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Fluorinated oxacalix[6]azacryptands: synthesis and complexation with propargyl alcohol

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Contents

1. General information

Octafluorotoluene was obtained from P&M Invest (Dolgoprudny, Russia). Resorcinol was obtained from AppliChem Panreac. Ethyl 3,5-dihydroxybenzoate was synthesized from 3,5-dihydroxybenzoic acid according to the standard procedure.

The ^{19}F spectra were recorded for solutions in $CDCl_3$, acetone-d_6 and C_6D_6 on a Bruker AV300 spectrometer at 282.36 MHz, ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AV-600 instrument at 600.18 MHz and 150.9 MHz. Chemical shifts are given in δ ppm from CCl_3F (^{19}F) and TMS (^{13}C), J values in Hz; C_6F_6 (–162.9 ppm from CCl_3F), $CDCl_3$ (76.90 ppm from TMS), C_6D_6 (128.39 ppm from TMS) were used as internal standards. IR spectra were taken on a Bruker Vector 22 IR spectrophotometer.

Gas chromatographic–mass spectrometric analysis (GC-MS) was performed on a Hewlett Packard HP 5890 Series II chromatograph coupled with an HP 5971 mass-selective detector [HP-5MS capillary column, 30 m × 0.25 mm, film thickness 0.25 μm; carrier gas helium, flow rate 1 mL/min; oven temperature programming from 50 °C (2 min) to 280 °C at a rate of 10 deg/min and finally 5 min at 280 °C; injector temperature 280 °C; ion source temperature 175 °C; electron impact, 70 eV; 1.2 scan/s, a.m.u. range 30–800].

The X-ray diffraction experiments for complex **2·8**·$4.5C_4H_8O_2$·$2H_2O$ were carried out on a Bruker KAPPA APEX II diffractometer with graphite monochromated MoKa (λ = 0.71073 A) radiation. The structures were solved by direct methods and refined by full-matrix least-squares method against all F^2 in anisotropic approximation using the SHELXT-2014 and SHELXL-2018 set of programs. Absorption corrections were applied using the empirical multi-scan method with the SADABS program.

The elemental compositions of macrocycles **2** and **6** were determined by classical method and for diaryloxybenzene **7** from the high-resolution mass spectra (electron impact, 70 eV) which were obtained with a Thermo Scientific DFS instrument. The progress of reactions was monitored by TLC on silica gel 60 F254 plates (Merck). Silica gel with a particle size of 0.063–0.200 mm (Merck) was used for column chromatography.

Experimental section

*Synthesis of 1,3-bis(3-ethoxy-2,4,5-trifluoro-6-(trifluoromethyl)phenoxy)benzene **7**.*

a) A solution of 23.6 g (0.1 mmol) of octafluorotoluene in 10 ml of ethanol was added dropwise under stirring at 5 °C to a solution of 4.1 g (0.1 mmol) of NaOH in 60 ml of ethanol. The mixture was stirred 4 h at 20°C and 0.5 h at 50°C, treated with 100 mL of 5% HCl, and then by CH_2Cl_2 (3x50 mL). The extract was dried with $CaCl_2$ and evaporated. Vacuum distillation of the residue yielded 22.2 g (85%) of pure 4-ethoxyheptafluorotoluene (NMR ^{19}F, ^{1}H, GC–MS, M^+ 262). The ^{19}F NMR spectrum of the product coincides with the spectrum of 4-ethoxyheptafluorotoluene described in the literature [L. S. Kobrina, G. G. Furin, G. G. Yakobson, *Zh. Org. Khim.*, 1970, **6**, 512 (in Russian)].

b) Potassium carbonate (2.76 g, 20 mmol) was added with stirring to a solution of 4-ethoxy-heptafluorotoluene (1.97 g, 7.5 mmol), resorcinol (0.41 g, 3.7 mmol) and 18-crown-6 ether (complex with MeCN, 0.23 g, 0.75 mmol) in 40 ml of acetone. The mixture was stirred at 56 °C for 30 h and the solvent was evaporated. Silica gel column chromatography (eluent CCl_4) gave 1.34 g of a viscous material containing (GC–MS, NMR ^{19}F, ^{1}H) 93% of diaryloxybenzene **7** and 6% of 4-ethoxy-heptafluorotoluene. HRMS, m/z: M^+ 594.0690. Calculated $C_{24}H_{14}F_{12}O_4$ M^+ 594.0695. $^{19}FNMR$ ($CDCl_3$), δ: -154.3 (dd, 2F, $J_{F(4)-F(5)}$ 21 Hz, $J_{F(4)-F(2)}$ 4 Hz, F-4,4´), -145.4 (dd, 2F, $J_{F(2)-F(5)}$ 9 Hz, $J_{F(2)-F(4)}$ 4 Hz, F-2,2´), -141.8 (qdd, 2F, $J_{F(5)-CF_3}$ 25 Hz, $J_{F(5)-F(4)}$ 21 Hz, $J_{F(5)-F(2)}$ 9 Hz F-5,5´), -55.4 (d, 6F, $J_{CF_3-F(5)}$ 25 Hz, CF_3(6,6´)). $^{1}HNMR$ ($CDCl_3$), δ: 1.42 (t, 6H, CH_3), 4.40 (q, 4H, OCH_2), 6.58 (m, 2H, H-4,6), 6.61 (m, 1H, H-2), 7.22 (t, 1H, H-5). NMR ^{1}H ($(CD_3)_2CO$), δ: 1.40 (t, 6H, CH_3), 4.44 (q, 4H, CH_2), 6.75 (m, 1H, H-2), 6.77 (m, 2H, H-4,6), 7.33 (t, 1H, H-5).

*Synthesis of oxacalix[6]azacryptand **2***

*Tris(2-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenoxy)ethyl)amine **1***. To *tert*-butyl alcohol (15 mL), finely ground NaOH (0.30 g, 7.5 mmol) and triethanolamine (0.30 g, 2 mmol) were added sequentially. The mixture was stirred for 0.5 h at 80 °C, octafluorotoluene (2.12 g, 9 mmol) was added and this was stirred further for 14 h. Solvent was evaporated. Silica gel column chromatography (eluent $CCl_4/CHCl_3$ 1:1) gave 1.25 g (78%) of tris(2-aryloxyethyl)amine **1** as a viscous material. IR (film) ν, cm^{-1}: w 2958, w 2896, w 2846 (C-H); s 1656, m 1614, vs 1504 (Ar_F); vs 1340, vs 1232 (Ar-O); s 1186-995 (group, C-F). $^{19}FNMR$ (CCl_4+$CDCl_3$), δ: -157.3 (m, 6F, F-2,6), -142.6 (m, 6F, F-3,5), -57.0 (t, 9F, *J* 21.6 Hz, CF_3). $^{1}HNMR$ (CCl_4+$CDCl_3$), δ: 3.17 (t, 6H, NCH_2), 4.42 (t, 6H, OCH_2). Found, %: C 40.53; H 1.58; F 50.11; N 1.78. M 794. $C_{27}H_{12}F_{21}NO_3$. Calculated, %: C 40.67; H 1.52; F 50.04; N 1.76. M 797.

*2,13,14,24,25,36,42,43-Octafluoro-12,26,40-tris(trifluoromethyl)-4,10,16,22,28,34,37-heptaoxa-19-azaheptacyclo[17.17.3.1^{3,35}.1^{5,9}.1^{11,15}.1^{23,27}.1^{29,33}]tetratetraconta-1,3(40),5(44),6,8,11(43),12,14,23(42),24,26,29(41),30,32,35-pentadecaene **3***. Potassium carbonate (2.10 g, 15 mmol) and 18-crown-6 ether (0.31 g, 1 mmol) were added under stirring to a solution of tris(aryloxy)ethylamine **1** (1.09 g, 1.3 mmol) and resorcinol (0.61 g, 5.5 mmol) in 300 mL acetonitrile. The mixture was stirred at 82 °C for 30 h, filtered and the solvent evaporated. Silica gel column chromatography (eluent CCl_4, then $CHCl_3$) gave 1.09 g of a product mixture containing (^{19}F NMR, ^{1}H) 17% oxacalix[6]azacryptand **2** and 83% bicycle **3**. $^{19}FNMR$ ($CDCl_3$), δ: -156.5 (d, 2F, *J* 20 Hz, F-14,24), -144.6 (m, 2F, F-42,43), -143.2 (s, 2F, F-2,36), -141.4 (m, 2F, F-13,25), -56.8 (d, 6F, *J* 27 Hz, CF_3-12,26), -56.6 (s, 3F, CF_3-40). $^{1}HNMR$

($CDCl_3$), δ: 3.09, 3.13 (m, 6H, NCH_2-18,20,39); 4.28, 4.33 (m, 6H, OCH_2-17,21,38); 6.12 (m, 2H, H-41,44); 6.92-7.00 (m, 4H, H-6,8,30,32); 7.35 (t, 2H, H-7,31).

2,12,14,24,26,36-Hexafluoro-47,49,51-tris(trifluoromethyl)-4,10,16,22,28,34,37,43,46-nonaoxa-40-azanonacyclo[23.11.7.3^{13,40}.1^{3,35}.1^{5,9}.1^{11,15}.1^{17,21}.1^{23,27}.1^{29,33}]dopentaconta-1,3(47),5(52),6,8,11(51),12,14,17(50),18,20,23(49),24,26,29(48),30,35-octadecaene ***2.***

