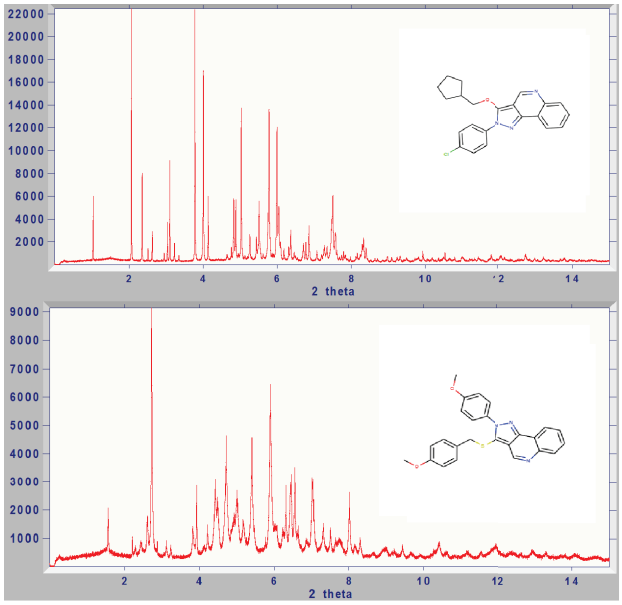

Figure 1. High resolution powder diffraction data of (a) crystalline and (b) poorly crystalline organic materials collected using ESRF beamline ID22.

Keywords: crystallization, prediction, powder diffraction, microstructure, machine learning

MS41-P7 Structural characterization of Re-substituted lanthanum tungstates $La_{5.4}W_{1-x}Re_xO_{12-\delta}$ ($0 \le x \le 0.2$) with Neutron and X-Ray Diffraction

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The presented work on proton conducting materials will show new results on the structural characterization of a substituted lanthanum tungstate: $La_{5.4}W_{1-x}Re_xO_{12-\delta}$ with $0 \le x \le 0.2$ in two different conditions (dry-Ar, wet-D2O). Among the eight specimens measured, the one without substitution ($La_{5.4}WO_{12-\delta}$) and the other with highest Re-substitution level ($x=0.2$) will be in the focus of the presentation and compared throughout the whole talk.

Their comparison will be achieved considering Neutron Diffraction results, due to the insufficient contrast between W (Z=74, b=4.86fm) and Re (Z=75, b=9.2fm) against X-Rays. The structural model of the undoped system $La_{5.4}WO_{12-\delta}$, as suggested recently by a coauthor [1] will be independently confirmed and taken as starting point to determine the position of Re-atoms in the crystal structure.

Neutron Diffraction data was obtained from D2B (ILL, Grenoble) and HRPT (SINQ, Villigen). At D2B, High-Resolution mode was employed for the two above-mentioned samples, measuring many temperature steps with a Displex-device (5K – 200 K range). At SINQ, 1.5K was reached in High-Intensity mode, using their peculiar orange cryostat ORI4.

Three models will be finally presented for the Re-substituted system within the Fm-3m space group: Re substituting W on the main W position (Wyckoff site 4a), Re substituting W on the shared La/W position (Wyckoff site 48h) and Re substituting W statistically on both sites. As low temperature minimizes thermal vibrations, structural modeling may exclude positional disorder contribution. Substitution amount in the shared sites is reached refining occupancies in a single-atom-per-site approach (average neutron scattering length). Results match with composition obtained from Electron-Micro-Probe-Analysis.

Due to the low Re amount (~1 atom per unit cell out of 32 cations and 55 oxygen anions) and the close neutron scattering length of Re to the main element La (Z=57, b=8.24fm) it is hard to determine unambiguously the very details of the structure. Finally, special importance is given to the refined oxygen occupancies in wet and dry condition and compared to thermo-gravimetric results.

[1] T. Scherb, PhD Thesis. Technische Universität Berlin, 2011, doi: http://dx.doi.org/10.5442/d0014.

Keywords: Proton Conductors, Lanthanum Tungstates, Neutron Diffraction