Supporting Informations

Synthesis of benzo[6]urils and their selective interactions with bipyridines

Man Chen, Hai-Ling Li, Hang Cong*, Yan Yan, Kai-Li Li, Ming-Qiong Li, Zhu Tao
Key Laboratory of Macrocyclic and Supramolecular Chemistry of Guizhou Province, Guizhou University, Guiyang 550025, China

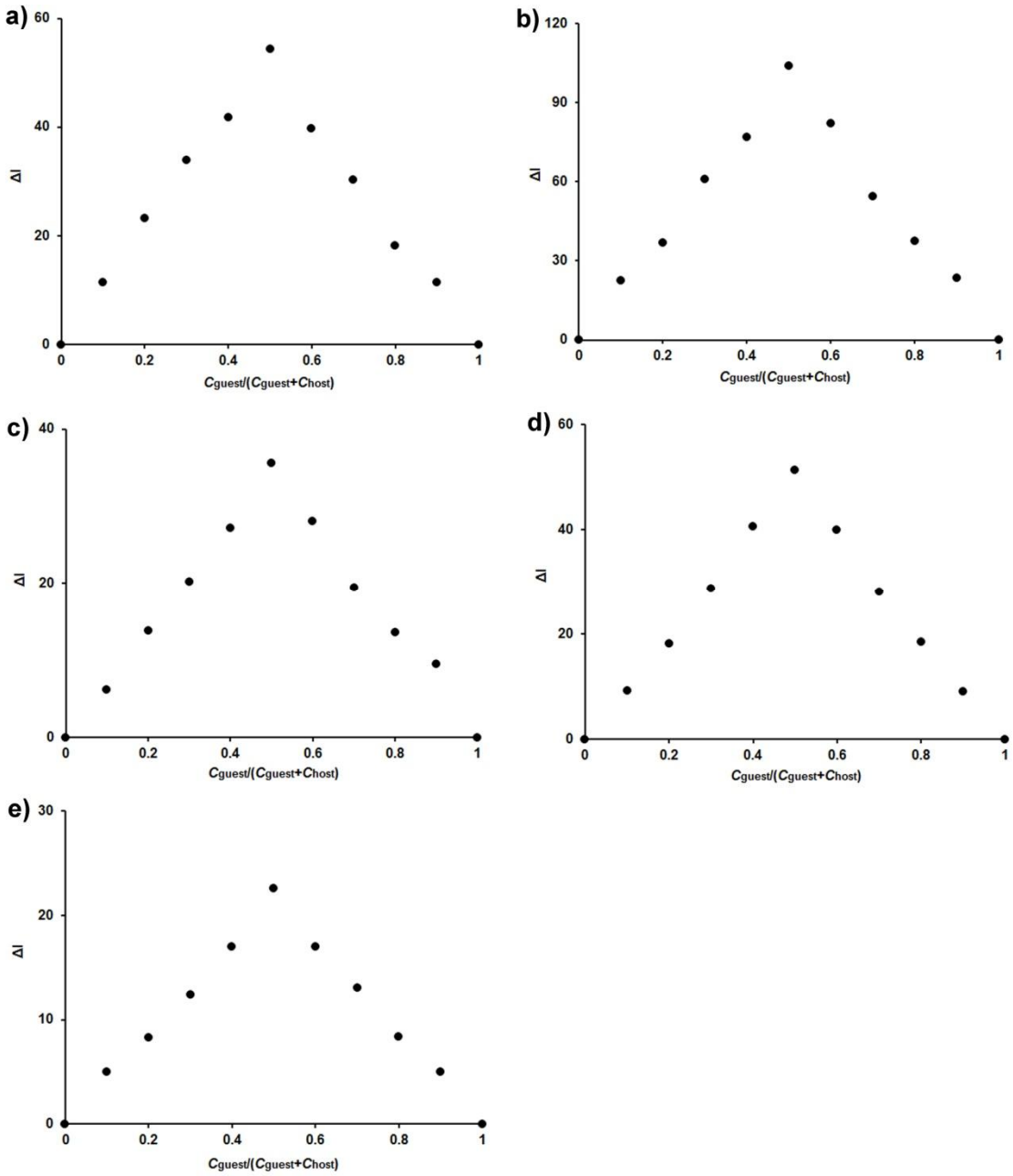

Figure S1 Job's plots of the interaction between benzo[6]uril **1** and a) guest **3**; b) guest **4**; c) guest **5**; d) guest **6**; e) guest **8**. The total concentration was maintained at 1.0×10^{-5} mol·L $^{-1}$, and fluorescence intensities were recorded at λem = 326 nm, λex = 300 nm.

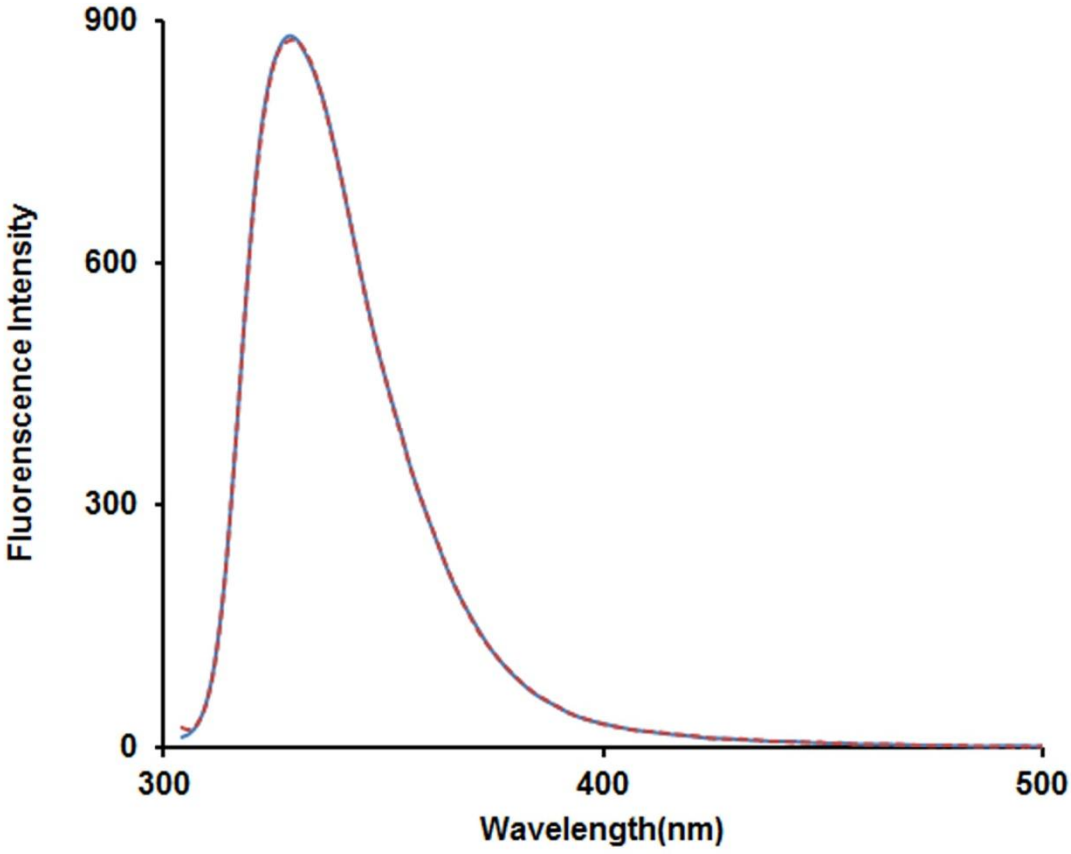

Figure S2 Fluorescence spectra of benzo[6]uril **1** (1.0×10^{-5} mol·L^{-1} in DMSO) in the absence (blue solid) and presence of 1 eq. guest **7** (red dash).

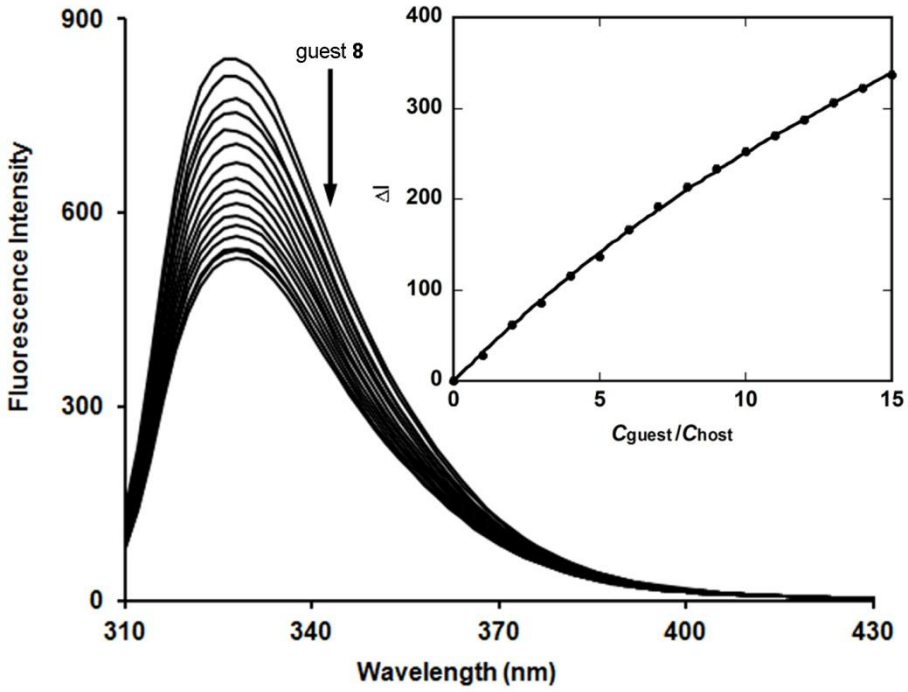

Figure S3 Fluorescence titration spectra of benzo[6]uril **1** with guest **8**. The host concentration was fixed at 1.0×10^{-5} mol·L^{-1} in DMSO. Insets: nonlinear fitting of the changes in fluorescence intensity upon addition of the guest (λ_{em} = 326 nm, λ_{ex} = 300 nm)

