ESRF	**Experiment title:** Measurement of long-range order parameter in substituted $LaNi_5$ compounds	**Experiment number:** CH 101
Beamline: BM 16	**Date of experiment:** from: **4th** June 96 to: 6th June 96	**Date of report:** 20th August 96
Shifts: 6	**Local contact(s):** **Eric** Dooryhee	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

J.-M. Joubert*, R. Cerny*, K. Yvon : Laboratoire de Cristallographie, Université de Genève, 24 Quai E. Ansermet,CH-1211 GENEVE 4.

M. Latroche*, A. Percheron-Guégan: LCMSTR, CNRS, 1 Place A. Briand, F-92195 MEUDON CEDEX.

Report:

$LaNi_5$ derivative compounds are widely studied for their ability to store hydrogen reversibly. Major applications are found in nickel-hydride batteries which are replacing nickel-cadmium ones at increased capacity and decreased toxicity.

The best compromise between hydrogen storage capacity, adequate thermodynamic properties and resistance to corrosion in highly alkaline electrolyte was obtained for multisubstituted compounds such as $LaNi_{3.55}Mn_{0.4}Al_{0.3}Co_{0.75}$ [1]. In order to understand the role of the different substituents in that compound, we have decided to study its crystallographic properties in details.

It crystallizes with the $CaCu_5$ structure type (space group P6/mmm, La on site la (0,0,0), Ni, Mn Al and Co on sites 2c (1/3, 2/3, O) and 3g (1/2, 1/2, 1/2)). The exact distribution of the transition elements and aluminium is unknown. Its determination *by* conventional diffraction experiment is impossible due to (i) the proximity of the elements in the periodic table and (ii) the fact that a single data set (even of neutron diffraction) is

not sufficient to determine the population rates of more than 3 elements over 2 sites. For this reason, we proposed to collect synchrotrons X-ray diffraction data recorded at the different K-edges of the transition elements in order to take advantage of anomalous dispersion effects.

Three patterns were collected at the powder diffraction beamline (BM16) in flat plate geometry at the K-edges of Ni (λ=1.49050 Å), Co (λ=1.61268 Å) and Mn (λ=1.89898 Å). The wavelengths were chosen not too far from the edges to obtain high dispersion terms ($f' \approx -6$ e-) but not too close to be confident in the theoretically calculated values [2], and to avoid the use of standard. The data reduction of the patterns continuously recorded by 9 detectors into a single step-by-step data set was made by using on-site developed software.

For the data treatment, we used the GSAS package [3] which allows joined Rietveld refinement, Despite of difficulties to model the line shapes which appeared to be super-lorentzian, we succeeded in refining simultaneously all the data. The refinement converged unambiguously to a solution in which the manganese and aluminium occupy only the 3g site and cobalt atoms are distributed statistically, according to their multiplicity, over the 2c (40%) and 3g (60%) sites. The results differ slightly from the previously reported results on single substituted compounds in which manganese was found to occupy both 2c and 3g sites [4] and cobalt to be preferentially distributed on the 3g site [5].

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[3] R. B. Von Dreele and A.C. Larson, General Structure Analysis System, University of California, 1995.

[4] A. Percheron-Guégan, C. Lartigue, J.-C. Achard, P. Germi and F. Tasset, *J. Less-Common Met., 74 (1980)1.*

[5] E. Gurewitz, H. Pinto, M.P. Dariel and H. Shaked, *J.Phys F'., 13 (1983) 545.*