Supporting Information

Synthesis of Upper Rim-Double-Bridged Calix[4]arenes Bearing Seven-Membered Rings and Related Compounds

Martin Tlustý,[†] Martin Babor,[§] Václav Eigner,[∫] Michal Kohout[†] and Pavel Lhoták[†*]

[†]Department of Organic Chemistry, University of Chemistry and Technology, Prague (UCTP), Technicka 5, 166 28 Prague 6, Czech Republic
[§] Department of Solid State Chemistry, UCTP, Technicka 5, 166 28 Prague 6, Czech Republic
[∫] Department of Structure Analysis, Institute of Physics of Czech Academy of Sciences, Na Slovance 1999, 182 21 Prague 8, Czech Republic

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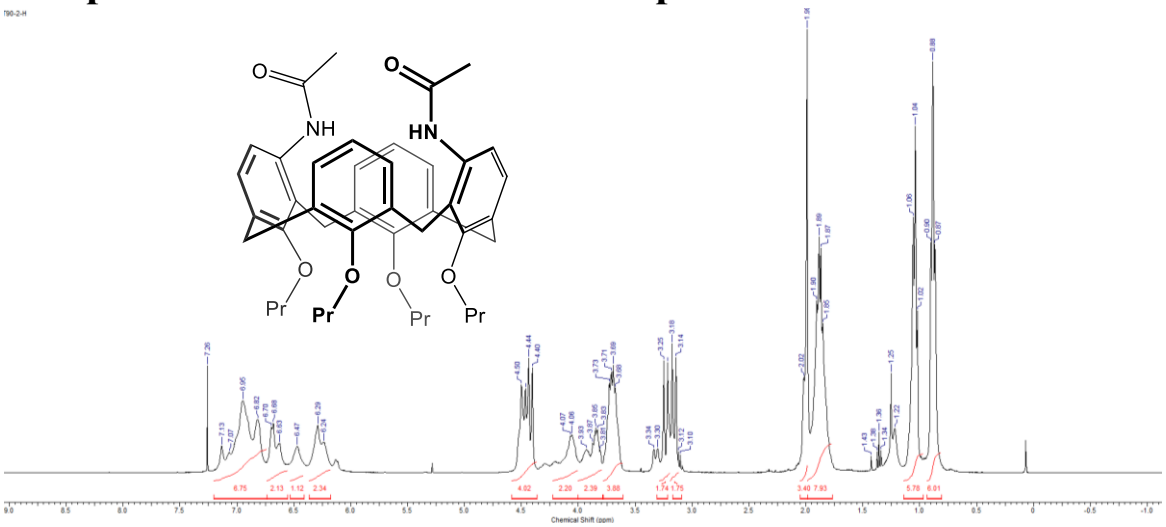


Figure 1: ^{1}H NMR of compound **6a** ($CDCl_3$, 400 MHz).

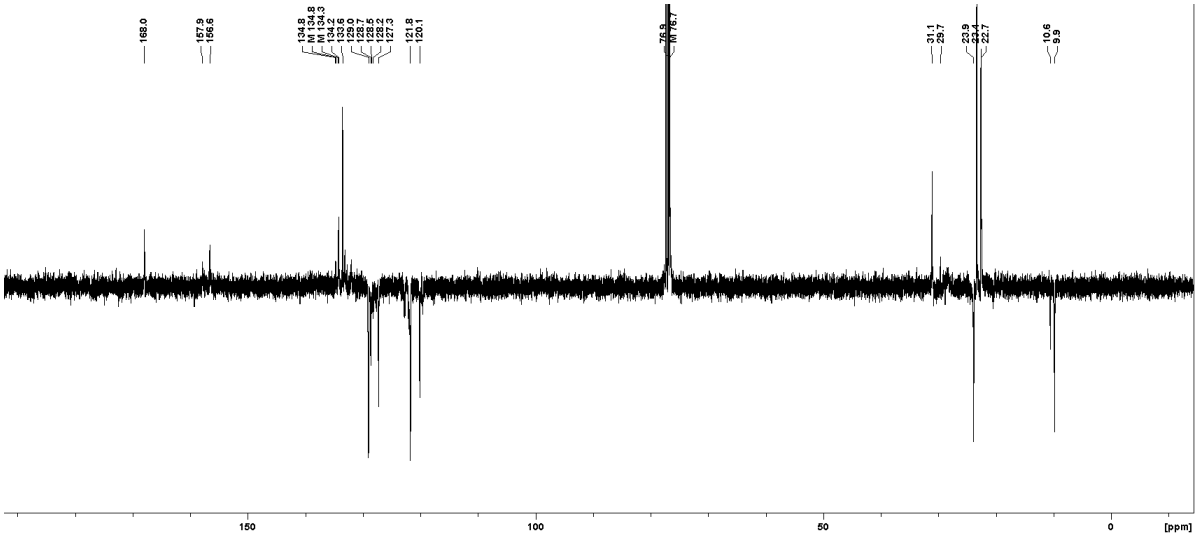


Figure 2: ^{13}C(APT) NMR of compound **6a** ($CDCl_3$, 100 MHz).

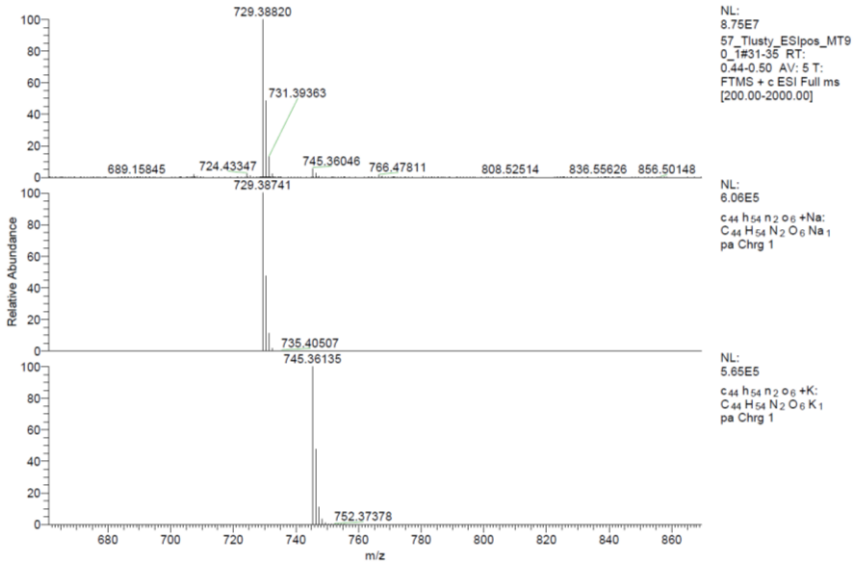


Figure 3: HRMS of compound **6a** (ESI+).

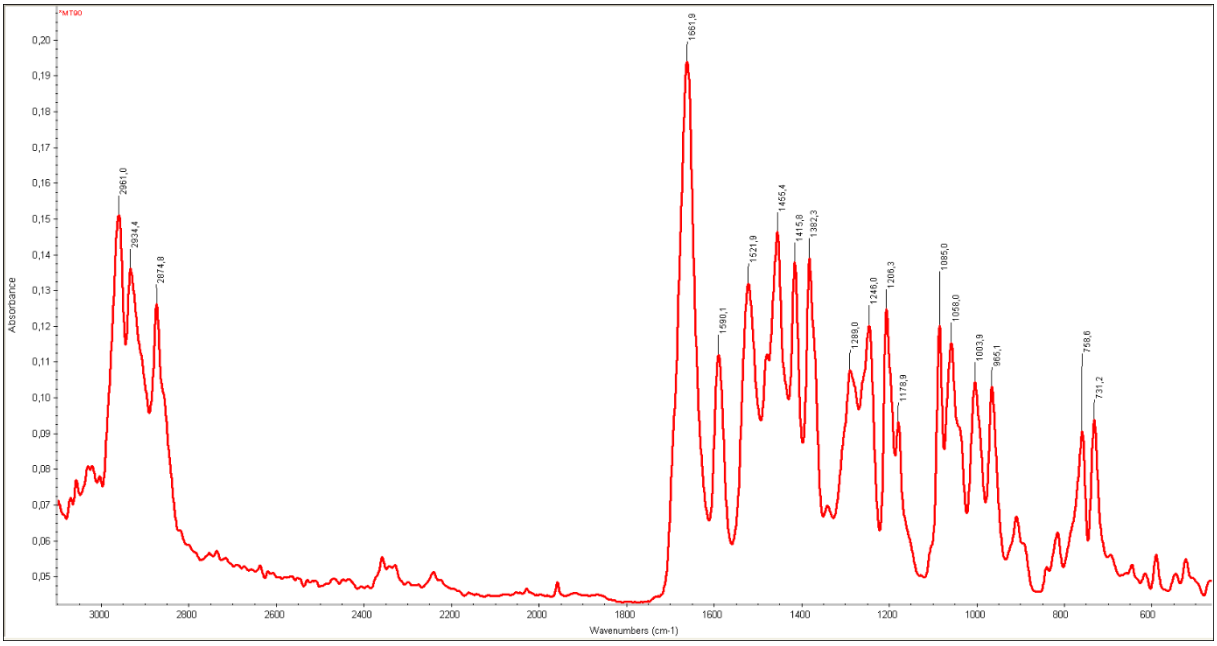
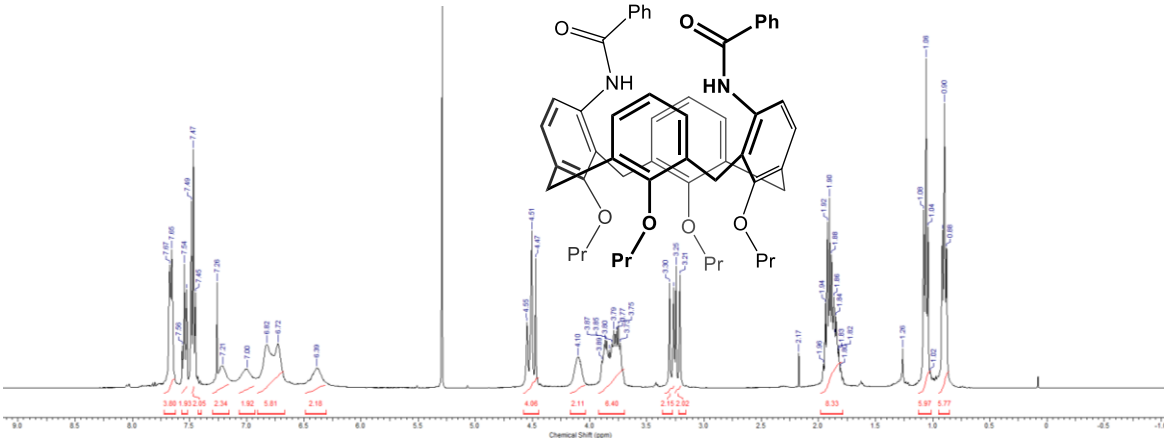


Figure 4: IR of compound **6a** (KBr).



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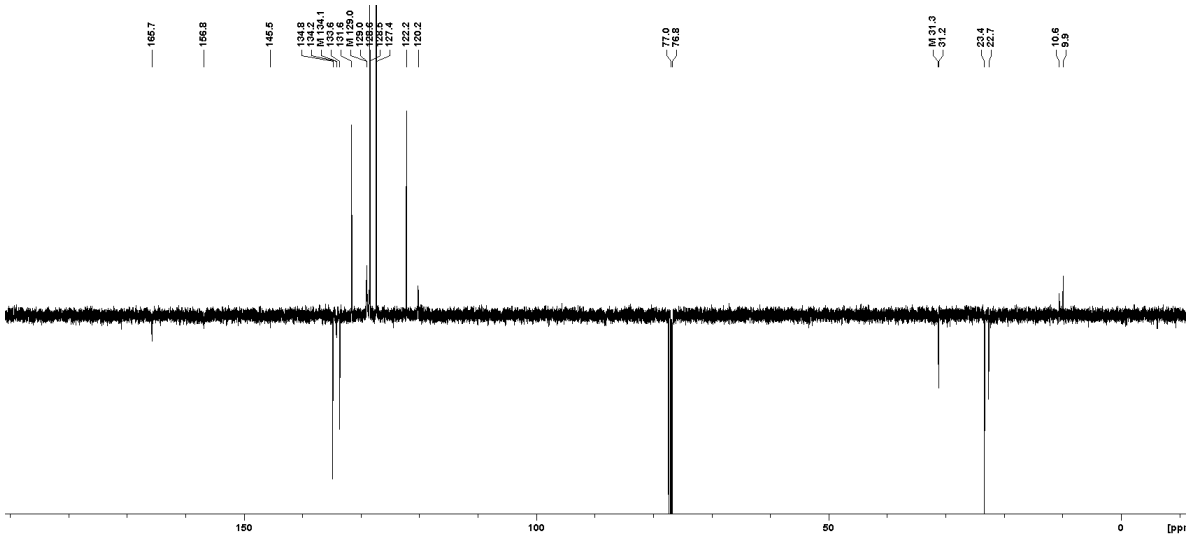

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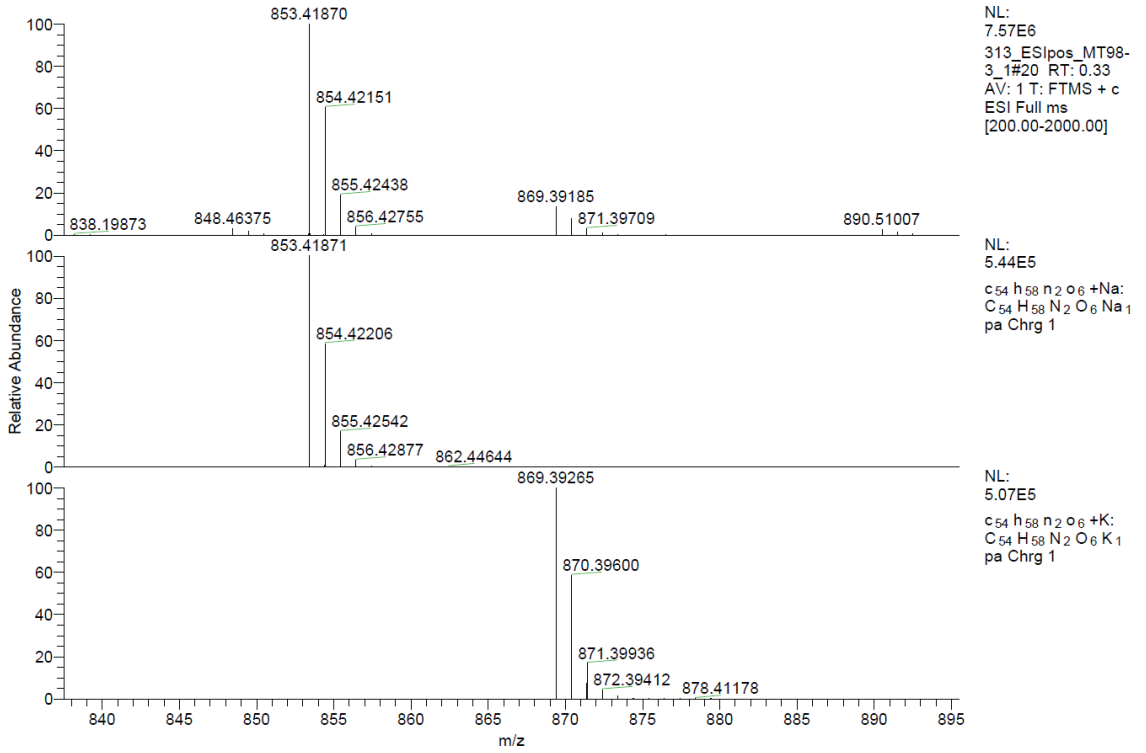

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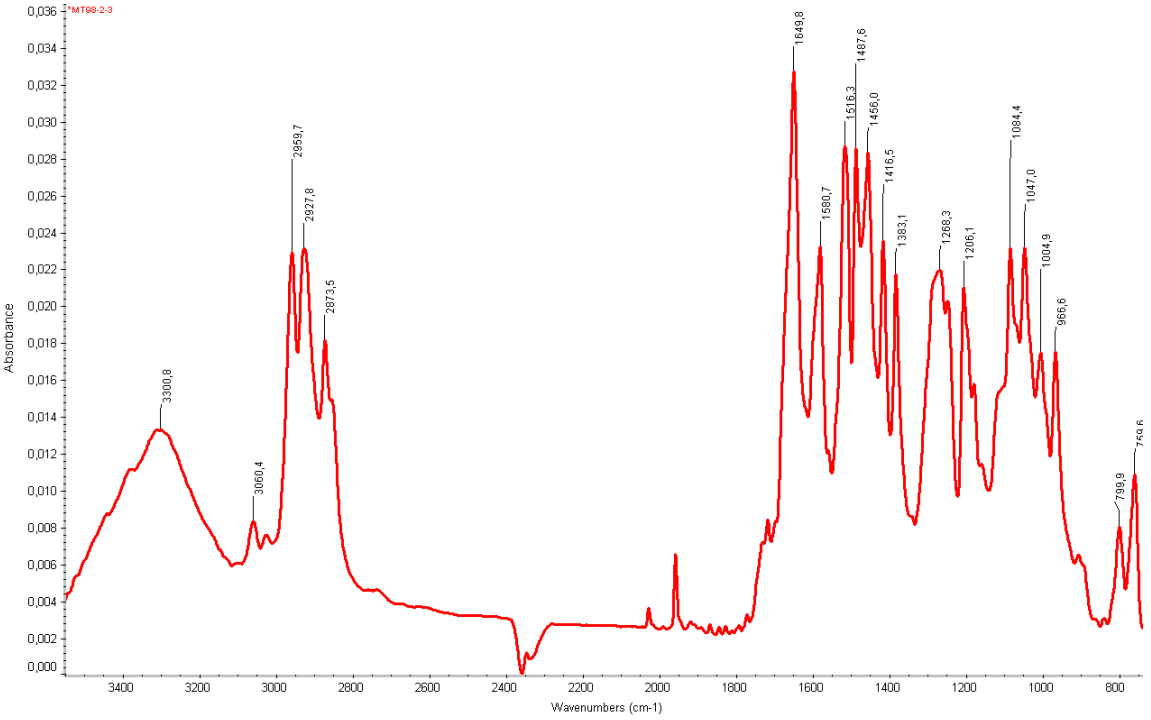

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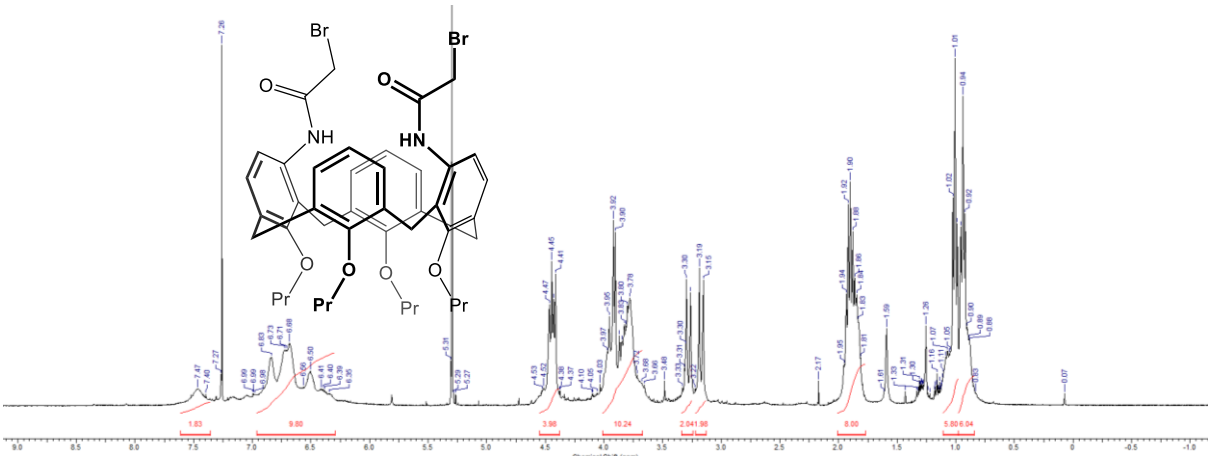

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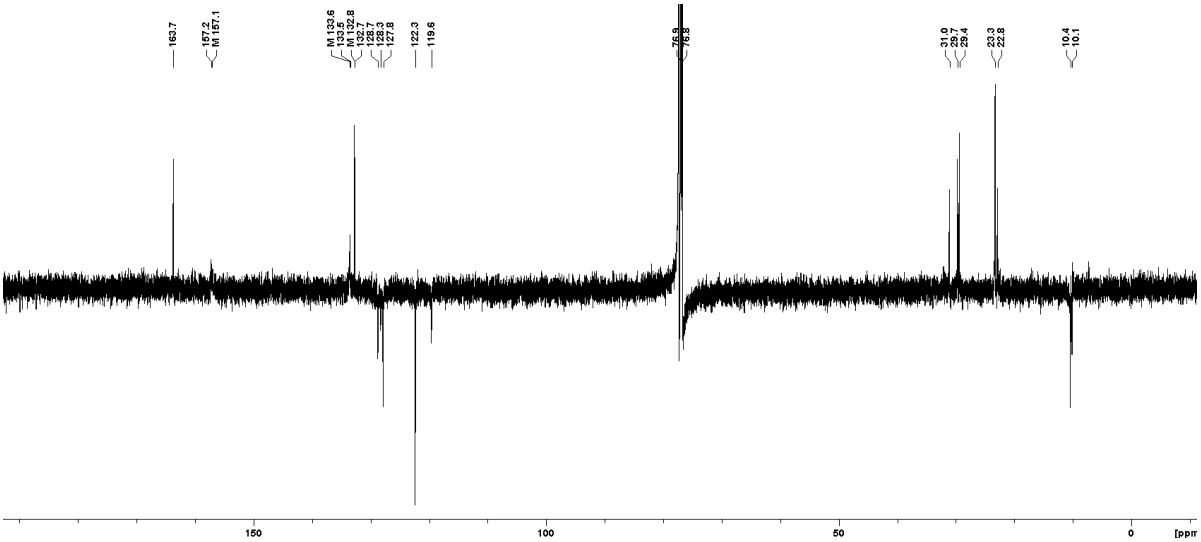
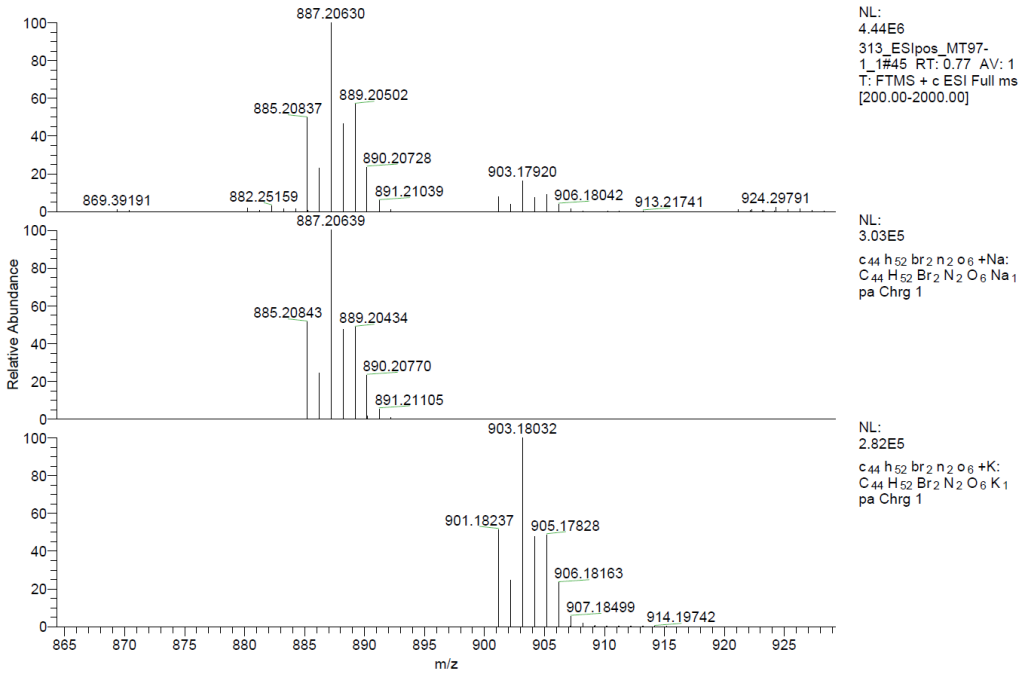


Figure 10: ^{13}C(APT) NMR of compound **6c** ($CDCl_3$, 100 MHz).



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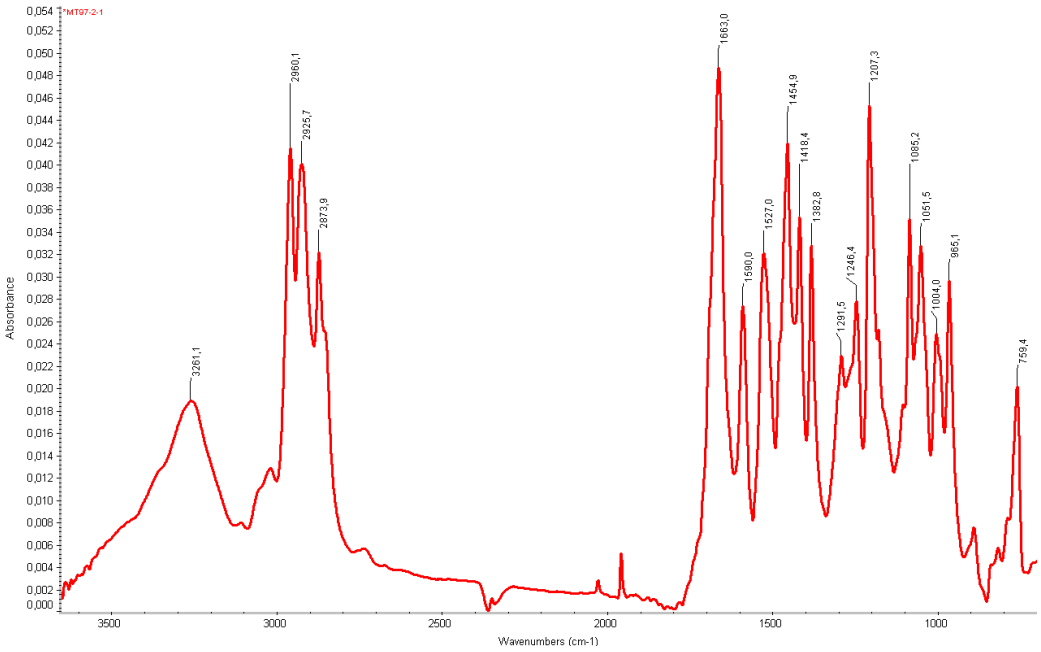

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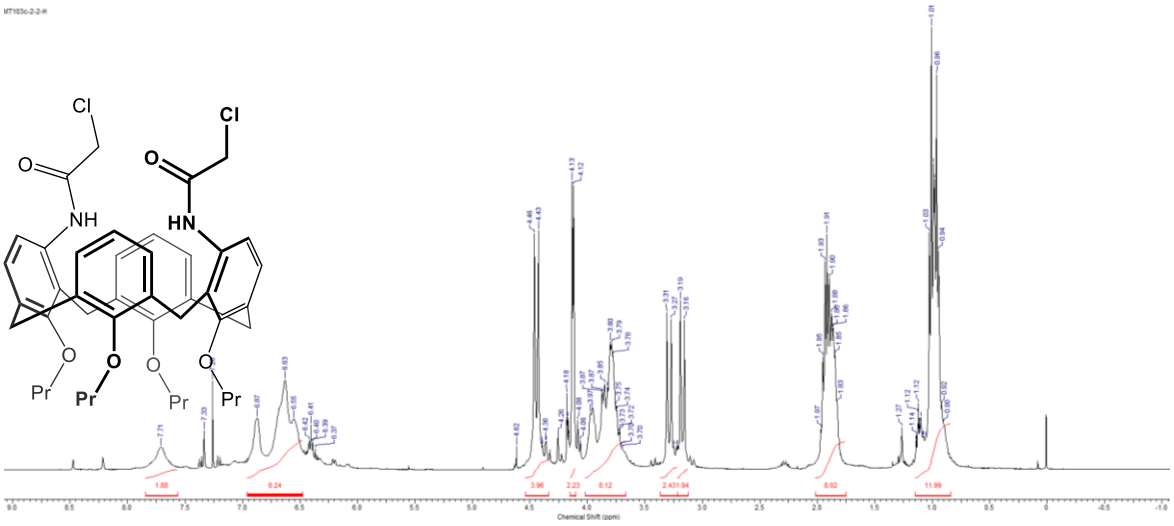

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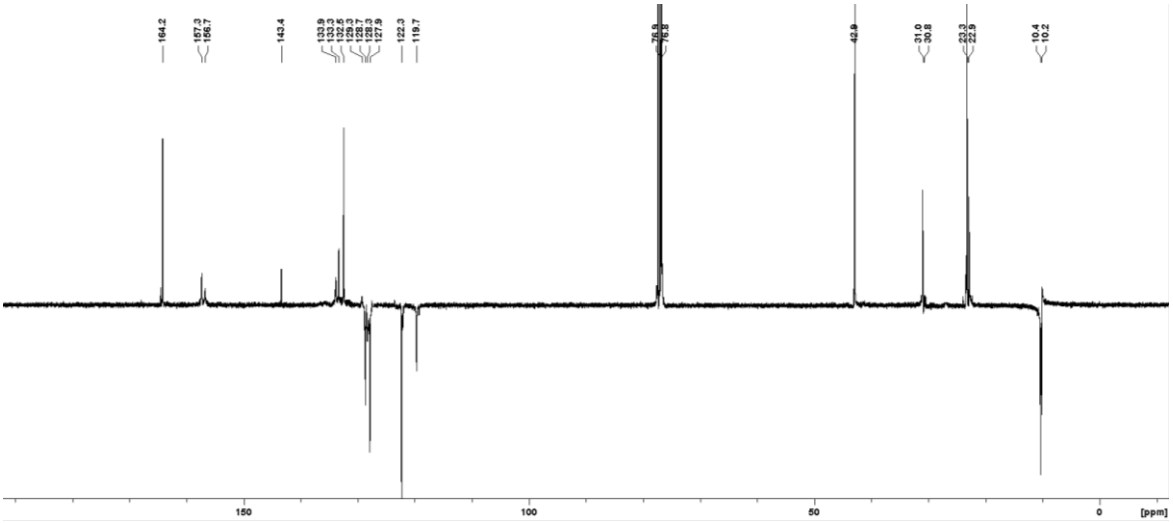

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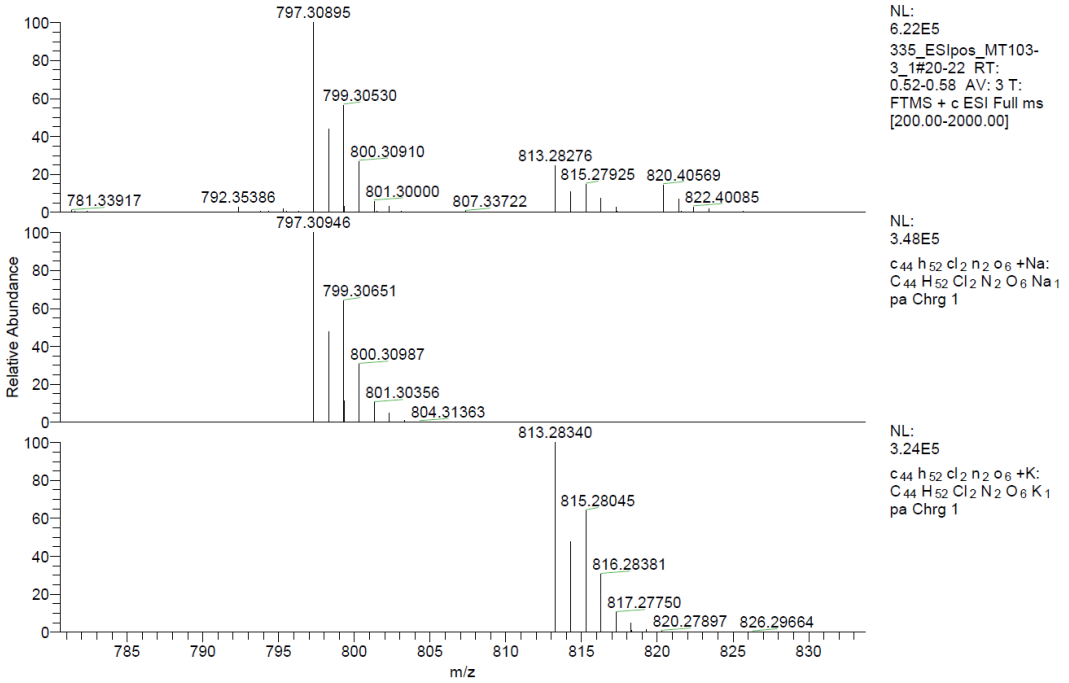

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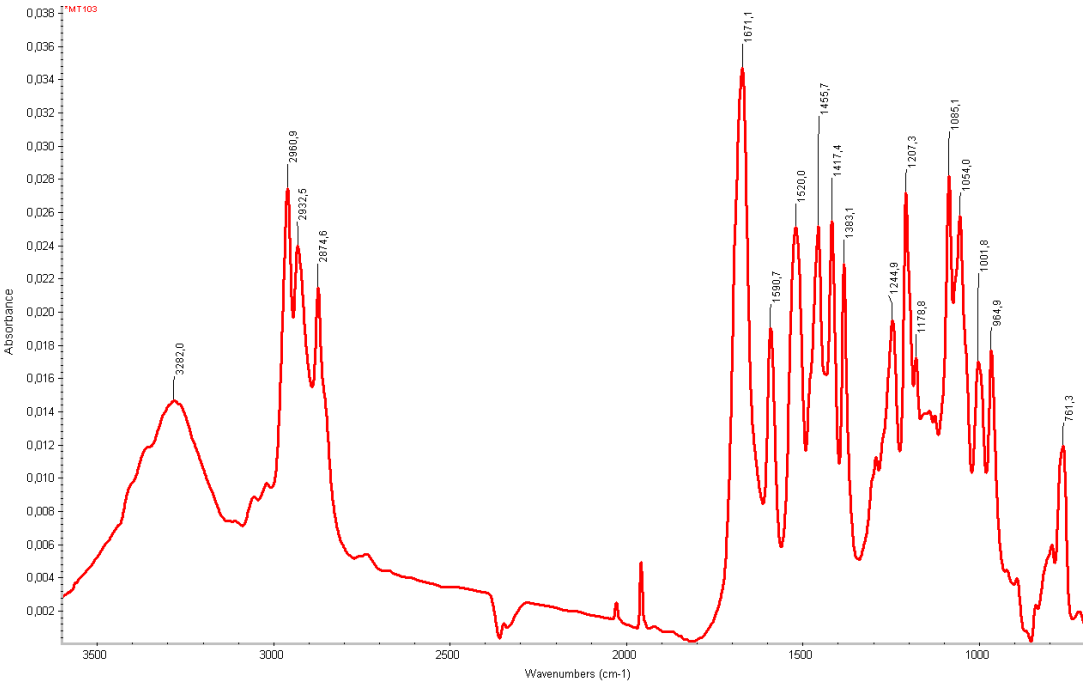

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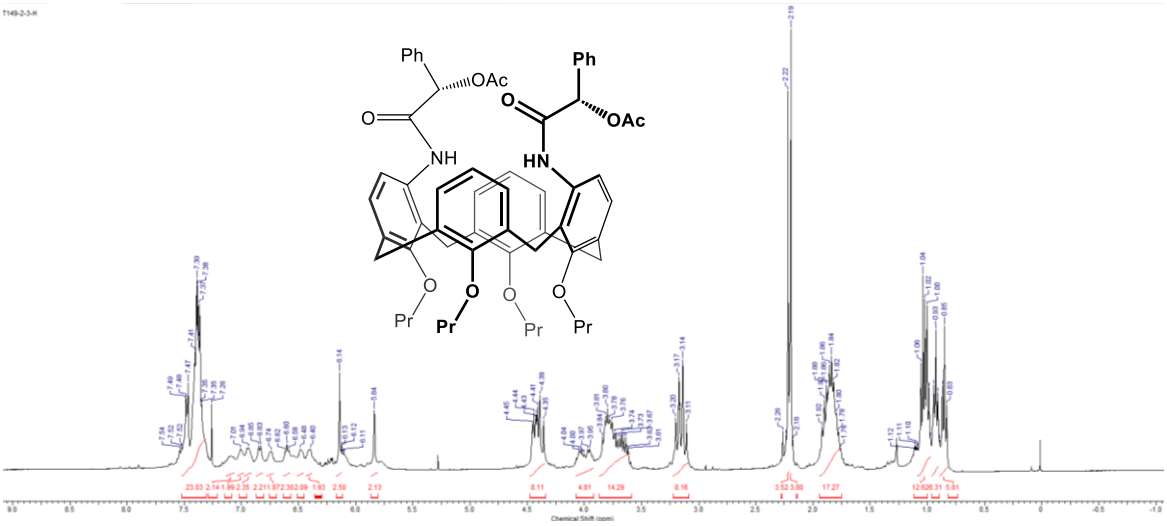

