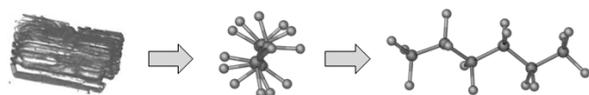


science [2]. We have used this interaction to realise the self-assembly of new families of liquid-crystalline materials based on perfluoroalkyl onium salts. An accurate choice of the starting components allowed us to produce superfluorinated ionic liquid crystalline materials with relatively high clearing points; some of those gave crystals of sufficiently good quality to be studied by single crystal X-ray diffraction. These compounds present an extensive disorder, mainly in the perfluorinated chains. In this contribution we present how to model the disorder, essential for a reasonable refinement of the structure, and which choices have driven our hands in selecting a good crystal for data collection. They are normally 'buttery' or at least very easily deformable, or extremely thin, or curved, twinned and often undergo phase transition at low temperatures.



[1] P. Metrangolo, G. Resnati, *Science* 2008, 321, 918. [2] P. Metrangolo, F. Meyer, T. Pilati, G. Resnati, G. Terraneo, *Angew. Chem. Int. Ed.* 2008, 47, 6114.

Keywords: Perfluoroalkyl Chains, Liquid Crystals, Disorder Modelling

FA4-MS31-P14

Synthesis and Crystal structure of Diethyltin bis-dihydrogenphosphate, (Et₂Sn)(H₂PO₄)₂ *Martin Reichelt^a, Hans Reuter^a,*
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The title compound was synthesized from Et₂SnO and H₃PO₄. Its colourless needle shaped crystals crystallise in the monoclinic space group C2/c (no. 15) with $a = 22.4786(8)$ Å, $b = 9.9382(3)$ Å, $c = 20.3812(7)$ Å, $\beta = 96.922(2)^\circ$, $V = 4519.9(3)$ Å³, $Z = 16$ and $d = 2.180$ g/cm³. For structure solution and refinement 82153 reflections in the θ -range 2.01°–28.00° were collected, resulting in 5450 unique reflections ($R_{int} = 0.0396$) after applying an empirical absorption correction ($T_{min} = 0.6360$, $T_{max} = 0.8309$). In summary 286 parameters (2 restraints) were refined to $R1 = 0.0212$, $wR2 = 0.0453$ for 5450 reflections with $I > 2\sigma(I)$ and $R1 = 0.0252$, $wR2 = 0.0465$ for all reflections.

The asymmetric unit consists of two formula units and contains {Et₂SnO₃} trigonal bipyramids and {PO₄} tetrahedra, which are connected with each other to rings and chains. Via hydrogen bonds an extended three-dimensional framework is built up with channels parallel to the crystallographic b axis filled by the organic groups.

Keywords: tin compounds, phosphates, organic-inorganic hybrid materials

FA4-MS31-P15

The crystal structure of α -sodium stearate NaC₁₈H₃₅O₂ · 0.5 H₂O. *Alexandra Christine Schaap, Walter Frank, Institut für Anorganische Chemie und*

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Sodium soaps are materials with a wide range of industrial applications for example as detergents, greases, lubricants and cosmetics. Hence, the knowledge of the structural behaviour of sodium soaps is of general interest. However, until today crystallography of neutral sodium soaps is restricted to the determination of unit cell parameters [1, 2, 3, 4, 5]. Here we present the results of the first crystal structure determination of a curd soap, α -sodium soap: NaC₁₈H₃₅O₂ · 0.5 H₂O (**1**). Extremely thin platelet crystals of **1** were obtained by slow cooling of a propylglycolic solution that contained 5% of water. Soap **1** crystallizes in the triclinic space group $P\bar{1}$ with unit cell parameters of $a = 7.847(2)$ Å, $b = 9.302(2)$ Å, $c = 51.100(10)$ Å, $\alpha = 91.68(3)^\circ$, $\beta = 92.46(3)^\circ$ and $\gamma = 90.11(3)^\circ$ and with $Z = 2$. The sodium soap can be described as a bilayered structure with a strict separation of hydrophobic and hydrophilic parts that can be considered as an arrangement of model membranes.

