SNBL	**Experiment title:** Structural characterisation of intermetallic compounds and metal hydrides by powder diffraction.	**Experiment number**: 01-01-274
Beamline: BM01B	**Date of experiment**: from: 16-sept-01 to: 18-sept-01 8-oct-01 10-oct-01	**Date of report**: 28-march-02
Shifts: 12	**Local contact(s)**: Wouter VAN BEEK	*Received at UNIL:*

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About nine powder patterns (samples from projects INTAS and FNS) were succesfully acquired during 12 shifts (9 resp. 3 shifts from projects 01-01-274 resp. 01-01-232). Here we give a short report on succesfully analyzed patterns and refined crystal structures.

Compound	space group	lattice parameters [Å]	impurities
$Mg_xTa_yH_z$	?	a=5.066, b=9.043, c=4.737	α-MgH_2, MgO, unknown imp.

Experimental conditions: λ=0.48562 Å, 1.53°≤2θ≤35.485°, step size 0.005°. New ternary hydride $Mg_xTa_yH_z$ of light-brown colour was studied. Preliminary calculations showed that the ternary hydride has an orthorhombic symmetry. However, multiphase character of the sample has not yet allowed to solve the structure.

Compound	space group	lattice parameters [Å]	impurities
$CeMn_{1.8}Al_{0.2}D_{3.5}$	$P6_3/mmc$	a=5.95060(12), c=9.7679(3)	CeO_{2-x}, CeO_{1-x}, β-Mn

Experimental conditions: λ=0.48562 Å, 3°≤2θ≤33.5°, step size 0.002°. The structure refinement performed by the Rietveld method (R_B=0.029, R_p=0.085, R_{wp}=0.098, χ^2=6.98) gave precise occupational parameters for Al and Mn atoms, distributed over two crystallographic positions (2*a* and 6*h*). Obtained results confirmed an assumption about local metal atom exchange during low(room)-temperature hydrogenation of $CeMn_{1.8}Al_{0.2}$ alloy, that we found recently from neutron powder diffraction experiments.

Compound	space group	lattice parameters [Å]	impurities
$Ca_4Mg_4Fe_3D_{22}$	*P*-43*m*	*a*=6.7016(4)	MgD_2

Experimental conditions: λ=0.48562 Å, 0.512°≤2θ≤35.49°, step size 0.002°. The purpose of this experiment was to investigate to what precision synchrotron X-rays are capable of locating hydrogen(deuterium) atoms in polycrystalline samples containing heavy elements. The data are currently used for Rietveld refinement to verify the crystallinity of the phase of interest.

Compound	space group	lattice parameters [Å]	impurities
$1.5MgH_2+0.75Fe+0.25Zn$	*Fm*-3*m*	*a*=6.44362(3)	Fe
$2MgH_2+0.75Fe+0.25Zn$	*Fm*-3*m*	*a*=6.44432(7)	Fe, unknown

The major phase found was Mg_2FeH_6 and no evidence of partial substition of Fe^{2+} ions by Zn^{2+} ions was clearly demonstrated.

Compound	space group	lattice parameters [Å]	impurities
$LiBH_4$	*Pnma*	*a*=7.17858(4), *b*=4.43686(2), *c*=6.80321(4)	-

The room temperature data were recorded at the wavelength λ=0.48562 Å (range of scattering angles 1°≤2θ≤28.5°, step size 0.002°). The structure was solved with the recently developed computer programme FOX [1] and found to contain one lithium, one boron and three hydrogen sites. Structure refinement was performed by the Rietveld method by using the program FullProf.2000 and converged to R_{Bragg}=0.035, R_{wp}=0.146 and χ^2=1.86. The present study provides the first example of a metal hydride structure for which the hydrogen atoms have been located unambiguously by X-ray synchrotron powder diffraction.

Compound	space group	lattice parameters [Å]	impurities
$CsOH \cdot H_2O$	*I*4$_1$/*amd*	*a*=4.38088(4), *c*=15.46525(17)	CsH, CsOH

The room temperature data were recorded at the wavelength λ=0.48562 Å (range of scattering angles 6°≤2θ≤36.5°, step size 0.002°). The structure was also solved with the programme FOX. Structure refinement (FullProf.2000) converged to R_{Bragg}=0.059, R_{wp}=0.133 and χ^2=5.23. Tetragonal caesium hydroxide monohydrate, a clathrate hydrate, is a polymorph of three known hexagonal or pseudo-hexagonal modifications. It was obtained as a by-product in a high pressure experiment. Whether it is a high pressure polymorph, however, stays to be verified.

Compound	space group	lattice parameters [Å]	impurities
$Mg_{\sim 1}Ir_{\sim 1}$	*Cmca*	*a*=18.4639(5), *b*=16.1699(4), *c*=16.8167(4)	-

The structure is currently analysed, the best structural model (22 independent atoms R_{Bragg}=0.26, χ^2=8.4) found by FOX is not yet refined.

[1] V. Favre-Nicolin and R. Černý, FOX & ObjCryst++ : new object-oriented tools for crystal structure determination. *J. Appl. Cryst*. – computer programs, in preparation.
see also : http://objcryst.sourceforge.net/, and Book of Abstract of the ECM 20, Krakow 2001, p. 135.