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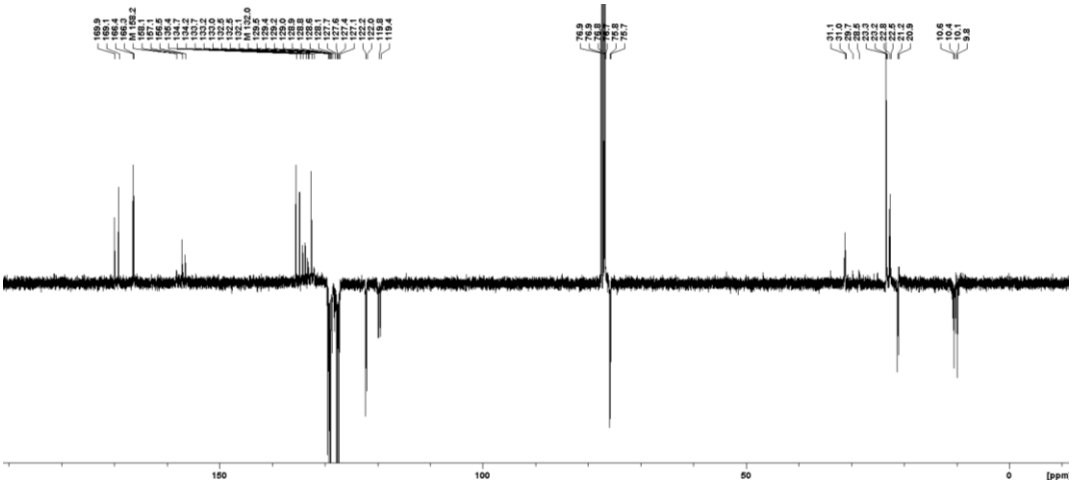

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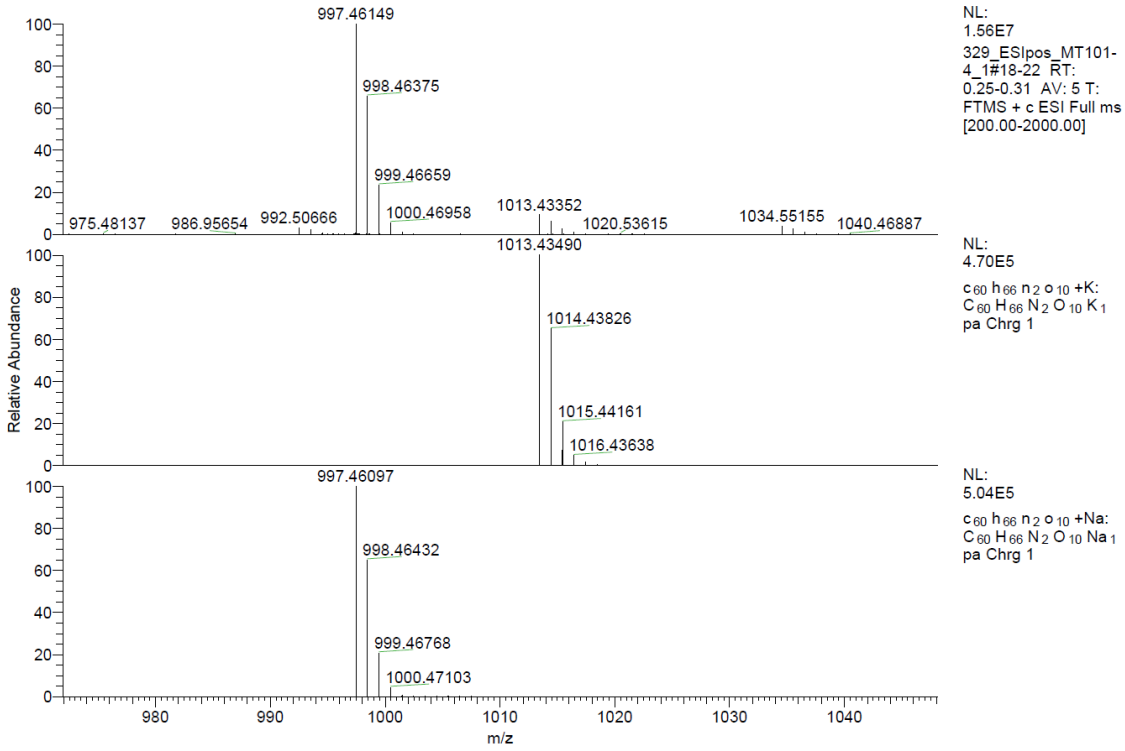

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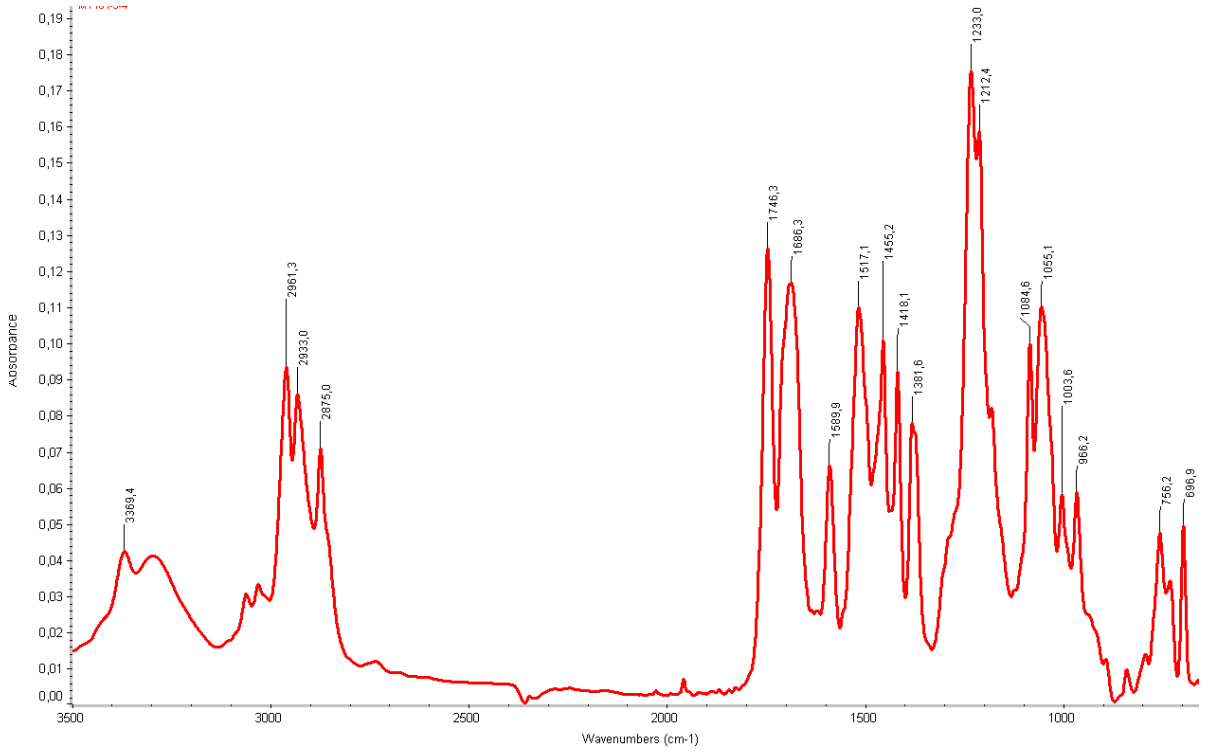

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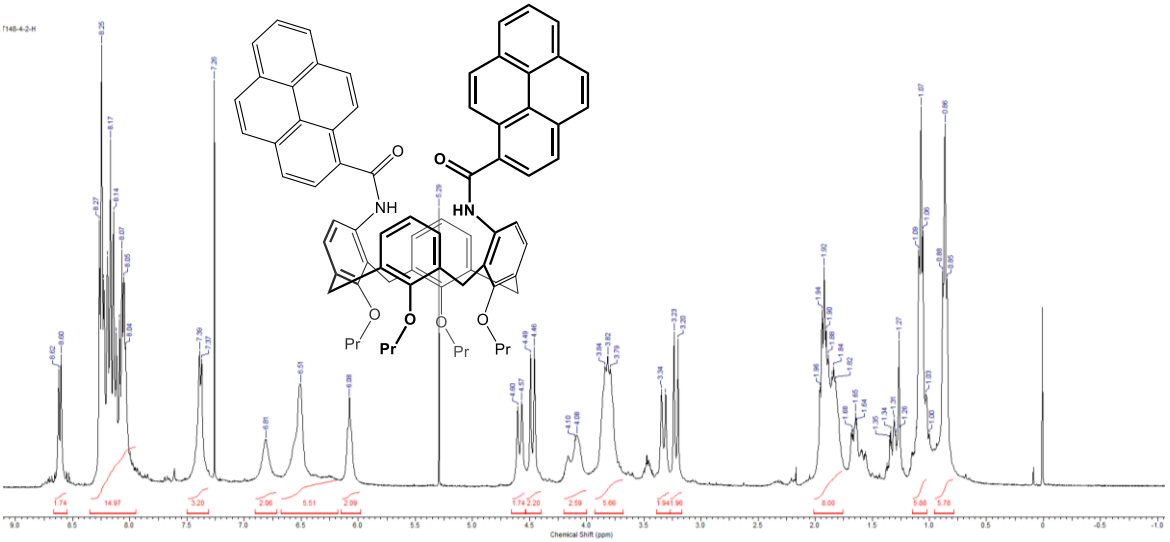
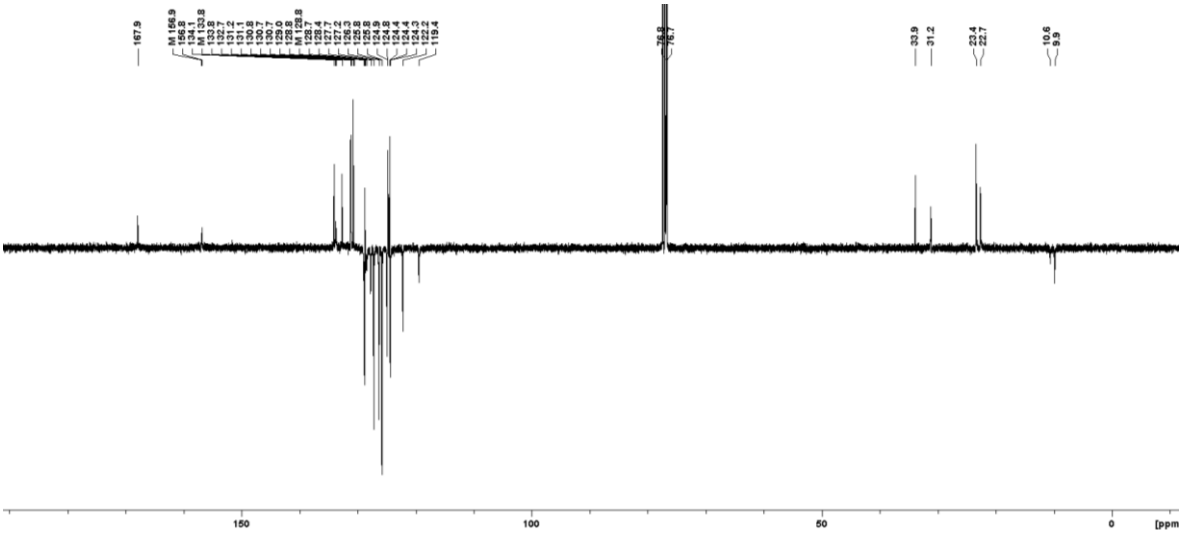


Figure 21: ^{1}H NMR of compound **6f** ($CDCl_3$, 400 MHz).



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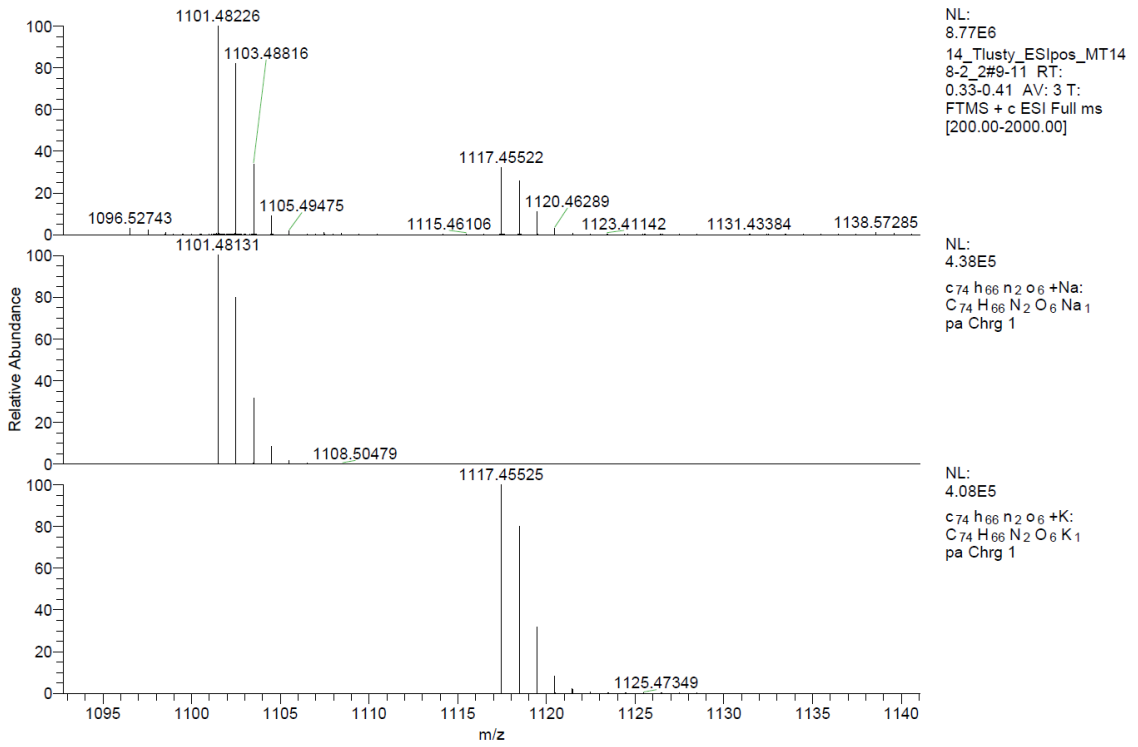

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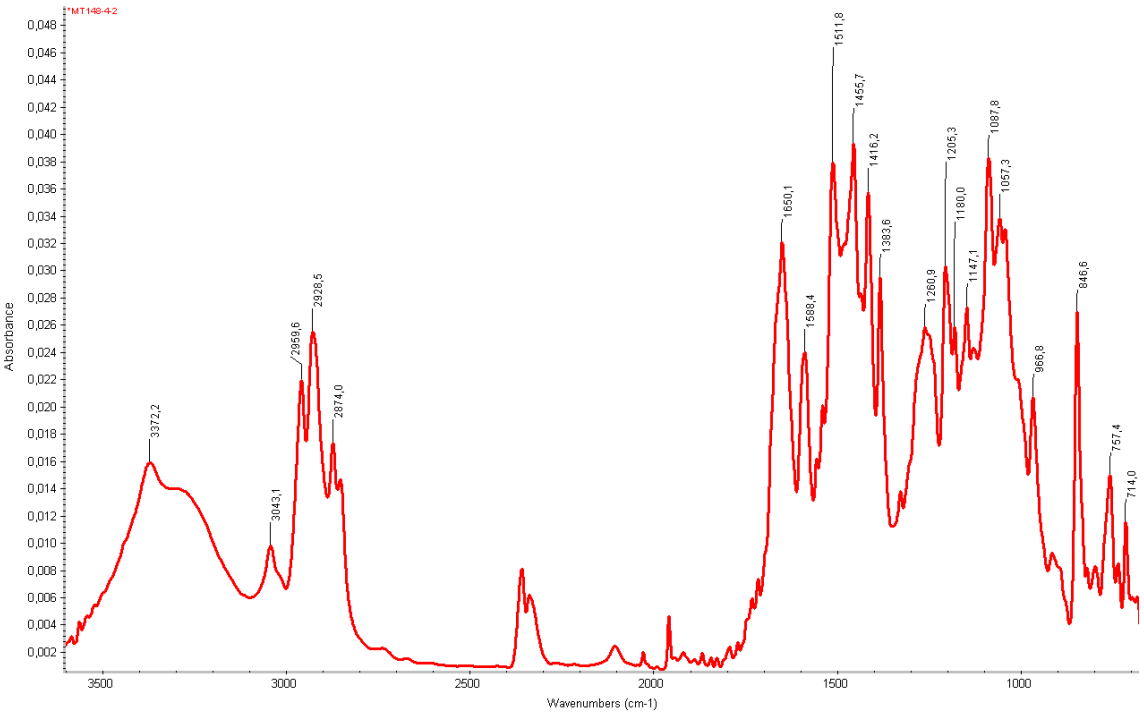

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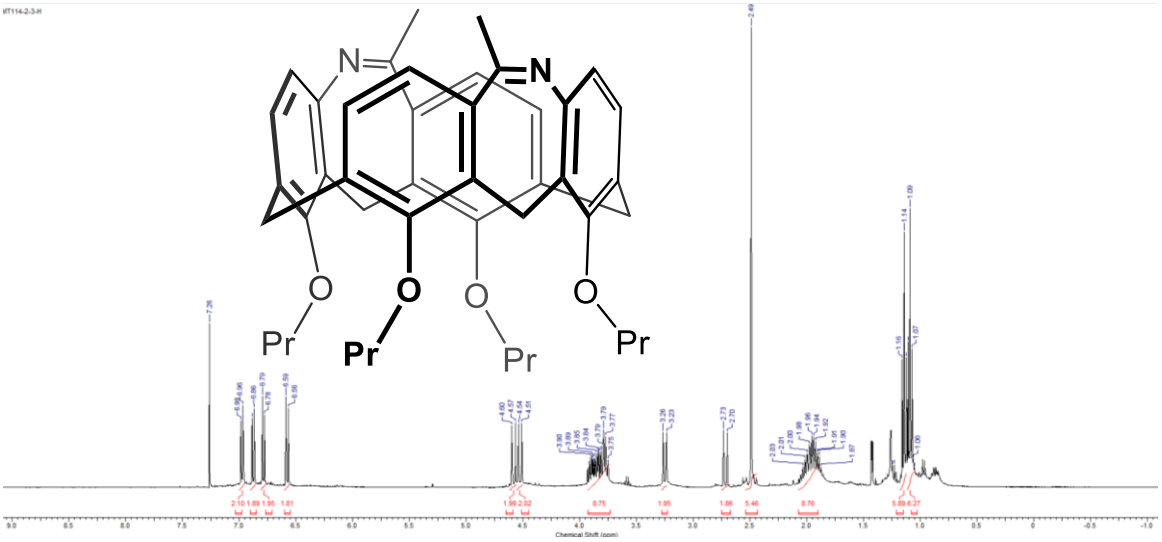

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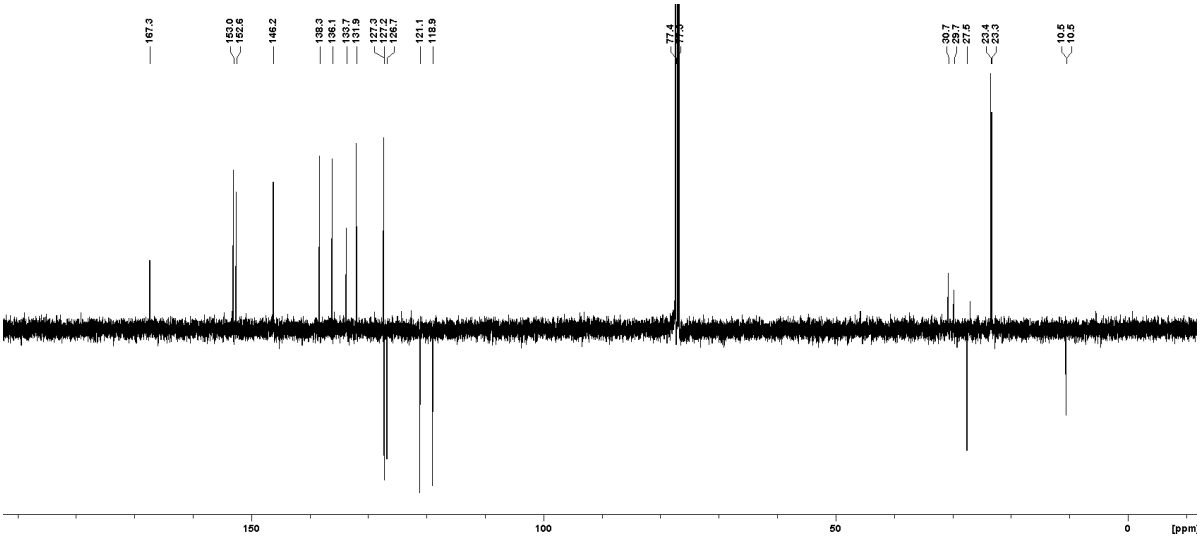

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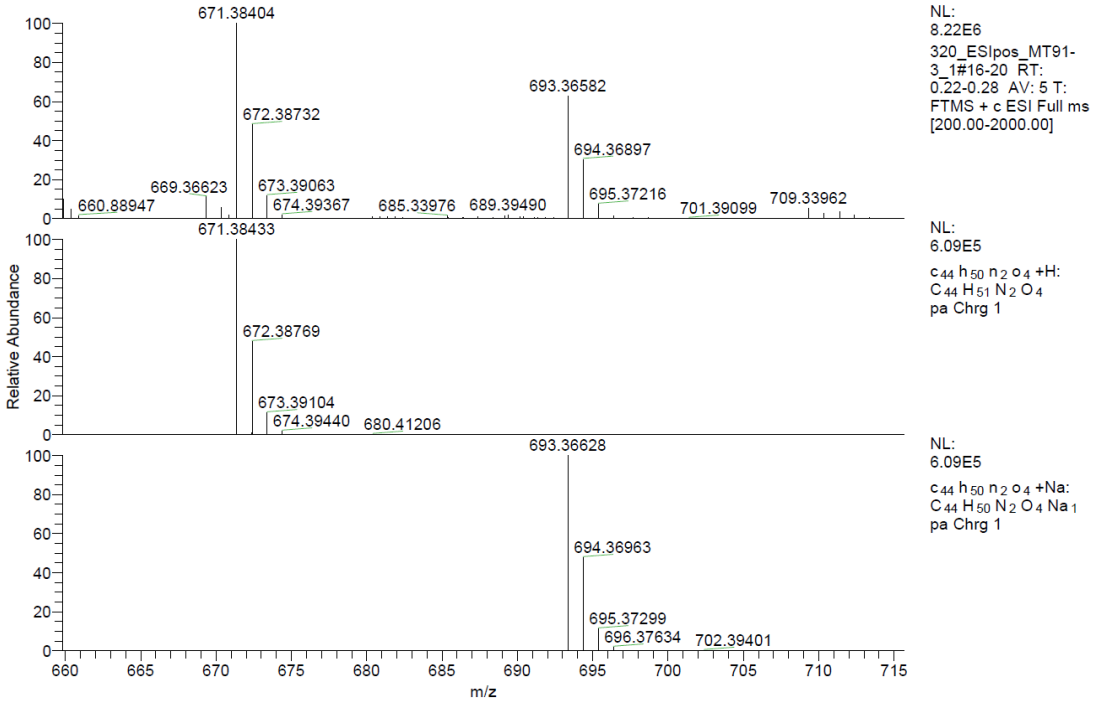

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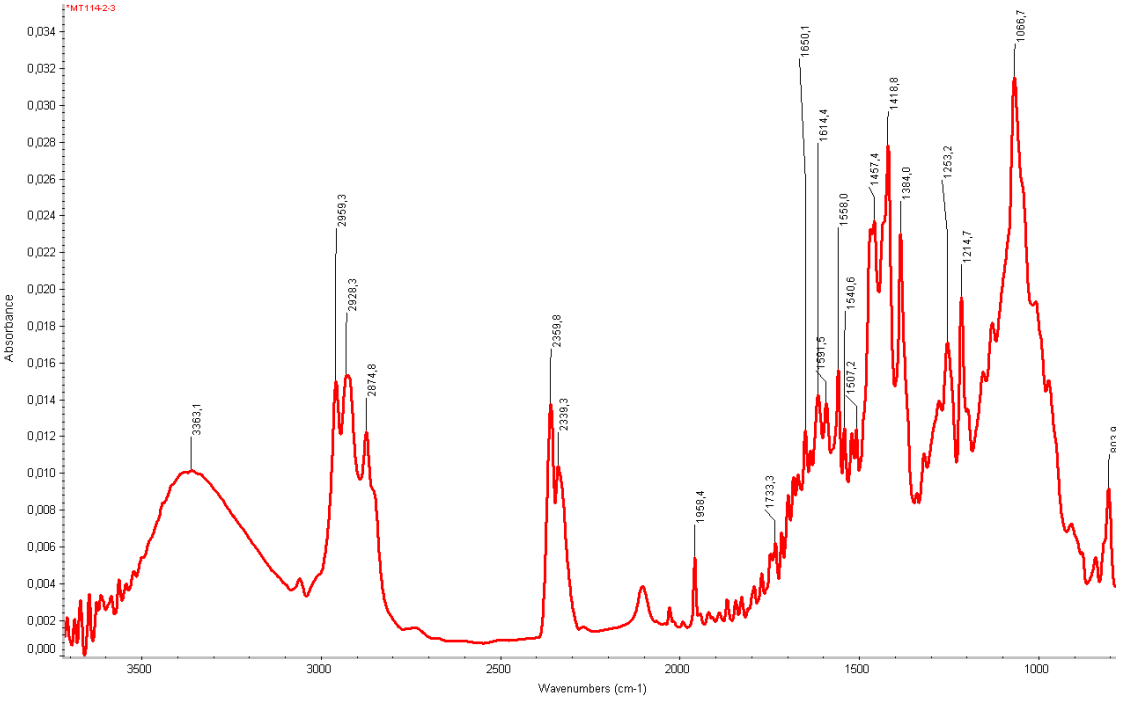

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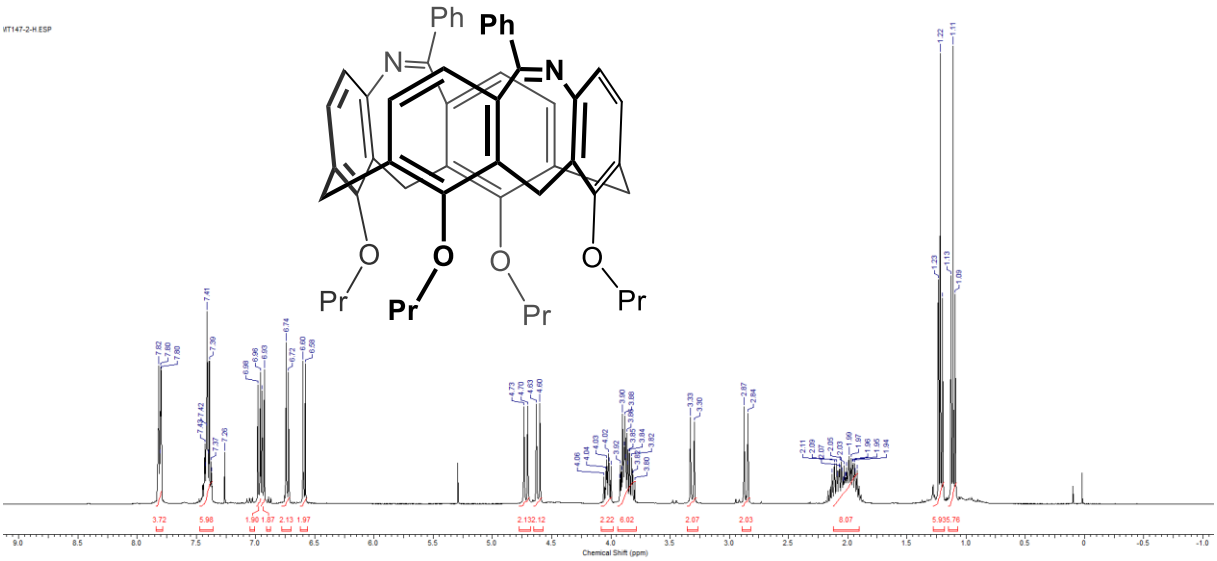

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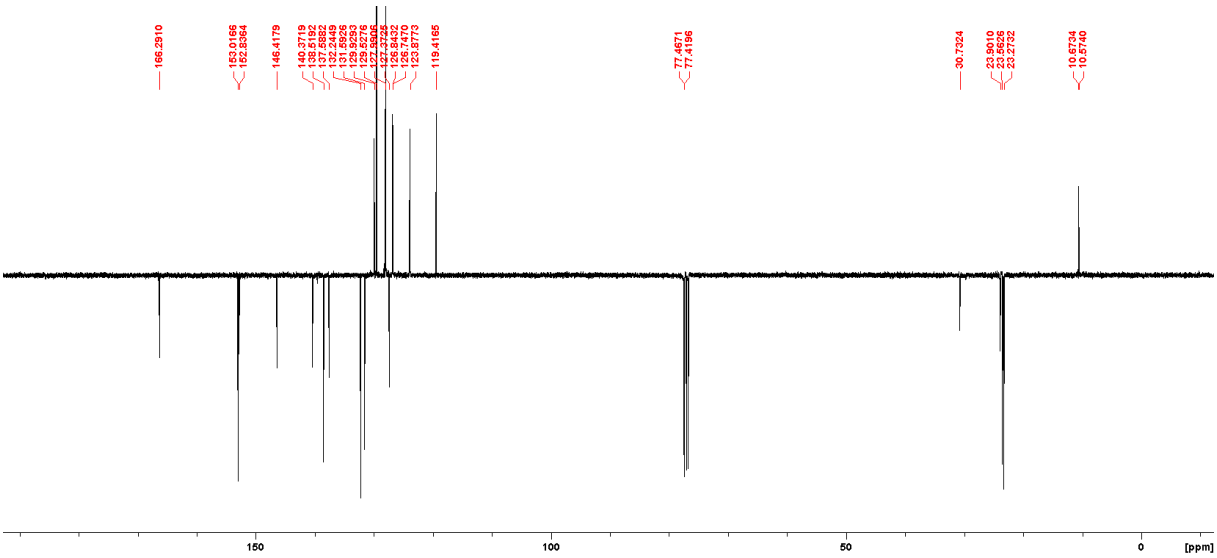

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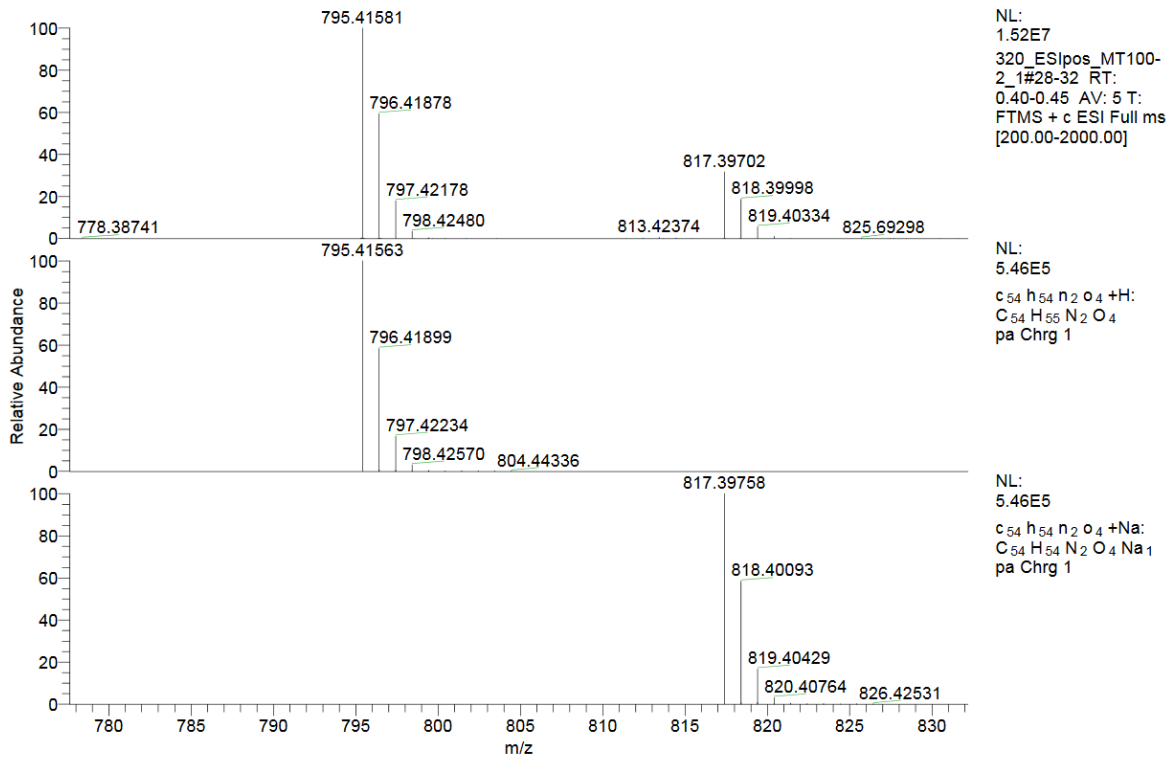

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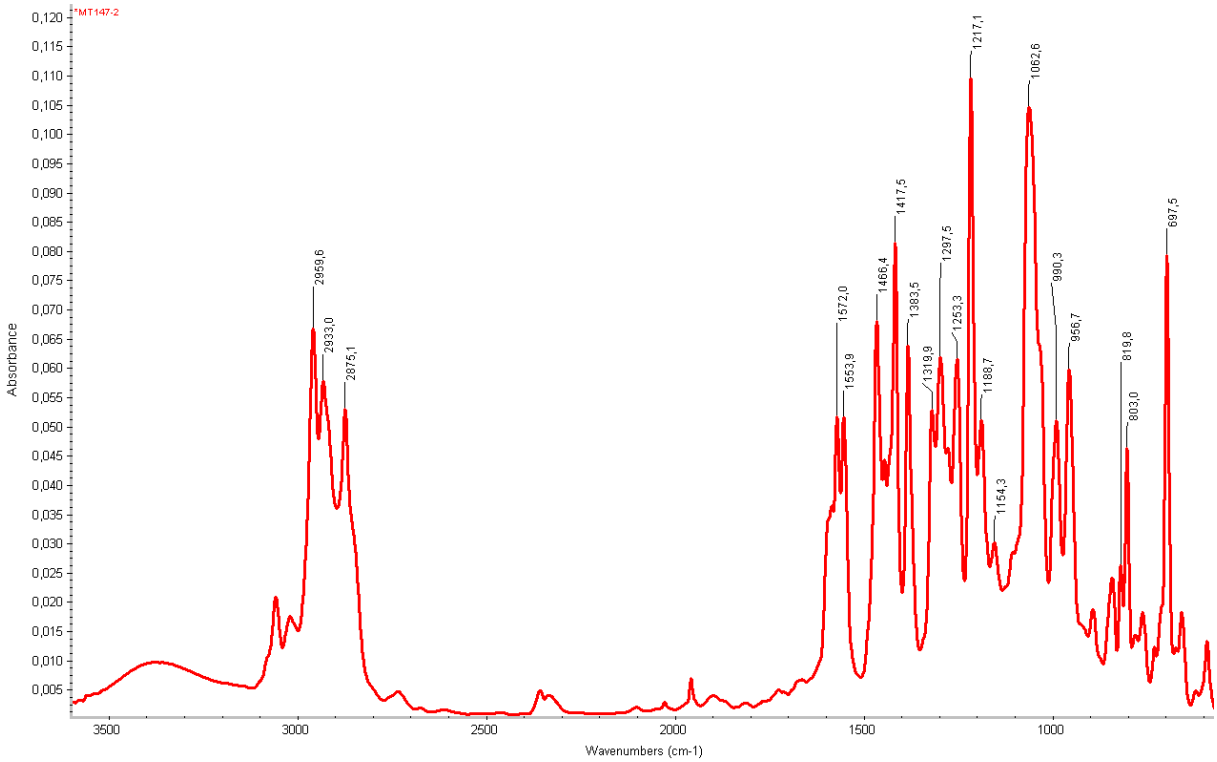

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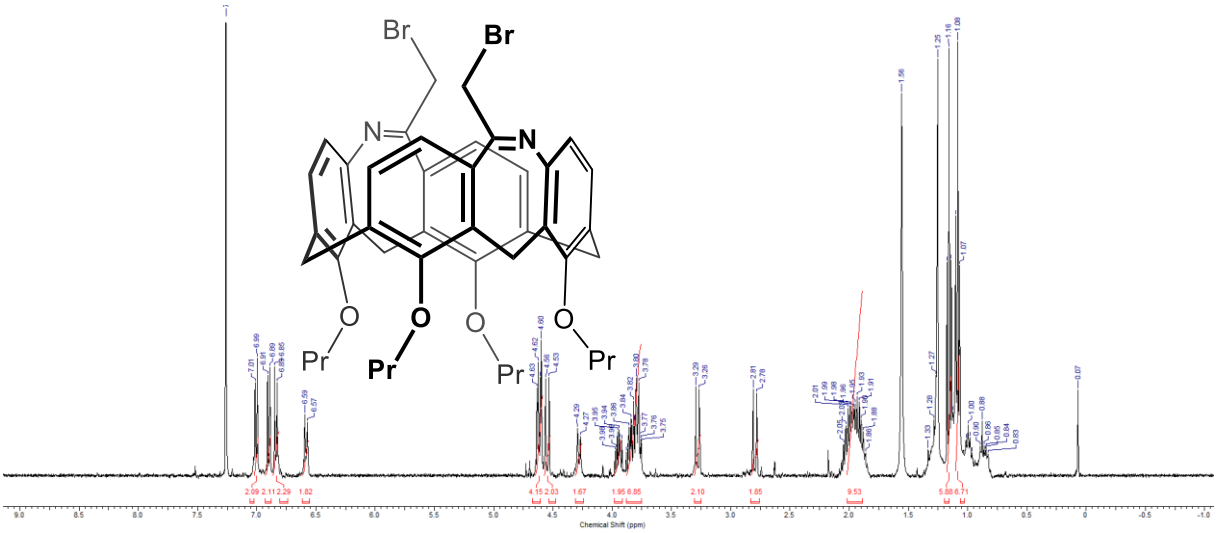