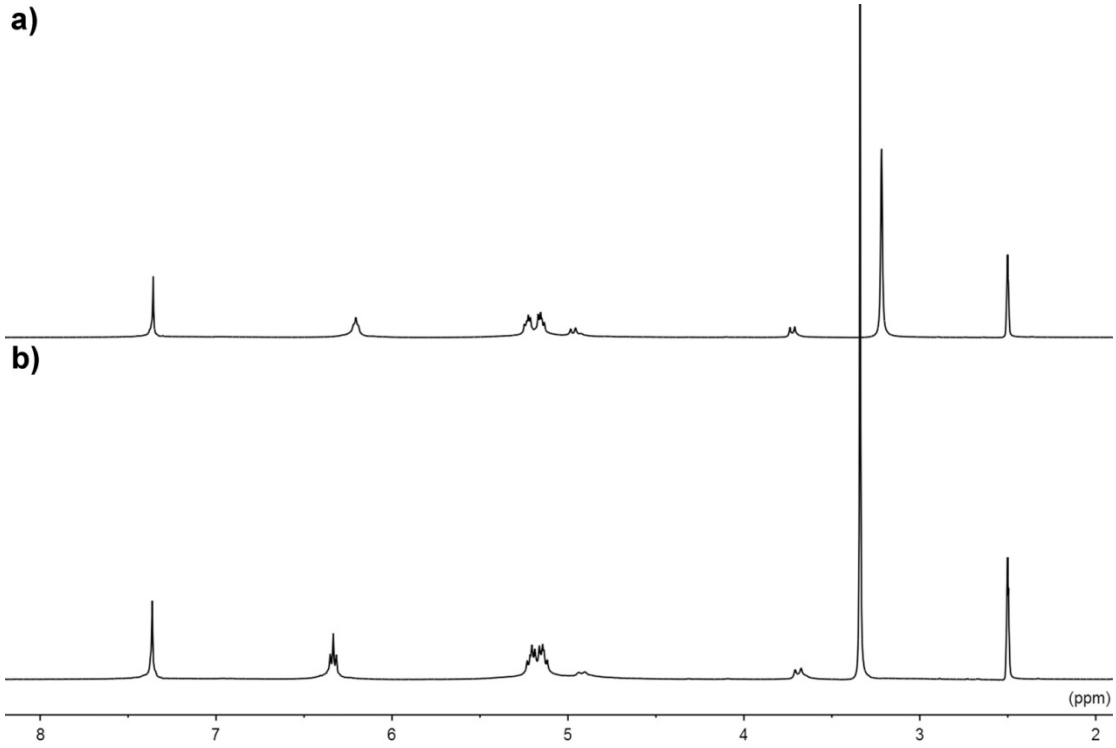

Figure S4 ^{1}H NMR spectra of benzo[6]uril **1** at a) 50 and b) 25 °C

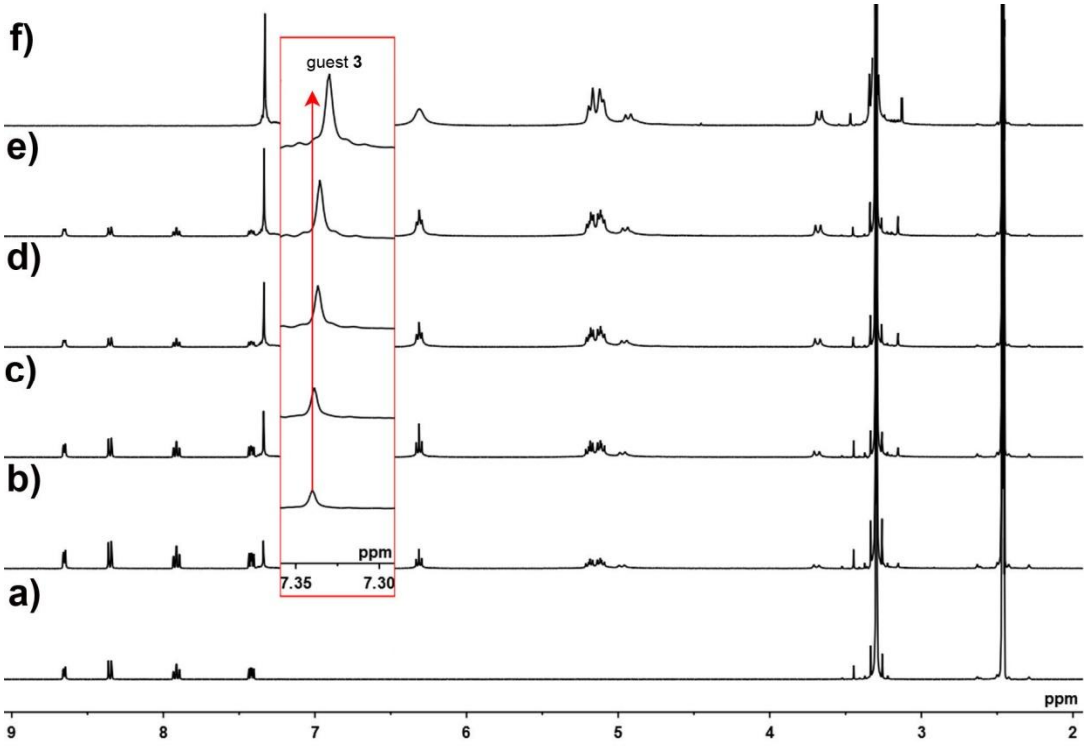

Figure S5 ^{1}H NMR spectra (400 MHz, [D_6]DMSO) of a) free guest **3**(1.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.5 equiv.; c) 1.0 equiv.; d) 2.0 equiv.; e) 3.0 equiv. of benzo[6]uril **1**, and f) free benzo[6]uril **1**.

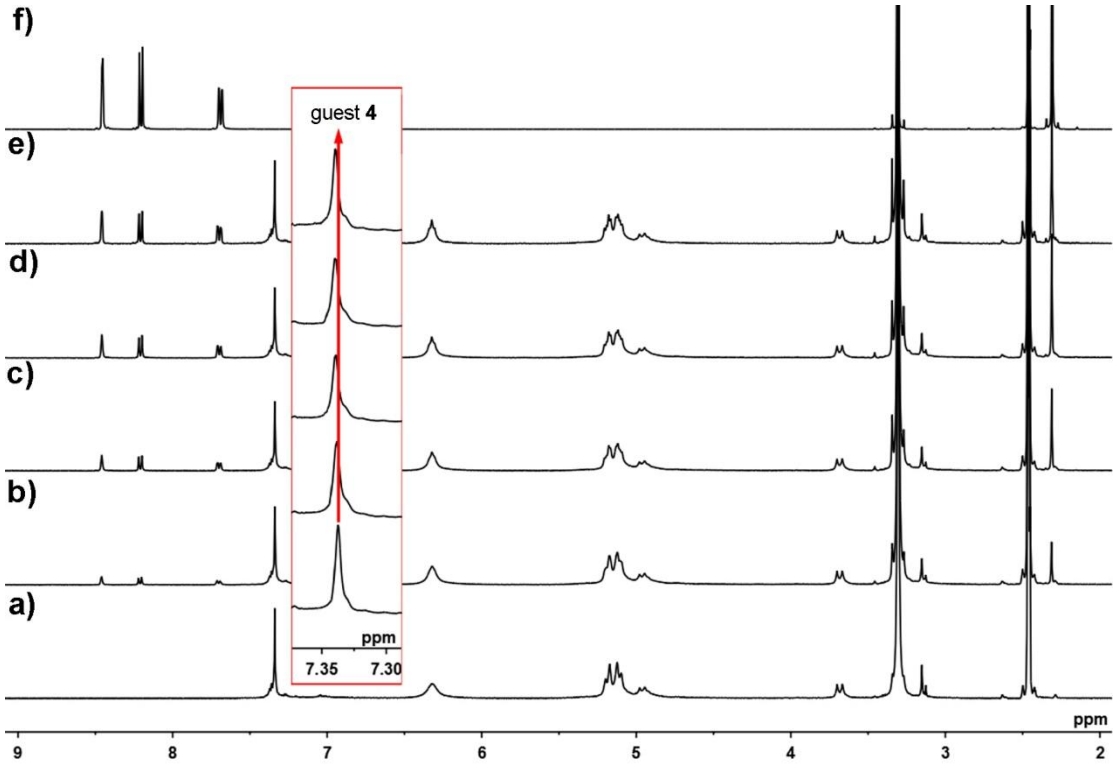

Figure S6 ^{1}H NMR spectra (400 MHz, [D_6]DMSO) of a) free benzo[6]uril **1** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.5 equiv.; c) 1.0 equiv.; d) 2.0 equiv.; e) 3.0 equiv. of guest **4**, and f) free guest **4**.

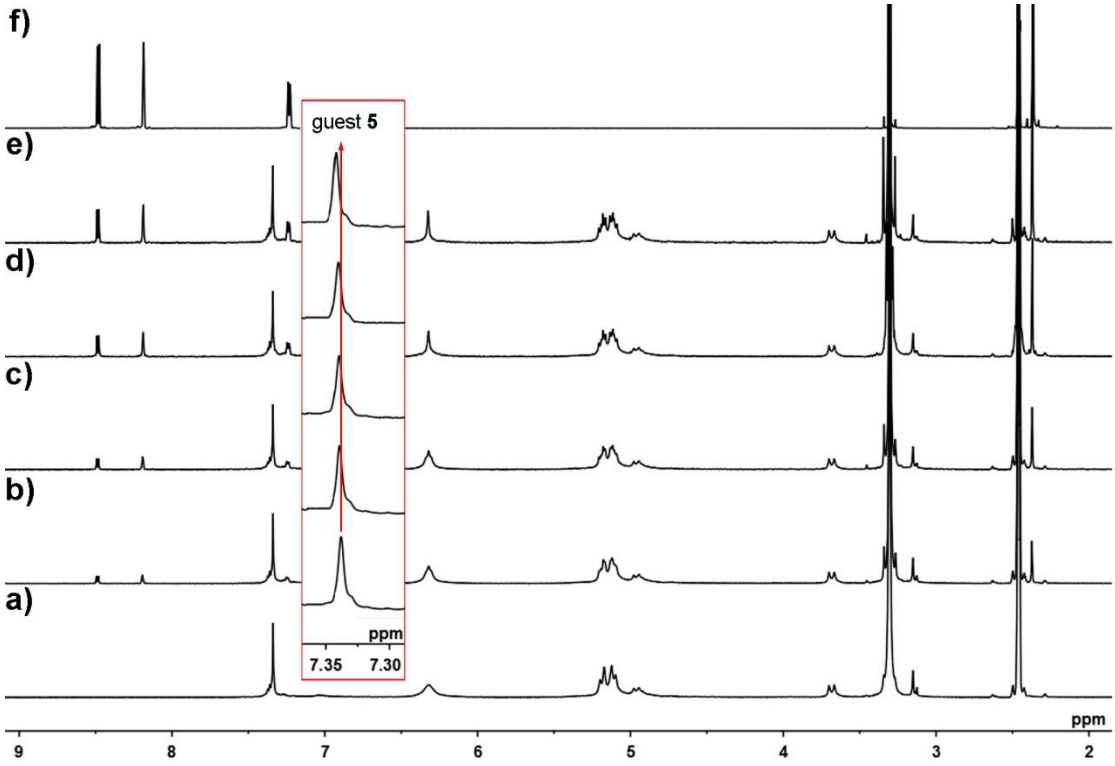

Figure S7 ^{1}H NMR spectra (400 MHz, [D_6]DMSO) of a) free benzo[6]uril **1** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.5 equiv.; c) 1.0 equiv.; d) 2.0 equiv.; e) 3.0 equiv. of guest **5**, and f) free guest **5**.

