Supporting Information

Ligand-Free Highly Effective Iron/Copper Co-catalyzed Formation of Dimeric Aryl Ethers or Sulfides

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Contents

Experimental Section

General experimental: All reactions were carried out under an argon atmosphere condition. Solvents were dried and degassed by the standard methods and all aryl halides were purchased from Aldrich or Alfa. Various alkynes, iron and copper salts were purchased from Aldrich, Acros or Alfa. Flash column chromatography was performed using silica gel (300–400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 200–300 mesh silica gel impregnated with a fluorescent indicator (254 nm). NMR spectra were recorded in $CDCl_3$ on a Varian Inova-400 NMR spectrometer (400 MHz) with TMS as an internal reference. Products were characterized by comparison of ^{1}H NMR, ^{13}C NMR and TOF-MS data in the literatures.

General procedure for iron/copper co-catalyzed formation of aryl ethers or sulfides: A mixture of aryl halide (0.5 mmol), phenol (2.5 mmol), $Fe(acac)_3$ (5 mol %), CuI (5 mol %), K_2CO_3 (2 equiv), and DMSO (2 mL) in a Schlenk tube was stirred under argon atmosphere at 140 °C for the desired time until complete consumption of starting material as monitored by TLC. After the reaction, the mixture was poured into ether, then washed with water, extracted with ethyl acetate, dried by anhydrous Na_2SO_4, then filtered and evaporated under vacuum, the residue was purified by flash column chromatography (petroleum ether or petroleum ether/ethyl acetate) to afford the corresponding coupling products.

Copy of Certificate of Analysis of CuI and $Fe(acac)_3$:

Certificate of Analysis

SIGMA-ALDRICH

Product Name	Copper(I) iodide, 99.999% trace metals basis
Product Number	215554
Product Brand	ALDRICH
CAS Number	7681-65-4
Molecular Formula	CuI
Molecular Weight	190.45

TEST	SPECIFICATION	LOT MKBB2521 RESULTS
Appearance (Color): Grey to Tan	Conforms to Requirements	Beige
Appearance (Form): Powder/Chunks	Conforms to Requirements	Powder
Complexometric EDTA: % Cu	31.0 - 34.0 %	33.7 %
ICP: Confirms Copper Component	Confirmed	Conforms
Trace Metal Analysis	≤20.0 ppm	3.7 ppm
Cesium (Cs)	ppm	0.4 ppm
Calcium (Ca)	ppm	2.1 ppm
Silver (Ag)	ppm	0.2 ppm
Zinc (Zn)	ppm	0.4 ppm
Lead (Pb)	ppm	0.3 ppm
Cobalt (Co)	ppm	0.2 ppm
Europium (Eu)	ppm	0.1 ppm
Purity	Meets Requirements 99.999% Based On Trace Metals Analysis	Meets Requirements
Specification Date:		MAR 2009
Date of QC Release:		SEP 2009
Print Date:		SEP 02 2009

Barbara Rajzer, Supervisor
Quality Control
Milwaukee, Wisconsin USA

Certificate of Analysis

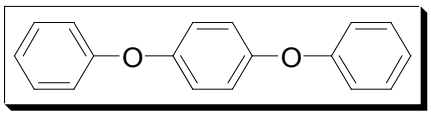
SIGMA-ALDRICH

Product Name	Iron(III) acetylacetonate, ≥99.9% trace metals basis
Product Number	517003
Product Brand	ALDRICH
CAS Number	14024-18-1
Molecular Formula	$Fe(C_5H_7O_2)_3$
Molecular Weight	353.17

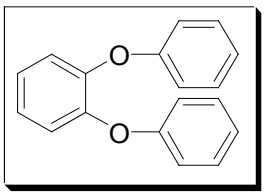
TEST	SPECIFICATION	LOT MKBD1863 RESULTS
Appearance (Color)	Red to Dark Red	Red
Appearance (Form)	Powder	Powder
Infrared spectrum	Conforms to Structure	Conforms
Titration by Na2S2O3: % Fe	15.3 - 16.4 %	15.8 %
ICP: Confirms Iron Component	Conforms	Conforms
Trace Metal Analysis	≤1000.0 ppm	371.5 ppm
Aluminum (Al)	ppm	238.2 ppm
Calcium (Ca)	ppm	31.1 ppm
Chromium (Cr)	ppm	7.9 ppm
Lead (Pb)	ppm	27.1 ppm
Lithium (Li)	ppm	1.5 ppm
Magnesium (Mg)	ppm	1.5 ppm
Manganese (Mn)	ppm	5.1 ppm
Sodium (Na)	ppm	6.0 ppm
Tin (Sn)	ppm	7.9 ppm
Titanium (Ti)	ppm	18.4 ppm
Vanadium (V)	ppm	0.6 ppm
Zinc (Zn)	ppm	26.2 ppm
Purity	Meets Requirements >=99.9% Based On Trace Metals Analysis	Meets Requirements
Specification Date:		AUG 2009
Date of QC Release:		MAR 2010
Print Date:		MAR 16 2010

Barbara Rajzer, Supervisor
Quality Control
Milwaukee, Wisconsin USA

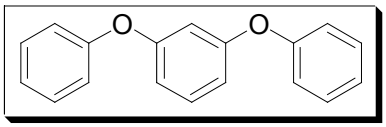
Characterization of the corresponding products:



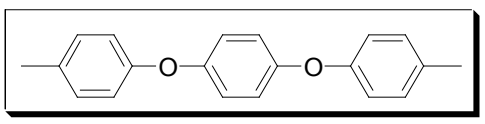
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.00 (t, J = 4.0 Hz, 8H), 7.07–7.11 (t, J = 7.2 Hz, 2H), 7.31–7.35 (t, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 118.5, 120.7, 123.2, 129.9, 152.9, 158.0; HRMS (ESI$^+$): calcd. for $[C_{18}H_{14}O_2]^+$ requires m/z 262.0994, found 262.0994.



Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.89 (d, J = 8.0 Hz, 4H), 7.02 (t, J = 7.6 Hz, 2H), 7.04–7.11 (m, 4H), 7.24 (d, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 117.2, 121.2, 122.4, 124.3, 129.0, 147.3, 157.0; HRMS (ESI$^+$): calcd. for $[C_{18}H_{14}O_2]^+$ requires m/z 262.0994, found 262.0995.

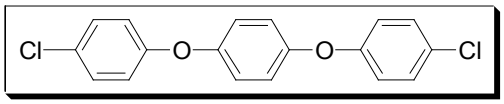


Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.69–6.72 (m, 3H), 7.02 (d, J = 8.0 Hz, 4H), 7.10 (t, J = 7.6 Hz, 2H), 7.23 (t, J = 8 Hz, 1H), 7.32 (t, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 110.9, 114.8, 120.8, 125.2, 131.4, 132.0, 158.3, 160.3; MS HRMS (ESI$^+$): calcd. for $[C_{18}H_{14}O_2]^+$ requires m/z 262.0994, found 262.0993.

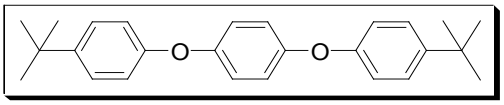


Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 2.33 (s, 6H), 6.91 (d, J = 8.0 Hz, 4H), 6.96 (s, 4H), 7.13 (d, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm)

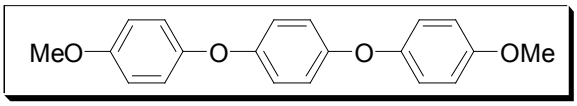
20.2, 118.0, 119.5, 129.8, 132.1, 152.5, 154.9; HRMS (ESI^+): calcd. for $[C_{20}H_{18}O_2]^+$ requires m/z 290.1307, found 290.1308.



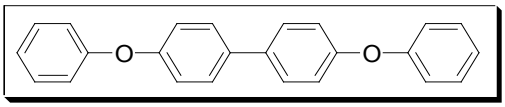
Sticky oil; ^1H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.93 (d, J = 8.8 Hz, 4H), 7.00 (s, 4H), 7.28 (d, J = 8.8 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 121.1, 122.1, 129.7, 131.3, 154.2, 157.90; HRMS (ESI^+): calcd. for $[C_{18}H_{12}O_2Cl_2]^+$ requires m/z 330.0214, found 330.0216.



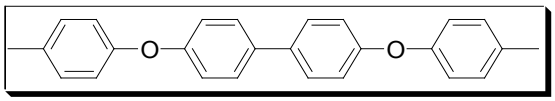
Mp: 124–125 ^oC;^1H NMR (400 MHz, $CDCl_3$) (δ, ppm) 1.32 (s, 18H), 6.93 (d, J = 8.8 Hz, 4H), 6.98 (s, 3H), 7.25 (m, 1H), 7.34 (d, J = 8.8 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 31.7, 34.5, 117.9, 120.4, 126.7, 146.0, 153.0, 155.5; HRMS (ESI^+): calcd. for $[C_{26}H_{30}O_2]^+$ requires m/z 374.2246, found 374.2245.



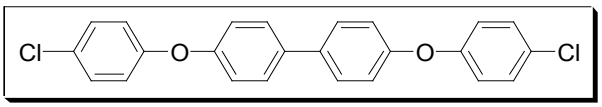
Mp: 147–148 ^oC; ^1H NMR (300 MHz, $CDCl_3$) (δ, ppm) 3.79 (s, 6H), 6.85–6.97 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 55.9, 115.0, 119.3, 120.2, 151.1, 153.6, 155.8.



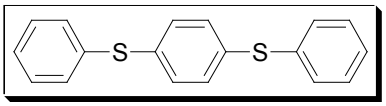
Mp: 153–154 ^oC; ^1H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.08–7.10 (m, 8H), 7.14 (t, J = 7.6 Hz, 2H), 7.38 (t, J = 7.6 Hz, 4H), 7.54 (d, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 119.2, 119.3, 123.5, 128.4, 130.0, 135.8, 156.8, 157.3; HRMS (ESI^+): calcd. for $[C_{24}H_{18}O_2]^+$ requires m/z 338.1307, found 338.1308.



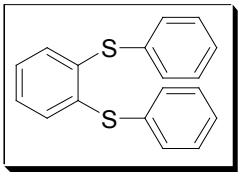
Mp: 179–180 °C; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 2.35 (s, 6H), 6.96 (d, J = 8.4 Hz, 4H), 7.03 (d, J = 8.8 Hz, 4H), 7.16 (d, J = 8.4 Hz, 4H), 7.49 (d, J = 8.8 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 20.3, 118.1, 118.7, 127.6, 129.8, 132.6, 134.9, 154.2, 156.7; HRMS (ESI$^+$): calcd. for $[C_{26}H_{22}O_2]^+$ requires m/z 366.1620, found 366.1623.



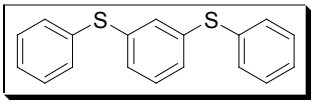
Mp: 182–183 °C; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.75 (d, J = 8.0 Hz, 4H), 6.98 (d, J = 8.4 Hz, 1H), 7.05 (d, J = 8.4 Hz, 2H), 7.16 (d, J = 8.4 Hz, 4H), 7.30 (d, J = 8.8 Hz, 2H), 7.51 (t, J = 8.0 Hz, 2H), 7.74 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 119.3, 120.3, 127.2, 127.4, 128.5, 130.0, 156.0, 156.5; HRMS (ESI$^+$): calcd. for $[C_{24}H_{16}O_2Cl_2]^+$ requires m/z 406.0527, found 406.0528.



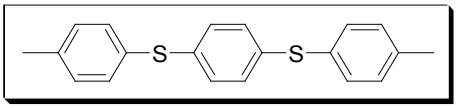
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.24 (s, 5H), 7.29–7.33 (m, 4H), 7.37 (t, J = 7.6 Hz, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 129.0, 130.9, 132.8, 133.1, 136.5, 136.6; HRMS (ESI$^+$): calcd. for $[C_{18}H_{14}S_2]^+$ requires m/z 294.0537, found 294.0536.



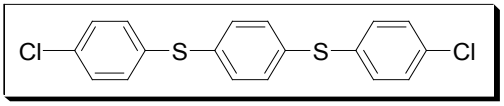
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.12 (s, 4H), 7.32–7.38 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 127.1, 128.9, 130.3, 131.0, 131.4, 134.0, 137.0; HRMS (ESI$^+$): calcd. for $[C_{18}H_{14}S_2]^+$ requires m/z 294.0537, found 294.0536.



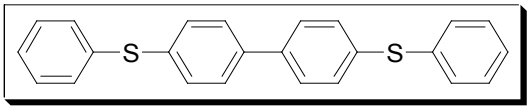
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.13–7.15 (m, 2H), 7.19–7.21 (m, 1H), 7.23 (s, 1H), 7.27–7.33 (m, 6H), 7.35–7.37 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 127.1, 127.9, 128.9, 129.2, 130.8, 131.4, 134.0, 137.2; HRMS (ESI^+): calcd. for $[C_{18}H_{14}S_2]^+$ requires m/z 294.0537, found 294.0535.



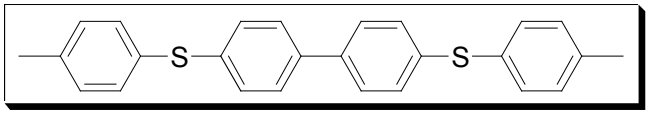
Mp: 99–100 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 2.34 (s, 6H), 7.13 (d, J = 5.6 Hz, 8H), 7.28 (d, J = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 20.7, 129.7, 129.8, 130.5, 131.9, 134.9, 137.4; HRMS (ESI^+): calcd. for $[C_{20}H_{18}S_2]^+$ requires m/z 322.0850, found 322.0844.



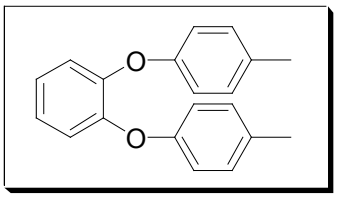
Mp: 135–137 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.22 (s, 4H), 7.28 (s, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 129.1, 130.9, 132.2, 133.1, 133.2, 134.4; HRMS (ESI^+): calcd. for $[C_{18}H_{12}S_2Cl_2]^+$ requires m/z 361.9757, found 361.9754.



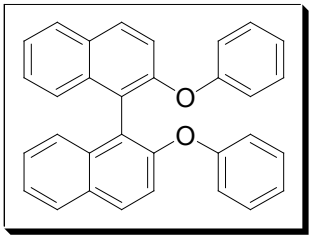
Mp: 117–118 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.28 (d, J = 7.6 Hz, 2H), 7.33 (t, J = 7.2 Hz, 4H), 7.37–7.40 (m, 8H), 7.49–7.51 (d, J = 8.4 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 126.8, 127.2, 128.8, 130.7, 130.9, 134.9, 134.9, 138.5; HRMS (ESI^+): calcd. for $[C_{24}H_{18}S_2]^+$ requires m/z 370.0850, found 370.0846.



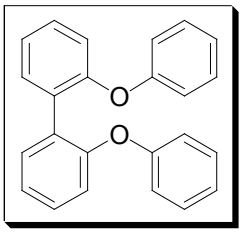
Mp: 165–166 °C; ^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 2.35 (s, 6H), 7.16 (d, J = 8.1 Hz, 4H), 7.28–7.35 (m, 8H), 7.45 (d, J = 8.1 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 21.3, 127.6, 130.1, 130.3, 131.1, 132.7, 136.7, 138.0, 138.6.



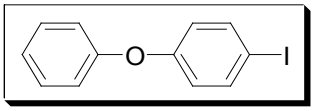
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 2.34 (s, 6H), 6.89 (d, J = 8.4 Hz, 4H), 7.07–7.08 (m, 4H), 7.12 (d, J = 7.6 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 22.3, 119.5, 122.5, 125.8, 131.6, 133.9, 149.7, 156.8; HRMS (ESI$^+$): calcd. for $[C_{20}H_{18}O_2]^+$ requires m/z 290.1307, found 290.1306.



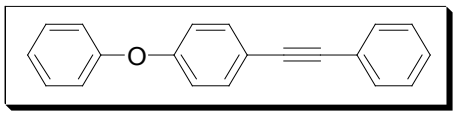
Mp: 225–226 °C; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.83 (d, J = 8.0 Hz, 4H), 6.96 (t, J = 7.6 Hz, 2H), 7.15 (t, J = 8.0 Hz, 4H), 7.22 (d, J = 7.6 Hz, 2H), 7.33 (s, 4H), 7.39–7.42 (m, 2H), 7.88 (d, J = 7.6 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 118.5, 118.9, 121.7, 122.2, 124.2, 125.4, 126.1, 127.6, 128.9, 129.2, 129.9, 133.8, 152.1, 157.1; HRMS (ESI$^+$): calcd. for $[C_{32}H_{22}O_2]^+$ requires m/z 438.1620, found 438.1620.



Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.88 (d, J = 8.4 Hz, 4H), 6.92(d, J = 8.0 Hz, 2H), 7.01 (t, J = 7.6 Hz, 2H), 7.14 (d, J = 7.6 Hz, 2H), 7.20–7.29 (m, 6H), 7.45 (dd, J = 7.6, 2.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 118.9, 119.0, 122.9, 123.3, 129.0, 129.6, 129.9, 132.2, 154.9, 157.6; HRMS (ESI^+): calcd. for $[C_{24}H_{18}O_2]^+$ requires m/z 338.1307, found 338.1306.

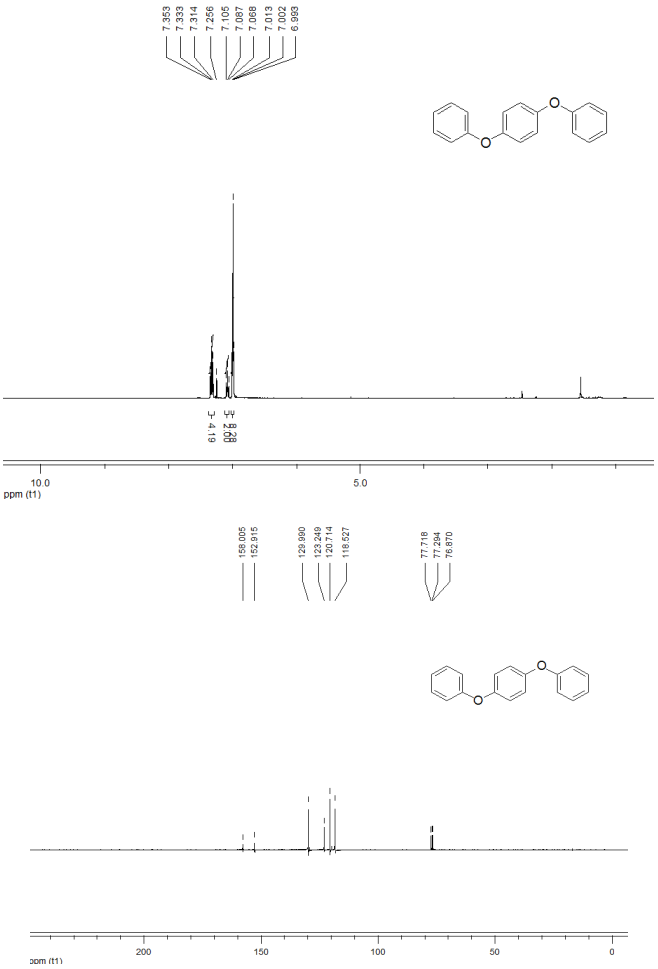


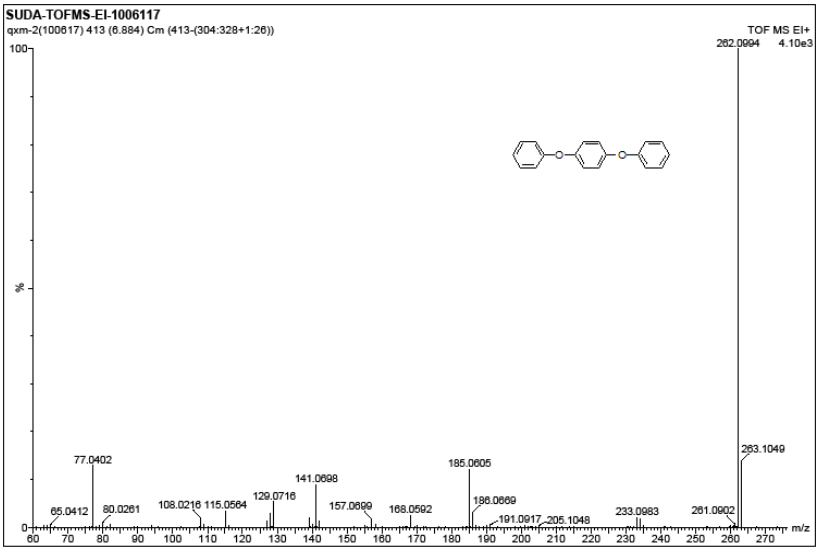
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.81 (d, J = 8.0 Hz, 2H), 7.04–7.07 (m, 2H), 7.14–7.19 (m, 1H), 7.35–7.40 (m, 2H), 7.64 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 86.1, 119.3, 121.0, 124.0, 130.1, 138.8, 156.7, 157.6; HRMS (ESI^+): calcd. For $[C_{12}H_9OI]^+$ requires m/z 295.9698, found 295.9699.

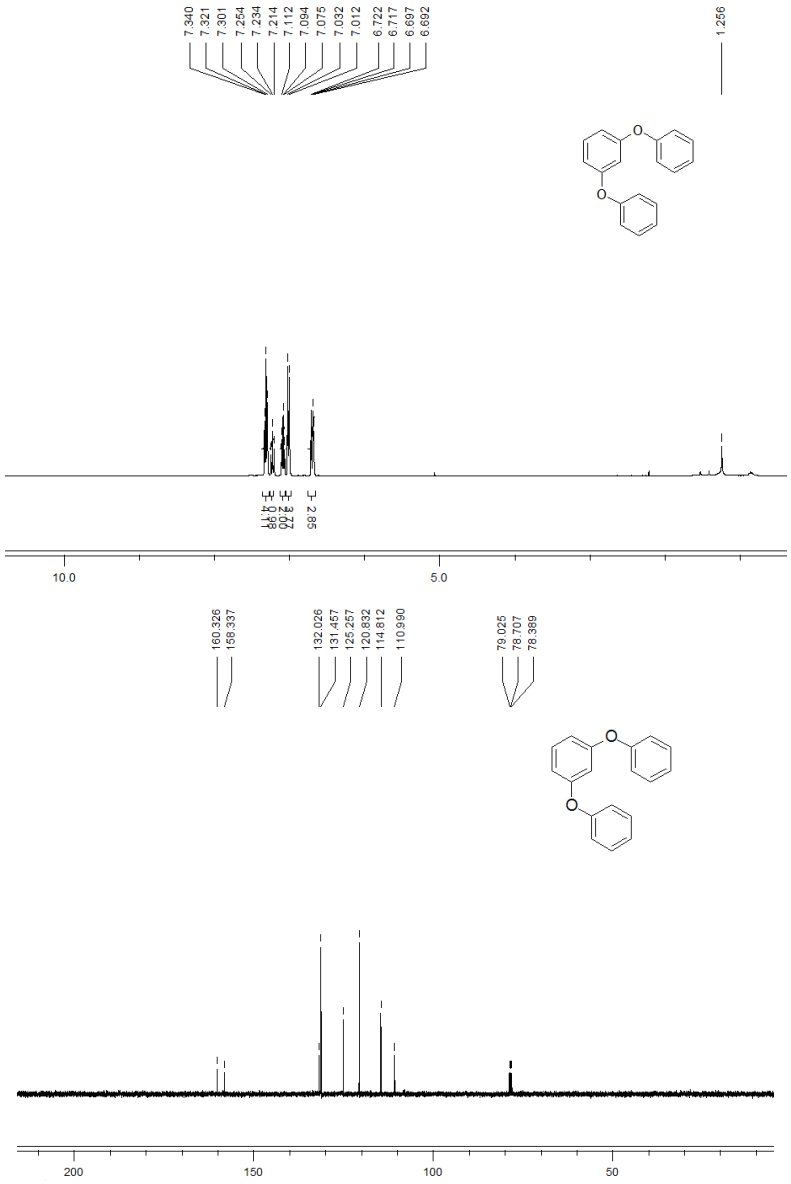


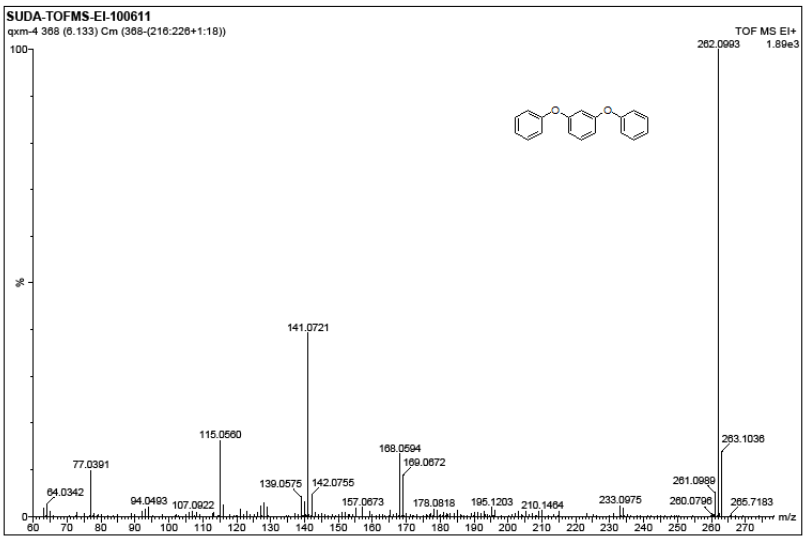
Sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 6.97 (d, J = 12.0 Hz, 2H), 7.05 (d, J = 12.0 Hz, 2H), 7.15 (t, J = 10.0 Hz, 1H), 7.32–7.39 (m, 5H), 7.48–7.54 (t, J = 12.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 88.9, 89.1, 118.0, 118.6, 119.6, 123.5, 124.0, 128.3, 128.5, 130.1, 131.7, 133.4, 156.6, 157.8; HRMS (ESI^+): calcd. For $[C_{20}H_{14}O]^+$ requires m/z 270.1045, found 270.1046.

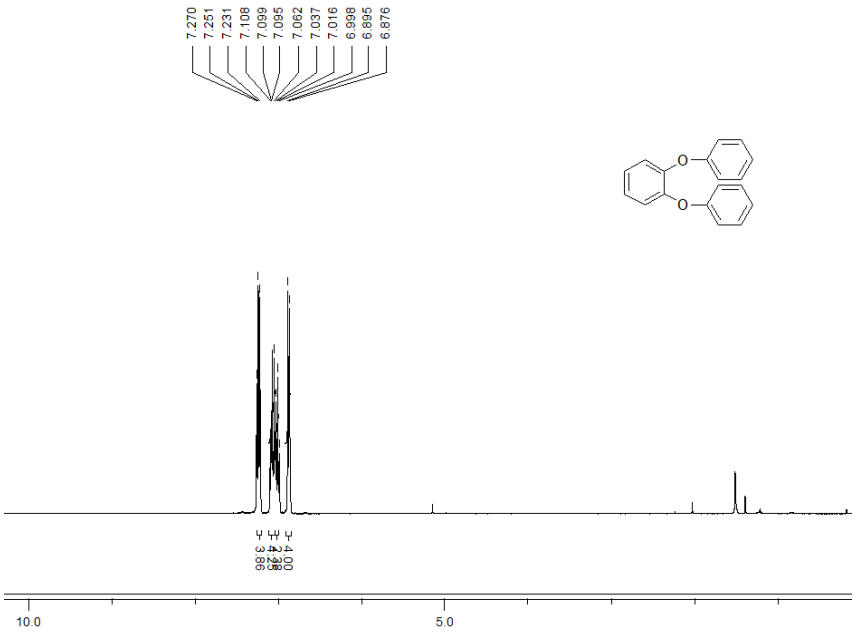
Copy of HRMS and NMR Spectra for desired products:

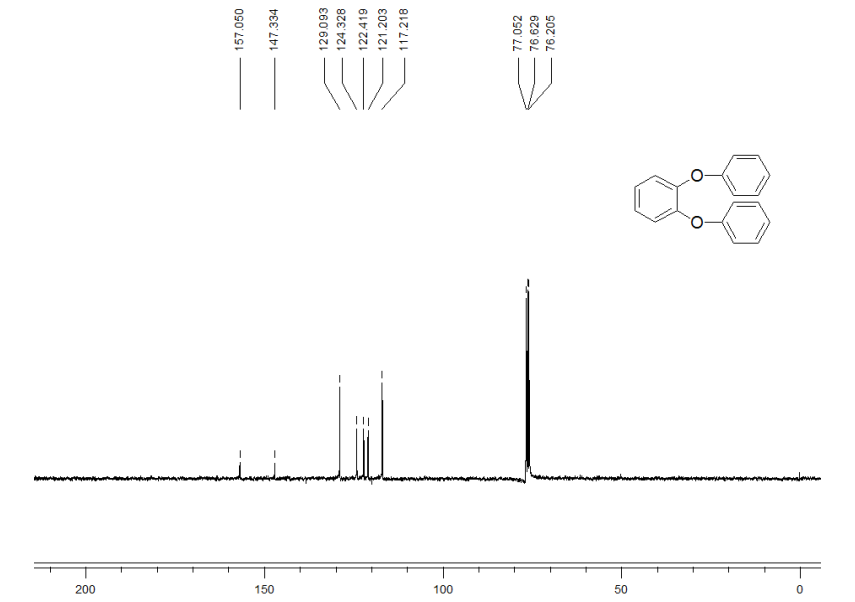


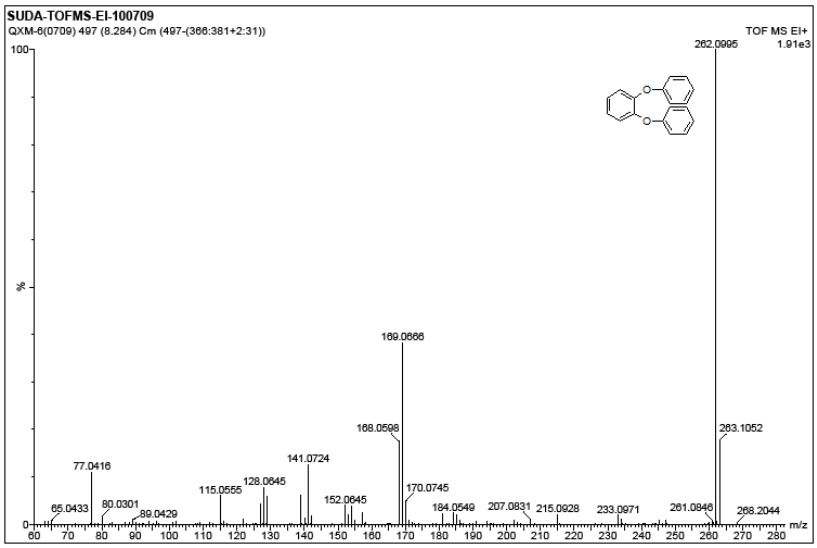



7.340
7.321
7.301
7.254
7.234
7.214
7.112
7.094
7.075
7.032
7.012
6.722
6.717
6.697
6.692
1.256
O
O
4.00
3.77
3.00
2.86
10.0
5.0
160.326
158.337
132.026
131.457
125.257
120.832
114.812
110.990
79.025
78.707
78.389
O
O
200
150
100
50

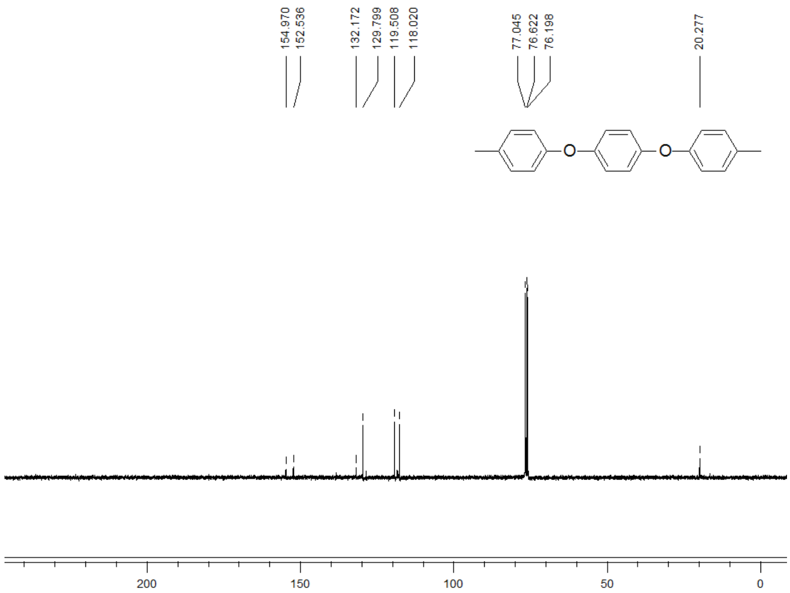

SUDA-TOFMS-EI-100611
qxm-4 368 (6.133) Cm (368-(216:226+1:18))
TOF MS EI+
1.89e3
100
%
262.0993
141.0721
115.0560
263.1036
168.0594
169.0672
77.0391
261.0989
64.0342
139.0575
142.0755
94.0493
107.0922
157.0673
178.0818
195.1203
210.1464
233.0975
260.0798
265.7183
0
60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270
m/z

