isotope is concerned and deuteration is unfeasible (true for many materials, e.g. hydrogen storage materials, proton conductors, ferroelectrics, etc.). Improvements in neutron and detector technology have changed this long-standing view and a range of hydrogenous materials have now been characterised successfully with PND. [1-6] There are limitations though, naturally dependent on the ^{1}H content, complexity and thermal motion of the material under study. The general reduction of information inherent in PND data (from both the nature of the powder data and the often high incoherent background contribution from hydrogen-containing materials) can be partially overcome by the inclusion of single crystal X-ray diffraction (SXD) data in joint PND and SXD refinements. Also, imaging proton densities from difference Fourier maps is an old but equally powerful tool to obtain qualitative information about proton behaviour. We intend to show what appears possible today, but also point out the limitations we have found on the basis of recent datasets collected at the high intensity powder diffractometer D20 at ILL, Grenoble, and the recently upgraded HRPD at ISIS, UK. The materials studied include both inorganic and organic, and vary in ^{1}H content, complexity and data collection temperature.

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**Keywords: powder neutron diffraction; hydrogen compounds; incoherent scattering**

### FA5-MS01-O10

**Crystal Structure and Phase Transitions of Gd(CO₃)OH Studied by Synchrotron PXRD.** Hwo-Shuenn Sheu[a], Wei-Ju Shih[a], Wei-Tsung Chuang[a], I-Fang Li[b], Chen-Sheng Yeh[b]. *[a]National Synchrotron Radiation Research Center, Hsinchu 300, Taiwan. [b]Department of Chemistry, National Cheng Kung University, Tainan 701, Taiwan.*
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The crystal structure of $Gd(CO_3)OH$ was solved using synchrotron powder X-ray diffraction (PXRD) (see attached Figure). $Gd(CO_3)OH$ has been known to exist in the form $Gd_2O(CO_3)_2 \cdot H_2O$ and its powder pattern is listed in JCPDF (#430604) for decades but the crystal structure has not yet been elucidated. The crystal structure is solved by simulated annealing method using the DASH program. The final Rietveld refinement converged to $R_{wp}$ = 6.28%, $R_p$ = 4.47% and $\chi^2$ = 1.348, using the GSAS program. $Gd(CO_3)OH$ crystallized in an orthorhombic crystal system with lattice constants a = 7.08109(9), b = 4.88436(7), c = 8.45010(13) Å, and space group *P nma*. $Gd(CO_3)OH$ forms a three-dimensional framework structure with an eight member-ring, one-dimensional channel and an OH anion in the cavity. XANES of Gd $L_{III}$–edge suggests that the oxidation

state of Gd is 3+. Two phase transitions of $Gd(CO_3)OH$ were found at 500 and 650 °C to yield $Gd_2O_2CO_3$ and $Gd_2O_3$ respectively.
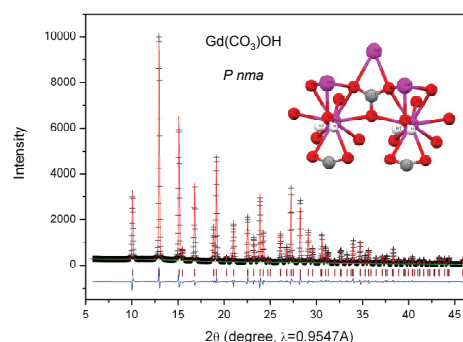


**Keywords: structure determination; phase transition; rare-earth compounds**