NMI3-FP7-J RA-II

WP20 “Advanced neutron tools for Soft and Bio-Materials”

Task 3 “Humidity Chamber”

**Report of 1st task meeting from 22nd of October 2012 at Helmholtz-Zentrum Berlin**

**Attendees:** A. Brûlet (LLB), N. Szekely (JNSC), Y. Gerelli (ILL), B. Demé (ILL), E. Lelièvre-Berna (ILL), S. Baudoin (ILL), N. Belkhier (ILL), M. Zolliker (PSI), O. Kirichek (ISIS), Z. Bowden (ISIS), B. Evans (ISIS), S. Cox (ISIS) D. Wallacher (HZB), K. Kiefer (HZB), N. Grimm (HZB), M. Barrett (HZB) , T. Hauß (HZB), B. Brüning (HZB)

**Presentations** (see attachment)**:**  D. Wallacher, T. Hauß, B. Demé, N. Szekely, M. Barrett

**Summary:**

Initially Dirk Wallacher gave a brief introduction about the goals and the timeline of the task as fixed in the NMI3 consortium contract. In the first year the specifications of the next generation humidity chambers should be fixed based on a detailed review of existing equipment. The new common development should at least combine enhanced and time efficient temperature and humidity control over a wide parameter range, sample change options, and different scattering geometries.

As typical scientific application of humidity chambers at HZB, Tomas Hauß (TH) presented investigations on lipid bio-membranes at the cold neutron diffractometer V1. Thereby double walled, thermostat-controlled chambers are used together with the saturated salt solution technique for high precision humidity control. TH proposed to combine the discrete salt solution technique with the temperature regulation approach to cover the whole humidity range. Automated change of the salt solution or H2O/D2O-mixture by robots or syringe pumps should also be included in future developments as well as an optional use of the fast gas-mixing technique for the lower humility range up to 95%RH. The calibration reliability and accuracy of humidity sensors and salt solutions, which are not much better than 1%, have been discussed. There was agreement, that better sensors and calibration standards are needed.

Bruno Demé (BD) reported afterwards about different generations of humidity chambers used on D16 including a detailed presentation and discussion of the present techniques and future needs. The necessity of well isolated and thermalized chambers has been stressed to achieve stable and defined humidity conditions in the sample region. An investigation under stepwise increased humidity using the T-regulation method showed up, that in particular for high humilities the equilibration times of the measured humidity become very long. It has been discussed, if this has thermodynamic reasons, or is a property of the regulation system, or given by the sensor dynamics. Since no clear answer could be given concerning this point, test experiments have been proposed, where different methods and sensors are used in the same (newest generation) chamber. In conclusion the establishments of perfectly insulated chambers, on-chip temperature sensors, exchangeable salt solutions or H2O/D2O mixtures, higher precision T & RH sensors, and multi-sample holders have been figured out as the future challenges for the project.

Neomi Szekely (NS) presented first results of a new humidity chamber at the KWS2-beamline at FRM-2. The chamber, purchased from “Anton Paar” in 2012, exhibits a neutron version of a commonly used SAXS cell, which allows 5 - 95% RH at 10 - 60°C based on the gas mixing technique. The small cell volume and a pre-heated gas flow allow fast changes of the sample conditions.

Finally, a review of existing humidity cells and techniques in neutron science was given by Matthew Barrett. He pointed out that by gas-flow-mixing a maximum RH of 95% can be reached. With a feedback control loop the accuracy of the method is implied in the sensor, which is in general not better than 1%. In contrast to this the salt solution technique exhibits a more precise and reliable method, where in principle no sensor is needed, if one relies on an exact temperature measurements and literature tables. Nevertheless a comparison of these tables shows up also an uncertainness of about 1.0%. The purity of the salts and the precision of the used sensors might be the reason for this. As the salt solution technique the temperature regulation method is slower than the gas flow mixing, but allows stable conditions at high humidity in the error range of the sensor calibration. Reaching 100% RH without condensation on the sample is in principle possible with this method even if challenging. A sponge close to the sample ensures in this case the saturated conditions by its huge surface. An example showing the strong dependence of the d-spacing of bilayer membranes (measured by neutron scattering) from the RH in the range 98 -100% stressed again the requirement of a high precision sensor technique for improved equipment. In summary a table with the properties of the different humidity regulation methods has been discussed with respect to the project goals to deliver a versatile, user friendly setup with fast equilibration times, precise in-situ measurement of RH and T, and high automation level.

Based on the information given by the speakers a discussion about the feasible designs, accuracy specifications started. There was agreement that the precision of any technique will at least be dependent by the accuracy of the sensor. It seems important to spend massive efforts on the improvement concerning this point in the initial phase of the project parallel to the design of the cell. Another subject, which should be clarified in the beginning, is the physical and technical understanding of the equilibrium formation in sample atmosphere and in the sensor. This is important for the right choice of the humidity control method (or possible combinations) with respect to time efficiency. It has been agreed by ILL and HZB that direct comparison-tests with different sensors and methods will be carried out.

Concerning the further organization of the project it has been agreed, that the partners (and observers) should specify their ideas and possibilities of contribution to the task until the next meeting, which will be at 7th December in Garching attached at the general assembly of the NMI3/FP7 program. Annie Brulet, as the coordinator of the “Bio-Soft Matter JRA”, will start a call for presentations among all participating tasks. A summary of this first humidity chamber task-meeting will be given then.