done to introduce carriers, holes or electrons, into these layers by chemical substitutions on the cations sites [6-7]. Some authors claimed the presence of superconductivity around 20 K, however this result was not clearly confirmed by other works. We report here on the synthesis of the compounds (Ba,Sr)\(_2\)Cu\(_4\)O\(_7\)-Cl\(_4\) and (Ba,Sr)Cu\(_4\)O\(_7\)-Cl\(_4\) in air and under oxygen flux, where the hole doping is respectively achieved by chemical substitution and copper site deficiency. Their structures are resolved by powder x-ray diffraction using the Rietveld method. The variations of the unit-cell parameters and the inter-atomic distances versus x and y are discussed according to the formal charge distributions in the structure [8]. Moreover, the averaged ionic sizes are also invoked to interpret the stability range of such phases.

Our study deals with the structural and micro-structural characterisations of the solid solution LiTi\(_2\)-Sn\(_4\)(PO\(_4\))\(_3\) using the powder X-ray diffraction and the Rietved refinements. A comparative study is made with the Na-analog phases [5-6] and a global approach is proposed to explain the structural features of the Nasicon phases, that is, the origin of unusual evolutions of the unit-cell constants.

**Keywords:** nasicon; crystal structures; powder diffraction analysis

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**Structure and Microstructure of the Solid Solution LiTi\(_2\)-Sn\(_4\)(PO\(_4\))\(_3\) and the Origin of Unusual Evolutions of the Unit-cell Constants of the Nasicon Structures.** Nedjemeddine Bounar, Abderrahim Benabbas, *L.I.M.E Laboratory. University Of Jijel. B.P. 98 Ouled-Aissa 18000 Jijel. Algeria. E-mail: nedjmibounar@yahoo.fr

Nasicon [1] compound have been extensively investigated owing to their interesting physical properties with potential applications as fast ion conductors and low thermal expansions ceramics [2-4]. The Nasicon structure is built up of corner-sharing BO\(_6\) octahedra and PO\(_4\) tetrahedra leading to a frame-work of B\(_2\)P\(_2\)O\(_7\) formulas with interconnected channels where cations can be inserted in two types of sites usually noted M\(_1\) at (0,0,0) and M\(_2\) at (2/3,0,1/4). The great flexibility of this structure allows large chemical substitutions and makes it possible that the sites M\(_1\) and M\(_2\) may be empty as in Nb\(_2\)(PO\(_4\))\(_3\) partially occupied or completely full as in Na\(_2\)Zr\(_2\)(SiO\(_4\))\(_3\). The ionic transport in these compounds is due to the migration of the cations through the cavities in the interconnected channels where an order-disorder phenomenon is possible in the partially filled cases. On the other hand, the thermal behaviour is related to the variation of lattice parameters vs. temperature generally variable in opposite senses. Thus, the detailed structural characterisation particularly of the atomic positions and the bond lengths is essential to understand the underlying mechanisms and to optimise such properties.

Metal phosphonate chemistry is of great interest in material science due to their potential applications in many fields such as ion-exchange, catalysis, and sensor devices. Often, metal phosphonates exhibit polymeric structures consisting of chains, layers, or three-dimensional networks, but monomeric structures are also known. In particular, the tunable organic units in diphenosphate [R(PO\(_3\))\(_2\)] allow the construction of metal phosphate materials with new architectures. We have recently reported the crystal structure of [H\(_3\)N(CH\(_2\))\(_4\)NH\(_3\)]La[hedph][hedph\(_2\)]. The crystal structure of this compound, which crystallize in space group P-1, is built up from one-dimensional covalent lanthanum phosphate cross-linked chains and [NH\(_3\)(CH\(_2\))\(_4\)NH\(_3\)] cations [1,2]. Following with the work previously developed [3] we presented here single crystal data of new members of this family, and new structures solved by combination of the known single crystal data and the powder data of the new structures. The powder data were measured in ESRF Synchrotron (Spline). It has been carried out a Rietveld refinement using FullProf with the heavy atoms being the geometry of the remaining H-atoms completed using Mercury. The final structural results show the isostructurality of all compounds, the differences on the intra and intermolecular network will be presented and discussed.