

Although from the same family, the two phosphates adopt two different crystal structures. $\text{Rb}_2\text{MnP}_2\text{O}_7 \cdot \text{H}_2\text{O}$ crystallizes in the monoclinic $P2_1/c$ space group with lattice parameters $a = 5.3845(5) \text{ \AA}$, $b = 10.241(1) \text{ \AA}$, $c = 15.566(2) \text{ \AA}$, $\beta = 93.15(1)^\circ$, $Z = 4$, $V = 857,00(4) \text{ \AA}^3$. $\text{Cs}_2\text{MnP}_2\text{O}_7$ crystallizes in the orthorhombic $Pnma$ ($n^\circ 62$) space group and the unit cell parameters are $a = 16.3398(3) \text{ \AA}$, $b = 5.3872(1) \text{ \AA}$, $c = 9.8872(2) \text{ \AA}$, $Z = 4$, $V = 870,33(3) \text{ \AA}^3$. Both structures are however closely related. They can be described by a single axis stacking of undulating $[\text{MnP}_2\text{O}_{10}]_\infty$ layers; alkaline cations occupy the interlayer spaces. The water molecule in $\text{Rb}_2\text{MnP}_2\text{O}_7 \cdot \text{H}_2\text{O}$ is positioned also between the $[\text{MnP}_2\text{O}_{10}]_\infty$ layers. Its presence is confirmed by the 1607cm^{-1} , 3173cm^{-1} and 3545cm^{-1} absorption bands observed in the infrared spectrum.

Some parallel can be made between the structures of $\text{Rb}_2\text{MnP}_2\text{O}_7 \cdot \text{H}_2\text{O}$ / $\text{Cs}_2\text{MnP}_2\text{O}_7$ [1] and some phosphates of the enlarged $\text{A}_2\text{MP}_2\text{O}_7$ family like $\text{K}_2\text{CuP}_2\text{O}_7$ [2]. On the other hand, within the entire $\text{A}_2\text{MnP}_2\text{O}_7$ ($A = \text{Li, Na, K, Rb, Cs}$) family itself, every phosphate adopts a different crystal structure with either layered or 3D $[\text{MnP}_2\text{O}_7]$ framework and various Mn coordination [1, 3 - 5].

[1] S. Kaoua, S. Péchev, S. Krimi, P. Gravereau, J.-P. Chaminade^a, A. El Jazouli (submitted). [2] A. ElMaadi, A. Boukhari, E.M. Holt, *Journal of Alloys and Compounds* **1995**, 223, 13. [3] L. Adam, A. Guesdon, B. Raveau, *J. Solid State Chem.* **2008**, 181, 3110-3115. [4] A. ElMaadi, A. Boukhari, E.M. Holt and S. Flandrois, *CR.Acad. Sci. Paris, Set. II*, **1994**, 318,765. [5] N.K.Lokanath, M.A. Sridhar, J. Shashidara Prasad, G.S. Gopalakrishnan, K.G. Ashamanjari, *Bulletin of Materials Science (Bangalore)* **2000**, 23(3), 175-178

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Three-dimensional lanthanide MOF based on a linear ditopic dicarboxylate

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Since its first appearance, the MOF-type materials (Metal-Organic Frameworks) has shown a promising future into the materials science, due to their big applications in all fields, from structural materials (giving light and resilient structures), to their magnetic properties (opening a new field of research in the information store devices.)

For the last ten years, our research group has centered its attention upon the field of MOF-type materials, with special interest into the magnetic properties. In the preparation of these materials our strategy has been based on carboxylic ligands, using transition metal and lanthanide ions as paramagnetic centres and organic molecules containing this substituent as ligands.

We have selected the *trans,trans*-muconic acid (**Figure 1**, H_2ttmuc) because it is a linear, ditopic ligand with a rigid rod-like configuration. This ligand has two connection sites at both extremes, giving as a result a ligand which can act as edges in three-dimensional MOFs where the formation of cavities is favoured. The muconic acid is a dicarboxylic acid. The double unsaturated muconic acid has three isomeric forms, designated *trans,trans*-muconic acid, *cis,trans*-muconic acid, and *cis,cis*-muconic acid, which differ by the geometry around the double bonds.

Previous results of this ligand include the preparation of transition metal complexes, such as Fe[1], Cu[1], Ni[2] and Co[3], to form networks of different dimensionalities. However, the use of lanthanides remains unexplored, and thus here in we present the preliminary results

on the crystal structure of the praseodymium(III) compound (**Figures 2a & 2b**), that show that the MOF formed is three-dimensional with cavities where water molecules are hosted.