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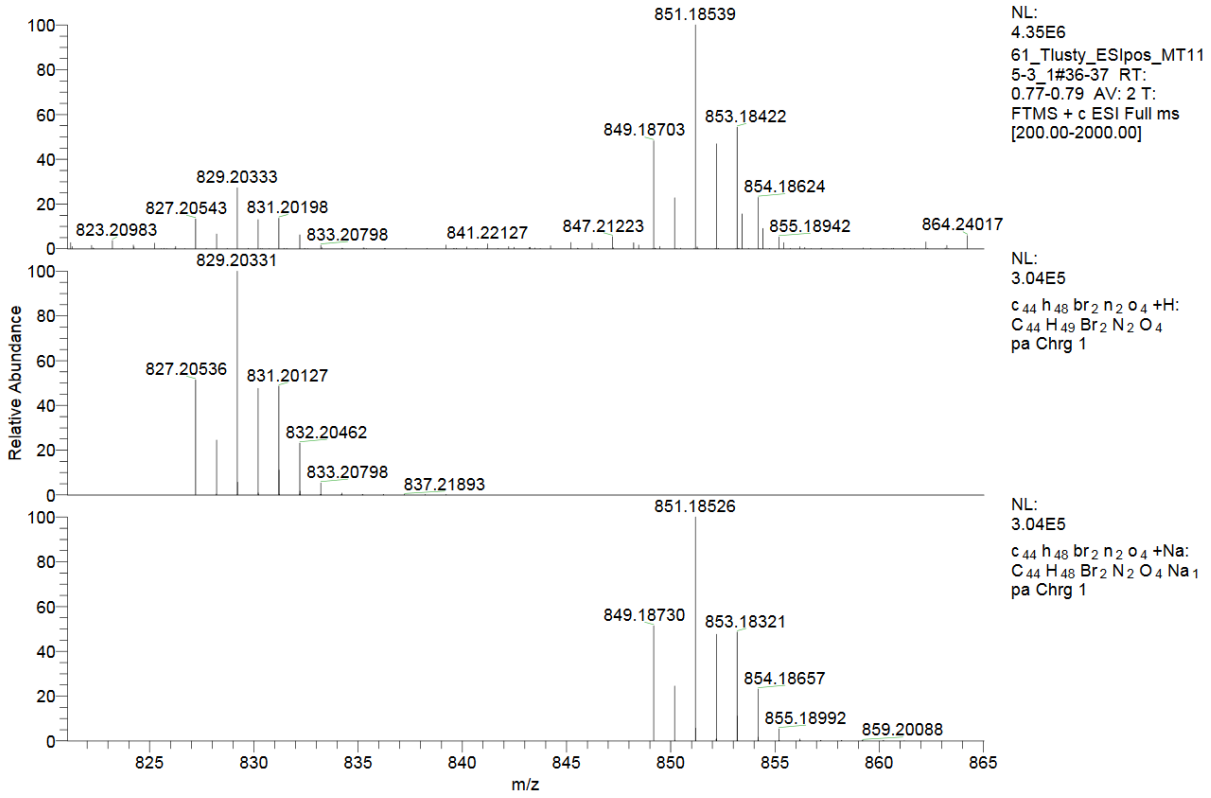

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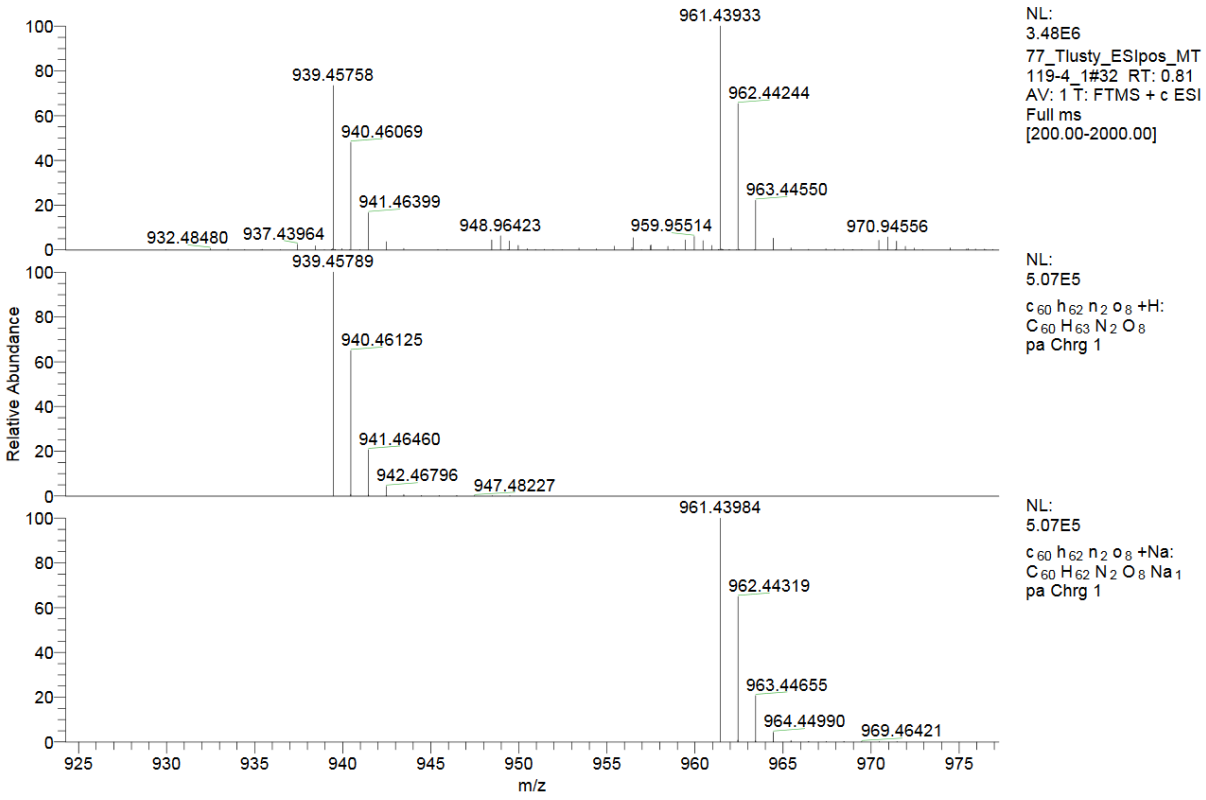

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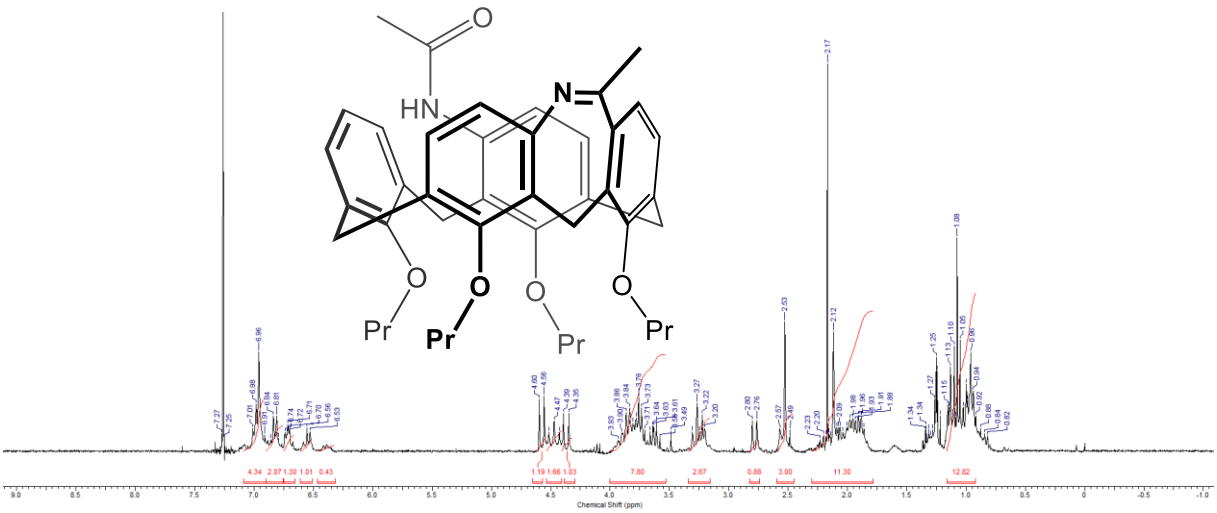

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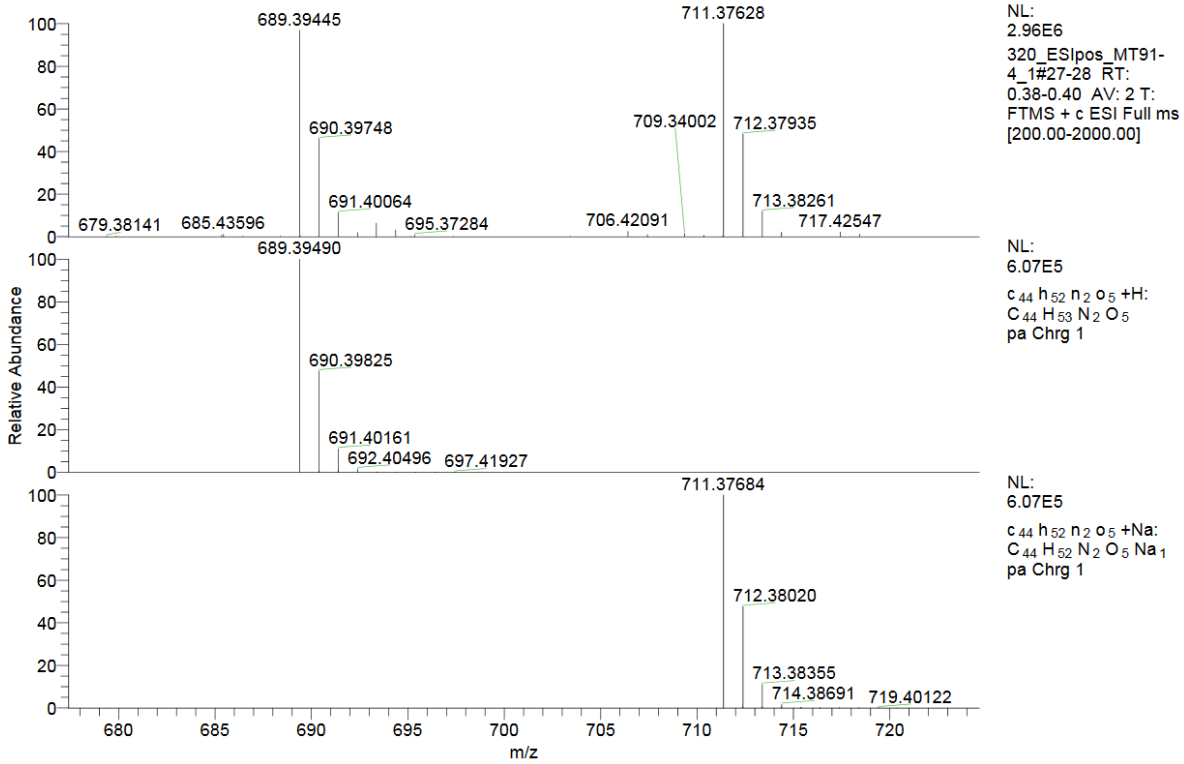

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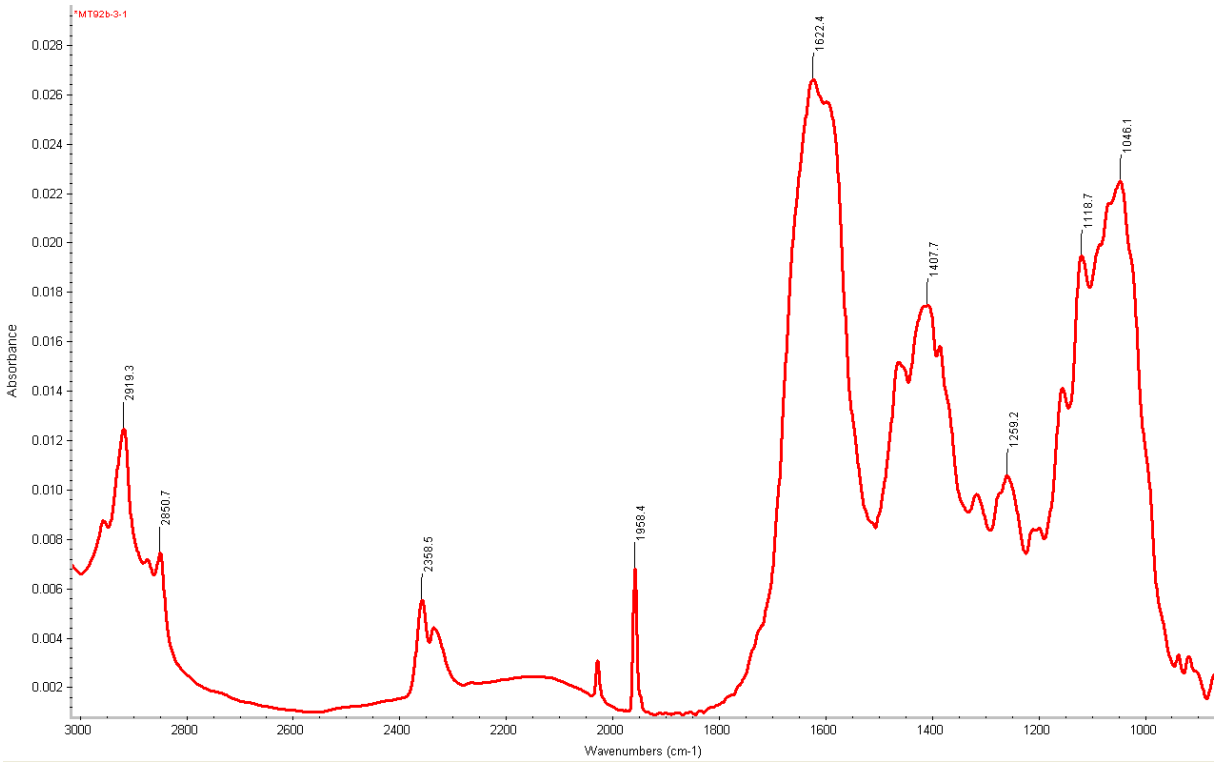
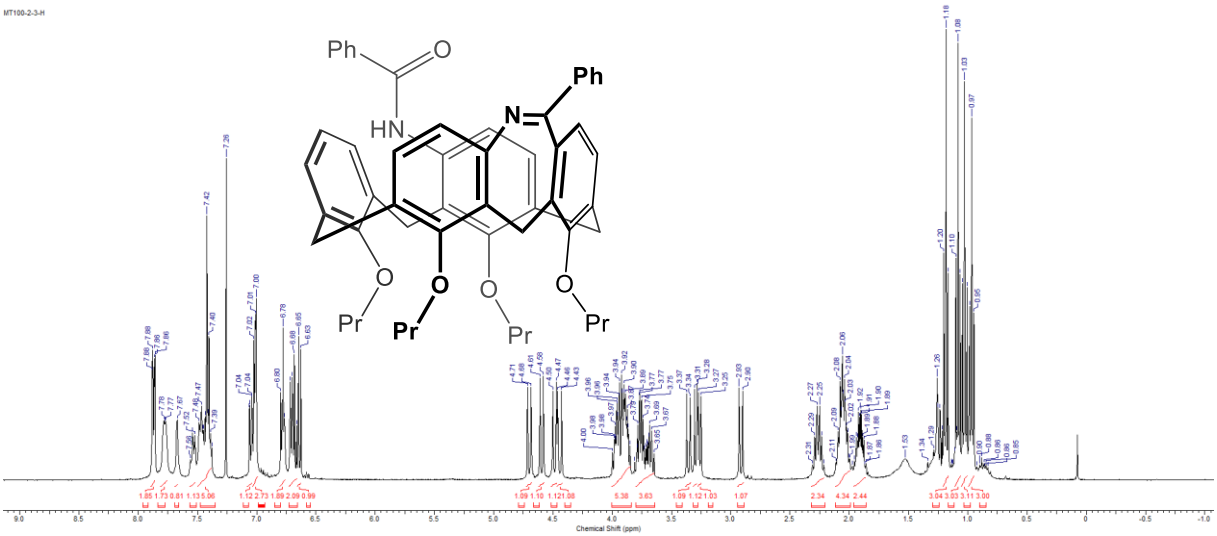


Figure 38: IR of compound **8a** (KBr).



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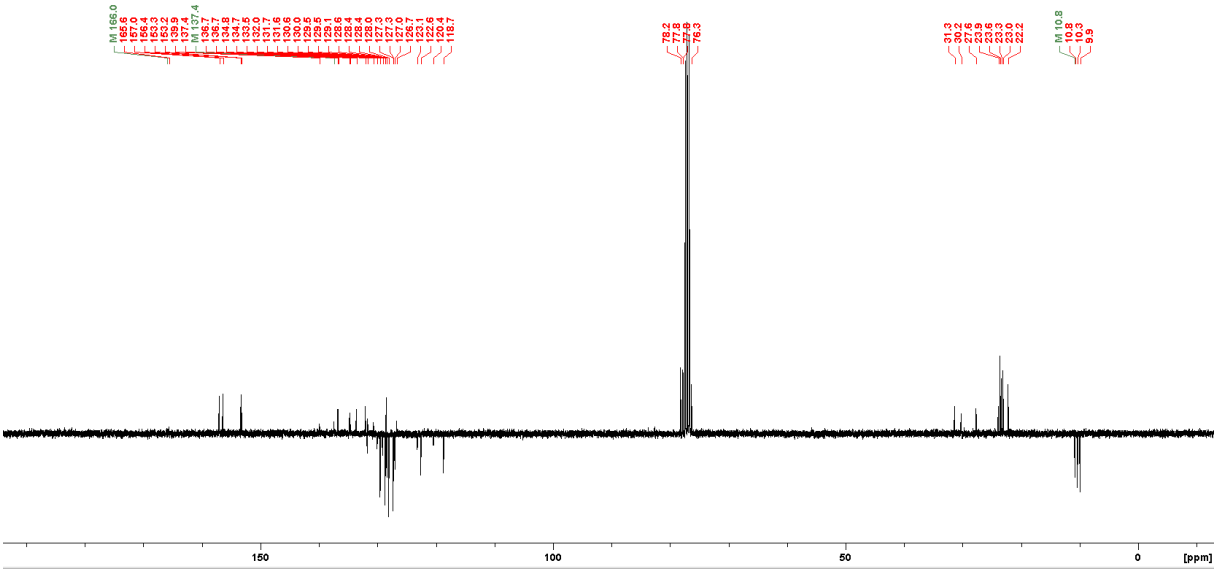
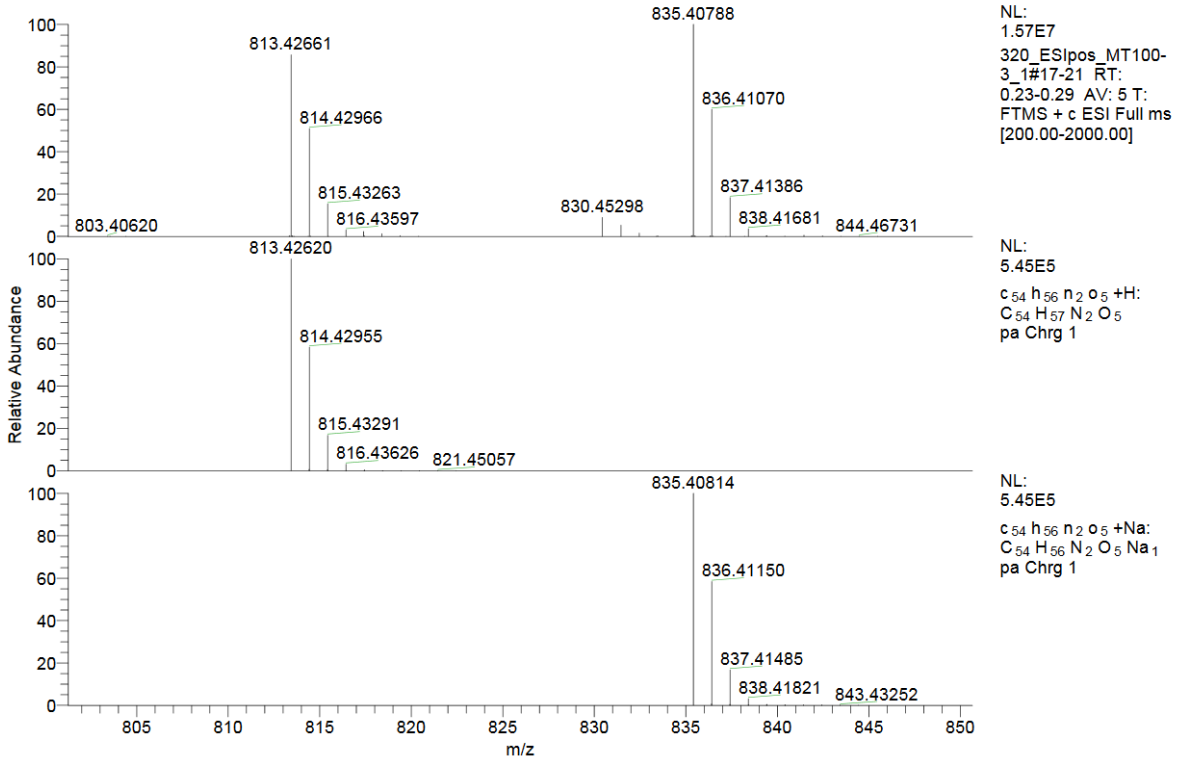


Figure 40: ^{13}C(APT) NMR of compound **8b** ($CDCl_3$, 100 MHz).



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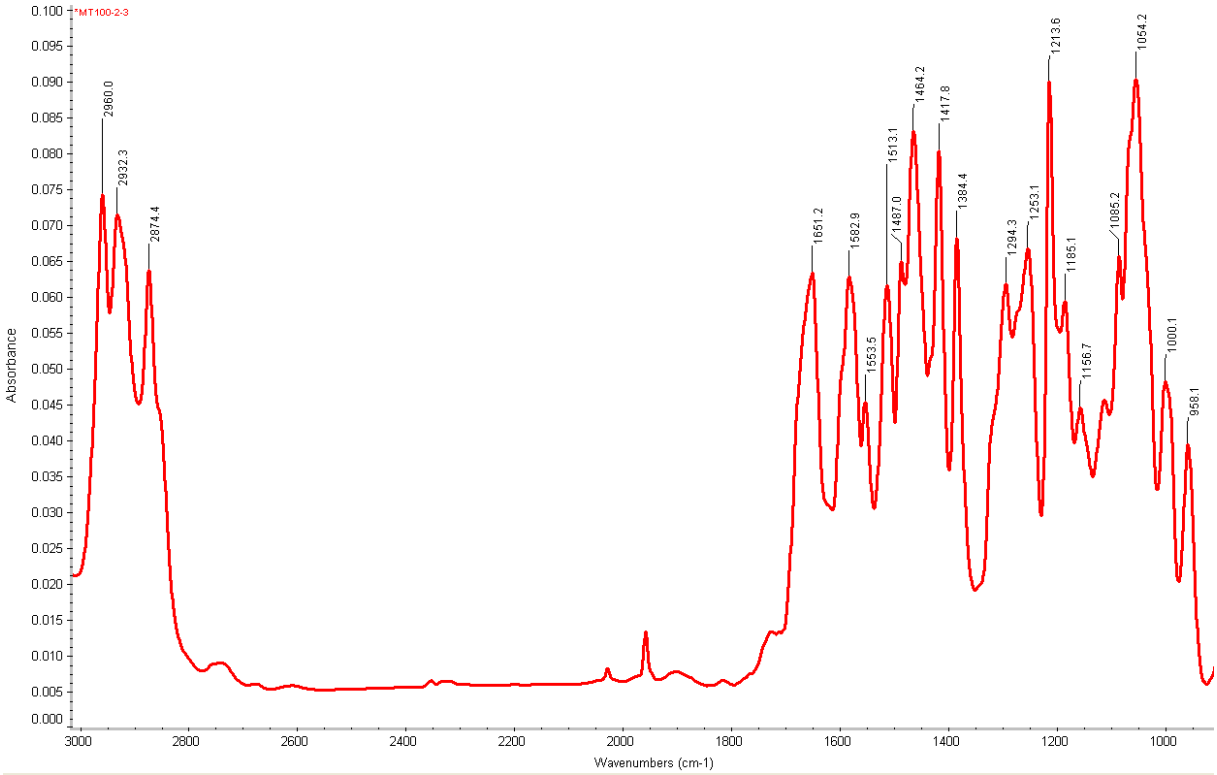


Figure 42: IR of compound **8b** (KBr).

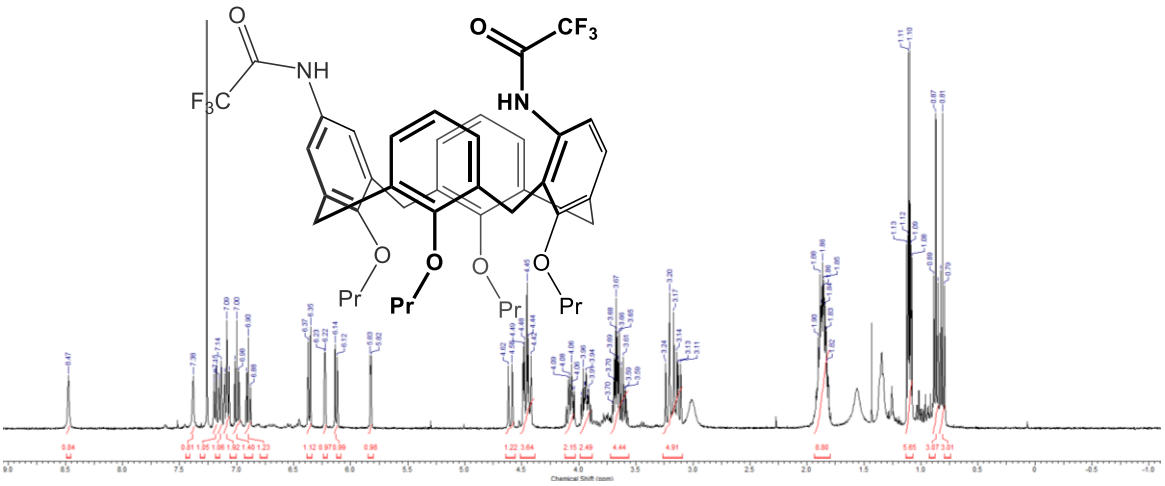


Figure 43: ^{1}H NMR of compound **11** ($CDCl_3$, 400 MHz).

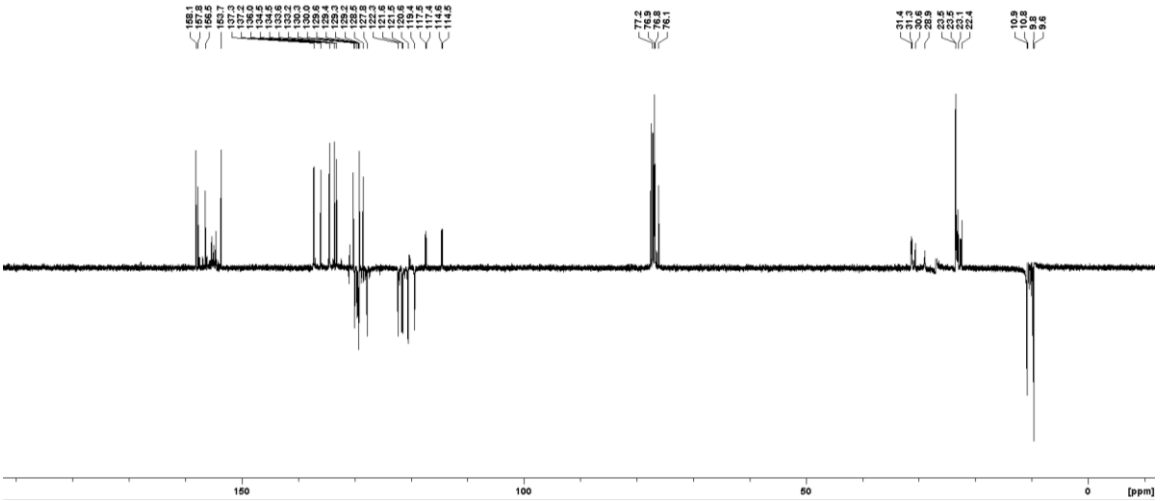


Figure 44: ^{13}C(APT) NMR of compound **11** ($CDCl_3$, 100 MHz).

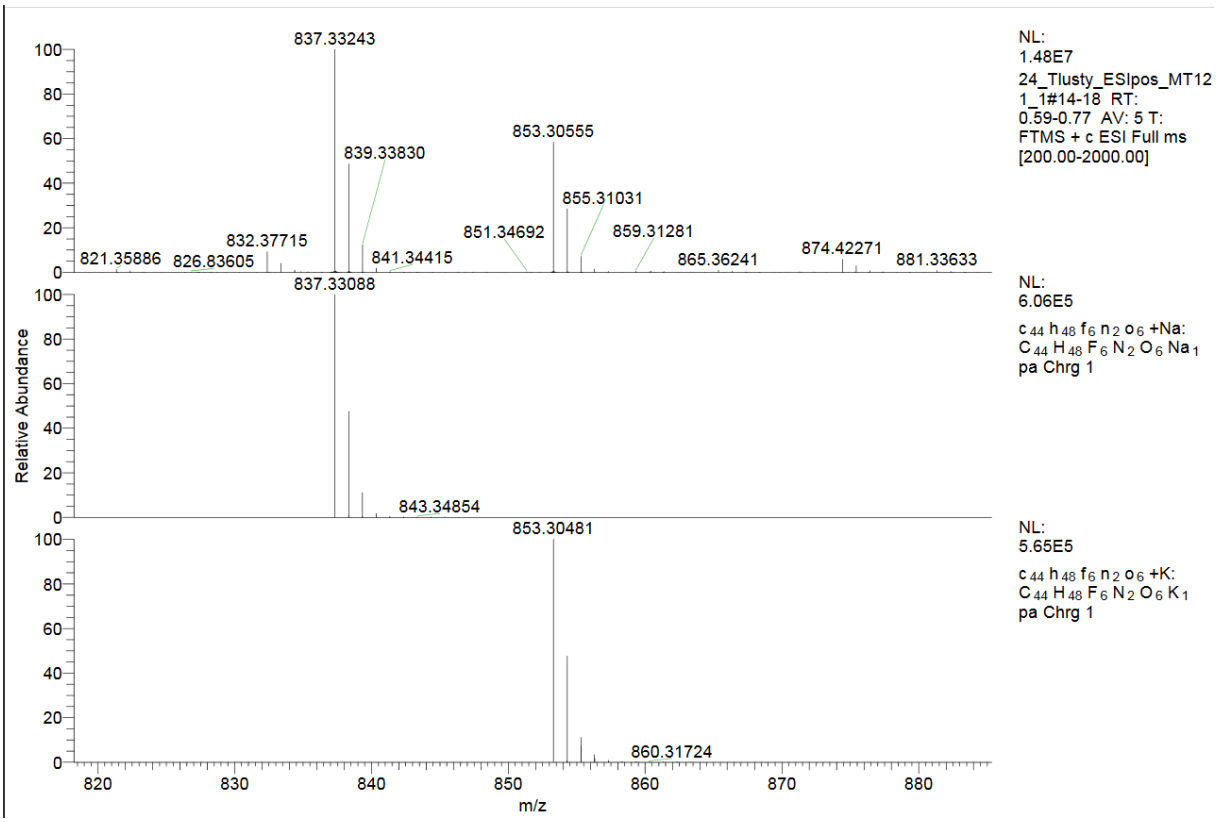


Figure 45: HRMS of compound **11** (ESI+).

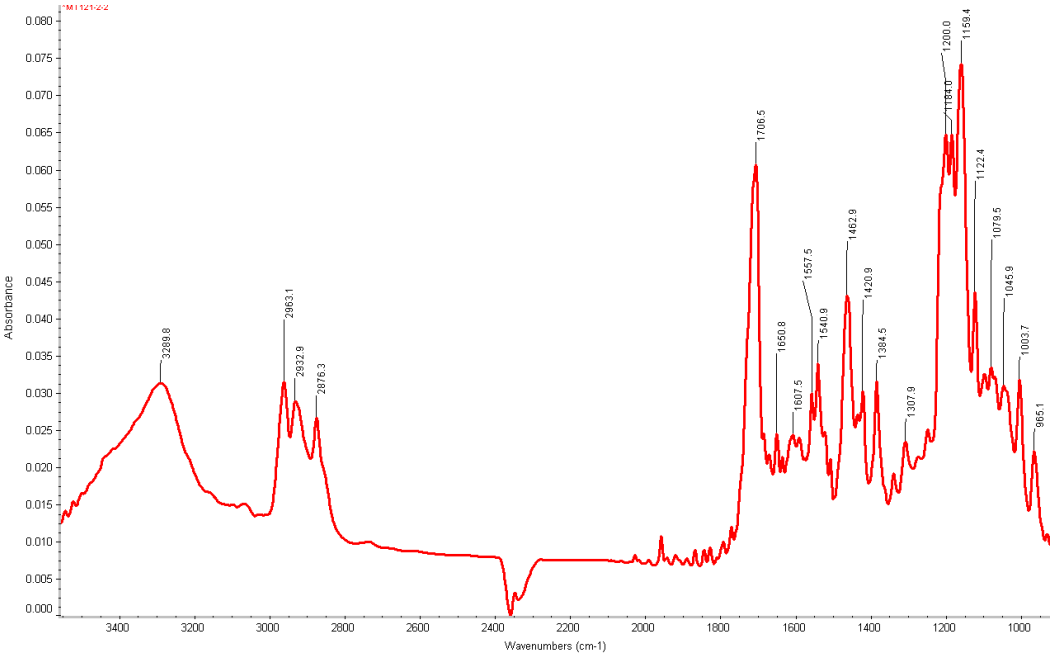


Figure 46: IR of compound **11** (KBr).

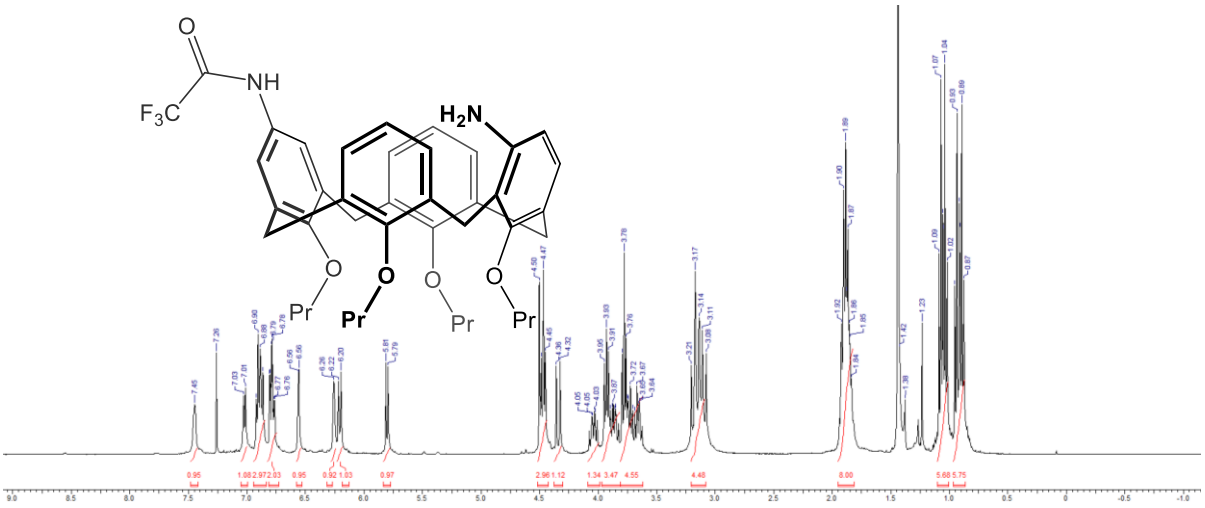


Figure 47: ^{1}H NMR of compound **12a** ($CDCl_3$, 400 MHz).

