ESRF	**Experiment title:** Structure refinement of palygorskites: chemical and structural correlations	**Experiment number**: CH-1732
Beamline:	**Date of experiment**: from: 30 April 2004 to: 3 May 2004	**Date of report**: 27 Feb 2006
Shifts: 6	**Local contact(s)**: Dr. Denis TESTEMALE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*** Dr Mercedes SUAREZ. Universidad de Salamanca, Spain**
Dr Lucia ALIANELLI, ILL Grenoble France
* Dr. Eric DOORYHEE, Lab. Cristallographie, CNRS, Grenoble France
* Dr Emilia GARCIA ROMERO, Universidad Complutense de Madrid, Spain
* Dr. Pauline MARTINETTO, Lab. Cristallographie, CNRS, Grenoble France
Dr Constantino REYES-VALERIO, INAH, Mexico
* Dr. Manuel SANCHEZ DEL RIO, ESRF, Granoble France

Report:

Powder diffraction data were recorded at BM01B on a collection of palygorskites with different chemical composition (characterized by Analytical Electron Microscopy) permitted to unambiguously relate the crystallographic parameters *a* (or *a* sin β if a monoclinic phase is considered) and *b* to the nature of the octahedral sheet.

High-resolution X-ray powder diffraction patterns (Fig. 1) were collected with a fixed wavelength of 0.79950±0.00001 Å at room temperature. Powdered samples were placed inside a 1 mm diameter capillary, which was rotated during exposure. Data collection was done in a continuous 2θ-scan mode from 3° to 28°.

The analysis of some peaks of the diffractogram, mainly the 110, 200 and 040 indicated a well defined dependence of the peak position versus both both the number of octahedral positions that are occupied and the type of octahedral cation. A complementary electron diffraction study concludes that there is no significant change in *c*. The unit cell modification essentially consists in an expansion in the <100>

direction as the number of cations with larger ionic radii (Mg^{2+} and Fe^{3+}) dominates with respect to smaller cations (Al^{3+}). We observed a linear dependency of *b* and *a* (or *a* sin β) versus the chemical composition of the octahedral layer (Fig. 2). This relationship can be used for assessing from conventional diffraction data, without the need of performing chemical analyses, the ratio of Al to Mg+Fe contents and the number of vacancies, in the octahedral sheets.

The results of this experiment have been submitted for publication:

Mercedes Suárez, Emilia García-Romero, Manuel Sánchez del Río, Pauline Martinetto and Eric Dooryhée

On The Structure of Palygorskite: Cell Parameters' Dependence on the Octahedral Contents

