# Experimental report

05/02/2016

**Proposal:** 6-02-549 **Council:** 10/2014

**Title:** A new approach to the structure of1H-containing materials using polarised neutrons: the cases of liquidmethanol and cyclohexane

**Research area:** Chemistry

**This proposal is a new proposal**


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**Samples:** C3H6O
CHCl3
deuterated methanol/CD3OD
cyclohexane/C6H12
methanol/CH3OH
deuterated cylohexane/C6D12

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D3 | 12 | 12 | 24/07/2015 | 05/08/2015 |


## Abstract:

The main difficulty with structure determination of 1H-containing ('protonated') liquids is the huge incoherent inelastic scattering that arises due to the exceptionally high level of spin-incoherency of the proton (1H). As a result, more than 90 % of the measured signal (using non-polarized neutron beams) from pure H2O, pure CH3OH (methanol) or C6H12 (cyclohexane) is useless ('background') from the structural point of view. Spin-incoherence, however, can be bypassed if the neutron beam is polarized. We have recently shown that using the D3 instrument, it is possible to measure accurate (coherent) static structure factors of water samples, containing a varying proportion of 1H, over a wide Q-range. Even more important perhaps is the fact that the incoherent 'background' could also be determined precisely. Here we propose an experiment for investigating whether the incoherent scattering depends on the chemical environment of the protons, using liquid methanol and cyclohexane. These materials contain the same amount of hydrogen as water, so that comparison of the incoherent backgrounds would provide direct information on the influence of the chemical environment of 1H atoms.

# A new approach to the structure of $^1$H-containing materials using polarised neutrons: the cases of liquid methanol and cyclohexane

## Introduction

Materials that contain hydrogen are undoubtedly among the most important substances in our world: it's sufficient to remember that life on Earth is based on water. For this reason, determining the structure of materials containing hydrogen is of utmost importance in various scientific fields, from basic chemistry and physics, through geochemistry, to biochemistry and soft-matter research.

X-ray diffraction is not a sufficiently sensitive probe for hydrogen in most of the cases (including the case of $H_2O$), so that neutron diffraction with H/D substitution seems to be the only feasible way of deriving more detailed information on the microscopic structure of hydrogenous (i.e., ones with $^1$H) systems. The main difficulty with structure determination of $^1$H-containing ('protonated') liquids is the huge incoherent inelastic scattering that arises due to the exceptionally high level of spin-incoherency of the proton ($^1$H). As a result, more than 90 % of the measured signal (using non-polarized neutron beams) from pure $H_2O$, pure $CH_3OH$ (methanol) or $C_6H_{12}$ (cyclohexane) is useless ('background') from the structural point of view.

Spin-incoherence, however, can be bypassed if the neutron beam is polarized. We have recently shown that using the D3 instrument, it is possible to measure accurate (coherent) static structure factors of water samples, containing a varying proportion of $^1$H, over a wide Q-range [1].

Even more important perhaps is the fact that the incoherent 'background' could also be determined precisely. Here we wished to investigate whether the incoherent scattering depends on the chemical environment of the protons, using liquid methanol, acetone, chloroform and cyclohexane. The final aim of this experiment was to provide a reference set of (spin-)incoherent backgrounds for hydrogenous materials as a function of $^1$H content, using polarised neutrons from the D3 instrument.

## The experiment

For being able to determine the coherent and incoherent structure factor of $^1$H containing liquid samples in which the chemical environment of the protons varies, we have measured structure factor of $CH_3OH$-$CD_3OD$ mixtures (protonated compound content: 0, 20, 40 and 100 %), by separating the coherent and spin-incoherent parts of the scattering, using the D3 instrument at ILL. Similar measurements have been conducted for acetone-D ($C_3D_6O$) and acetone-H ($C_3H_6O$), as well for cyclohaxane-H ($C_6H_{12}$) and chloroform-H ($CHCl_3$). The separated incoherent and coherent contributions are shown in Figures 1 and 2, respectively.

The idea was that by measuring the entire series will make it possible to compare the incoherent backgrounds for compounds of different chemical nature, also as a function of the isotopic composition. It is now feasible to establish whether the incoherent background is the same for identical proton contents, or it depends on the environment of the protons. This knowledge would facilitate the handling of spin-incoherent backgrounds of any hydrogenous materials, without actually performing polarisation analyses for every single sample.

The next step will be to set up an appropriate protocol for comparing the incoherent intensities, shown in Figure 1.

