Experimental Report

Proposal: **1-10-20** **Council:** 10/2012

Title: Acoustic levitation apparatus for neutron investigations on liquids with low melting point.

This proposal is a new proposal

Researh Area: Methods and instrumentation

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Samples: ibuprofen-water-tert-butanol
sorbitol-water
water
Si powder
trehalose/lysozyme/Water

Instrument	Req. Days	All. Days	From	To
D16	5	3	05/04/2013	09/04/2013
D1B	2	2	07/08/2013	09/08/2013

Abstract:

The sample environment is a critical component of research programs in advanced materials, geological systems, biology, and energy-related research and development. At high temperatures, various limitations are due to reactions with crucibles. At lower temperatures, heterogeneous nucleation by containers limits the ability to supercool liquids below their equilibrium melting point or to avoid crystallization in supersaturated solutions.
In the past, various contactless techniques have been developed to overcome these problems. The objective of the present work is to optimize a new tool for neutron investigations of liquids at temperatures between -40 and +40 °C. This temperature range covers the liquid and supercooled state of water and many salt and biological solutions that are important in oceanic and biological processes, as well as in many industrial applications

The objective of the present work is to optimize a new tool based on acoustic levitation for neutron investigations of liquids at temperatures between -40 and +40 °C. This temperature range covers the liquid and supercooled state of water and many salt and biological solutions that are important in oceanic and biological processes, as well as in many industrial applications (cosmetics, pharmaceutics...).

A single droplet is held in a node of a standing acoustic wave, avoiding hereby any contact with a thermally conducting holding-device. The droplet size can be adjusted in a wide range, up to 3 mm. The surrounding drying-gas can be conditioned in order to control accurately the temperature, relative humidity, and flow rate applied on the droplet are accurately controlled.

By eliminating contact with container walls, sources of extraneous nucleation are reduced and it becomes possible to study liquids at temperatures substantially below their equilibrium melting point.

For D16 experiment, only H_2O and D_2O were used. The main goal was to test the whole system during cooling by a cryostream flux and exposing to neutrons, obtain diffraction patterns for water and heavy water droplets under different temperatures (liquid and solid state). At room temperature the volume of 40 µL was used for both – water and heavy water, in all other cases the volume of 30 µL was used.

For temperature measurements, a FLIR i60 Infrared Camera was used (Figure 10).

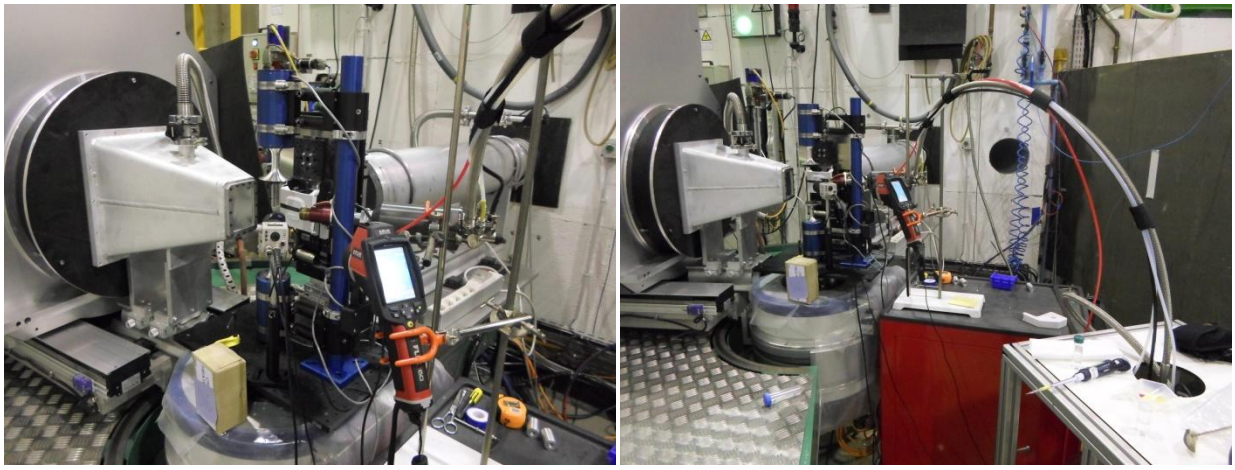

Figure 10. All the equipment installed for the experiments on D16.

In the experiments, the minimum temperature wasn't achieved due to the following reason: as AD51 Dry Air Unit used surrounding air and the air was hot those days, so it warmed up nitrogen flux more than before. During experiments samples were very unstable and it was very difficult to introduce samples and manipulate the levitator-heater system's settings simultaneously.

To prevent icing the method with periodical turning on and off of the heater was used. Usually there was no icing in the first 10 minutes but then it appeared and the heater had to be used almost every minute until the end of a single experiment.

For the experiment on D1b the following solutions in D_2O were prepared: Sorbitol (10 and 65%), BSA (1, 5 and 10%), Lysozyme (2 and 10%). The main goal of these measurements was to freeze a levitating droplet of prepared solutions and expose it to neutrons while the temperature was slowly increased. There were three intervals of temperature: -100 – -60°C, -60 – 0°C and 0°C – r.t. Samples were introduced into the levitator at nitrogen flux's temperature -100°C.