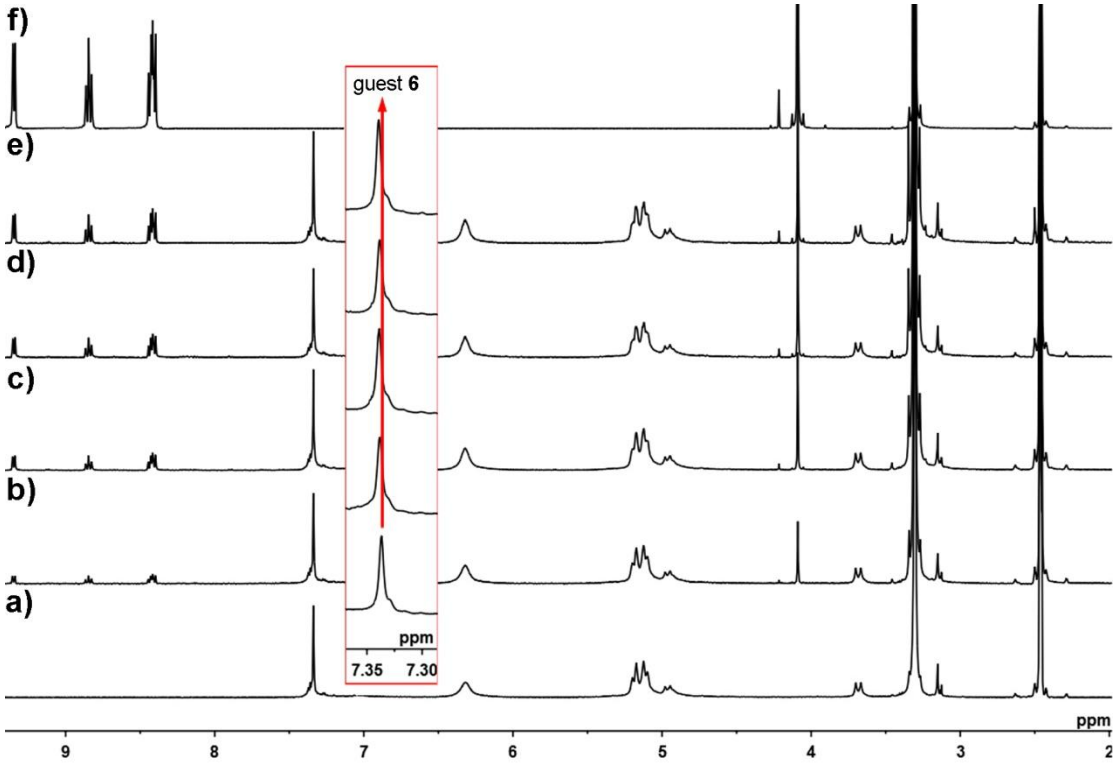

Figure S8 ^{1}H NMR spectra (400 MHz, [D_6]DMSO) of a) free benzo[6]uril **1** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.5 equiv.; c) 1.0 equiv.; d) 2.0 equiv.; e) 3.0 equiv. of guest **6**, and f) free guest **6**.

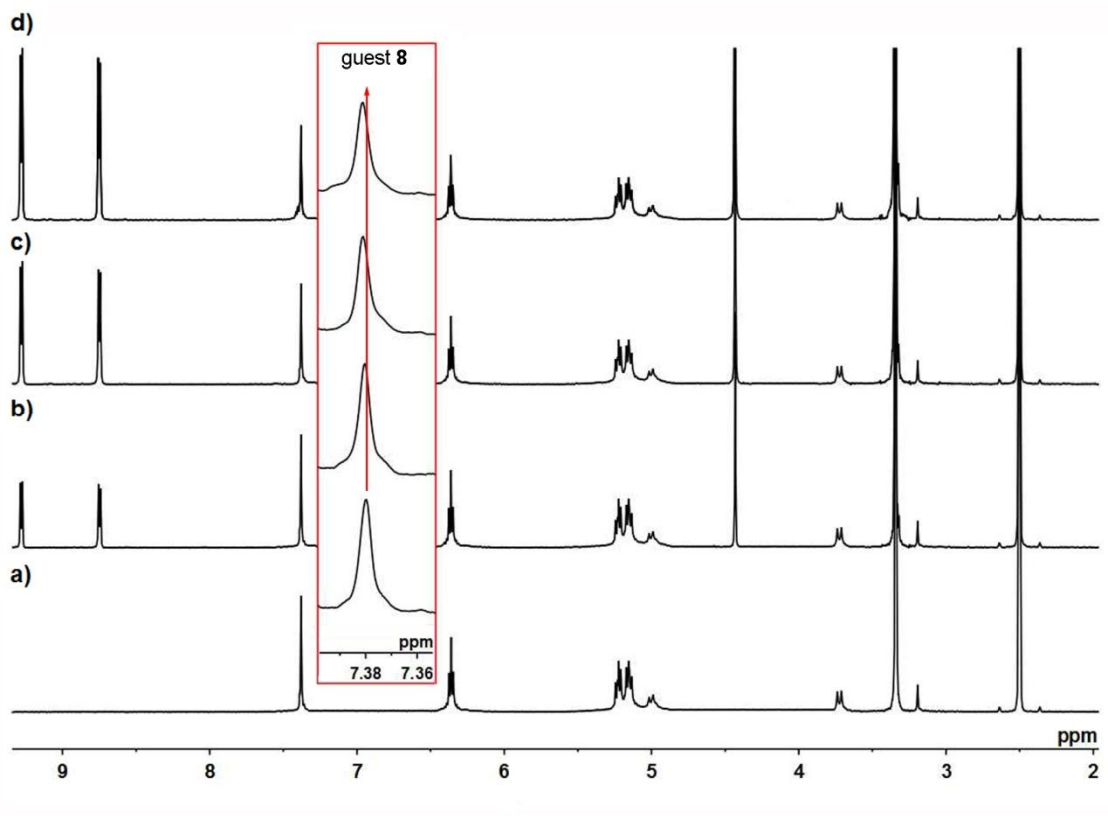

Figure S9 ^{1}H NMR spectra (400 MHz, [D_6]DMSO) of a) free benzo[6]uril **1** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 2.0 equiv.; c) 4.0 equiv.; d) 6.0 equiv. of guest **8**.

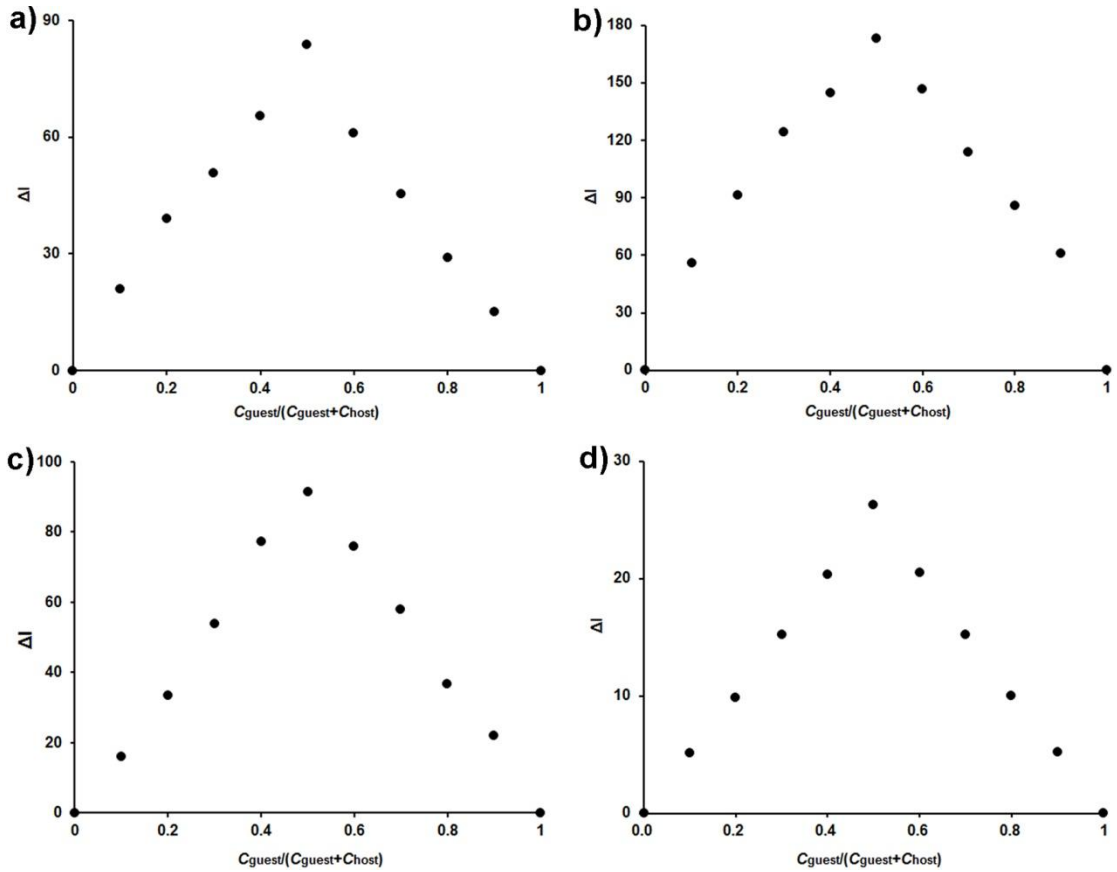

Figure S10 Job's plots of the interaction between benzo[6]uril **2** and a) guest **3**; b) guest **4**; c) guest **5**; d) guest **8**. The total concentration was maintained at 1.0×10^{-5} mol·L $^{-1}$, and fluorescence intensities were recorded at λ em = 327 nm, λ ex = 304 nm for host **2** system.