[1] Buerger, M.J., Smith L.B., de Bretteville, A., Ryer, F.V., *Proc. Natl. Acad. Sci. USA*, 1942, 28, 526. [2] Buerger, M.J., *Proc. Natl. Acad. Sci. USA*, 1942, 28, 529. [3] Buerger, M.J., Smith L.B., Ryer, F.V., Spike, J.E., *Proc. Natl. Acad. Sci. USA*, 1945, 31, 226. [4] Buerger, M.J., *Am. Mineral.*, 1945, 30, 551. [5] Minor, J.E., Lingafelter, E.C., *J. Am. Chem. Soc.* 1949, 71, 1145.

Keywords: soap, α -sodium stearate, crystal structure determination

FA4-MS31-P16

Engineering Metal Organic Frameworks with a Triazole-Tetrazole Ligand. *Bernard Tinant^a, Anil D. Naik^b, Mathieu Monaux^a, Yann Garcia^a, ^aInstitut de la Matière Condensée et des Nanosciences, Université Catholique de Louvain, Belgium*
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Cavernous solids at the designer's aspiration have found applications in separation science, gas storage technology, sensors, catalysis, drug delivery [1]. In continuation of our modular approach to functional materials of metal-organic frameworks, [2] we now direct our interest to the coordination chemistry of a novel asymmetric tecton (L1H = (5-(4H-1,2,4-triazol-yl)-2H-tetrazole), whose crystal structure was solved. This molecule was designed based on a conceivable transamination method [3] and actually represents the first molecular tecton wherein two flagged motifs of 1,2,4-triazole and tetrazole were brought on the same platform in a conciliation conformation. This unique molecule can behave as bifunctional bridging ligand as well as anion. [Zn(phen)₂(L1)]BF₄ (**1**) crystallises in a centrosymmetric space group (Pnma) with unsymmetrical L1 acting as 'interpolating' pillars and chelating phen as a capping ligand of hexa-coordinated Zn^{II} ions thus affording a robust 2D zig-zag network propagated by π - π interactions from heterocycles. The inclusion complex [Zn(bpy)₃]₂(L1) · (BF₄)₃ · 10H₂O (**2**) crystallises in a non-centrosymmetric space group (P2₁2₁2₁). It is a mononuclear octahedral complex presenting trapped L1 molecules in the water occupied channels by multiple secondary interactions also acting as an anion. TGA-DTA of **1**

shows exceptional thermal stability up to 200°C whereas **2** which is fully dehydrated at 100°C, begins to collapse after 110°C.

[1] Yaghi O., Li Q., *MRS Bulletin* 2009, 34, 682. [2] Naik A. D., Dürst M. M., Léonard A., Tinant B., Marchand-Brynaert, Su B. L., Garcia Y., *Cryst. Growth Des.* 2010, 10.1021/cg901473. [3] Naik A. D., Marchand-Brynaert J., Garcia Y., *Synthesis* 2008, 149.

Keywords: MOF, crystal engineering, supramolecular chemistry

FA4-MS31-P17

Zn₂(O₂CCF₃)₄ and Zn₇O₂(O₂CCF₃)₁₀ – Syntheses and Crystal Structures. Nina van Gellecom^a, Georgi Genchev^a, Walter Frank^a, ^a*Institut für Anorganische und Strukturchemie II, Heinrich-Heine Universität Düsseldorf, Germany*
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The first examples of zinc trifluoroacetates were reported in 1939 by F. Swarts [1]. These salts were found to be suitable precursors for the preparation of ZnO:F, which is of interest for the semiconductor research [2]. So far, only micro-crystalline samples or solvates of zinc trifluoroacetates have been described. Here we report on the syntheses and the crystal structures of the parent Zn₂(O₂CCF₃)₄ (**1**) and of Zn₇O₂(O₂CCF₃)₁₀ (**2**). Single-crystals of **1** have been obtained by the thermolysis of In(O₂CCF₃)₃ in the presence of elemental zinc at 240 °C under static vacuum. **1** crystallizes in the monoclinic space group *P*2₁/*c* with lattice parameters of *a* = 11.558(2) Å, *b* = 12.561(3) Å, *c* = 16.602(5) Å, and β = 134.121(17) ° with *Z* = 4. The structure is composed of two crystallographically independent Zn atoms that are linked by three trifluoroacetate groups. These fragments are connected by a further trifluoroacetate anion to form chains along [010]. Single-crystals of **2** have been prepared by the thermolysis of Ce(O₂CCF₃)₃ in the presence of zinc powder under reduced pressure at 320 °C. **2** crystallizes in the triclinic space group *P*1̄ with lattice parameters of *a* = 10.754(3) Å, *b* = 13.543(5) Å, *c* = 17.634(5) Å, α = 68.323(5) °, β = 89.772(5) ° and γ = 66.634(4) ° and with *Z* = 2. In the crystal of **2** the Zn₇O₂ fragments, which are composed of two edge-sharing Zn₄O tetrahedrons, are coordinated by ten trifluoroacetate groups. These fragments are linked by a bridging trifluoroacetate group to form chains along [001].

