## Methyl-Substituted Cucurbit[6]uril-Based Microporous Supramolecular Frameworks for Highly Selective Et<sub>2</sub>O/CH<sub>3</sub>OH Adsorption

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## **EXPERIMENTAL SECTION**

Fig. S1. The sorption isotherms of N<sub>2</sub> at 77 K by a micrometrics ASAP2020HD88 automated sorption analyser.  $\blacksquare$  symbol = adsorption and,  $\bullet$  symbol = desorption of the AE<sup>2+</sup>-HMeQ[6]-[CdCl<sub>4</sub>]<sup>2</sup>-based microporous materials.



Fig. S2. Recyclability tests for methanol adsorption of the  $Ba^{2+}-HMeQ[6]-[CdCl_4]^{2-}$  system microporous material.



Fig. S3. Powder X-ray diffraction (PXRD) of the coordination complexes 1, 2 and the corresponding comparison with simulation.



Fig. S4. TG curves of the coordination complexes 1, 2 and the corresponding comparison with HMeQ[6] powder in N<sub>2</sub> respectively.