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**Synthesis and structural study of photoluminescent lanthanide-organic frameworks**

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During the last few decades, worldwide researchers have been devoting significant efforts to the design, synthesis and characterization of new multi-dimensional Coordination Polymers, also known as Metal-Organic Frameworks (MOFs). These compounds have been extensively studied for gas storage and separation, ion exchange, in optical sensors, catalysis, among several others [1].

Our research group has been interested in the construction of Lanthanide-Organic Frameworks (LnOFs) using phosphonate ligands and various lanthanide centers [2]. In this communication we report the synthesis and structural characterization of micro- and nano-sized LnOFs obtained using distinct synthetic approaches (hydrothermal, ultrasonic- and microwave-assisted synthesis). Crystalline and phase-pure [Ln(Hpmd)(H$_2$O)]$_2$ materials [where Ln$^{3+}$ = Eu$^{3+}$ (1), Tb$^{3+}$ (2), (Tb$_3$b$_2$En)$_{10}$$^{3+}$ (3); H$_2$hpmd = 1,4-phenilenbis(methylene)diphosphonic acid] were fully studied and characterized by standard solid-state techniques and their structures unveiled by powder X-ray diffraction.

All prepared LnOFs exhibit similar structural features to the previously reported [Ce(Hpmd)(H$_2$O)] and [Pr(Hpmd)(H$_2$O)] compounds [3]. Compound I has a single Eu$^{3+}$ site coordinated to one water molecule and to five phosphate groups, overall describing a {EuO$_7$} seven-coordination environment resembling a significantly distorted monocapped trigonal prism. The two independent phosphonate moieties promote the formation of lanthanide dimers with Eu–Eu intermetallic distances of 4.05 Å. This terminal group also connects adjacent dimers along the [010] direction of the unit cell, leading to the formation of a 1D zigzag array. Bridges between this array are ensured by strong O–H∙∙∙O hydrogen bonds involving the hydrogenophosphate and the coordinated water molecule. This arrangement of supramolecular interactions promotes structural cohesion within the inorganic lanthanide (hydrogeno)phosphonate layer. Connections between adjacent layers are ensured by the Hpmd$^-$ organic linkers leading to an alternation along the c-axis of organic and inorganic fragments. All compounds exhibit intense photoluminescence under UV excitation.

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**Synthesis, crystal structure and thermal decomposition of NaSc[PO$_4$]$_2$[HPO$_4$(OH)$_2$]$_n$$^{2n}$**

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Na$_2$Sc[PO$_4$]$_2$[HPO$_4$(OH)$_2$]$_n$ was prepared by use of a phosphorous acid flux route (H$_2$PO$_3$, mp: 73.6 °C). [1] The crystal structure was determined from single-crystal X-ray diffraction data: triclinic, space group P1 (No. 2), $a = 7.4507(1)$ Å, $b = 9.6253(17)$ Å, $c = 9.6141(16)$ Å, $\alpha = 115.798(4)$°, $\beta = 101.395(4)$°, $\gamma = 101.136(3)$°, $V = 577.29(16)$ Å$^3$ and $Z = 1$. The crystal structure of Na$_2$Sc[PO$_4$]$_2$[HPO$_4$(OH)$_2$]$_n$ contains two kinds of phosphate(III) groups: HPO$_4$$^{2-}$ and HPO$_4$(OH)$_2$$^{-}$. 

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