# Experimental report

04/03/2021

**Proposal:** 8-04-803 **Council:** 10/2016

**Title:** Hierarchical mobility in silk proteins - implication for storage and spinning

**Research area:** Biology

**This proposal is a resubmission of 8-04-775**


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**Samples:** Silk proteins

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| IN16B | 3 | 2 | 06/02/2017 | 08/02/2017 |


## Abstract:

The formation of silk fibres in both spiders and silkworms is characterized by a controlled conversion of short range ordered structures in solution into long range ordered beta-sheet rich structures in the final fibre. The dynamics of the water and protein chains involved in the conversion remains, however, unknown. Building upon our newly developed deuterium exchange method; and, the performance of the IN16B spectrometer (faster, good dynamic range allowing access to both the internal and center-of-mass diffusion of proteins in solution); we wish to determine the global and internal dynamics leading to conformational changes in silk proteins in aqueous solution (D2O and H2O). The expected results will determine how hierarchical mobility plays a role in the control of silk proteins storage, aggregation, and spinning. The data analysis will build upon recently published frameworks to separate the contributions from the solvent water, internal, and global motions [M.Grimaldo et al., EPJ Web conf.83, 02005 (2015) and references therein].

**Report on Experiment 8-04-803 on IN16b, 2017**

## Hierarchical mobility in silk proteins – implication for storage and spinning

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We have carried out the experiment 8-04-803 (IN16b) on native silk protein solutions extracted from the *Bombyx mori* silkworm silk species. Our principal aim was to describe the dynamics events taking place during silk conformational change as a function of temperature.

The main challenge of having the silk proteins in $D_2O$ was solved earlier using a desalting column to exchange the $H_2O$ to $D_2O$.

Figure 1 shows the transmission from SANS experiment of different silk protein solutions.

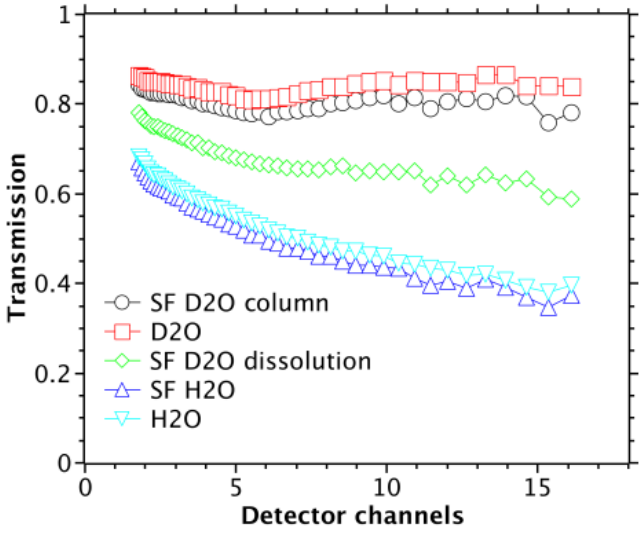



Figure 1. Neutron transmission of silk proteins (SF) in $H_2O$, dissolved in $D_2O$ and exchanged in $D_2O$ by means of an exchange column, $D_2O$ buffer and $H_2O$ buffer.

At IN16b, we prepared and collected the following samples:

| Sample | QENS (280 K) | FWS | Inelastic (1.3ueV) | QENS (353K) |
|---|---|---|---|---|
| SF_$D_2O$_3 | 30 min | 30s | 90s | 30min |
| SF_$H_2O$_1 | 30min | 30s | 90s | 30min |
| TSF_$D_2O$_1c | 30min | 30s | 90s | 30min |

Other samples were run, but failed. Mainly in the signal optimisation phase.

