Experimental Report

02/07/2014

Proposal: LTP-9-3 **Council:** 10/2009

Title: Combined in-situ neutron and optical spectroscopy of complex bio-related soft matter

This proposal is a new proposal

Researh Area:

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Samples: different proteins in solution, but also other (bio-inspired) soft-matter systems
Bovine serum Albumin protein in aqueous (D2O) solution
salts NaCl, CaCl2, YCl3 up to 2M concentration
ovalbumin proteins

Instrument	Req. Days	All. Days	From	To
IN6	8	4		
IN16	12	6	16/12/2011	22/12/2011
IN10	21	14	26/08/2010	02/09/2010
			30/06/2011	07/07/2011
IN11	16	11	26/07/2012	01/08/2012
			27/06/2013	02/07/2013

Abstract:

One of the major trends in neutron science, in particular in the area of soft matter and biology, is the study of increasingly complex systems, for which the control of environmental parameters (e.g., temperature, pressure, pH, salt concentration, light stimulus etc.) is crucial and the use of complementary characterization techniques is not only helpful, but frequently essential. We have recently made significant progress in the identification of important effects of charges on the phase behaviour of protein solutions, including reentrant condensation, which we propose to study systematically in this project by a combination of spectroscopic techniques traditionally used in the lab and neutron methods. While the materials used in this project are of biological origin, the issues are rather related to the physics of soft condensed matter. We propose to establish simultaneous combined in-situ neutron and optical spectroscopy techniques (e.g. UV/vis, IR, fluorescence, Raman). This will greatly enhance the accuracy with which the state of a fragile biological or soft-matter sample can be defined during neutron spectroscopy experiments.

Report on the Long Term Proposal LTP 9-3 for the year 2011/2012

For the allocation period of 2010/2011 we refer to our previous report.

We focus here mostly on our design efforts of optical access sample environment, which are at the centre of the LTP.

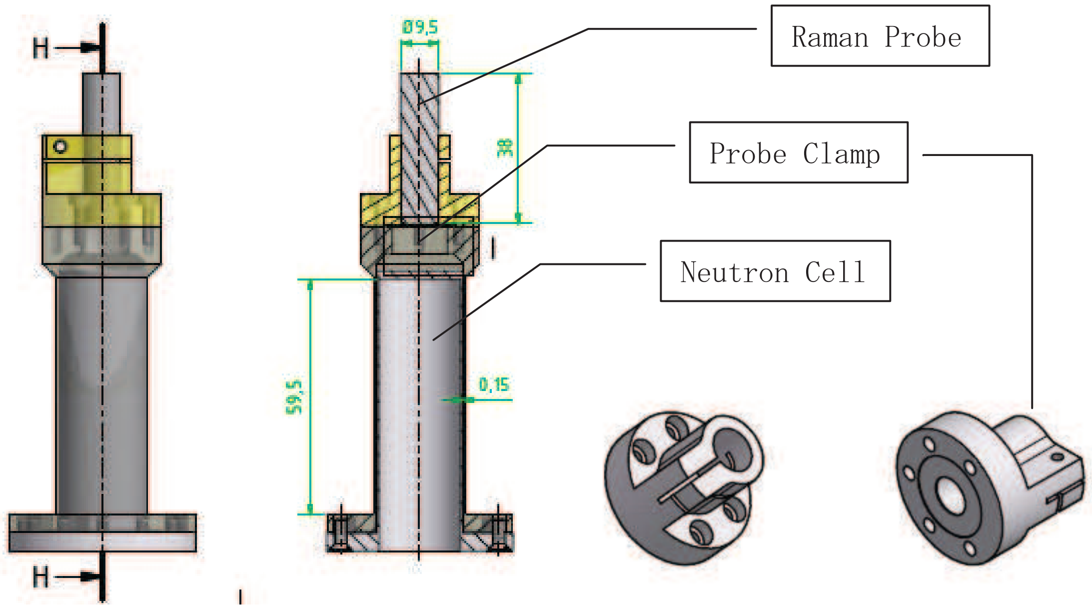
For the allocation period of 2011/2012 we had **not asked** for allocation of beam time. We have only carried out an experiment in commissioning time on IN11 using beam time in backlog from the allocation period 2010/2011. This IN11 experiment took place during the last days of the cycle 2012-1 at the end of July 2012. The spin-echo experiment addressed the possibility to systematically explore the phase diagram of protein solution samples with neutrons and optics. It has therefore been an important precursor experiment for systematic in situ combinations of neutrons with optical methods. A detailed data analysis is in progress.

Besides this one experiment on IN11, all our efforts during 2011/2012 went into the development and improvement of the optical methods and sample environment *ex-situ*, i.e. without using neutrons in order to be efficient. This has involved multiple tests of several optical cells for UV/vis and laser transmission experiments. The cell has been designed in both reflection and transmission mode. For the reflection mode, the UV light for the optical fiber is focused on a mirror after the sample solution, and the transmitted light is reflected back and recorded by the detector. In this case, we have tested the *in-situ* determination of protein concentration by UV-vis spectroscopy. In another test, laser transmission has been used to determine the phase transition of a protein solution as a function of temperature. In this case a pin diode is embedded at the bottom of the inner cylinder to determine the sample transmission. Bovine serum albumin (BSA) in the presence of YCl3 has a lower critical solution temperature in the condensed region. Increasing temperature crossing the critical point, the liquid-liquid phase separation occurs. Laser transmission measurement is therefore crucial for the precise control of the status of the system and the onsite temperature of phase transition.

In addition to adsorption spectroscopy (as tested by us in combination with neutrons before), *in-situ* optical scattering methods would be of very significant interest to the neutron user community. Our general (improved) design concept of the optical access sample containers used for neutron experiments is of course intentionally compatible with different types of optical methods. In order to enable scattering measurements we have designed a Raman Cell that is currently being manufactured in the Tuebingen workshop. This will utilize the PSCM Ocean Optics Raman system. The technical drawings of this new version are shown in the figure below (next page). We intent to test this system initially *ex-situ*, and then, if beamtime is allocated, also simultaneously with various neutron techniques as described in the initial proposal.

Furthermore, *in-situ* FTIR (Fourier Transform Infra-Red) spectroscopy is being investigated. Pike Technologies offer a suitable fiber (“FlexIR”). This is compatible with almost all IR machines and could be specified with various ATR (Attenuated total reflectance) crystals. The newly designed Raman Cell could in principle be adapted for FTIR if it proves to be successful for Raman measurements *in-situ*.

In conclusion, our design efforts of optical access sample environment, which are at the centre of the LTP, are progressing, but require a substantial amount testing, which in the last period we have done mostly ex-situ.


Raman Probe
Probe Clamp
Neutron Cell
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H
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38
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