

Smart control of calixarene polymorphic states

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Abstract

© The Royal Society of Chemistry. An efficient strategy of polymorph screening is offered for tert-butylthiacalix[4]arene tetrasubstituted with phenacyloxy groups (**1**), which is based on saturation of its guest-free solid samples and inclusion compounds with guest vapors inducing solid-solid phase transition, complete or partial exchange or repulsion of included guests. The offered approach makes the preparation of polymorphs more reproducible and predictable as the performed phase transitions are under mostly thermodynamic control even when giving metastable forms. Smart transformation of calixarene states was demonstrated where stable and metastable polymorphs are formed in a kind of on/off switching. Guest repulsion or solid-phase exchange with another guest vapor gives a guest-free form directly, or a less stable inclusion compound is prepared that can release guests by drying under milder conditions without host collapse to a stable packing. In total, seven polymorphs of **1** were identified and characterized. Single-crystal data reveal the polymorphophore features of **1**, which help to control its polymorphic state. The observed control of polymorphic transitions contributes to the comprehension of their causes and mechanism. The elaborated procedures enable an effective screening of polymorphs through a standardized treatment of the target compound on a click basis with a flexible variation of conditions, which may be scaled up to find more crystal forms. This journal is

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