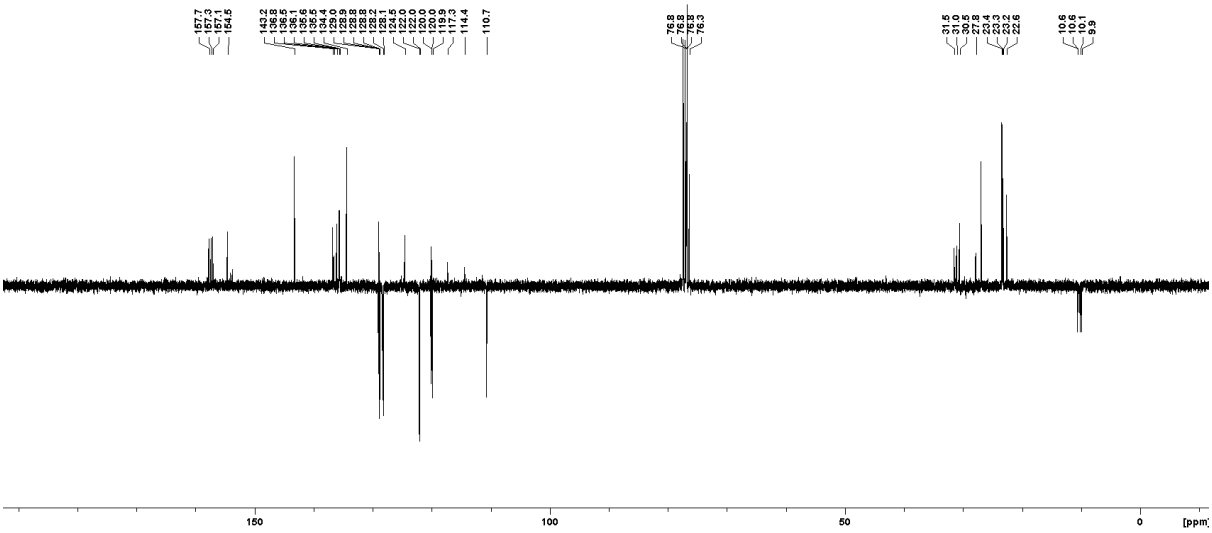


Figure 48: ^{13}C(APT) NMR of compound **12a** ($CDCl_3$, 100 MHz).

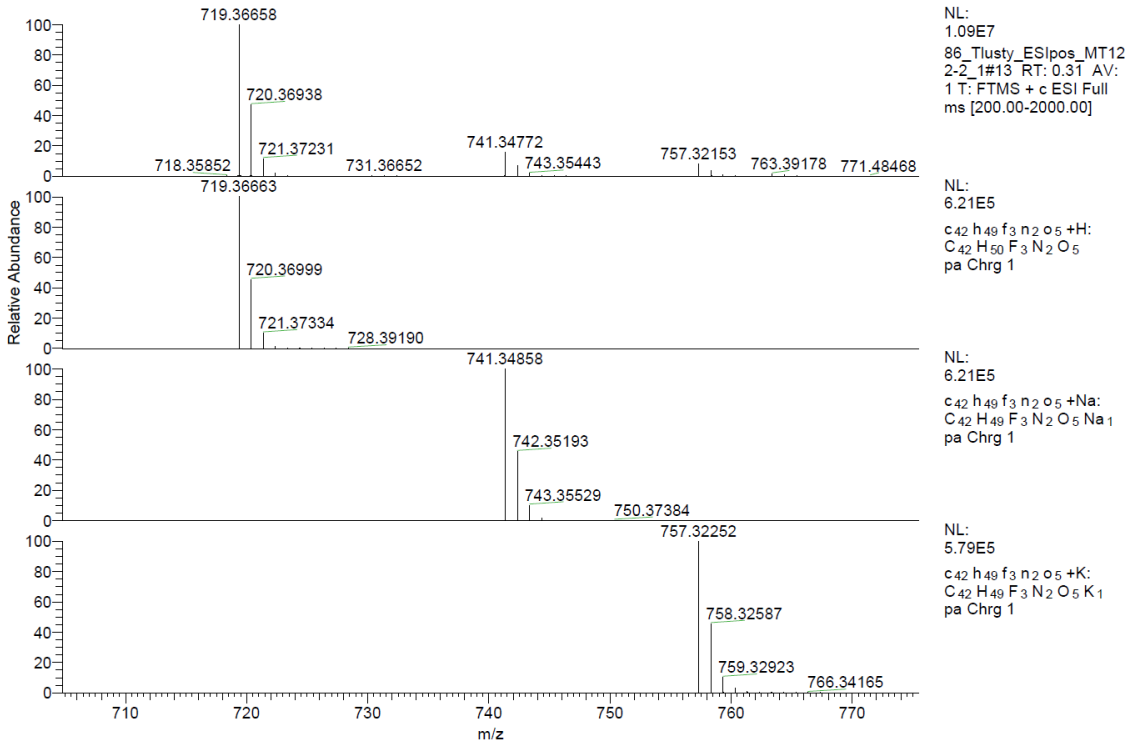


Figure 49: HRMS of compound **12a** (ESI+).

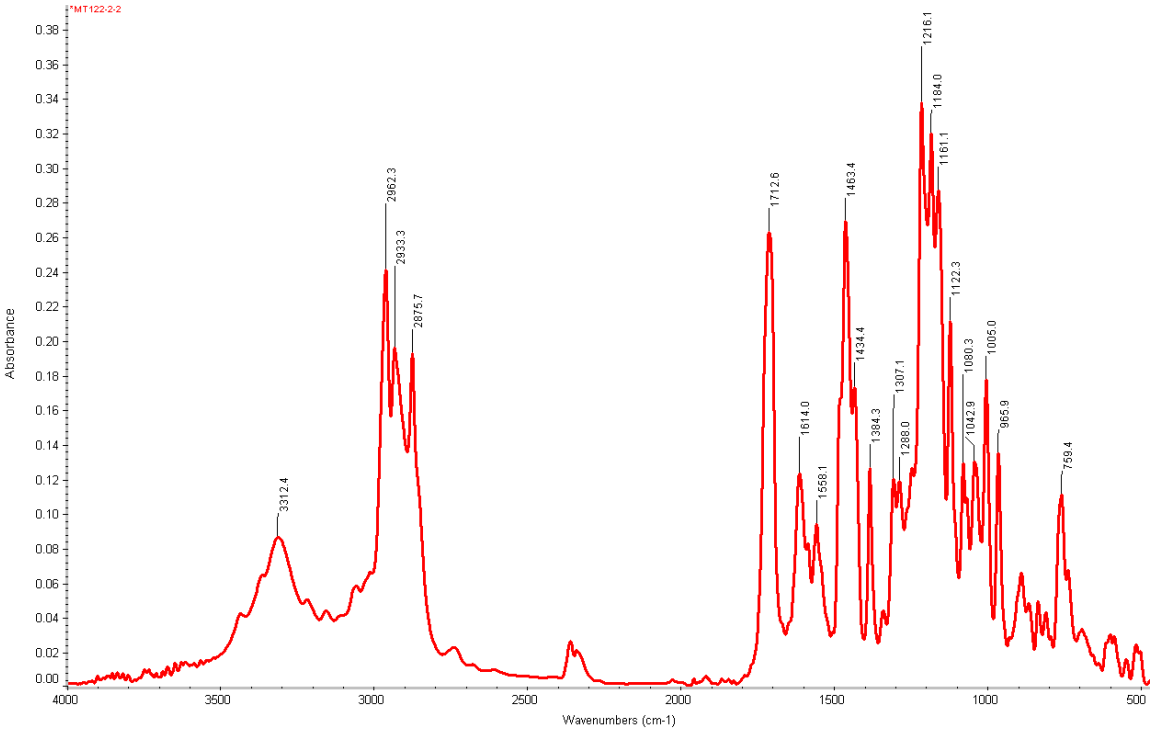


Figure 50: IR of compound **12a** (KBr).

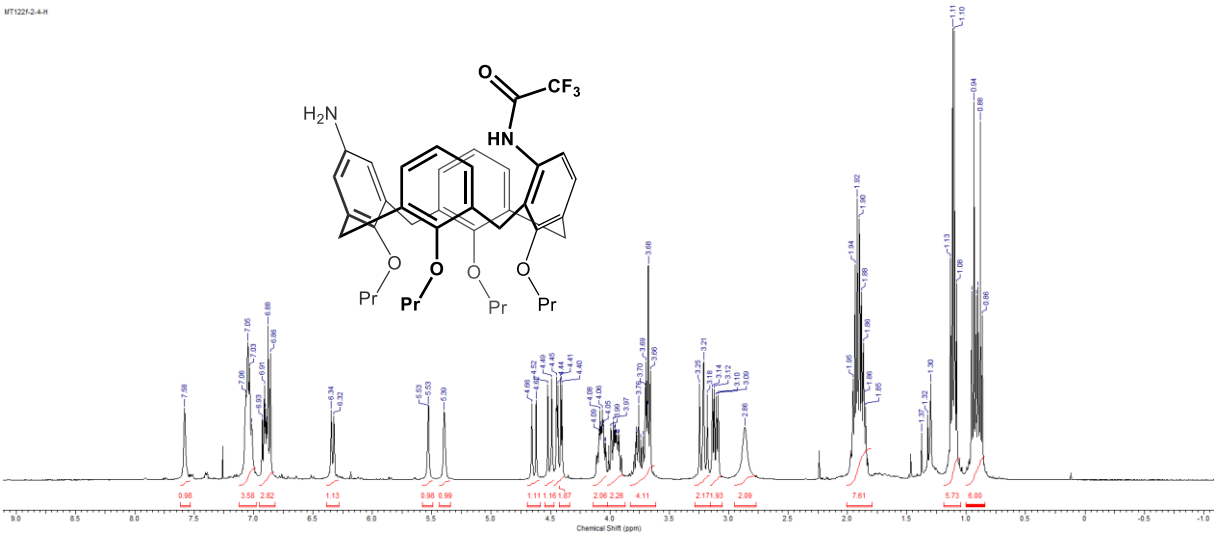


Figure 51: ^{1}H NMR of compound **12b** ($CDCl_3$, 400 MHz).

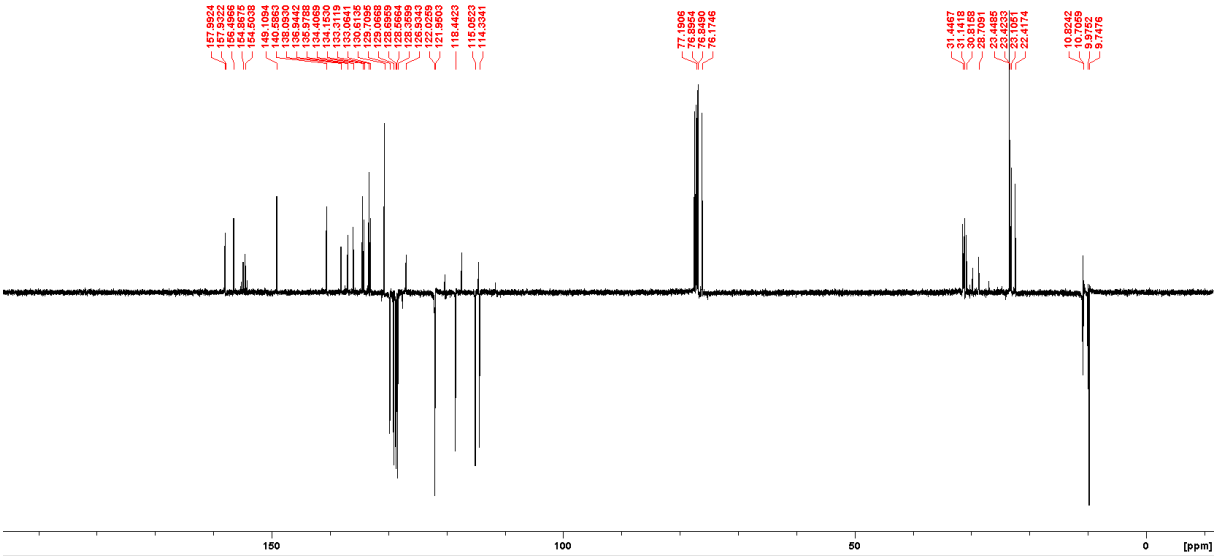


Figure 52: ^{13}C(APT) NMR of compound **12b** ($CDCl_3$, 100 MHz).

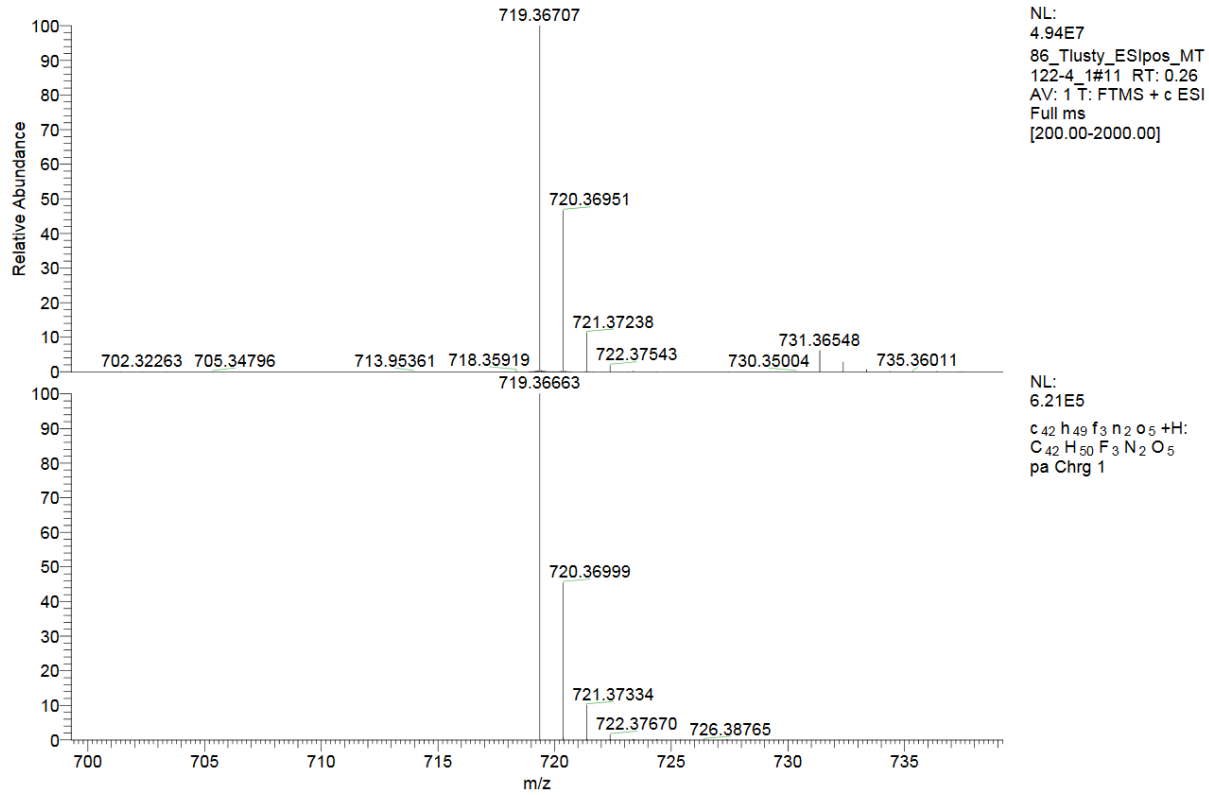


Figure 53: HRMS of compound **12b** (ESI+).

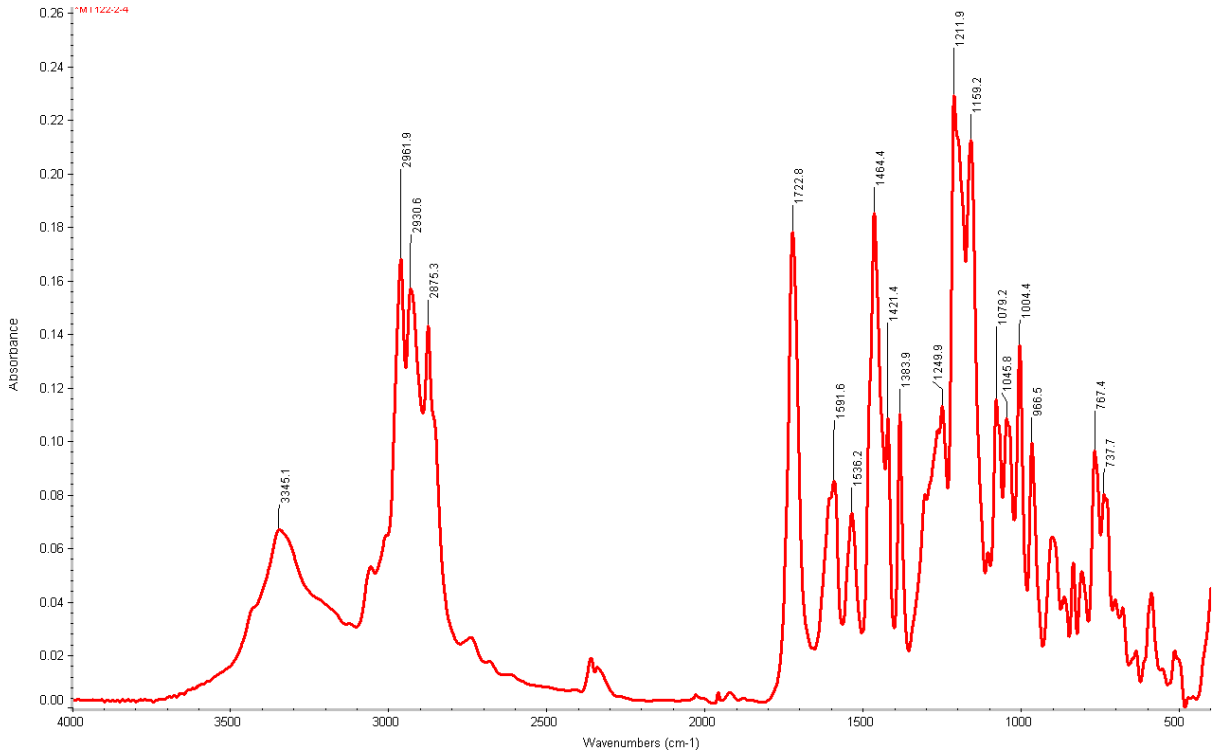


Figure 54: IR of compound **12b** (KBr).

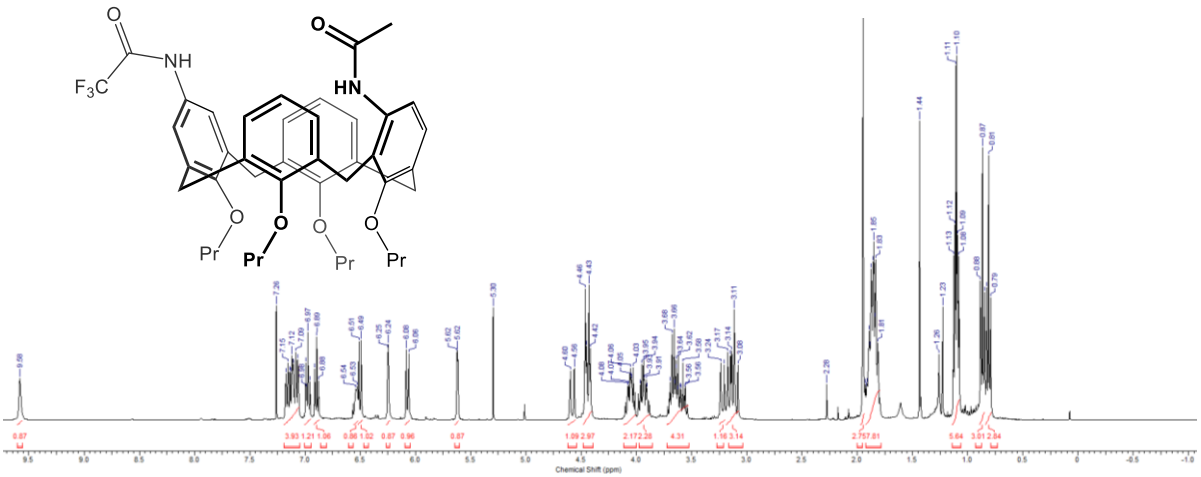


Figure 55: ^{1}H NMR of compound **13a** ($CDCl_3$, 400 MHz).

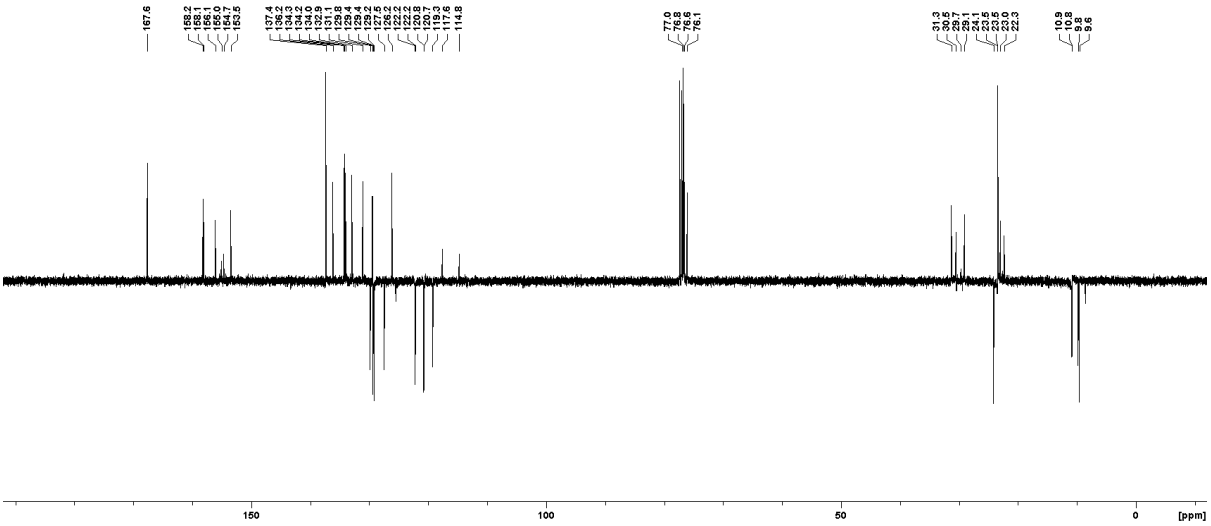


Figure 56: ^{13}C(APT) NMR of compound **13a** ($CDCl_3$, 100 MHz).

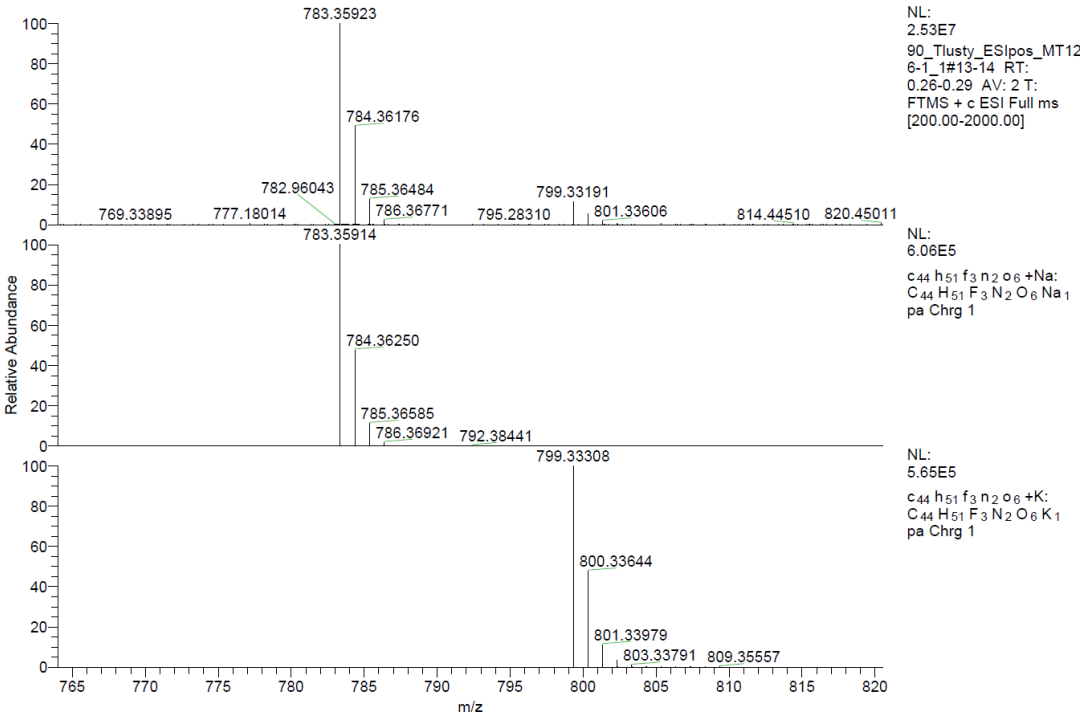


Figure 57: HRMS of compound **13a** (ESI+).

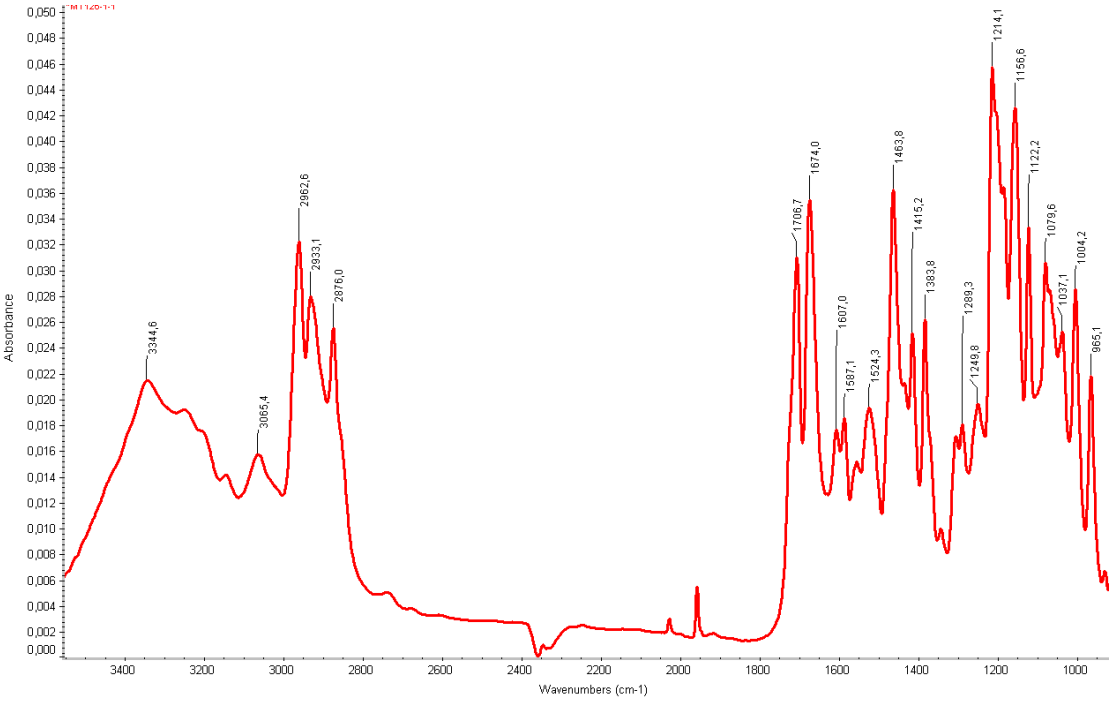


Figure 58: IR of compound **13a** (KBr).

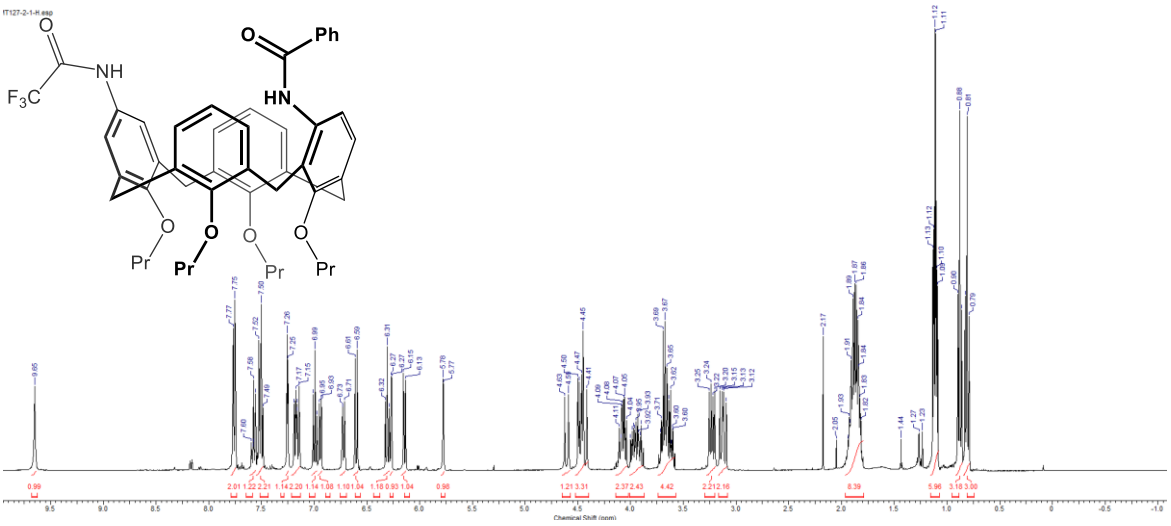


Figure 59: ^{1}H NMR of compound **13b** ($CDCl_3$, 400 MHz).

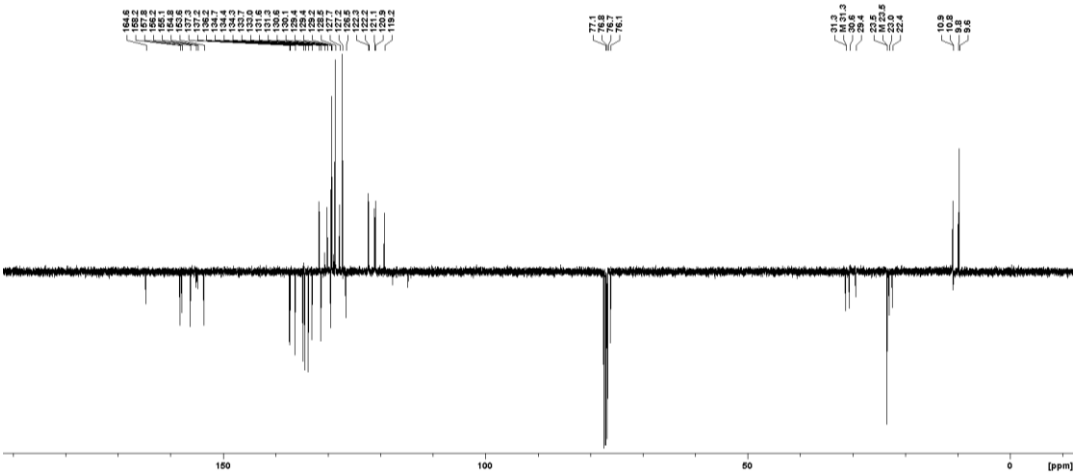


Figure 60: ^{13}C(APT) NMR of compound **13b** ($CDCl_3$, 100 MHz).

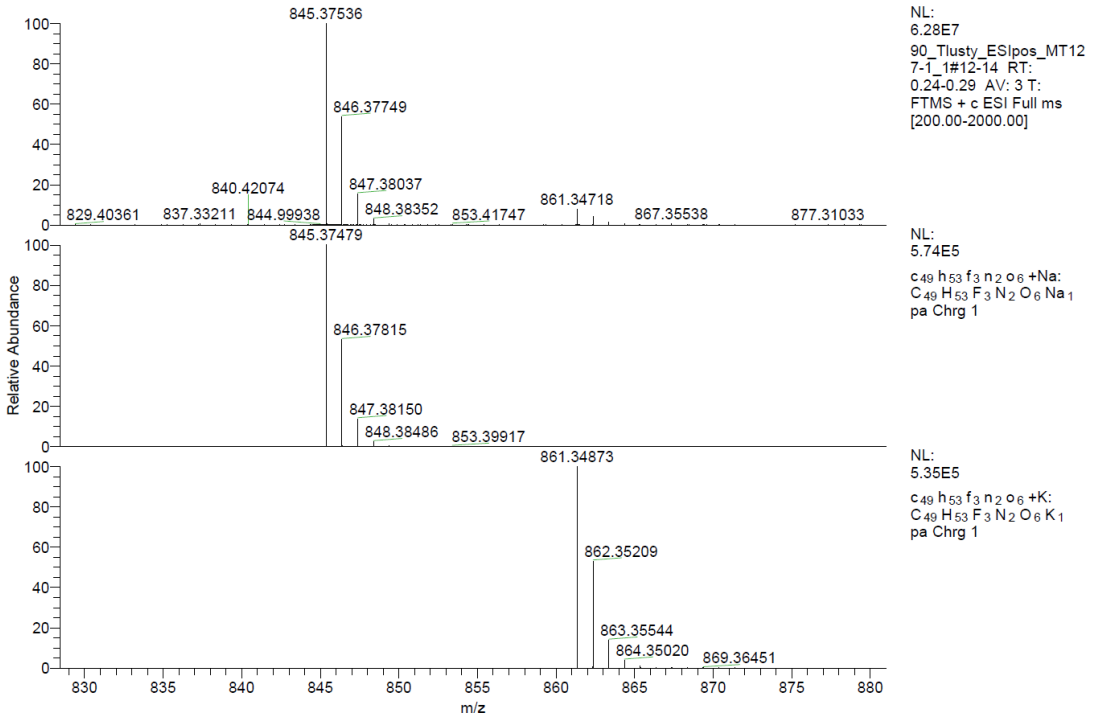


Figure 61: HRMS of compound **13b** (ESI+).

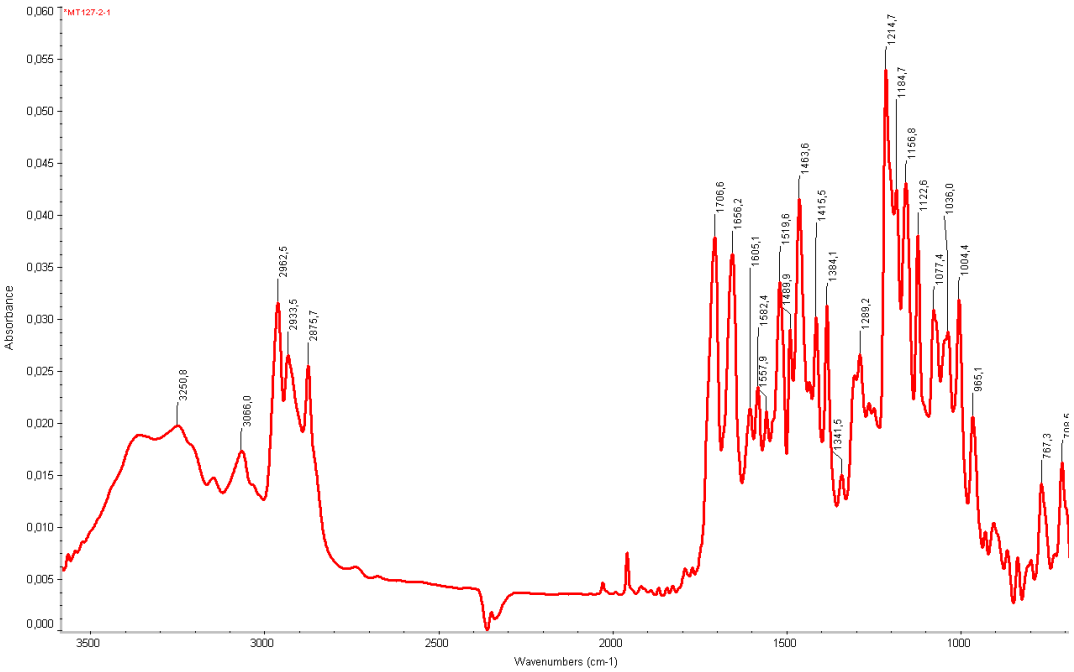


Figure 62: IR of compound **13b** (KBr).

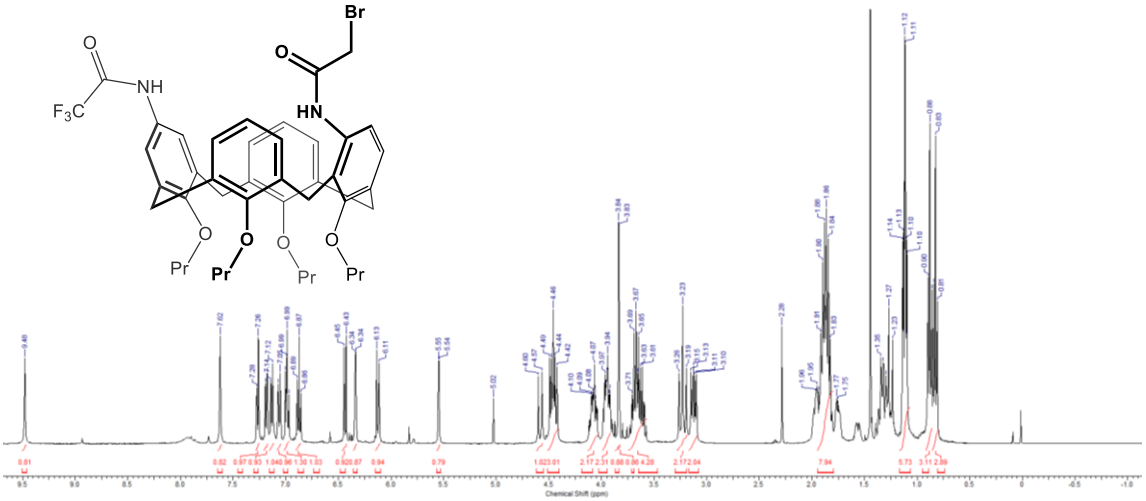


Figure 63: ^{1}H NMR of compound **13c** ($CDCl_3$, 400 MHz).