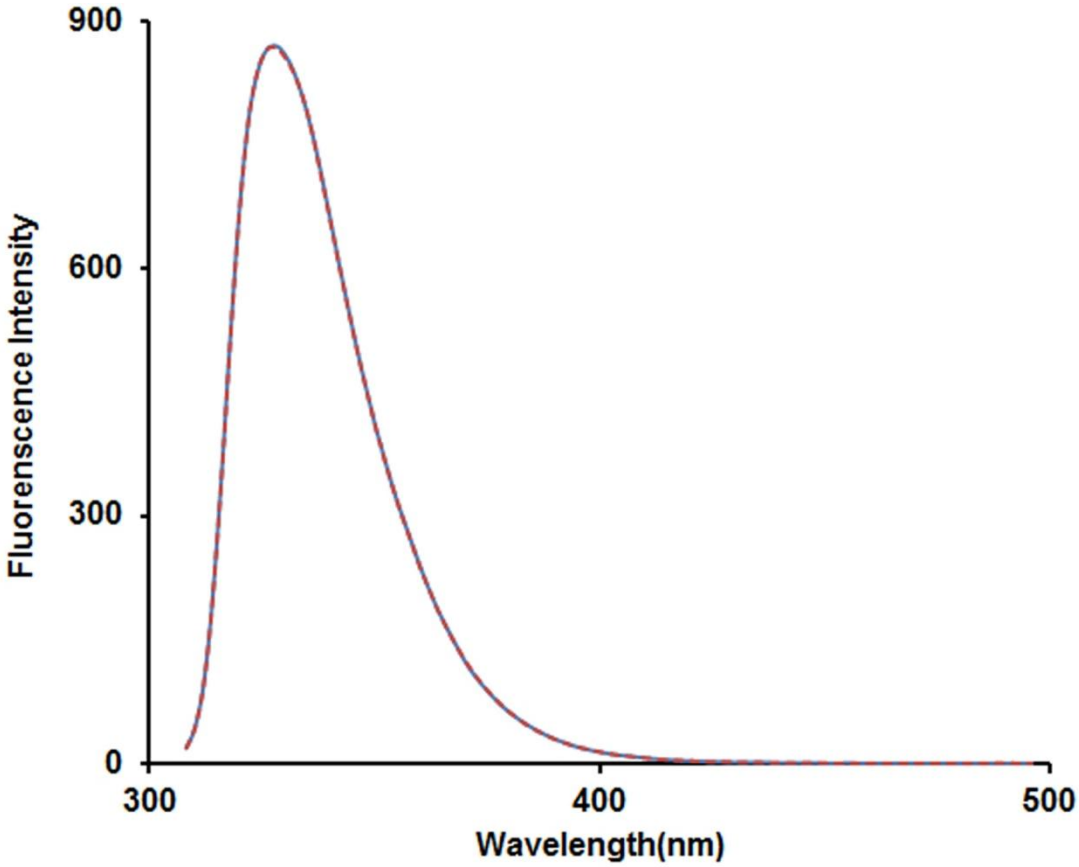

Figure S11 Fluorescence spectra of benzo[6]uril **2** (1.0×10^{-5} mol·L^{-1} in CH_2Cl_2/CH_3OH, 5:1, v/v) in the absence (blue solid) and presence of 1 eq. guest **7** (red dash).

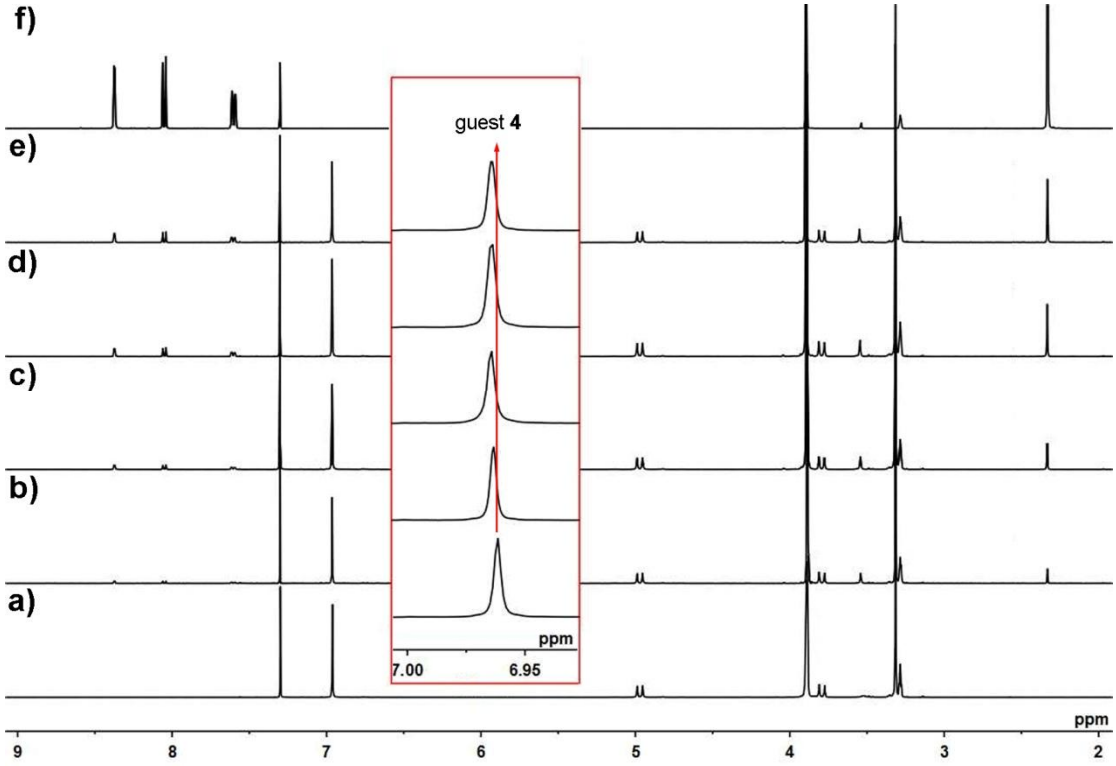

Figure S12 ^{1}H NMR spectra (400 MHz, $CDCl_3/CD_3OD$, 5:1, v/v) of a) benzo[6]uril **2** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.5 equiv.; c) 1.0 equiv.; d) 2.0 equiv.; and e) 3.0 equiv. of guest **4**, and f) free guest **4**.

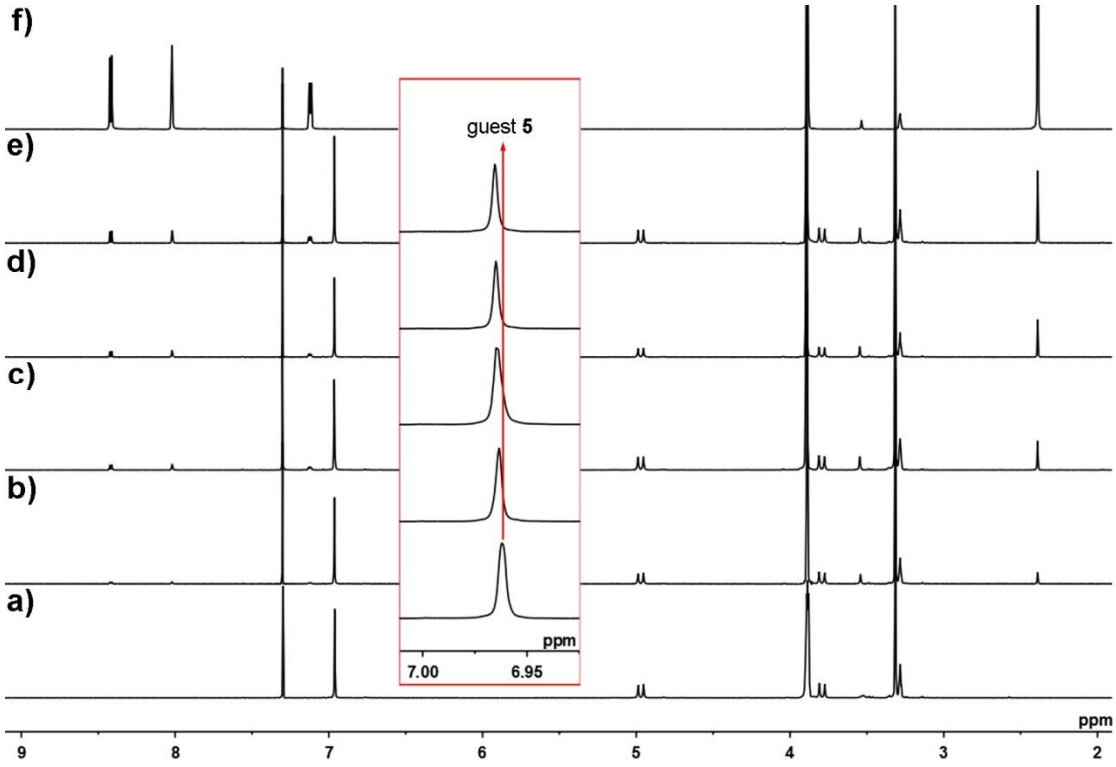

Figure S13 ^{1}H NMR spectra (400 MHz, $CDCl_3/CD_3OD$, 5:1, v/v) of a) benzo[6]uril **2** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.5 equiv.; c) 1.0 equiv.; d) 2.0 equiv.; and e) 3.0 equiv. of guest **5**, and f) free guest **5**.

