**Supporting Information for**

# *Meta*-Arylation of Calixarenes using Organomercurial Chemistry

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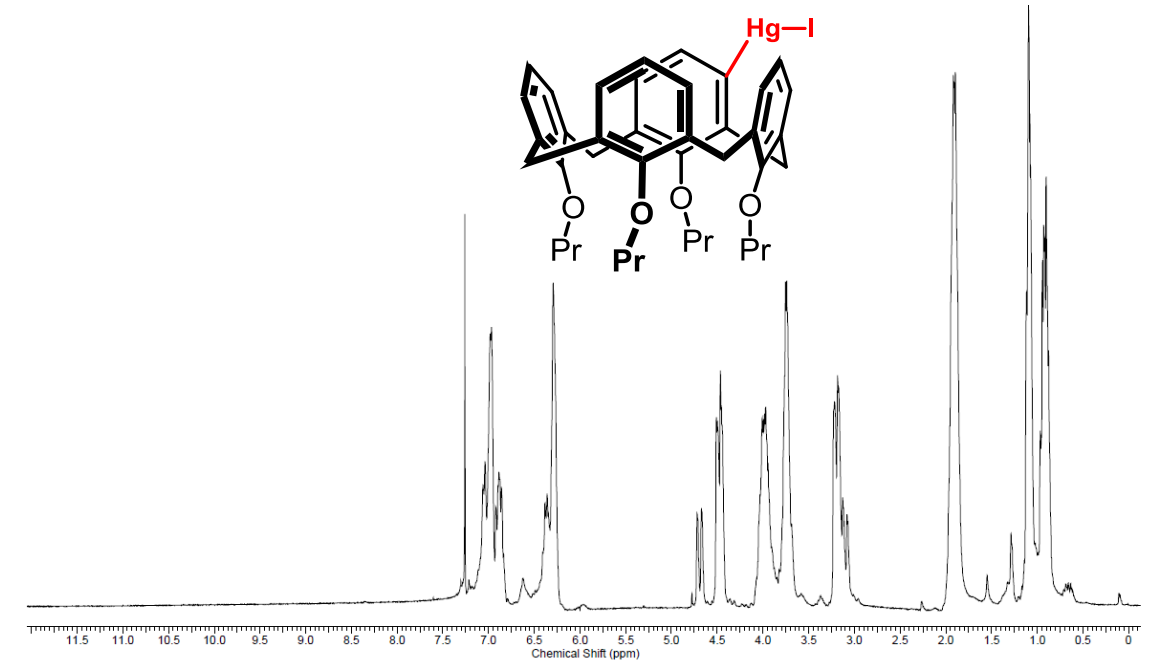
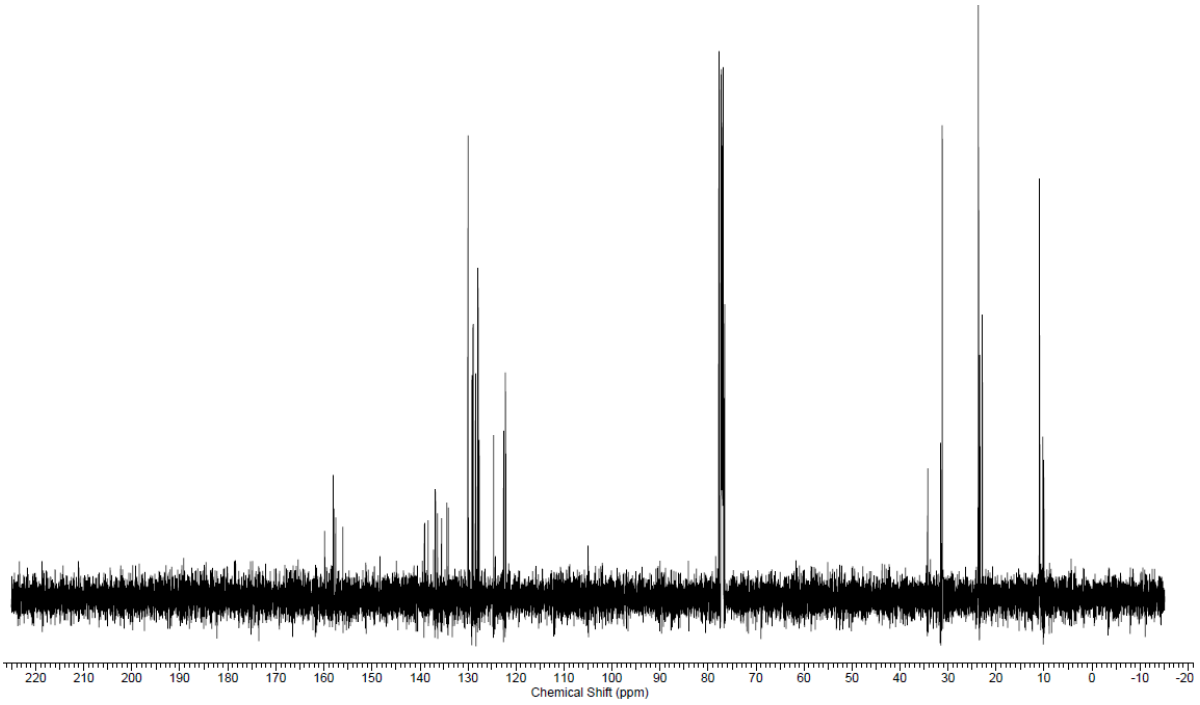
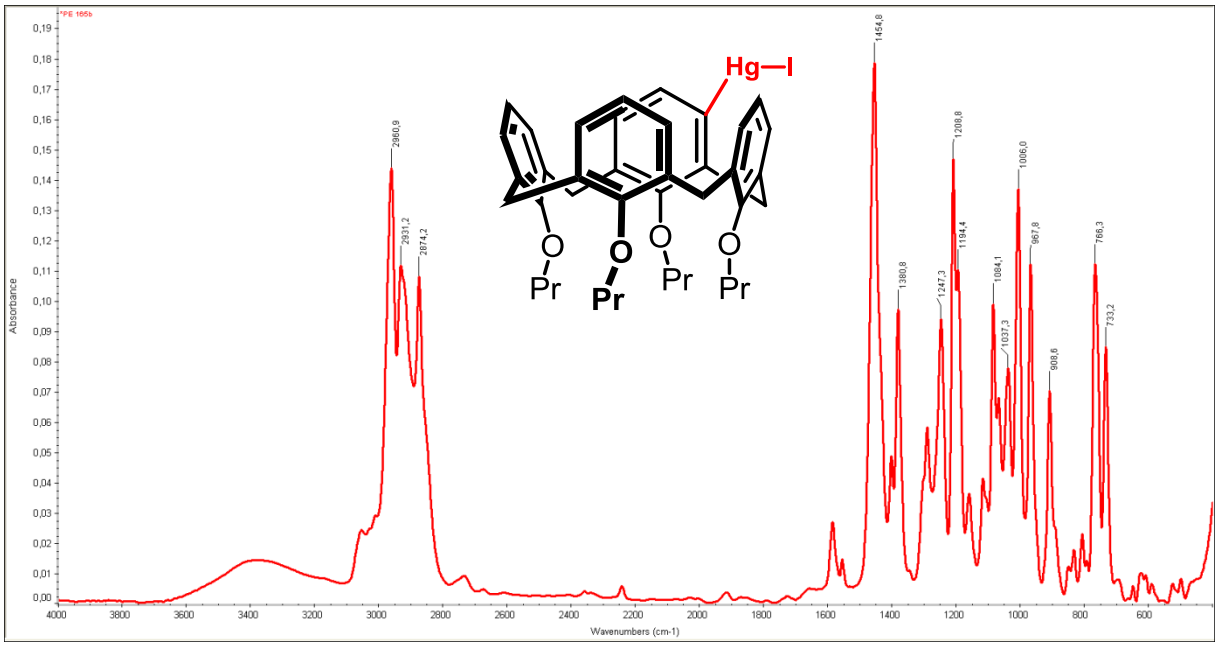
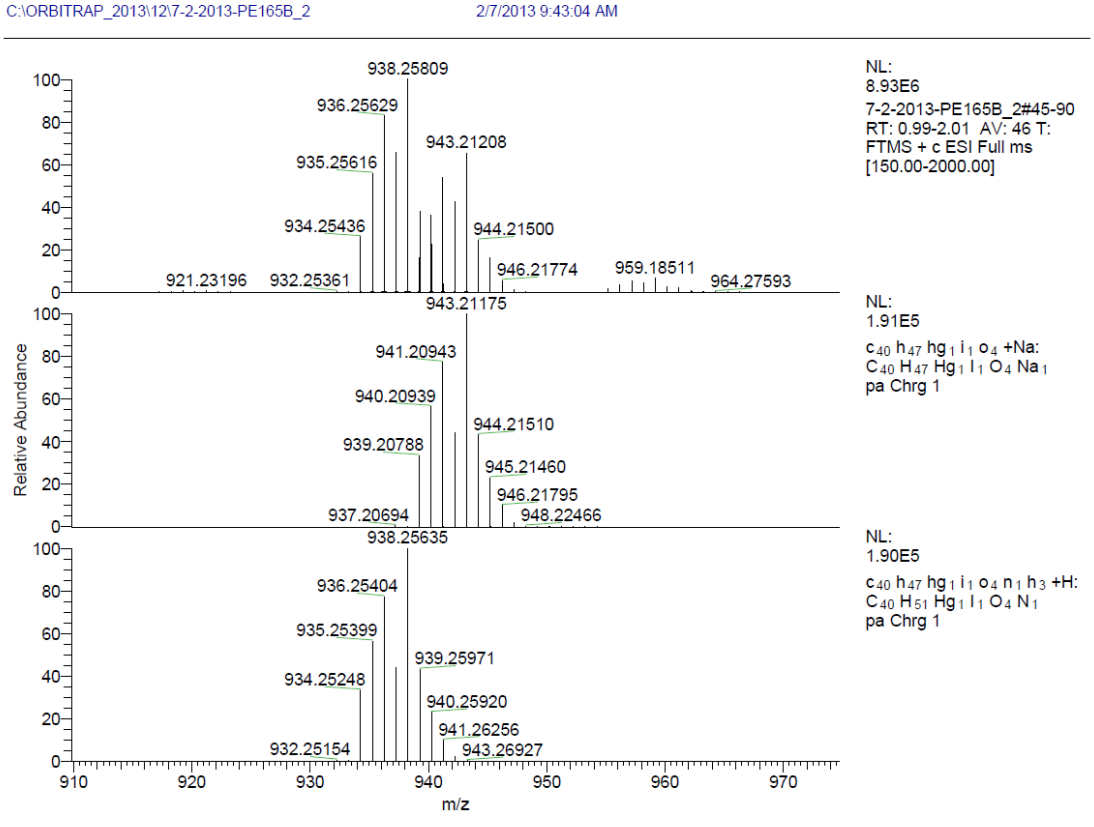
**Compound 3b**



## Compound 3b


Hg—I
O
Pr
O
Pr
O
Pr
O
Pr
Absorbance
Wavenumbers (cm-1)
2960,9
2931,2
2874,2
1454,8
1380,8
1247,3
1208,8
1194,4
1084,1
1066,0
967,8
766,3
733,2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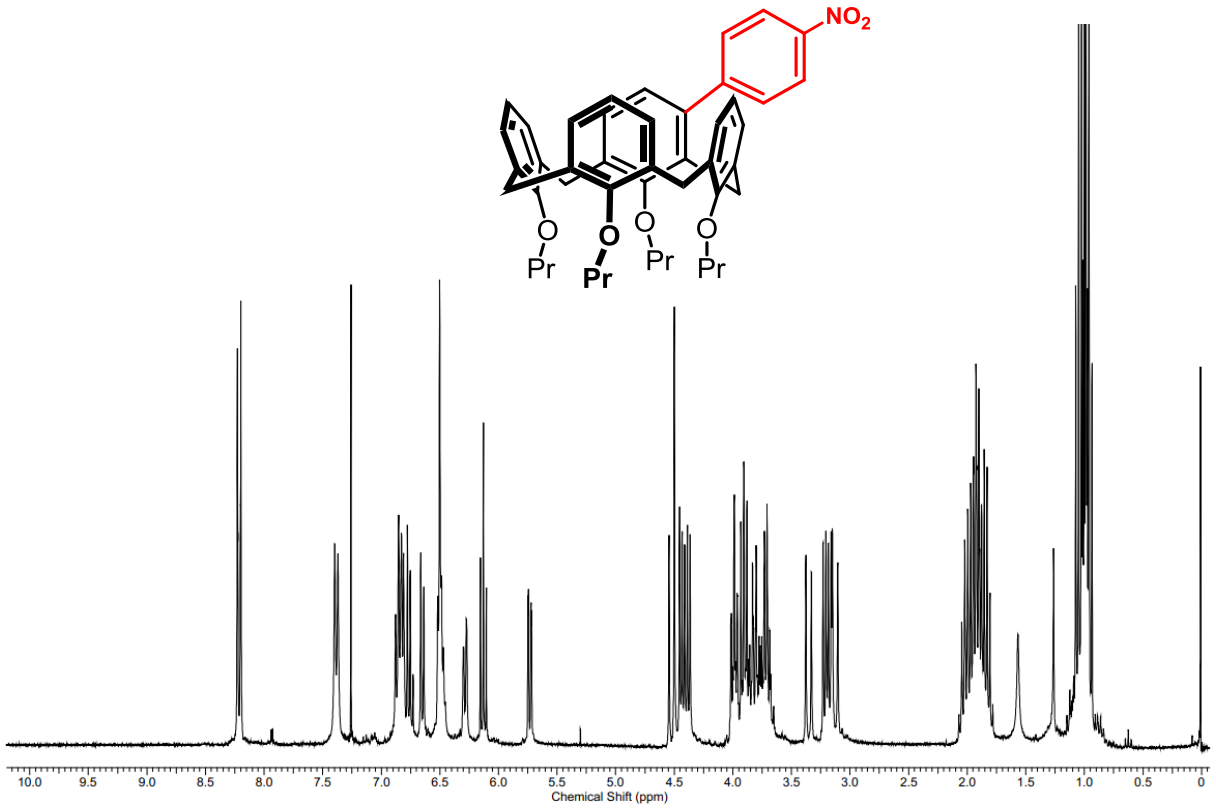
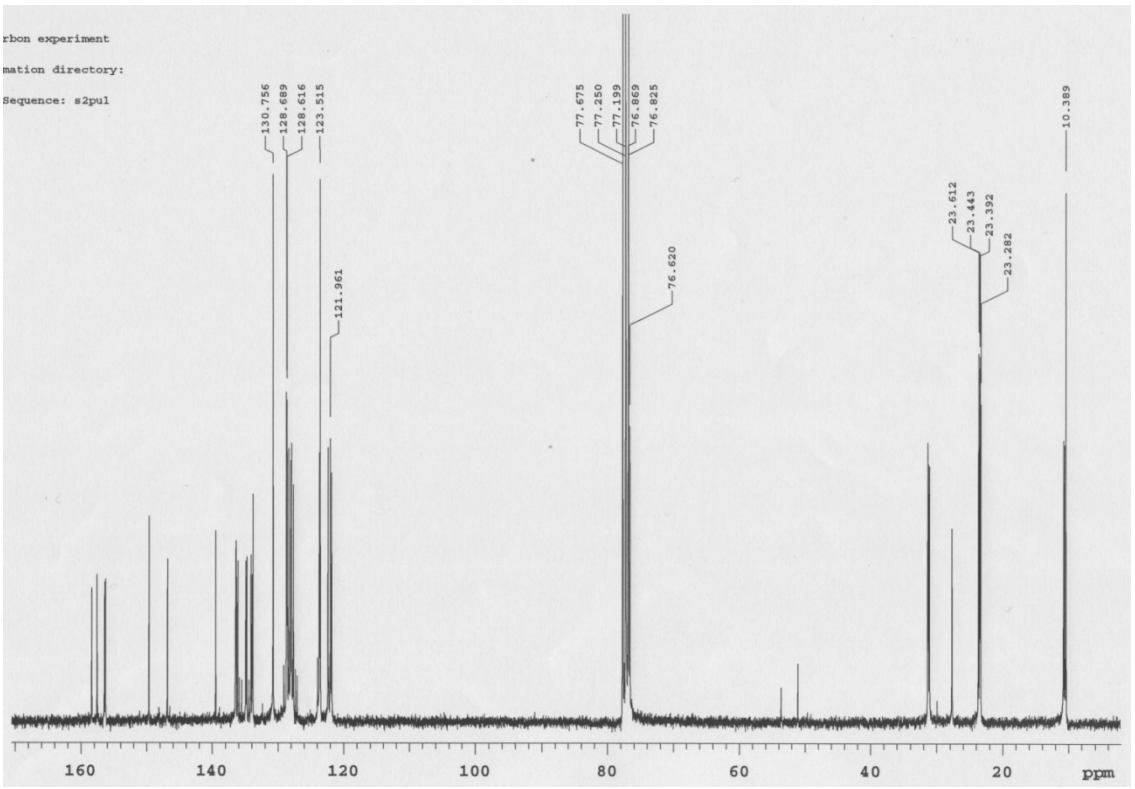
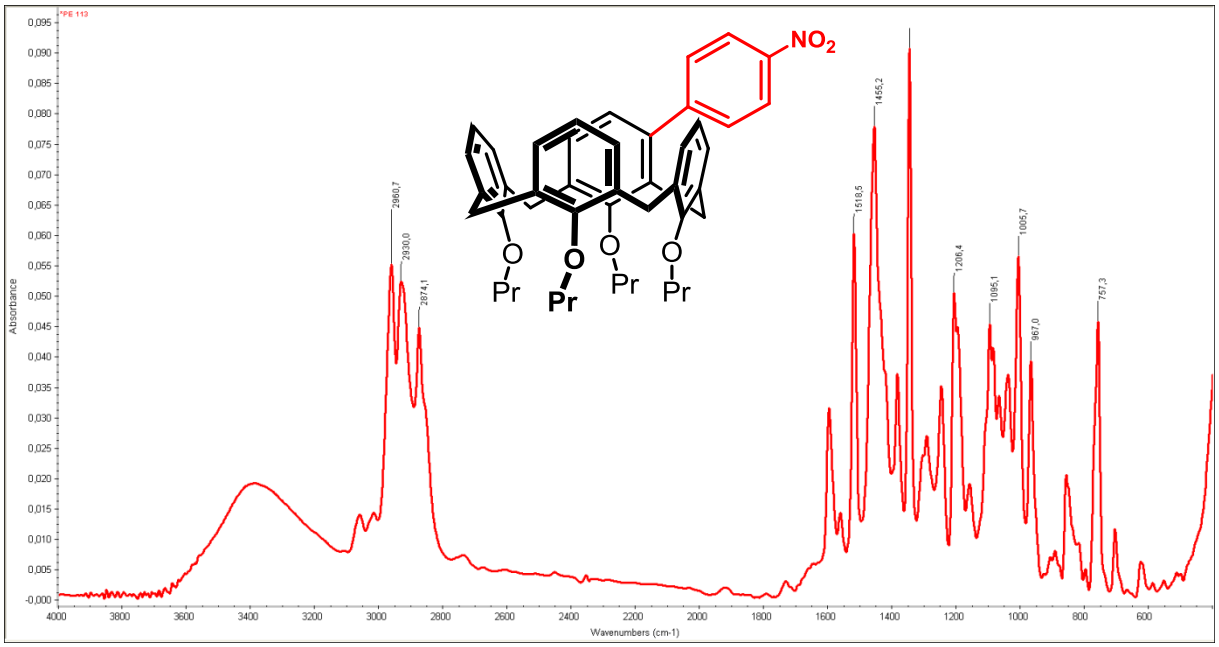
938.25809
936.25629
943.21208
935.25616
934.25436
944.21500
921.23196
932.25361
946.21774
959.18511
964.27593
NL:
8.93E6
7-2-2013-PE165B_2#45-90
RT: 0.99-2.01 AV: 46 T:
FTMS + c ESI Full ms
[150.00-2000.00]
943.21175
941.20943
940.20939
944.21510
939.20788
945.21460
946.21795
937.20694
948.22466
NL:
1.91E5
c40 h47 hg1 i1 o4 +Na:
C40 H47 Hg1 I1 O4 Na1
pa Chrg 1
938.25635
936.25404
935.25399
939.25971
934.25248
940.25920
941.26256
932.25154
943.26927
NL:
1.90E5
c40 h47 hg1 i1 o4 n1 h3 +H:
C40 H51 Hg1 I1 O4 N1
pa Chrg 1
Relative Abundance
m/z