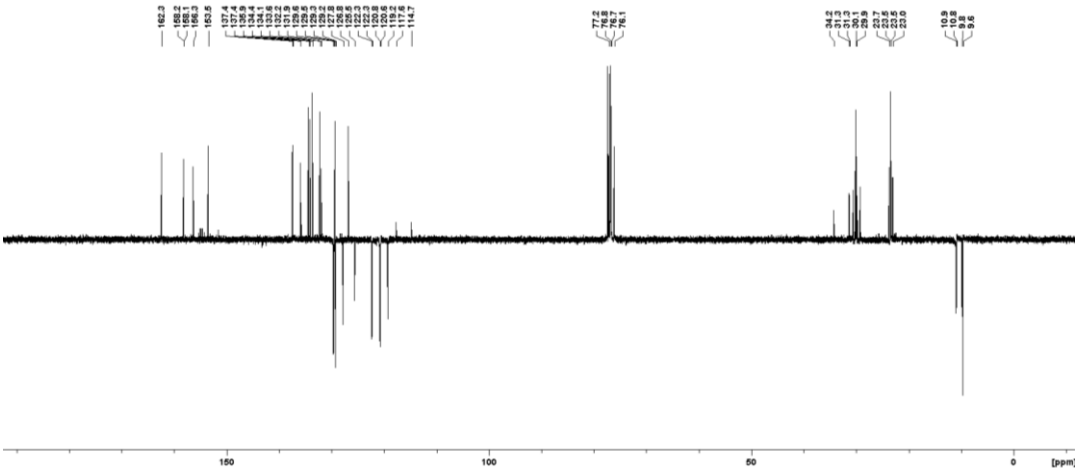


Figure 64: ^{13}C(APT) NMR of compound **13c** ($CDCl_3$, 100 MHz).

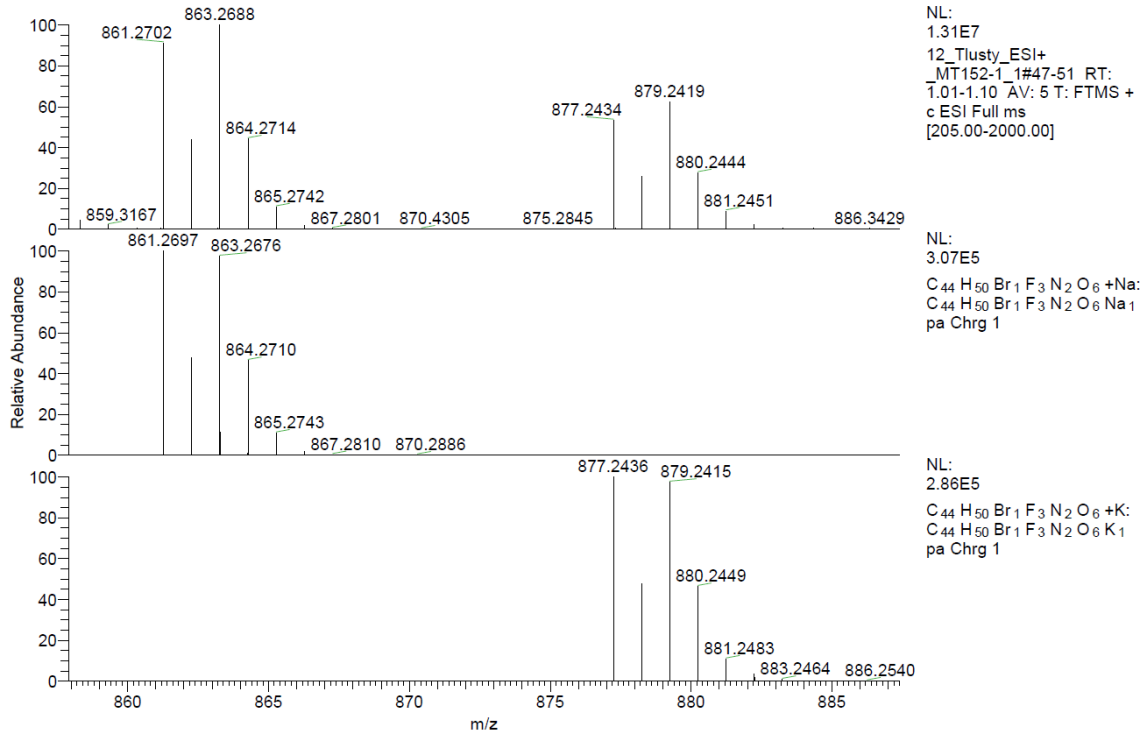


Figure 65: HRMS of compound **13c** (ESI+).

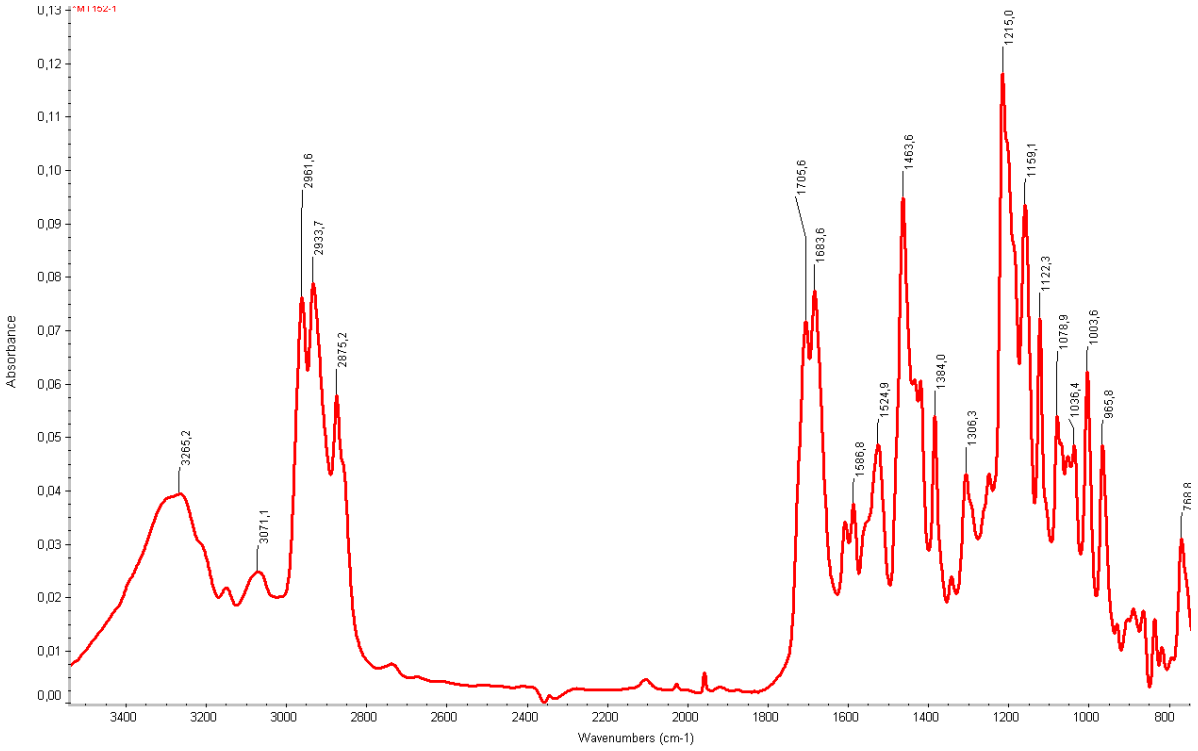


Figure 66: IR of compound **13c** (KBr).

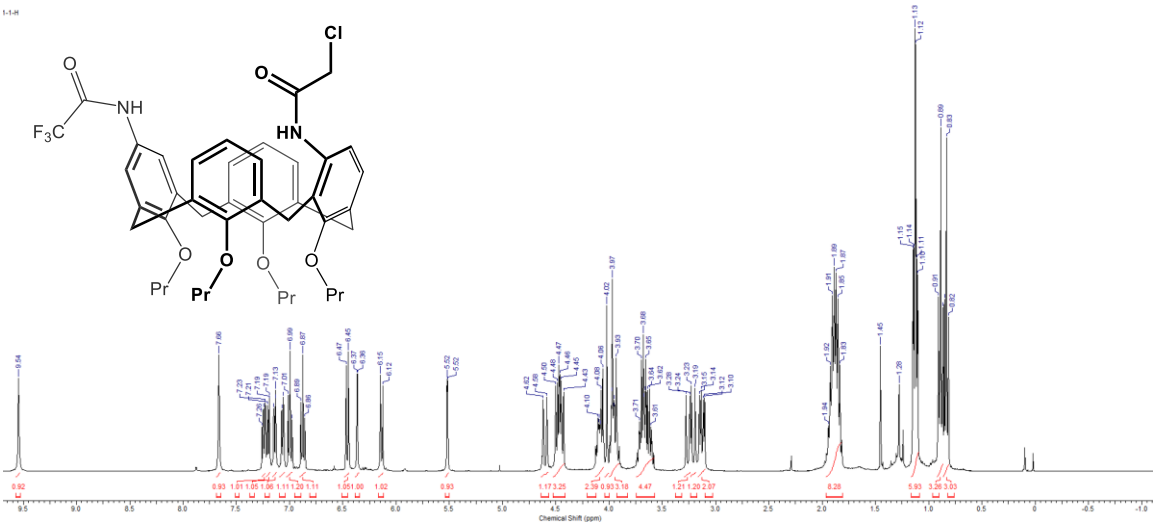


Figure 67: ^{1}H NMR of compound **13d** ($CDCl_3$, 400 MHz).

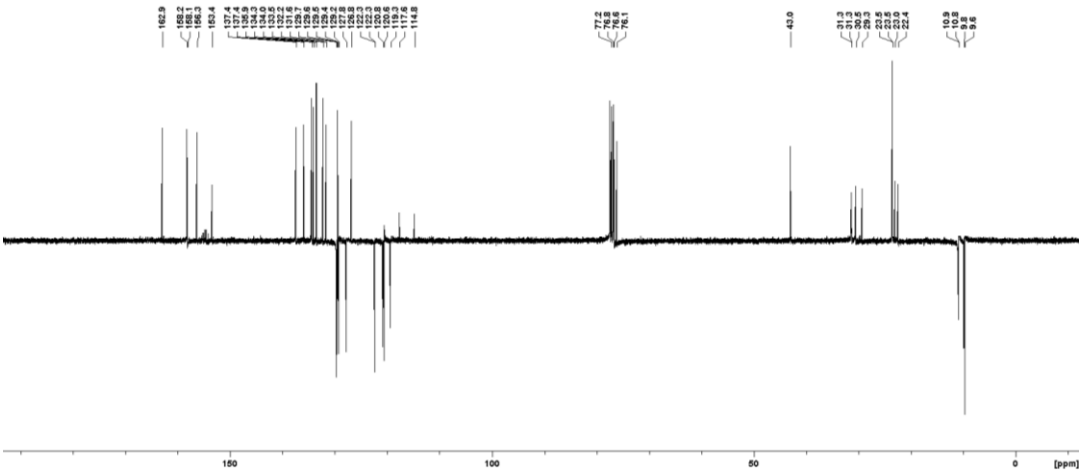


Figure 68: ^{13}C(APT) NMR of compound **13d** ($CDCl_3$, 100 MHz).

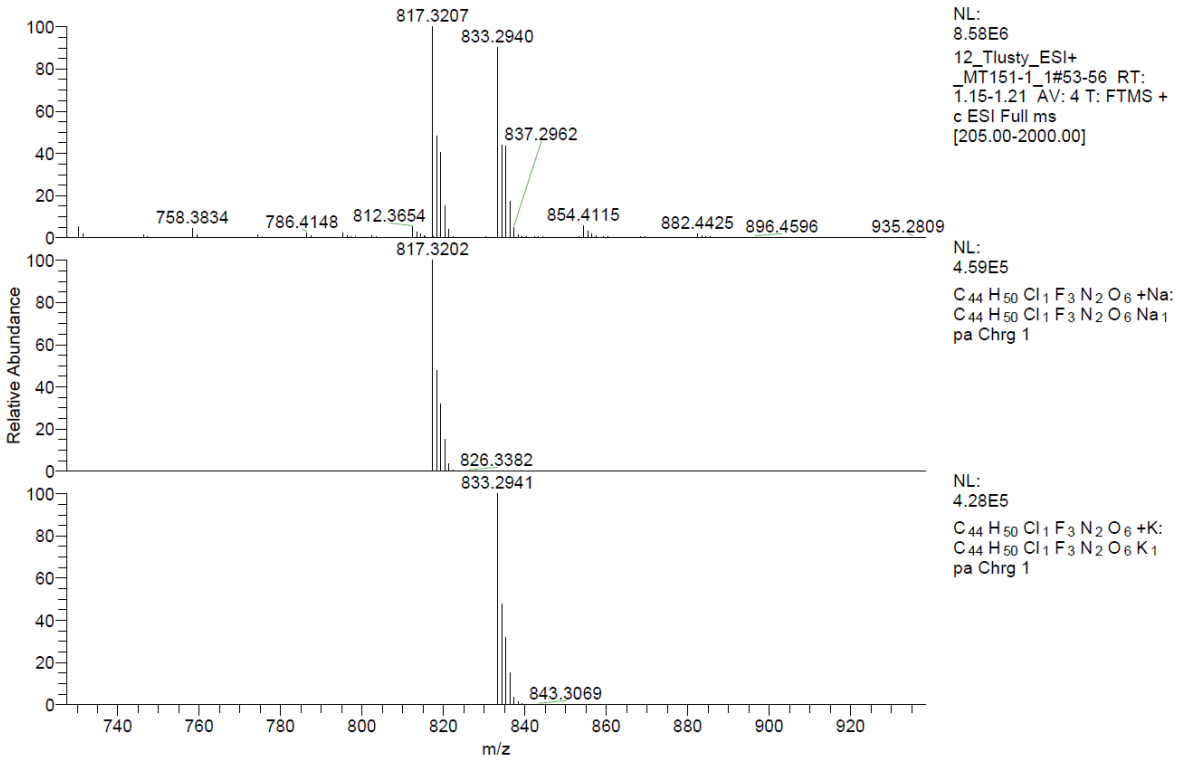


Figure 69: HRMS of compound **13d** (ESI+).

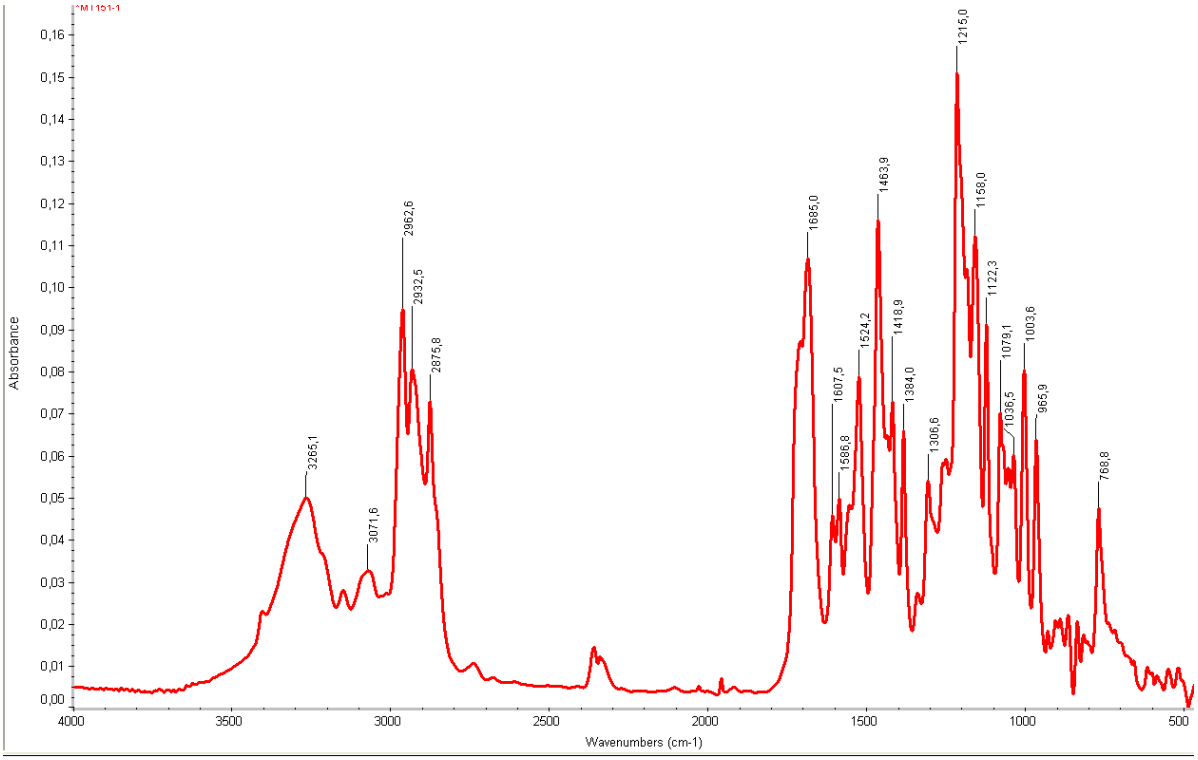


Figure 70: IR of compound **13d** (KBr).

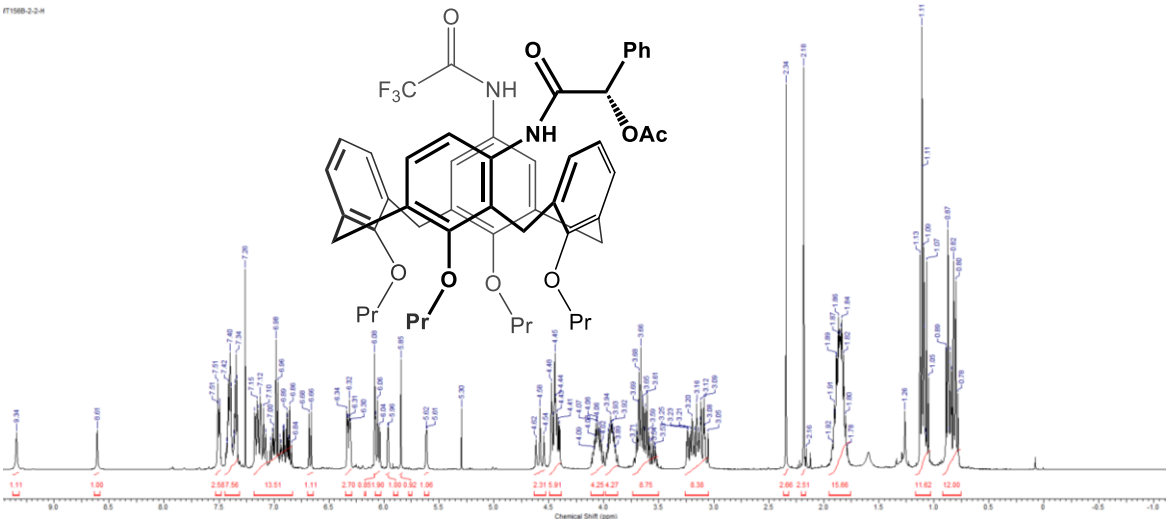


Figure 71: ^{1}H NMR of compound **13e** ($CDCl_3$, 400 MHz).

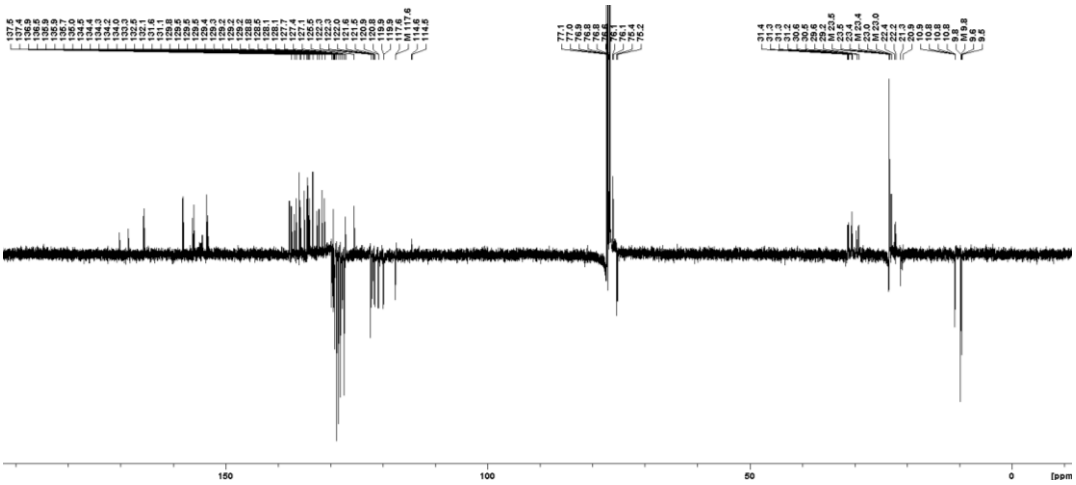


Figure 72: ^{13}C(APT) NMR of compound **13e** ($CDCl_3$, 100 MHz).

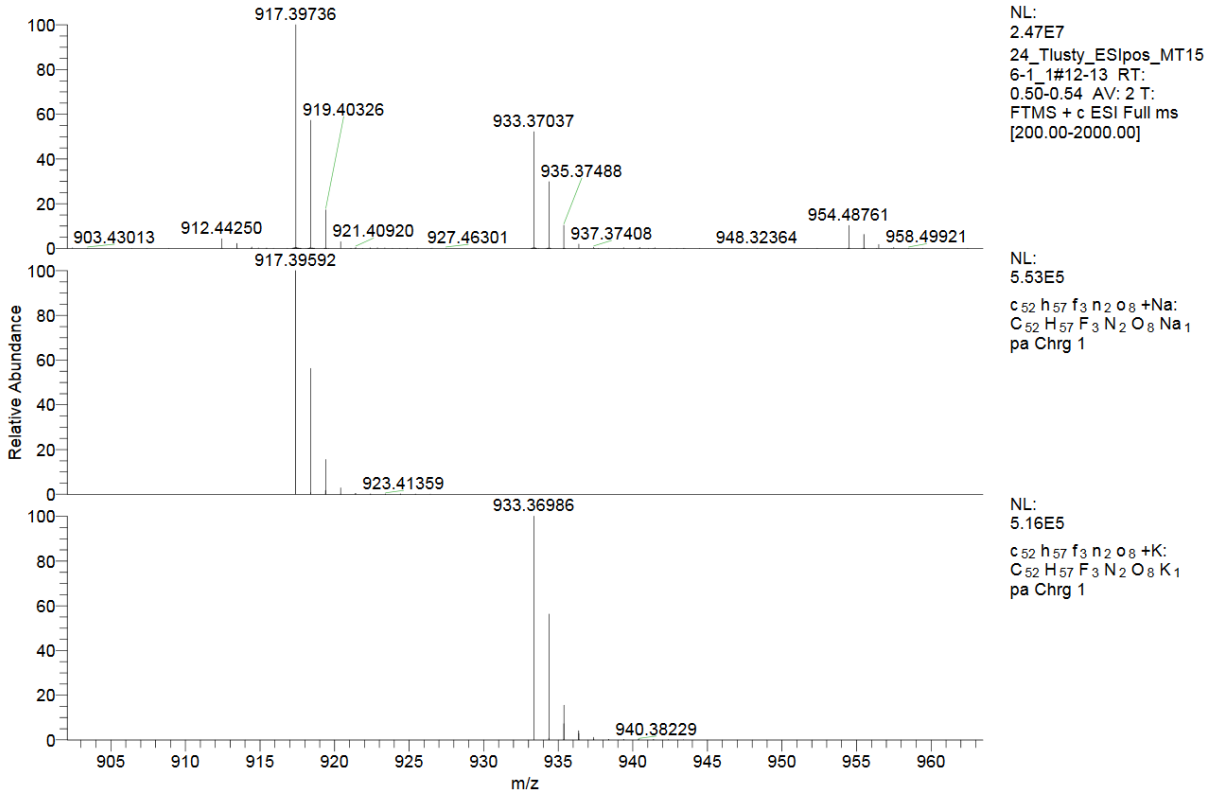


Figure 73: HRMS of compound **13e** (ESI+).

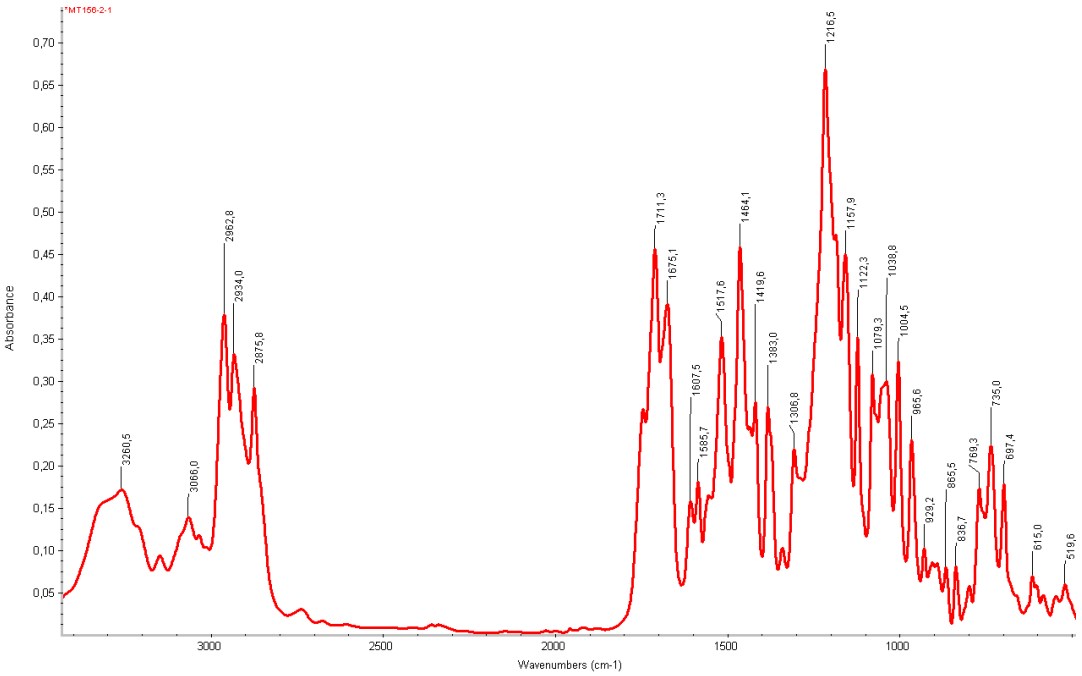


Figure 74: IR of compound **13e** (KBr).

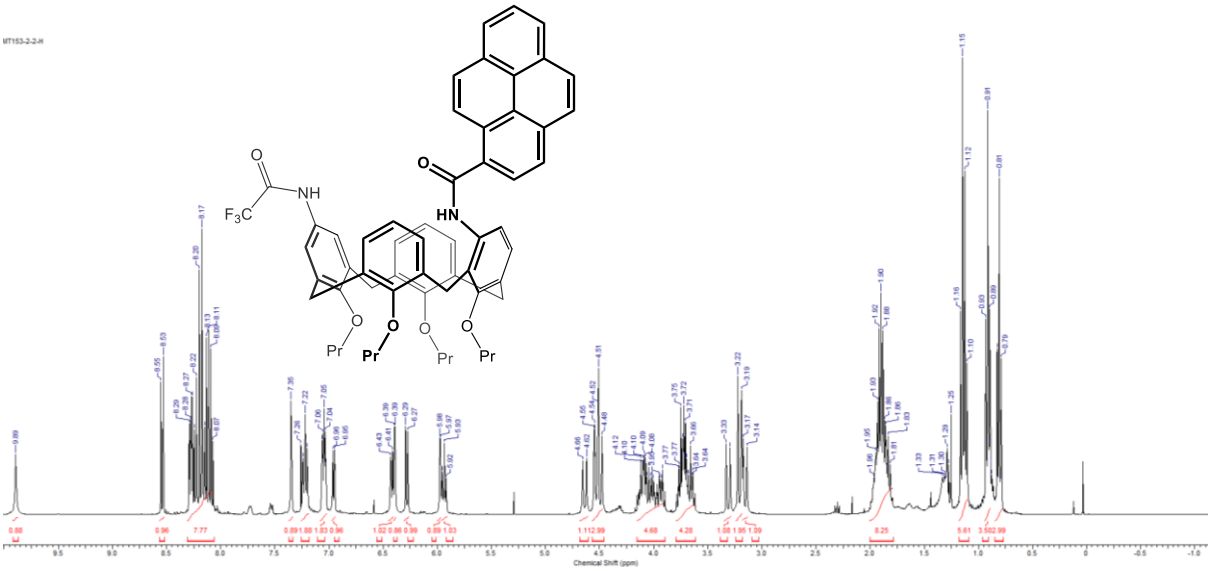


Figure 75: ^{1}H NMR of compound **13f** ($CDCl_3$, 400 MHz).

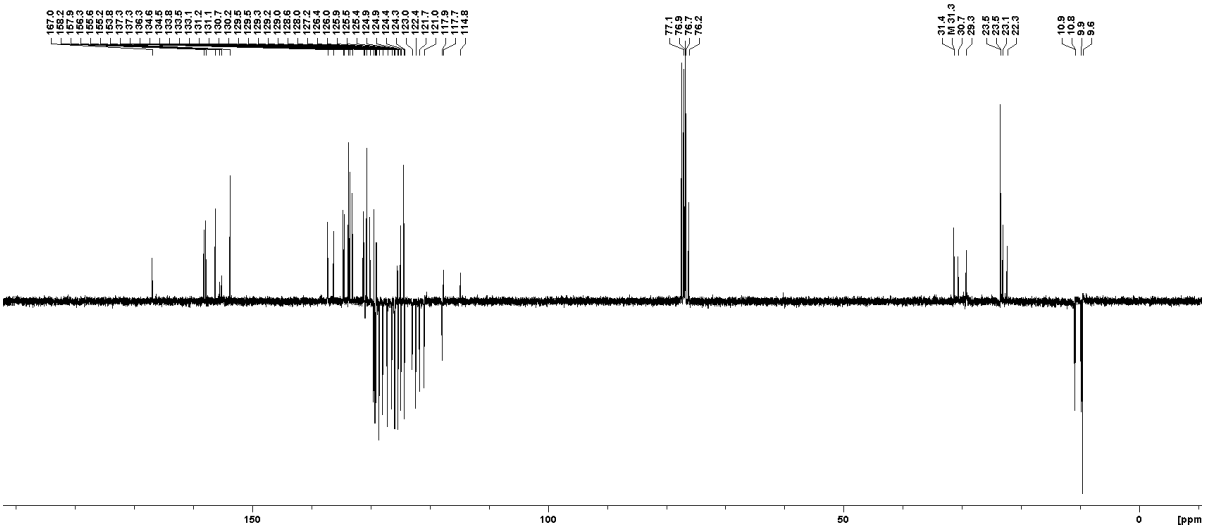


Figure 76: ^{13}C(APT) NMR of compound **13f** ($CDCl_3$, 100 MHz).

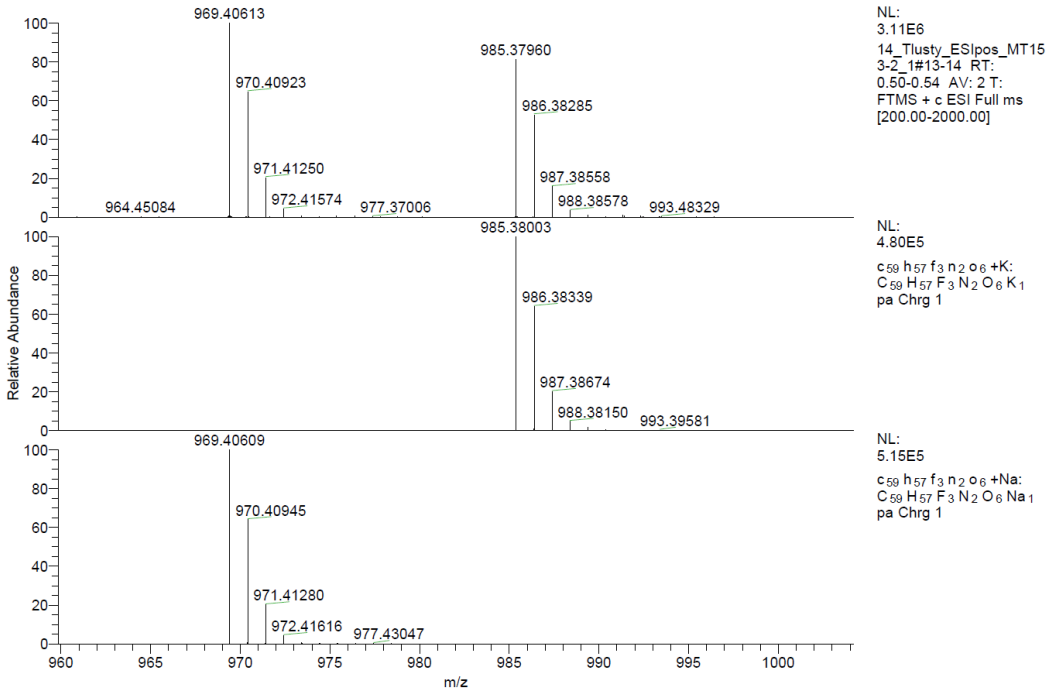


Figure 77: HRMS of compound **13f** (ESI+).

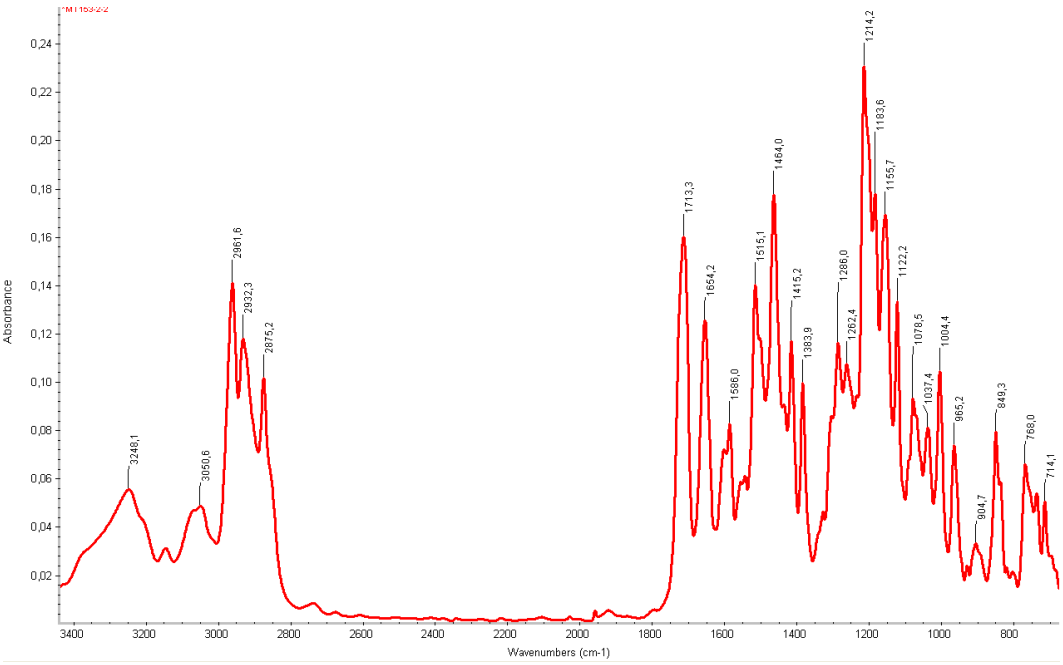


Figure 78: IR of compound **13f** (KBr).

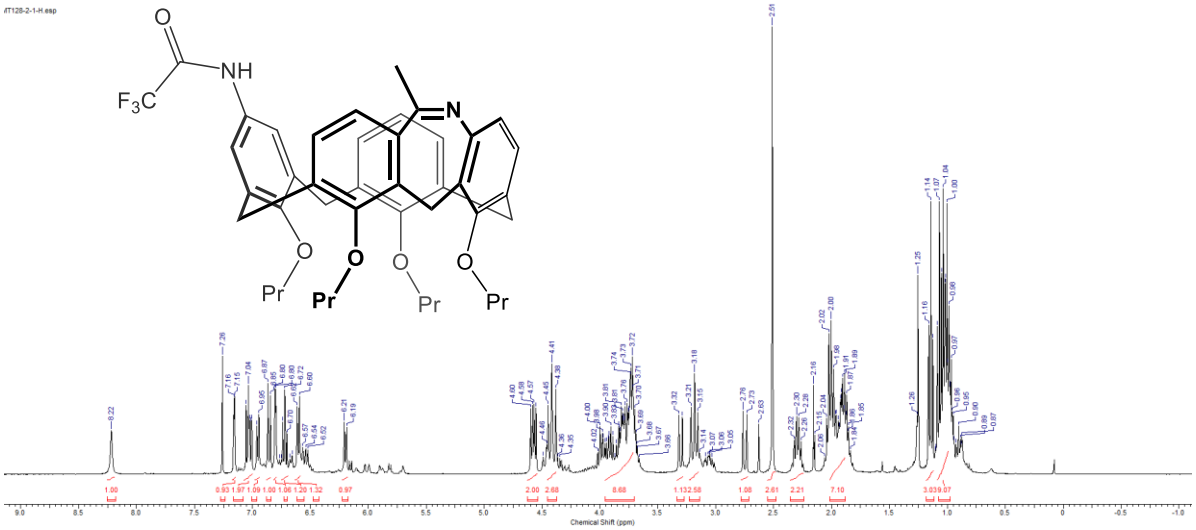


Figure 79: ^{1}H NMR of compound **14a** ($CDCl_3$, 400 MHz).

