

Smart Polymorphism of Thiacalix[4]arene with Long-Chain Amide Containing Substituents

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Abstract

© 2017 American Chemical Society. A problem of controlled (smart) formation of polymorphs was solved for a set of tert-butylthiacalix[4]arene derivatives with four N-(*n*-acetoxyethyl)carbamoylmethoxy substituents at the lower rim with 1,3-alternate, cone, and partial cone conformations. For this, an effective polymorph screening with a reproducible influence of preparation history was achieved using guest vapor inclusion and a standard state of host glass powder. By this procedure with consequent guest release and heating, the ability of the studied calixarenes for polymorphism was investigated and compared as a function of their macrocycle conformation. The data of simultaneous thermogravimetry and differential scanning calorimetry with mass spectrometry of evolved vapors were determined together with the data of powder X-ray diffraction for the initial host samples, intermediate clathrates, and final polymorphs. In addition, single crystal X-ray diffraction data were obtained for four crystalline forms of the studied calixarenes. The results yielded a structure-property relationship, where 1,3-alternate calixarene without an extended H-bonded supramolecular network at least in one crystalline form has a much higher ability for polymorphism than the other two conformations. Thus, 10 polymorphs with essentially different crystal packing were found for this calixarene including a unique tetramorphism with four consecutive melting points of guest-free polymorph and corresponding three crystallization ranges. This ability of 1,3-alternate calixarene is linked with its other smart property: selective crystallization of its compact glass in vapors of binary liquid mixtures, which can be used for visual detection of very small benzene impurities (0.5% (v/v)) in cyclohexane.

<http://dx.doi.org/10.1021/acs.cgd.7b00463>

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