Elastic window scans:

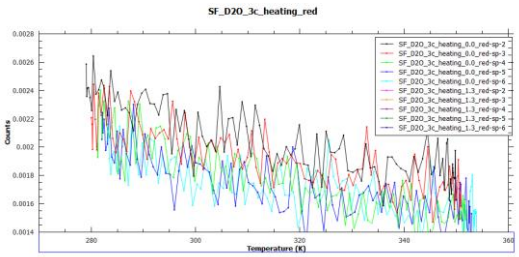
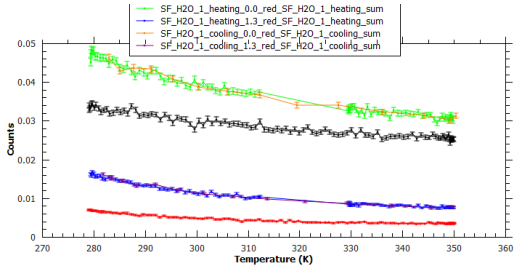



Figure 2. FWS at 0 and 1.3ueV for SF_$D_2O$




FWS at 0 and 1.3ueV for SF_$H_2O$

QENS:

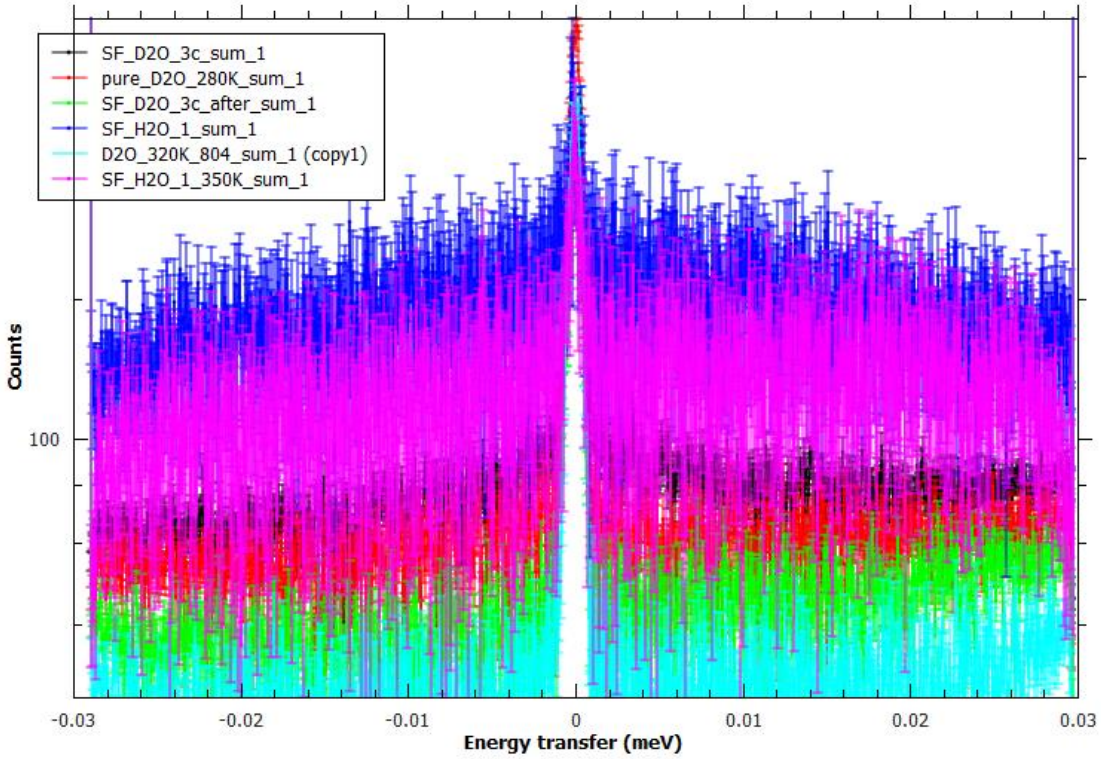



Figure 4. QENS comparison plot between SF_$D_2O$ and SF_$H_2O$

In conclusion, we have successfully exchanged native silk protein from $H_2O$ to $D_2O$. The FWS and QENS, to explore the temperature induced gelation did not work however. The weak signal intensities and slow conversion precluded any further useful analysis.