**References**

[1] ILL Experimental Report 6-02-519 (by L. Temleitner); L. Temleitner et al., Phys. Rev. B 91, 014201 (2015)

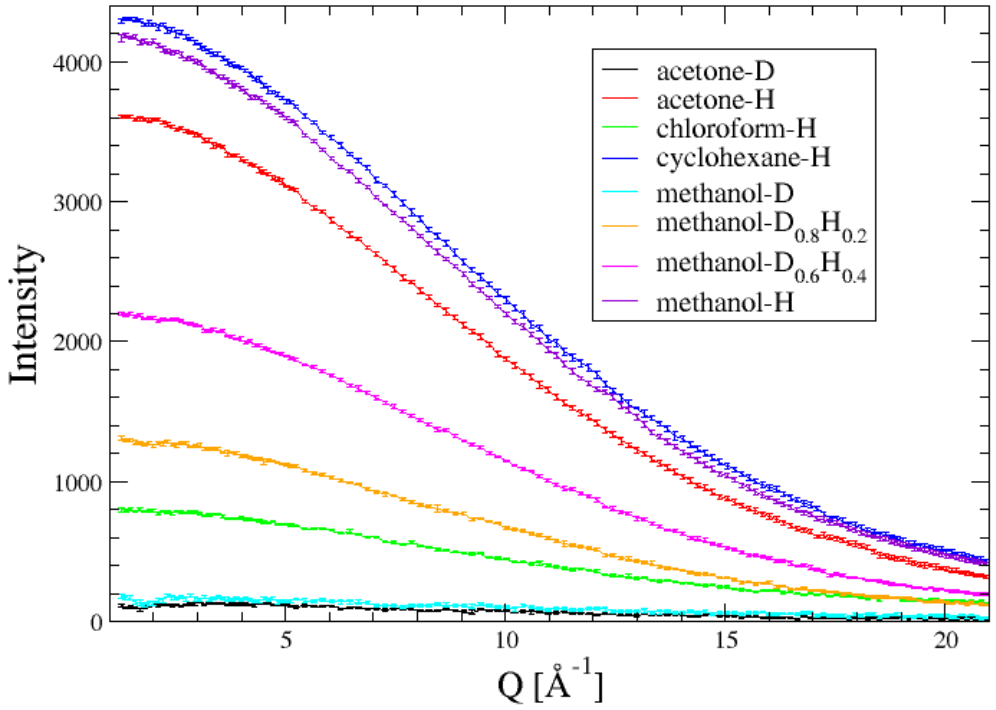


Figure 1: Measured incoherent intensities for acetone, chloroform, cyclohexane and methanol, with varying H/D ratios

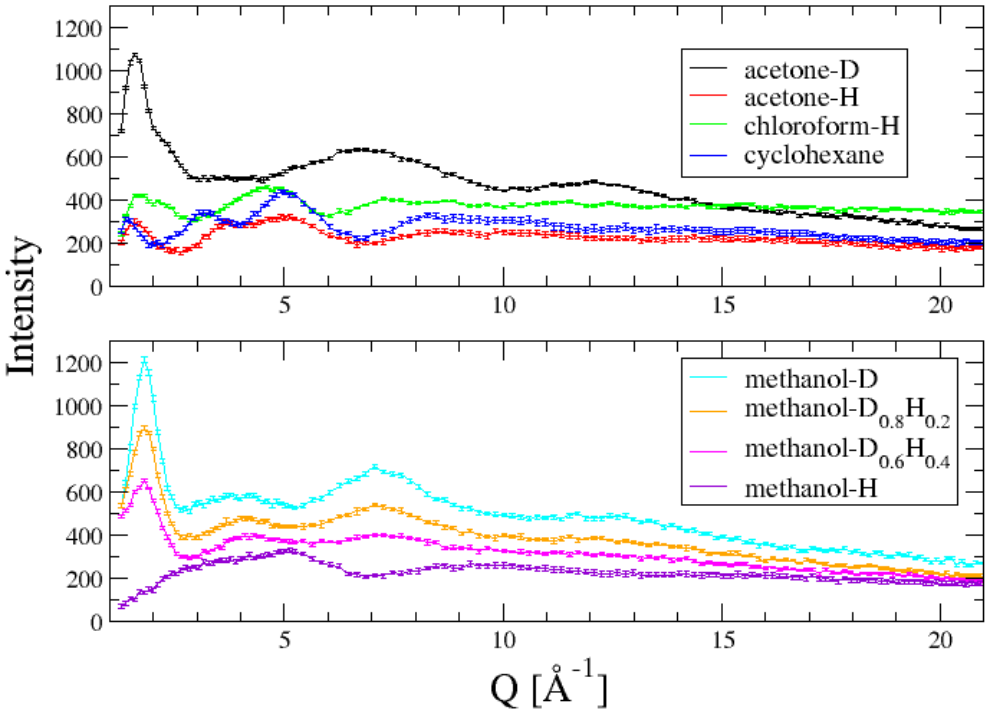


Figure 2: Measured coherent intensities for acetone, chloroform, cyclohexane and methanol, with varying H/D ratios