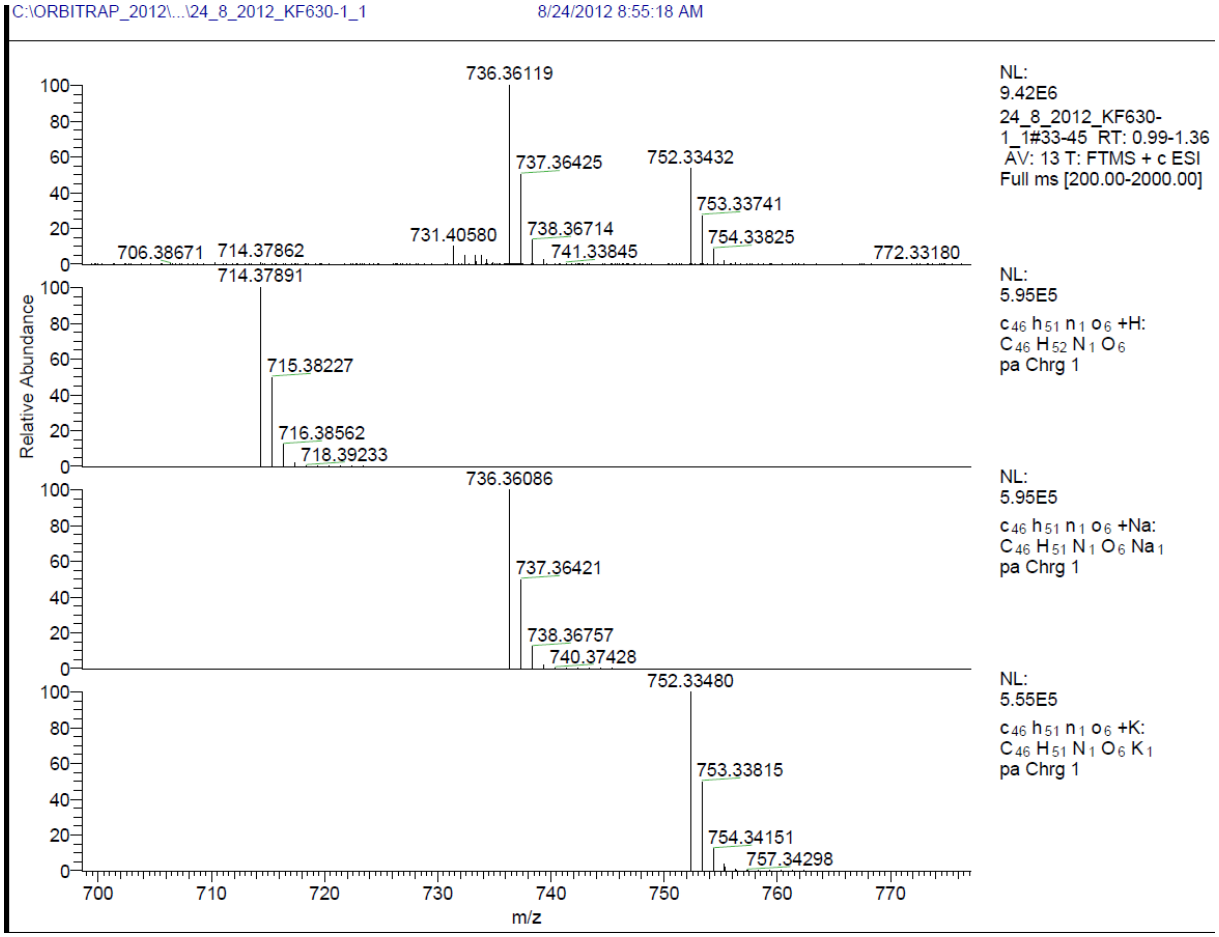
**Compound 5a**



## Compound 5a


NO2
O
Pr
O
Pr
O
Pr
O
Pr
2960,7
2930,0
2874,1
1518,5
1455,2
1206,4
1095,1
1005,7
967,0
757,3
Absorbance
Wavenumbers (cm-1)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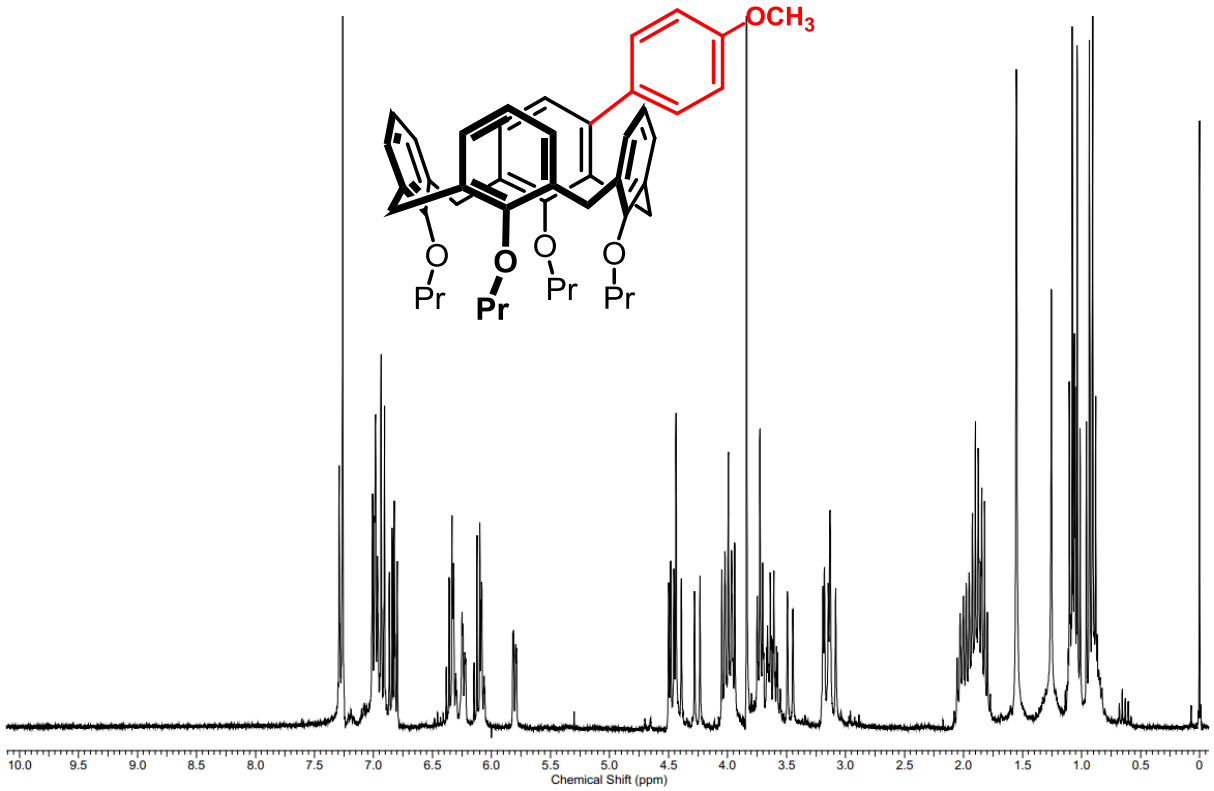
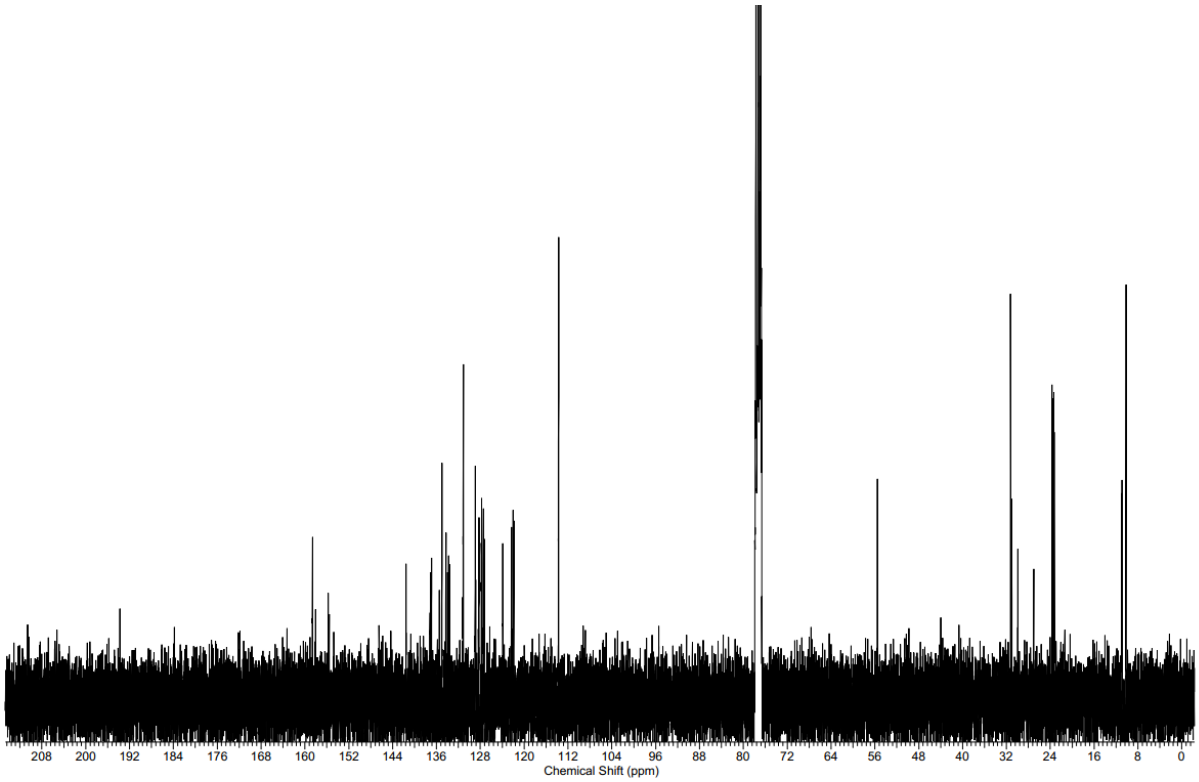
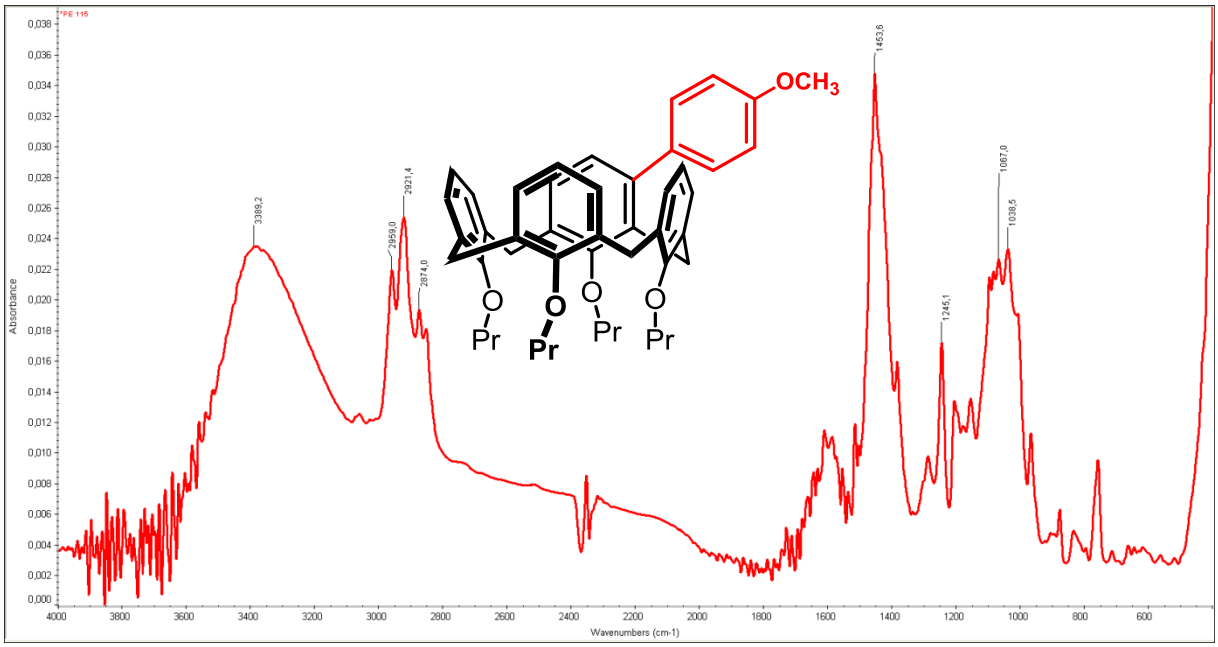
8/24/2012 8:55:18 AM
736.36119
737.36425
752.33432
753.33741
731.40580
738.36714
754.33825
706.38671
714.37862
741.33845
772.33180
NL:
9.42E6
24_8_2012_KF630-1_1#33-45 RT: 0.99-1.36 AV: 13 T: FTMS + c ESI Full ms [200.00-2000.00]
714.37891
715.38227
716.38562
718.39233
NL:
5.95E5
C46 H51 N1 O6 +H:
C46 H52 N1 O6
pa Chrg 1
736.36086
737.36421
738.36757
740.37428
NL:
5.95E5
C46 H51 N1 O6 +Na:
C46 H51 N1 O6 Na1
pa Chrg 1
752.33480
753.33815
754.34151
757.34298
NL:
5.55E5
C46 H51 N1 O6 +K:
C46 H51 N1 O6 K1
pa Chrg 1
Relative Abundance
m/z

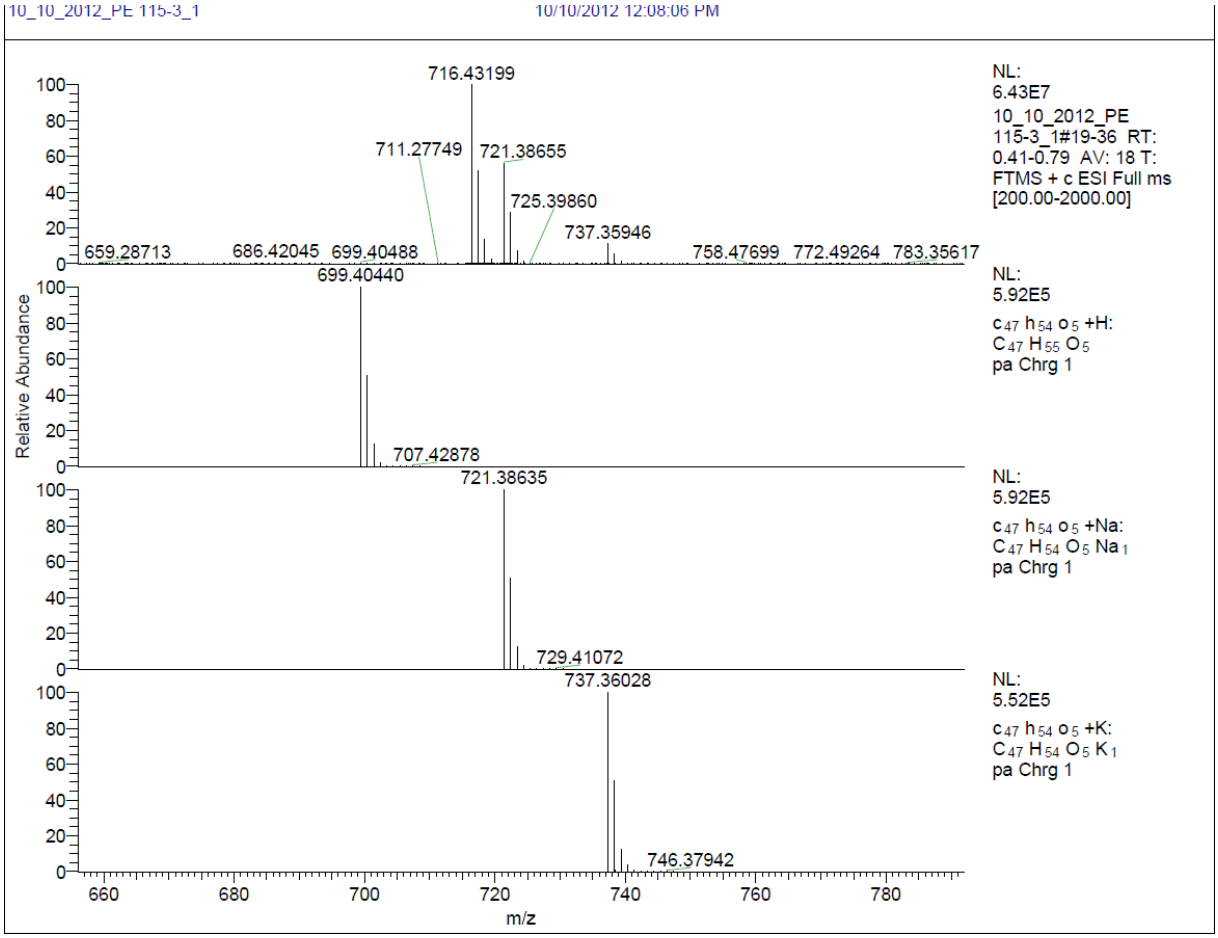
**Compound 5b**



## Compound 5b


PE 115
Absorbance
Wavenumbers (cm-1)
3389,2
2959,0
2921,4
2874,0
1453,6
1245,1
1067,0
1038,5
OCH3
O
Pr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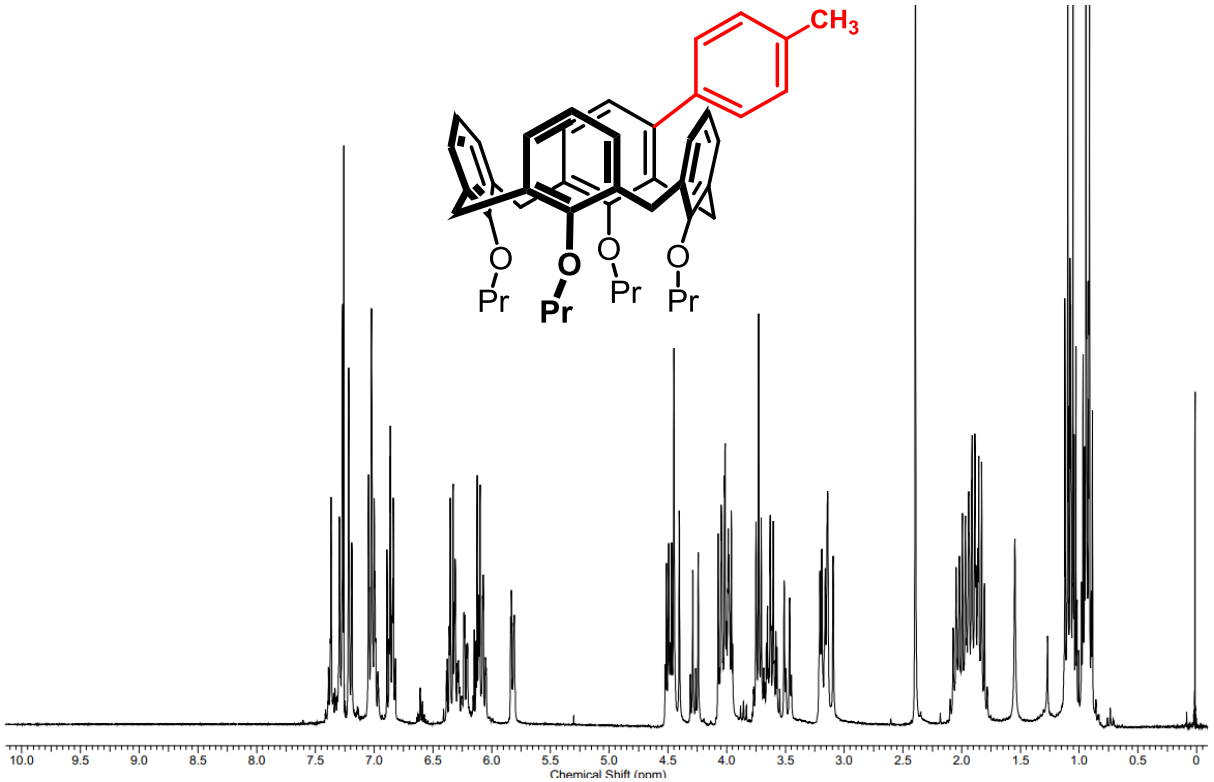
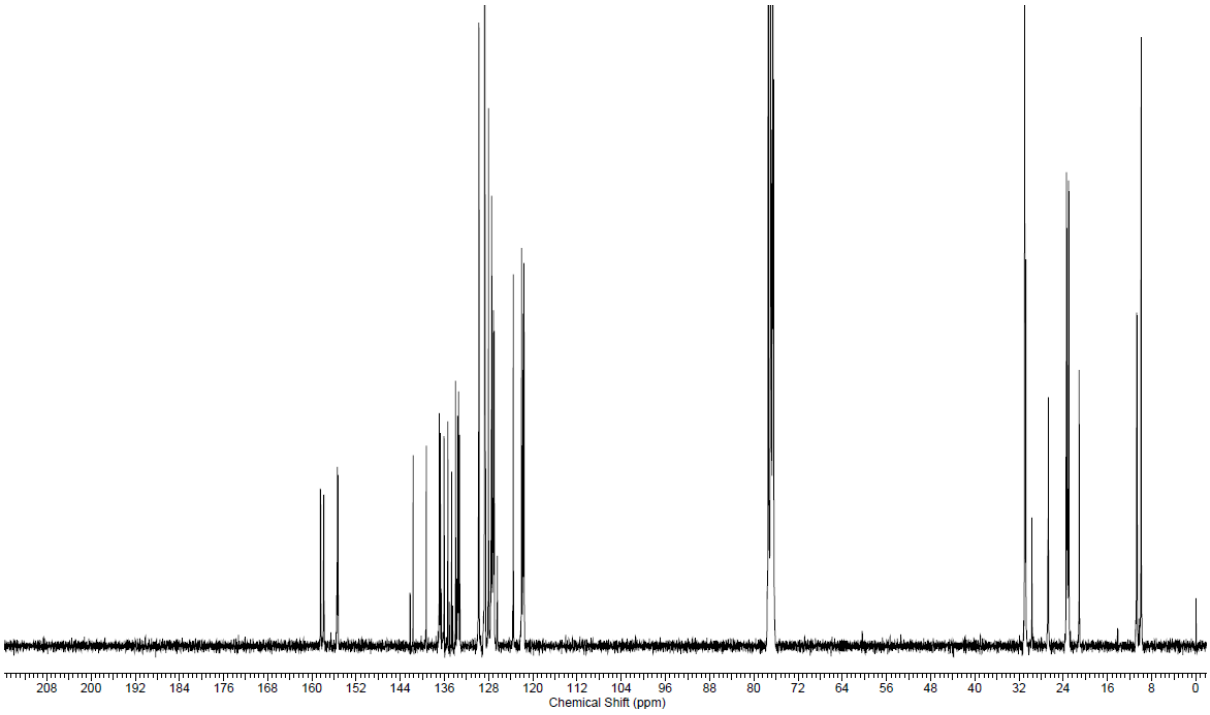
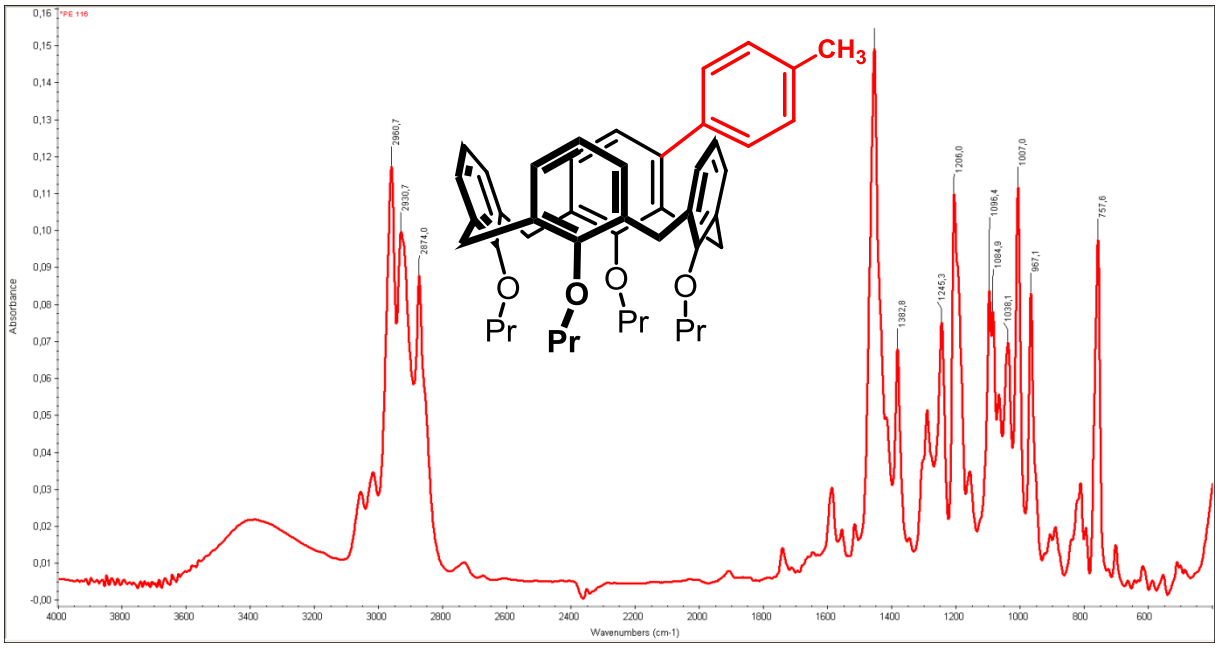
716.43199
711.27749
721.38655
725.39860
737.35946
659.28713
686.42045
699.40488
758.47699
772.49264
783.35617
NL:
6.43E7
10_10_2012_PE
115-3_1#19-36 RT:
0.41-0.79 AV: 18 T:
FTMS + c ESI Full ms
[200.00-2000.00]
699.40440
707.42878
NL:
5.92E5
C47 H54 O5 +H:
C47 H55 O5
pa Chrg 1
721.38635
729.41072
NL:
5.92E5
C47 H54 O5 +Na:
C47 H54 O5 Na1
pa Chrg 1
737.36028
746.37942
NL:
5.52E5
C47 H54 O5 +K:
C47 H54 O5 K1
pa Chrg 1
Relative Abundance
m/z

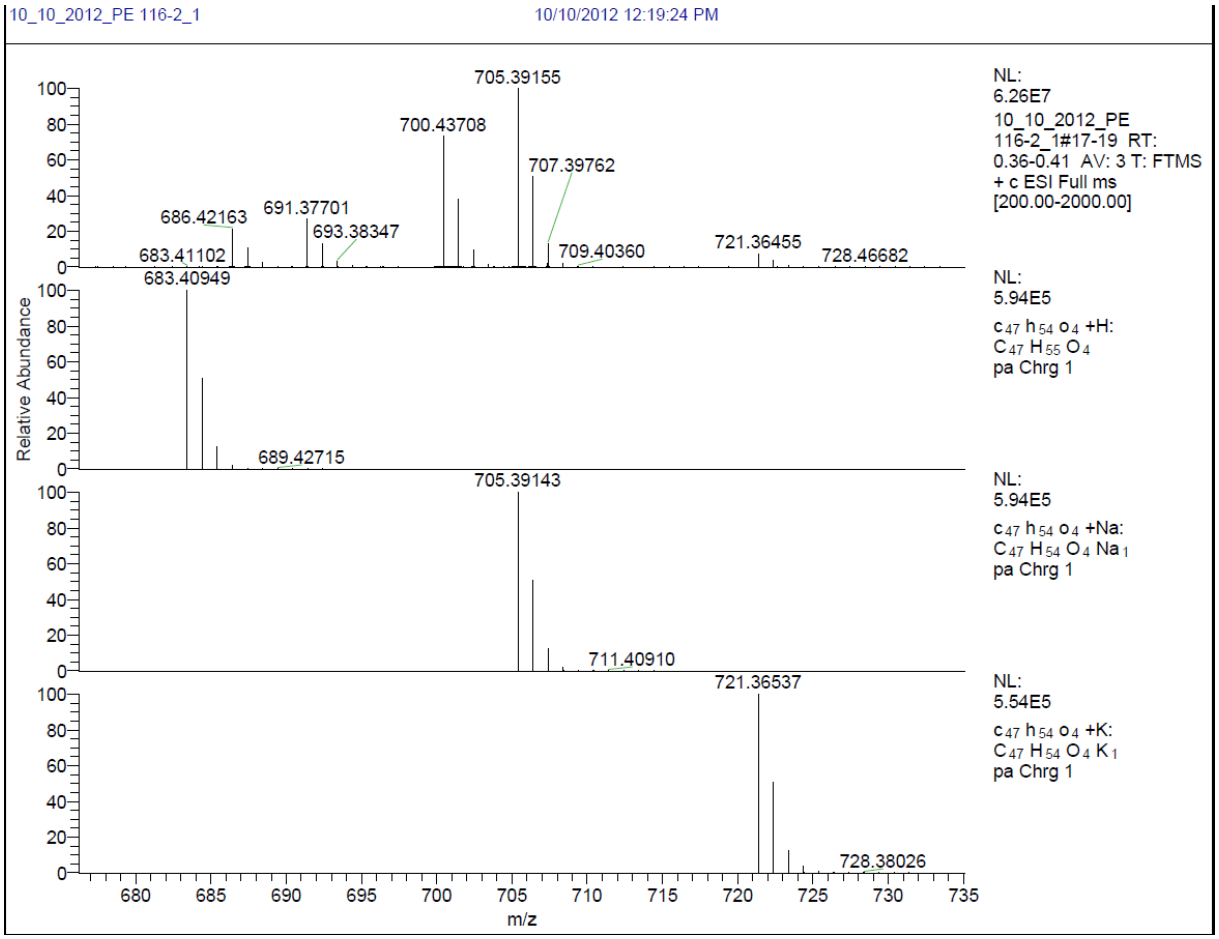
**Compound 5c**



**Compound 5c**


CH3
O
Pr
O
Pr
O
Pr
O
Pr
Absorbance
Wavenumbers (cm-1)
2960,7
2930,7
2874,0
1382,8
1245,3
1206,0
1096,4
1084,9
1038,1
1007,0
967,1
757,6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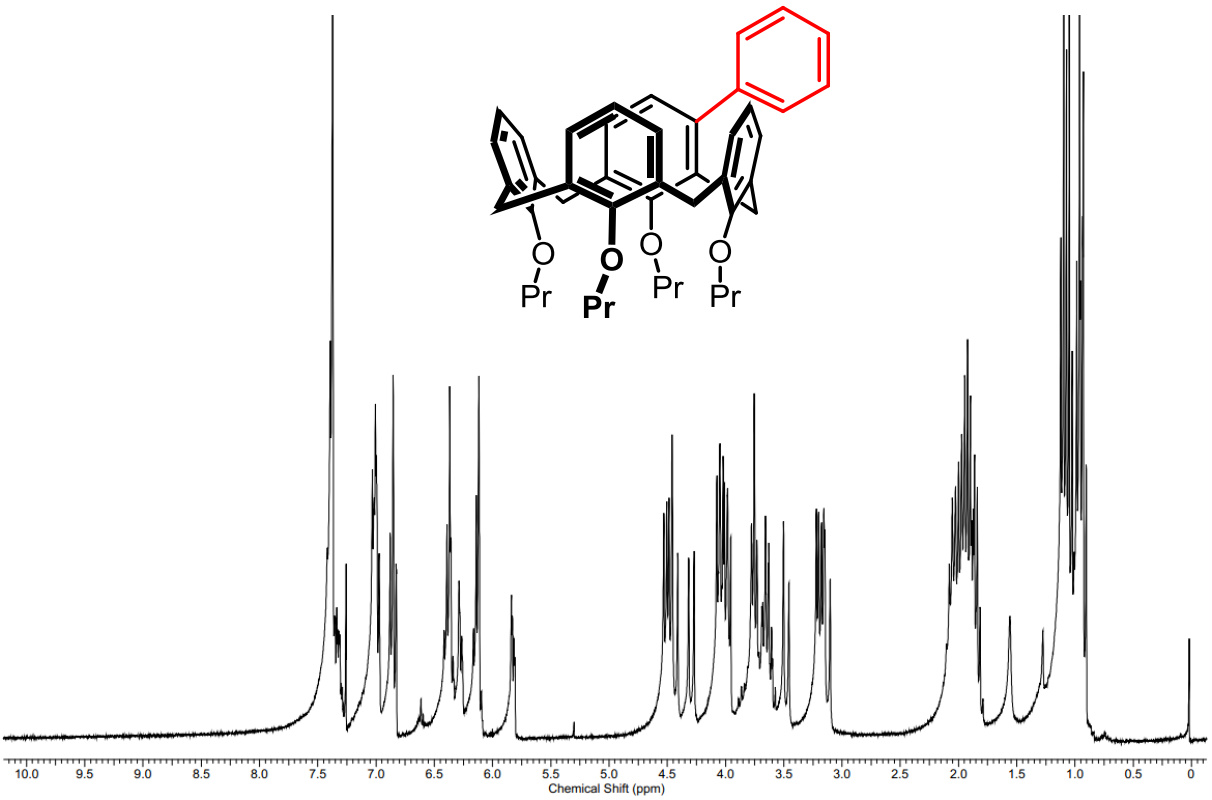
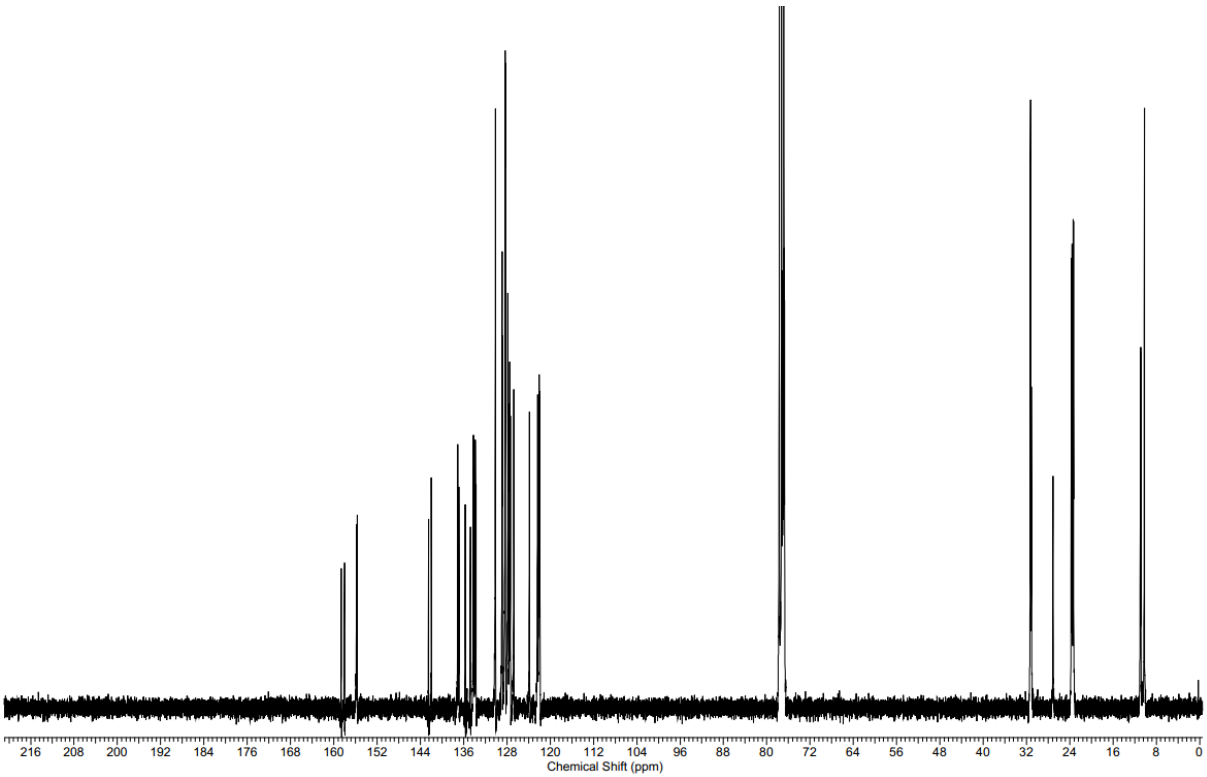
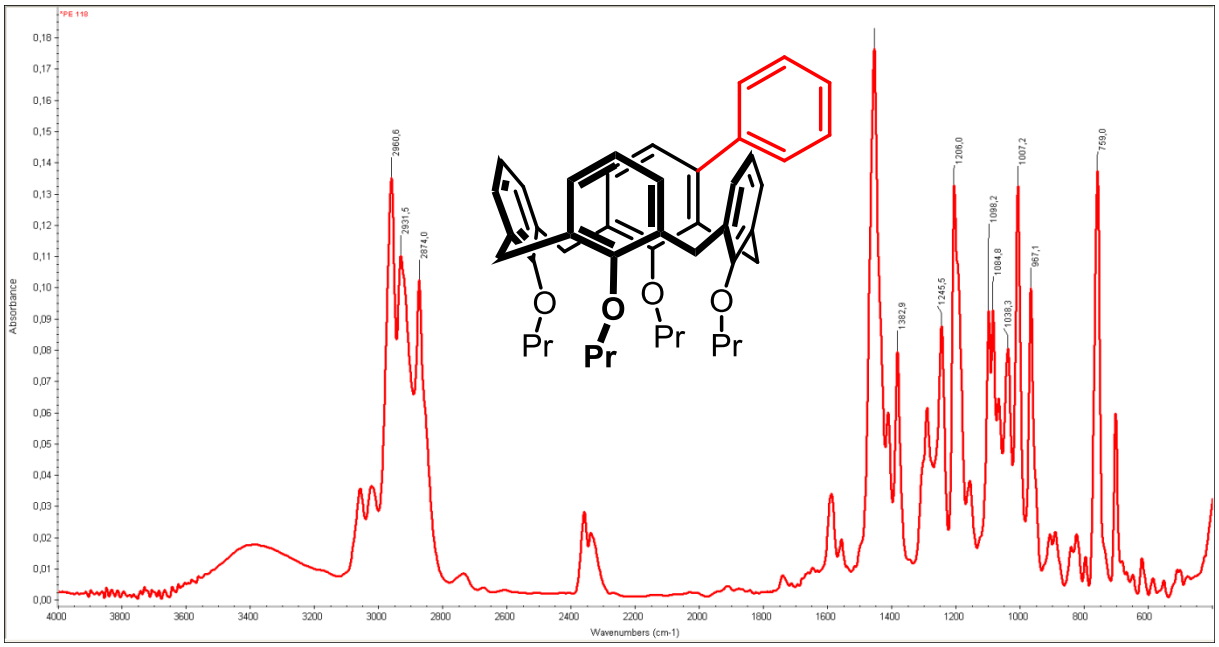
10/10/2012 12:19:24 PM
705.39155
700.43708
707.39762
686.42163
691.37701
693.38347
683.41102
709.40360
721.36455
728.46682
683.40949
689.42715
705.39143
711.40910
721.36537
728.38026
Relative Abundance
m/z
NL:
6.26E7
10_10_2012_PE
116-2_1#17-19 RT:
0.36-0.41 AV: 3 T: FTMS
+ c ESI Full ms
[200.00-2000.00]
NL:
5.94E5
c47 h54 o4 +H:
C47 H55 O4
pa Chrg 1
NL:
5.94E5
c47 h54 o4 +Na:
C47 H54 O4 Na1
pa Chrg 1
NL:
5.54E5
c47 h54 o4 +K:
C47 H54 O4 K1
pa Chrg 1

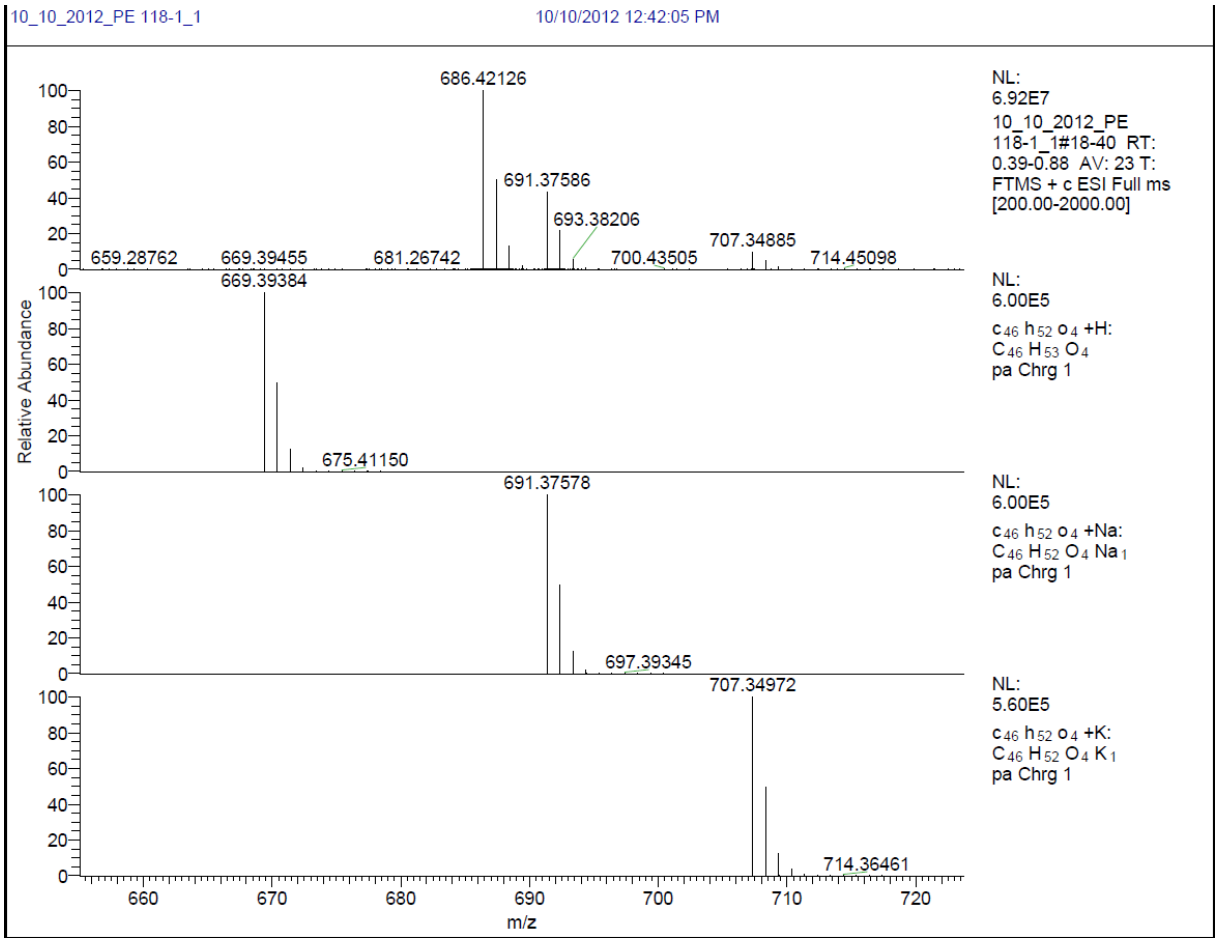
**Compound 5d**



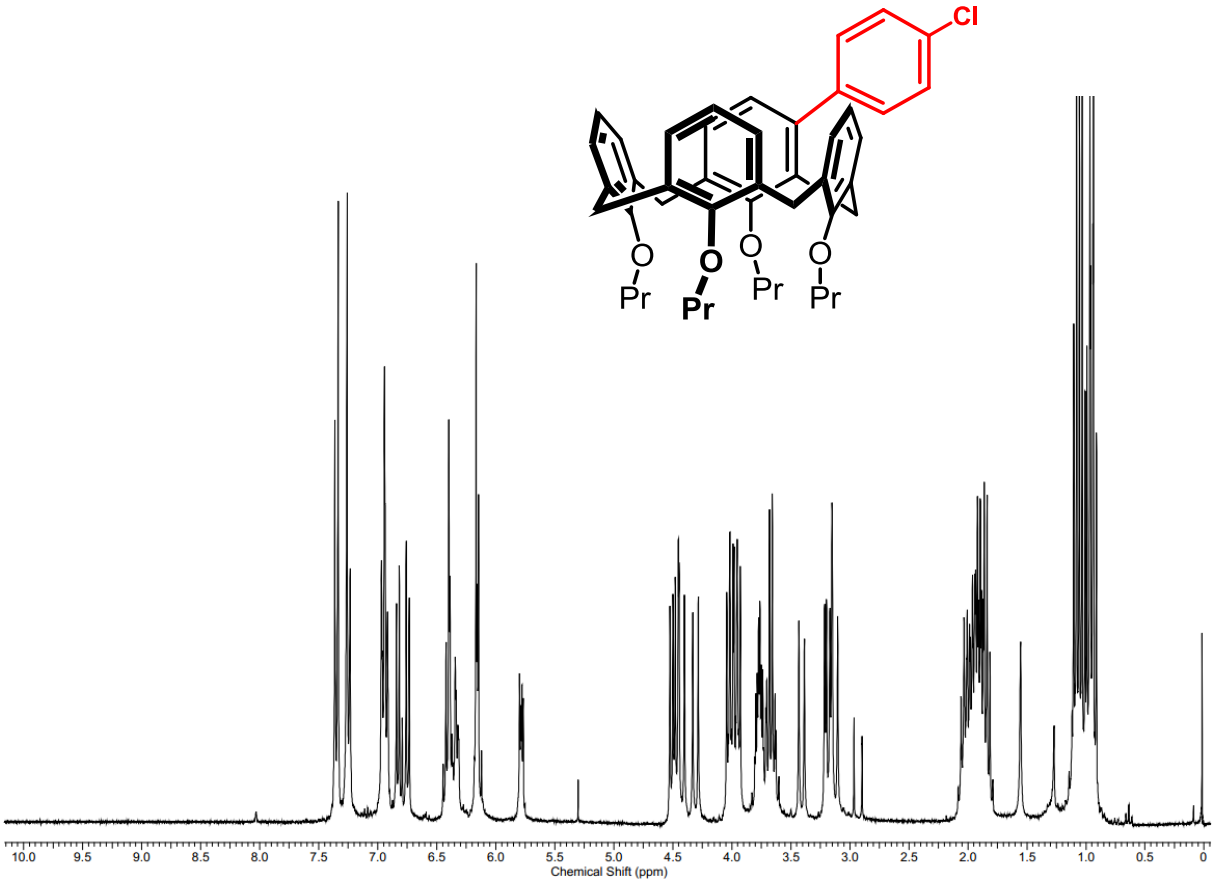



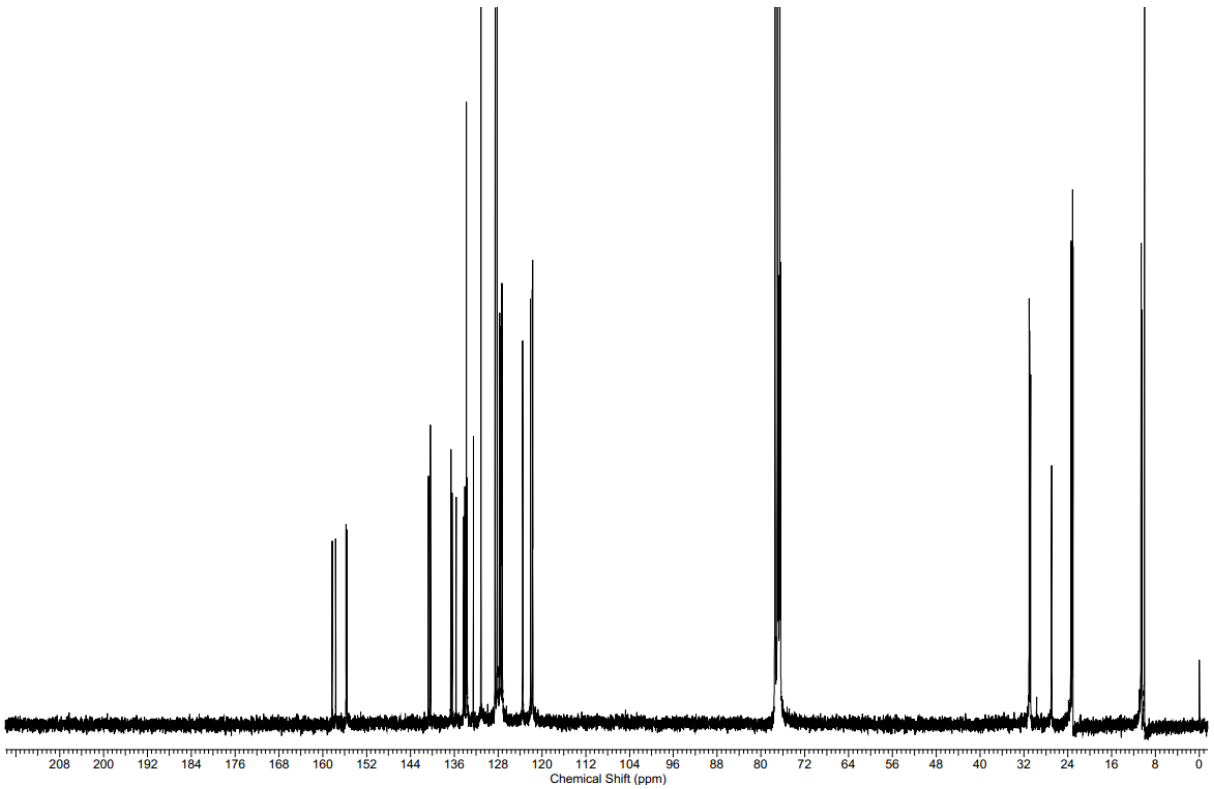
## Compound 5d


Absorbance
Wavenumbers (cm-1)
2960,6
2931,5
2874,0
1382,9
1245,5
1206,0
1098,2
1084,8
1038,3
1007,2
967,1
759,0
Pr
Pr
Pr
Pr

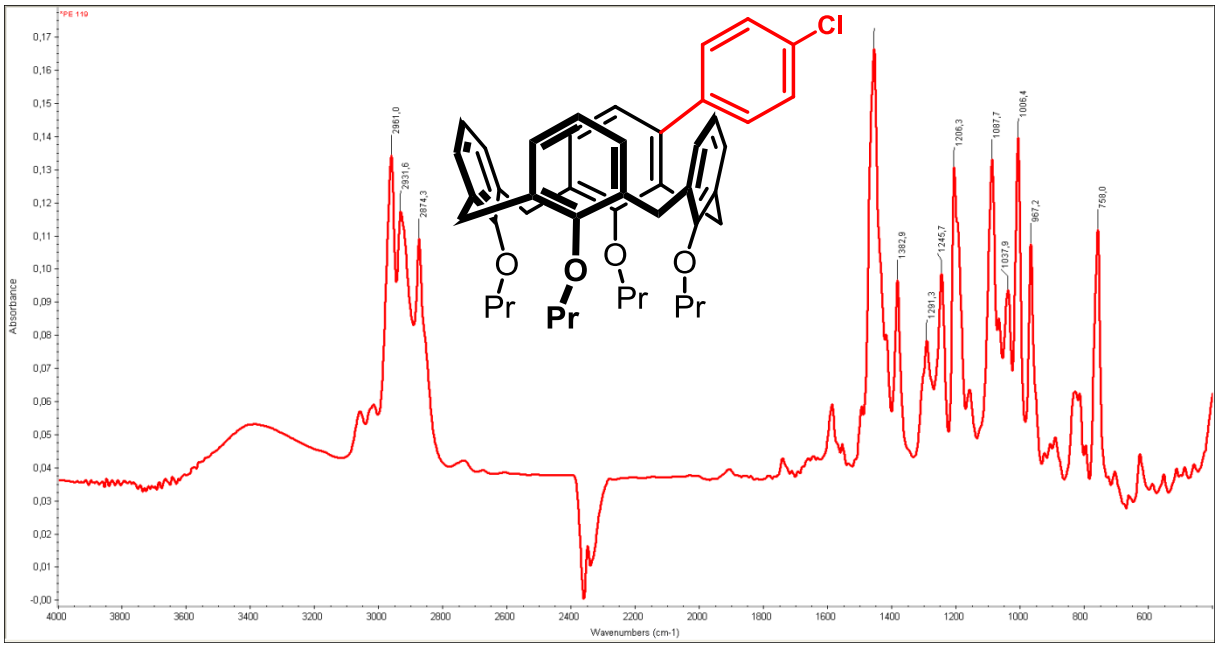

10_10_2012_PE 118-1_1
10/10/2012 12:42:05 PM
686.42126
691.37586
693.38206
659.28762
669.39455
681.26742
700.43505
707.34885
714.45098
NL:
6.92E7
10_10_2012_PE
118-1_1#18-40 RT:
0.39-0.88 AV: 23 T:
FTMS + c ESI Full ms
[200.00-2000.00]
669.39384
675.41150
NL:
6.00E5
c46 h52 o4 +H:
C46 H53 O4
pa Chrg 1
691.37578
697.39345
NL:
6.00E5
c46 h52 o4 +Na:
C46 H52 O4 Na1
pa Chrg 1
707.34972
714.36461
NL:
5.60E5
c46 h52 o4 +K:
C46 H52 O4 K1
pa Chrg 1
Relative Abundance
m/z