a) Potassium carbonate (3.01 g, 22 mmol) and tetrabutylammonium chloride monohydrate (0.42 g, 1.5 mmol) were added under stirring to a solution of tris(aryloxy)ethylamine **1** (0.66 g, 0.8 mmol) and resorcinol (0.79 g, 7.2 mmol) in 200 mL acetonitrile. The mixture was stirred at 82 ^oC for 30 h, filtered and the solvent evaporated. Silica gel column chromatography (eluent CCl_4, then $CHCl_3$) gave 0.41 g (51%, 0.41mmol) of pure oxacalix[6]azacryptand **2** (^{19}F NMR, ^1H). White powder. Mp not determined, gradual softening. IR (KBr) ν, cm^{-1}: w 2964, w 2939, w 2883, w 2844 (C-H); m 1641, m 1614, m 1598, vs 1481 (Ar, Ar_F); s 1330, vs 1303, s 1249, s 1222 (Ar-O); s 1160-995 (group, C-F). $^{19}FNMR$ ($CDCl_3$), δ: -143.1 (s, 6F, F-2,12,14,24,26,36); -56.6 (s, 9F, CF_3). ^1HNMR ($CDCl_3$), δ: 3.21 (m, 6H, NCH_2-39,41,44); 4.48 (m, 6H, OCH_2-38,42,45); 6.46 (m, 3H, H-48,50,52); 6.94 (dd, 6H, *J* 8.2 Hz, *J* 2.3 Hz, H-6,8,18,20,30,32); 7.36 (t, 3H, *J* 8.2 Hz, H-7,19,31). NMR ^1H (acetone-d_6), δ: 3.31 (m, 6H, NCH_2-39,41,44); 4.59 (m, 6H, OCH_2-38,42,45); 6.56 (m, 3H, H-48,50,52); 7.01 (dd, 6H, *J* 8.2 Hz, *J* 2.3 Hz, H-6,8,18,20,30,32); 7.48 (t, 3H, *J* 8.2 Hz, H-7,19,31). $^{13}CNMR$ ($CDCl_3$), δ: 62.21(s, N<u>C</u>H_2); 76.07 (s, O<u>C</u>H_2); 98.97 (s, C-48,50,52); 109.08 (q, $J_{C(47)-F(CF3)}$ 31.6 Hz, C-47,49,51); 112.27 (s, C-6,8,18,20,30,32); 122.06 (q, J_{C-F} 275 Hz, CF_3); 130.57 (s, C-7,19,31); 137.54 (d, $J_{C(3)-F}$ 10 Hz, C-3,11,15,23,27,35); 137.54 (t, $J_{C(1)-F}$ 8 Hz, C-1,13,25); 142.48 (d, $J_{C(2)-F}$ 251 Hz, C-2,12,14,24,26,36); 156.47 (s, C-5,9,12,21,29,33). Found, %: C 53.65; H 2.50; N 1.32; F 28.08. $C_{45}H_{24}F_{15}NO_9$. Calculated, %: C 53.64; H 2.40; N 1.39; F 28.28. M 1007.

b) Potassium carbonate (1.40 g, 10 mmol) and 18-crown-6 ether (0.10 g, 0.33 mmol) were added under stirring to a solution of resorcinol (0.16 g, 1.5 mmol) and mixture (0.80 g), containing 10% of oxacalix[6]azacryptand **2** and 90% of bicycle **3,** in 200 mL acetonitrile. The mixture was stirred at 82 ^oC for 25 h, filtered and the solvent evaporated. Silicagel column chromatography (eluent CCl_4, then $CHCl_3$) gave 0.63 g of oxacalix[6]azacryptand **2** (^{19}F NMR, ^1H).

Triethyl 2,12,14,24,26,36-hexafluoro-47,49,51-tris(trifluoromethyl)-4,10,16,22,28,34,37,43,46-nonaoxa-40-azanonacyclo[23.11.7.3^{13,40}.1^{3,35}.1^{5,9}.1^{11,15}.1^{17,21}.1^{23,27}.1^{29,33}]dopentaconta-1,3(47),5(52),6,8,11(51),12,14,17(50),18,20,23(49),24,26,29(48),30,32,35-octadecaene-7,19,31-tricarboxylate ***6.*** Potassium carbonate (2.10 g, 15 mmol) and tetrabutylammonium chloride monohydrate (0.30 g, 1 mmol) were added under stirring to a solution of tris(aryloxy)ethylamine **1** (1.24 g, 1.5 mmol) and ethyl 3,5-dihydroxybenzoate (1.09 g, 6 mmol) in 300 mL acetonitrile. The mixture was stirred at 82 ^oC for 24 h, then potassium carbonate (1.38 g, 10 mmol) and ethyl 3,5-

dihydroxybenzoate (0.72 g, 4 mmol) were added and stirred at 82 ºC for another 30 h. The mixture was filtered and the solvent evaporated. Silicagel column chromatography (eluent CCl_4, then $CHCl_3$) gave 0.65 g (36%, 0.53 mmol) of pure oxacalix[6]azacryptand **6** (^{19}F NMR, ^{1}H). White powder. Mp not determined, gradual softening. ^{19}FNMR ($CDCl_3$), δ: -142.7 (s, 6F, F-2,12,14,24,26,36); -56.5 (s, 9F, CF_3). ^{1}HNMR ($CDCl_3$), δ: 1.40 (t, 9H, $OCH_2C\underline{H}_3$); 3.22 (m, 6H, $NC\underline{H}_2$); 4.40 (q, 6H, $OC\underline{H}_2CH_3$); 4.48 (m, 6H, OCH_2); 6.59 (m, 3H, H-48,50,52); 7.62 (d, 6H, *J* 2.3 Hz, H-6,8,18,20,30,32). Found, %: C 53.45; H 3.29; N 1.17; F 23.27. $C_{54}H_{36}F_{15}NO_{15}$. Calculated, %: C 53.00; H 2.97; N 1.14; F 23.29. M 1223.

*NMR data for complex **2·8***

*Pure propargyl alcohol **8**:* ^{1}HNMR (C_6D_6), δ: 0.66 (t, OH); 1.98 (t, 1H, H-C≡); 3.71 (d, 2H, CH_2). ^{13}CNMR (C_6D_6), δ: 50.94 (s, $\underline{C}H_2$); 73.76 (s, ≡$\underline{C}$-H); 83.01 (s, ≡$\underline{C}$-CH_2).

*Pure oxacalix[6]azacryptand **2**:* ^{1}HNMR (C_6D_6), δ: 2.46 (m, 6H, NCH_2-39,41,44); 4.12 (m, 6H, OCH_2-38,42,45); 6.59 (m, 3H, H-48,50,52); 6.85 (m, 6H, H-6,8,18,20,30,32); 6.88 (m, 3H, H-7,19,31). ^{13}CNMR (C_6D_6), δ: 62.09 (s, N$\underline{C}H_2$); 76.40 (s, O$\underline{C}H_2$); 99.54 (s, C-48,50,52); 109.84 (q, C-47,49,51); 113.23 (s, C-6,8,18,20,30,32); 123.28 (q, CF_3); 131.62 (s, C-7,19,31); 138.43 (d, C-3,11,15,23,27,35); 141.26 (t, C-1,13,25); 143.25 (d, C-2,12,14,24,26,36); 157.60 (s, C-5,9,12,21,29,33). NMR ^{19}F (C_6D_6), δ: -142.38 (s, 6F, F-2,12,14,24,26,36); -55.61 (s, 9F, CF_3).

*Mixture of **8** and **2** (**8:2** = 1:2.2):* ^{1}HNMR (C_6D_6) of **8**, δ: 1.96 (t, 1H, H-C≡); 3.48 (d, 2H, CH_2). NMR ^{13}C (C_6D_6) of **8**, δ: 50.41 (s, $\underline{C}H_2$); 72.25 (s, ≡$\underline{C}$-H); 83.69 (s, ≡$\underline{C}$-CH_2).

^{1}HNMR (C_6D_6) of **2**, δ: 2.42 (m, 6H, NCH_2-39,41,44); 4.10 (m, 6H, OCH_2-38,42,45); 6.65 (m, 3H, H-48,50,52). ^{13}CNMR (C_6D_6) of **2**, δ: 62.53 (s, N$\underline{C}H_2$); 76.24 (s, O$\underline{C}H_2$); 99.64 (s, C-48,50,52); 109.92 (q, C-47,49,51); 113.17 (s, C-6,8,18,20,30,32); 123.38 (q, CF_3); 131.57 (s, C-7,19,31); 138.43 (d, C-3,11,15,23,27,35); 141.24 (t, C-1,13,25); 143.36 (d, C-2,12,14,24,26,36); 157.72 (s, C-5,9,12,21,29,33). ^{19}FNMR (C_6D_6) of **2**, δ: -142.80 (s, 6F, F-2,12,14,24,26,36); -55.65 (s, 9F, CF_3).

*Mixture of **8** and **2** (**8:2** = 1:1.2):* ^{1}HNMR (C_6D_6) of **8**, δ: 1.97 (t, 1H, H-C≡); 3.50 (d, 2H, CH_2). ^{13}CNMR (C_6D_6) of **8**, δ: 50.47 (s, $\underline{C}H_2$); 72.44 (s, ≡$\underline{C}$-H); 83.62 (s, ≡$\underline{C}$-CH_2).

^{1}HNMR (C_6D_6) of **2**, δ: 2.40 (m, 6H, NCH_2-39,41,44); 4.08 (m, 6H, OCH_2-38,42,45); 6.70 (m, 3H, H-48,50,52). ^{13}CNMR (C_6D_6) of **2**, δ: 62.83 (s, N$\underline{C}H_2$); 76.15 (s, O$\underline{C}H_2$); 99.72 (s, C-48,50,52); 110.01 (q, C-47,49,51); 113.13 (s, C-6,8,18,20,30,32); 123.44 (q, CF_3); 131.53 (s, C-7,19,31); 138.45 (d, C-3,11,15,23,27,35); 141.27 (t, C-1,13,25); 143.46 (d, C-2,12,14,24,26,36); 157.82 (s, C-5,9,12,21,29,33). ^{19}FNMR (C_6D_6) of **2**, δ: -143.11 (s, 6F, F-2,12,14,24,26,36); -55.70 (s, 9F, CF_3).

*Mixture of **8** and **2** (**8:2** = 1:0.7):* ^{1}HNMR (C_6D_6) of **8**, δ: 1.97 (t, 1H, H-C≡); 3.55 (d, 2H, CH_2). ^{13}CNMR (C_6D_6) of **8**, δ: 50.57 (s, $\underline{C}H_2$); 72.70 (s, ≡$\underline{C}$-H); 83.50 (s, ≡$\underline{C}$-CH_2).

^{1}HNMR (C_6D_6) of **2**, δ: 2.38 (m, 6H, NCH_2-39,41,44); 4.07 (m, 6H, OCH_2-38,42,45); 6.73 (m, 3H, H-48,50,52). ^{13}CNMR (C_6D_6) of **2**, δ: 63.05 (s, $N\underline{C}H_2$); 76.06 (s, $O\underline{C}H_2$); 99.78 (s, C-48,50,52); 110.07 (q, C-47,49,51); 113.10 (s, C-6,8,18,20,30,32); 123.49 (q, CF_3); 131.50 (s, C-7,19,31); 138.45 (d, C-3,11,15,23,27,35); 141.29 (t, C-1,13,25); 143.52 (d, C-2,12,14,24,26,36); 157.89 (s, C-5,9,12,21,29,33). ^{19}FNMR (C_6D_6) of **2**, δ: -143.32 (s, 6F, F-2,12,14,24,26,36); -55.73 (s, 9F, CF_3).

References

S1. L. S. Kobrina, G. G. Furin, G. G. Yakobson, *Zh. Org. Khim.*, 1970, **6**, 512 (in Russian).

