Supplementary Information

Hexameric assembly of 5,17-di-substituted calix[4]arene in the solid state

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Figure S1. $^1$H (500 MHz, DMSO-$d_6$, 293 K) and $^{13}$C (125 MHz, DMSO-$d_6$, 293 K) NMR spectra of (S,S)-1.

Figure S2. X-ray crystal structure of (S,S)-1·(MeOH) viewed down along the crystallographic $c$ axis. Color scheme: gray (carbon), white (hydrogen), blue (nitrogen), red (oxygen).
Figure S3. X-ray crystal structure of (S,S)-1-(1-PrOH) viewed down along the crystallographic c axis. Color scheme: gray (carbon), white (hydrogen), blue (nitrogen), red (oxygen).

Figure S4. X-ray crystal structure of (S,S)-1-(CH3CN) viewed down along the crystallographic c axis. Color scheme: gray (carbon), white (hydrogen), blue (nitrogen), red (oxygen).
Figure S5. Head-to-tail columnar structures consisting of \((R,R)-1\) (left) and \((S,S)-1\) (right) found in \textit{rac}-1. Color scheme: gray (carbon), white (hydrogen), blue (nitrogen), red (oxygen).

Figure S6. \(^1\text{H} \text{NMR} \) spectrum (300 MHz, DMSO-\(d_6\), 293 K) of \((S,S)-1_{\text{apo}}\).

Figure S7. \(^1\text{H} \text{NMR} \) spectrum (300 MHz, DMSO-\(d_6\), 293 K) of \((S,S)-I_{\text{apo}}\) after contacting benzene vapor with \((S,S)-I_{\text{apo}}\) for 3 days at room temperature. Open circle denotes the signal of benzene.
Figure S8. $^1$H NMR spectrum (300 MHz, DMSO-$d_6$, 293 K) of (S,S)-I$_{apo}$ after contacting a mixed vapor of benzene and acetone with (S,S)-I$_{apo}$ for 3 days at room temperature. Open circle denotes the signal of benzene.

Figure S9. $^1$H NMR spectrum (300 MHz, DMSO-$d_6$, 293 K) of (S,S)-I$_{apo}$ after contacting a mixed vapor of benzene and ethyl acetate with (S,S)-I$_{apo}$ for 3 days at room temperature. Open circle denotes the signal of benzene.