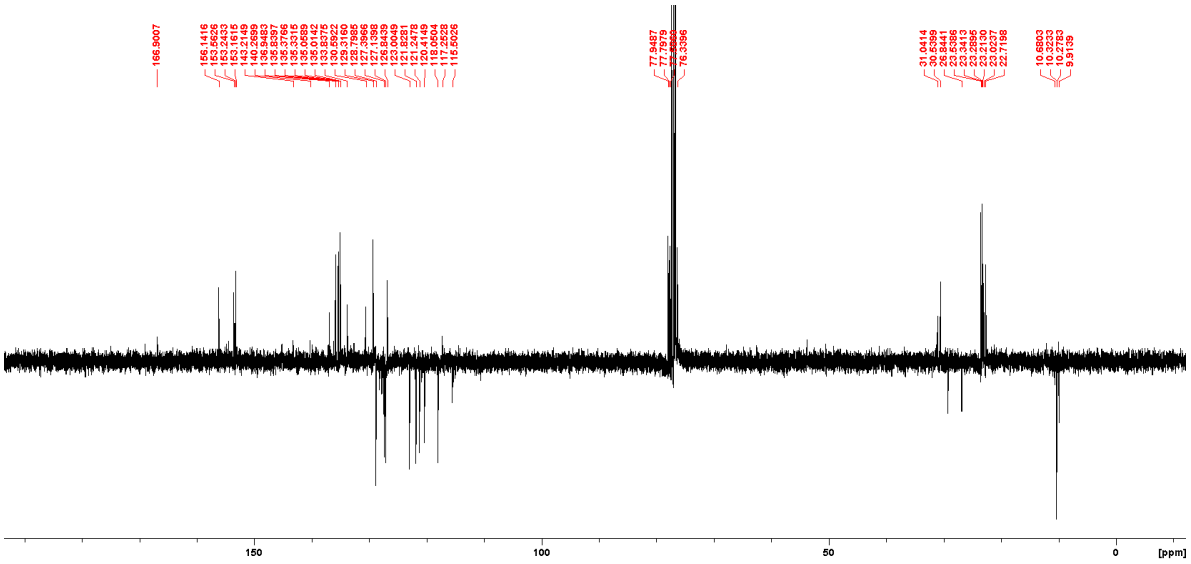


Figure 80: ^{13}C(APT) NMR of compound **14a** ($CDCl_3$, 100 MHz).

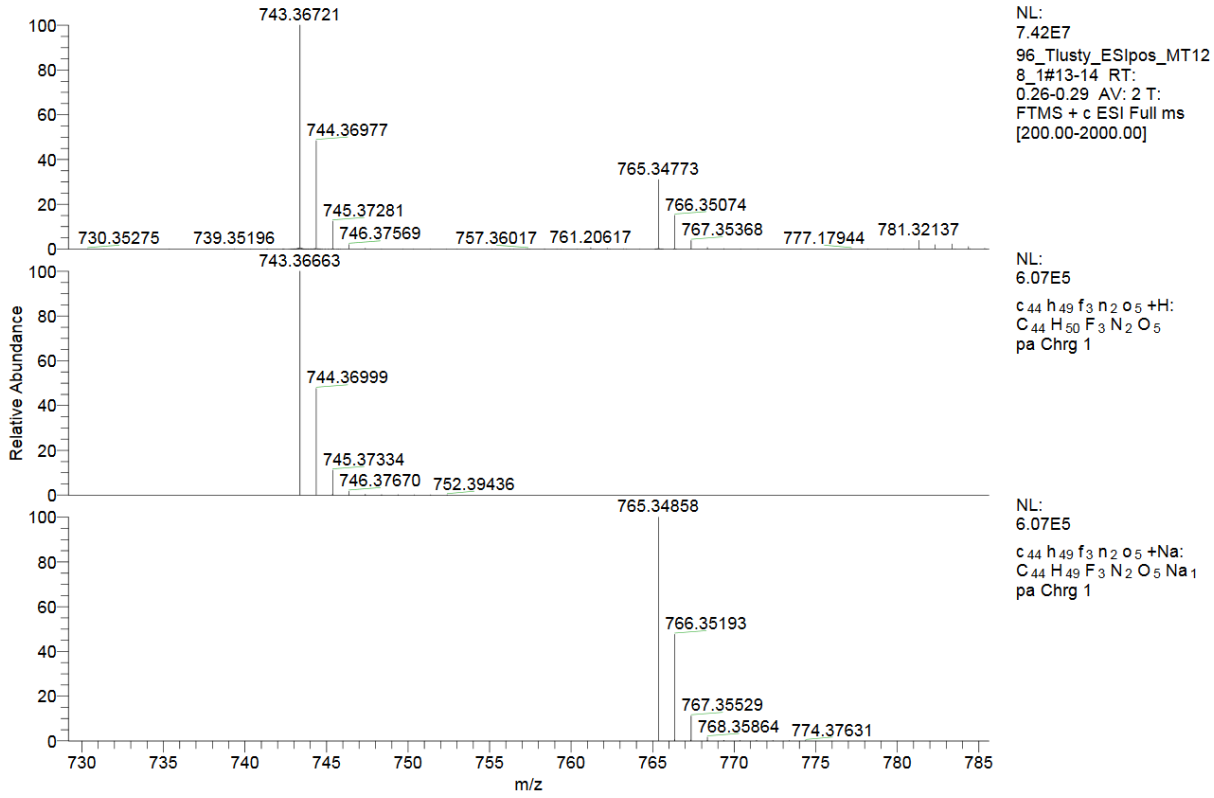


Figure 81: HRMS of compound **14a** (ESI+).

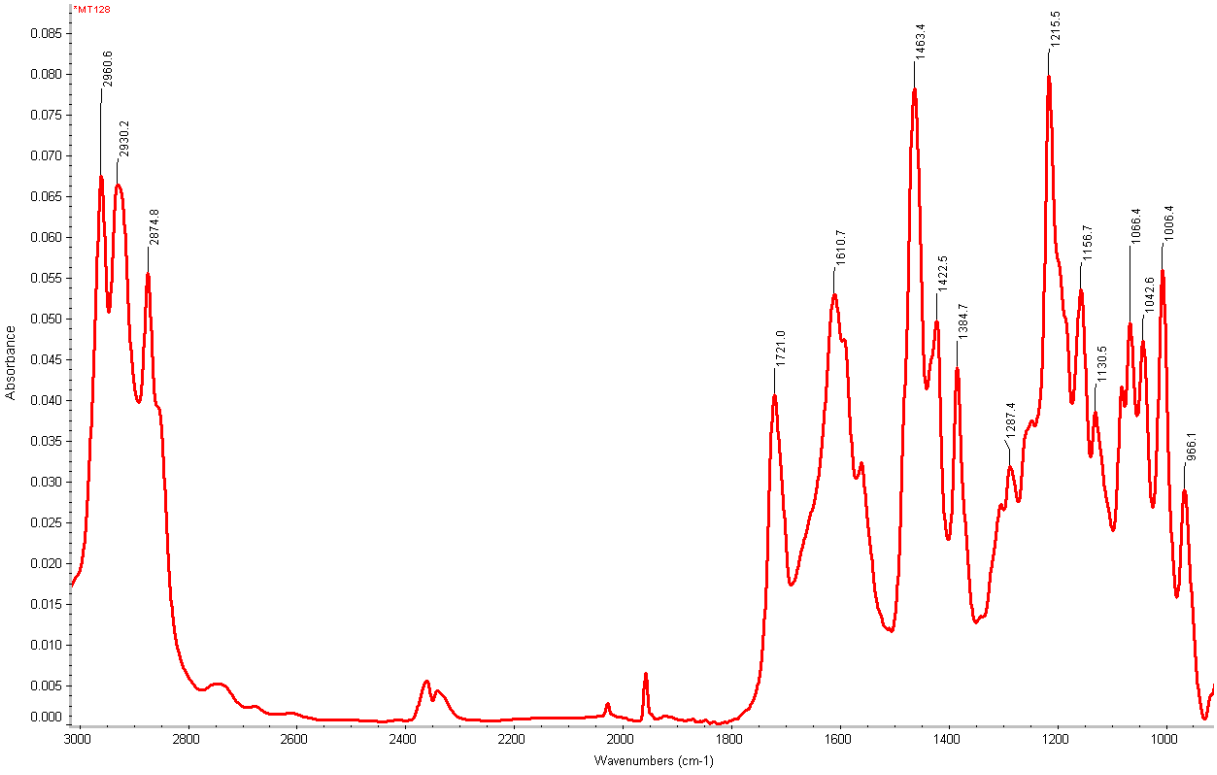


Figure 82: IR of compound **14a** (KBr).

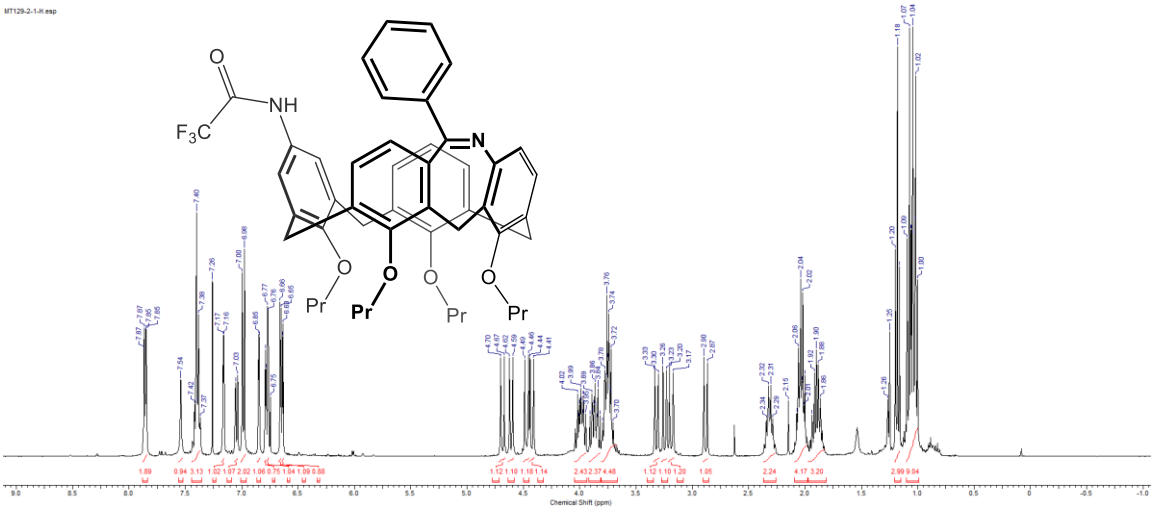


Figure 83: ^{1}H NMR of compound **14b** ($CDCl_3$, 400 MHz).

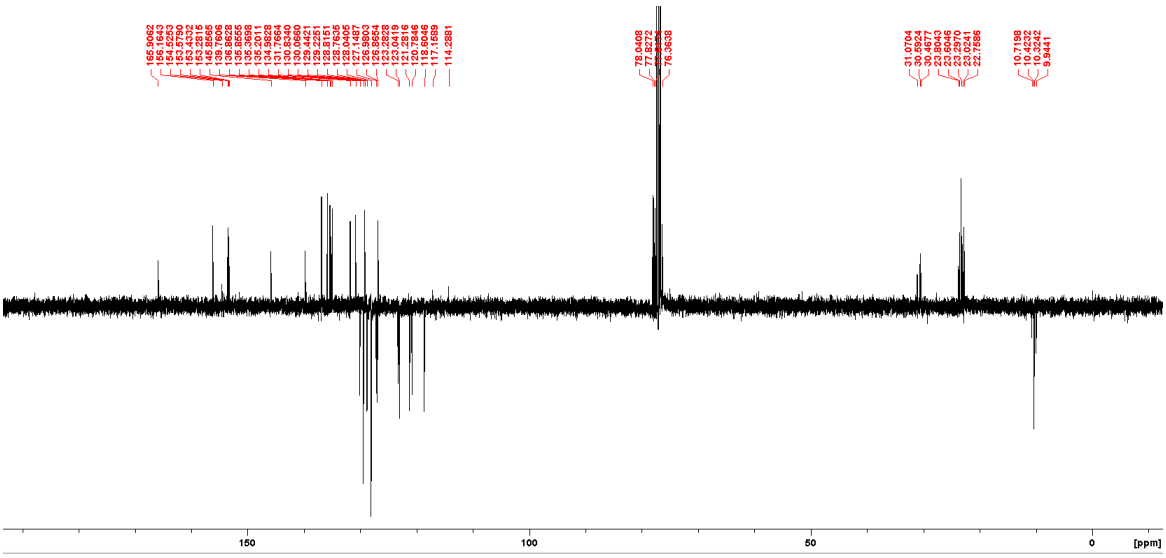


Figure 84: ^{13}C(APT) NMR of compound **14b** ($CDCl_3$, 100 MHz).

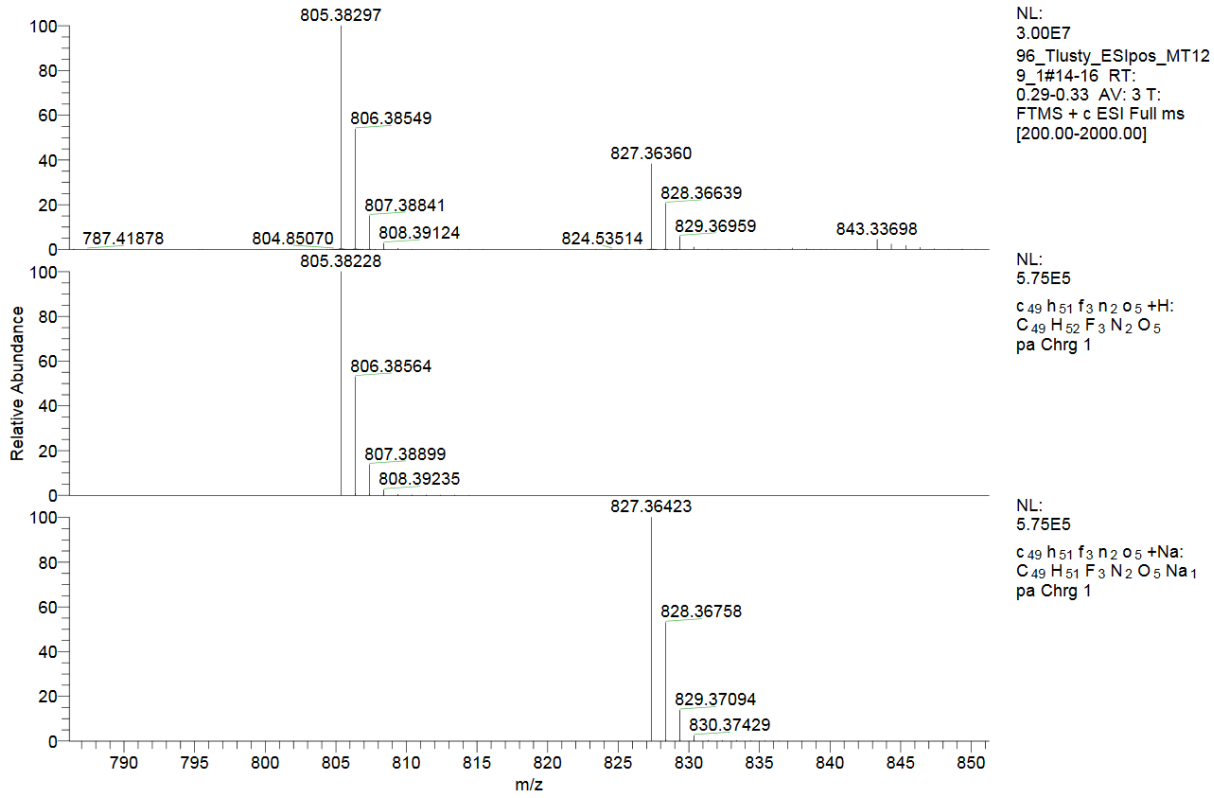


Figure 85: HRMS of compound **14b** (ESI+).

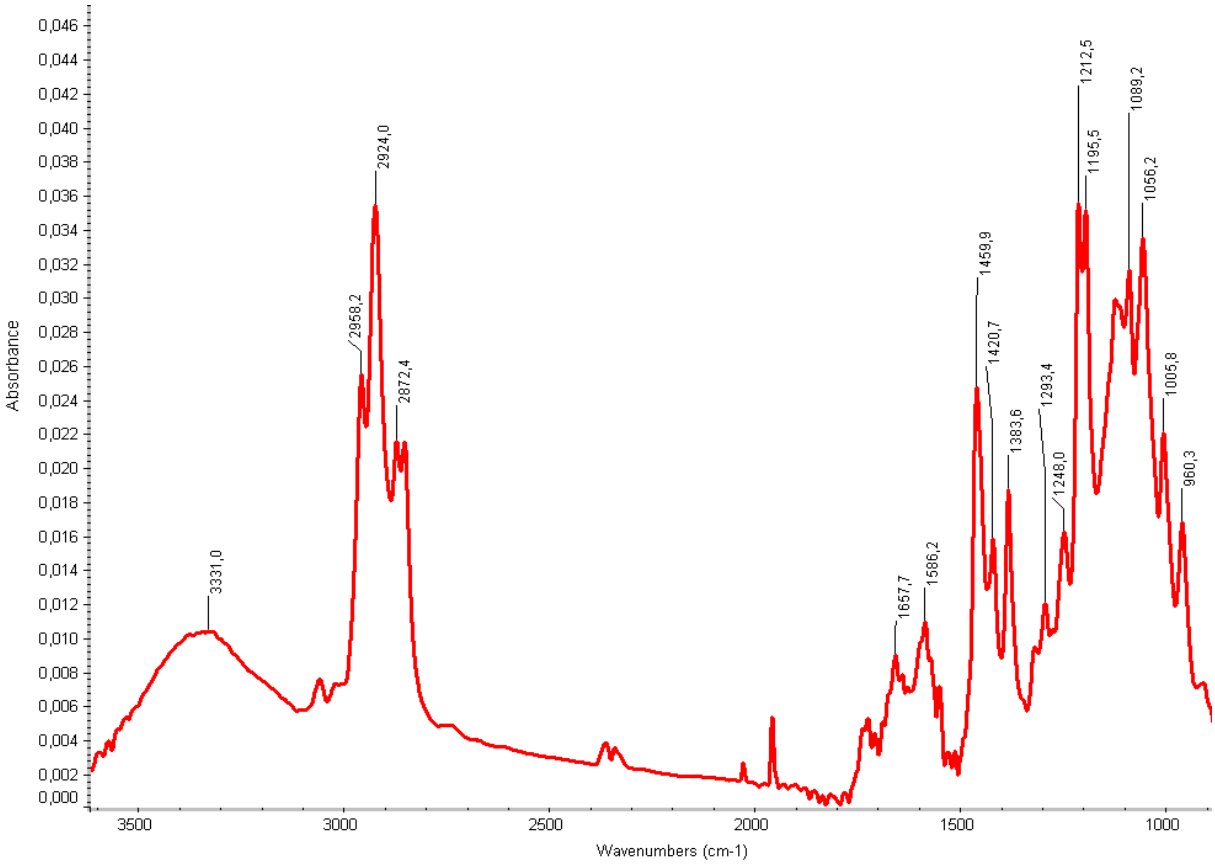


Figure 86: IR of compound **14b** (KBr).

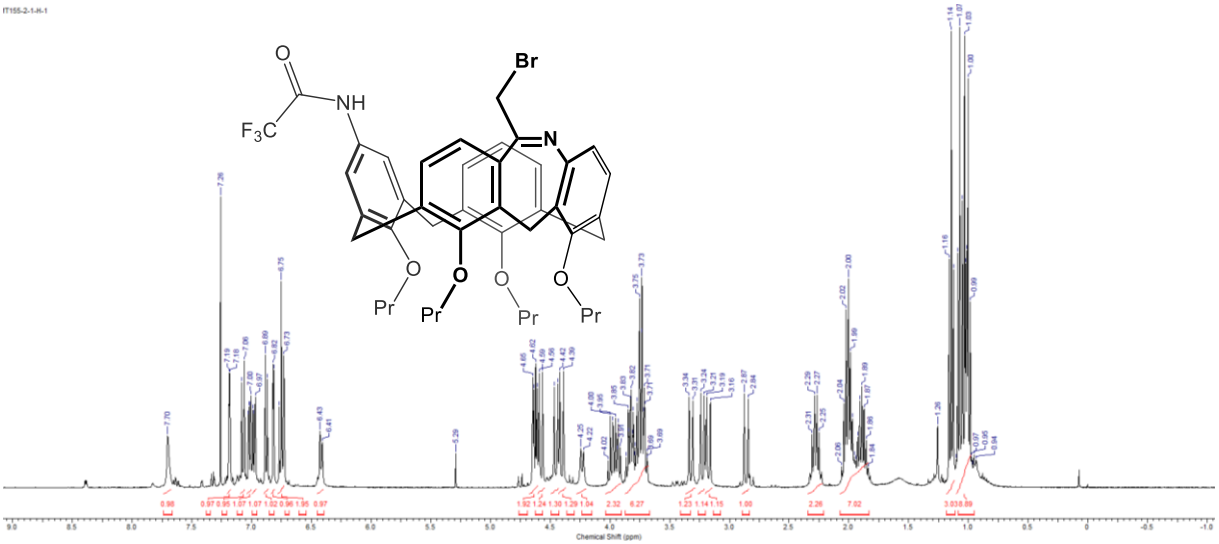


Figure 87: ^{1}H NMR of compound **14c** ($CDCl_3$, 400 MHz).

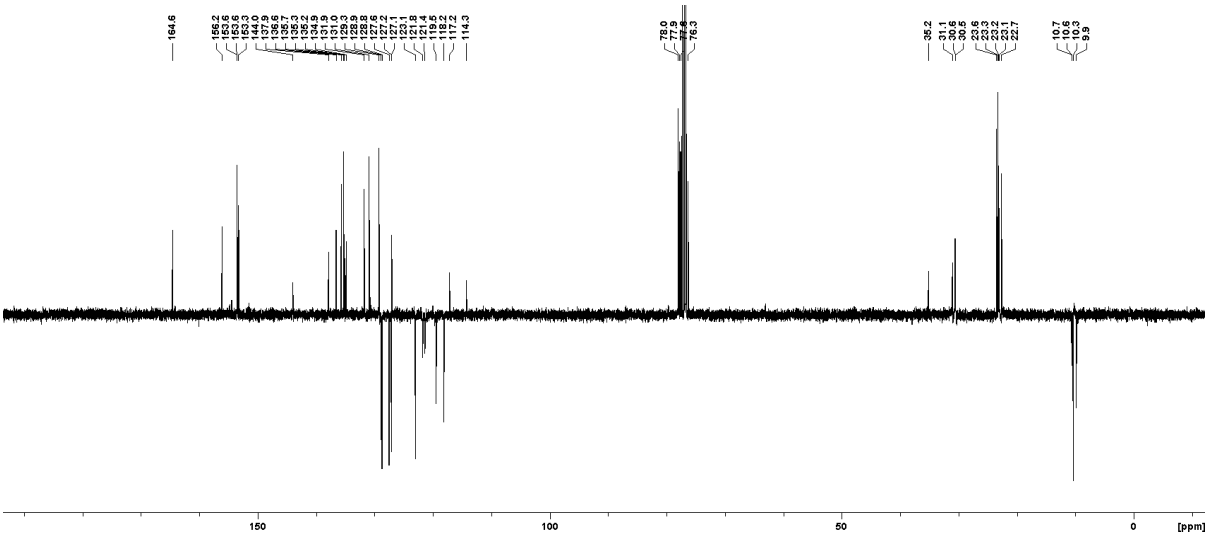


Figure 88: ^{13}C(APT) NMR of compound **14c** ($CDCl_3$, 100 MHz).

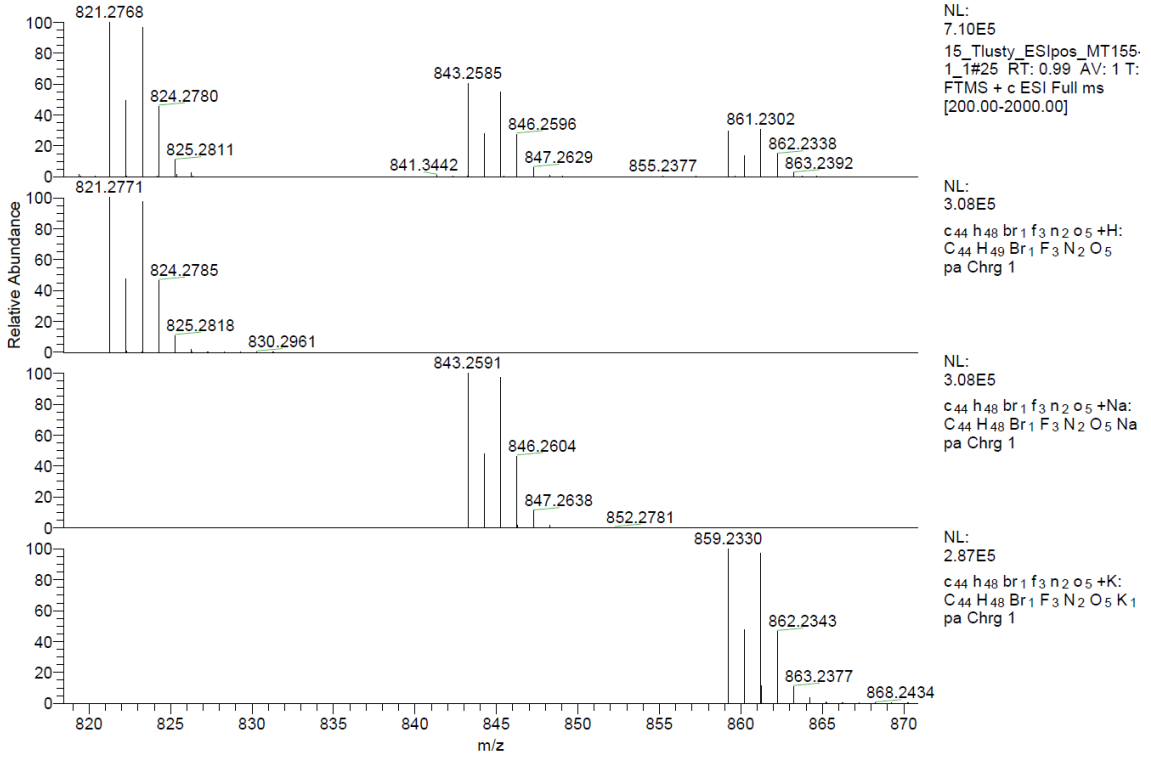


Figure 89: HRMS of compound **14c** (ESI+).

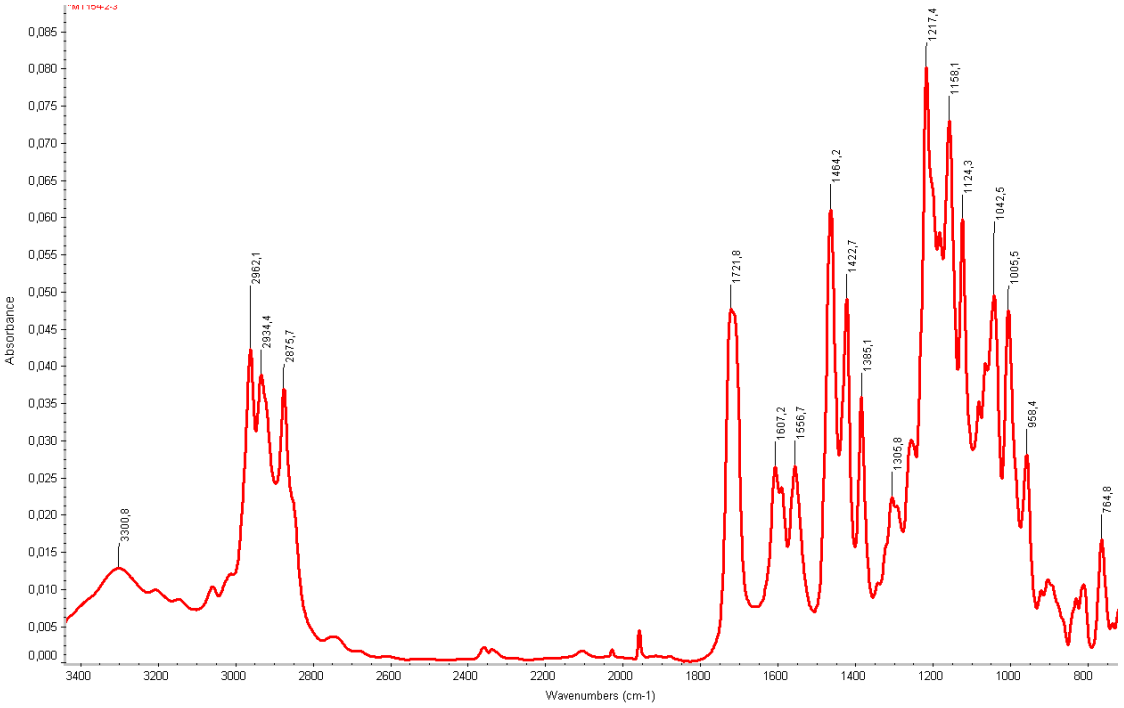


Figure 90: IR of compound **14c** (KBr).

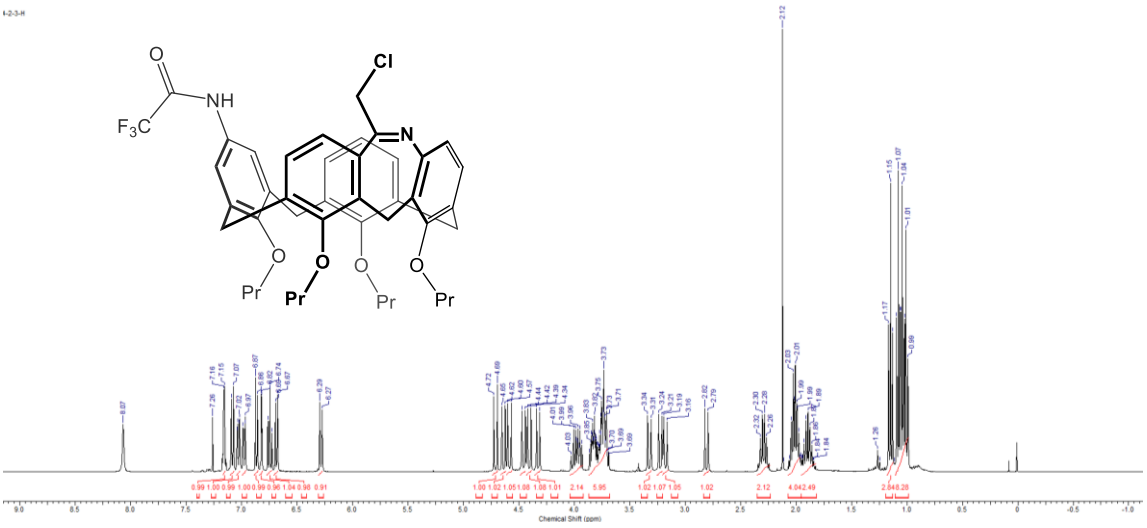


Figure 91: ^{1}H NMR of compound **14d** ($CDCl_3$, 400 MHz).

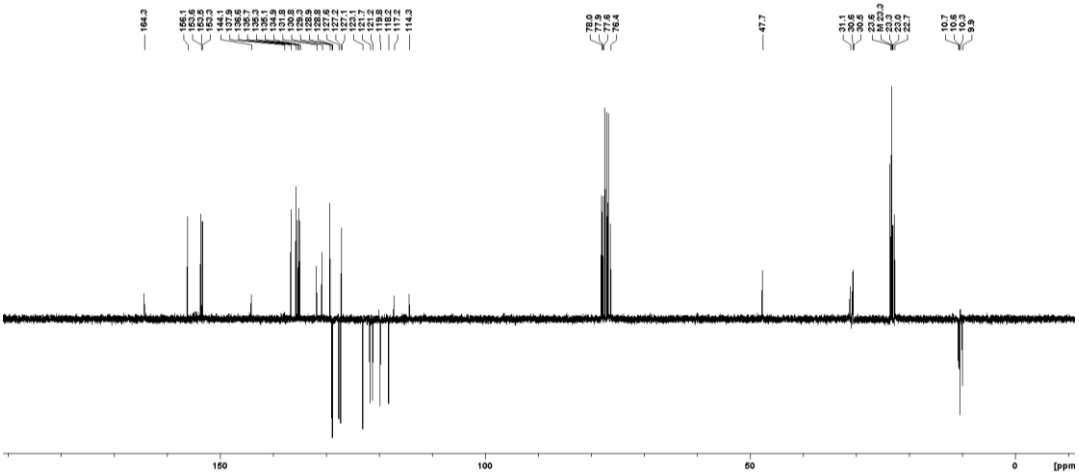


Figure 92: ^{13}C(APT) NMR of compound **14d** ($CDCl_3$, 100 MHz).

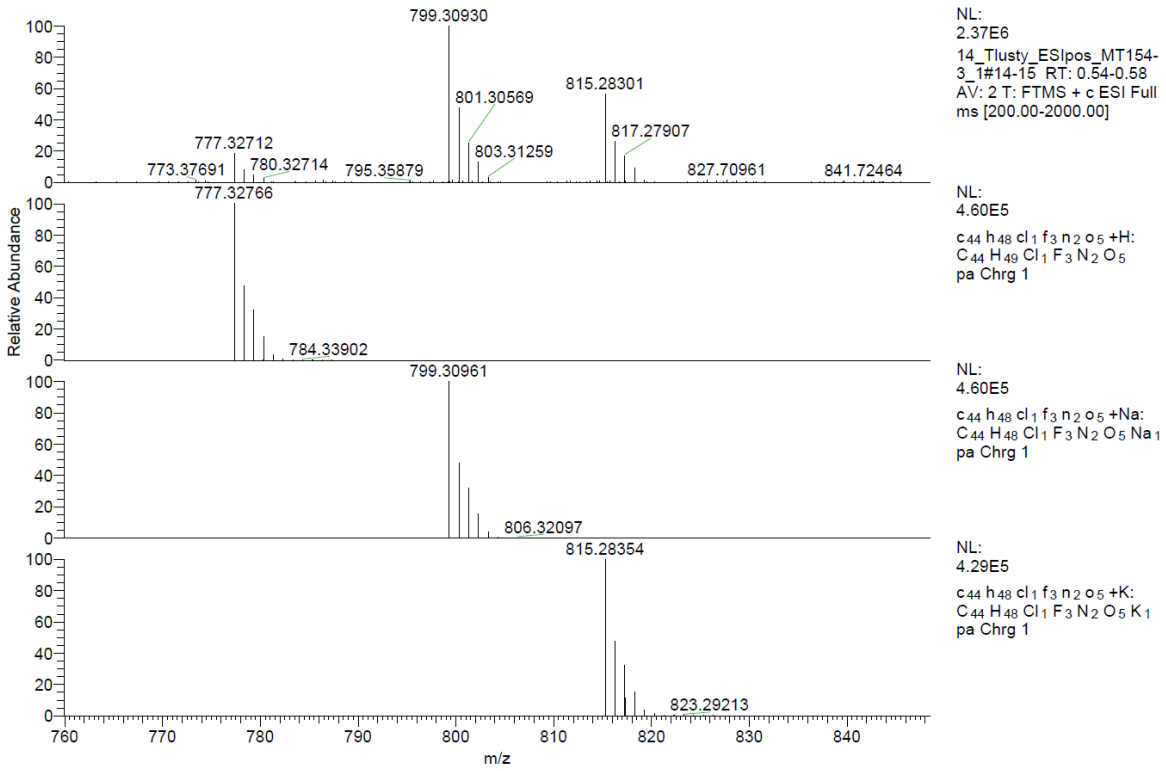


Figure 93: HRMS of compound **14d** (ESI+).

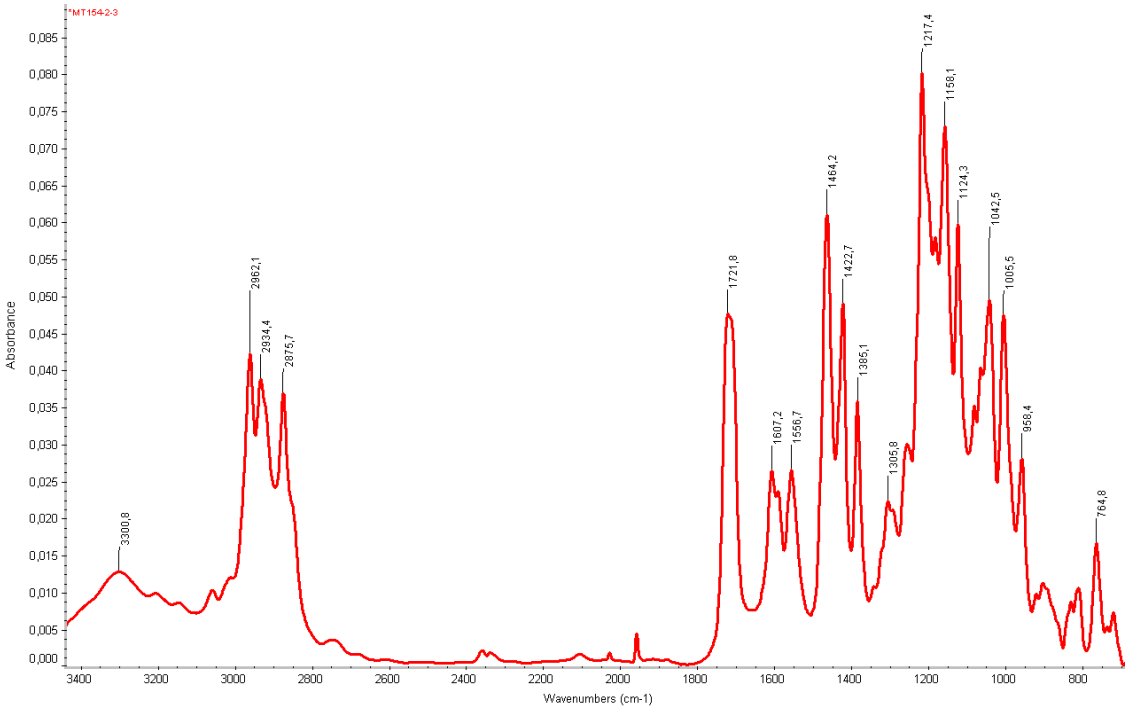


Figure 94: IR of compound **14d** (KBr).

