A Combined Raman-X-ray Powder Diffraction Study at Non-ambient Condition

Marco Milanesio[a], Enrico Boccaleri[a], Gianluca Croce[a], Leonardo Marchese[a], Davide Viterbo[a], Wouter van Beek[b], Hermann Emerich[b], [a]*DISTA, Università del Piemonte Orientale, Alessandria, Italy.* [b]*SNBL/ESRF Grenoble, France.* E-mail: marco.milanesio@mfn.unipmn.it.

Raman spectroscopy can often help to overcome the well-known limitations of X-Ray Powder Diffraction (XRPD) alone by providing additional information on samples containing light elements and/or disordered moieties and/or amorphous phases. We designed and carried out a combined Raman-XRPD *in situ* experiment to exploit the complementarities of the two techniques in investigating the kinetics of a transformation occurring in the solid state at non-ambient conditions. The experimental setup was tested on a simple solid-state to solid-state transformation, consisting in a phase transition occurring at 330K to octakis(isobutyl)-octasilsesquioxane. The crystal structure of the RT phase was solved by single crystal X-Ray diffraction [1], whereas the high temperature (HT) phase was only recently discovered by some of us. The *ab initio* solution of the high T crystal structure was hampered by the poorness of the 330 K XRPD pattern. Monitoring the transformation simultaneously using the Raman technique combined to XRPD allowed a full structural characterization of the HT phase and in particular:

i) to understand that the phase transition was strongly correlated to the features of the isobutyl moieties;

ii) to monitor the possible radiation damage;

iii) to carefully check the temperature of the system (exploiting the Stokes/anti-Stokes ratio of the Raman bands).

[1] Bassindale, et al., *Dalton Trans.*, 2003, 2945.

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