Experimental report

20/09/2022

Proposal: 1-10-42 **Council:** 4/2019

Title: Absolute inelastic neutron scattering cross sections of clathrate hydrate compounds for new very-cold-neutron moderators

Research area: Methods and instrumentation

This proposal is a new proposal

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Samples: clathrate hydrates

Instrument	Requested days	Allocated days	From	To
D20	2	2	21/08/2020	23/08/2020
IN5	4	4	04/02/2021	08/02/2021
D7	3	0		

Abstract:

The goal of this proposal is the measurement of inelastic neutron scattering cross sections (S(q,omega)) of clathrate hydrate compounds in absolute units. These materials are particularly promising candidates for new moderators for very cold neutrons. The expected enhancement of intensities at wavelengths larger than from existing cold sources has high impact on the capabilities of neutron scattering instrumentation which is of high interest for several neutron centers. A large range of q and omega shall be covered in experiments at IN5 and IN4/Panther, complemented with measurements for sample characterisation at D20 and D7, to provide a valuable data base for model calculations of moderation efficiencies in realistic configurations.

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Experimental team: T. Hansen, M. Koza, R. Wagner, O. Zimmer

Time-of-flight measurements on clathrate hydrate samples with Tetrahydrofuran (THF) as guest molecule have been performed at IN5, with the goal to determine the scattering function $S(q, \omega)$ in absolute units. Samples of stoichiometric composition have been prepared as protonated and deuterated variants:

1) $17D_20$:THF-d

2) $17H_20$:THF-d

3) $17D_20$:THF

4) $17H_20$:THF

This choice enables a contrast variation for separation of the scattering contributions from the host and the guest molecules. While not being essential for the application of the material as a moderator for VCN (for which the weakly absorbing, fully deuterated variant is of highest interest), the additional information will be of great value for comparison of the data with simulations of the motional modes. The mixing of the liquids to prepare the samples has been done in the ILL chemistry laboratory shortly before the measurements. The weighted mixtures were filled in hollow cylinders of 15 mm inner diameter as shown in Figure 1, with a filling height of 2 cm.

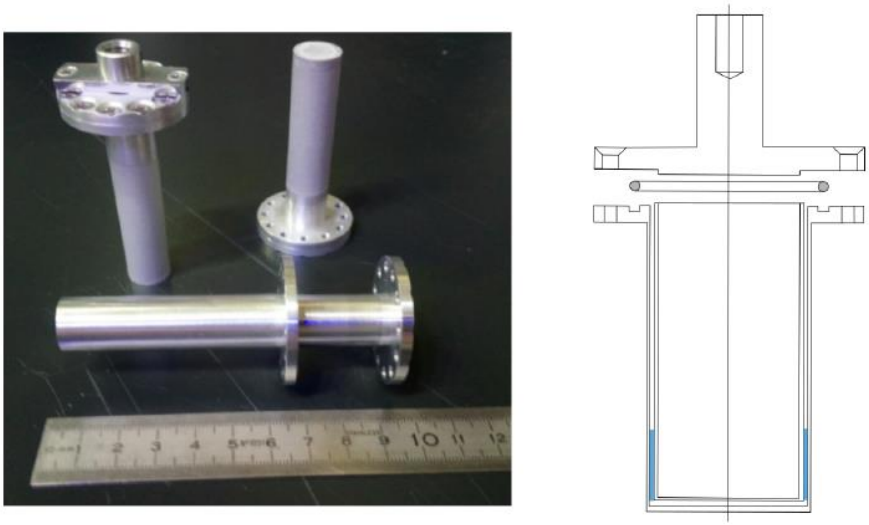

Figure 1: Sample holder used for the experiment (right picture from Physica B 266 (1999) 112" by J. Wuttke).

A scan of the beam profile (Figure 2) allowed us to place the sample in a position, where the uncertainties of the exact filling height and the sample location had the smallest impact on the systematic error. Measurements with the samples were performed at different

wavelengths (2 Å and 3 Å) and temperatures, with emphasis on measurements at the base temperature of the cryostat (1.5 K), at which the local modes of the guest molecules are frozen out, resulting in only neutron up-scattering contributions to the signal.

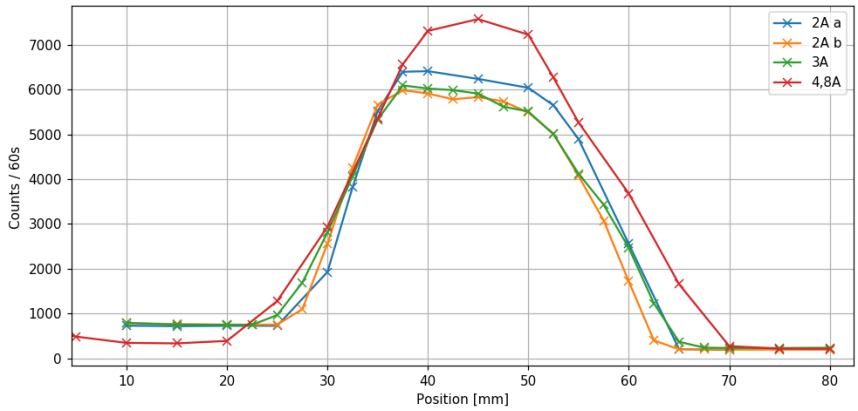

Figure 2: Vertical beam profile at IN5 for several wavelengths.

For absolute calibration of the scattering signals, complementary measurements were performed with a vanadium sample (rolled foil) of known mass in the same geometry as the samples, and with the empty cells for subtraction of backgrounds. Figure 3 shows a cut through the raw data of $S(q, \omega)$ for runs at 2 Å, from which one can already see the variations of the signal shapes due to the contrast variation.

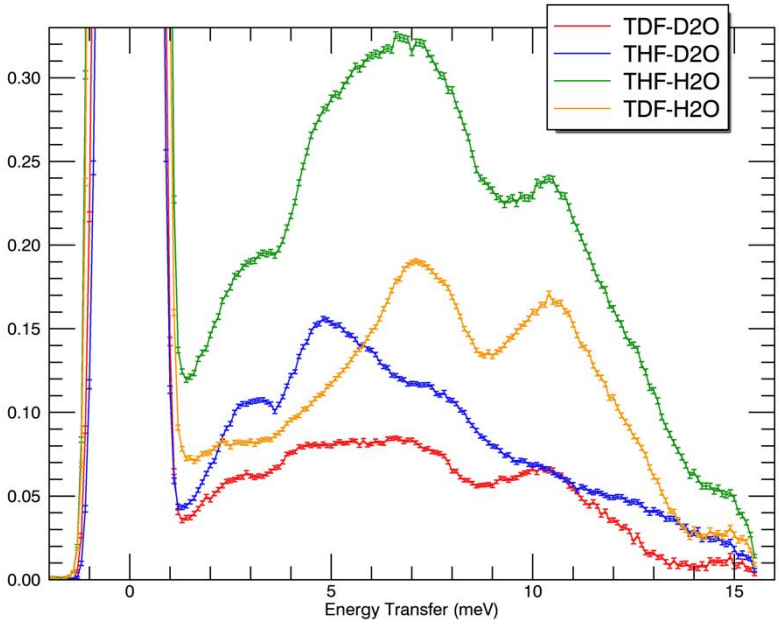

Figure 3: Scattering Data at 2 Å for the differently deuterated clathrate samples.