Spectral data of obtained compounds

Figure S1. ^{19}F NMR spectrum ($CDCl_3$) of 1,3-bis(3-ethoxy-2,4,5-trifluoro-6-(trifluoromethyl)phenoxy)benzene **7.**

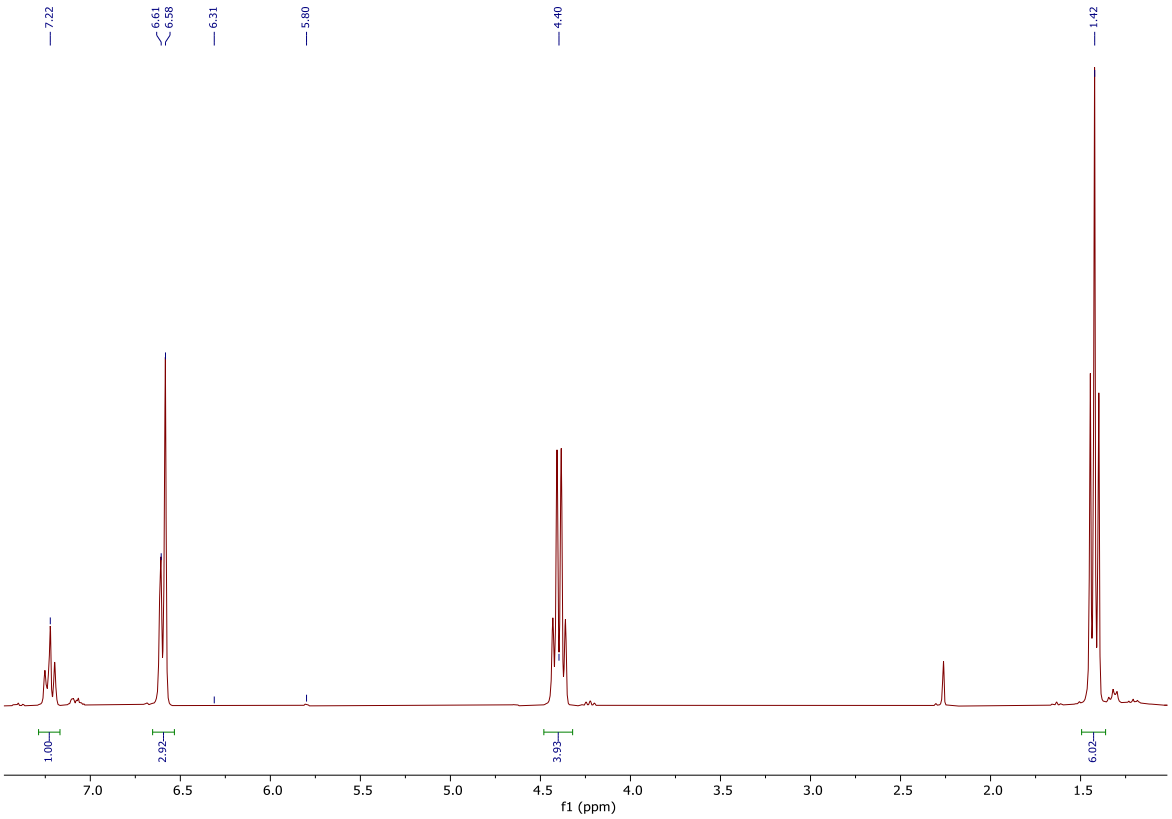

Figure S2. ^{1}H NMR spectrum ($CDCl_3$) of 1,3-bis(3-ethoxy-2,4,5-trifluoro-6-(trifluoromethyl)phenoxy)benzene **7.**

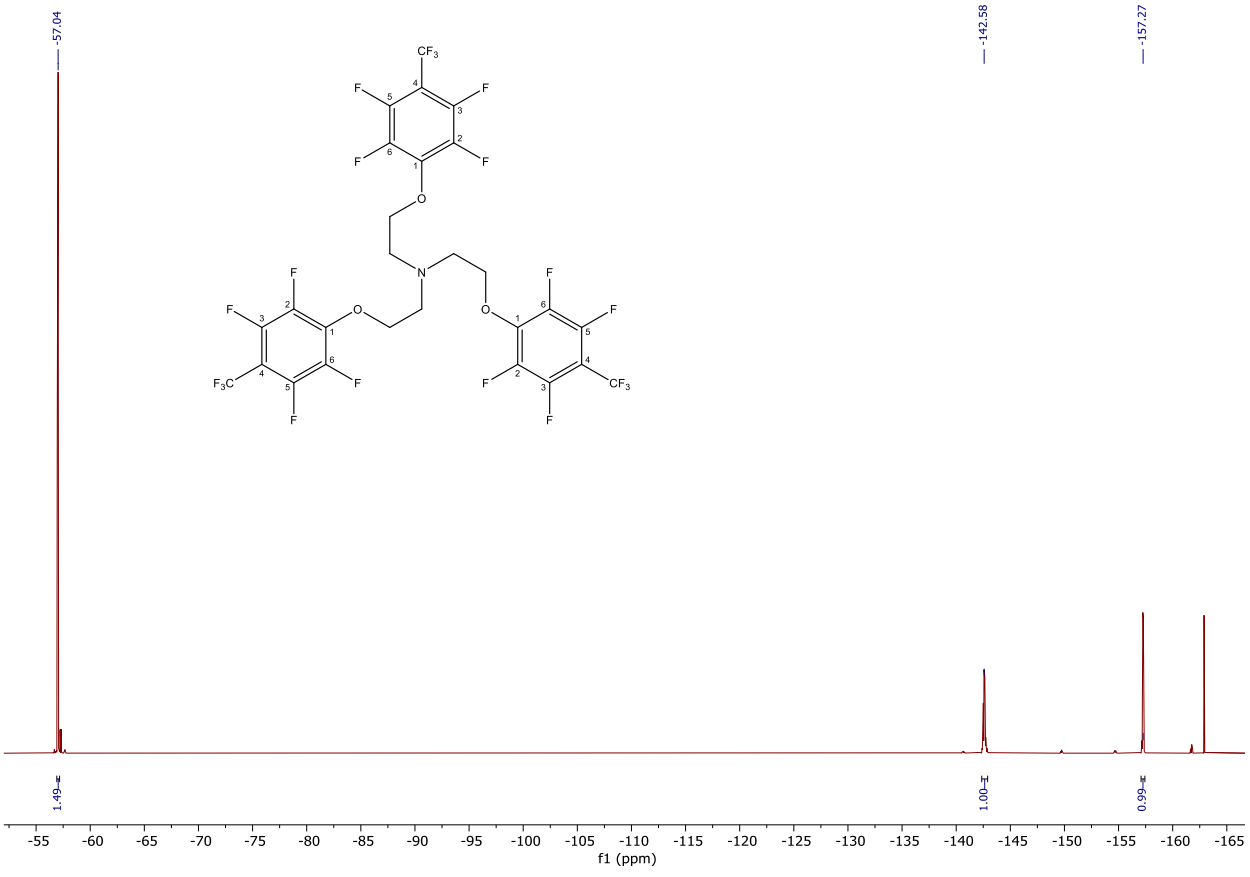

Figure S3. ^{19}F NMR spectrum (CCl_4 +$CDCl_3$) of tris(2-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenoxy)ethyl)amine **1**.

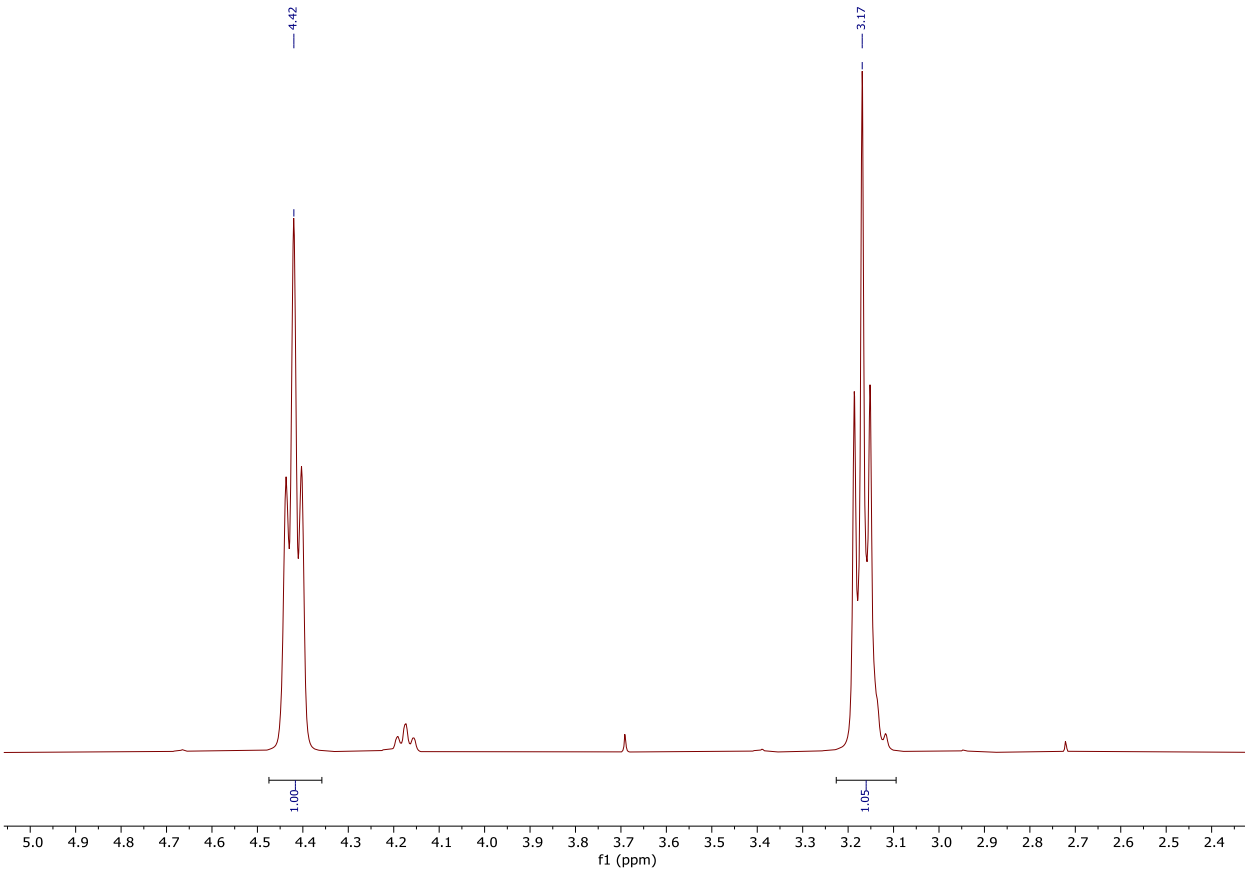

Figure S4. ^{1}H NMR spectrum (CCl_4 +$CDCl_3$) of tris(2-(2,3,5,6-tetrafluoro-4-(trifluoromethyl)phenoxy)ethyl)amine **1**.