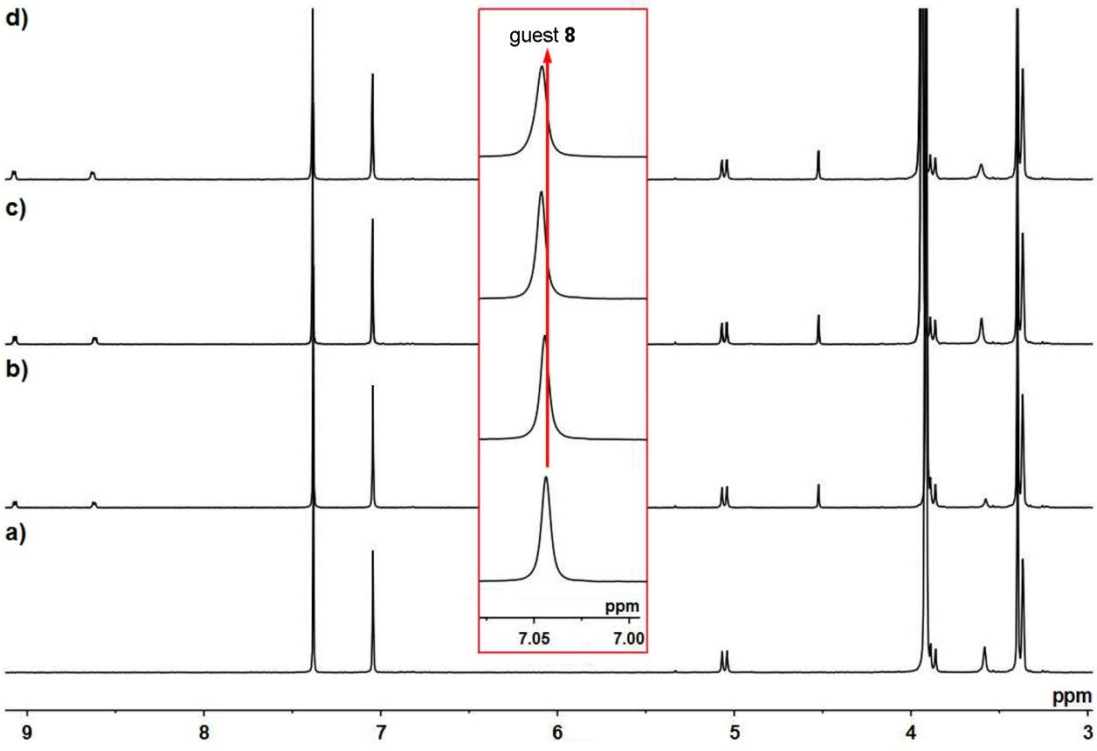

Figure S14 ^{1}H NMR spectra (400 MHz, $CDCl_3/CD_3OD$, 5:1, v/v) of a) benzo[6]uril **2** (2.0×10^{-3} mol·L^{-1}), and in the presence of b) 0.2 equiv.; c) 0.4 equiv.; and d) 0.6 equiv. of guest **8**.

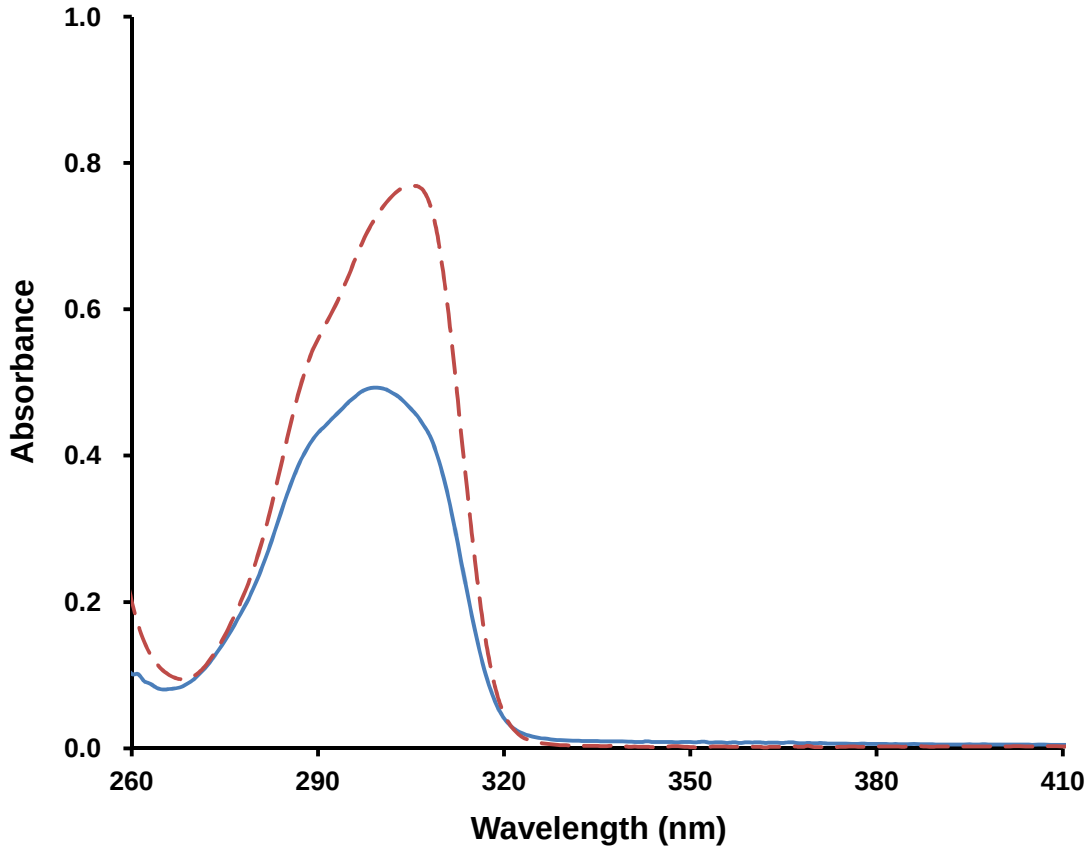

Figure S15 UV-vis spectra of benzo[6]uril **1** in DMSO (1.0×10^{-5} mol·L^{-1}, solid line) and benzo[6]uril **2** in CH_2Cl_2/CH_3OH (v/v, 5:1; 1.5×10^{-5} mol·L^{-1}, dash line).

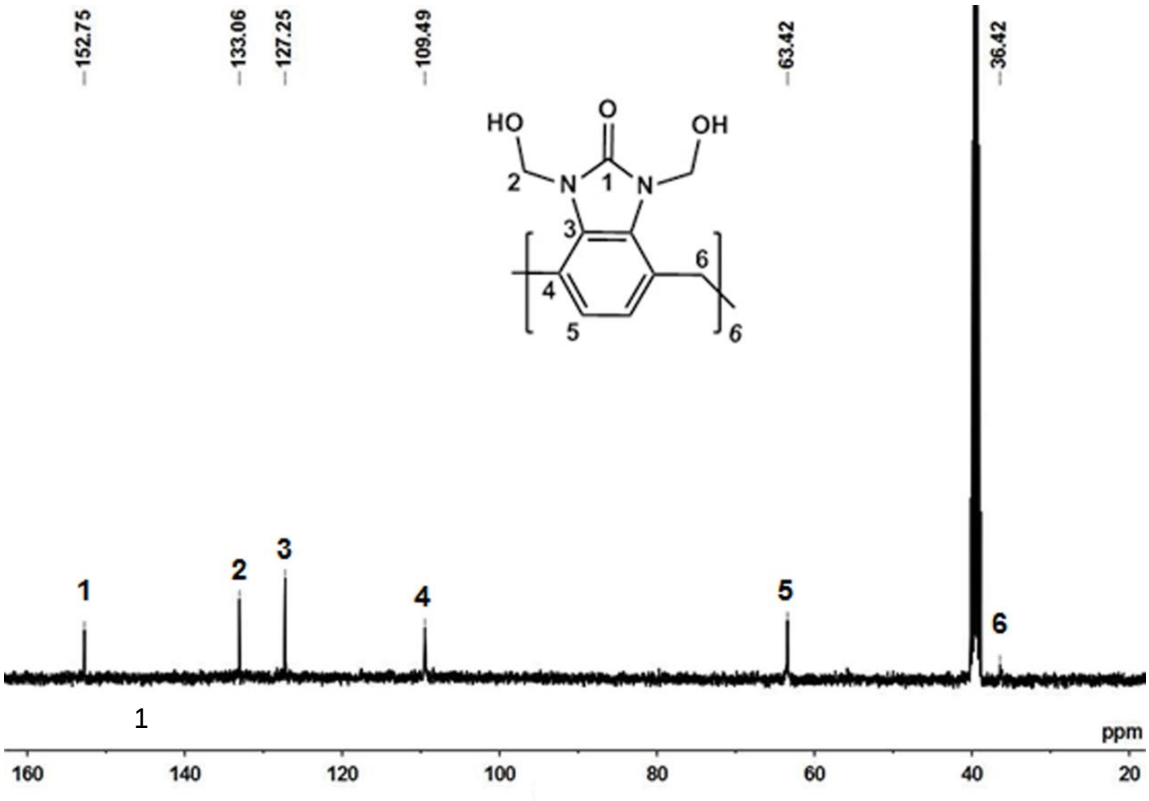

Figure S16 ^{13}CNMR spectrum of benzo[6]uril **1**.

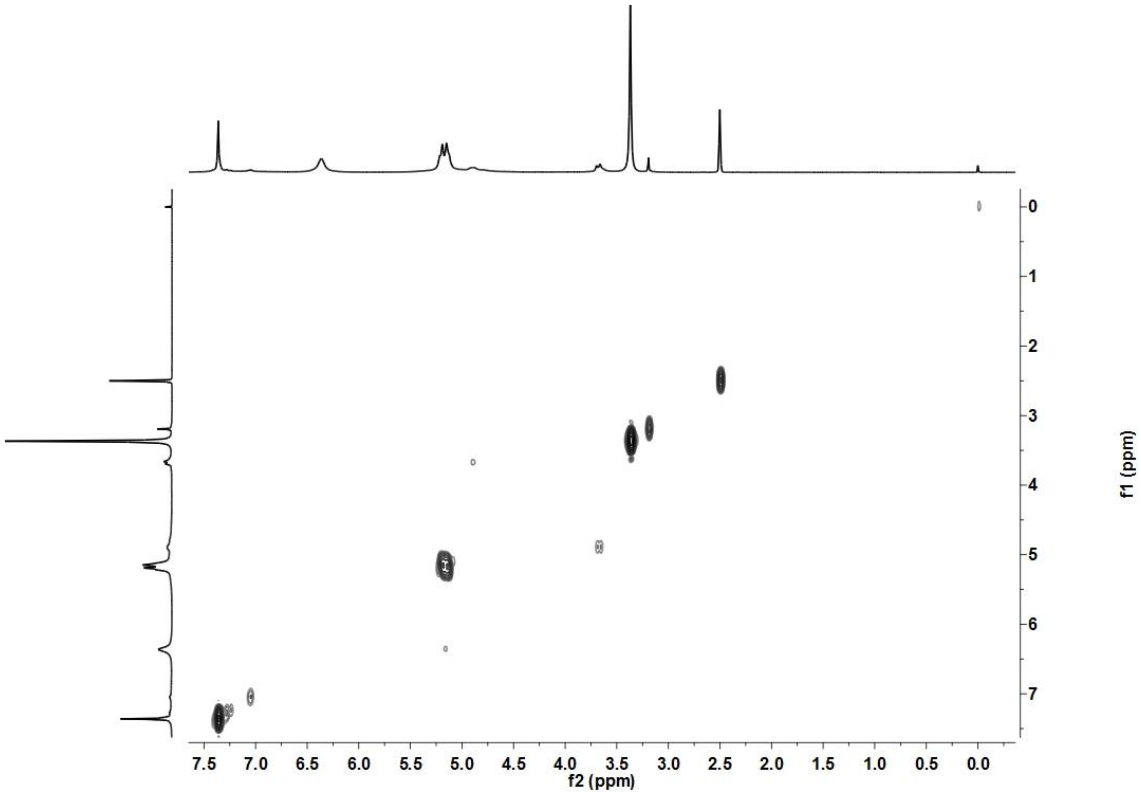

Figure S17 gCOSY NMR spectrum of benzo[6]uril **1**.

