Supplementary Information Presented with the Paper Entitled

Supramolecular Assembly Based on *p*-Sulfonatothiacalix[6]arene with Sodium and Water molecules

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Experimental Section for General Procedures

Table 1 Effect of yield for **1** by various reaction temperatures.

General Procedure

^{1}H NMR and ^{13}C NMR spectra were taken on Bruker DPX 300 spectrometer. IR spectra were obtained with a Perkin-Elmer SPECTRUM 2000 spectrophotometer.

Referring a procedure of synthesis of **2**, we tried to synthesize **1**. Unfortunately, there was something problems, because salting out with excess of NaCl gave impured precipitates containing **1**. To obtain **1** as pure, we modified purification method. The obtained precipitates were washed with a small amount of conc. H_2SO_4 to remove impure substances. After that precipitates was resolved in water, and insoluble fractions was separated by filtration. Pure **1** was obtained from the resulting water soluble fractions by addition of excess NaCl. Table 1 show that the reaction temperature for sulfonation of *p-tert*-butylthiacalix[6]arene (TC6A). In the case of Run 1, it was observed that almost unreacted TC6A was isolated. In the case of Run 2, amount of unreacted TC6A decreased and the yield was raised up 20%, which means elevation of 10°C of a reaction temperature works effectively. In the case of Run 3, it was observed a yield came up more 10% increase. However, in the cases of Run 4 and 5, the yields go down, which means decomposition reaction proceeded than that of sulfonation. The most favourable reaction condition was determined as reaction temperature and reaction time are 100°C and 3 h, respectively.

Table 1 (Supplementary Information)

Run	Temperature (ºC)	Unreacted TC6A (g)	yield (%)
1	80	0.71	8 (0.09 g)
2	90	0.46	28 (0.32 g)
3	100	0.27	38 (0.43 g)
4	110	0.10	36 (0.41 g)
5	120	0	0

Reaction time: 3 h, Starting material: TC6A 1.0 g (0.925 mmol), Reagent: conc. H_2SO_4 10 mL