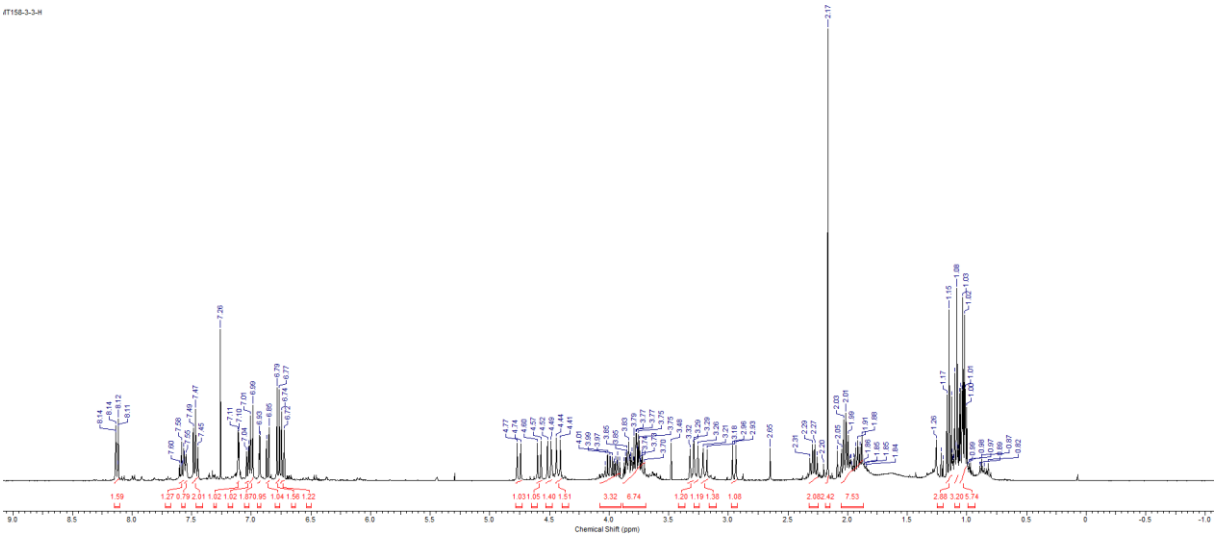


Figure 95: ^{1}H NMR of compound **14e** ($CDCl_3$, 400 MHz).

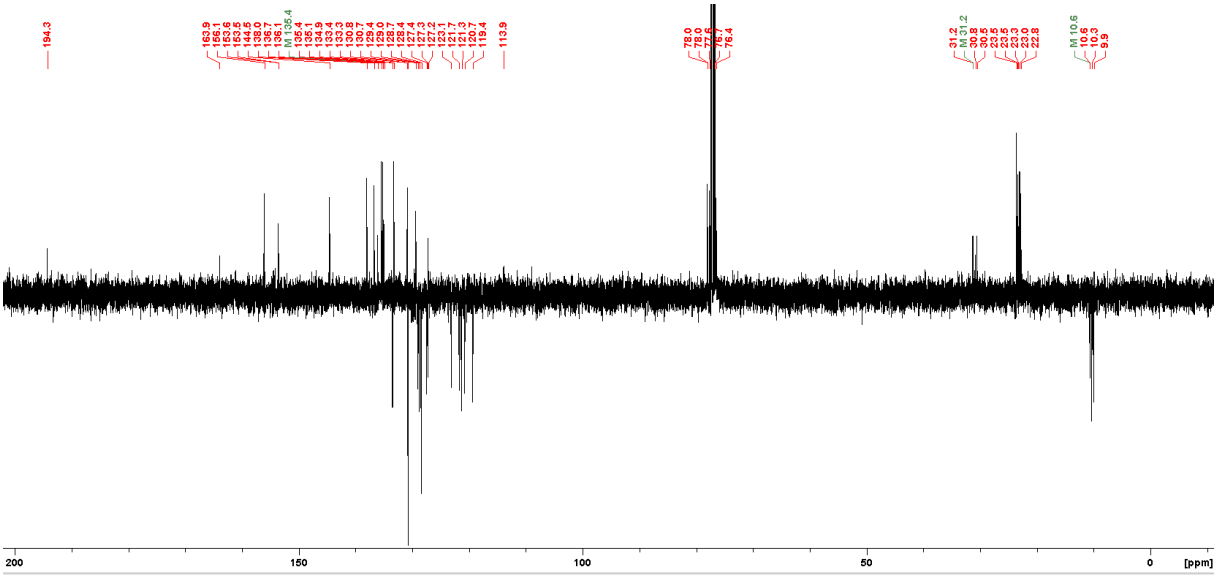


Figure 96: ^{13}C(APT) NMR of compound **14e** ($CDCl_3$, 100 MHz).

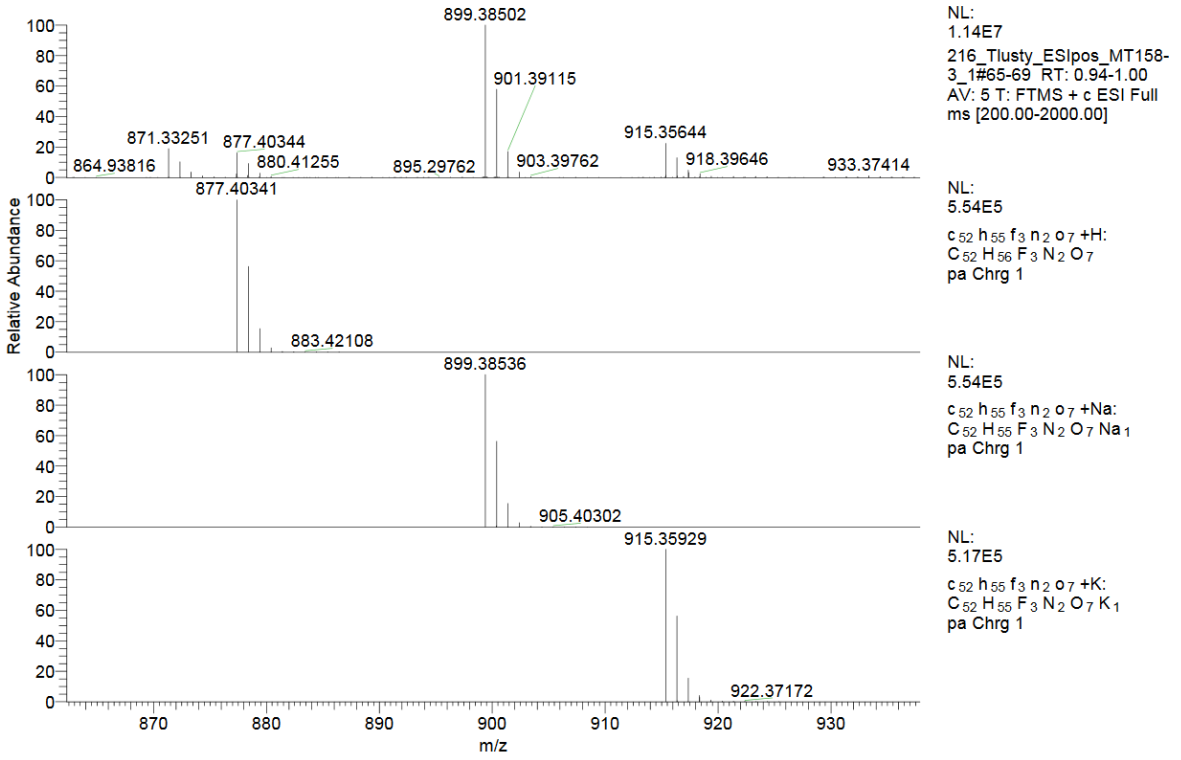


Figure 97: HRMS of compound **14e** (ESI+).

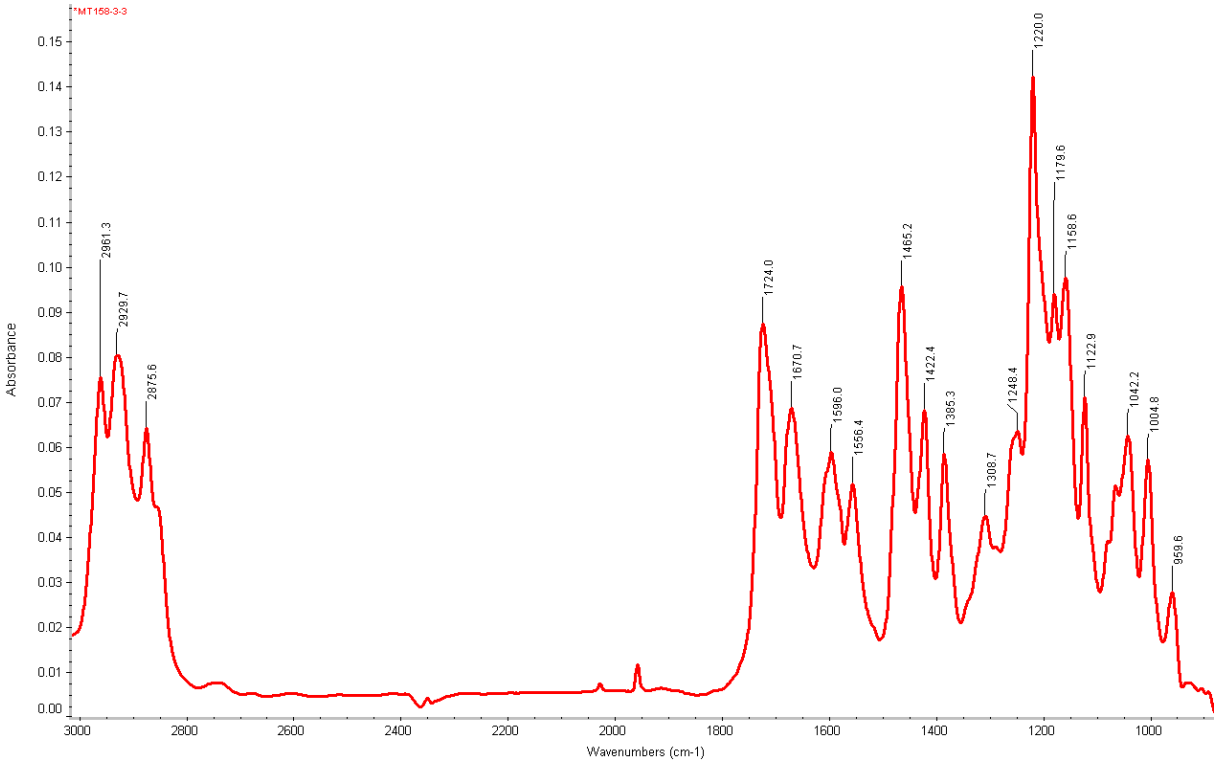


Figure 98: IR of compound **14e** (KBr).

2. Chiral separation and ECD spectra

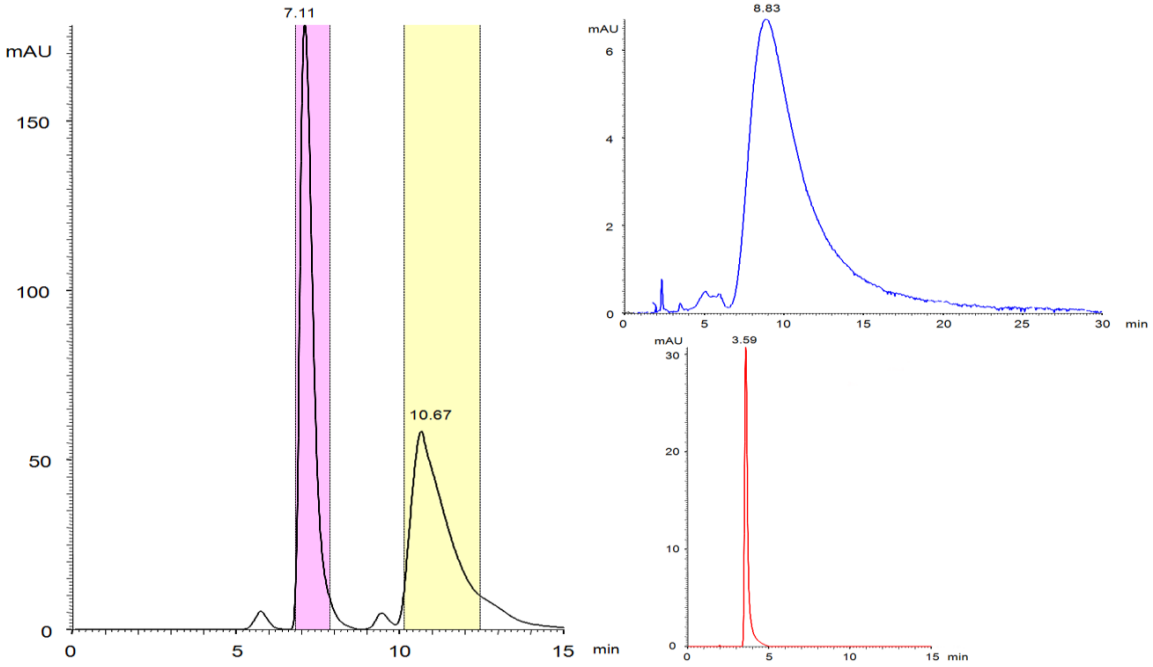


Figure 99: Preparative enantioseparation of **7b** on Chiralpak IA (250×20 mm i.d., 5 µm).

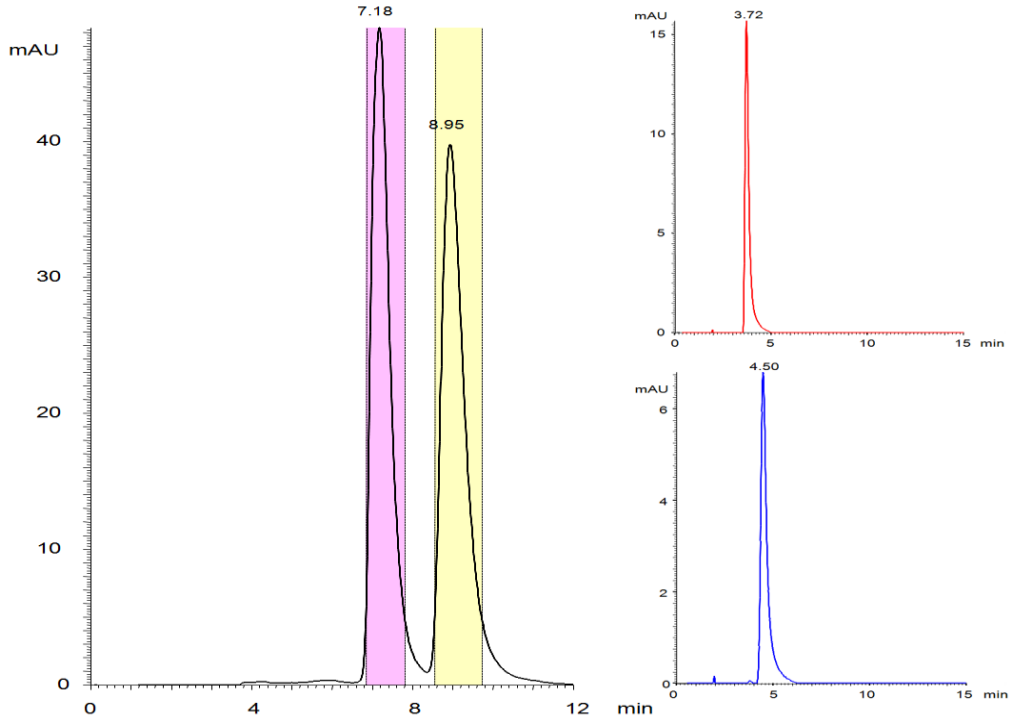


Figure 100: Preparative enantioseparation of **14b** on Chiralpak IA (250×20 mm i.d., 5 µm).

The enantiomeric nature of the separated substances was confirmed by ECD spectroscopy using Jasco J-810 (Jasco).

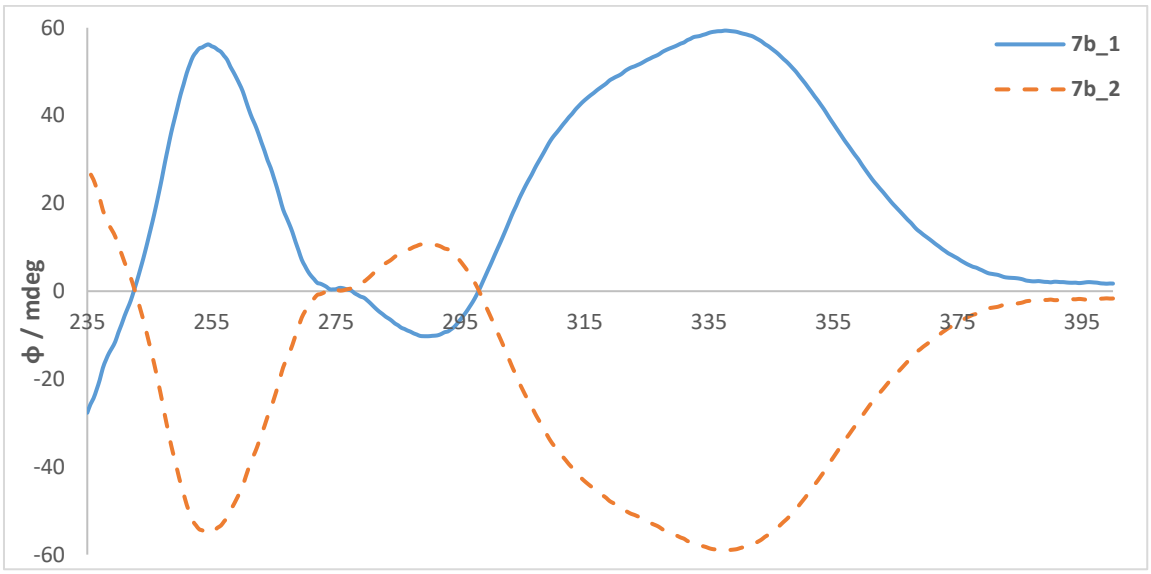


Figure 101: ECD spectra of both separated enantiomers of **7b** in MeOH, the first eluting enantiomer **7b_1** full (blue) line, the second eluting enantiomer **7b_2** dashed (orange) line.

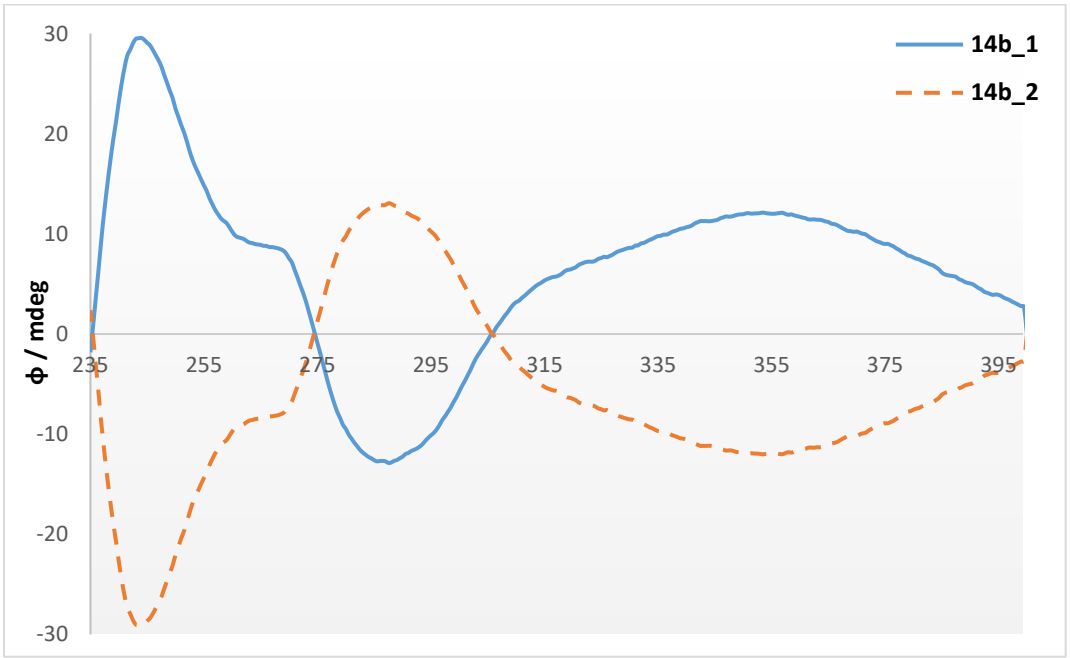


Figure 102: ECD spectra of both separated enantiomers of **14b** in MeOH, the first eluting enantiomer **14b_1** full (blue) line, the second eluting enantiomer **14b_2** dashed (orange) line.

3. Crystallographic data

Crystallographic data for ***7b-1***

The structure of **7b** was measured using D8 VENTURE equipped with Photon CMOS detector with Cu-Ka (λ = 1.54178 Å) radiation at 180 K. The structure was in hexagonal system, *P*61 space group with lattice parameters *a*=14.5403(3) Å, *b*=14.5403(3) Å, *c*=36.4429(8) Å, *α*=90° *β*=90° *γ*=120°, *Z* = 6, *V* = 6672.5(4) Å^3, D_c= 1.199 g/cm^3, *μ* (Cu-K α) = 0.713 mm^{-1}. The data reduction and absorption correction were done with Apex3 software. The structure was solved by chargeflipping methods using SIR92 software and refined by full matrix least squares on *F* squared value using Crystals software to final values *R* = 0.0438 and *wR* =0.1004 using 8653 independent reflections (Θ_{max}= 72.100°), 609 parameters and 42 restraint. MCE software was used for visualization of residual electron density maps. According to common practice the hydrogen atoms attached to carbon atoms were place geometrically with U_{iso}(H) in range 1.2–1.5 U_{eq} of parent atom (C). The crystal is a solid solution of two chemical entities.[x1] The difference between them is the chlorine atom bonded in the para position to the one of propoxy group. The occupancy of the molecule, which contains chlorine, is 0.23. The ordering is random, there are no peaks in the pattern which can show the supercell. The disordered functional groups were refined with restrained geometry and occupancy constrained to full for each atomic position. The structure was deposited into Cambridge Structural Database under number CCDC 1918213.

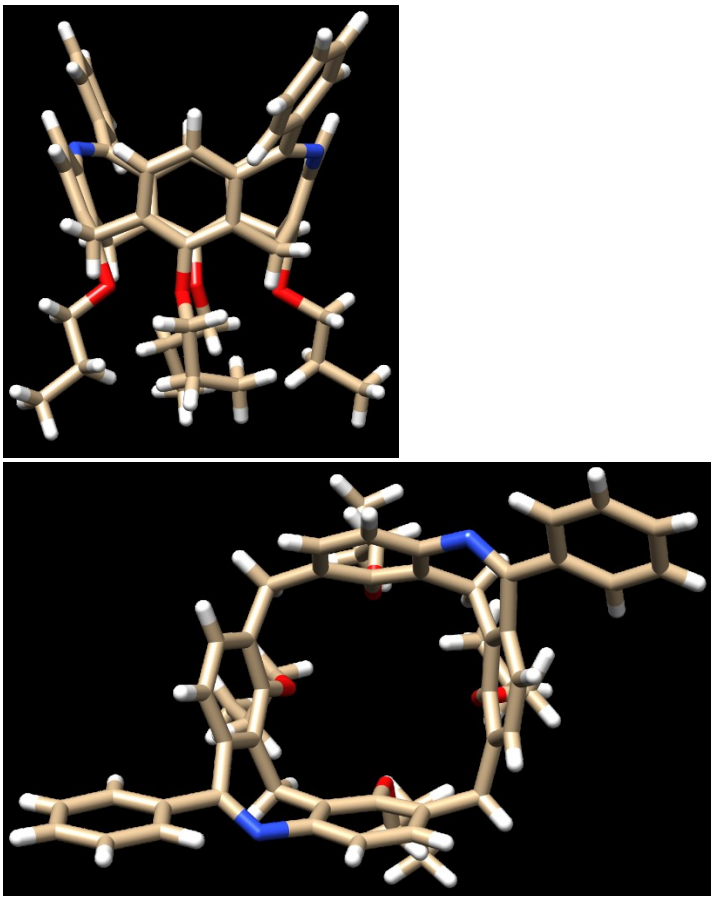


Figure 103: X-ray structure of compound **7b-1**.

*Crystallographic data for **8b***

Larger prism crystal of **8b** was selected, immersed in high viscosity PEG oil and cut to size appropriate for data collection. Data were collected at 180 (2) K on a D8 Venture Photon CMOS diffractometer with Incoatec microfocus sealed tube Cu-Kα radiation. The crystal s found to be in monoclinic space group P21/c with lattice parameters $a = 12.0248$ (3) Å, $b = 20.0249$ (4) Å, $c = 40.0398$ (9) Å, $\beta = 93.8505$ (12) °, $V = 9619.6$ (4) Å^3, $Z = 8$. The structure was solved by charge flipping[x2] and anisotropically refined by full matrix least squares on F squared using the CRYSTALS suite of programs[x3] to final value $R = 0.064$ and $wR = 0.153$ using 17623 independent reflections ($\Theta_{max} = 68.5°$), 1456 parameters and 491 restrains. The disordered propoxy groups and solvent were refined with restrained geometry and thermal parameters. The sum occupancy of disordered positions was restrained to 1 for each group. The hydrogen atoms attached to carbon atoms were placed in calculated positions. The hydrogen atoms attached to oxygen and nitrogen atoms were found in difference electron density maps. In both cases were the hydrogen atoms refined with riding constrains after initial refinement of geometry. The MCE program[x4] was used for visualization of residual electron density maps. The structure was deposited into Cambridge Structural Database under number CCDC 1918371.

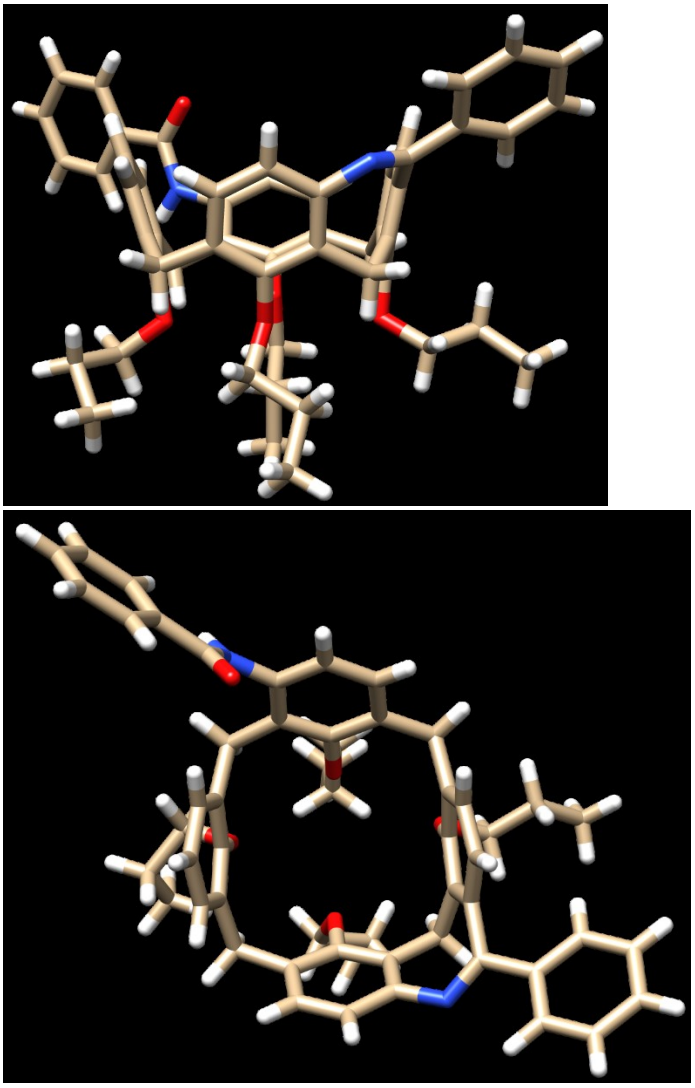
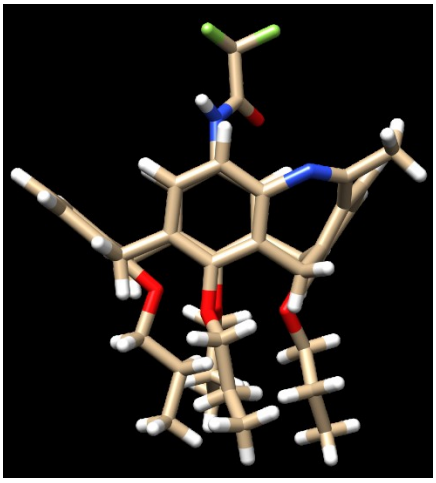


Figure 104: X-ray structure of compound **8b**.

*Crystallographic data for **14a***

Larger prism crystal of **14a** was selected, immersed in high viscosity PEG oil and cut to size appropriate for data collection. Data were collected at 180 (2) K on a D8 Venture Photon CMOS diffractometer with Incoatec microfocus sealed tube Cu-Kα radiation. The crystal s found to be in monoclinic space group $P2_1/c$ with lattice parameters $a = 13.9434$ (6) Å, $b = 17.1880$ (8) Å, $c = 17.1967$ (8) Å, $\beta = 105.4889$ (17) °, $V = 3971.7$ (3) Å^3, $Z = 4$. The structure was solved by charge flipping[x2] and anisotropically refined by full matrix least squares on F squared using the CRYSTALS suite of programs[x3] to final value $R = 0.045$ and $wR = 0.113$ using 7210 independent reflections ($\Theta_{max} = 68.4°$), 546 parameters and 58 restrains. The disordered propoxy groups were refined with restrained geometry and thermal parameters. The sum occupancy of disordered positions was restrained to 1 for each group. The hydrogen atoms attached to carbon atoms were placed in calculated positions. The hydrogen atoms attached to nitrogen atoms were found in difference electron density maps. In both cases were the hydrogen atoms refined with riding constrains after initial refinement of geometry. The MCE program[x4] was used for visualization of residual electron density maps. The structure was deposited into Cambridge Structural Database under number CCDC 1918372.



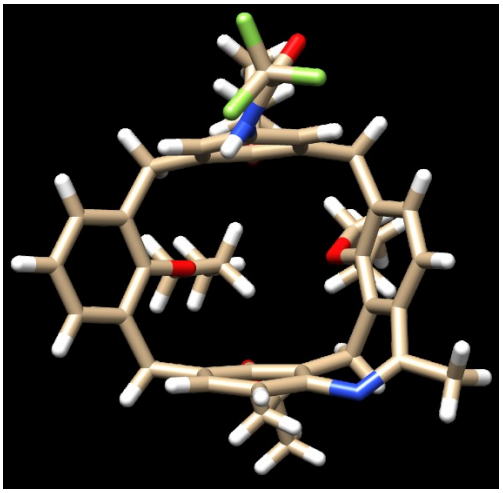


Figure 105: X-ray structure of compound **14a**.

[x1] Lusi, M. (2018). CrystEngComm 20, 7042-7052.

[x2] Palatinus, L., Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790.

[x3] Betteridge, P.W., Carruthers, J.R., Cooper, R.I., Prout, K. & Watkin, D.J. (2003). J. Appl. Cryst. 36, 1487.

[x4] Rohlicek J., Husak M.: J. Appl. Cryst. 40, 600 (2007)

4. Titration experiments

Nitromethane was mixed with specified amount of $CDCl_3$. 0.5 ml of nitromethane solution was put in NMR tube. To 0.12 ml of the nitromethane solution a specific amount of calixarene **7a** was added. The aliquots of **7a** were gradually added to NMR tube to achieve different guest / calixarene ratios (1:0.000-150.714), ensuring constant guest concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the guest molecule using nonlinear curve-fitting procedure (program BindFit).

	V(CH3NO2)	0.0041	ml				
	M(CH3NO2)	61.04	g/mol	M(**7a**)	670.894	g/mol	
	m(CH3NO2)	0.00466211	g	m(**7a**)	0.01102	g	
	c(CH3NO2)	0.000152756	mol/l	c(**7a**)	0.136882	mol/l	
	V(CHCl3_CH3NO2)	500	ml	V(CHCl3_**7a**)	0.12	ml	
	ρ(CH3NO2)	1.1371	g/ml				

	V (total) [m	V(addition, total) [n	V(addition) [ml]	c(ACN) [mol/l]	c(**7a**) [mol/l]	c(**7a**)/c(CH3N	shift [Hz]
1	0.500000	0.000000	0.000000	0.000153	0.000000	0.000000	617.93
2	0.500300	0.000300	0.000300	0.000153	0.000082	0.537328	617.54
3	0.500600	0.000600	0.000300	0.000153	0.000164	1.074011	617.54
4	0.501400	0.001400	0.000800	0.000153	0.000382	2.502028	618.32
5	0.502800	0.002800	0.001400	0.000153	0.000762	4.990123	619.10
6	0.505600	0.005600	0.002800	0.000153	0.001516	9.924975	621.45
7	0.508500	0.008500	0.002900	0.000153	0.002288	14.978779	623.80
8	0.511400	0.011400	0.002900	0.000153	0.003051	19.975266	625.75
9	0.517300	0.017300	0.005900	0.000153	0.004578	29.967608	630.06
10	0.523400	0.023400	0.006100	0.000153	0.006120	40.061812	634.75
11	0.529600	0.029600	0.006200	0.000153	0.007651	50.083214	641.40
12	0.563100	0.063100	0.033500	0.000153	0.015339	100.413542	669.18
13	0.601100	0.101100	0.038000	0.000153	0.023022	150.713752	683.31

K	2.78 M^{-1}
Error	0.1608981 M^{-1}

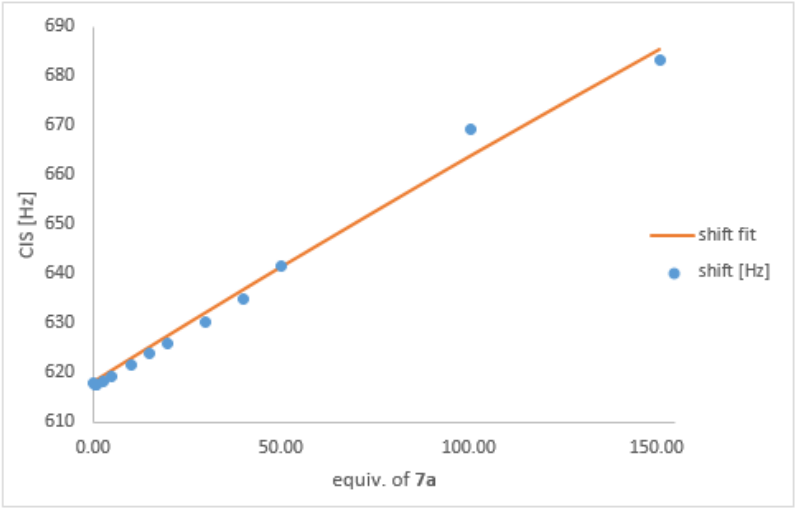


Figure 106: ^{1}H NMR titration of compound **7a** with nitromethane ($CDCl_3$, 400 MHz).