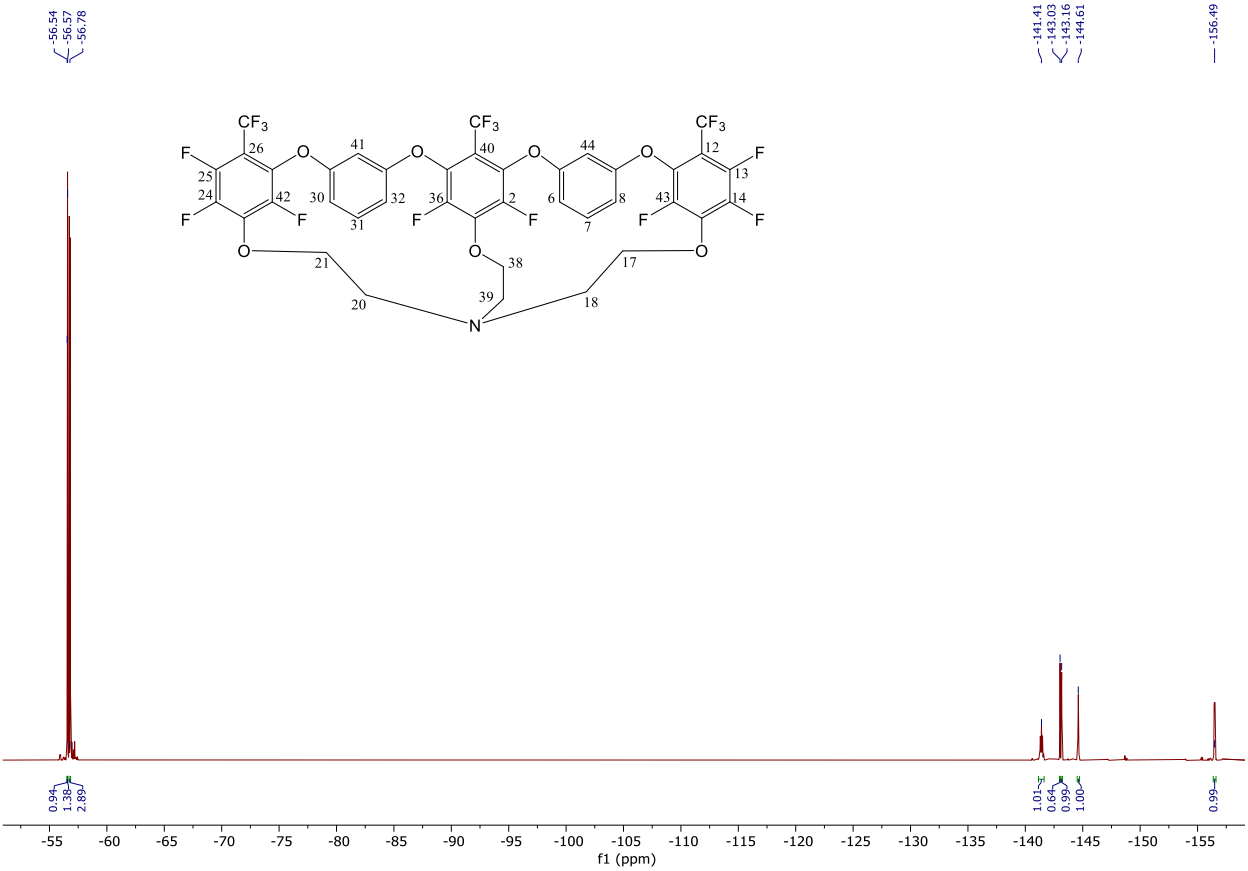

Figure S5. ^{19}F NMR spectrum ($CDCl_3$) of mixture bicycle **3** and oxacalix[6]azacryptand **2** **(3:2** = 83:17)

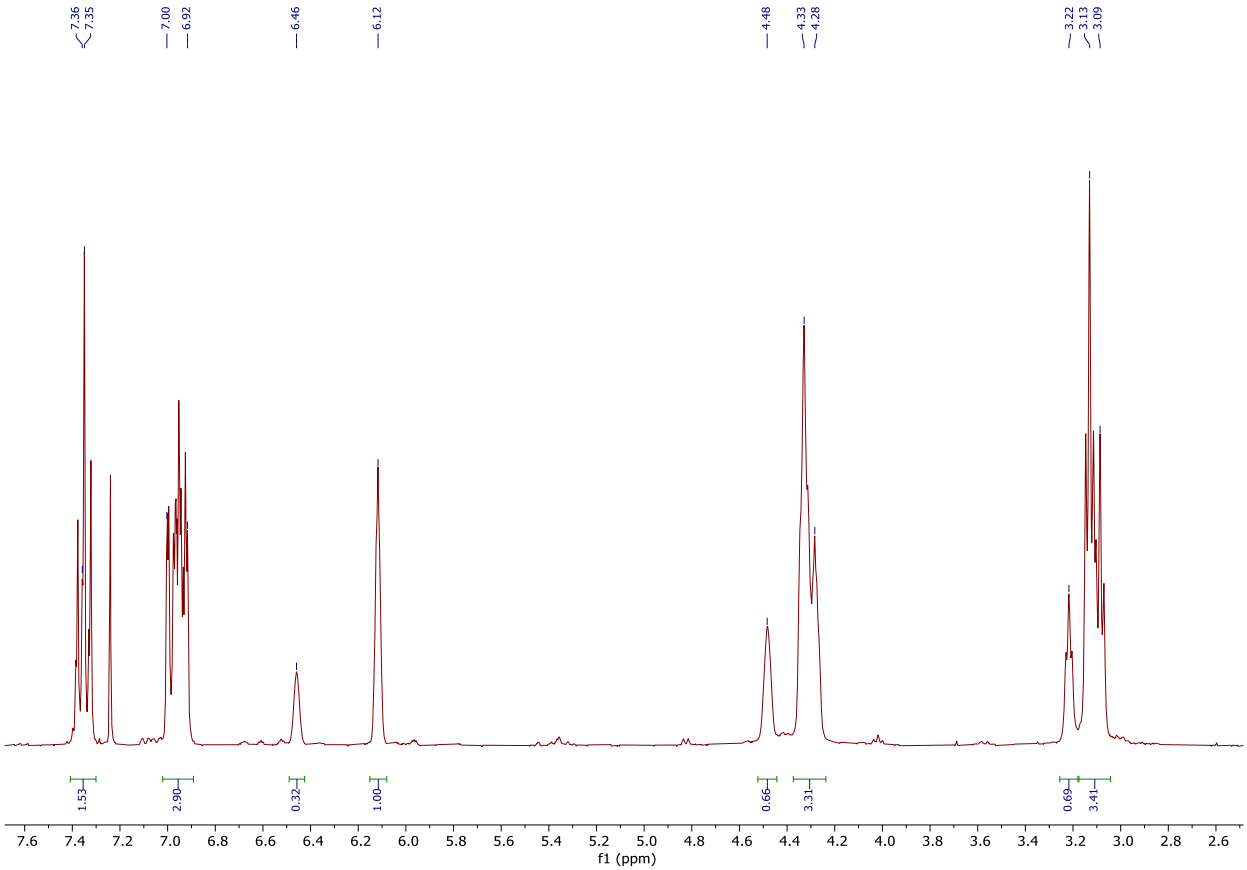

Figure S6. ^{1}H NMR spectrum ($CDCl_3$) of mixture bicycle **3** and oxacalix[6]azacryptand **2** **(3:2** = 83:17)

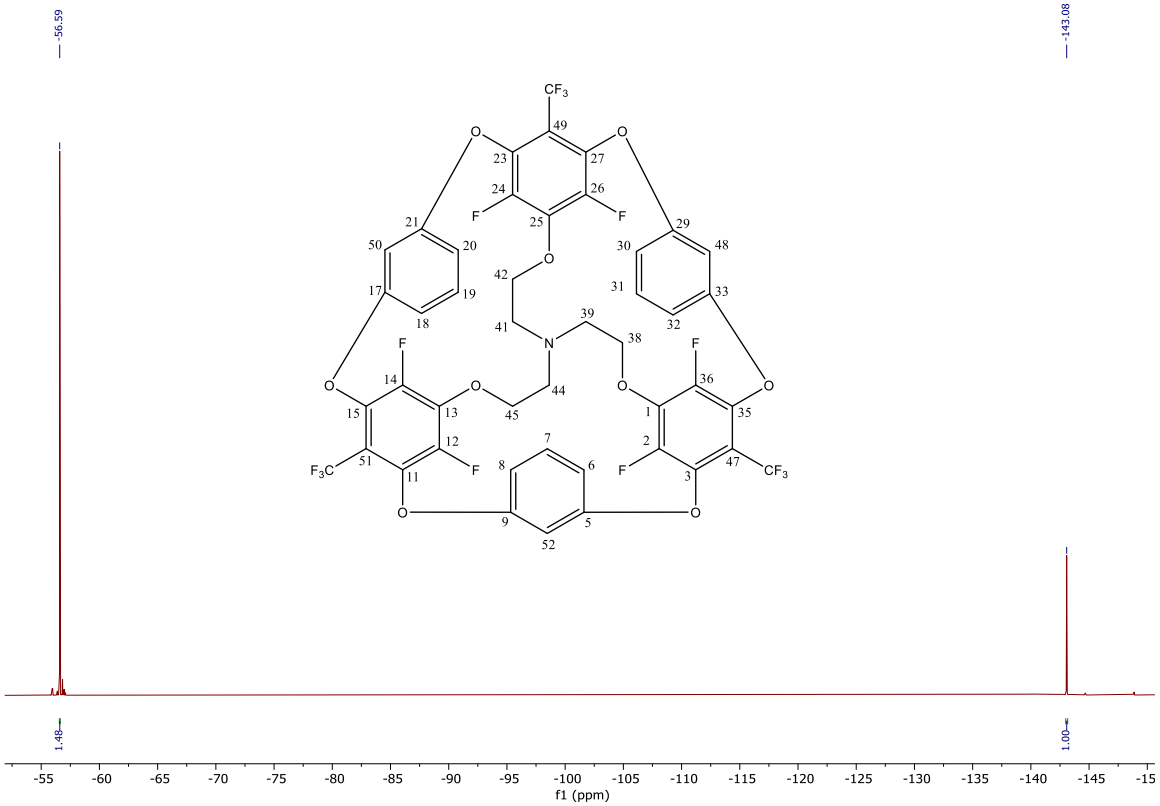

Figure S7. ^{19}F NMR spectrum ($CDCl_3$) of oxacalix[6]azacryptand **2**

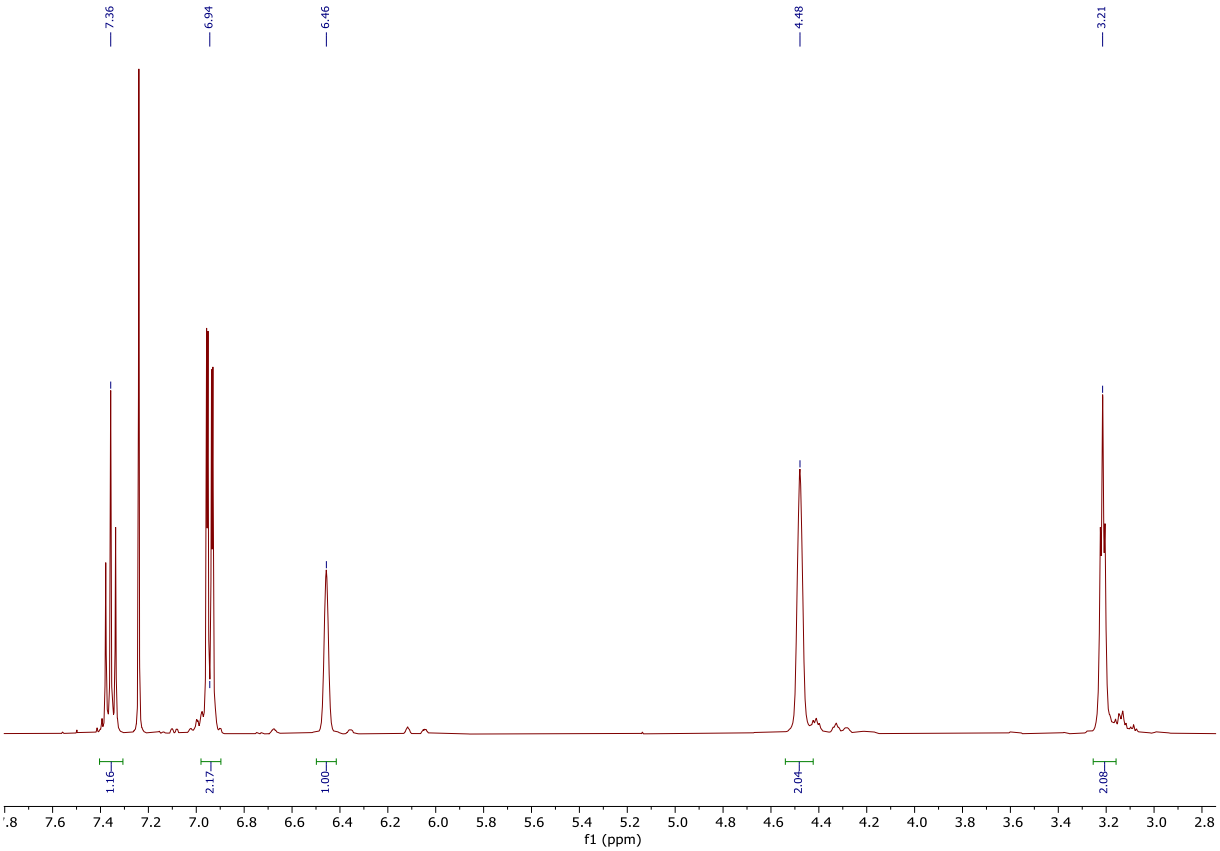

Figure S8. ^{1}H NMR spectrum ($CDCl_3$) of oxacalix[6]azacryptand **2**

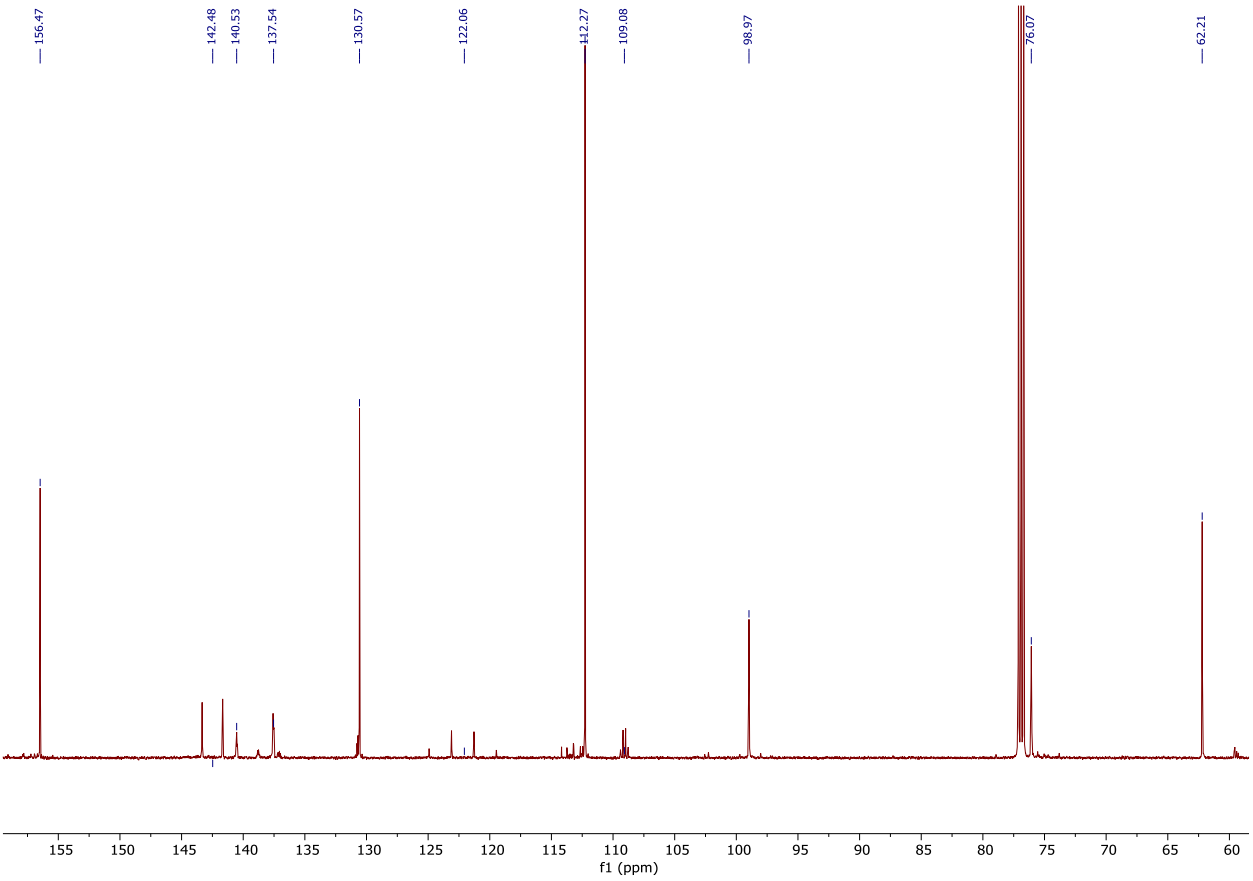

Figure S9. ^{13}C NMR spectrum ($CDCl_3$) of oxacalix[6]azacryptand **2**

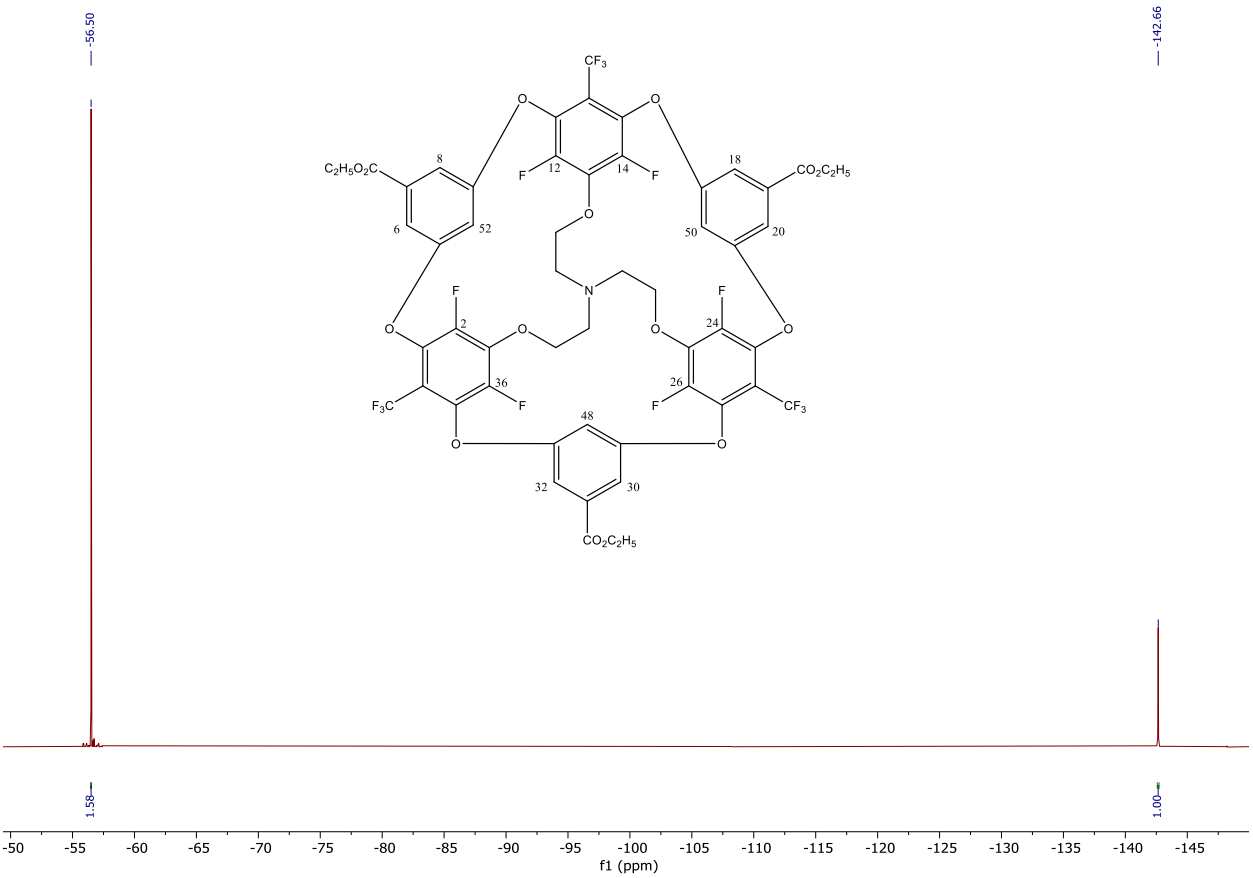

Figure S10. ^{19}F NMR spectrum ($CDCl_3$) of oxacalix[6]azacryptand **6**

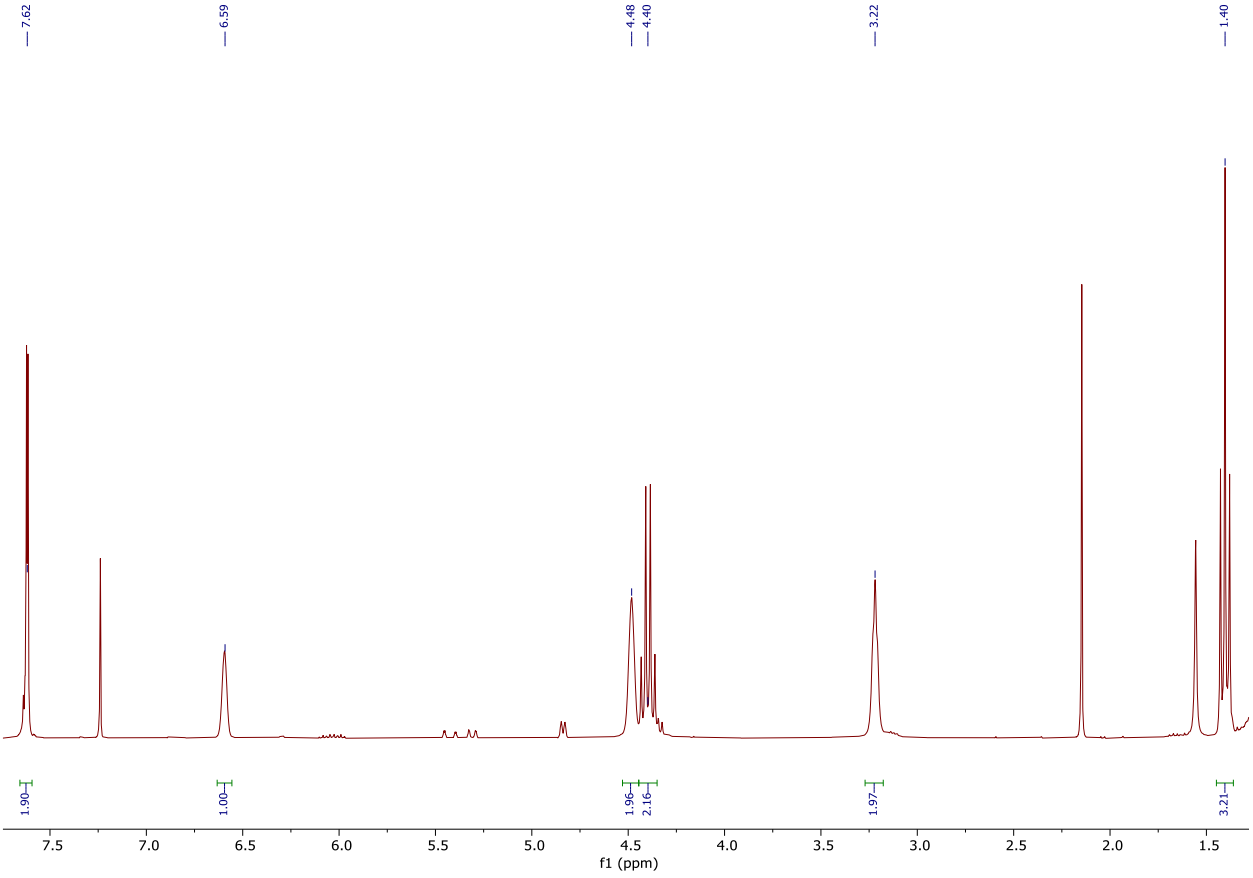