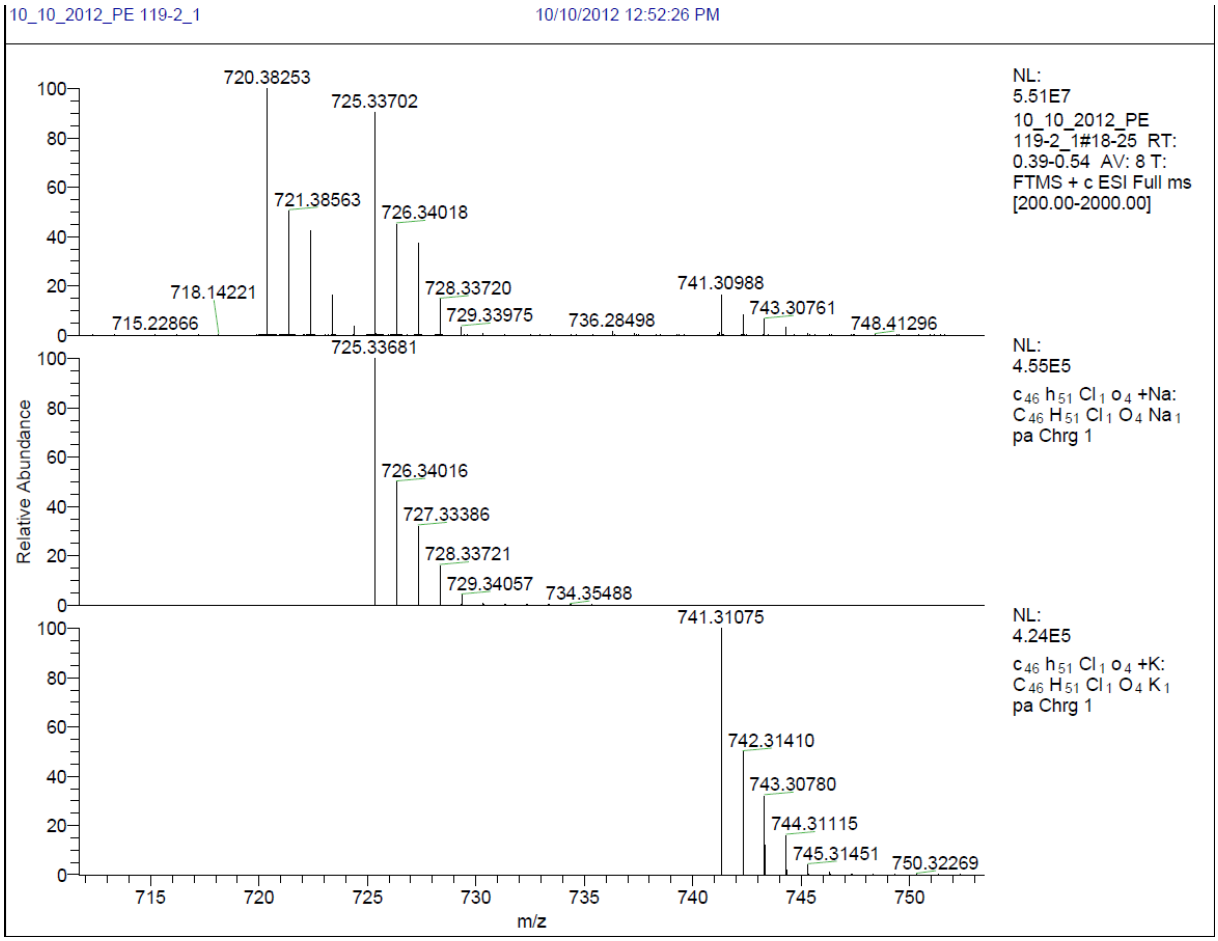
**Compound 5e**



**Compound 5e**


Cl
O
Pr
O
Pr
O
Pr
O
Pr
2961,0
2931,6
2874,3
1382,9
1391,3
1245,7
1206,3
1097,7
1037,9
1006,4
967,2
758,0
Absorbance
Wavenumbers (cm-1)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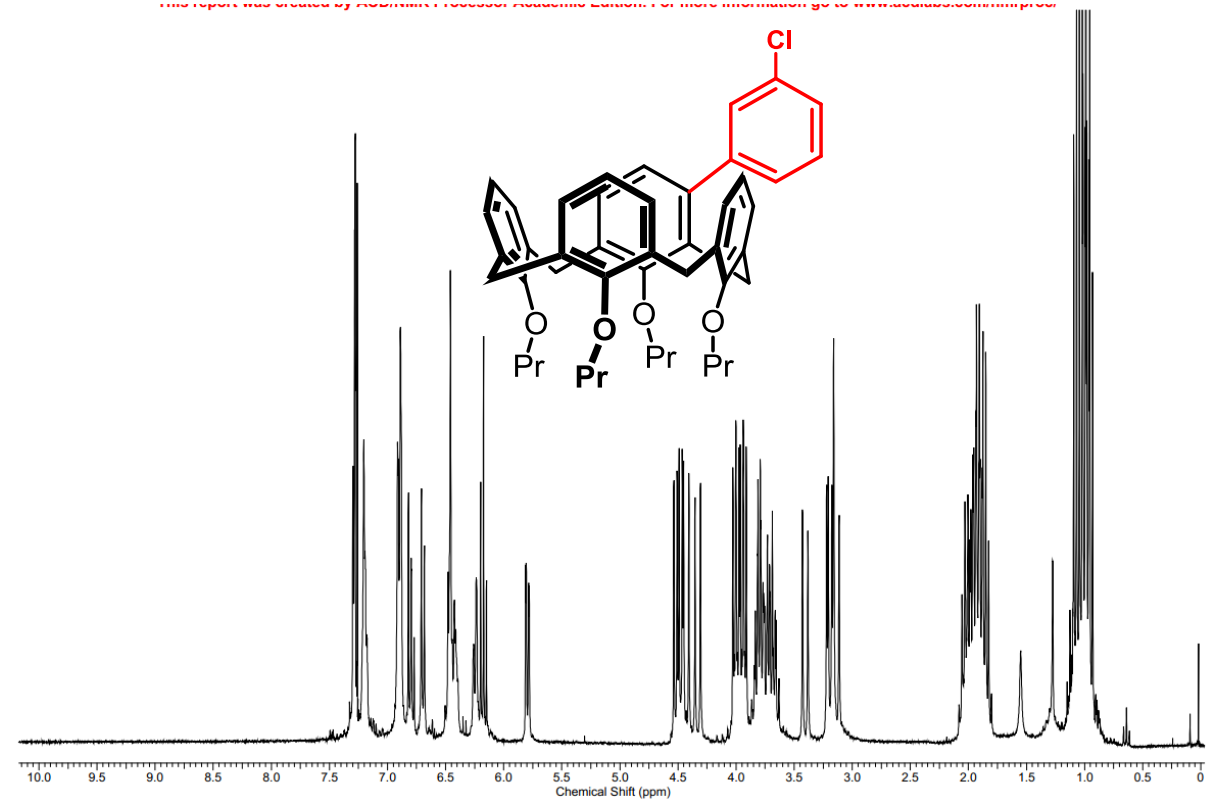
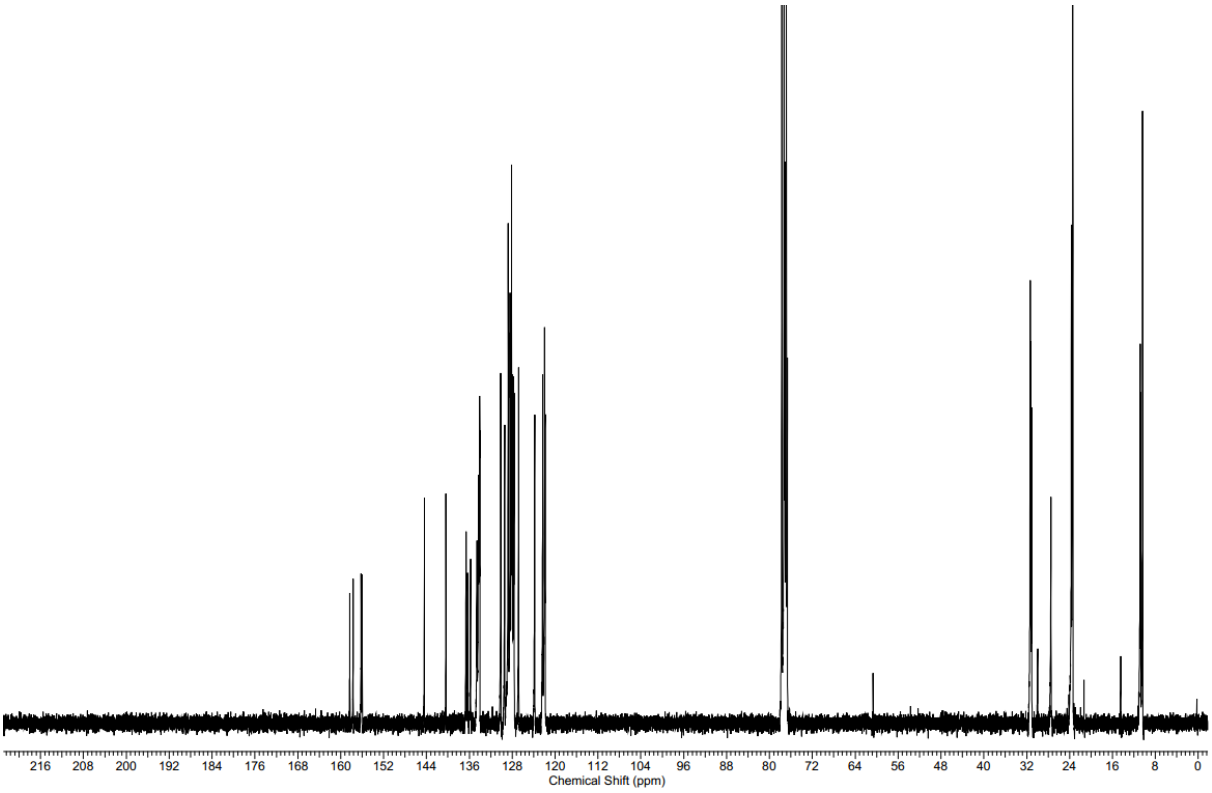
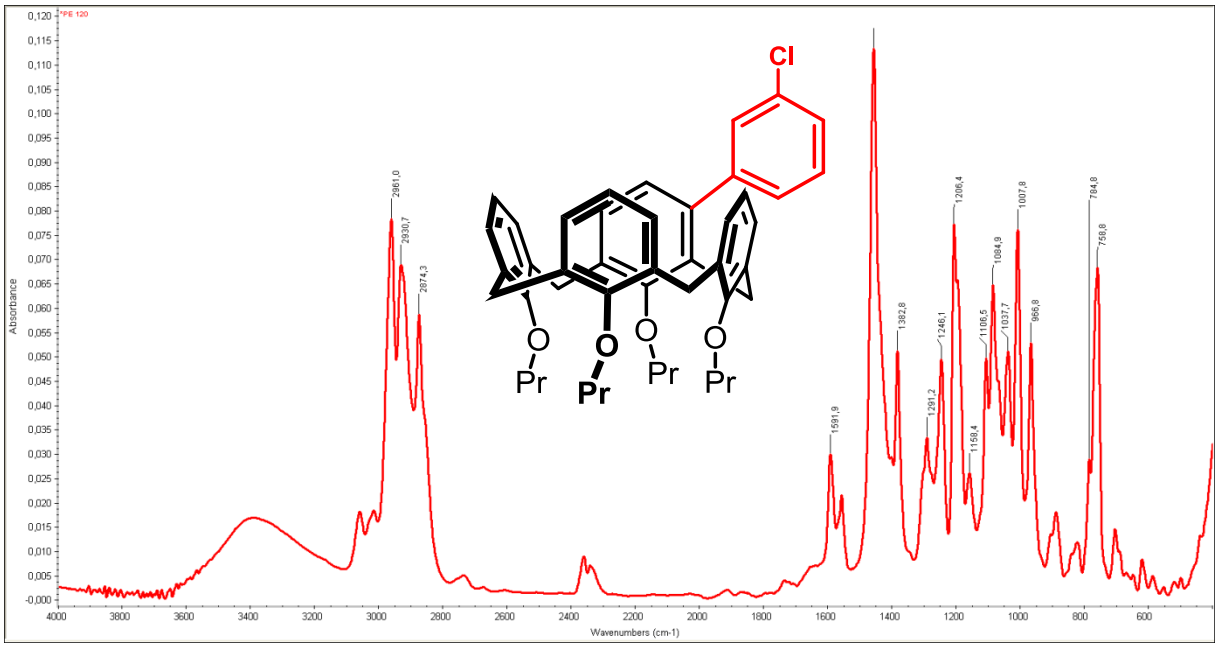
720.38253
725.33702
721.38563
726.34018
718.14221
728.33720
729.33975
715.22866
736.28498
741.30988
743.30761
748.41296
NL:
5.51E7
10_10_2012_PE
119-2_1#18-25 RT:
0.39-0.54 AV: 8 T:
FTMS + c ESI Full ms
[200.00-2000.00]
725.33681
726.34016
727.33386
728.33721
729.34057
734.35488
NL:
4.55E5
c46 h51 Cl1 o4 +Na:
C46 H51 Cl1 O4 Na1
pa Chrg 1
741.31075
742.31410
743.30780
744.31115
745.31451
750.32269
NL:
4.24E5
c46 h51 Cl1 o4 +K:
C46 H51 Cl1 O4 K1
pa Chrg 1
Relative Abundance
m/z

**Compound 5f**


Cl
O
Pr
O
Pr
O
Pr
O
Pr
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
Chemical Shift (ppm)


216 208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

**Compound 5f**

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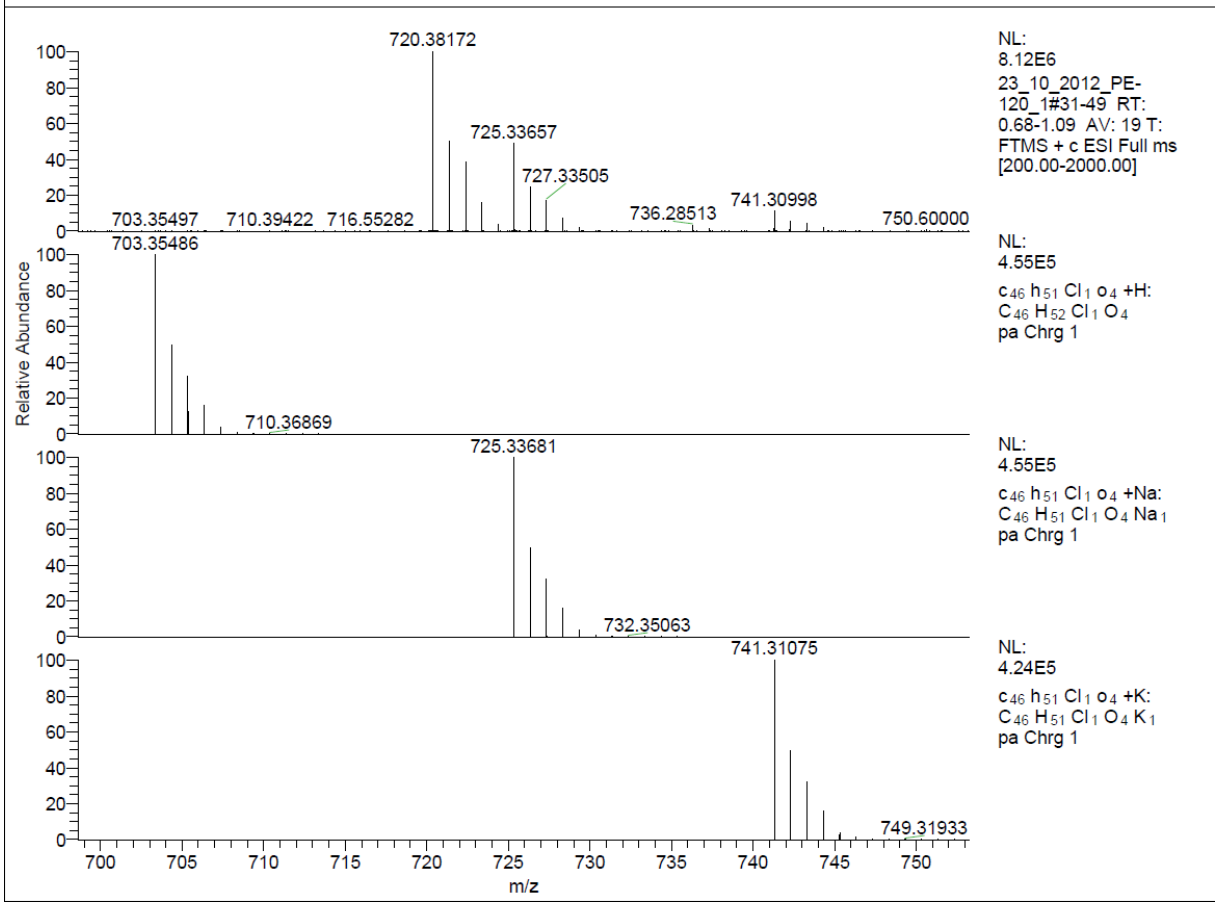
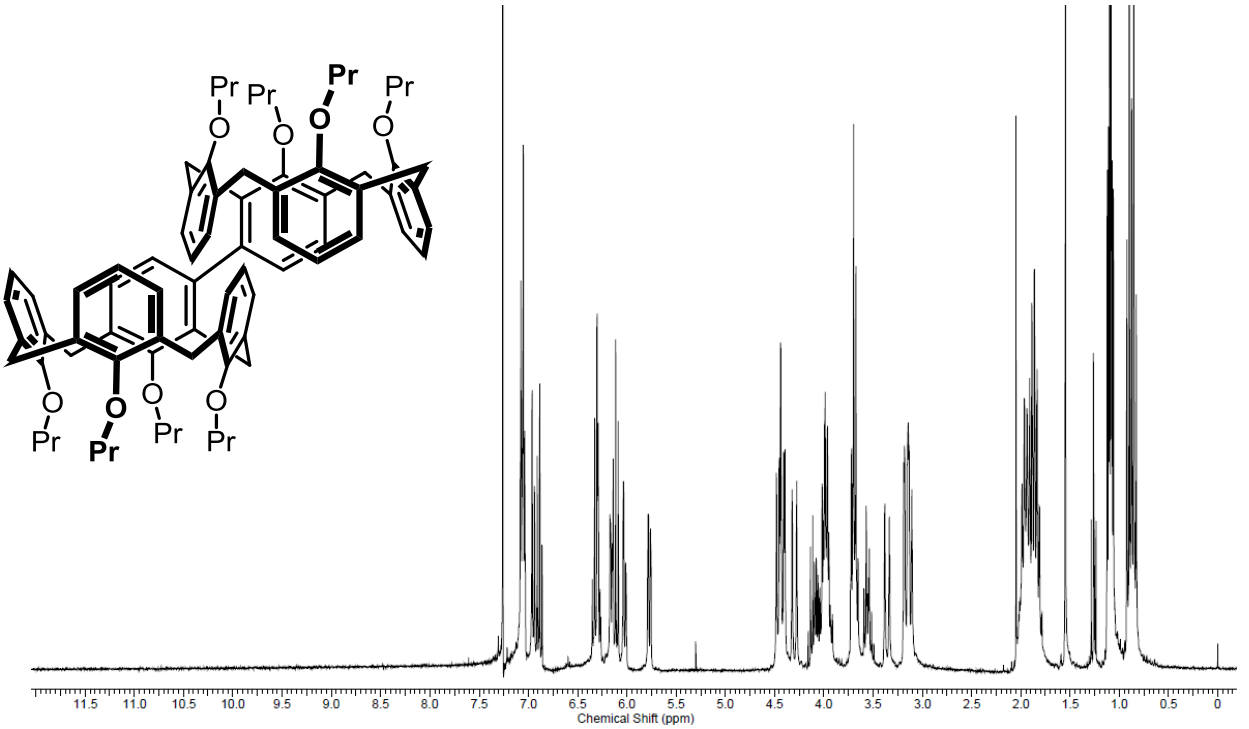
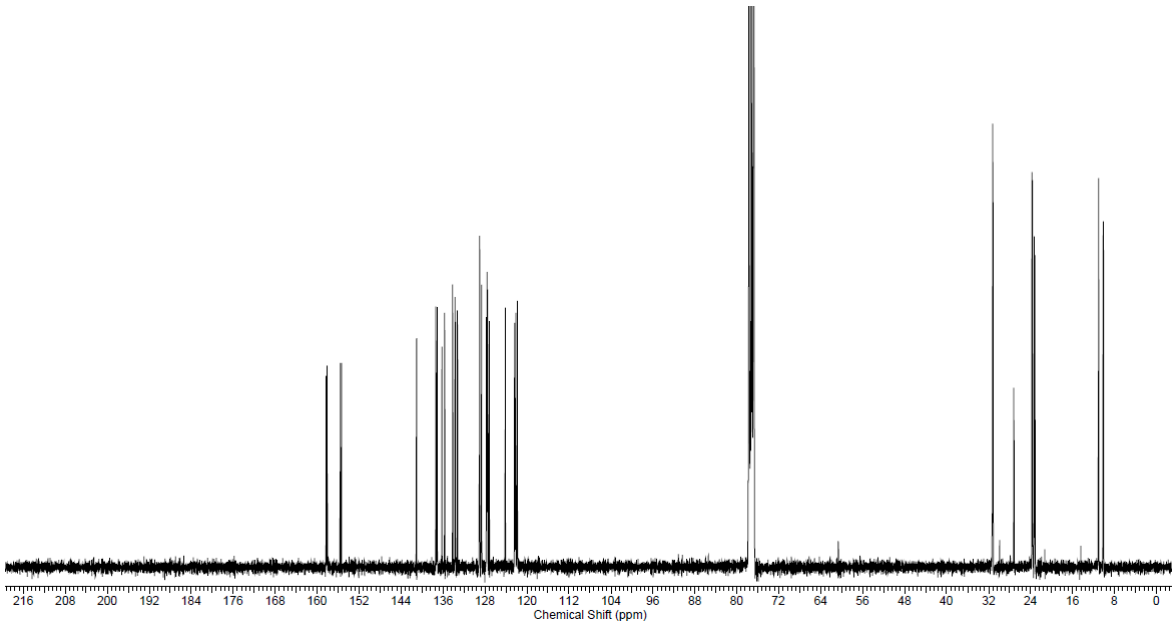
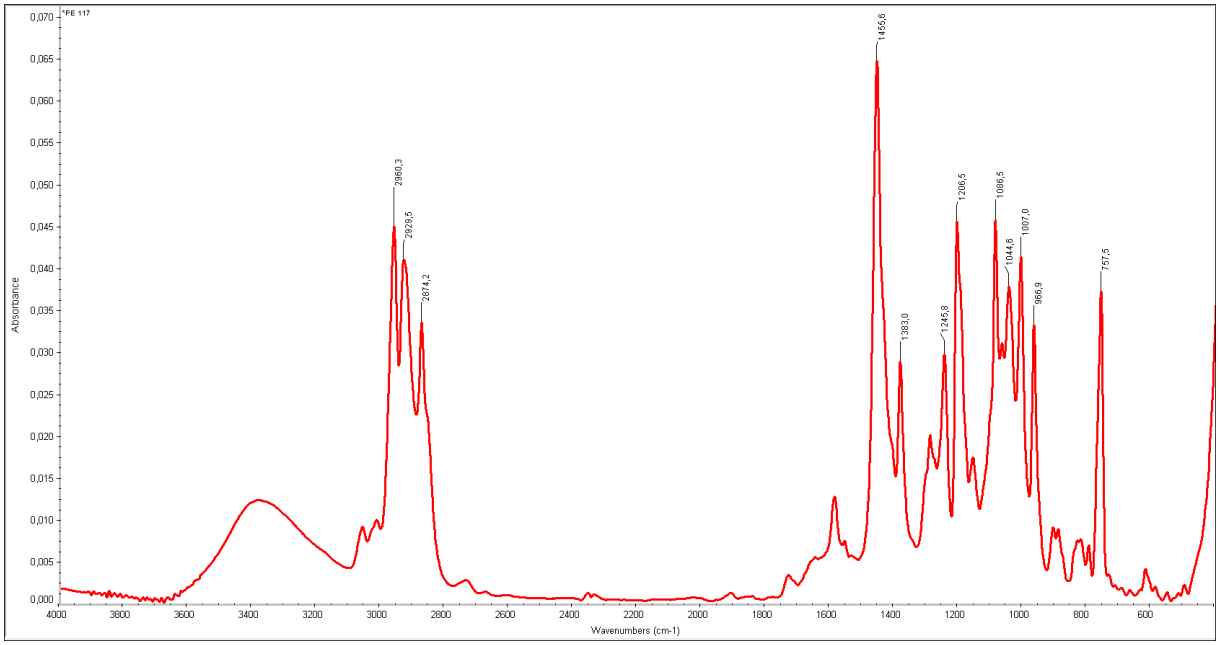