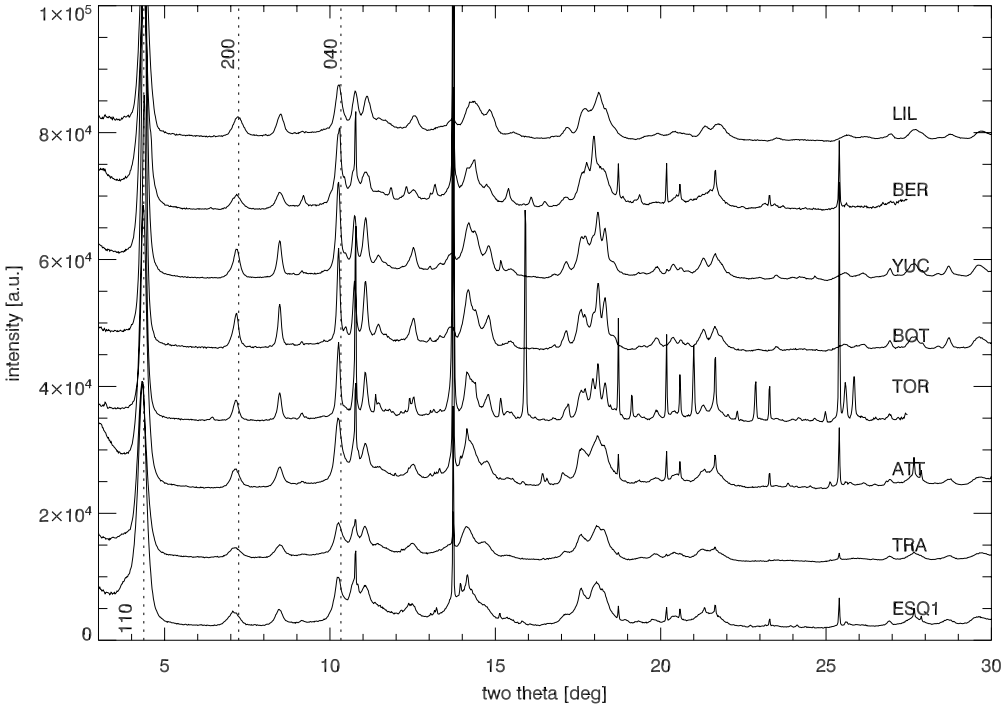

Fig 1 Synchrotron XRD patterns versus 2θ for the eight palygorskite samples studied. Photon wavelength is 0.8 Å. Dotted lines indicated the approximated positions of the 110, 200 and 040.peaks, which are discussed in the paper.

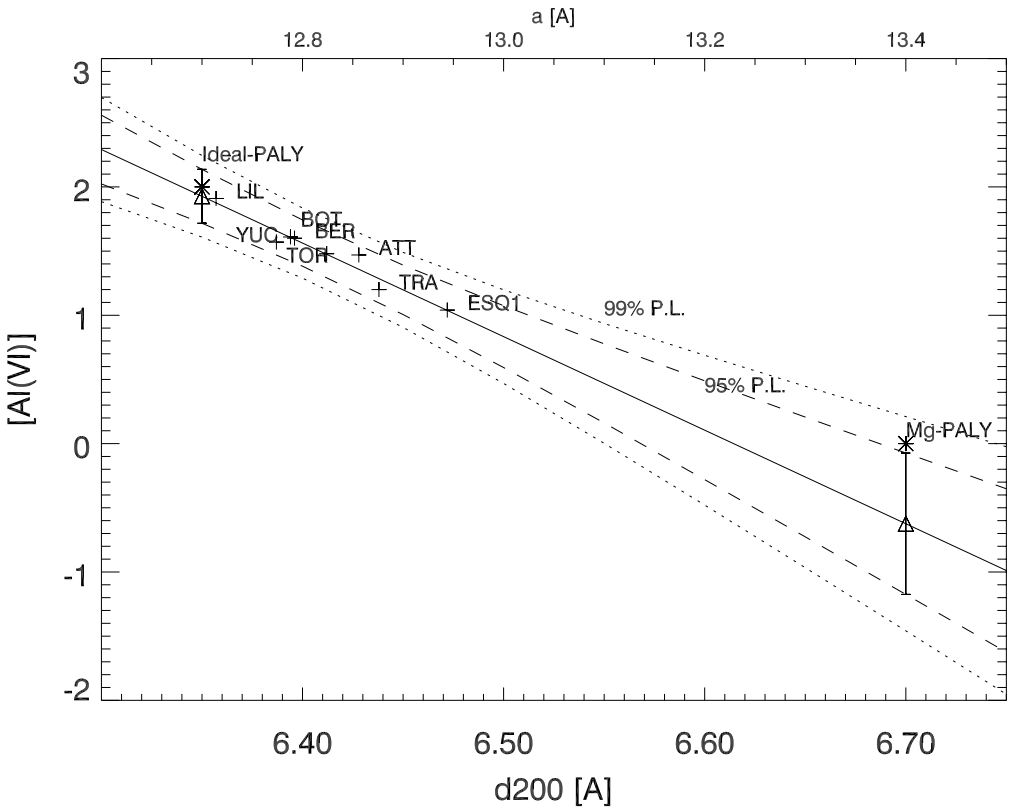

Fig. 2 Relationship between the number of Al occupying octahedral positions in palygorskites and the d200 spacing (also a or $a \sin \beta$ calculated from this reflection). Solid line: regression fit with the bands of prediction limits at 95% confidence level (dashed) and 99% confidence level (dotted). The experimental points are plotted together with a theoretical "imaginary Mg-palygorskite" with five octahedral positions occupied by Mg and "ideal palygorskite" (asterisks). The predicted value for these palygorskites (triangles) using the regression fit are also plotted.