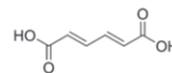


Figure 1. *trans,trans*-Muconic Acid (H_2ttmuc)

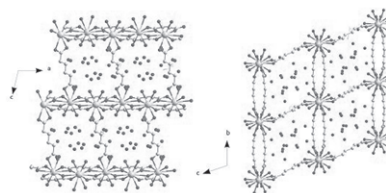


Figure 2a & 2b. Crystalline structure of *Pr [tt-muc]*. a, c

[1] C.Serre, F.Millange, S.Surble, G.Ferey, *Angew.Chem.,Int.Ed.* **2004**, 43, 6286. [2] M.H.Mir, Li Wang, Ming Wah Wong, J.J.Vittal; *Chem.Comm.* **2009**, 4539. [3] K.Yoneda, M.Ohba, T.Shiga, H.Oshio, S.Kitagawa; *Chem.Lett.* **2007**, 36, 1184.

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Formation of LC-phase in concentrated solutions of mono-carboxylic acids by SANS

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Mono-carboxylic acids (bent non-saturated oleic acid (OA) and strictly linear saturated stearic (SA) and myristic (MA) acids) show different efficiency in stabilization of ferrofluids, in particular, regarding nano-magnetite in organic non-polar carriers [1]. The difference can be related to the interaction between acid molecules in organic non-polar solutions added to stabilized ferrofluids at the preparation stage. To clarify this point we study the concentration effects in pure solutions of OA, MA, SA based on deuterated benzene and decalin by means of small-angle neutron scattering (SANS). The behavior of the studied acids in the two solvents is quite similar. The increase in the concentration of MA and SA results in vigorous growth of solution viscosity followed by the formation of a new jelly-like phase. We associate this effect with the transition into the liquid crystalline (LC) state, which is well-known in the thermodynamics of stiff rod-like polymers. At the same time, such transition is not observed in the OA solutions.

The characteristic size of the acid molecules (2 nm) is close to the resolution limit of the SANS method. This means that only the Guinier region of the scattering curves can be reliably treated in the case of diluted isotropic solutions [2]. The important feature of the used analysis is that the dependence of the forward scattering intensity on the solute concentration is determined only by the isotropic part of the particle interaction potential [3]. The excluded volume repulsion is found to dominate over the attraction (presumably van der Waals forces) for OA and MA. In its turn, for SA the attractive component is higher than the repulsive one. This results in a shift of the transition into the liquid

crystalline state towards smaller concentrations as compared to the MA case. At some concentration of SA/MA a partial aggregation (regions with liquid crystals of acid molecules in the solution) is observed as a deviation of the scattering curves from the Guinier law at the smallest q -values. As the concentration increases, the SANS signal from these aggregates becomes rather distinguishable, and the corresponding characteristic size can be roughly estimated from an additional Guinier-type term as ~ 10 nm. The further increase in SA/MA concentration leads to the alignment of these aggregates (transition to a smectic phase), which is reflected in the appearance of the diffraction peak at $q \sim 2 \text{ nm}^{-1}$ (corresponding correlation length ~ 3.2 nm). The position of the peak shifts to higher q -values (smaller distance between aggregates) with an increase in the acid concentration. The possibilities of the wide-angle diffraction for the study of the observed liquid crystalline phase in solutions of linear molecules with comparatively short alkane chains are considered.

The formation of the found LC-phase in bulk solutions of mono-carboxylic acids is an important factor, which influences the stabilization efficiency of the studied acids in colloidal solutions of magnetic nanoparticles.

[1] M.V. Avdeev, D. Bica, L. Vekas, V.L. Aksenov, A.V. Feoktystov, O. Marinica, L. Rosta, V.M. Garamus, R. Willumeit *J. Coll. Inter. Sci.* **2009**, *334*, 37-41. [2] V.I. Petrenko, M.V. Avdeev, L. Almasy, L.A. Bulavin, V.L. Aksenov, L. Rosta, V.M. Garamus *J. Col. Surf. A* **2009**, *337*, 91-95. [3] T. Shimada, M. Doi, K. Okano *J. Chem. Phys.* **1988**, *88*, 2815-2821.