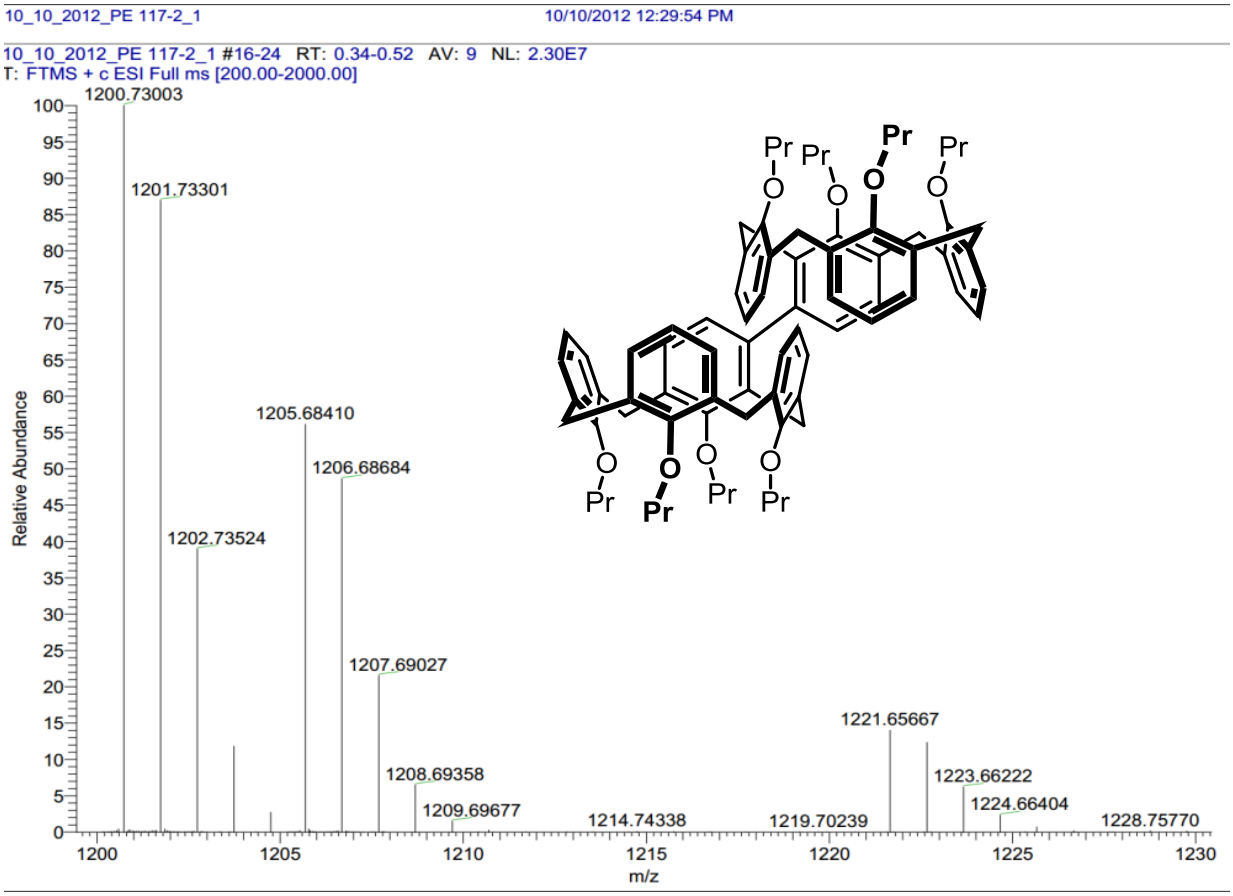
**Compound 6**



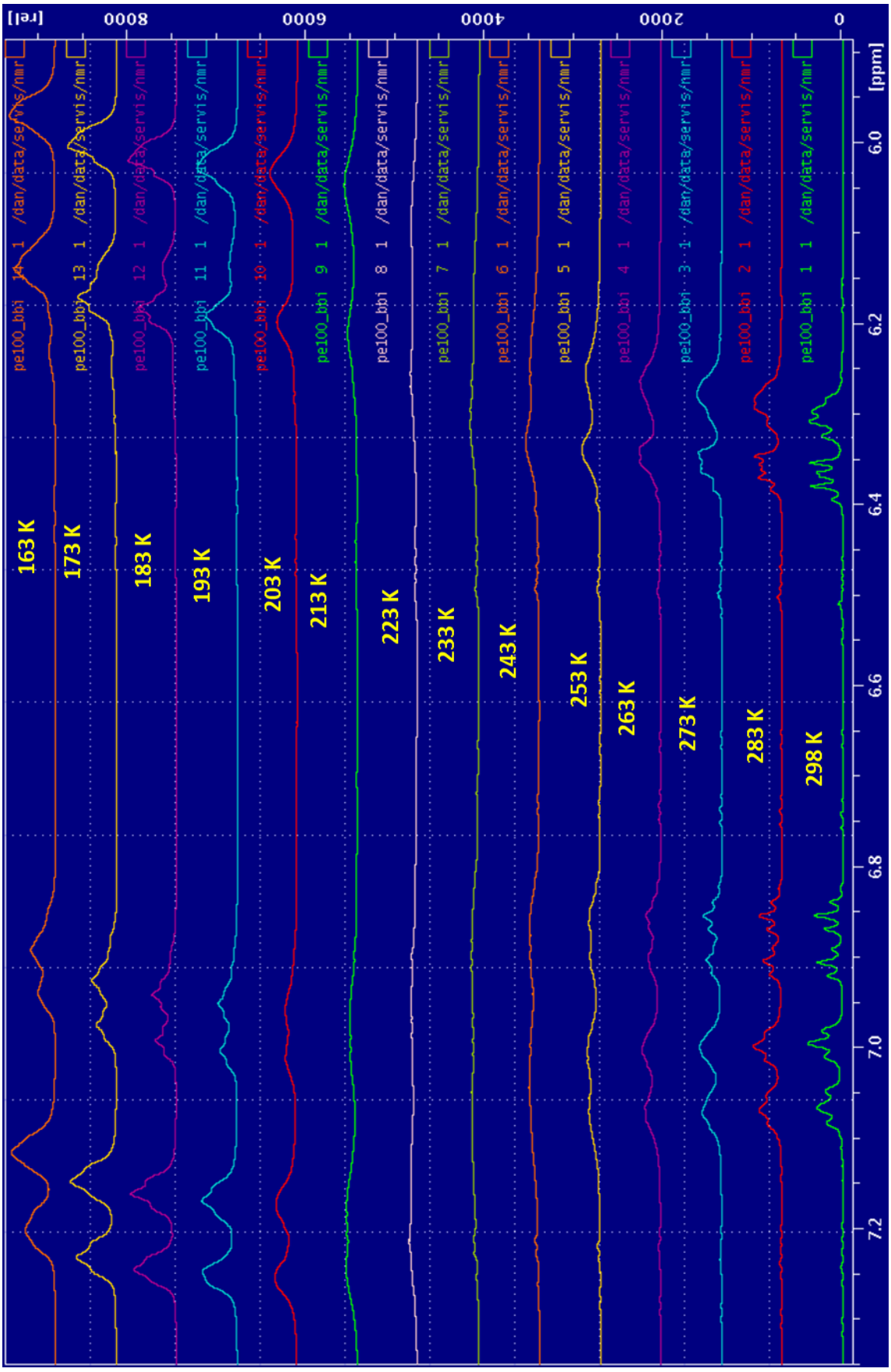



## Compound 6

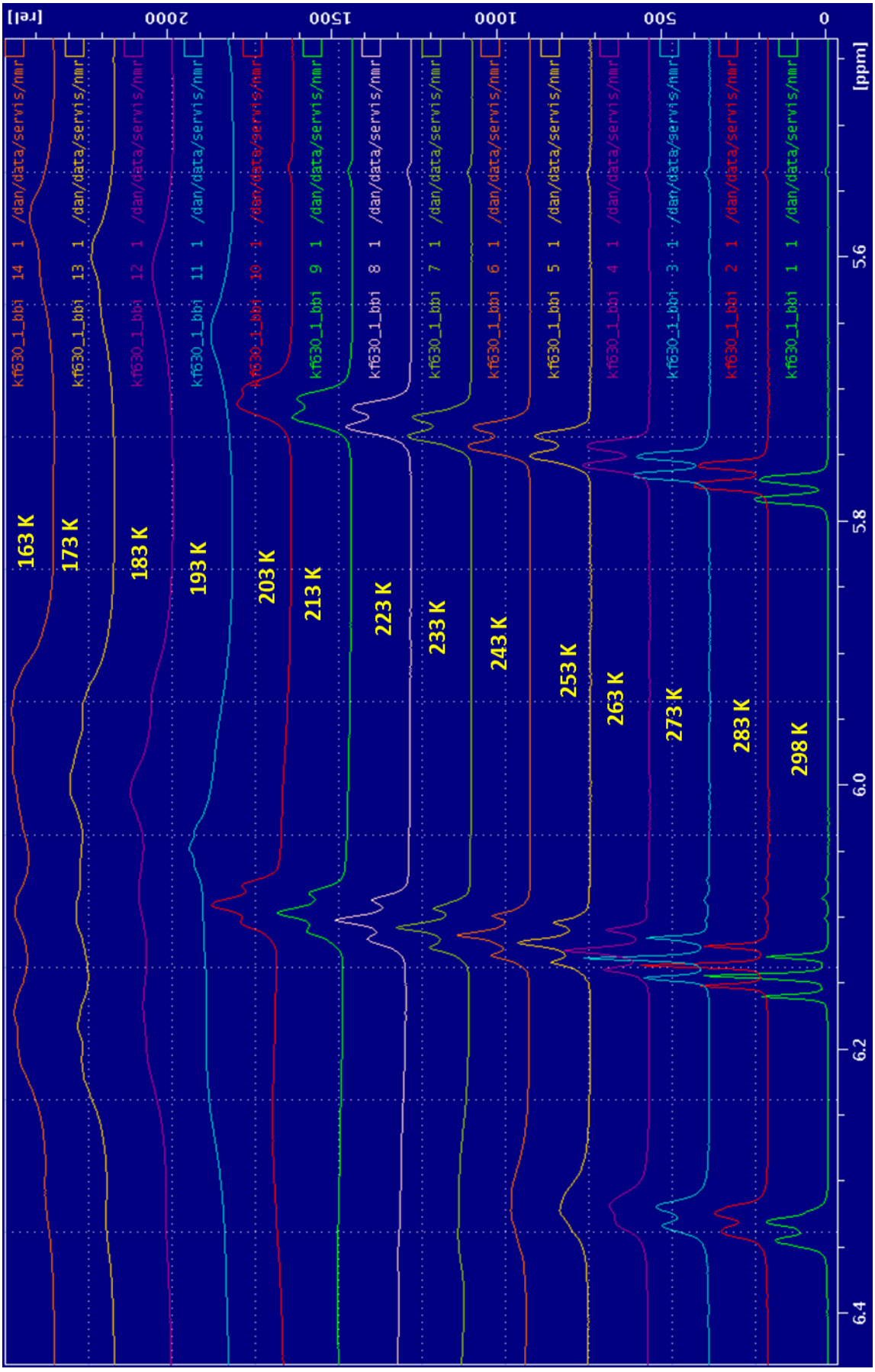

*PE 117
Absorbance
Wavenumbers (cm-1)
2960,3
2929,5
2874,2
1455,6
1383,0
1245,8
1206,5
1086,5
1044,6
1007,0
966,9
757,5


10_10_2012_PE 117-2_1
10/10/2012 12:29:54 PM
10_10_2012_PE 117-2_1 #16-24 RT: 0.34-0.52 AV: 9 NL: 2.30E7
T: FTMS + c ESI Full ms [200.00-2000.00]
Relative Abundance
m/z
1200.73003
1201.73301
1205.68410
1206.68684
1202.73524
1207.69027
1208.69358
1209.69677
1214.74338
1219.70239
1221.65667
1223.66222
1224.66404
1228.75770
Pr
O
O
Pr
O
Pr
O
Pr
O
Pr
O
Pr
O
Pr
O
Pr

Compound **3a**, 298 – 163 K, $CD_2Cl_2$, 500 MHz



Compound 5**a**, 298 – 163 K, $CD_2Cl_2$, 500 MHz



Crystallographic data for derivative **3a** ($C_{40}H_{47}ClHgO_4$):

M = 827.85 g.mol$^{-1}$, monoclinic system, space group *C2/c*, *a* = 38.668 (1) Å, *b* = 9.91309 (18) Å, *c* = 38.2857 (9) Å, β = 99.843 (2)°, Z = 16, *V* = 14459.6 (6) Å$^3$, Dc = 1.521 g.cm$^{-3}$, μ(Cu-Kα) = 8.62mm$^{-1}$, crystal dimensions of 0.58 × 0.24 × 0.16mm. Data were collected at 190 (2) K on Xcalbur Onyx CCD diffractometer with graphite monochromated Cu-Kα radiation. The structure was solved by charge flipping methods[1] using the CRYSTALS suite of programs[2] and anisotropically refined by full matrix least squares on F squared value to final R = 0.041 and $R_w$ = 0.094 using 15129 independent reflections ($\Theta_{max}$ = 77.5°), 910 parameters and 114 restrains. The positions of disordered propoxy groups were found from the electron density maps and then placed in appropriate positions. All distances between neighbouring atoms and angles were restrained. Site occupancies were assigned resulting in similar thermal parameters for each of disordered groups. The hydrogen atoms were placed in calculated positions. The structure was deposited into Cambridge Structural Database under number CCDC 926581.