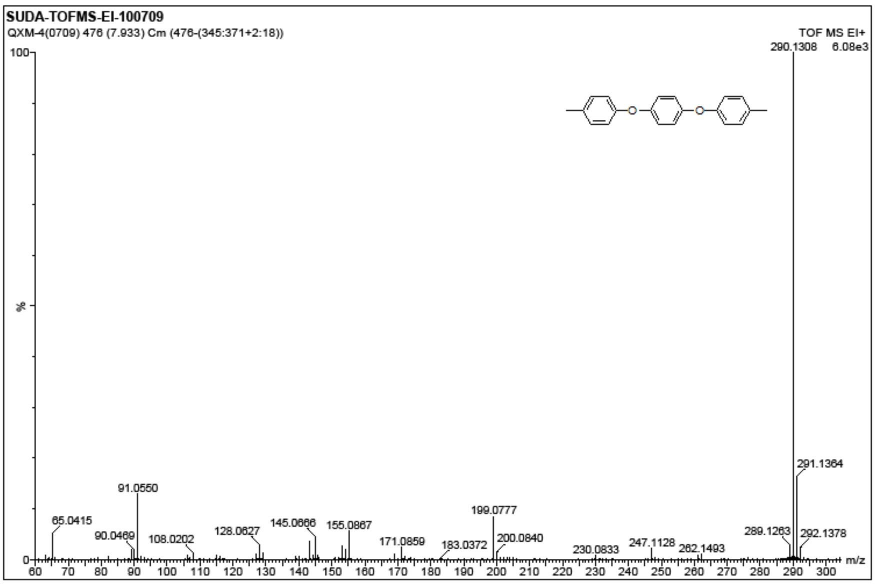

7.270
7.251
7.231
7.108
7.099
7.095
7.062
7.037
7.016
6.998
6.895
6.876
7.00
7.42
7.43
7.86
10.0
5.0

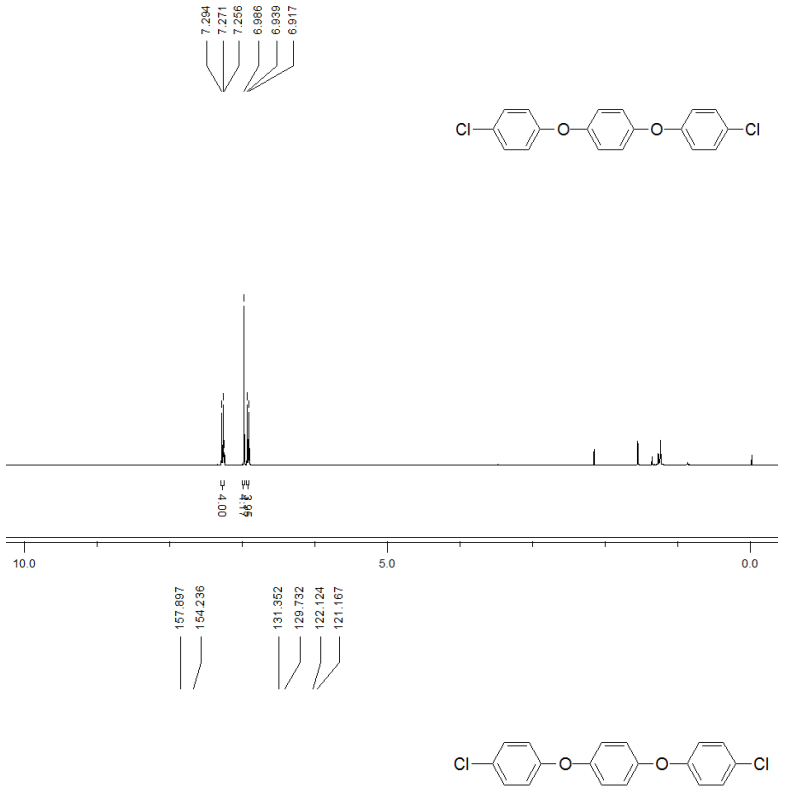

157.050
147.334
129.093
124.328
122.419
121.203
117.218
77.052
76.629
76.205
200
150
100
50
0

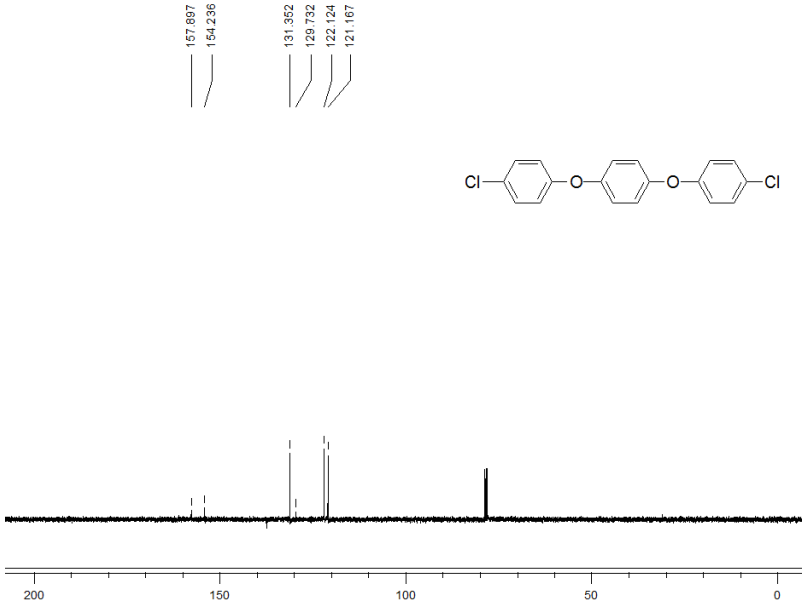

SUDA-TOFMS-EI-100709
QXM-8(0709) 497 (8.284) Cm (497-(366:381+2:31))
TOF MS EI+
1.91e3
100
%
262.0995
263.1052
169.0666
168.0598
141.0724
77.0416
128.0645
115.0555
170.0745
65.0433
80.0301
89.0429
152.0645
184.0549
207.0831
215.0928
233.0971
261.0846
268.2044
m/z

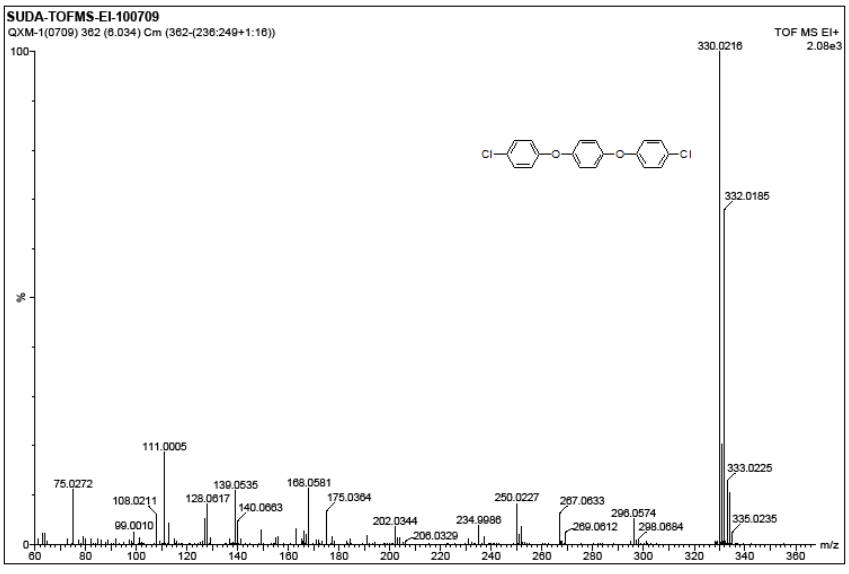

7.262
7.142
7.121
6.957
6.916
6.895
2.331
3.97
3.89
6.00
10.0
5.0
0.0

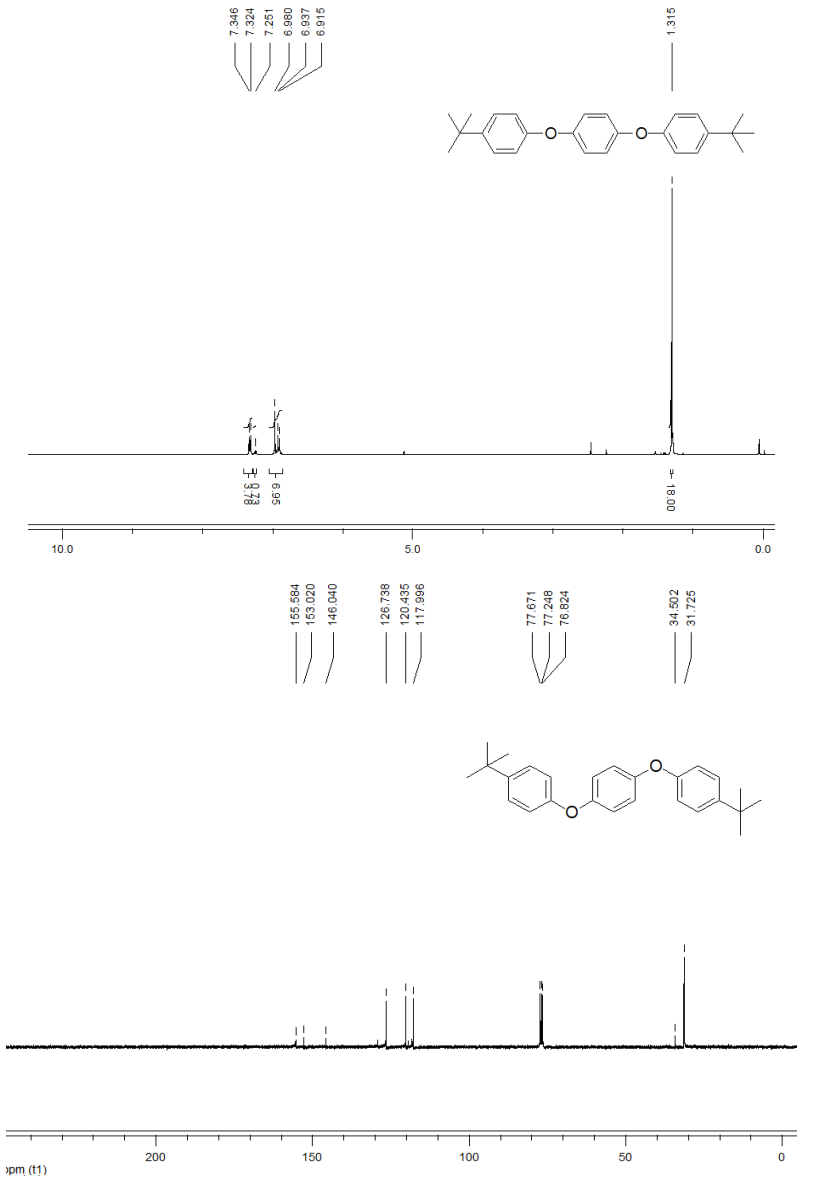

154.970
152.536
132.172
129.799
119.508
118.020
77.045
76.622
76.198
20.277
200
150
100
50
0

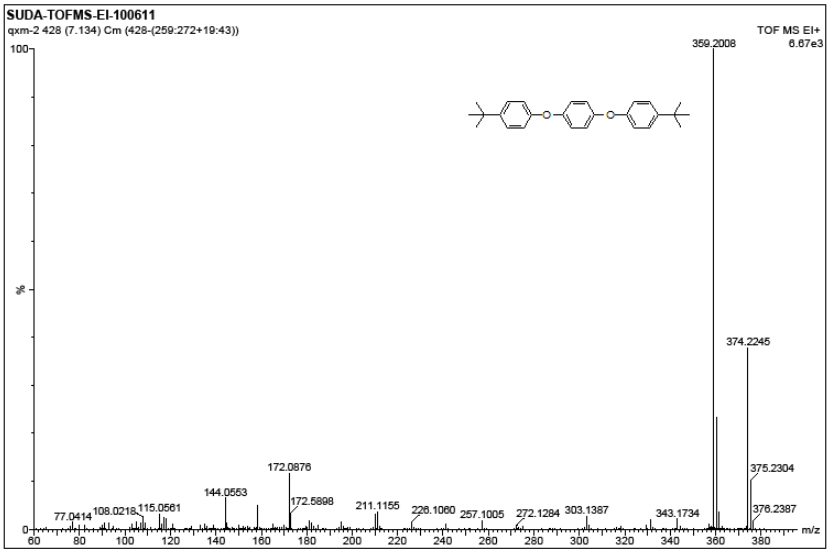

SUDA-TOFMS-EI-100709
QXM-4(0709) 476 (7.933) Cm (476-(345:371+2:18))
TOF MS EI+
290.1308 6.08e3
100
%
0
291.1364
91.0550
199.0777
65.0415
90.0469
108.0202
128.0627
145.0686
155.0867
171.0859
183.0372
200.0840
230.0833
247.1128
262.1493
289.1263
292.1378
m/z

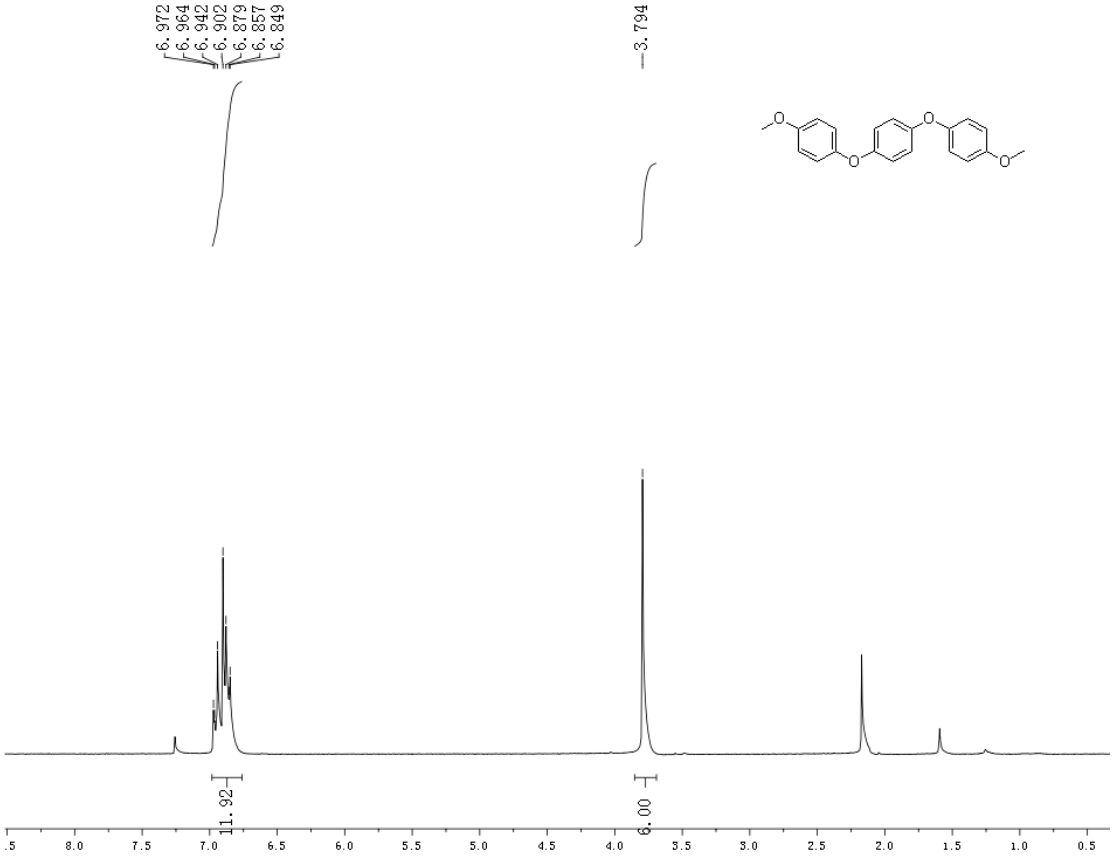

7.294
7.271
7.256
6.986
6.939
6.917
Cl
O
O
Cl
4.00
3.95
10.0
5.0
0.0

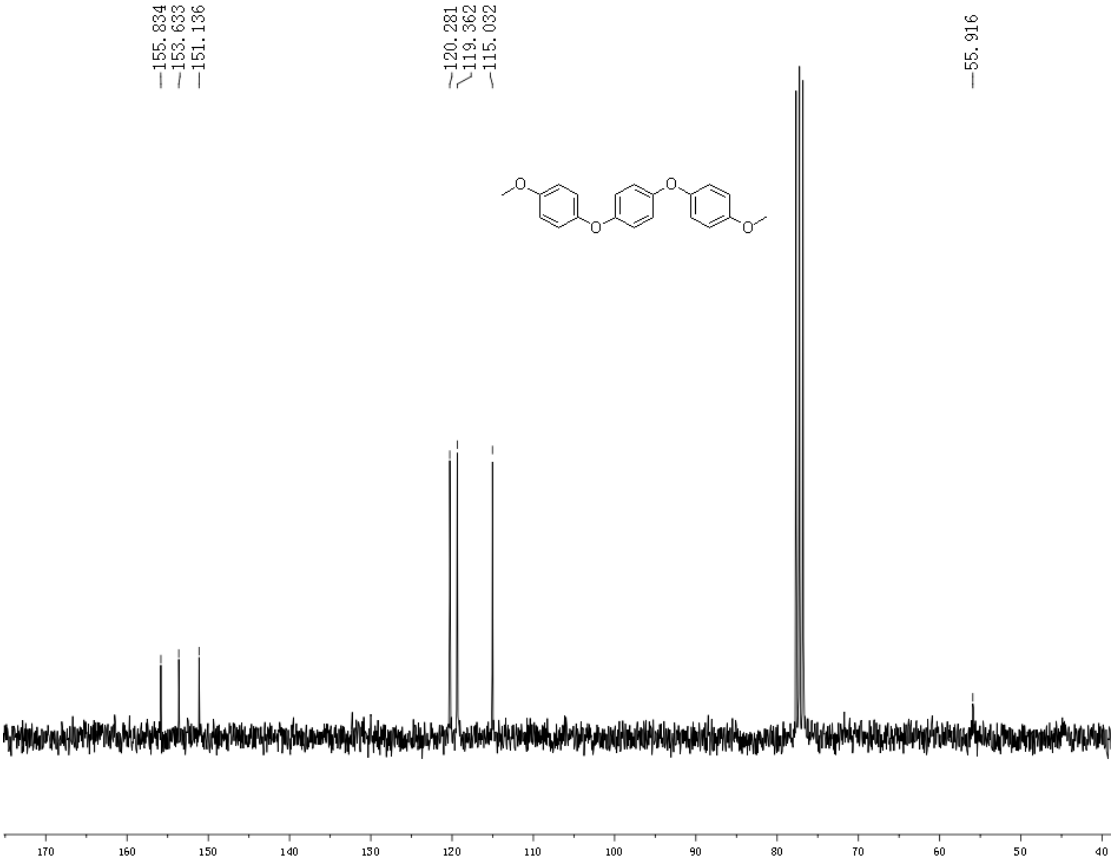

157.897
154.236
131.352
129.732
122.124
121.167
Cl
O
O
Cl
200
150
100
50
0

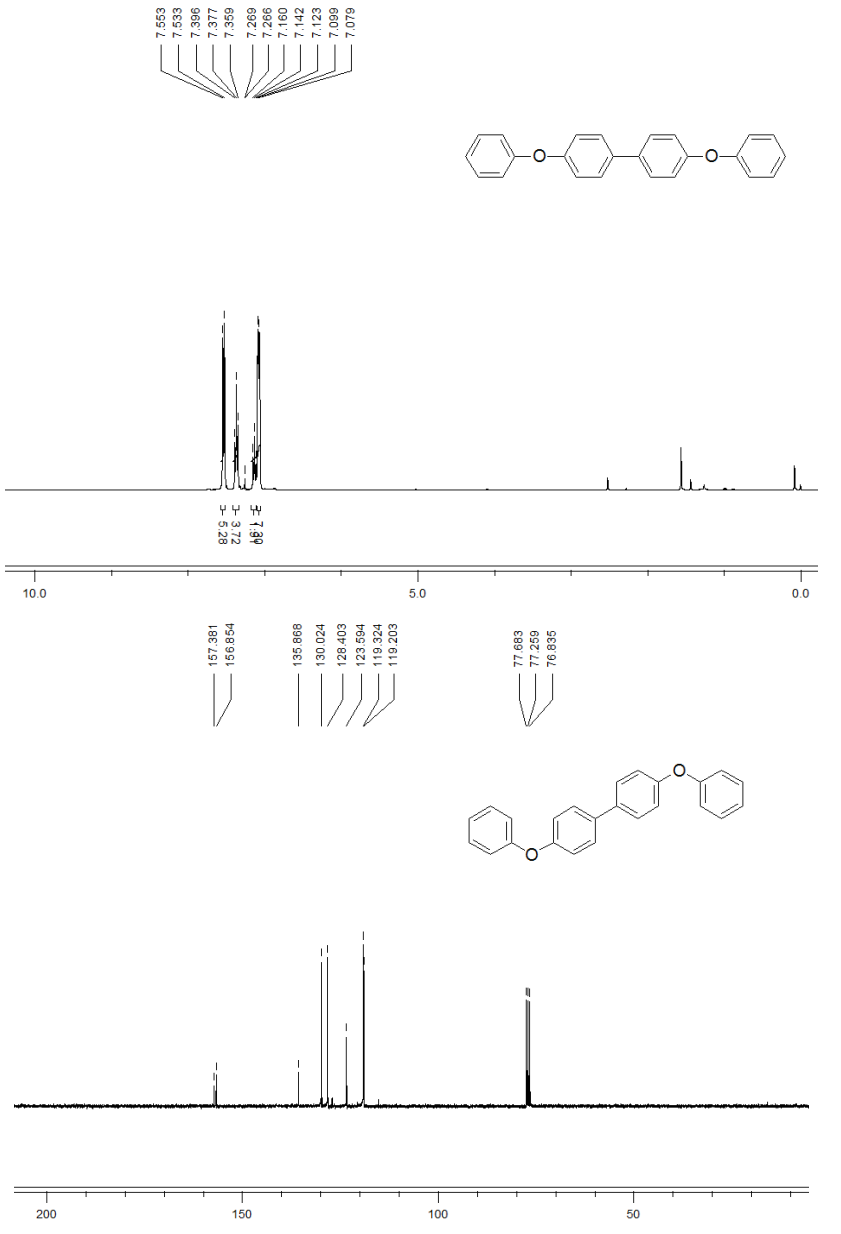

SUDA-TOFMS-EI-100709
QXM-1(0709) 362 (6.034) Cm (362-(236:249+1:16))
TOF MS EI+
2.08e3
330.0216
332.0185
333.0225
335.0235
111.0005
75.0272
139.0535
168.0581
108.0211
128.0617
175.0364
140.0683
99.0010
250.0227
267.0633
296.0574
202.0344
234.9986
206.0329
269.0612
298.0684
%
m/z

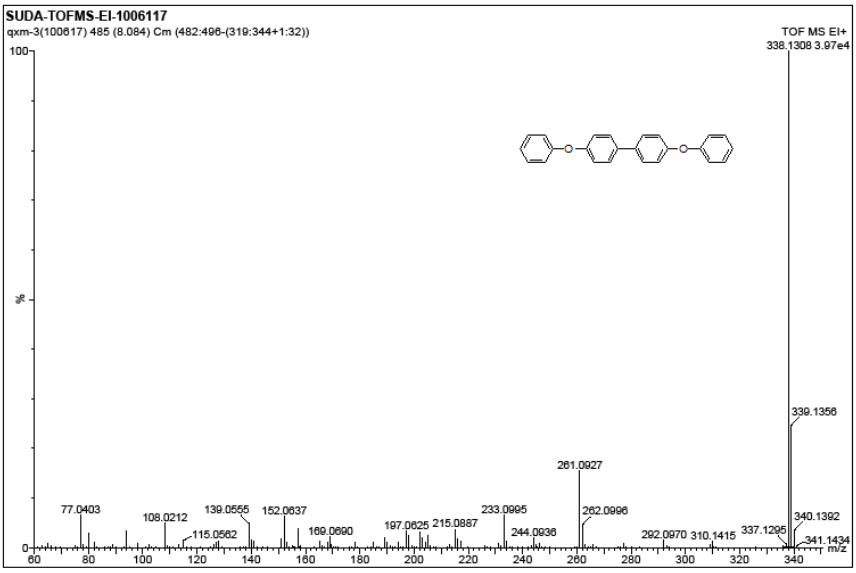

7.346
7.324
7.251
6.980
6.937
6.915
1.315
3.78
6.95
18.00
10.0
5.0
0.0
155.584
153.020
146.040
126.738
120.435
117.996
77.671
77.248
76.824
34.502
31.725
200
150
100
50
0
ppm (t1)

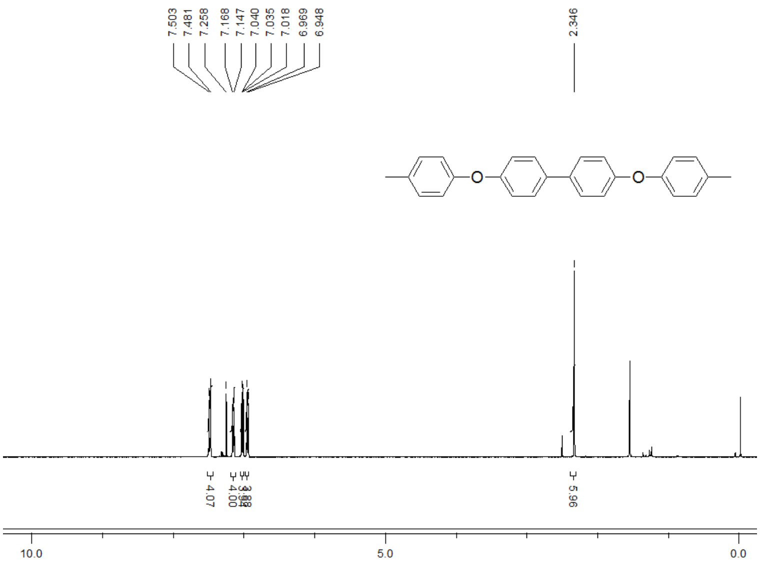

SUDA-TOFMS-EI-100611
qxm-2 428 (7.134) Cm (428-(259:272+19:43))
TOF MS EI+
6.67e3
359.2008
374.2245
375.2304
376.2387
172.0876
144.0553
172.5898
77.0414
108.0218
115.0561
211.1155
226.1060
257.1005
272.1284
303.1387
343.1734
%
m/z

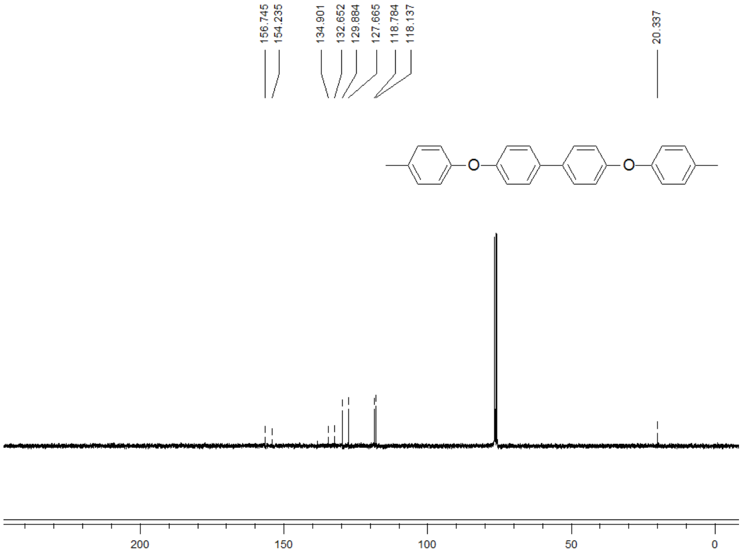

6.972
6.964
6.942
6.902
6.879
6.857
6.849
3.794
11.92
6.00

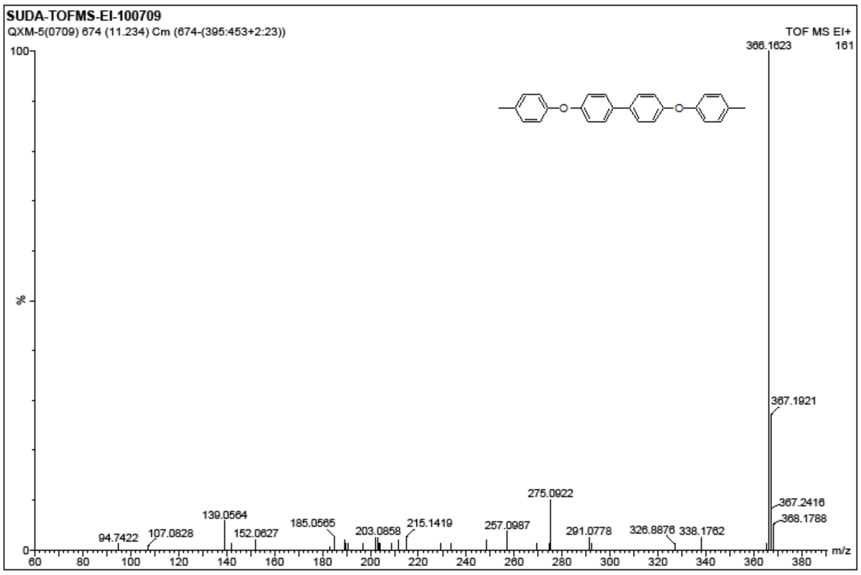

155.834
153.633
151.136
120.281
119.362
115.032
55.916

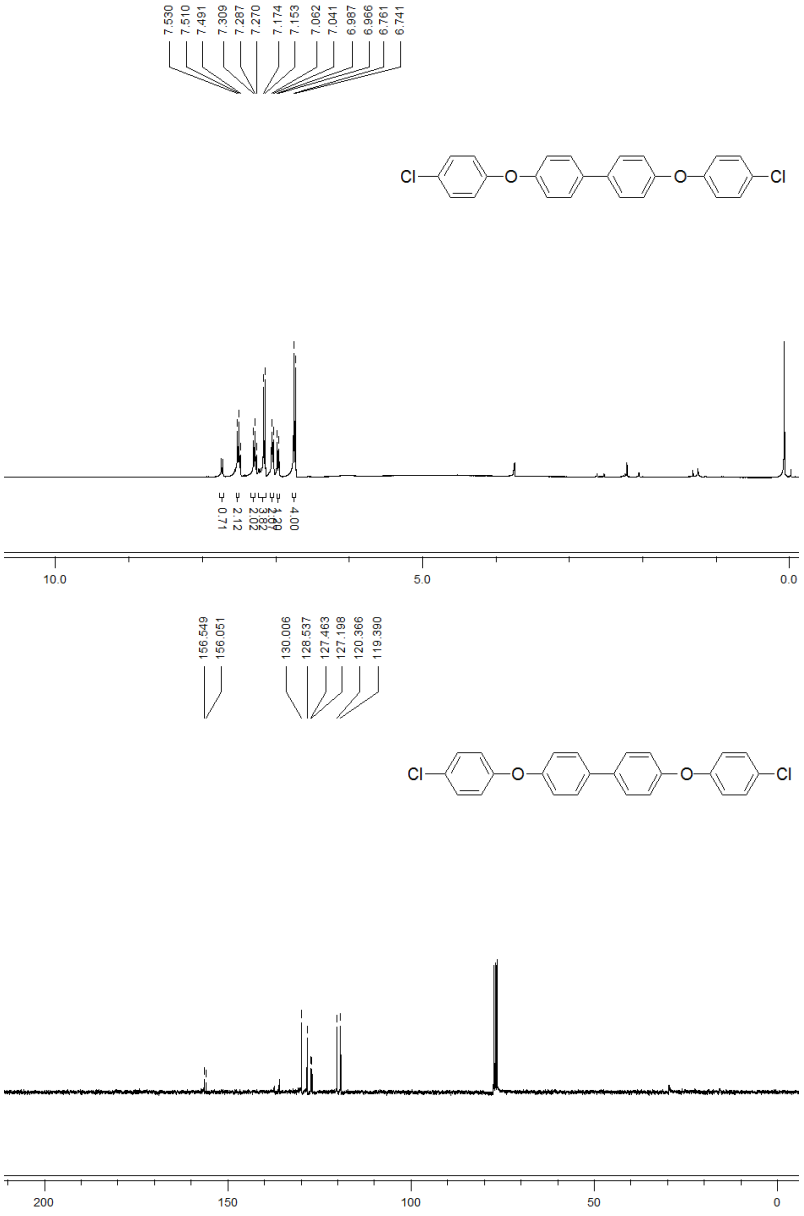

7.553
7.533
7.396
7.377
7.359
7.269
7.266
7.160
7.142
7.123
7.099
7.079
5.28
2.72
1.00
10.0
5.0
0.0
157.381
156.854
135.868
130.024
128.403
123.594
119.324
119.203
77.683
77.259
76.835
200
150
100
50

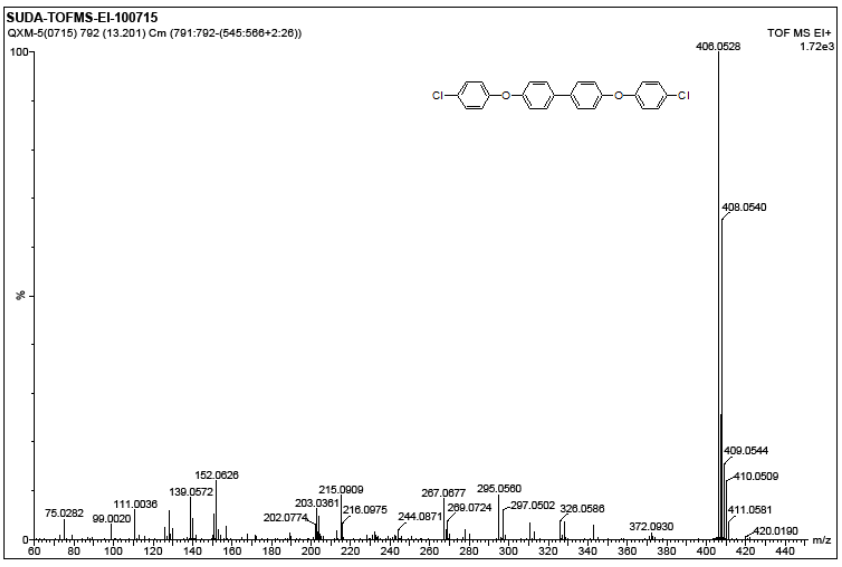

SUDA-TOFMS-EI-1006117
qxm-3(100617) 485 (8.084) Cm (482:496-(319:344+1:32))
TOF MS EI+
338.1308 3.97e4
100
%
0
339.1356
261.0927
77.0403
108.0212
139.0555
152.0637
115.0562
169.0690
197.0625
215.0887
233.0995
244.0936
262.0996
292.0970
310.1415
337.1295
340.1392
341.1434
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
m/z

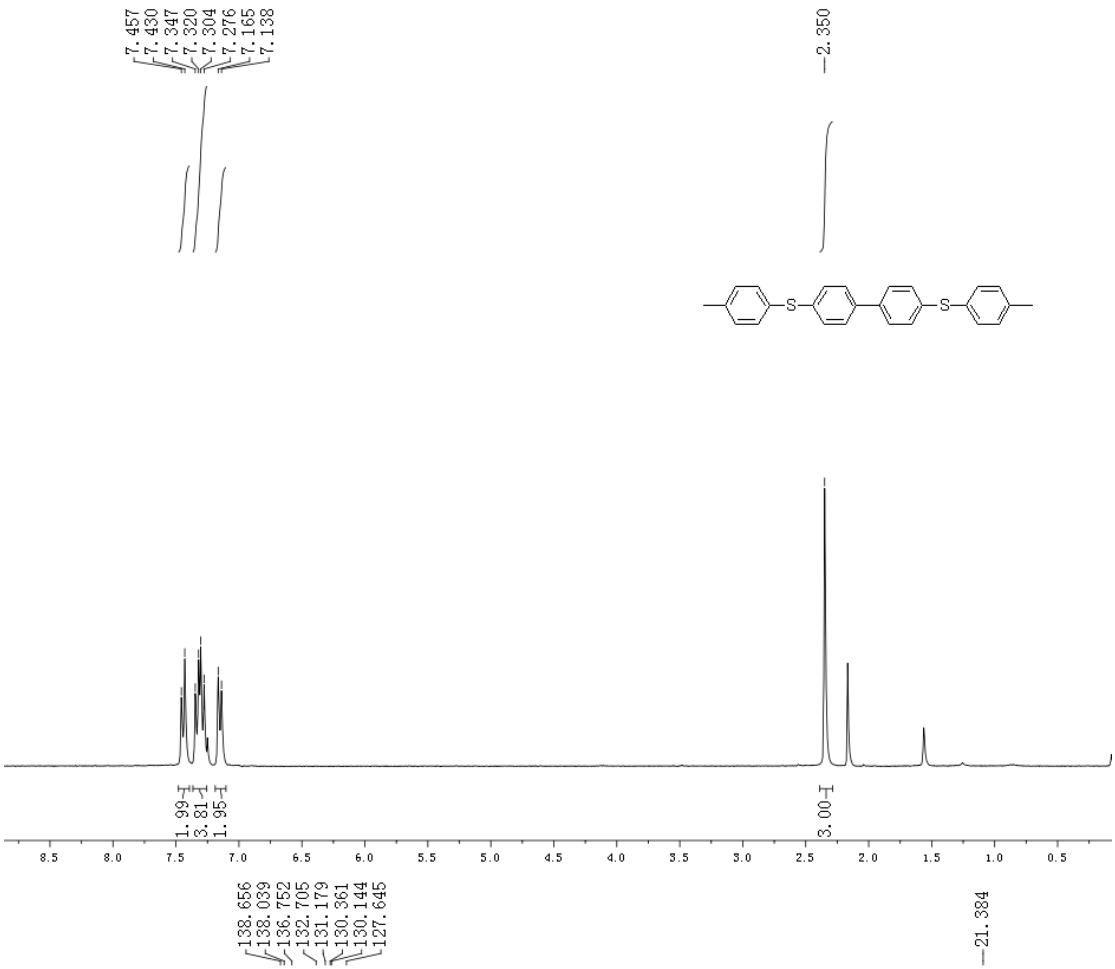

7.503
7.481
7.258
7.168
7.147
7.040
7.035
7.018
6.969
6.948
2.346
O
O
4.07
4.00
3.98
3.88
5.96
10.0
5.0
0.0

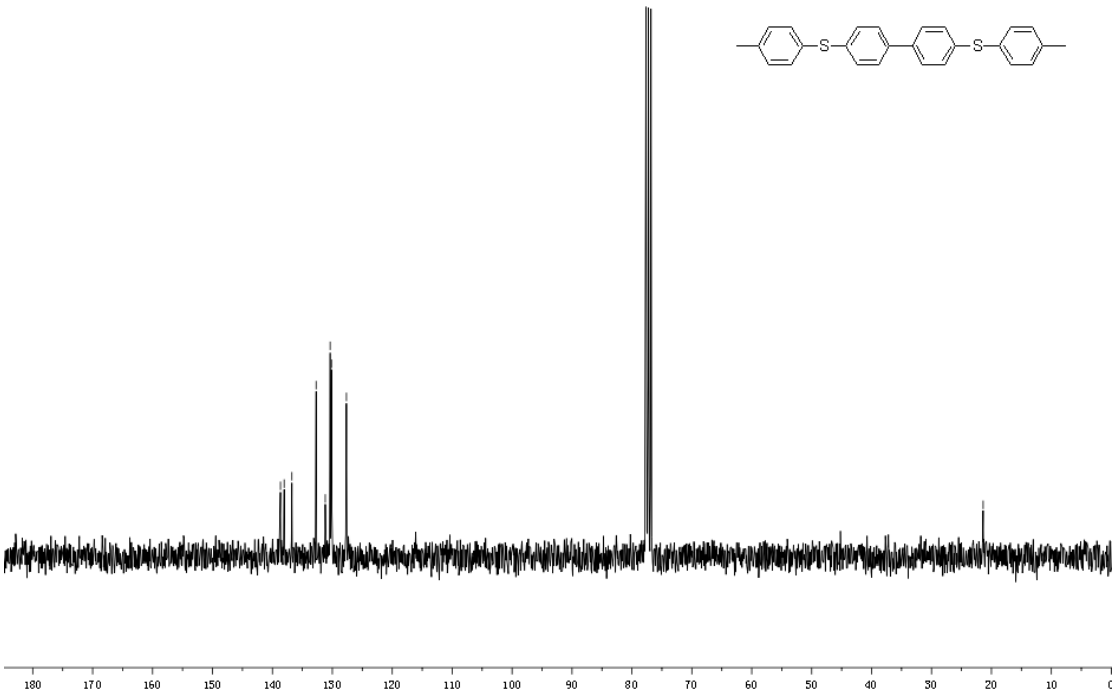

156.745
154.235
134.901
132.652
129.884
127.665
118.784
118.137
20.337
O
O
200
150
100
50
0

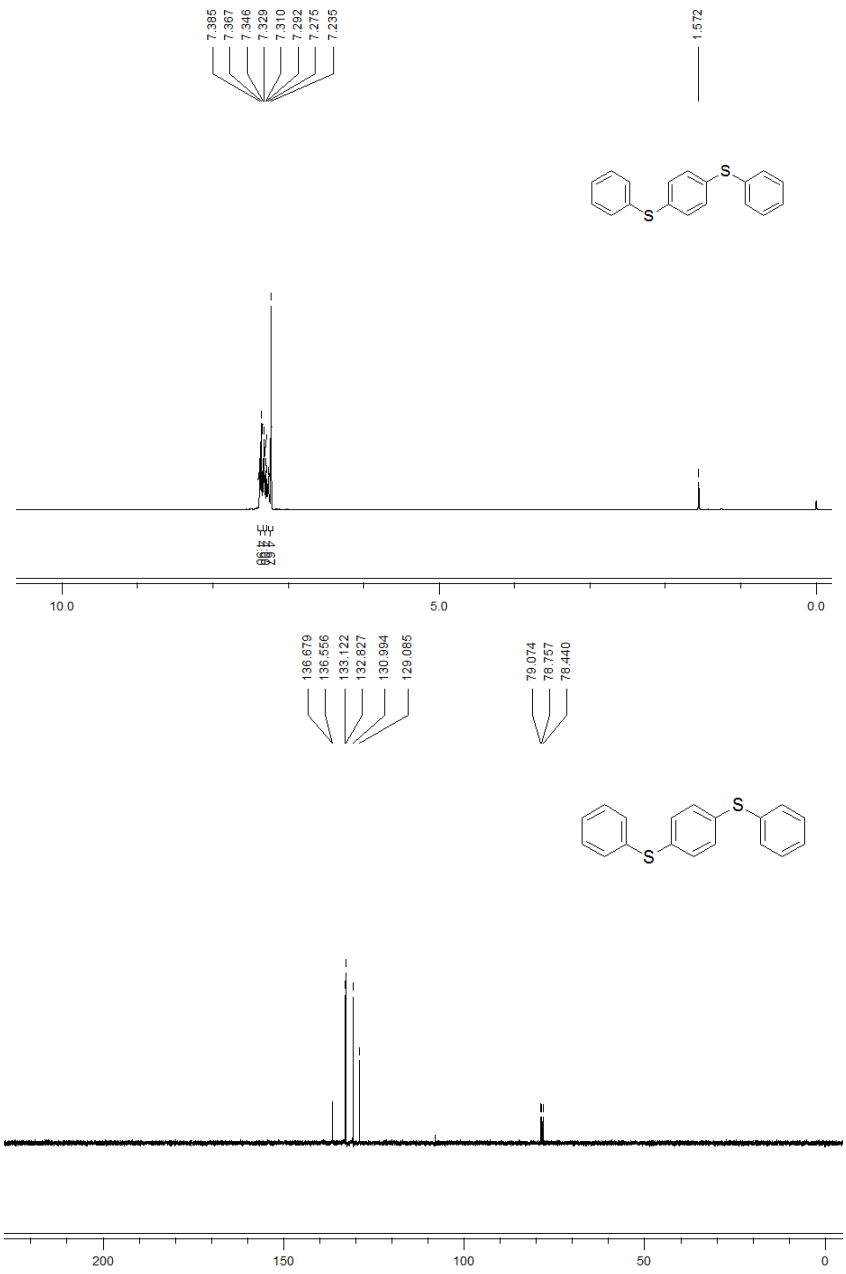

SUDA-TOFMS-EI-100709
QXM-5(0709) 674 (11.234) Cm (674-(395:453+2:23))
TOF MS EI+
366.1623
161
100
%
0
367.1921
367.2416
368.1788
275.0922
139.0564
185.0565
215.1419
257.0987
94.7422
107.0828
152.0627
203.0858
291.0778
326.8876
338.1762
m/z

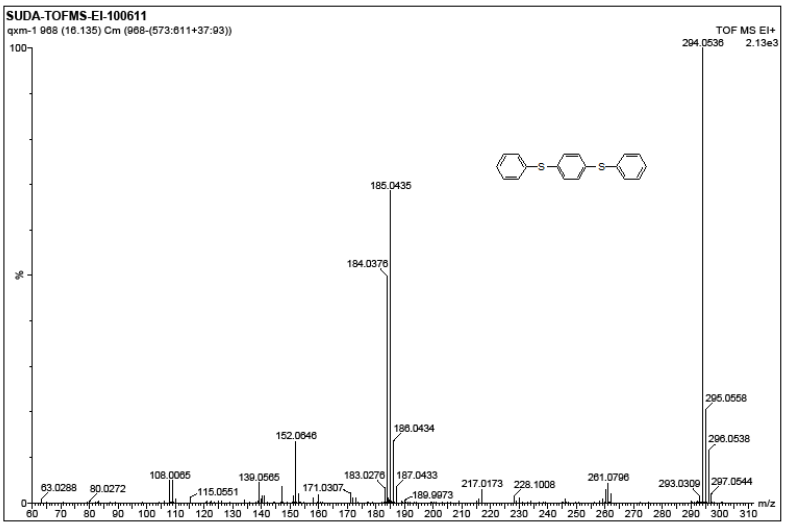

7.530
7.510
7.491
7.309
7.287
7.270
7.174
7.153
7.062
7.041
6.987
6.966
6.761
6.741
Cl
O
O
Cl
10.0
5.0
0.0
156.549
156.051
130.006
128.537
127.463
127.198
120.366
119.390
Cl
O
O
Cl
200
150
100
50
0

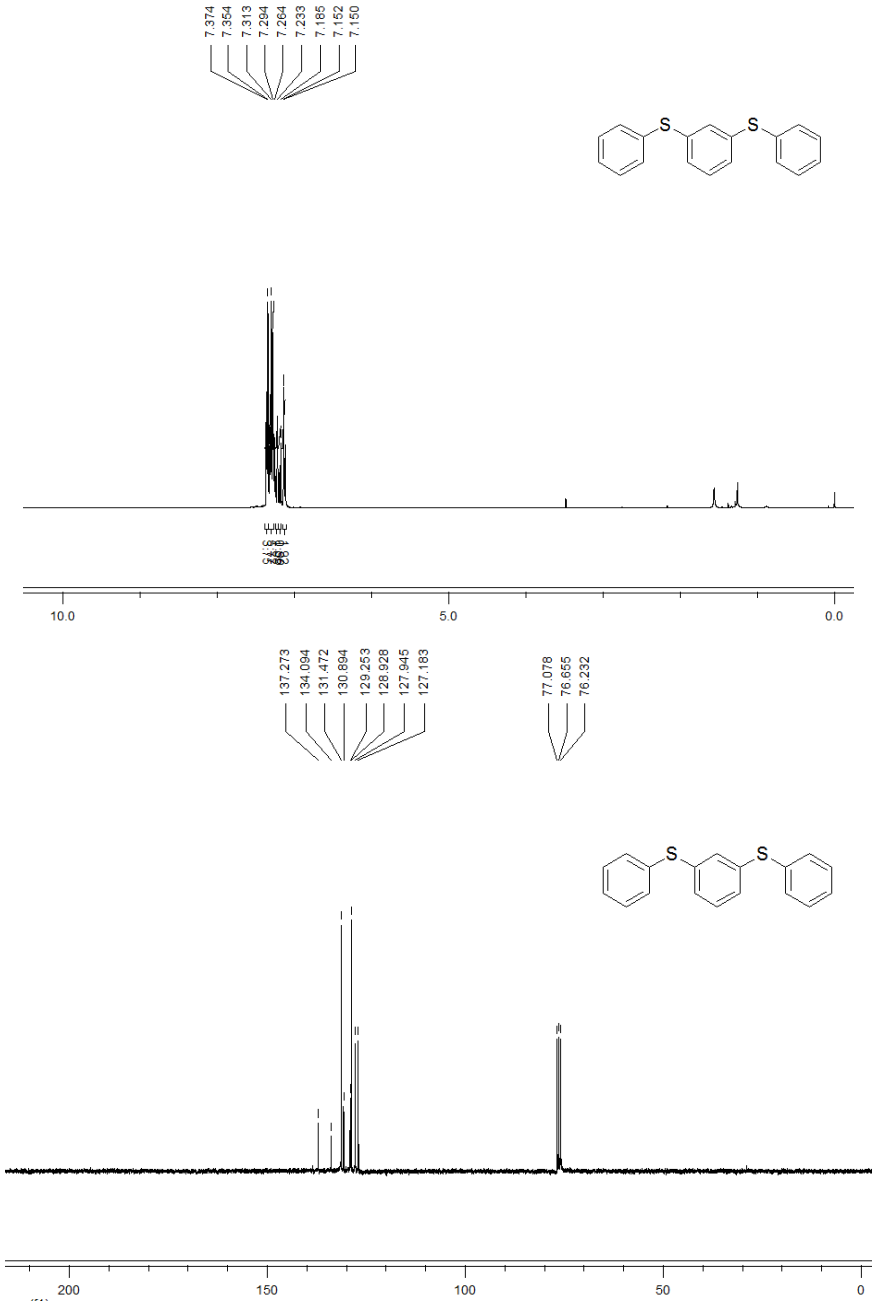

SUDA-TOFMS-EI-100715
QXM-5(0715) 792 (13.201) Cm (791:792-(545:566+2:26))
TOF MS EI+
1.72e3
406.0528
408.0540
409.0544
410.0509
411.0581
420.0190
372.0930
326.0586
297.0502
295.0560
269.0724
267.0677
244.0871
215.0909
216.0975
203.0361
202.0774
152.0626
139.0572
111.0036
99.0020
75.0282
%
m/z

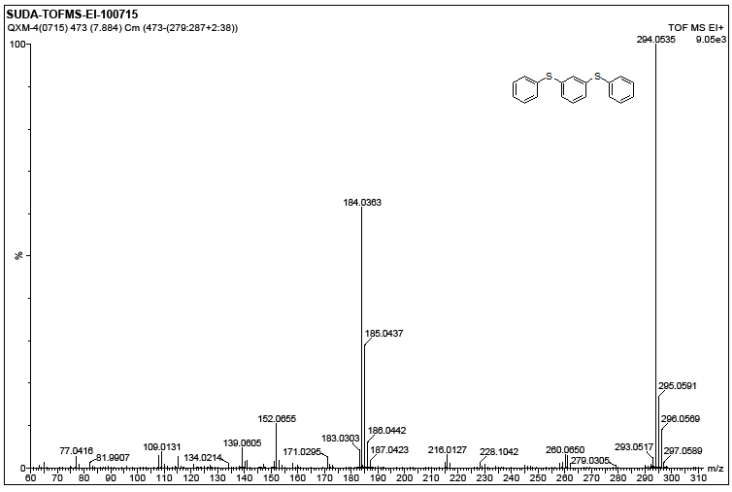


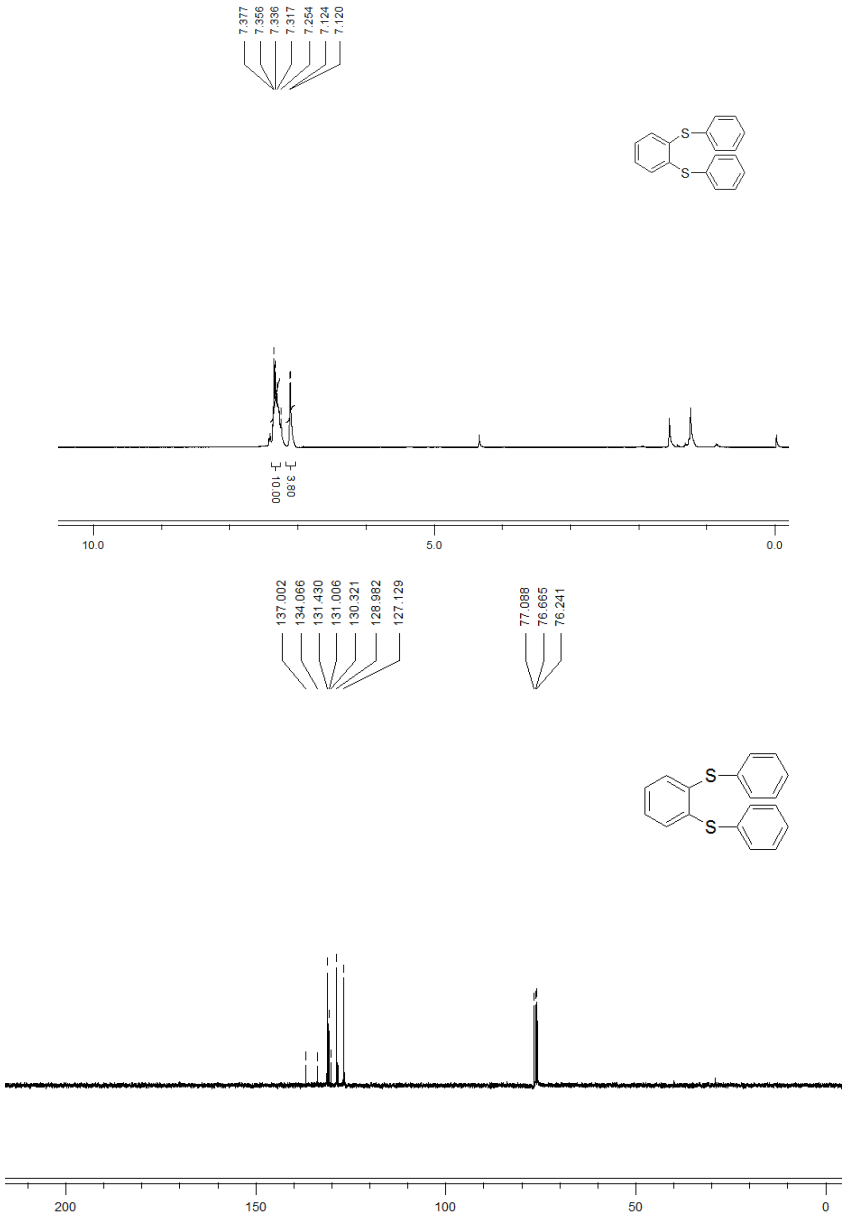


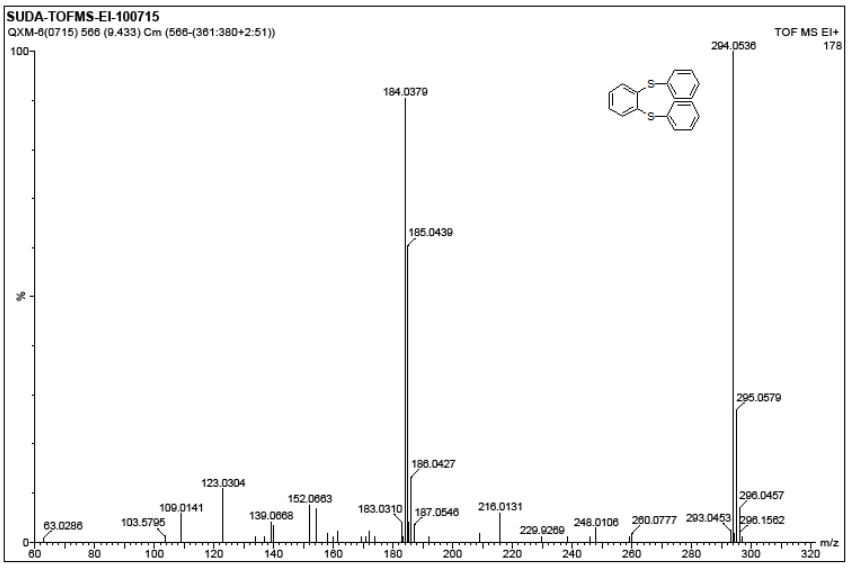

7.385
7.367
7.346
7.329
7.310
7.292
7.275
7.235
1.572
S
S
10.0
5.0
0.0
136.679
136.556
133.122
132.827
130.994
129.085
79.074
78.757
78.440
S
S
200
150
100
50
0

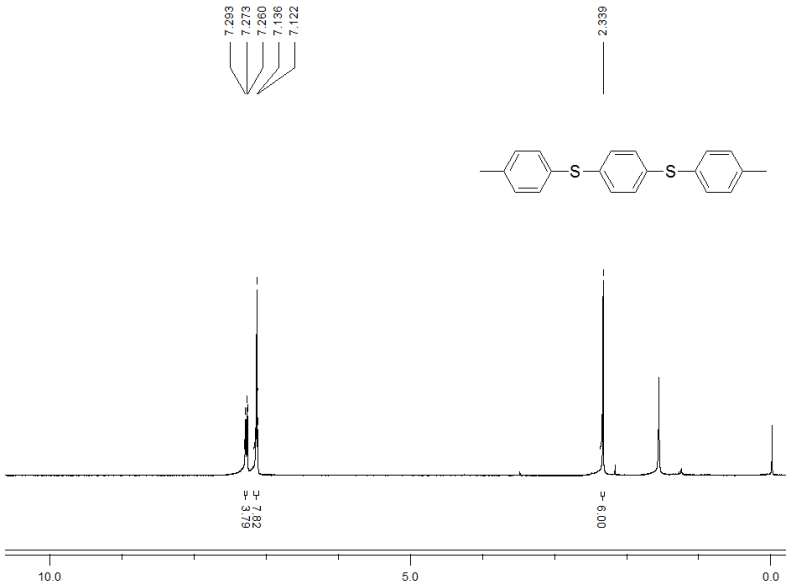

SUDA-TOFMS-EI-100611
qxm-1 968 (16.135) Cm (968-(573:611+37:93))
TOF MS EI+
294.0536 2.13e3
100
%
S
S
185.0435
184.0376
295.0558
296.0538
186.0434
152.0646
297.0544
108.0065
139.0565
183.0276
187.0433
261.0796
63.0288
80.0272
115.0551
171.0307
189.9973
217.0173
228.1008
293.0309
0
60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310
m/z

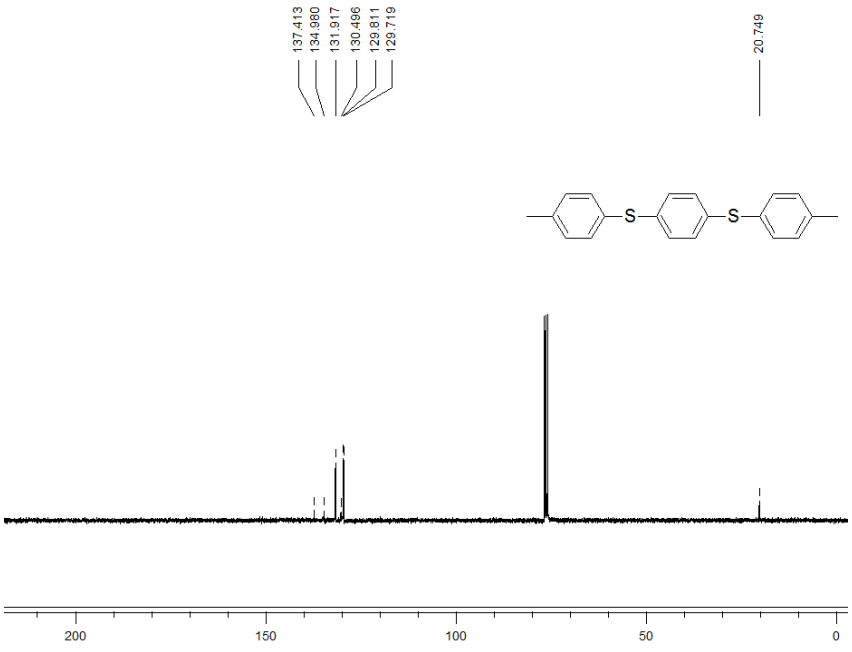

7.374
7.354
7.313
7.294
7.264
7.233
7.185
7.152
7.150
S
S
10.0
5.0
0.0
137.273
134.094
131.472
130.894
129.253
128.926
127.945
127.183
77.078
76.655
76.232
S
S
200
150
100
50
0

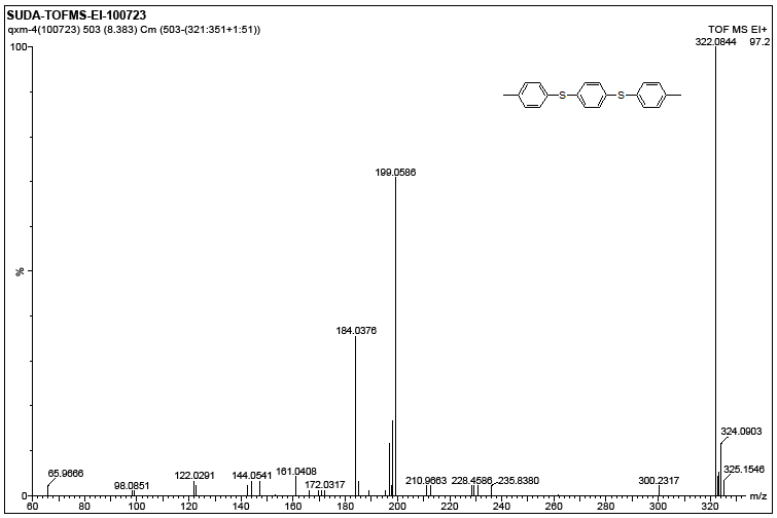

SUDA-TOFMS-EI-100715
QXM-4(0715) 473 (7.884) Cm (473-(279:287+2:38))
TOF MS EI+
294.0535 9.05e3
100
%
184.0363
185.0437
295.0591
296.0569
152.0655
186.0442
183.0303
139.0605
109.0131
77.0416
81.9907
134.0214
171.0295
187.0423
216.0127
228.1042
260.0650
279.0305
293.0517
297.0589
0
60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310
m/z

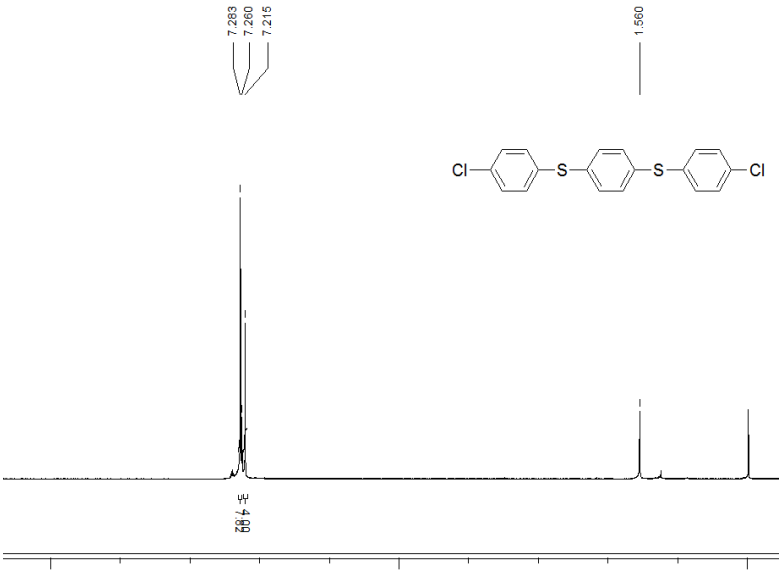

7.377
7.356
7.336
7.317
7.254
7.124
7.120
S
S
10.00
3.80
10.0
5.0
0.0
137.002
134.066
131.430
131.006
130.321
128.982
127.129
77.088
76.665
76.241
S
S
200
150
100
50
0

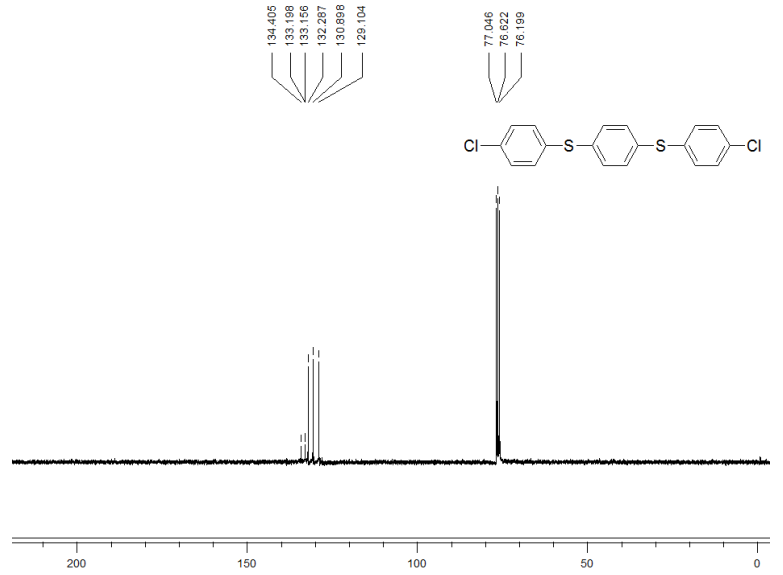

SUDA-TOFMS-EI-100715
QXM-6(0715) 566 (9.433) Cm (566-(361:380+2:51))
TOF MS EI+
178
294.0536
184.0379
185.0439
295.0579
186.0427
296.0457
123.0304
152.0663
109.0141
139.0668
183.0310
187.0546
216.0131
103.5795
63.0286
229.9269
248.0106
260.0777
293.0453
296.1562
%
m/z

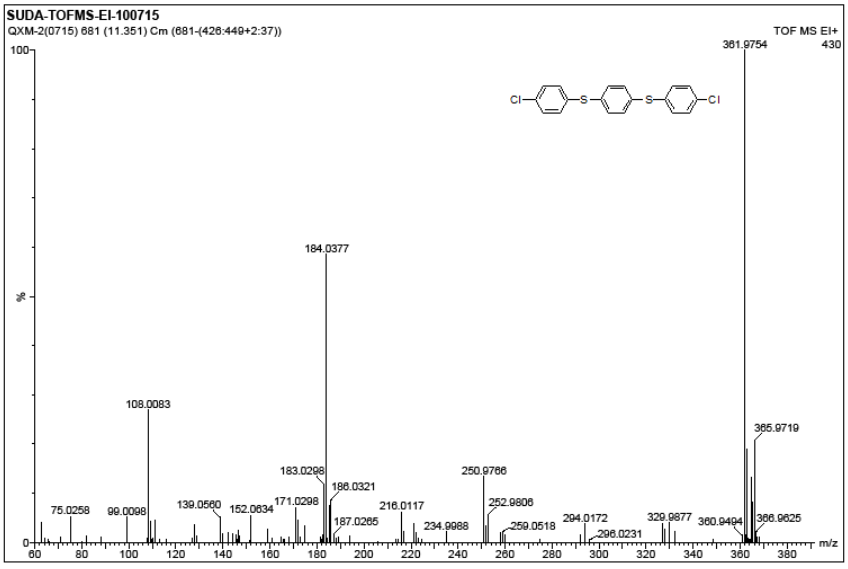

7.293
7.273
7.260
7.136
7.122
2.339
S
S
7.379
7.382
6.00
10.0
5.0
0.0

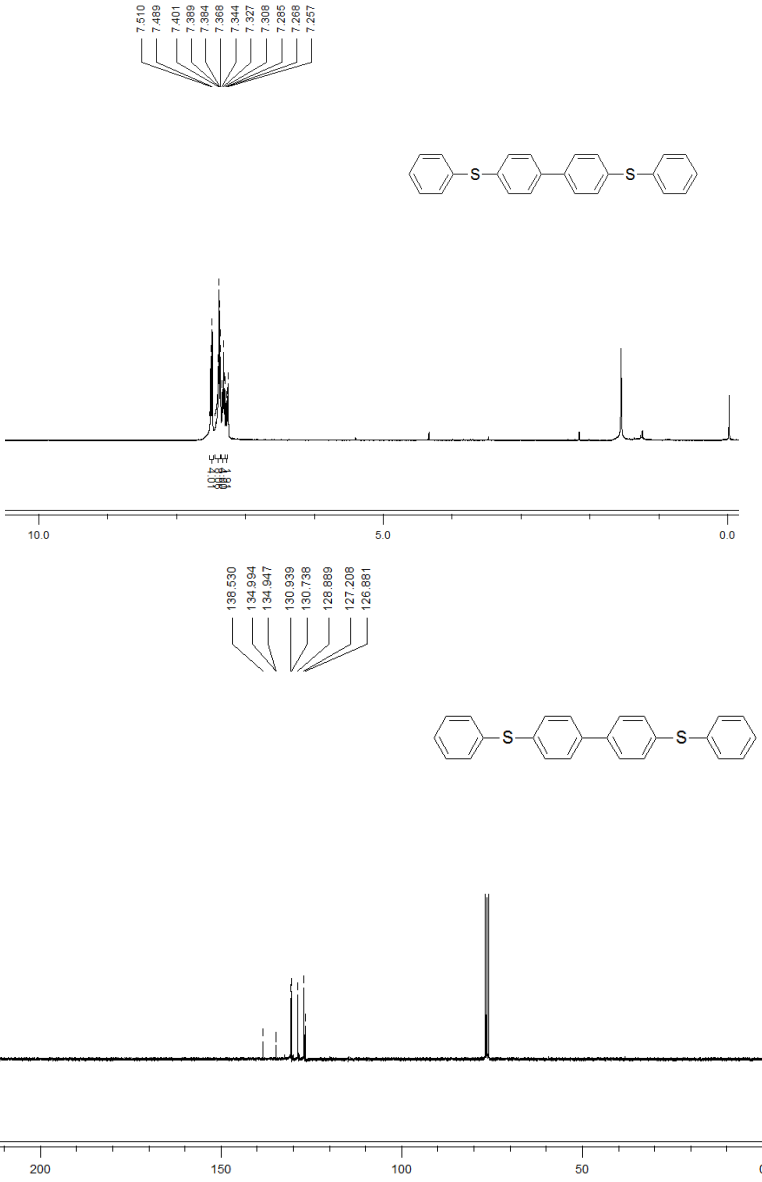

137.413
134.980
131.917
130.496
129.811
129.719
20.749
S
S
200
150
100
50
0

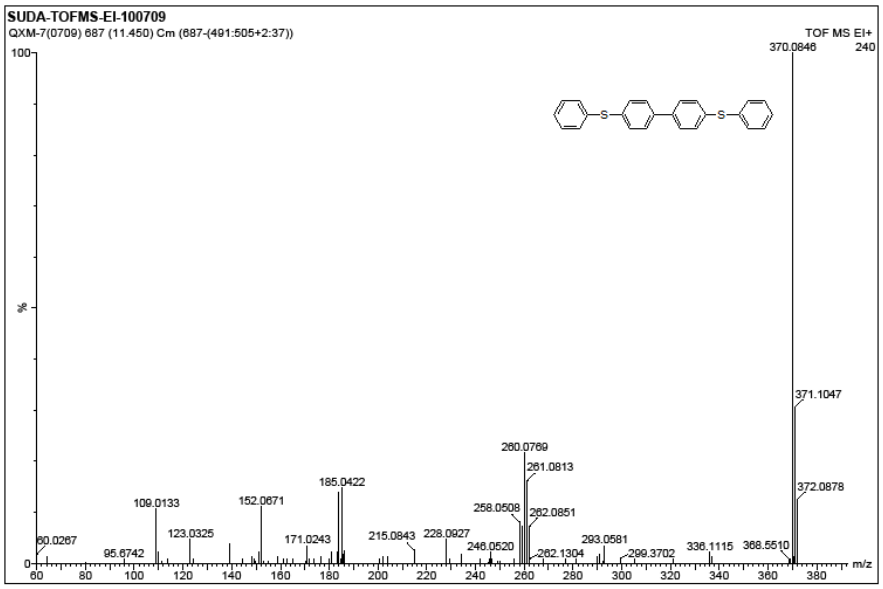

SUDA-TOFMS-EI-100723
qxm-4(100723) 503 (8.383) Cm (503-(321:351+1:51))
TOF MS EI+
322.0844 97.2
100
%
0
S
S
199.0586
184.0376
324.0903
325.1546
65.9666
98.0851
122.0291
144.0541
161.0408
172.0317
210.9863
228.4586
235.8380
300.2317
60
80
100
120
140
160
180
200
220
240
260
280
300
320
m/z


7.283
7.260
7.215
1.560
Cl
S
S
Cl
3.482
10.0
5.0
0.0

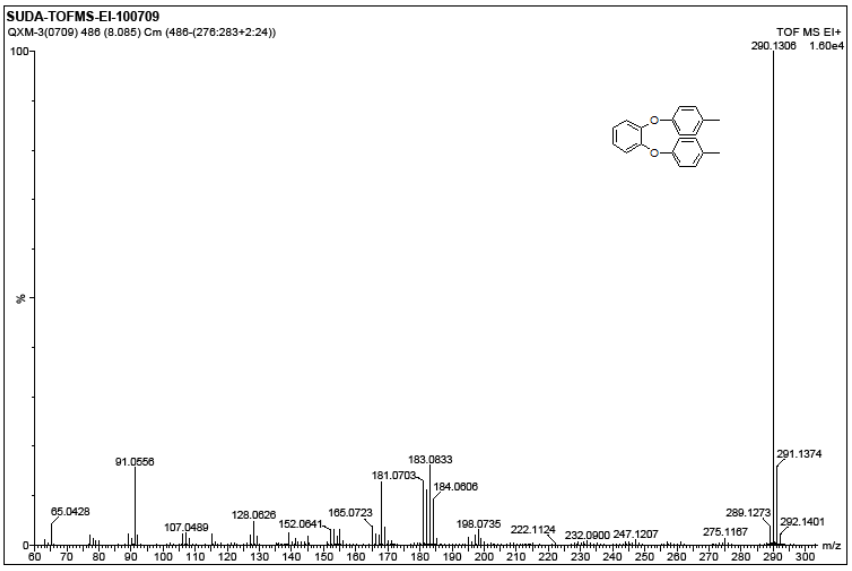

134.405
133.198
133.156
132.287
130.898
129.104
77.046
76.622
76.199
Cl
S
S
Cl
200
150
100
50
0

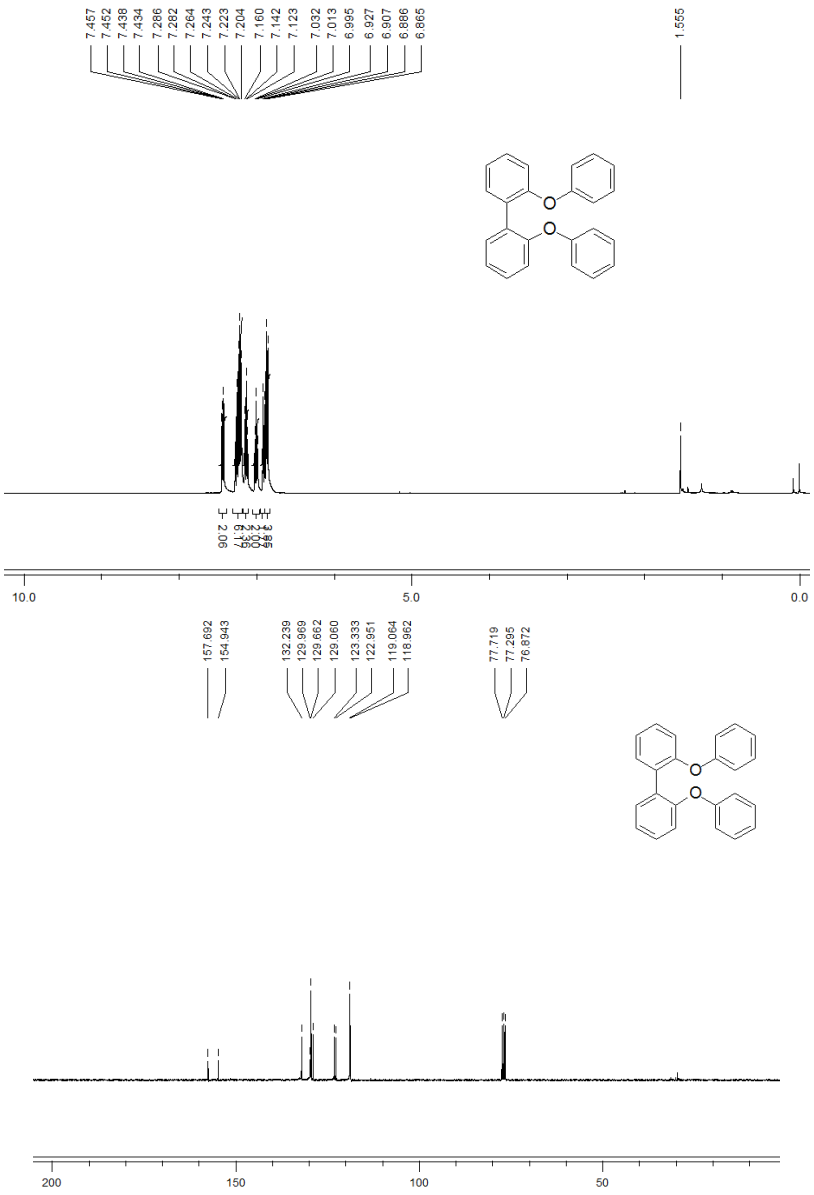

SUDA-TOFMS-EI-100715
QXM-2(0715) 681 (11.351) Cm (681-(426:449+2:37))
TOF MS EI+
361.9754
430
100
%
184.0377
108.0083
365.9719
183.0298
186.0321
250.9766
75.0258
99.0098
139.0560
152.0634
171.0298
216.0117
252.9806
187.0265
234.9988
259.0518
294.0172
329.9877
360.9494
366.9625
296.0231
0
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
m/z

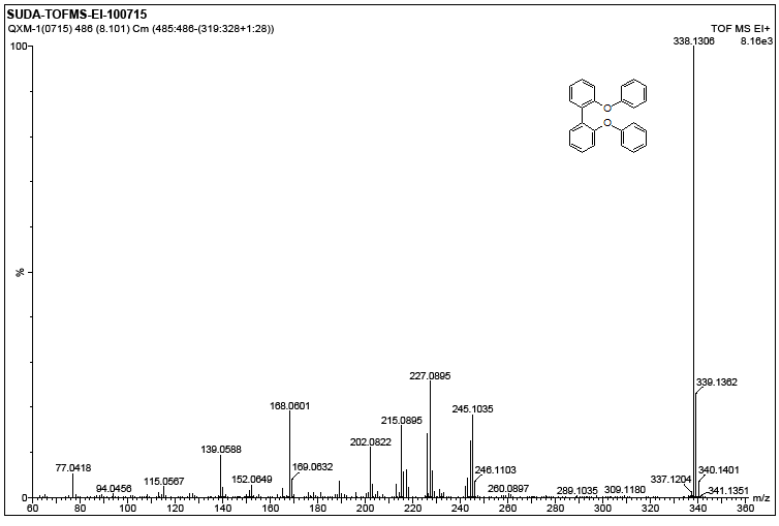

7.510
7.489
7.401
7.389
7.384
7.366
7.344
7.327
7.308
7.285
7.268
7.257
S
S
10.0
5.0
0.0
136.530
134.994
134.947
130.939
130.738
128.889
127.208
126.881
S
S
200
150
100
50
0

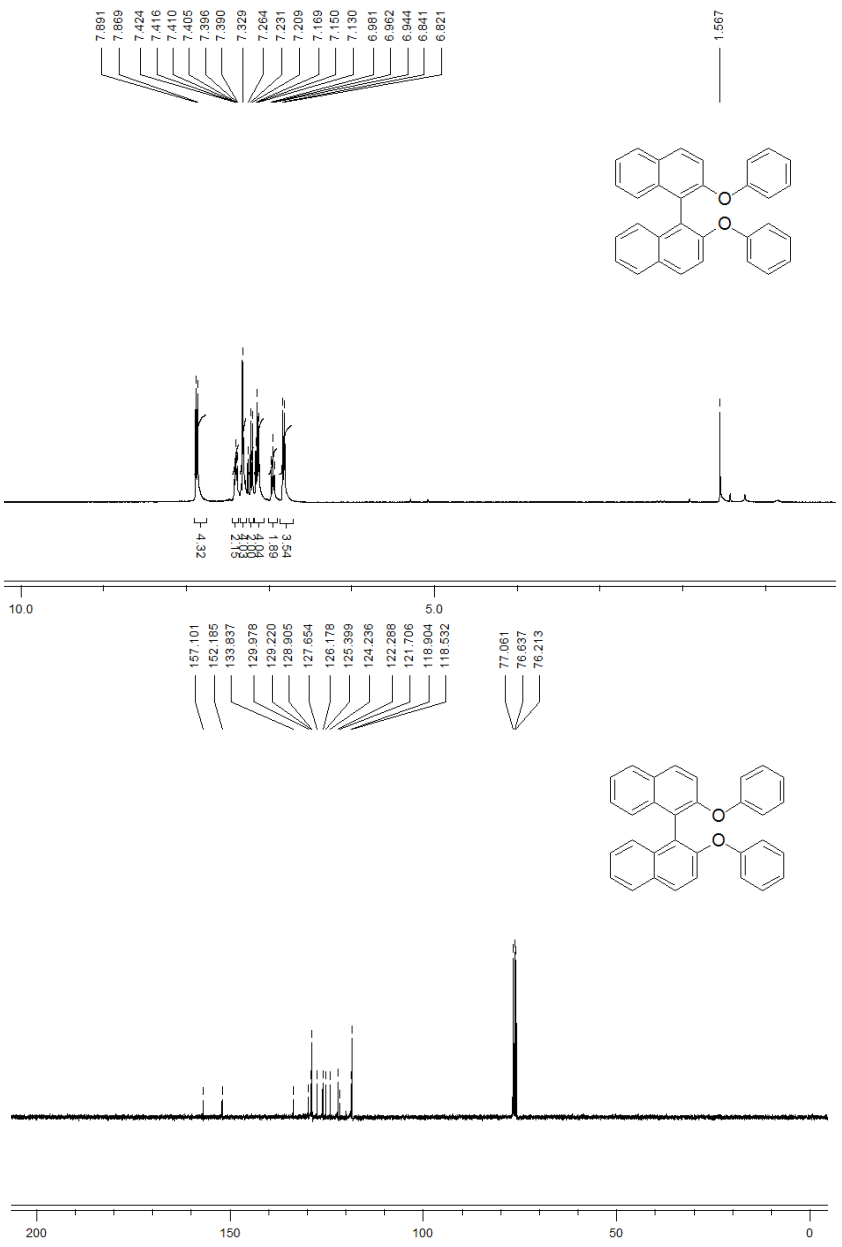

SUDA-TOFMS-EI-100709
QXM-7(0709) 687 (11.450) Cm (687-(491:505+2:37))
TOF MS EI+
370.0846
240
100
%
S
S
371.1047
260.0769
261.0813
372.0878
185.0422
109.0133
152.0671
258.0508
262.0851
123.0325
171.0243
215.0843
228.0927
293.0581
60.0267
95.6742
246.0520
262.1304
299.3702
336.1115
368.5510
0
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
m/z

