ESRF	**Experiment title:** Structure Refinement of Lanthanum-Hexaboride to obtain an Instrument Parameters File for GSAS	**Experiment number:** CH-189
Beamline: BM16	**Date of experiment:** from: 23-Nov-96 to: 04-Dec-96	**Date of report:** 10-Apr-97
Shifts: 12	**Local contact(s):** Andy Fitch	*Received at ESRF:* 17 AVR. 1997

Names and affiliations of applicants (* indicates experimentalists):

* Markus Wunschel [1]
* Robert E. Dinnebier [1]
* Falk Olbrich [2]

[1] Laboratory of Crystallography, University of Bayreuth, D-95440 Bayreuth, Germany
[2] Institute of Inorganic Chemistry, University of Hamburg, D-20146 Hamburg, Germany

Report:

Data collection of LaB_6 was carried out at room temperature during single bunch time in transmission mode (0.3mm glass capillary) using 0.84979 Å from double monochromator and 9 parallel Ge (111) analyzers. The data was collected in continuous mode and later recalculated to equal step size in 2Θ of 0.0025°.

We used the program package GSAS [l] for the Rietveld [2] refinement, shown in Fig. 1. The peak profile function was modeled using a multiterm Simpson's rule integration of the pseudo-Voigt function [3]. The strong asymmetry in the low angle region was modeled by a lately implemented function [4] which accounts for the asymmetry due to axial divergence, leading to a strongly improved fit and therefore better profile R-factors. A manual fit background was used in combination with a refinable 4-term cosine-series to give some flexibility back to the program.

Interestingly, the profile came out to be almost 100% Lorentzian with LX= 0.899 and LY= 1.56, with the Lorentzian coefficient defined as $\gamma = LX/\cos\Theta + LY\tan\Theta$. The asymmetry parameters due to axial divergence remained close to their calculated positions (S/L=0.0080 and H/L=0.0080, where S is the sample length, H is the detector slit length and L is the distance sample-detector).

Due to the high crystallinity of the material, primary extinction had to be taken into account for the Rietveld refinement.

The final R-values are R-p=10.39% and R-wp=13.22%. and R-F=2.82% [Figl].

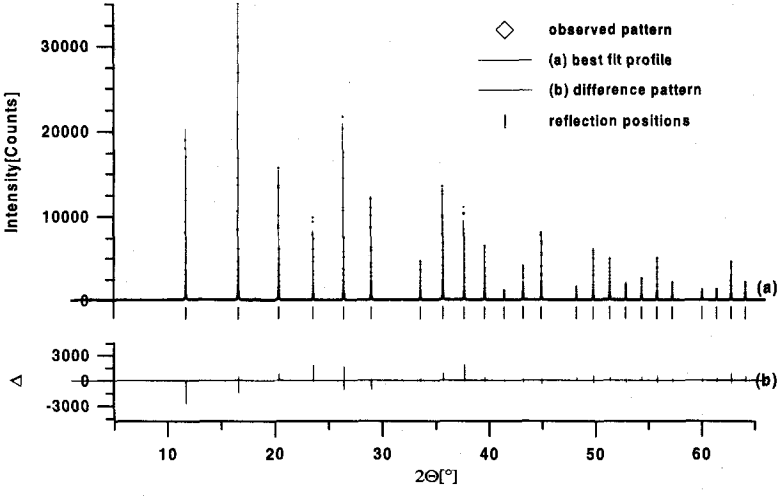

Fig 1:
Actual Rietveld plot of LaB_6 ($Pm\bar{3}m$ symmetry).

References:

[l] Von Dreele,R.B. & Larson, A.C. (1987) GSAS, Generalized Crystal Structure Analysis System. LAUR-86-748, Los Alamos National Laboratory, Los Alamos, NM, USA.

[2] Rietveld, H.M. (1969) J. Appl. Cryst. 2, 65-71.

[3] Thompson,P., Cox, D.E. & Hastings, L.B. (1987) J. Appl. Cryst., 20,79-83.

[4] Finger, L.W., Cox, D.E. & Jephcoat, A.P. (1994) J. Appl. Cryst., 27, 892-900