Keywords: SANS, surfactant_solutions, liquid_crystalline_phase

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Nanostructures in a supramolecular side-chain liquid crystalline block copolymer

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Hydrogen-bonding amphiphilic nonmesogen 4'-(3,4,5-trioctyloxybenzoyloxy)benzoic acid (TOB), of a wedge shape, is bound to P4VP block for supramolecular side-chain liquid crystalline blocks in polystyrene-*block*-poly(4-vinylpyridine) (PS-*b*-P4VP). We have demonstrated that rich phase transitions of hierarchical structures can be manipulated by adjusting the mesomorphic ordering of P4VP(TOB)_x block via the TOB grafting fraction ratio, x , in the PS-*b*-P4VP(TOB)_x thin films under solvent-casting and stretch-annealing treatments. Using synchrotron-based small- and wide- angle X-ray scattering and transmission electron microscopy (SAXS, WAXS and TEM), we observed global phase transitions of the PS-*b*-P4VP(TOB)_x compound from lamellar (LAM), hexagonal-packed cylinder (HC), undulating hexagonal-packed cylinder (UHC), face-centered cubic sphere (FCC), to tetragonally perforated layer (TPL) structures, upon increasing the TOB content for an increasingly ordered mesomorphic phase of the hydrogen-bonded complex of P4VP(TOB)_x, from nematic (N), smectic A (SmA), hexagonal columnar disordered (Col_{hd}), to hexagonal columnar ordered (Col_{ho}) phases. The mechanism of stretch-induced phase transition from FCC to TPL in the PS-*b*-P4VP(TOB)_{0.7} thin film was also investigated by simultaneous SAXS and in-situ stretching mechanical measurement. When the SAXS patterns from the stretching film of PS-*b*-P4VP(TOB)_{0.7} were examined, we found that the FCC spheres become oblate ellipsoids by the initial deformation and than merging of each oblate ellipsoids along $\langle 110 \rangle$ direction of FCC form TPL mesophase by thermal annealing. The TPL, a kinetic trapped phase, is directed by strong local ordering columns of TOB with π - π interactions at a sufficient grafting density. The FCC-to-TPL phase

transition is described for the first time in block copolymers.

[1] W.T. Chuang, H.S. Sheu, U.S. Jeng, H.H. Wu, P.D. Hong, J.J. Lee *Chem. Mater.* **2009**, *21*, 975-978.

Keywords: SAXS, polymer, superstructure

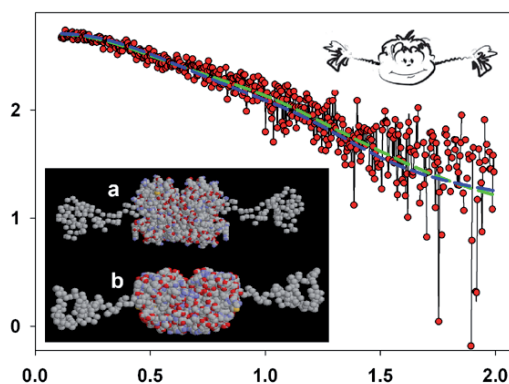
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Virus matrix protein M1: SAXS data analysis and modeling

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Structural analysis of the influenza A virus full-length matrix M1 protein was performed using small-angle X-ray scattering (SAXS). The structure of the M1 protein macromolecules in solution was for the first time reconstructed using advanced methods of SAXS data analysis and interpretation [1-6]. The detailed analysis of the scattering data and modeling revealed a structurally polarized molecule with a compact NM-fragment and weakly ordered C-terminal domain. These structural peculiarities explain the ability of the matrix M1 protein to mediate the multistep process of cell infection due to flexibility of the C-terminal regions.



[1] L.A. Feigin, D.I. Svergun, *Plenum Press*, New York, **1987**. [2] P.V. Konarev, V.V. Volkov, A.V. Sokolova, M.H.J. Koch, D.I. Svergun, *J. Appl. Crystallogr.* **2003**, *36*, 1277-1282. [3] D.I. Svergun, *J. Appl. Crystallogr.* **1992**, *25*, 495. [4] D.I. Svergun, *Biophys. J.* **1999**, *76*, 2879-2886. [5] M.V. Petoukhov, D.I. Svergun, *Biophys. J.* **2005**, *89*, 1237-1250. [6] D.I. Svergun, C. Barberato, M.H.J. Koch, *J. Appl. Cryst.* **1995**, *28*, 768-773.

Keywords: protein, modelling, structure

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SAXS study of phase separation process in the SiO₂-SnO₂ nanostructured materials

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