

## Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.**

Once completed, the report should be submitted electronically to the User Office via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

### ***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### ***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



	<b>Experiment title:</b> Structural changes in AlH <sub>3</sub> under pressure	<b>Experiment number:</b> HC809
<b>Beamline:</b> ID09A	<b>Date of experiment:</b> from: 03/27/2013 to: 03/29/2013	<b>Date of report:</b> 06/11/2013
<b>Shifts:</b> 6	<b>Local contact(s):</b> Lucile Bezacier and Michel Hanfland	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): <b>Charles Pépin*</b> <b>Gunnar Weck*</b> <b>Paul Loubeyre</b>		

## Report:

### Scientific background

High-pressure theoretical<sup>1,2</sup> and experimental<sup>3</sup> investigations of AlH<sub>3</sub> reported that the application of pressure transformed the rhombohedral alpha-phase of alane into high pressure phases hp1 and hp2. However discrepancies still remained and the structure of the hp1 phase was not yet solved experimentally. The aim of this project was to solve the structure of the hp1 phase using single-crystal angle dispersive diffraction and to propose a new equation of state of alane in the pressure range 0-120 GPa.

### Experimental technique

Wide aperture (35° cone) membrane diamond anvil cells (MDAC) equipped with Almax-Boehler anvils of 100\*300 microns culets have been used. Single crystals of AlH<sub>3</sub> had been previously loaded in neon in our laboratory (see figure 1a). Angle-dispersive single-crystal diffraction was carried out using a monochromatic beam (0.414036 Å) focused to 10-15 microns. The samples were rotated about a vertical axis from -20° to 20° to take advantage of the wide cell aperture while not contaminating the CCD with scattering from the experimental apparatus. Images were collected in steps of 1 degree using the online MAR555 detector.

## Results

Single-crystal angle dispersive diffraction allowed us to determine that phase hp1 had a very poor crystallization rate, which can explain the difficulties encountered by the previous studies to determine its structure. However we found that this phase could be described with a monoclinic unit cell.

Upon increasing pressure we confirmed the phase hp2 previously solved though it can be noted that the transition appeared at a pressure 7 GPa higher than what was previously reported.

Using the results collected on ID9 we were able to propose a slightly revised equation of state of alane shown on figure 1b.

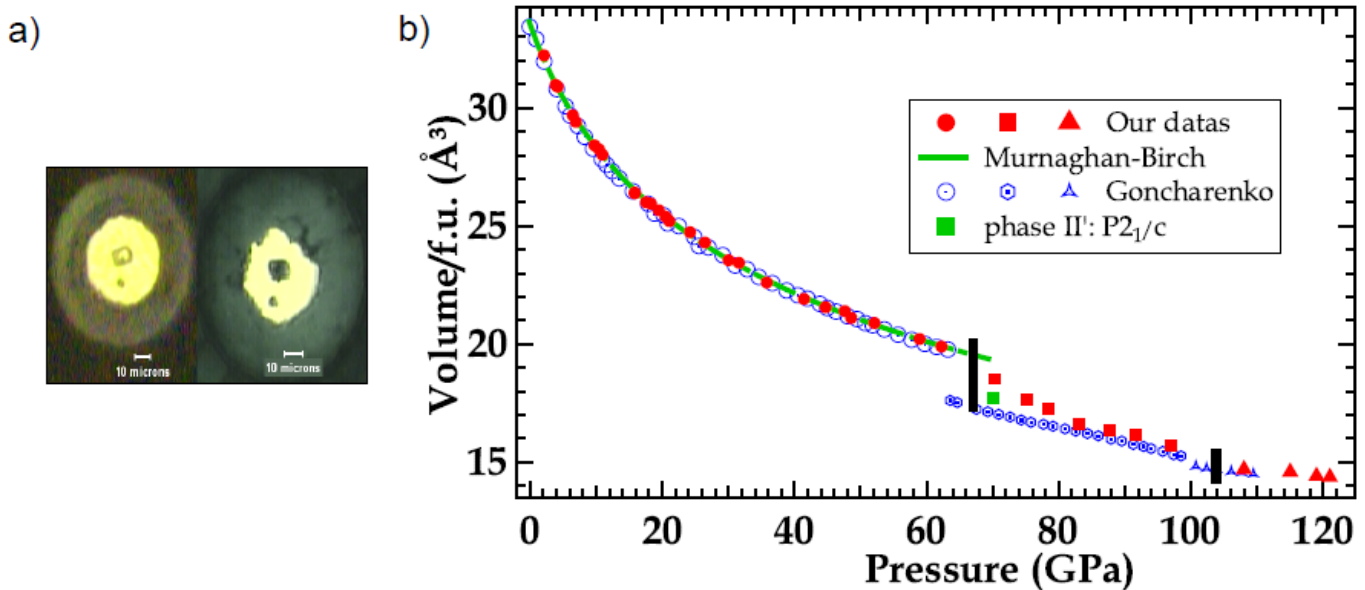


Figure 1: a) View of the sample used on ID9, left: alane at 8 GPa, right: the same sample at 120 GPa. b) Equation of state deduced from our measurements

## References

- [1]C. Pickard et al., Phys. Rev. B 76, 144114 (2007)
- [2]P. Vajeeston et al., J. Phys. Chem. A115, 10709 (2011)
- [3]I. Goncharenko et al., Phys. Rev. Lett. 100, 045504 (2008)