[1] Swarts F., *Bull. Soc. Chim. Belg.*, 1939, 48, 176. [2] Klingshirn C., *ChemPhysChem*, 2007, 8, 782.

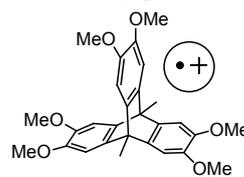
Keywords: zinc compounds, single-crystal X-ray diffraction, thermal behaviour

FA4-MS31-P18

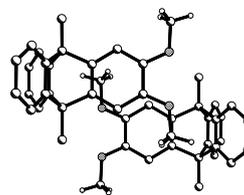
Novel 2D Assembly of Triptycene Cation Radicals vs. Traditional 1D Motifs Sergey V. Lindeman, Khushabu Thakur, Rajendra Rathore *Department of Chemistry, Marquette University, Wisconsin, USA*
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A series of triptycene derivatives with varying number of *o*-dimethoxy substituents along with their corresponding cation

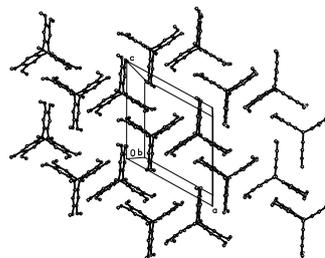
radical salts has been synthesized. The methoxy-substituted benzene rings possess a lower oxidation potential whereas the lengths of their C_{Ar}-O bonds provide a very sensitive indicator of the positive charge associated with the corresponding arene ring (i.e. 5.3 pm bond contraction per unit charge). The triptycene molecules were designed as a 3-



dimensional π-delocalized alternative to traditional planar 2-dimensional ion-radical substrates. Using highly precise low-temperature (100 K) X-ray structural data, the intra-molecular electronic interaction/delocalization was identified both in the neutral molecules and in the corresponding cation radicals. Specifically, C_{Ar}-O bonds in the neutral triptycenes are progressively elongated by up to 1.0 pm with increasing electron donicity of the molecules whereas C_{Ar}-O bonds in the corresponding cation radicals are additionally shortened by 2.0 pm (per unit charge) for interacting dimethoxy-substituted benzene rings. Importantly, both the neutral molecules and their cation-radical salts have a very uniform/conserved association mode of the dimethoxy-substituted arene rings in



crystals. They form close anti-parallel couples that are strengthened by multiple C-H...π hydrogen bonds and dipole-dipole interactions. These forces are further strengthened in the crystals of the cation radicals being assisted by favorable π-π interactions. The regular association of triptycene rings produces 2-dimensional layers with continuous electronic π-π delocalization throughout the plane of the layers via combination of intra- and inter-molecular charge resonance. This packing mode is principally different from the traditional



1-dimensional stacking of the planar aromatic ion radicals and hence opens new avenues in supramolecular design of the next-generation electronic materials for photovoltaic applications.

Keywords: supramolecular self-assembly, cation radical salts, charge resonance

FA4-MS31-P19

Synthetic Polymers in Biological Environment. Jindřich Hašek, Jan Dohnálek, Tereza Skálová, Jarmila Dušková, Andrea Štěpánková, Petr Kolenko *Institute of Macromolecular Chemistry, Academy of Science, 16206 Praha 6, Czech Republic*
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Many practical applications where synthetic polymers are in a direct contact with bio-macromolecules are known. Positive and also some negative consequences of polymer activity in