Figure S11. ^{1}H NMR spectrum ($CDCl_3$) of oxacalix[6]azacryptand **6**

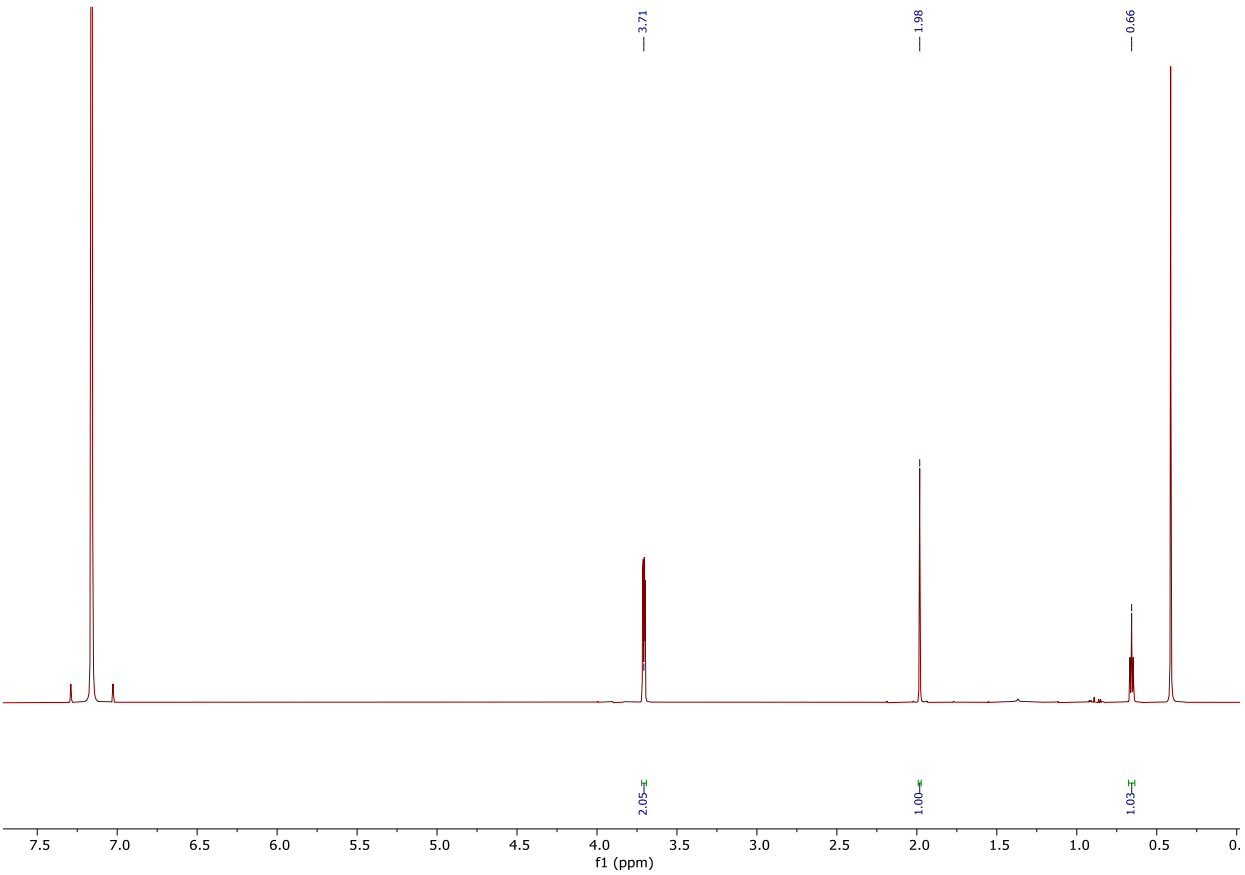

Figure S12. ^{1}H NMR spectrum (C_6D_6) of propargyl alcohol **8**

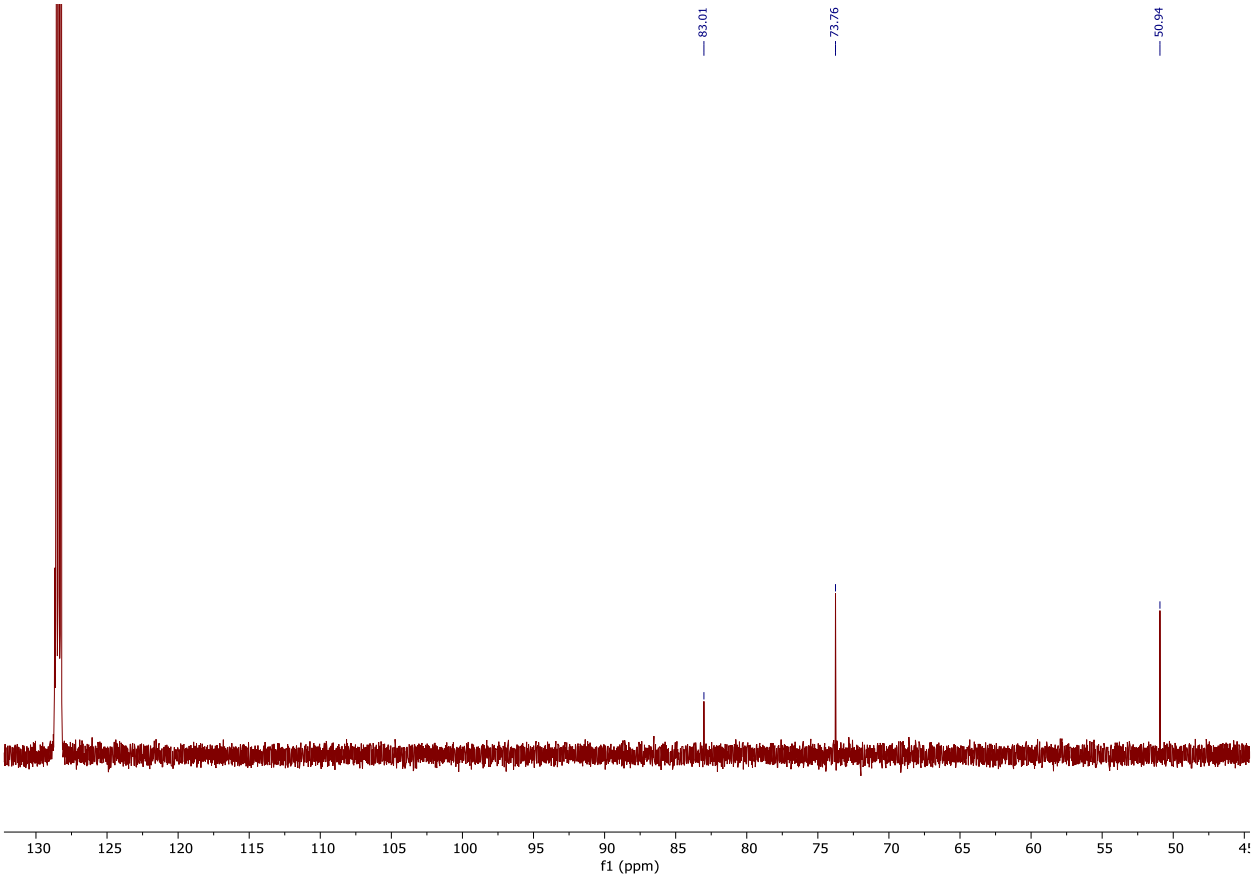

Figure S13. ^{13}C NMR spectrum (C_6D_6) of propargyl alcohol **8**

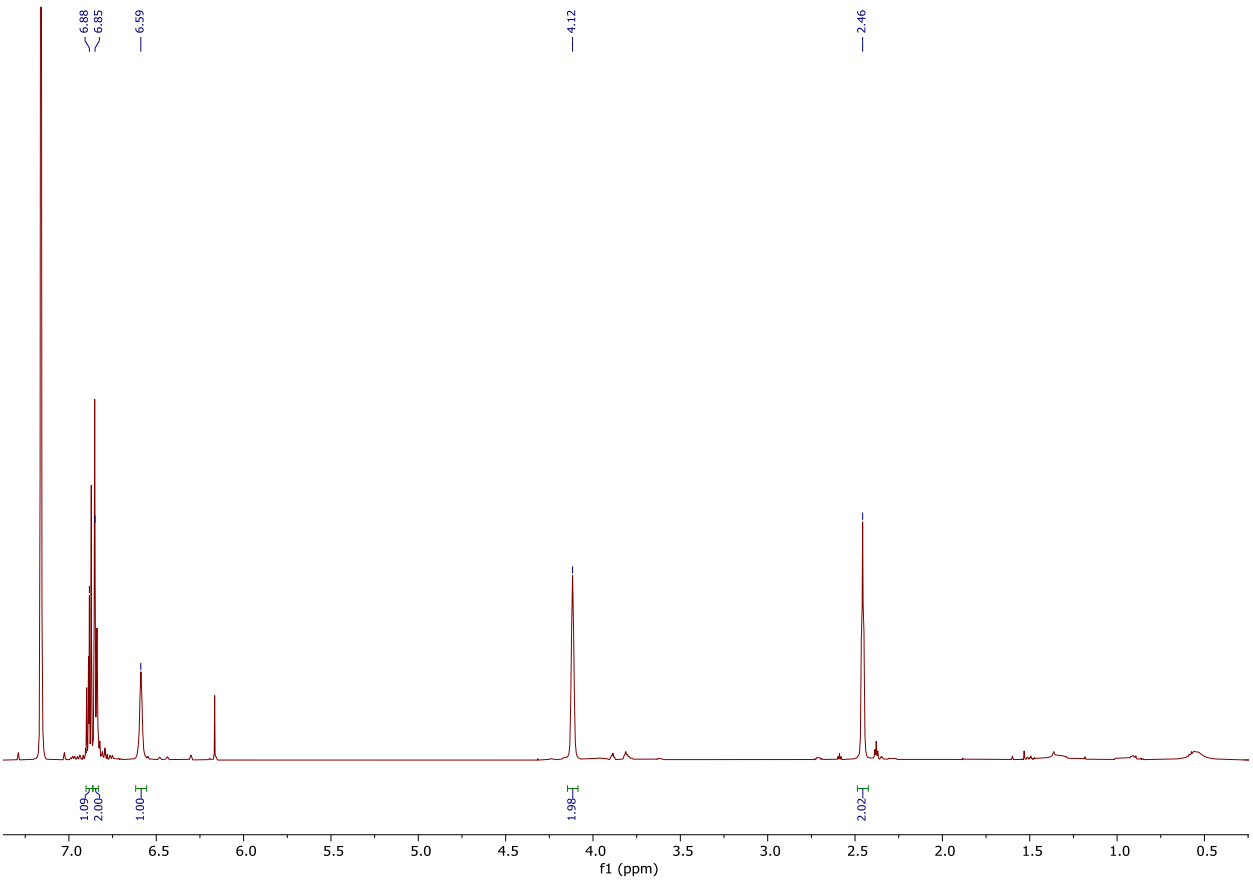

Figure S14. ^{1}H NMR spectrum (C_6D_6) of oxacalix[6]azacryptand **2**

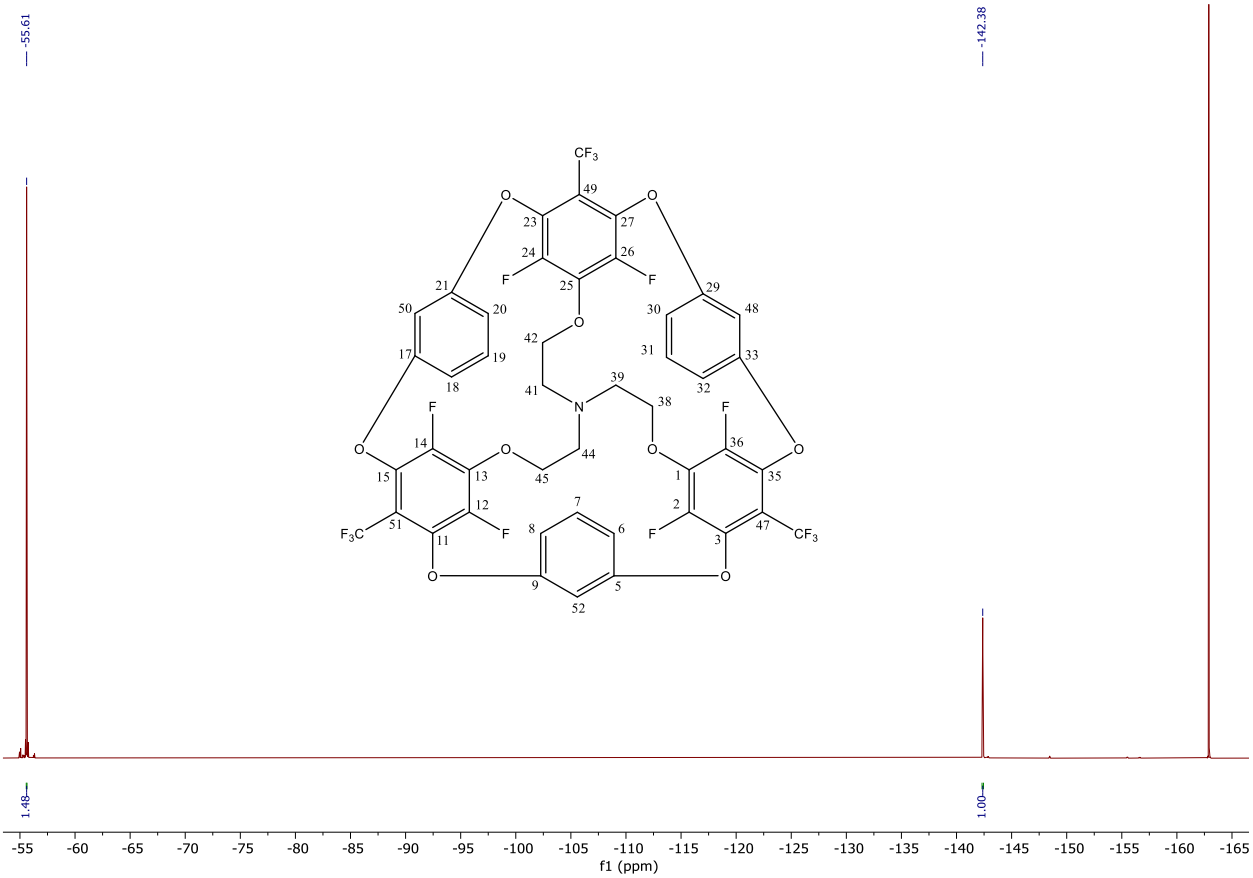

