

were synthesized and their crystal structures were determined. Unexpectedly all such molecules form interlocked *Piedfort*-associates. Thus the two superposed molecules of a *Piedfort*-unit serve as supramolecular *synthon*[1]. We can also clearly see *intramolecular halogen-halogen interactions* which are developed at the external surface of the *Piedfort*-pair.

The effect of the halogen atom on the *Piedfort* stacking distances and the dimensions of the halogen *synthon* will be described in details. The authors acknowledge *Hungarian Scientific Research Fund* (OTKA grants T042642).

[1] Czugler M., Weber E., Párkányi L., Korkas P.P., Bombicz P., *Chem. Eur. J.*, 2003, **9**, 3741-3747.

**Keywords:** *Piedfort association, halogen-halogen interaction, supramolecular chemistry*

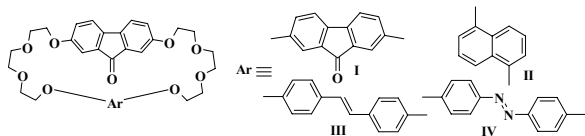
#### P.06.06.2

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#### Crystal Structures of Some Representatives of the Fluorenonocrownophanes

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Four new crownophanes containing the 2,7-dioxyfluorenone fragment, bridged with another aromatic units (I – IV) via triethylene glycol residues were synthesized as the perspective complexing agents for the metal and organic cations [1-2].



The molecular geometry of crownophanes and their packing in crystals are governed by the combination of intra- and intermolecular C–H...O hydrogen-bonding, C–H... $\pi$  and  $\pi$ – $\pi$  stacking interaction that provide the T-shape for all but bisfluorenone crownophane (Ar = I) which exhibits the antiparallel alignment of the fluorenone units in the centrosymmetric molecule. The incorporation of fluorenone building block in crownophane molecule ensures the advantages, such as strong hydrogen bonding of the substrate, often dominating in the processes of molecular recognition. Fluorenone and its derivatives have good luminescence properties that are important for the development of sensitive fluorescence-based chemosensors.

[1] Lukyanenko N.G., Kirichenko T.I., Lyapunov A.Yu., Mazepa A.V., Simonov Yu.A., Fonari M.S., Botoshansky M.M., *Chem. Eur. J.*, 2005, **11**, 262. [2] Lukyanenko N.G., Kirichenko T.I., Lyapunov A.Yu., Kulygina C.Yu., Simonov Yu.A., Fonari M.S., Botoshansky M.M., *Tet. Lett.*, 2004, **45**, 2927.

**Keywords:** *crown compounds, macrocycles, intermolecular interactions*

#### P.06.06.3

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#### Crystal Structure of Isosteviol and its Derivatives

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Crystal structure determination of 33 derivatives of plant terpenoid isosteviol and its molecular complexes with small molecules were performed. It was analyzed a molecular geometry and intermolecular interactions in the crystal, and types of crystalline packing. The absolute configuration of chiral centers of the molecule of isosteviol was established.

Shown that for single framework isosteviol derivatives, solvates, or complexes of including are got only at presence of not modified acid group.

For bis-framework derivatives – «tweezers» structures, built on

the type «head to head», molecular complexes is not observed, while in crystals of structures, having type of buildings «head to tail» even at short connecting bridge, received solvates with small molecules.

It was established that molecular complexes of isosteviol with small aromatic molecules (benzene, toluene, aniline, dimethylaniline, naphthalene, etc.) are isostructural in the space group  $P4_32_12$ . The supramolecular structure of these complexes is a double spiral around an axis of fourth order; branches, which formed by hydrogen bonded molecules of isosteviol. Molecules of aromatic «guests» are located in the same areas of crystal, but differently oriented for molecules of isosteviol. Shown that forming of complexes with technical mixtures of nitroanilines and toluene runs regioselective.

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**Keywords:** *absolute configuration, crystal and molecular structure, hydrogen bonds in organic crystals*

#### P.06.06.4

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#### Inclusion Compounds from a Decomposing Host: a Cautionary Tale

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5-Naphthalen-1-yl-5H-dibenzo[a,d]cyclohepten-5-ol (1) has been synthesised from bromo-naphthalene and 5H-Dibenzo[a,d]cyclohepten-5-one (2). Analysis (IR, NMR, TLC) showed the product to be pure. It was stored in a dark bottle in an inert atmosphere. However, when recrystallised from EtOH, we found that the crystal structure contained 1, 2 and a dimer of 2 (referred as 3). Upon recrystallisation from toluene, we obtained a crystal containing 1 and 2, while from hot DMSO we obtained different crystals of 1·DMSO and 3.

When recrystallised from either pyridine or 1,4-dioxane, we obtained crystals of the respective solvates, and we employed the dioxane solvate in order to purify the mixture of the contaminated host which contains 1 + 2 + 3.

Pure 1 was obtained by desorption of its dioxane inclusion compound, and allowed us to estimate that it is present in 59% quantity in the original mixture.

**Keywords:** *supramolecular assemblies, host-guest structures, inclusion complexes*

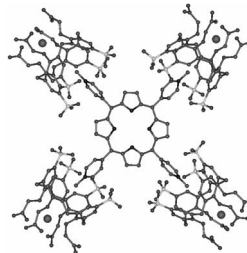
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#### Design and Crystallographic Characterization of Multi-Porphyrins Complexes

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The interactions of the tetracationic *meso*-tetrakis(4-N-methylpyridyl)porphyrin (H<sub>2</sub>T4) and its metallo-derivatives (MT4) with the octa-anionic form (at neutral pH) of 5,11,17,23-tetrakisulfonato-25,26,27,28-tetrakis(hydroxycarbonylmethoxy)calix[4]arene (C<sub>4</sub>TsTc) leads to a series of complex species whose stoichiometry and



porphyrin sequence can be easily tuned. X-ray solid state and solution studies suggest a picture where a central 1:4 (porphyrin to calixarene) unit (Figure) serves as template for obtaining more complex species. The latter arise by step-wise addition of porphyrin molecules above and below the plane of the 1:4 core, allowing to tune the species stoichiometry up to 7:4

[1]. Structural results, reported here strongly suggest that the interactions between the anionic calixarenes and cationic porphyrins are not significantly influenced by the presence of tetra-coordinated metals in the porphyrin core. Finally, an application of the homo-