Crystallographic data for compound **6** ($C_{80}H_{94}O_8$):

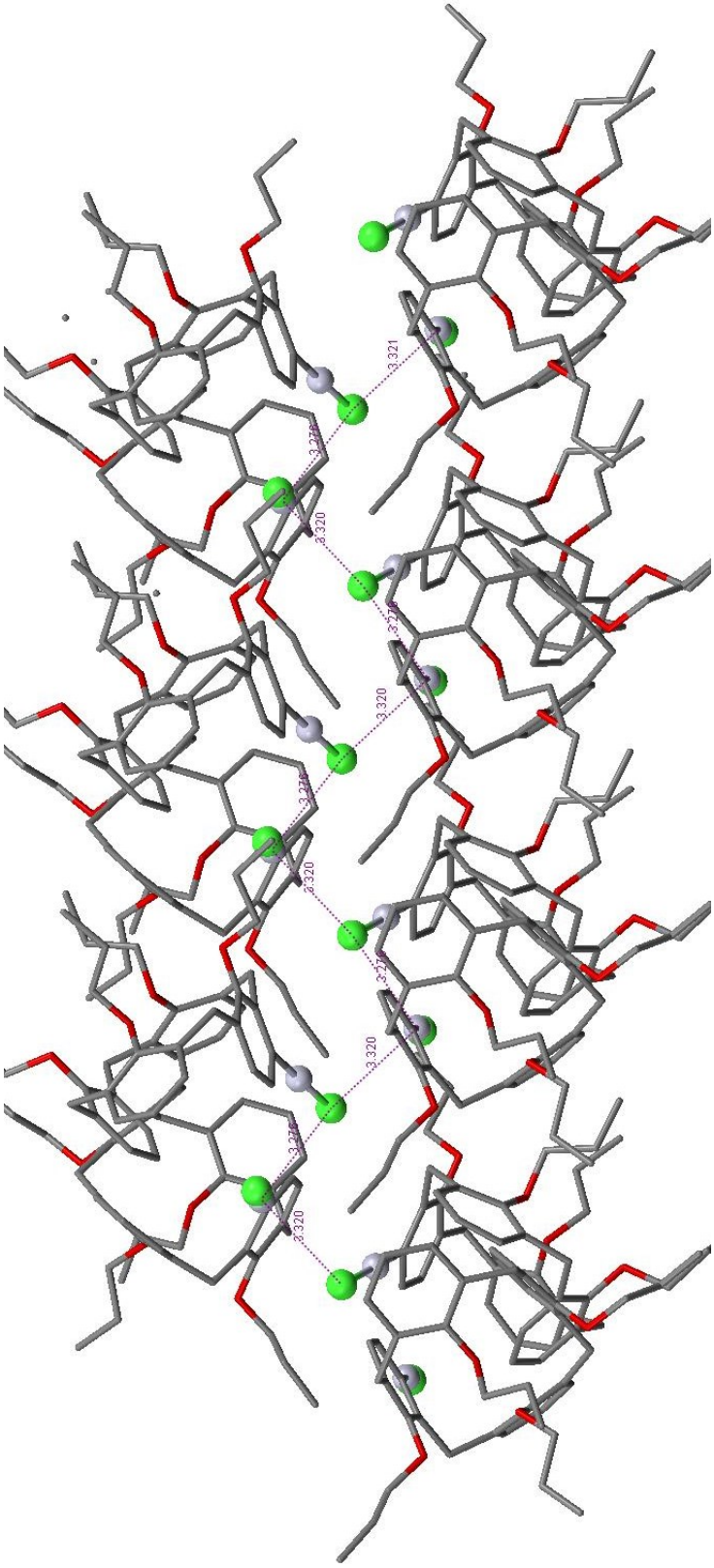
M = 1183.62 g.mol$^{-1}$, monoclinic system, space group *P21/c*, *a* = 13.4473 (2) Å, *b* = 30.0322 (5) Å, *c* = 16.7673 (3) Å, β = 92.7714 (14)°, Z = 4, *V* = 6763.55 (19) Å$^3$, Dc = 1.162 g.cm$^{-3}$, μ(Cu-Kα) = 0.57mm$^{-1}$, crystal dimensions of 0.47 × 0.26 × 0.12mm. Data were collected at 190 (2) K on Xcalbur Onyx CCD diffractometer with graphite monochromated Cu-Kα radiation. The structure was solved by charge flipping methods[1] using the CRYSTALS suite of programs[2] and anisotropically refined by full matrix least squares on F squared value to final R = 0.055 and $R_w$ = 0.101 using 13670 independent reflections ($\Theta_{max}$ = 75.9°), 919 parameters and 223 restrains. The positions of disordered propoxy groups were found from the electron density maps and then placed in appropriate positions. All distances between neighbouring atoms and angles were restrained. The site occupancies were refined to have summary full occupancy. The hydrogen atoms were placed in calculated positions. The structure was deposited into Cambridge Structural Database under number CCDC 926580.

Crystallographic data for compound **5a** ($C_{46}H_{51}NO_6$):

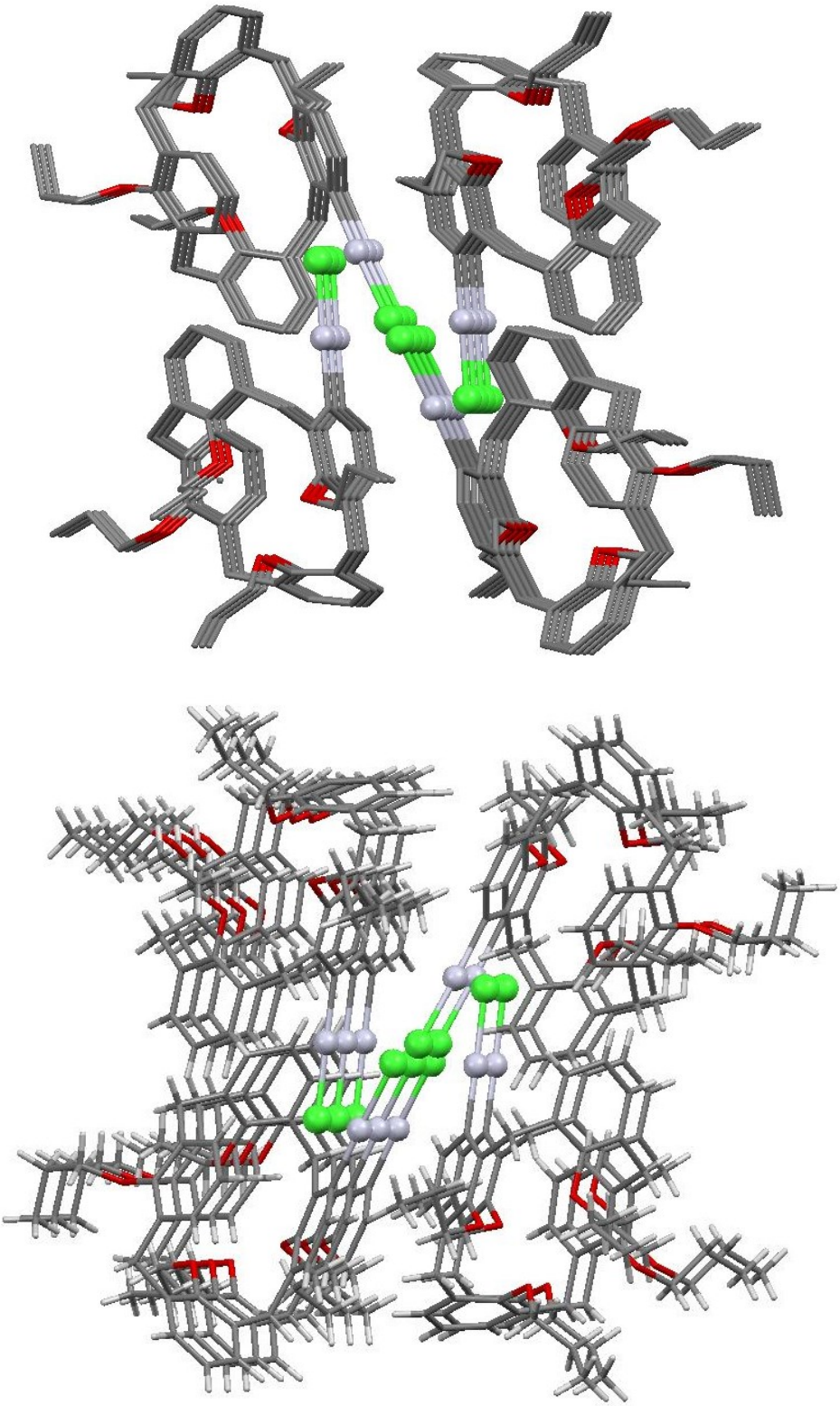
M = 713.9 g.mol$^{-1}$, triclinic system, space group *P-1*,*a* = 11.6871 (5) Å, *b* = 12.8055 (7) Å,*c* = 14.7108 (6) Å, α = 94.246 (4)°, β = 91.367 (3)°, γ = 113.935 (5)°, Z = 2,*V* = 2003.25 (18) Å$^3$, Dc = 1.183 g.cm$^{-3}$, μ(Cu-Kα) = 0.62 mm$^{-1}$, crystal dimensions of

0.54 × 0.35 × 0.22mm. Data were collected at 120 (2) K on Gemini Atlas CCD diffractometer with graphite monochromated Cu-Kα radiation. The structure was solved by charge flipping methods[1] using the Jana2006 suite of programs[3] and anisotropically refined by full matrix least squares on F squared value to final R = 0.037 and $R_w$ = 0.108 using 6940 independent reflections ($\Theta_{max}$ = 66.9°), 498 parameters and 10 restrains. The positions of disordered propoxy group was found from the electron density maps and then placed in appropriate positions. All distances between neighbouring atoms and angles were restrained to have bond lengths and angles to have same value for each disordered position. The site occupancies were refined to have summary full occupancy. The hydrogen atoms were placed in calculated positions. The structure was deposited into Cambridge Structural Database under number CCDC926579.

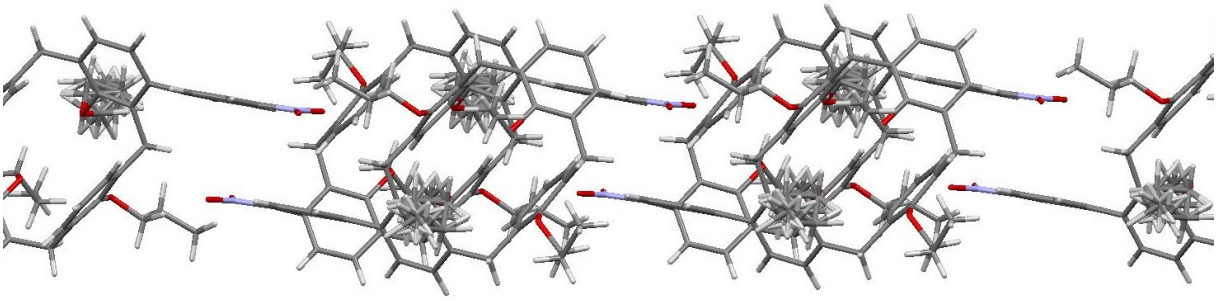
The crystal packing of compound **3a**



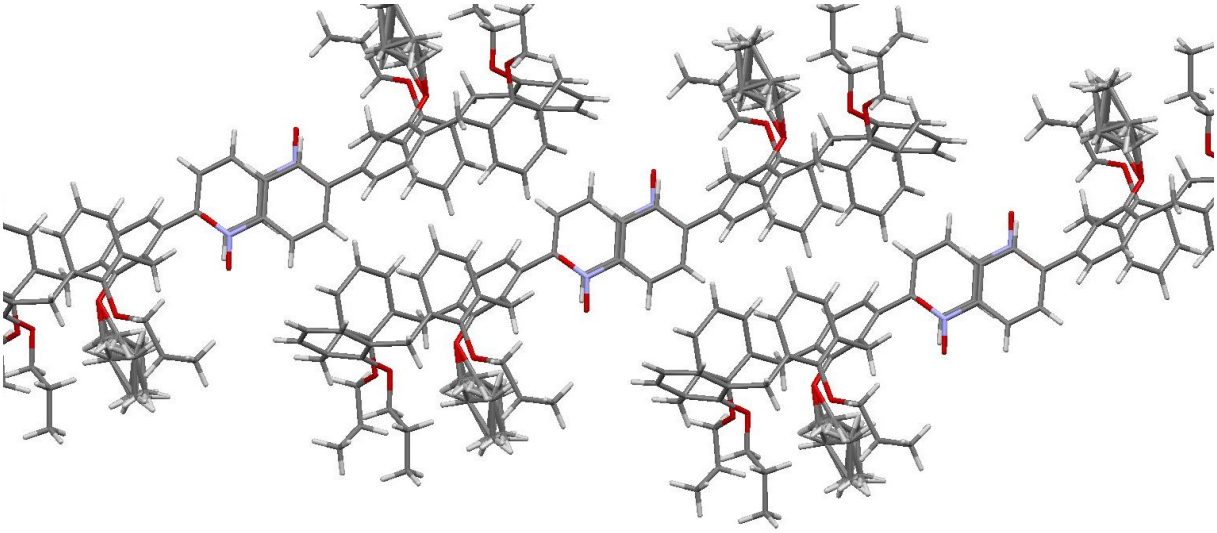
Crystal packing of compound **3a** – the channel-like arrangement of Hg-Cl bonds.



Crystal packing of compound **5a**:



a) The interactions between nitrophenyl subunits (side view)



b) The interactions between nitrophenyl subunits (upper view)