Figure S15. ^{19}F NMR spectrum (C_6D_6) of oxacalix[6]azacryptand **2**

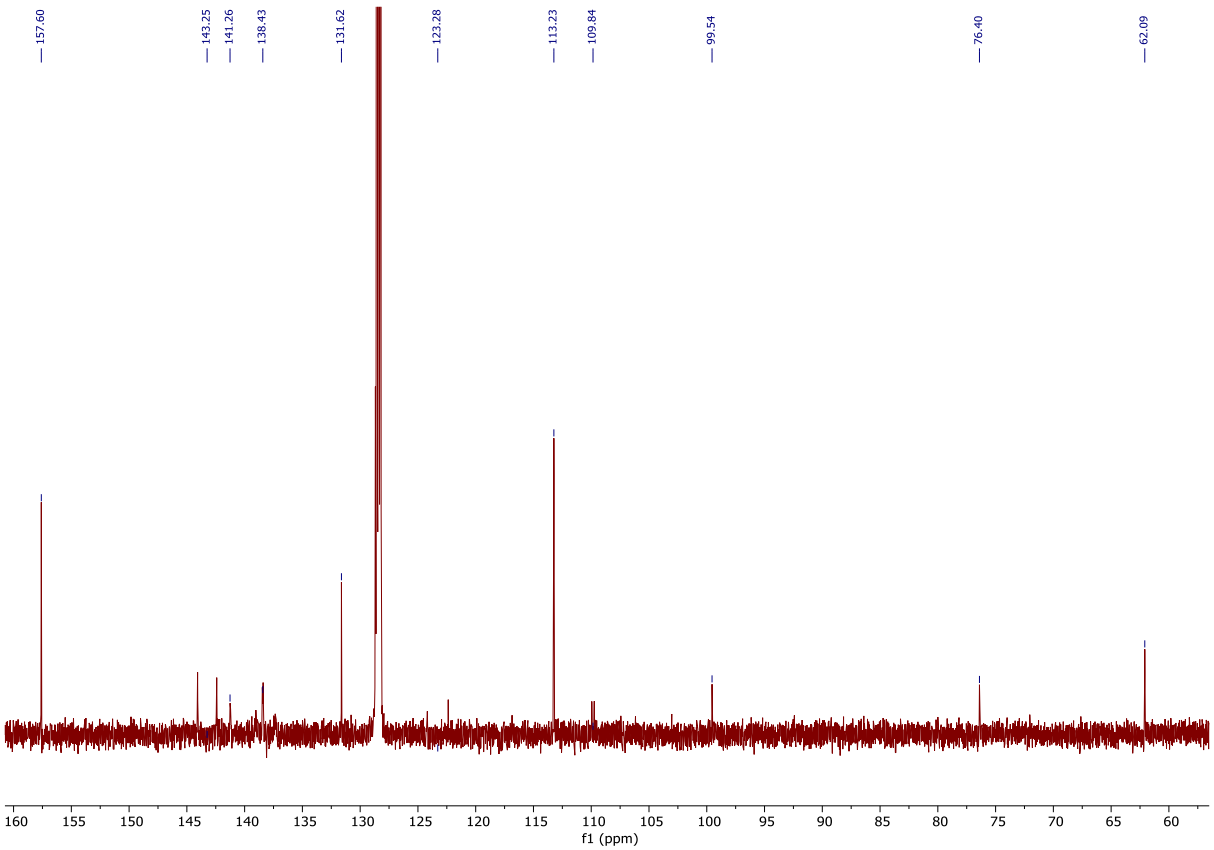

Figure S16. ^{13}C NMR spectrum (C_6D_6) of oxacalix[6]azacryptand **2**

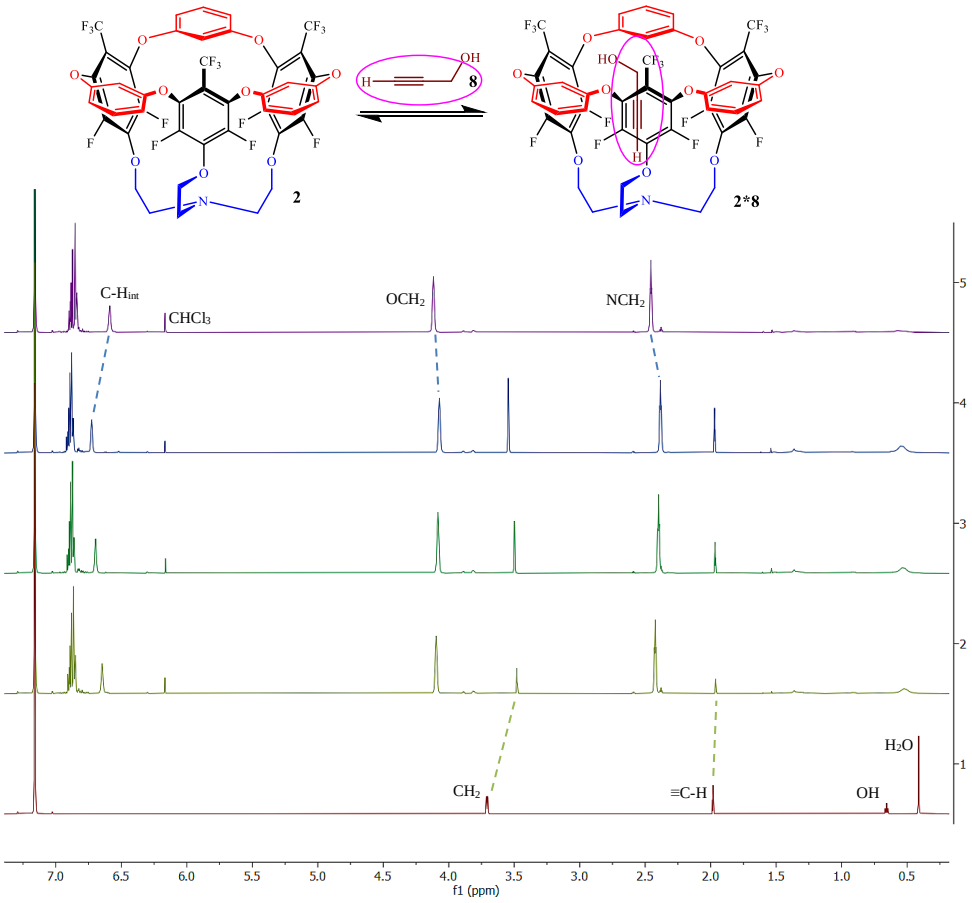

Figure S17. ^1H NMR spectra (benzene d_6): 1. propargyl alcohol **8**; 2-4. mixture of propargyl alcohol **8** and oxacalix[6]azacryptand **2** (2. **8:2** = 1:2.2, 3. **8:2** = 1.2, 4. **8:2** = 1:0.7); 5. oxacalix[6]azacryptand **2**.

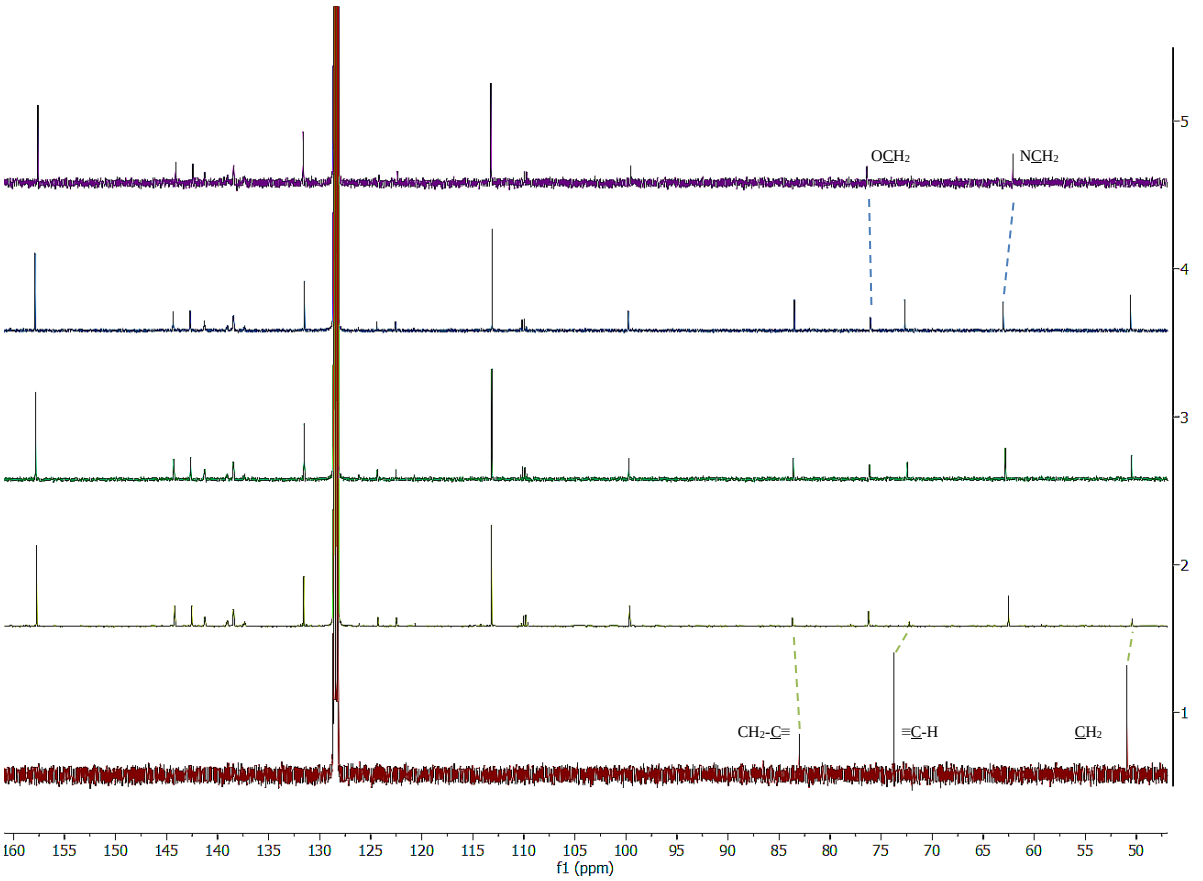