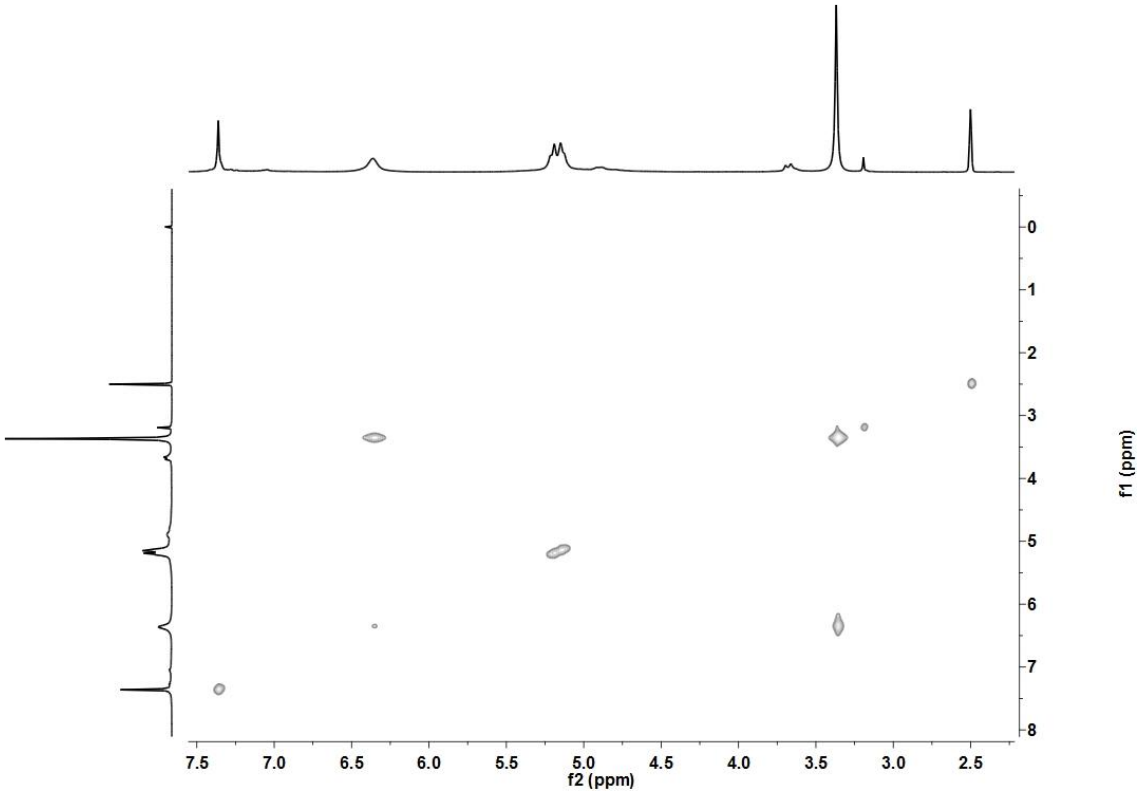

Figure S18 NOESY NMR spectrum of benzo[6]uril **1**.

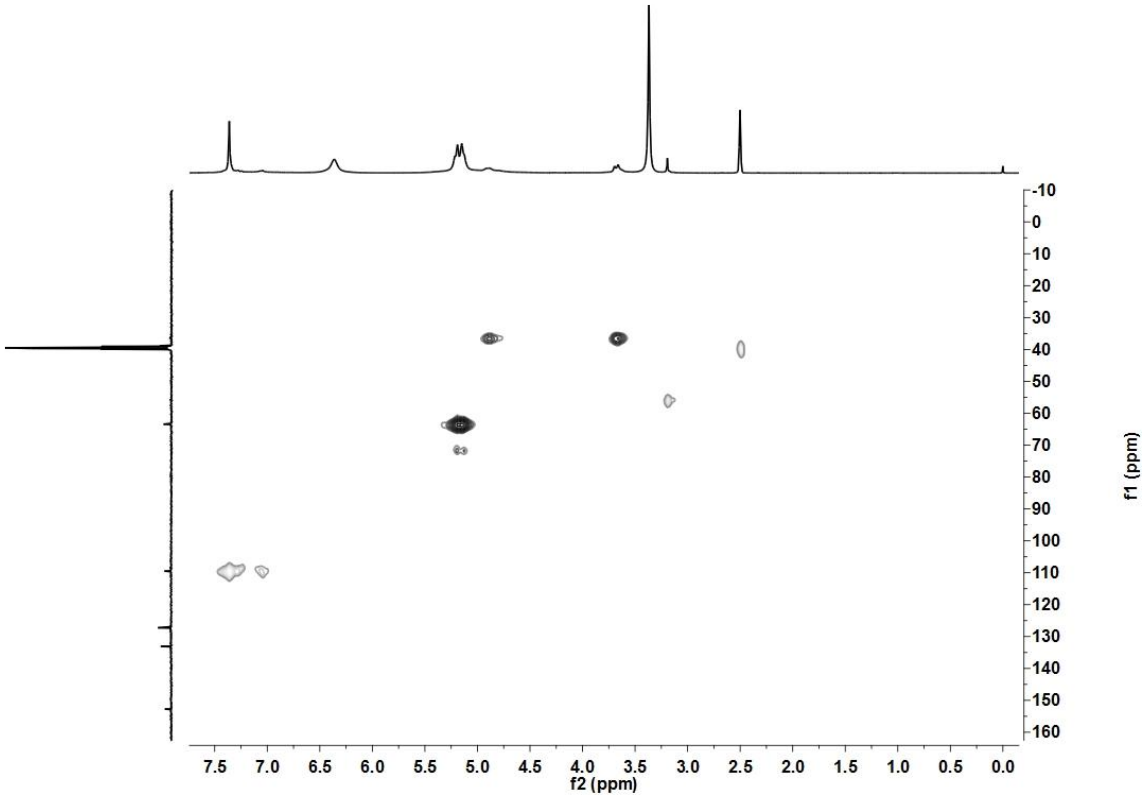

Figure S19 gHSQC (Heteronuclear Single Quantum Correlation) NMR spectrum of benzo[6]uril **1**.

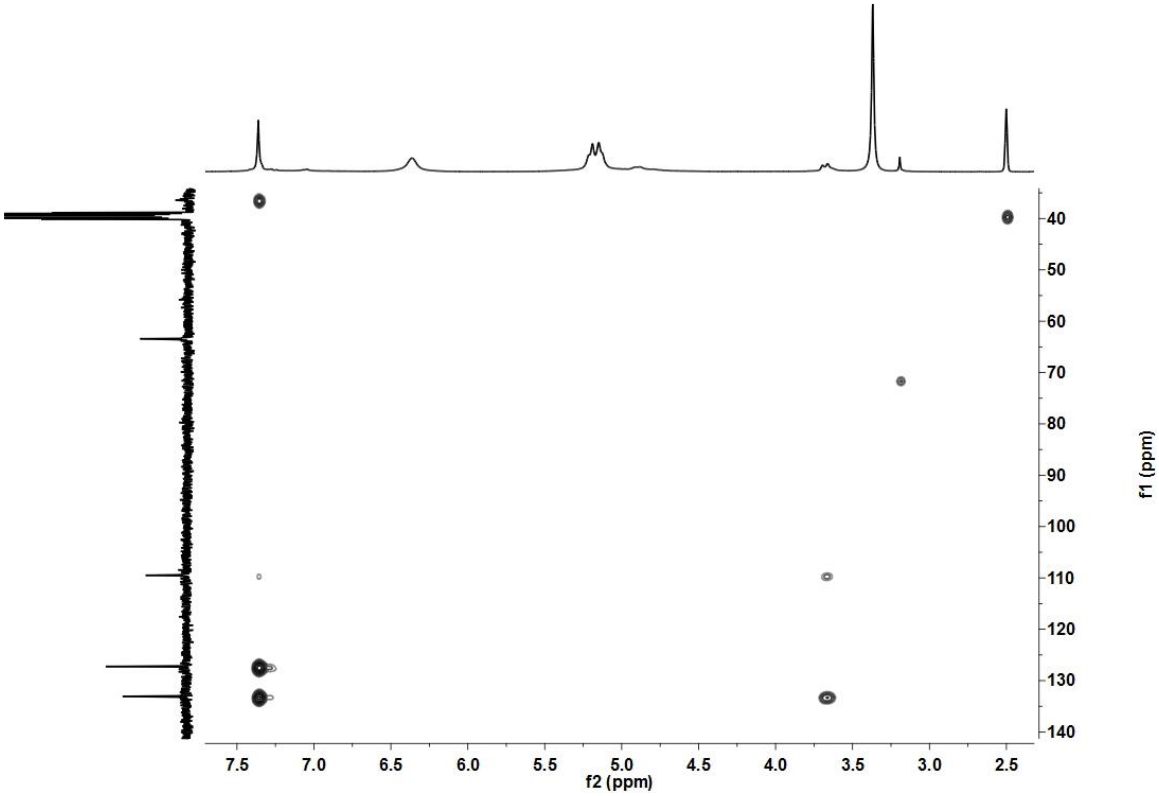

Figure S20 gHMBC (Heteronuclear Multiple Bond Correlation) NMR spectrum of benzo[6]uril **1**.

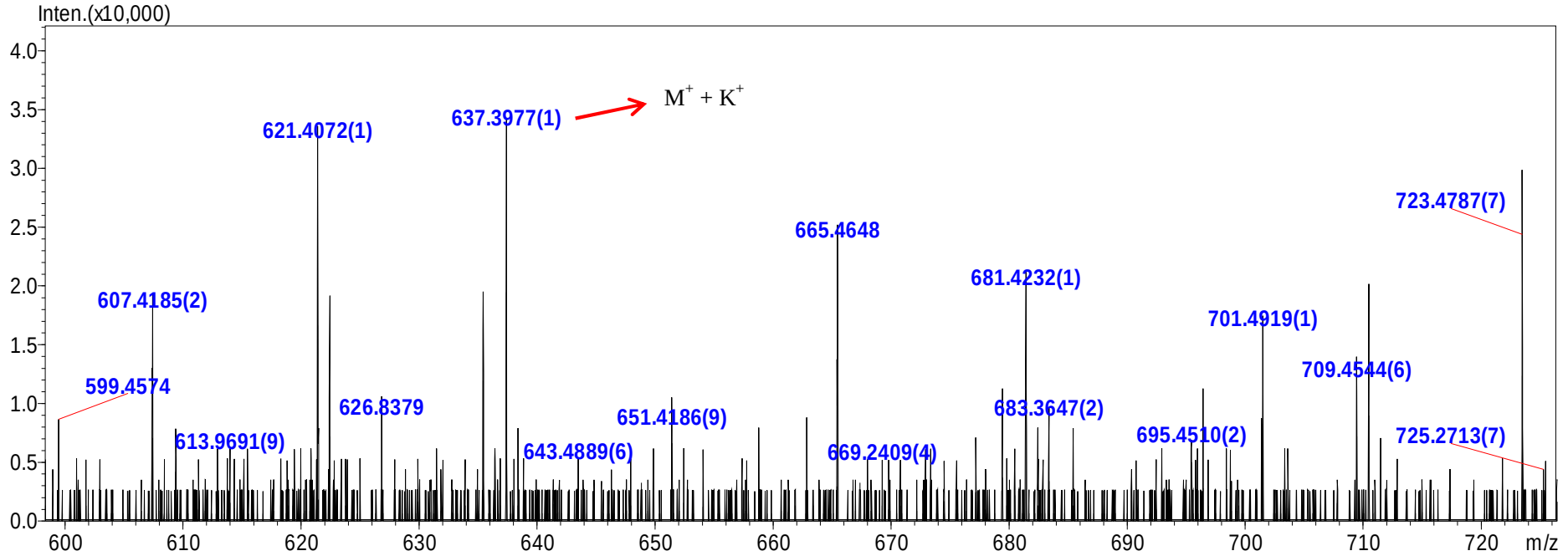

Figure S21 High resolution MS of benzo[6]uril **1**.

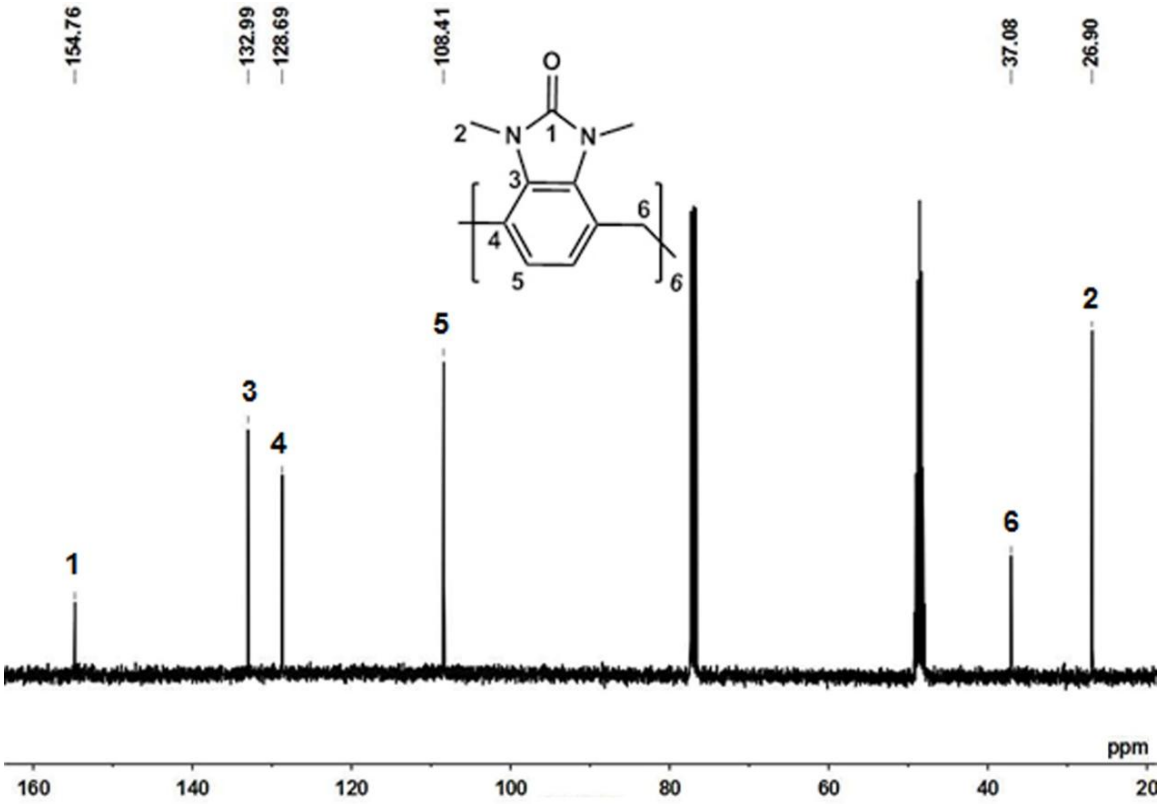

Figure S22 [13] CNMR spectrum of benzo[6]uril **2**.

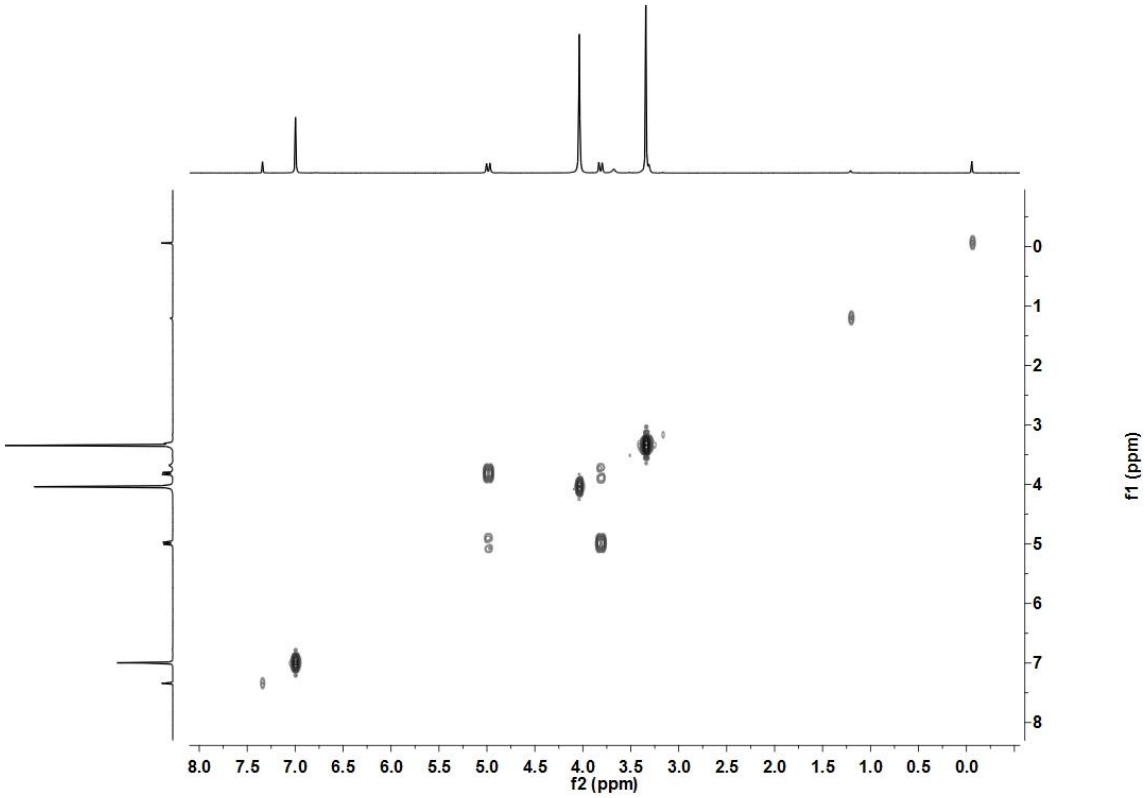

Figure S23 gCOSY NMR spectrum of benzo[6]uril **2**.

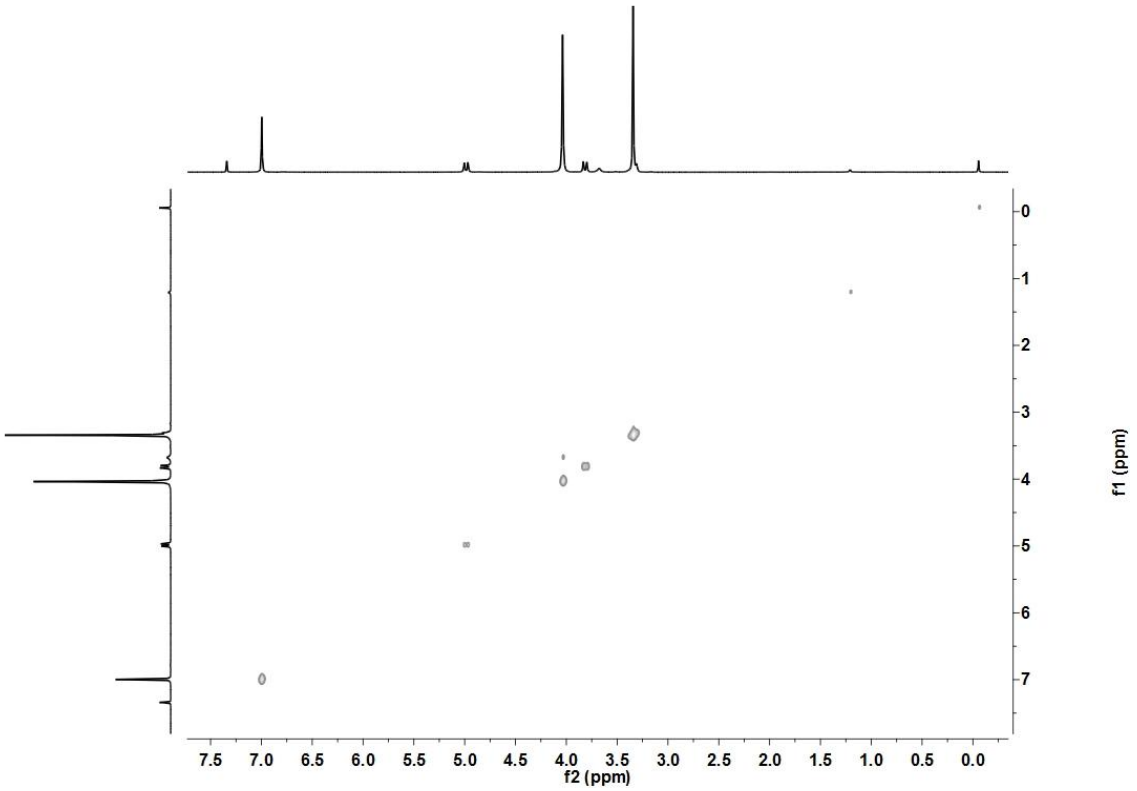

Figure S24 NOESY NMR spectrum of benzo[6]uril **2**.