The heating of cold nitrogen flux was achieved by means of the cryostream. The heating rate was chosen 5°C/min (300°C/h), so, for example, if a sample was warmed from -100 to -60 (8 minutes with such rate) and scan time was 2min x5 (10 minutes overall), then 2 minutes after reaching -60°C sample levitated at this temperature. Only for 65% solution of Sorbitol there was observed a glass-like solid at -100°C that started to "crystallize" at temperature above -30°C and became liquid-like at -20°C. For other solutions, there was no glass-like state, just crystallized samples at -100°C and above.

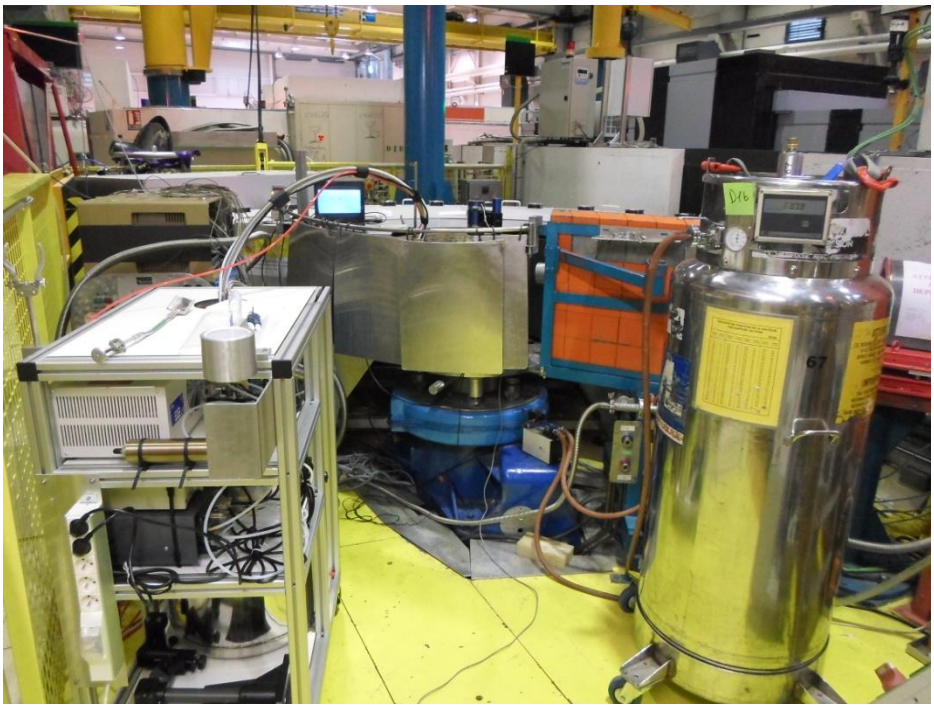

Figure 12. D1b environment with the levitator installed on.

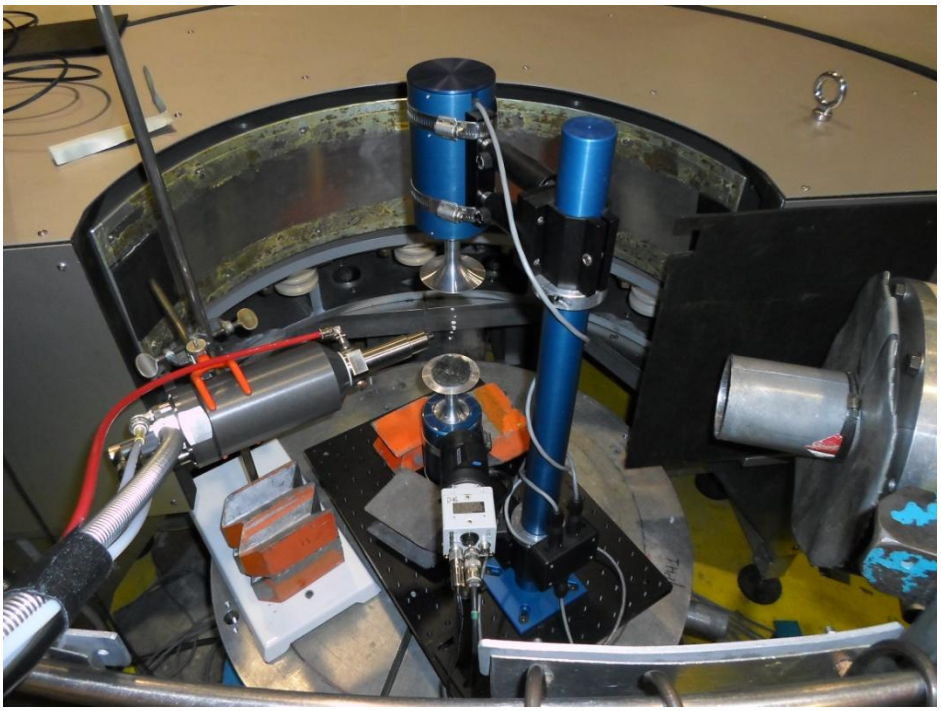

Figure 13. An example of icing during levitation at D1b