# Experimental report

29/05/2020

**Proposal:** 5-31-2674 **Council:** 4/2019

**Title:** Crystal and magnetic structures ofA-site manganites with B = Te, Ta and Nb with perovskite and corundumrelated structures.

**Research area:** Physics

**This proposal is a new proposal**


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**Samples:** Mn4Nb2O9-II
Mn3TeO6-II
Mn11Ta4O21
Mn3-xCoxTeO6

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D20 | 3 | 3 | 20/09/2019 | 23/09/2019 |


## Abstract:

ABO3 oxides are intensively studied materials as they exhibit useful chemical and physical properties. Amongst these, the use of high pressure synthesis techniques has shown that the small Mn2+ cation can be located in the A-site with several structures in competition. This study is on three different structures with Mn2+ in the A-site. The goal is to determine their magnetic structures and to compare the different structural factors that rule over the magnetic interactions in these exotic materials.

**Crystal and magnetic structures of A-site manganites with B = Te, Ta and Nb with perovskite and corundum related structures.**

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$ABO_3$ oxides are intensively studied materials as they exhibit useful chemical and physical properties. Amongst these, the use of high pressure synthesis techniques has shown that the small $Mn^{2+}$ cation can be located in the A-site with several structures in competition. For instance, the spintronic perovskite $MnVO_3$-II or the multiferroic $MnTiO_3$-II in the $LiNbO_3$-type. [1,2] More recently, $Mn_3WO_6$ has shown multiferroic properties along with an incommensurate magnetic order, therefore the study of the magnetic structures is important in order to understand the coupling between the different ferroic orders.[3]

In this experiment, two high pressure and one ambient pressure A-site manganites with B = Te, Ta and Nb were proposed as potential multiferroics. Among them, the ambient pressure sample $Mn_{11}Ta_4O_{21}$ and the high pressure $Mn_3TeO_6$ had been previously registered and measured on the D1B experiment 5-31-2624. The collected data, detailed in the experimental report for the indicated experiment allowed the accurate determination of both nuclear and magnetic structures and their thermal evolution. The results for $Mn_3TeO_6$ were reported in *Chem. Comm.* 2019, **55**, 14470-14473 (doi: 10.1039/c9cc07733b) and those for $Mn_{11}Ta_4O_{21}$ are currently under review in *Inorganic Chemistry*.

About 70 mg of $Mn_3NbO_6$ were measured on D20 using a 3 mm V-foil can combining several high pressure products together. Long scans were collected at 5 and 80 K using $\lambda = 2.41$ Å. Additional short scans were taken every 0.1 K at intermediate temperatures. The resulting features of a complex modulated structure motivated the collection of additional 100 K pattern in the highest resolution mode, using the 90˚ take off angle and $\lambda = 1.54$ Å. The results, in combination with synchrotron data, allowed the complete structural and magnetic characterisation of this sample, with a complex magnetic thermal evolution (Fig.1). The publication of these results is currently under preparation.

Additional related samples with general formula $Mn_{3-x}Co_xTeO_6$, from the accepted proposal 5-31-2731, were registered for this experiment and different temperature data sets were collected for each of them using $\lambda = 2.41$ Å and 42˚ take off angle as detailed below. In all cases, small amounts of sample between 50 and 120 mg combining several high pressure products together were scanned using a 3 mm V-foil can.

- x = 0.5: 2 h scans collected at 1.5, 35 and 100 K with additional short scans collected every 0.1 K between 1.5 and 35 K.
- x = 1: 1.5 h scans were collected at 1.5 and 50 K and additional short scans every 0.1 K.
- x = 1.5: 2.5 h scans were collected at 1.5, 40 and 80 K with additional short scans taken every 0.1 K.
- x = 2.5: 2 h scans were collected at 1.5, 35 and 90 K with additional short scans every 0.1 K.
- x = 3: 1 h and 45 min scans collected at 1.5, 40 and 80 K with additional short scans every 0.1 K.

The results confirm that the $Mn_{3-x}Co_xTeO_6$ solid solution shows a structural evolution from the double perovskite structure of $Mn_3TeO_6$[4] ($x < 1.5$) to the $Ni_3TeO_6$ related superstructure of $Mn_3NbO_6$ (for $x > 1.5$). $x = 1.5$ shows a phase coexistence between both polymorphs. The $x = 2$ member of the series was left to be measured during experiment 5-31-2731. The magnetic structures of the DPv polymorphs show a coherent evolution from the AFM $k = [½\ 0\ ½]$ of $Mn_3TeO_6$[4] to $k = [0\ 0\ 0]$ with spins confined to the ac plane in all cases (Fig. 2). Among the ordered corundum related structures, complex magnetic behaviours are observed, including incommensurate T-dependent propagation vectors similar to those observed for $Mn_3NbO_6$ in Fig. 1.

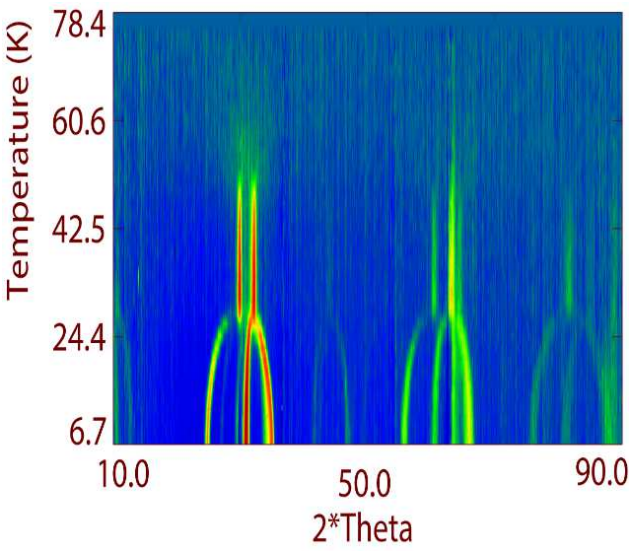


Fig. 1. 2D plot showing the complex thermal evolution of the $Mn_3NbO_6$ ordered corundum related high pressure phase.

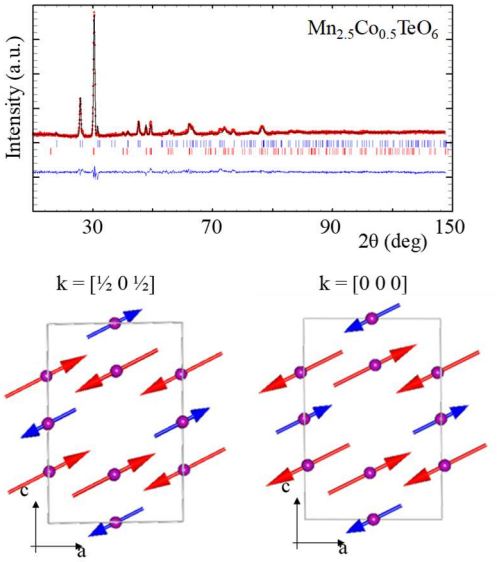


Fig. 2. Magnetic structures of the high pressure double perovskite polymorphs of $Mn_{3-x}Co_xTeO_6$.

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[1] M. Markkula, *et al. Phys. Rev. B.* **84**, 094450, 2011.
[2] A. M. Arevalo-Lopez and J. P. Attfield. *Phys. Rev. B.* **88**, 104416, 2013.
[3] M-R. Li *et al. Nat. Comm.* **8**, 2037, 2017.
[4] A. M. Arevalo-Lopez *et al. Chem. Comm.* **55**, 14470-14473, 2019.