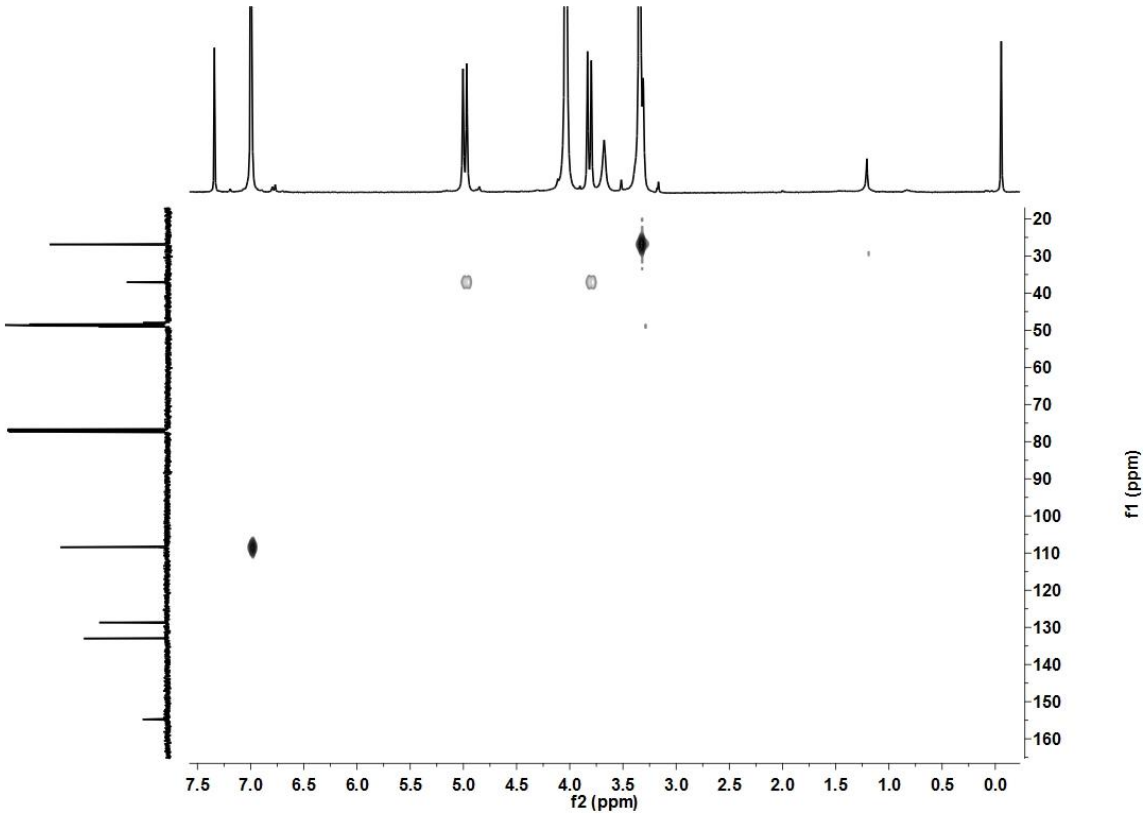

Figure S25 gHSQC (Heteronuclear Single Quantum Correlation) NMR spectrum of benzo[6]uril **2**.

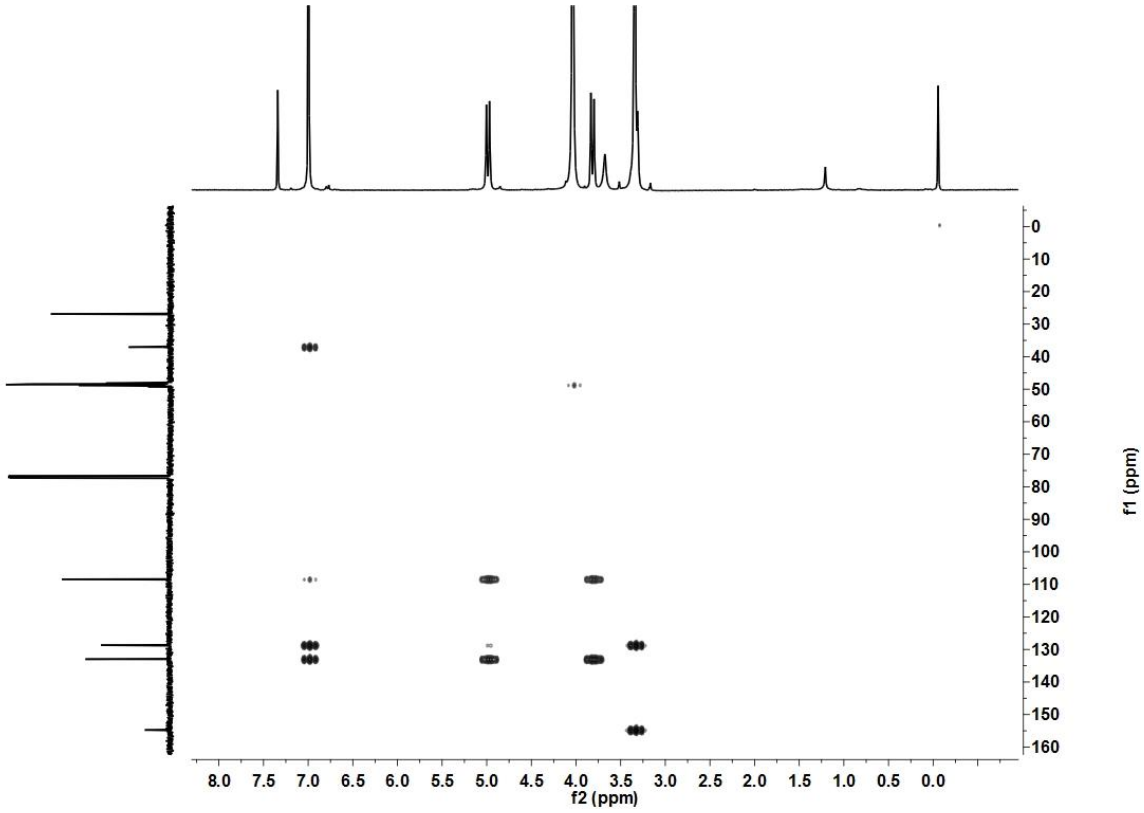

Figure S26 gHMBC (Heteronuclear Multiple Bond Correlation) NMR spectrum of benzo[6]uril **2**.

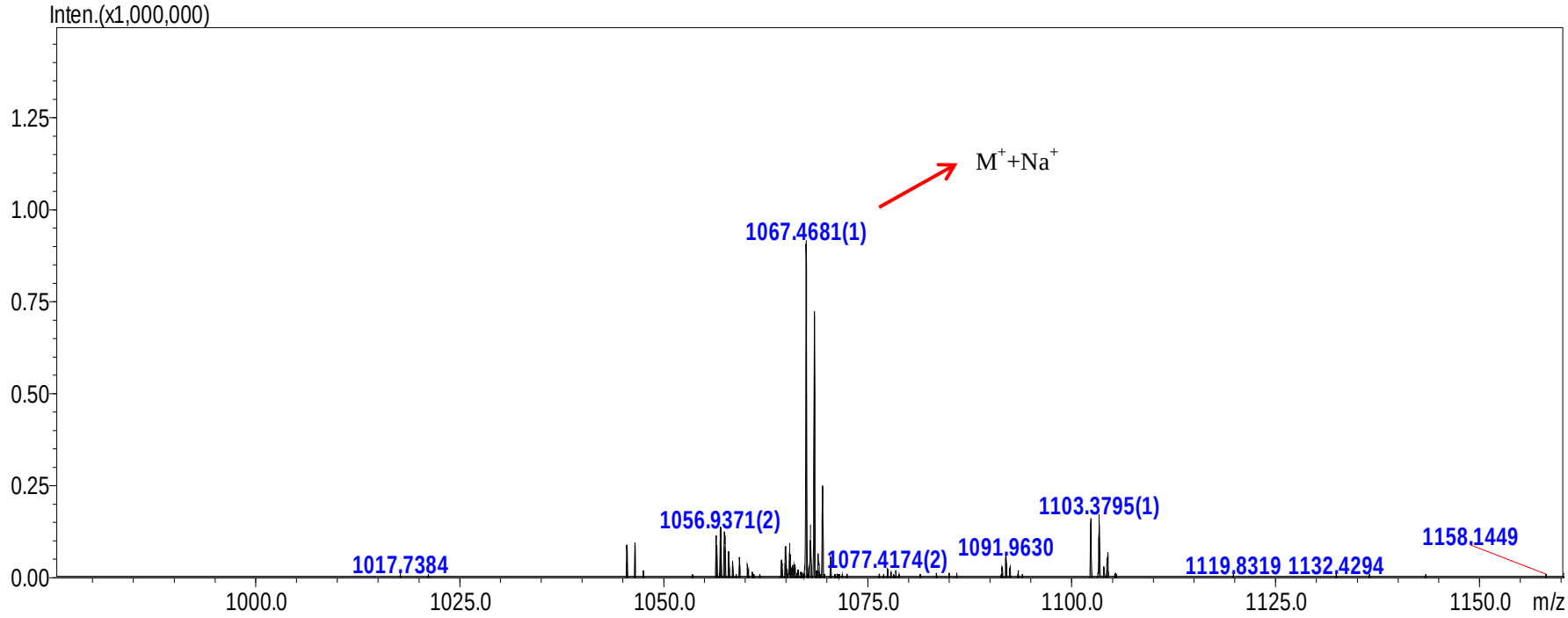

Figure S27 High resolution MS of benzo[6]uril **2**.