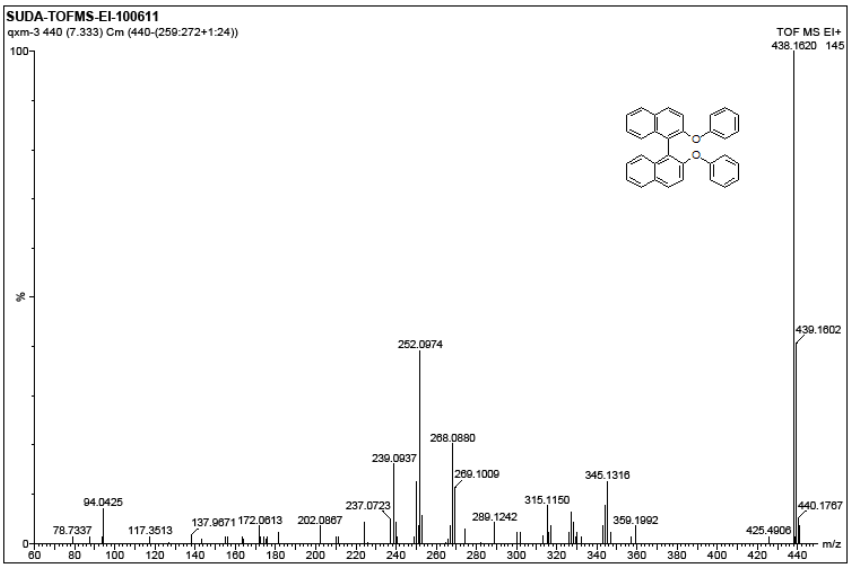

7.131
7.110
7.084
7.066
6.900
6.879
2.342
1.605
8.00
4.00
6.16
10.0
5.0
0.0
156.822
149.780
133.992
131.652
125.877
122.550
119.514
79.029
78.711
78.393
22.323
200
150
100
50

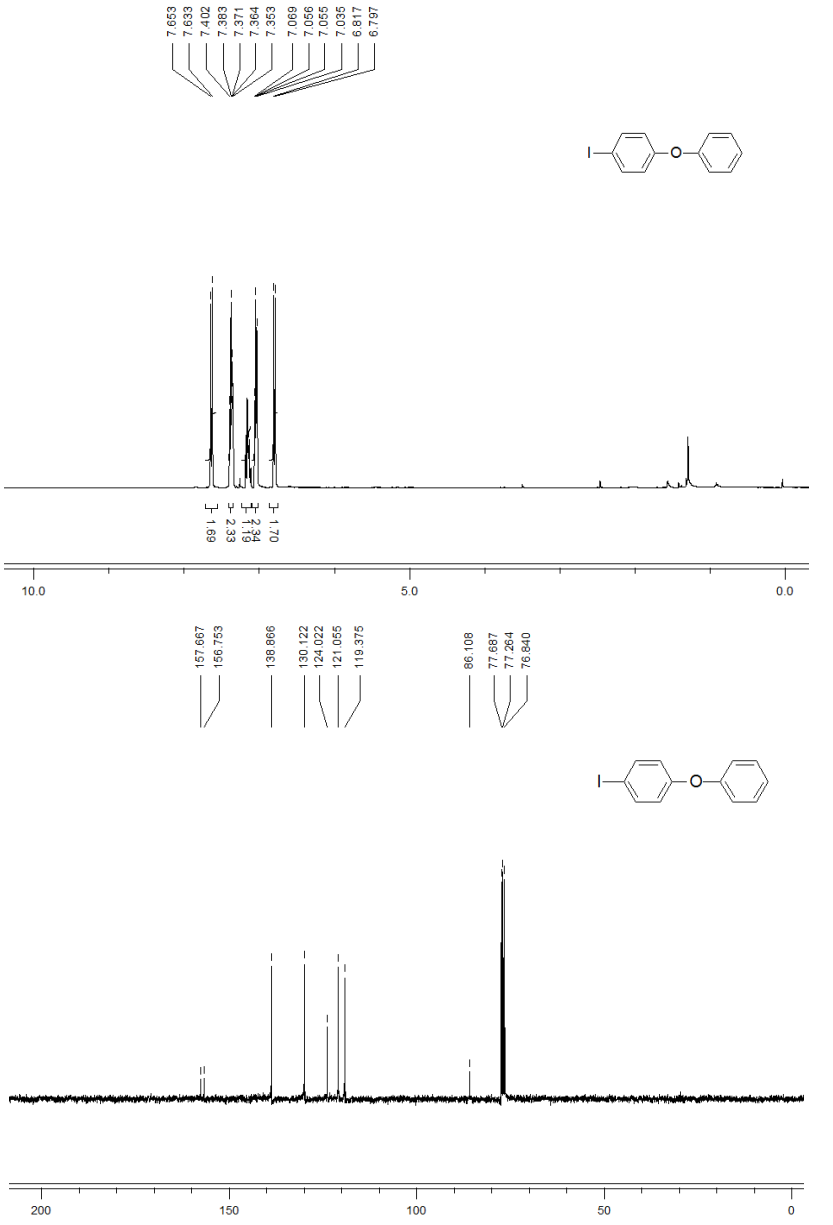

SUDA-TOFMS-EI-100709
QXM-3(0709) 486 (8.085) Cm (486-(276:283+2:24))
TOF MS EI+
290.1306 1.60e4
100
%
0
91.0556
183.0833
181.0703
184.0806
291.1374
65.0428
128.0626
107.0489
152.0641
165.0723
198.0735
222.1124
232.0900
247.1207
289.1273
275.1167
292.1401
m/z
60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300

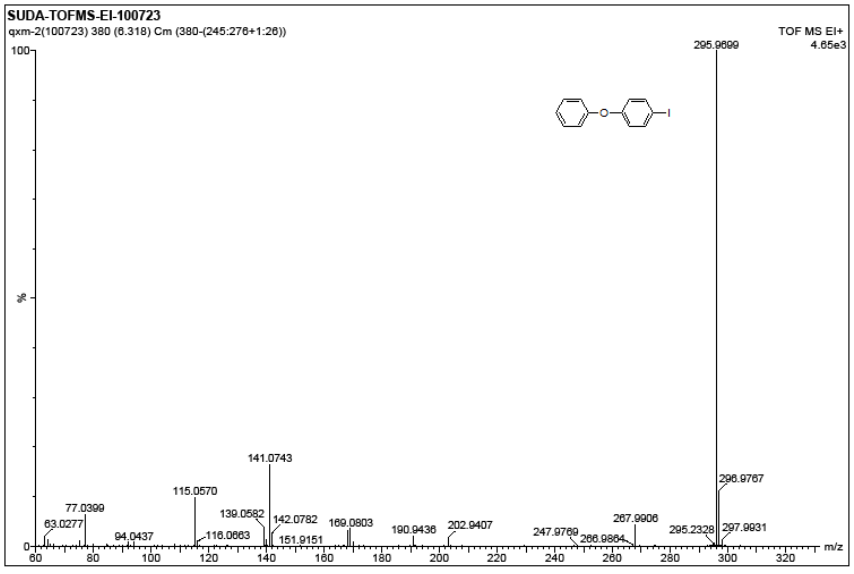


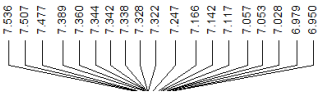


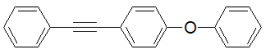

7.891
7.869
7.424
7.416
7.410
7.405
7.396
7.390
7.329
7.264
7.231
7.209
7.169
7.150
7.130
6.981
6.962
6.944
6.841
6.821
1.567
4.32
3.54
10.0
5.0
157.101
152.185
133.837
129.978
129.220
128.905
127.654
126.178
125.399
124.236
122.288
121.706
118.904
118.532
77.061
76.637
76.213
200
150
100
50
0

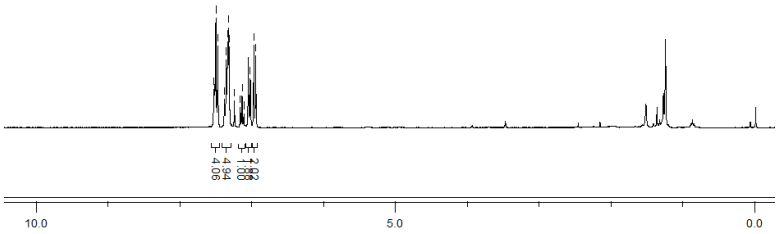

SUDA-TOFMS-EI-100611
qxm-3 440 (7.333) Cm (440-(259:272+1:24))
TOF MS EI+
438.1620 145
100
%
0
439.1602
252.0974
268.0880
239.0937
269.1009
345.1316
315.1150
94.0425
237.0723
289.1242
359.1992
440.1767
78.7337
117.3513
137.9671
172.0613
202.0867
425.4906
m/z
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440


7.653
7.633
7.402
7.383
7.371
7.364
7.353
7.069
7.056
7.055
7.035
6.817
6.797
I
O
1.69
2.33
2.24
1.69
1.70
10.0
5.0
0.0
157.667
156.753
138.866
130.122
124.022
121.055
119.375
86.108
77.687
77.264
76.840
I
O
200
150
100
50
0

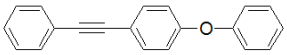

SUDA-TOFMS-EI-100723
qxm-2(100723) 380 (6.318) Cm (380-(245:276+1:26))
TOF MS EI+
4.65e3
100
%
0
295.9699
O
I
141.0743
296.9767
115.0570
77.0399
139.0582
142.0782
169.0803
63.0277
94.0437
116.0663
151.9151
190.9436
202.9407
247.9769
266.9864
267.9906
295.2328
297.9931
60
80
100
120
140
160
180
200
220
240
260
280
300
320
m/z

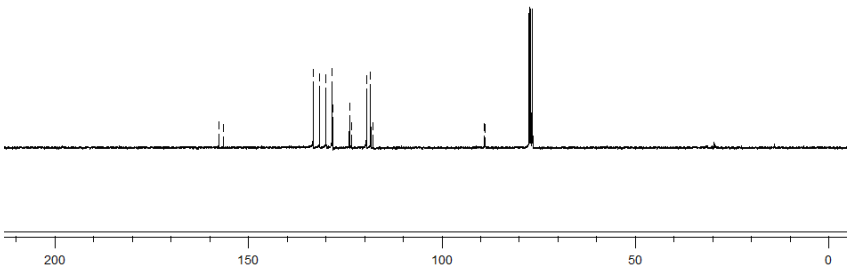

7.536
7.507
7.477
7.399
7.360
7.344
7.342
7.338
7.328
7.322
7.247
7.166
7.142
7.117
7.057
7.053
7.028
6.979
6.950

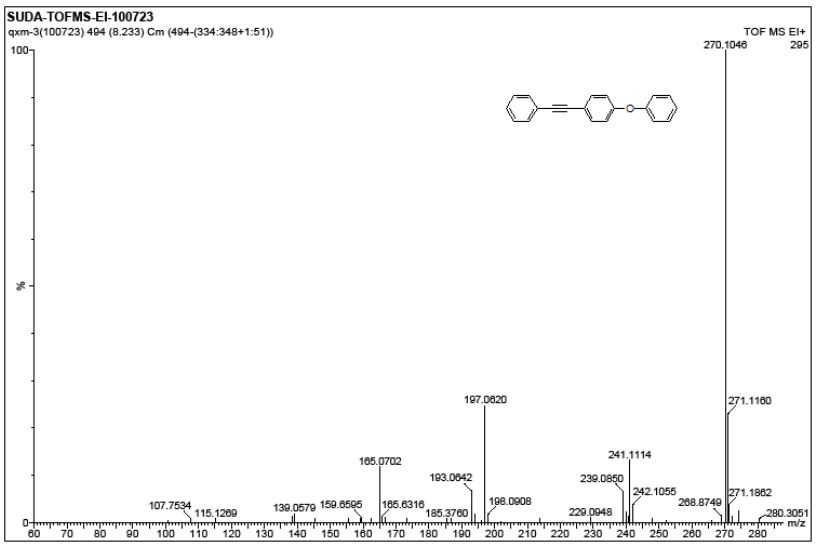



4.06
4.04
2.00
1.98
1.93
10.0
5.0
0.0


157.816
156.660
133.435
131.751
130.121
128.575
128.375
124.080
123.570
119.664
118.605
118.034
89.186
88.982




200
150
100
50
0


SUDA-TOFMS-EI-100723
qxm-3(100723) 494 (8.233) Cm (494-(334:348+1:51))
TOF MS EI+
295
270.1046
271.1160
197.0620
241.1114
165.0702
193.0642
239.0850
242.1055
271.1862
198.0908
107.7534
115.1269
139.0579
159.8595
165.6316
185.3760
229.0948
268.8749
280.3051
m/z
%

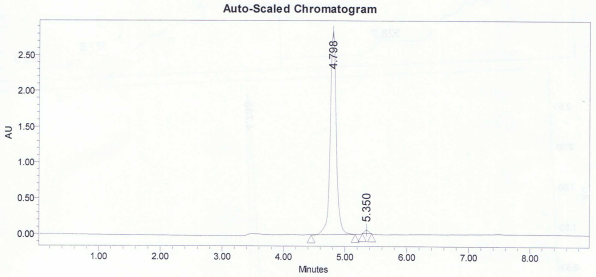
Empower 2 software

zyw

SAMPLE INFORMATION

Sample Name:	qxm-1	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	Untitled
Injection #:	3	Processing Method:	1111
Injection Volume:	10.00 ul	Channel Name:	221.0nm
Run Time:	30.0 Minutes	Proc. Chnl. Descr.:	PDA 221.0 nm
Date Acquired:	2010-6-29 20:03:09 CST		
Date Processed:	2010-6-29 20:13:46 CST		

Auto-Scaled Chromatogram



Peak Results

	Name	RT	Area	% Area	Height	Amount	Units
1		4.798	19664091	98.85	2861936		
2		5.350	228629	1.15	47891		

Reported by User: System
Report Method: zyw
Report Method ID 1398
Page: 1 of 2

Project Name: zyw
Date Printed:
2010-6-29
20:15:09 PRC

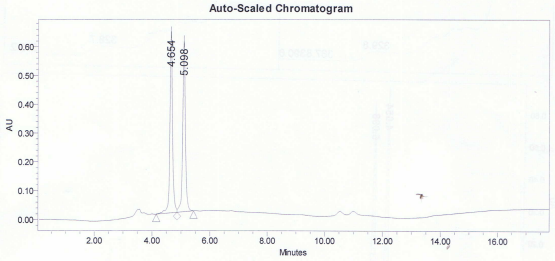
Empower 2 software

zyw

SAMPLE INFORMATION

Sample Name:	qxm-rac	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	
Vial:	1	Acq. Method Set:	Untitled
Injection #:	1	Processing Method:	111
Injection Volume:	10.00 ul	Channel Name:	221.0nm
Run Time:	60.0 Minutes	Proc. Chnl. Descr.:	PDA 221.0 nm
Date Acquired:	2010-6-29 19:35:20 CST		
Date Processed:	2010-6-29 20:11:49 CST		

Auto-Scaled Chromatogram



Peak Results

	Name	RT	Area	% Area	Height	Amount	Units
1		4.654	3910513	50.51	630843		
2		5.098	3830970	49.49	590307		

Reported by User: System
Report Method: zyw
Report Method ID 1398
Page: 1 of 2

Project Name: zyw
Date Printed:
2010-6-29
20:14:14 PRC