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Ternary inclusion compounds based on the gossypol-chloroform host matrix - stabilizing effect of the aromatic guest component

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Gossypol (Gp), the natural product of a polyphenolic nature, is known to form a large variety of inclusion compounds with solvent molecules as the guest components. From aprotic solvents Gp inclusion compounds are formed that are based on the host matrix constructed from columnar Gp assemblies, that in the highest symmetrical case belong to the $P12/c1$ rod group [1]. This group of compounds is represented by unstable Gp/CHCl₃ (1/1) inclusion compound that crystallizes in the C2/c space group with $Z'=1$. It has been proposed that desolvation of this compound proceeds stepwise *via* crystalline forms with the 4:3, 4:2 and 4:1 host:guest ratio [2]. Indeed, inclusion compounds having these stoichiometries and constructed from similar host columnar aggregates were prepared when Gp was recrystallized from toluene, benzene and p-xylene, respectively. Moreover, the intermediate form of Gp/CHCl₃ inclusion compound with the 4:2 host:guest ratio was also obtained.

To our surprise, when Gp was recrystallized from a mixture of solvents: CHCl₃/benzene, CHCl₃/toluene, CHCl₃/p-xylene, stable ternary inclusion compounds were formed with the aromatic molecules (Ar) occupying every second centrosymmetric void in the basic Gp/CHCl₃ (1/1) matrix. These inclusion compounds have Gp/CHCl₃/Ar ratio of 4/4/1 and crystallize in the P-1 or P2₁/c space groups with $Z'=4$. Similar ternary compounds were also obtained with naphthalene or pentafluoroaniline as the third component. In turn, when Gp crystallization was performed from mixtures of CHCl₃ with o- or m-xylenes another group of ternary inclusion compounds was obtained with Gp/CHCl₃/Ar ratio of 4/2/1 (P2₁/c, $Z'=2$). Structure of these compounds is to a large extent isostructural with the binary inclusion compound Gp/toluene (4/3), but with two symmetry independent guest sites occupied in ternary compounds by different guest components.

In addition to structural studies, stability data for the ternary Gp compounds will be presented.

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Exploring the assembly possibilities of calix[4]dihydroquinone

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The strong interest towards the synthesis of new building blocks in the field of the Crystal Engineering is related to the aim of this discipline that is "the rational design of functional molecular solids" [1].

Calix[4]arenes are a class of organic compounds investigated to meet this aim. 4,4'-Bipyridine is a rigid linker frequently used in the assembly of supramolecular architectures [2]. In fact it is able to act as hydrogen bond acceptor and to give rise to stacking $\pi - \pi$ or to other van der Waals-like interactions. Despite of these features, only few

examples of co-crystals between calix[4]arenes and 4,4'-bipyridine have been reported [3].

At the Dept. of Chemistry and Biology of the University of Salerno we prepared and characterized a new crystalline solid based on *p*-Bu'-calix[4]dihydroquinone **1**. The molecule, thanks to the presence of two vicinal hydroquinone moieties and to the large *t*-butyl substituents, is able to form a zeolite-like network [4].

Recently we have shown that **1** exhibits a certain variety of assembly possibilities, which can be triggered by appropriate solvent choice. [5] In particular, by using as crystallization solvents chloroform and water, it is possible to obtain a triclinic system. The unit cell contains two equivalent calixarene molecules, two water molecules and two chloroform molecules. The water molecules bridge the calixarene molecules through hydrogen bonds. The chloroform molecule is located inside the cavity of the calixarene molecule (Figure 1)

Considering the packing features we decided to replace the water molecules with the 4,4'-bipyridine molecules and to verify if the general arrangement was preserved. Triclinic single crystals have been obtained. The unit cell contains two equivalent calixarene molecules, one 4,4'-bipyridine molecule and two ethyl acetate molecules. Thanks to the similar molecular interactions the packing of the new structure is very similar to the one containing water. Nevertheless the 4,4'-bipyridine molecules show additional stacking $\pi - \pi$ interactions with the calixarene's aromatic rings (Figure 2).

These first results on the rational study of the possible intermolecular interactions could define a design strategy that could lead to predictable packing modes and be useful to obtain peculiar properties.

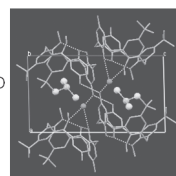
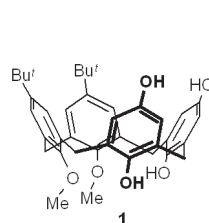


Figure 1

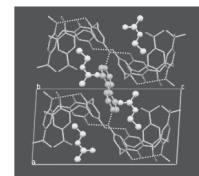


Figure 2

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Ultra high porosity, multi-variant MOFs and conducting porous materials

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In the present abstract we summarize part of the work that we are currently carrying out within the reticular chemistry field. In particular, some of the objectives in which we have focused our efforts are preparing materials with ultra high porosity, functionalizing the open spaces of MOFs in a controlled manner, and providing crystalline porous materials with new properties: