

**Experiment title:**

Thermostructural properties of a new vanadate, $\text{Ca}_{9.5}\text{Mn}(\text{VO}_4)_7$: thermal expansion and search for substitutional disorder of manganese

Experiment number:

MA-5010

Beamline:

ID22

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Local contact(s): FITCH Andrew

Tel: +33(0)4 76 88 25 32, 25-32, 19-92

Email: fitch@esrf.fr

Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

Houri S Rahimi Mosafer, PhD student at Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

Wojciech Paszkowicz, **Professor** at Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

Roman Minikayev, **PhD** at Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

Report:

The goal of experiment was investigation of thermal expansion coefficients and the structural properties of new calcium orthovanadates which is doped by transition metal and to check if there is any order-disorder transition at high temperature.

Calcium orthovanadate ($\text{Ca}_3(\text{VO}_4)_2$), are known to crystallize in $R3c$ space group. A small fraction (typically up to $\sim 10\%$) of Ca atoms can be replaced by other ones, of valences from +1 to +4, without a change of structure. The substitution is site-selective. Namely, the corresponding structural modification consists in a partial or full replacement of Ca atoms at one (case of substitutionally ordered compound, e.g. for the transition metal doping,) or more of Ca sites (disordered compound case, e.g. for lanthanide doping).

We used the multianalyzer detector option and studied the transition metal doped samples at a series of temperatures with excellent statistics and resolution. The applied mode of measurement provided data allowing for precise Lattice parameter determination and thus for understanding subtle differences, among variously doped samples, between the derived thermal expansion coefficients behavior as function of temperature. Other details of the crystal structure could be understood, as well, in particular the variation of site occupations of the M1-M5 sites high temperatures. the obtained data allow for analysing the type of substitutional disorder at the specific Ca/TM sites.

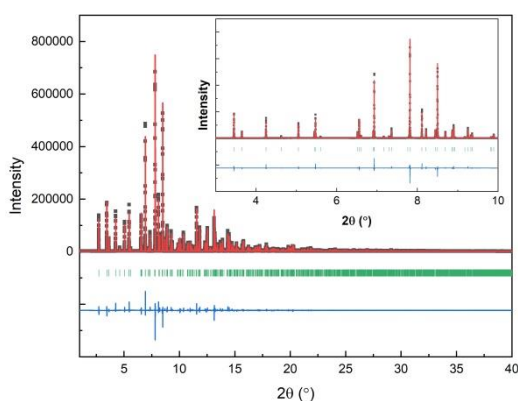


Figure 1 Rietveld refinement of $\text{Ca}_{9.5}\text{Mn}(\text{VO}_4)_7$ at room temperature, the inset illustrates the excellent resolution constituting a critical factor allowing for refinement of structural's details