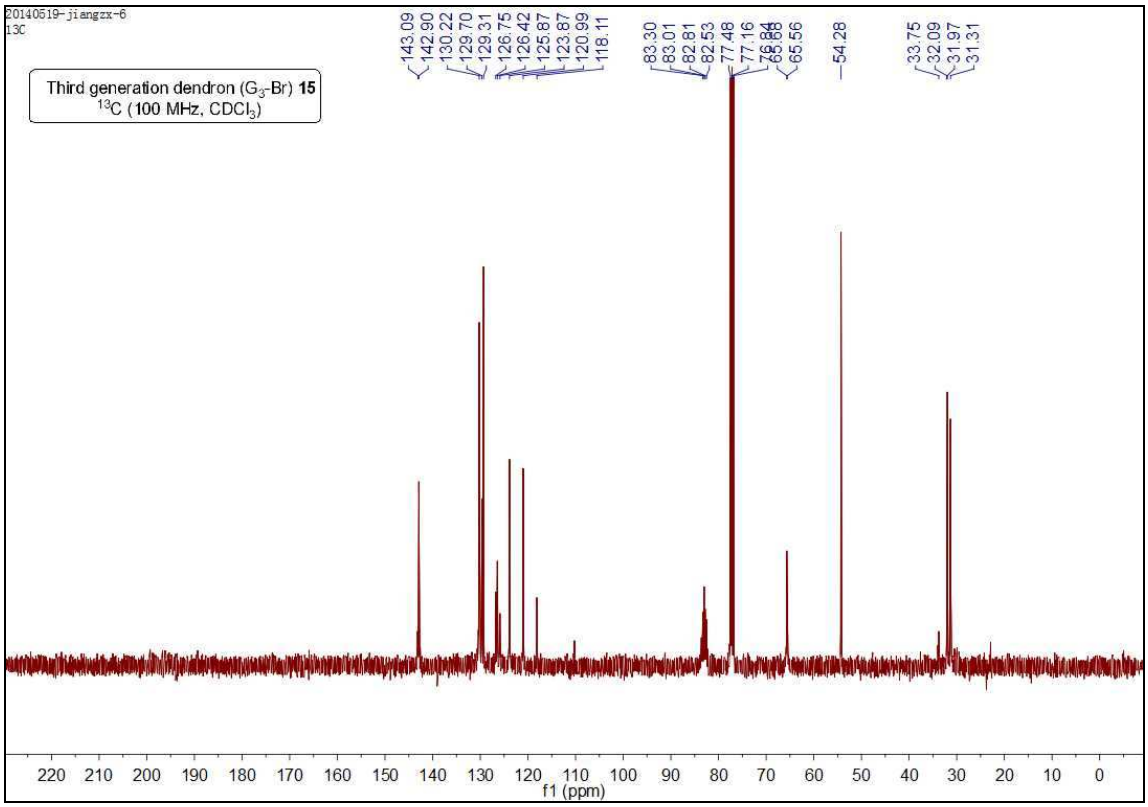

20140514-jiangzx-23
1H
Third generation dendron (G3-OH) 14
1H (400 MHz, CDCl3)
7.78
7.73
7.67
7.52
7.26
3.71
3.70
3.68
3.62
3.46
2.88
2.86
2.84
2.19
2.07
1.93
1.91
1.89
1.29
1.27
0.88
0.87
0.86
0.85
15.86
29.86
31.70
48.00
29.06
29.44
f1 (ppm)