Figure S18. ^{13}C NMR spectra (benzene d_6): 1. propargyl alcohol **8**; 2-4. mixture of propargyl alcohol **8** and oxacalix[6]azacryptand **2** (2. **8:2** = 1:2.2, 3. **8:2** = 1.2, 4. **8:2** = 1:0.7); 5. oxacalix[6]azacryptand **2**.

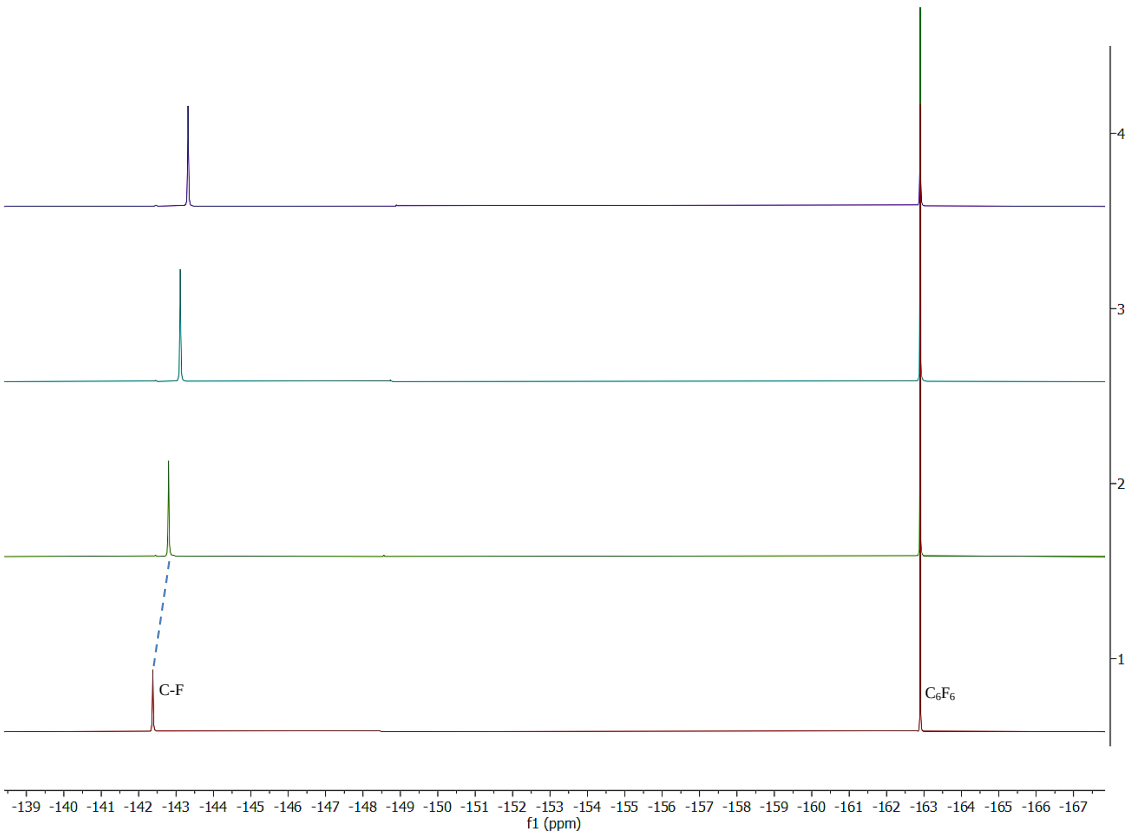

Figure S19. ^{19}F NMR spectra (benzene d_6): 1. oxacalix[6]azacryptand **2**; 2-4. mixture of propargyl alcohol **8** and oxacalix[6]azacryptand **2** (2. **8:2** = 1:2.2, 3. **8:2** = 1.2, 4. **8:2** = 1:0.7).