Calixarene **7b-1** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of (*S*)-*N*-methylnicotinium iodide (NMNI) was added. The aliquots of NMNI were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-11.896), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMNI)	304.1755 g/mol	M(**7b-1**)	795.036 g/mol
m(NMNI)	0.00595 g	m(**7b-1**)	0.0013125 g
c(NMNI)	0.032602 mol/l	c(**7b-1**)	0.0013757 mol/l
V(CHCl3_I	0.6 ml	V(CHCl3_7	1.2 ml

	V (total) [ml]	V(additio	V(additio	c(NMNI) [	c(**7b-1**) [m	c(**7b-1**)/c(N	shift 1 [Hz	shift 2 [Hz	shift 3 [Hz	shift 4 [Hz	shift 5 [Hz]
1	0.500000	0.000000	0.000000	0.000000	0.001376	0.000000	3230.07	3295.41	2404.08	3478.92	2339.92
2	0.501200	0.001200	0.001200	0.000078	0.001376	0.056739	3226.15	3295.41	2403.30	3478.53	2339.52
3	0.502400	0.002400	0.001200	0.000156	0.001376	0.113207	3221.85	3294.23	2400.95	3477.35	2338.74
4	0.505900	0.005900	0.003500	0.000380	0.001376	0.276374	3210.89	3293.06	2397.82	3475.40	2337.57
5	0.511800	0.011800	0.005900	0.000752	0.001376	0.546376	3197.20	3291.11	2393.52	3472.66	2336.00
6	0.517800	0.017800	0.006000	0.001121	0.001376	0.814645	3190.55	3290.71	2391.56	3471.48	2335.22
7	0.524000	0.024000	0.006200	0.001493	0.001376	1.085401	3185.46	3290.32	2390.39	3470.70	2334.83
8	0.554000	0.054000	0.030000	0.003178	0.001376	2.309906	3176.46	3290.71	2388.83	3469.92	2335.22
9	0.584000	0.084000	0.030000	0.004689	0.001376	3.408605	3172.55	3291.50	2389.22	3469.53	2335.61
10	0.624000	0.124000	0.040000	0.006479	0.001376	4.709202	3169.42	3292.28	2389.22	3469.53	2336.39
11	0.674000	0.174000	0.050000	0.008416	0.001376	6.117861	3166.29	3293.06	2389.61	3469.53	2336.79
12	0.734000	0.234000	0.060000	0.010393	0.001376	7.554923	3163.94	3294.23	2389.61	3469.53	2337.18
13	0.814000	0.314000	0.080000	0.012576	0.001376	9.141459	3161.59	3295.02	2390.00	3469.53	2337.57
14	0.904000	0.404000	0.090000	0.014570	0.001376	10.590664	3160.03	3295.80	2390.39	3469.53	2337.96
15	1.004000	0.504000	0.100000	0.016366	0.001376	11.896168	3158.07	3296.58	2390.78	3469.53	2338.35

K11	329.42	M^{-1}
Error	10.07037	M^{-1}
K21	5895.99	M^{-1}
Error	270.6672	M^{-1}

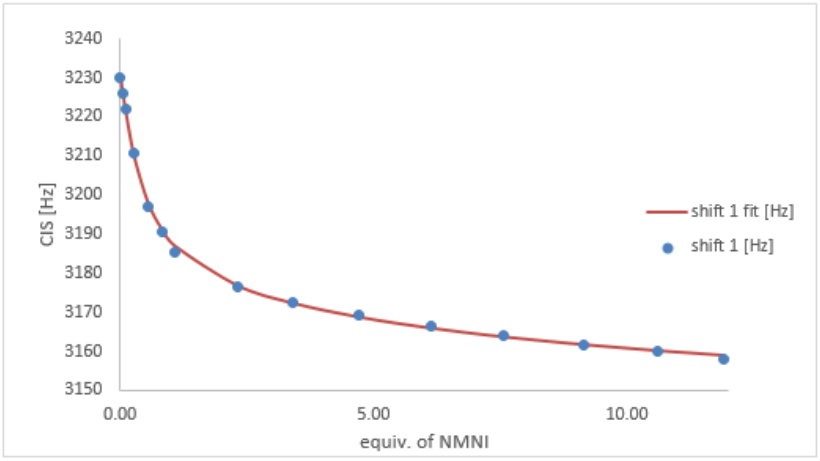


Figure 107: ^{1}H NMR titration of compound **7b-1** with NMNI ($C_2D_2Cl_4$, 400 MHz).

Calixarene **7b-2** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of (*S*)-*N*-methylnicotinium iodide (NMNI) was added. The aliquots of NMNI were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-11.715), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMNI)	304.1755 g/mol	M(**7b-2**)	795.036 g/mol
m(NMNI)	0.00625 g	m(**7b-2**)	0.0014 g
c(NMNI)	0.034246 mol/l	c(**7b-2**)	0.0014674 mol/l
V(CHCl3_I	0.6 ml	V(CHCl3_T	1.2 ml

	V (total) [ml]	V(additio	V(additio	c(NMNI) [	c(**7b-2**) [m	c(**7b-2**)/c(N	shift [Hz]	shift 2 [Hz	shift 3 [Hz	shift 4 [Hz	shift 5 [Hz	shift 6 [Hz]
1	0.500000	0.000000	0.000000	0.000000	0.001467	0.000000	3220.78	3135.09	3273.99	3471.97	2403.40	2339.23
2	0.501200	0.001200	0.001200	0.000082	0.001467	0.055875	3216.86	3135.48	3273.21	3471.58	2402.23	2339.23
3	0.502400	0.002400	0.001200	0.000164	0.001467	0.111482	3212.17	3135.48	3272.03	3470.80	2400.66	2338.84
4	0.505900	0.005900	0.003500	0.000399	0.001467	0.272165	3199.65	3134.70	3268.51	3467.28	2395.97	2336.49
5	0.511800	0.011800	0.005900	0.000790	0.001467	0.538055	3187.52	3134.30	3265.77	3465.71	2392.45	2335.32
6	0.517800	0.017800	0.006000	0.001177	0.001467	0.802237	3180.87	3133.91	3264.60	3464.54	2390.49	2335.32
7	0.524000	0.024000	0.006200	0.001569	0.001467	1.068869	3176.17	3133.52	3263.42	3463.76	2389.32	2334.54
8	0.554000	0.054000	0.030000	0.003338	0.001467	2.274723	3169.13	3132.35	3262.25	3462.58	2388.14	2334.54
9	0.584000	0.084000	0.030000	0.004926	0.001467	3.356688	3166.00	3131.56	3262.25	3462.58	2388.14	2334.93
10	0.624000	0.124000	0.040000	0.006805	0.001467	4.637476	3164.04	3130.78	3262.64	3462.19	2388.53	2335.71
11	0.674000	0.174000	0.050000	0.008841	0.001467	6.024679	3162.08	3130.39	3262.64	3462.19	2388.93	2336.10
12	0.734000	0.234000	0.060000	0.010918	0.001467	7.439853	3160.52	3129.22	3263.03	3462.19	2388.93	2336.49
13	0.814000	0.314000	0.080000	0.013210	0.001467	9.002224	3158.56	3128.43	3263.42	3462.19	2389.32	2337.28
14	0.904000	0.404000	0.090000	0.015304	0.001467	10.429357	3157.39	3128.04	3263.82	3462.19	2389.71	2337.28
15	1.004000	0.504000	0.100000	0.017191	0.001467	11.714976	3156.61	3127.65	3264.21	3462.19	2390.10	2337.67

K11	447.73	M^{-1}
Error	15.62981	M^{-1}
K21	5755.38	M^{-1}
Error	265.2079	M^{-1}

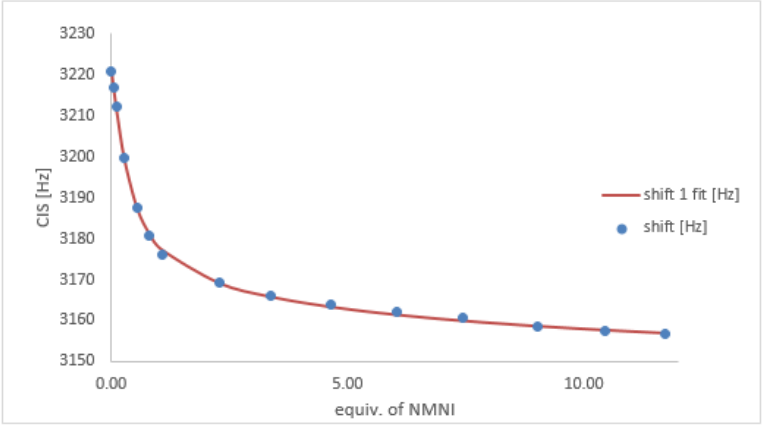


Figure 108: ^{1}H NMR titration of compound **7b-2** with NMNI ($C_2D_2Cl_4$, 400 MHz).

Calixarene **11** was dissolved in specified amount of $CDCl_3$. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of tetrabutylammonium acetate (TBAA) was added. The aliquots of TBAA were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-39.765), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of fluorines of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(TBAA)	301.51 g/mol	M(**11**)	814.866 g/mol
m(TBAA)	0.02384 g	m(**11**)	0.0008088 g
c(TBAA)	0.131781146 mol/l	c(**11**)	0.0008271 mol/l
V(CHCl3_TBA	0.6 ml	V(CHCl3_**11**	1.2 ml

	V (total) [m	V(addition, t	V(addition) [	c(TBAA) [mo	c(**11**) [mol/l	c(TBAA)/c(1	shift [Hz]	shift 2 [Hz]
1	0.500000	0.000000	0.000000	0.000000	0.000827	0.000000	-21280.1	-21430.7
2	0.500300	0.000300	0.000300	0.000079	0.000827	0.095537	-21279.1	-21430.7
3	0.500700	0.000700	0.000400	0.000184	0.000827	0.222741	-21280.1	-21431.7
4	0.501300	0.001300	0.000600	0.000342	0.000827	0.413167	-21279.1	-21430.7
5	0.501900	0.001900	0.000600	0.000499	0.000827	0.603137	-21276.1	-21429.7
6	0.502500	0.002500	0.000600	0.000656	0.000827	0.792654	-21275.2	-21429.7
7	0.503800	0.003800	0.001300	0.000994	0.000827	1.201725	-21272.2	-21427.7
8	0.507600	0.007600	0.003800	0.001973	0.000827	2.385457	-21263.3	-21423.8
9	0.512800	0.012800	0.005200	0.003289	0.000827	3.976871	-21249.4	-21414.9
10	0.519500	0.019500	0.006700	0.004947	0.000827	5.980378	-21234.5	-21406.9
11	0.526300	0.026300	0.006800	0.006585	0.000827	7.961629	-21220.7	-21400
12	0.555300	0.055300	0.029000	0.013124	0.000827	15.866350	-21173.1	-21373.2
13	0.588300	0.088300	0.033000	0.019779	0.000827	23.913406	-21134.5	-21352.4
14	0.666300	0.166300	0.078000	0.032891	0.000827	39.765094	-21083.9	-21322.7

K	21.70	M^{-1}
Error	0.3892763	M^{-1}

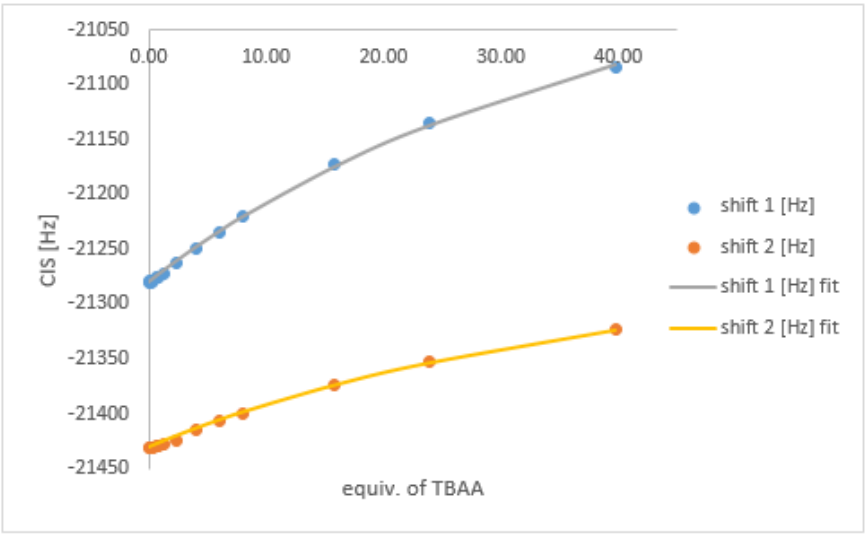


Figure 109: ^{19}F NMR titration of compound **11** with TBBA ($CDCl_3$, 376 MHz).

Calixarene **12a** was dissolved in specified amount of $CDCl_3$. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of tetrabutylammonium acetate (TBAA) was added. The aliquots of TBAA were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-82.716), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of fluorines of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(TBAA)	301.51 g/mol	M(**12a**)	718.858 g/mol
m(TBAA)	0.0486 g	m(**12a**)	0.0014 g
c(TBAA)	0.26864781 mol/l	c(**12a**)	0.001622945 mol/l
V($CHCl_3$_TBA	0.6 ml	V($CHCl_3$_**12a**)	1.2 ml

	V (total) [m	V(addition,	V(addition)	c(TBAA) [m	c(**12a**) [mol/l]	c(TBAA)/c(**12**	shift [Hz]
1	0.500000	0.000000	0.000000	0.000000	0.001623	0.000000	-21354.4
2	0.500200	0.000200	0.000200	0.000107	0.001623	0.066186	-21355.4
3	0.500600	0.000600	0.000400	0.000322	0.001623	0.198399	-21354.4
4	0.501200	0.001200	0.000600	0.000643	0.001623	0.396323	-21353.4
5	0.501800	0.001800	0.000600	0.000964	0.001623	0.593774	-21351.4
6	0.502400	0.002400	0.000600	0.001283	0.001623	0.790754	-21350.5
7	0.503600	0.003600	0.001200	0.001920	0.001623	1.183304	-21347.5
8	0.507300	0.007300	0.003700	0.003866	0.001623	2.381977	-21337.6
9	0.512400	0.012400	0.005100	0.006501	0.001623	4.005827	-21324.7
10	0.518800	0.018800	0.006400	0.009735	0.001623	5.998429	-21308.8
11	0.525400	0.025400	0.006600	0.012988	0.001623	8.002455	-21294
12	0.553400	0.053400	0.028000	0.025923	0.001623	15.972825	-21238.5
13	0.584400	0.084400	0.031000	0.038799	0.001623	23.906272	-21191.9
14	0.660400	0.160400	0.076000	0.065250	0.001623	40.204709	-21120.6
15	0.999400	0.499400	0.339000	0.134243	0.001623	82.715864	-21024.5

K	9.5	M^{-1}
Error	0.3033065	M^{-1}

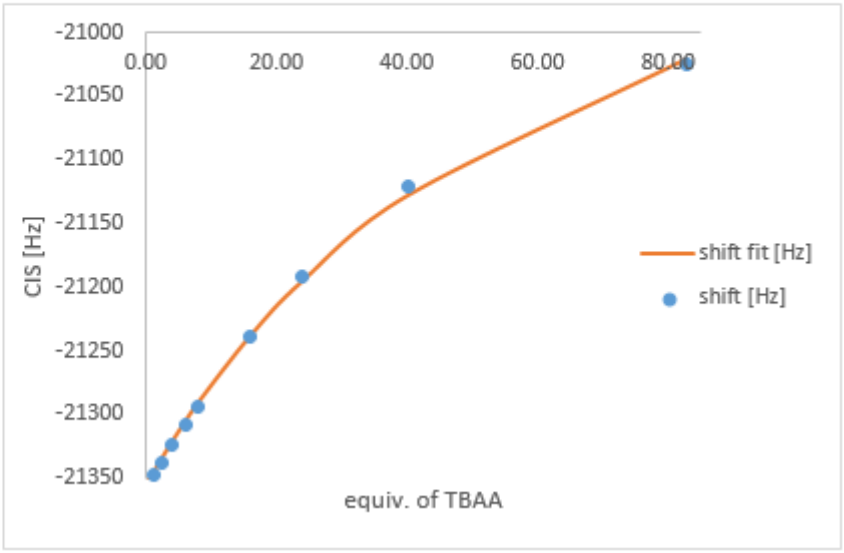


Figure 110: ^{19}F NMR titration of compound **12a** with TBBA ($CDCl_3$, 376 MHz).

Calixarene **14b** was dissolved in specified amount of $CDCl_3$. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of acetonitrile (ACN) was added. The aliquots of ACN were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-101.038), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

V(ACN)	0.008	ml			
M(ACN)	41.05	g/mol	M(**14b**)	804.93	g/mol
m(ACN)	0.006288	g	m(**14b**)	0.001218	g
c(ACN)	0.255298417	mol/l	c(**14b**)	0.001261	mol/l
V(CHCl3_ACN)	0.6	ml	V(CHCl3_**14b**)	1.2	ml
ρ(ACN)	0.786	g/ml			

	V (total) [m	V(addition, total) [n	V(addition) [ml]	c(ACN) [mol/l]	c(**14b**) [mol/l]	c(**14b**)/c(ACN	shift [Hz]	shift 2 [Hz	shift 3 [Hz
1	0.500000	0.000000	0.000000	0.000000	0.001261	0.000000	2674.46	3174.51	2856.01
2	0.500300	0.000300	0.000300	0.000153	0.001261	0.121403	2674.06	3174.11	2856.40
3	0.500900	0.000900	0.000600	0.000459	0.001261	0.363774	2672.11	3172.55	2854.44
4	0.501600	0.001600	0.000700	0.000814	0.001261	0.645807	2672.50	3172.55	2854.83
5	0.502200	0.002200	0.000600	0.001118	0.001261	0.886924	2671.33	3171.38	2853.27
6	0.503500	0.003500	0.001300	0.001775	0.001261	1.407372	2668.20	3167.85	2850.53
7	0.507300	0.007300	0.003800	0.003674	0.001261	2.913387	2665.07	3165.90	2846.62
8	0.512600	0.012600	0.005300	0.006275	0.001261	4.976593	2663.11	3162.38	2844.66
9	0.519300	0.019300	0.006700	0.009488	0.001261	7.524527	2658.02	3157.29	2839.97
10	0.526100	0.026100	0.006800	0.012665	0.001261	10.044132	2658.02	3157.68	2839.57
11	0.556100	0.056100	0.030000	0.025755	0.001261	20.424440	2648.24	3147.90	2832.14
12	0.588100	0.088100	0.032000	0.038245	0.001261	30.329477	2645.50	3144.38	2829.40
13	0.668100	0.168100	0.080000	0.064235	0.001261	50.940879	2641.98	3140.07	2827.05
14	0.998100	0.498100	0.330000	0.127406	0.001261	101.037520	2637.28	3133.42	2824.31

K	71.92 M^{-1}
Error	4.55908072 M^{-1}

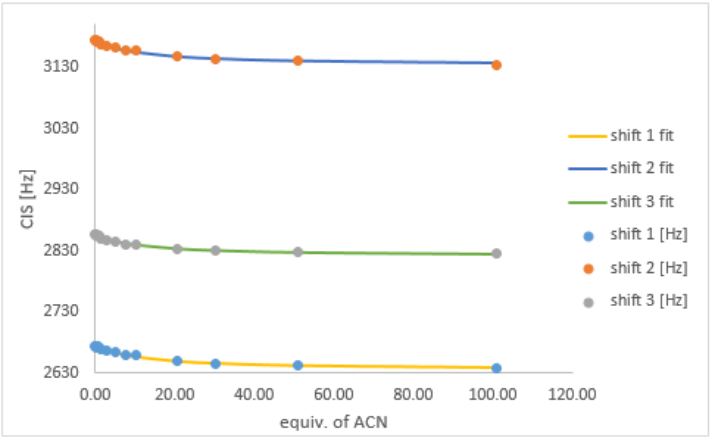


Figure 111: ^{1}H NMR titration of compound **14b** with ACN ($CDCl_3$, 400 MHz).

Calixarene **14b** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of *N*-methylpyridinium iodide (NMPI) was added. The aliquots of NMPI were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-11.955), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMPI)	221.04147 g/mol	M(**14b**)	804.93 g/mol
m(NMPI)	0.00436 g	m(**14b**)	0.001333333 g
c(NMPI)	0.032874676 mol/l	c(**14b**)	0.001380 mol/l
V(C2D2Cl4_NMPI)	0.6 ml	V(C2D2Cl4_**14b**)	1.2 ml

	V (total) [m	V(addition, total) [n	V(addition) [ml]	c(NMPI) [mol/l]	c(**14b**) [mol/l]	c(**14b**)/c(NMI	shift [Hz]	shift 2 [Hz	shift 3 [Hz]
1	0.500000	0.000000	0.000000	0.000000	0.001380	0.000000	3275.85	3349.95	3230.46
2	0.501200	0.001200	0.001200	0.000079	0.001380	0.057021	3279.37	3352.54	3226.15
3	0.502400	0.002400	0.001200	0.000157	0.001380	0.113769	3282.11	3358.01	3221.46
4	0.505900	0.005900	0.003500	0.000383	0.001380	0.277747	3293.09	3373.27	3213.63
5	0.511800	0.011800	0.005900	0.000758	0.001380	0.549090	3307.54	3394.01	3199.55
6	0.517800	0.017800	0.006000	0.001130	0.001380	0.818691	3320.06	3412.40	3193.29
7	0.524000	0.024000	0.006200	0.001506	0.001380	1.090792	3329.84	3427.66	3183.15
8	0.554000	0.054000	0.030000	0.003204	0.001380	2.321379	3359.58	3471.87	3159.25
9	0.584000	0.084000	0.030000	0.004729	0.001380	3.425536	3375.62	3493.79	3149.46
10	0.624000	0.124000	0.040000	0.006533	0.001380	4.732593	3385.01	3507.87	3141.25
11	0.674000	0.174000	0.050000	0.008487	0.001380	6.148249	3390.88	3518.83	3136.16
12	0.734000	0.234000	0.060000	0.010480	0.001380	7.592449	3397.53	3525.48	3132.64
13	0.814000	0.314000	0.080000	0.012681	0.001380	9.186865	3402.62	3531.35	3129.90
14	0.904000	0.404000	0.090000	0.014692	0.001380	10.643269	3404.97	3535.26	3126.77
15	1.004000	0.504000	0.100000	0.016503	0.001380	11.955257	3406.14	3537.10	3124.81

K	615.49 M^{-1}
Error	14.86962291 M^{-1}

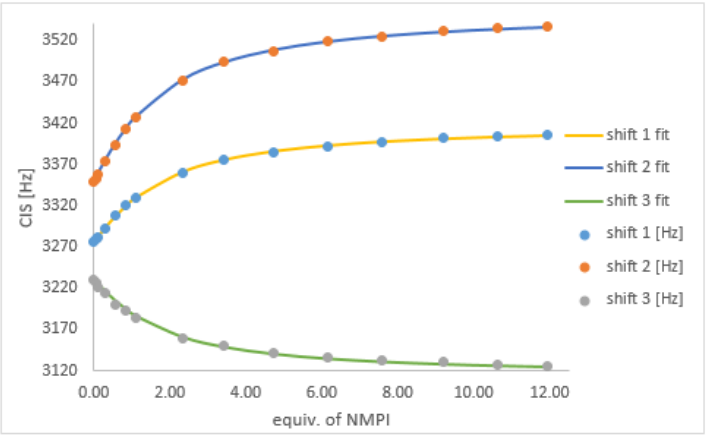


Figure 112: ^{1}H NMR titration of compound **14b** with NMPI ($CDCl_2$-$CDCl_2$, 400 MHz).

Calixarene **14b** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of *N*-methylisoquinolinium iodide (NMII) was added. The aliquots of NMII were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-12.867), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMII)	271.10147 g/mol	M(**14b**)	804.93 g/mol
m(NMII)	0.00524 g	m(**14b**)	0.001214 g
c(NMII)	0.03221426 mol/l	c(**14b**)	0.001257 mol/l
V(C2D2Cl4_NMII)	0.6 ml	V(C2D2Cl4_**14b**	1.2 ml

	V (total) [m	V(addition, total) [n	V(addition) [ml]	c(NMII) [mol/l]	c(**14b**) [mol/l]	c(14b)/c(NMI	shift [Hz]	shift 2 [Hz
1	0.500000	0.000000	0.000000	0.000000	0.001257	0.000000	3218.33	3192.11
2	0.501200	0.001200	0.001200	0.000077	0.001257	0.061368	3217.94	3193.29
3	0.502400	0.002400	0.001200	0.000154	0.001257	0.122442	3215.98	3194.85
4	0.505900	0.005900	0.003500	0.000376	0.001257	0.298921	3213.24	3199.16
5	0.511800	0.011800	0.005900	0.000743	0.001257	0.590950	3208.94	3205.02
6	0.517800	0.017800	0.006000	0.001107	0.001257	0.881103	3203.85	3211.28
7	0.524000	0.024000	0.006200	0.001475	0.001257	1.173948	3200.33	3215.59
8	0.554000	0.054000	0.030000	0.003140	0.001257	2.498347	3188.20	3232.80
9	0.584000	0.084000	0.030000	0.004634	0.001257	3.686679	3179.20	3244.15
10	0.624000	0.124000	0.040000	0.006402	0.001257	5.093378	3171.77	3253.93
11	0.674000	0.174000	0.050000	0.008316	0.001257	6.616955	3166.29	3261.37
12	0.734000	0.234000	0.060000	0.010270	0.001257	8.171253	3161.20	3267.24
13	0.814000	0.314000	0.080000	0.012427	0.001257	9.887217	3157.68	3271.93
14	0.904000	0.404000	0.090000	0.014397	0.001257	11.454649	3154.55	3275.85
15	1.004000	0.504000	0.100000	0.016171	0.001257	12.866655	3152.20	3277.80

K	211.16 M^{-1}
Error	2.1686132 M^{-1}

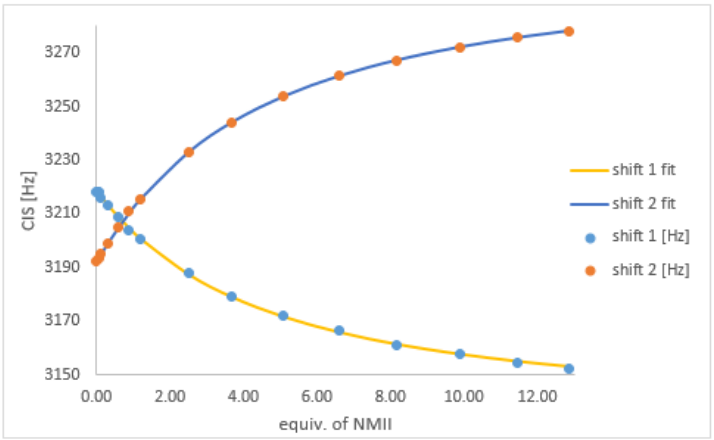


Figure 113: ^{1}H NMR titration of compound **14b** with NMII ($CDCl_2$-$CDCl_2$, 400 MHz).

Calixarene **14b** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of *N*-methylquinolinium iodide (NMQI) was added. The aliquots of NMQI were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-12.351), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMQI)	271.10147 g/mol	M(**14b**)	804.93 g/mol
m(NMQI)	0.00503 g	m(**14b**)	0.001214 g
c(NMQI)	0.030923231 mol/l	c(**14b**)	0.001257 mol/l
V(C2D2Cl4_NMQI)	0.6 ml	V(C2D2Cl4_**14b**	1.2 ml

	V (total) [m	V(addition, total) [n	V(addition) [ml]	c(NMQI) [mol/l]	c(**14b**) [mol/l]	c(14b)/c(NM	shift [Hz]	shift 2 [Hz]
1	0.500000	0.000000	0.000000	0.000000	0.001257	0.000000	3217.55	3192.50
2	0.501200	0.001200	0.001200	0.000074	0.001257	0.058908	3212.46	3196.03
3	0.502400	0.002400	0.001200	0.000148	0.001257	0.117535	3207.37	3199.16
4	0.505900	0.005900	0.003500	0.000361	0.001257	0.286941	3193.68	3208.16
5	0.511800	0.011800	0.005900	0.000713	0.001257	0.567267	3173.72	3221.85
6	0.517800	0.017800	0.006000	0.001063	0.001257	0.845792	3156.90	3232.80
7	0.524000	0.024000	0.006200	0.001416	0.001257	1.126900	3142.42	3242.59
8	0.554000	0.054000	0.030000	0.003014	0.001257	2.398223	3093.90	3273.89
9	0.584000	0.084000	0.030000	0.004448	0.001257	3.538930	3066.90	3292.28
10	0.624000	0.124000	0.040000	0.006145	0.001257	4.889254	3045.78	3305.97
11	0.674000	0.174000	0.050000	0.007983	0.001257	6.351772	3030.12	3316.54
12	0.734000	0.234000	0.060000	0.009858	0.001257	7.843779	3018.00	3323.58
13	0.814000	0.314000	0.080000	0.011929	0.001257	9.490974	3009.78	3328.28
14	0.904000	0.404000	0.090000	0.013820	0.001257	10.995588	3003.13	3332.97
15	1.004000	0.504000	0.100000	0.015523	0.001257	12.351007	2998.43	3334.93

K	394.45 M^{-1}
Error	3.8529876 M^{-1}

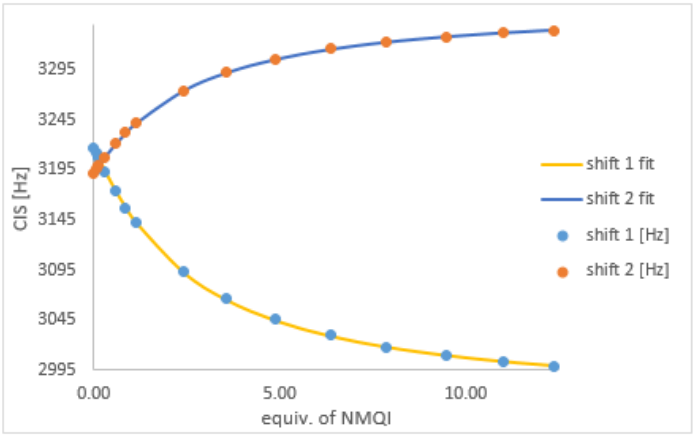


Figure 114: ^{1}H NMR titration of compound **14b** with NMQI ($CDCl_2$-$CDCl_2$, 400 MHz).

Calixarene **14b-1** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of (*S*)-*N*-methylnicotinium iodide (NMNI) was added. The aliquots of NMNI were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-11.008), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMNI)	304.1755 g/mol	M(**14b-1**)	804.93 g/mol
m(NMNI)	0.00503 g	m(**14b-1**)	0.001214 g
c(NMNI)	0.027561 mol/l	c(**14b-1**)	0.001257 mol/l
V(C2D2Cl4	0.6 ml	V(C2D2Cl4	1.2 ml

	V (total) [ml]	V(additio	V(additio	c(NMNI) [	c(**14b-1**) [	c(**14b-1**)/c(	shift 1 [Hz	shift 2 [Hz	shift NH
1	0.500000	0.000000	0.000000	0.000000	0.001257	0.000000	3155.33	3360.36	3528.22
2	0.501200	0.001200	0.001200	0.000066	0.001257	0.052503	3161.20	3360.36	3557.95
3	0.502400	0.002400	0.001200	0.000132	0.001257	0.104755	3167.46	3361.53	3588.87
4	0.505900	0.005900	0.003500	0.000321	0.001257	0.255741	3183.90	3363.88	3668.69
5	0.511800	0.011800	0.005900	0.000635	0.001257	0.505586	3200.72	3372.46	3759.85
6	0.517800	0.017800	0.006000	0.000947	0.001257	0.753826	3207.76	3381.1	3811.11
7	0.524000	0.024000	0.006200	0.001262	0.001257	1.004369	3211.68	3390.1	3846.32
8	0.554000	0.054000	0.030000	0.002686	0.001257	2.137456	3221.46	3420.62	3943.36
9	0.584000	0.084000	0.030000	0.003964	0.001257	3.154131	3226.15	3440.57	4013.01
10	0.624000	0.124000	0.040000	0.005477	0.001257	4.357630	3229.67	3458.96	4043.14
11	0.674000	0.174000	0.050000	0.007115	0.001257	5.661123	3231.24	3473.44	4080.31
12	0.734000	0.234000	0.060000	0.008786	0.001257	6.990899	3232.41	3484.79	4106.91
13	0.814000	0.314000	0.080000	0.010632	0.001257	8.458989	3233.59	3492.61	4130.39
14	0.904000	0.404000	0.090000	0.012317	0.001257	9.800002	3233.98	3501.22	4148.39
15	1.004000	0.504000	0.100000	0.013835	0.001257	11.008041	3234.37	3508.26	4162.87

K11	723.04	M^{-1}
Error	24.94271	M^{-1}
K21	4892.32	M^{-1}
Error	262.2968	M^{-1}

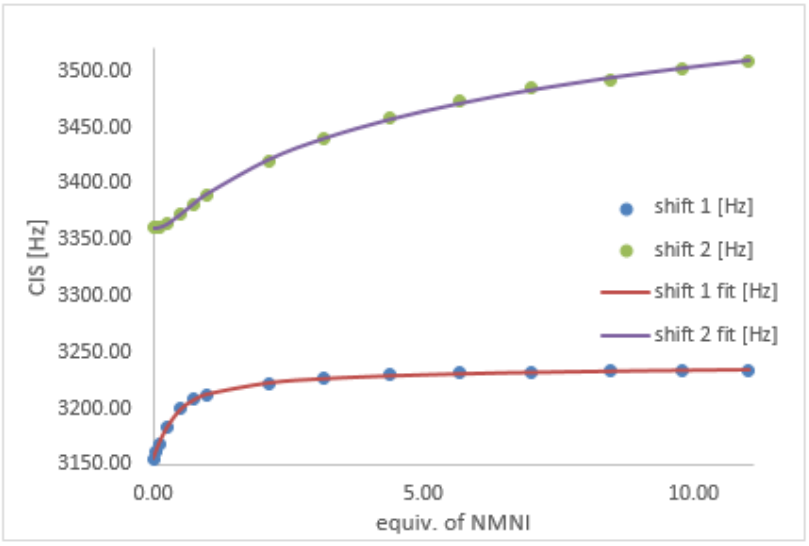


Figure 115: ^{1}H NMR titration of compound **14b-1** with NMNI ($CDCl_2$-$CDCl_2$, 400 MHz).

Calixarene **14b-2** was dissolved in specified amount of 1,1,2,2-tetrachloroethane. 0.5 ml of calixarene solution was put in NMR tube. To 0.6 ml of the calixarene solution a specific amount of (*S*)-*N*-methylnicotinium iodide (NMNI) was added. The aliquots of NMNI were gradually added to NMR tube to achieve different calixarene/guest ratios (1:0.000-12.077), ensuring constant host concentration during the experiment. The complexation constants were determined by analyzing CIS of protons of the host molecule using nonlinear curve-fitting procedure (program BindFit).

M(NMNI)	304.1755 g/mol	M(**14b**-2)	804.93 g/mol
m(NMNI)	0.00626 g	m(**14b**-2)	0.0013771 g
c(NMNI)	0.0343 mol/l	c(**14b**-2)	0.001426 mol/l
V(C2D2Cl4	0.6 ml	V(C2D2Cl4	1.2 ml

	V (total) [ml]	V(additio	V(additio	c(NMNI) [	c(**14b**-2) [	c(**14b**-2)/c(	shift 1 [Hz	shift 2 [Hz	shift NH
1	0.500000	0.000000	0.000000	0.000000	0.001426	0.000000	3155.33	3358.8	3528.22
2	0.501200	0.001200	0.001200	0.000082	0.001426	0.057601	3158.07	3360.75	3548.56
3	0.502400	0.002400	0.001200	0.000164	0.001426	0.114927	3160.42	3362.71	3570.08
4	0.505900	0.005900	0.003500	0.000400	0.001426	0.280573	3166.68	3369.75	3622.51
5	0.511800	0.011800	0.005900	0.000791	0.001426	0.554678	3176.46	3384.23	3682.38
6	0.517800	0.017800	0.006000	0.001179	0.001426	0.827022	3180.37	3393.62	3741.07
7	0.524000	0.024000	0.006200	0.001571	0.001426	1.101892	3185.07	3403.4	3778.24
8	0.554000	0.054000	0.030000	0.003343	0.001426	2.345002	3193.29	3437.44	3897.58
9	0.584000	0.084000	0.030000	0.004934	0.001426	3.460395	3199.94	3457.01	3964.49
10	0.624000	0.124000	0.040000	0.006816	0.001426	4.780754	3205.02	3476.57	4030.22
11	0.674000	0.174000	0.050000	0.008855	0.001426	6.210815	3208.55	3487.92	4077.18
12	0.734000	0.234000	0.060000	0.010935	0.001426	7.669712	3210.50	3500.05	4111.61
13	0.814000	0.314000	0.080000	0.013231	0.001426	9.280353	3212.46	3506.7	4139.00
14	0.904000	0.404000	0.090000	0.015329	0.001426	10.751578	3213.24	3514.52	4157.00
15	1.004000	0.504000	0.100000	0.017219	0.001426	12.076917	3214.42	3518.44	4171.47

K11	439.66 M^{-1}
Error	16.49077 M^{-1}
K21	1149.97 M^{-1}
Error	85.97636 M^{-1}

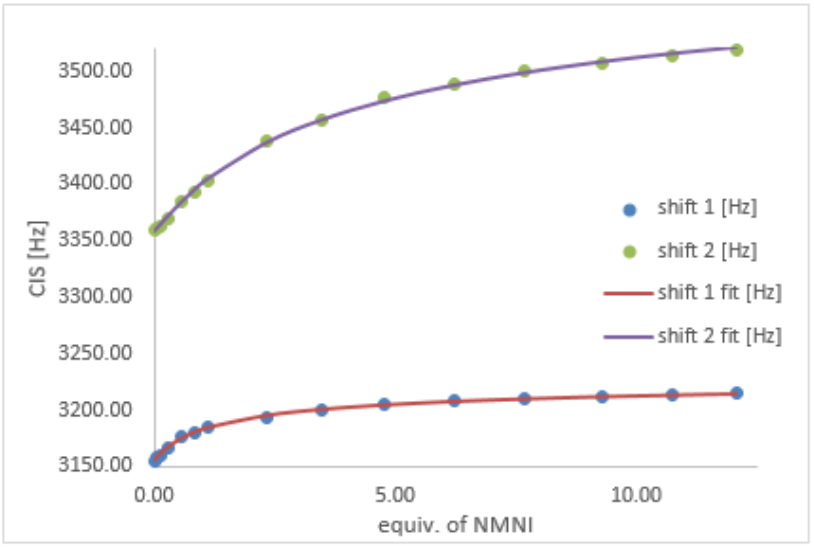


Figure 116: ^{1}H NMR titration of compound **14b-2** with NMNI ($CDCl_2$-$CDCl_2$, 400 MHz).