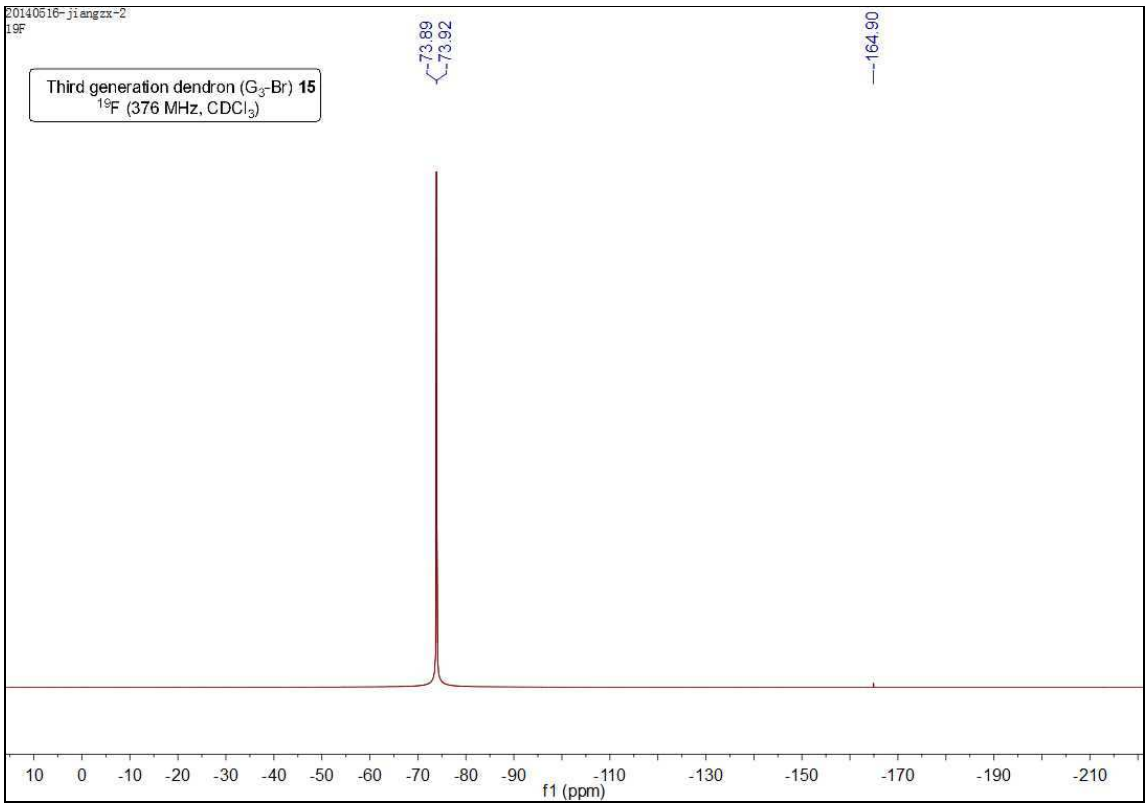

20140516-jiangzx-8
13C
Third generation dendron (G3-OH) 14
13C (100 MHz, CDCl3)
143.15
142.95
130.26
129.74
129.34
126.80
126.46
125.94
123.91
121.03
118.16
83.63
83.33
83.06
82.77
82.58
77.48
77.16
76.84
65.71
65.57
61.68
54.28
34.13
31.99
31.34
f1 (ppm)


20140514-jiangzx-23
19F
Third generation dendron (G_3-OH) **14**
^{19}F (376 MHz, $CDCl_3$)
-73.91
-73.95
-73.99
-164.90
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)


Data: NAX15-00203-CHCA0001.G8[c] 11 Mar 2015 15:44 Cal: LSH-12362-16952 11 Mar 2015 15:43
Shimadzu Biotech Axima Performance 2.8.4.20081127: Mode Linear, Power: 108, Blanked, P.Ext. @ 6992 (bin 129)
%Int. 76 mV[sum= 1520 mV] Profiles 34-53 Smooth Av 5 -Baseline 80
Third generation dendron (G_3-OH) **14**
7015.2
7040.2
5464.4
6088.5
6834.1
7330.0
7478.4
7941.3
8338.5
8867.0
9478.6
100 90 80 70 60 50 40 30 20 10 0
6000 7000 8000 9000 10000
m/z


20140516-jiangzx-2
1H
Third generation dendron (G3-Br) 15
1H (400 MHz, CDCl3)
7.73
7.68
7.56
7.53
7.25
3.63
3.46
3.39
3.38
3.36
2.93
2.88
2.87
2.85
2.07
0.00
15.86
29.39
28.51
48.00
2.00
29.80
30.50
f1 (ppm)


20140519-jiangzx-6
13C
Third generation dendron (G3-Br) 15
13C (100 MHz, CDCl3)
143.09
142.90
130.22
129.70
129.31
126.75
126.42
125.87
123.87
120.99
118.11
83.30
83.01
82.81
82.53
77.48
77.16
65.56
54.28
33.75
32.09
31.97
31.31
f1 (ppm)


20140516-jiangzx-2
19F
Third generation dendron (G3-Br) 15
19F (376 MHz, CDCl3)
-73.89
-73.92
-164.90
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)