Experimental report

17/02/2020

Proposal: 6-05-998 **Council:** 10/2018

Title: Recovering of density scaling and isochronal superposition by globular dynamics of dipropylene glycol

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: d6-dipropylene glycol
Glycerol with deuterated water, C3H8O3 + D2O

Instrument	Requested days	Allocated days	From	To
IN6-SHARP	4	0		
IN16B	4	3	07/10/2019	10/10/2019

Abstract:

In the proposed experiment we wish to study the temperature and pressure dependence of the centre of mass motion on partly deuterated dipropylene glycol (DPG), i.e. the dynamics which is not from methyl-group rotation. In particular, we want to study whether the observed breakdown for the hydrogen-bonding liquid DPG close to the glass transition of density scaling and isochronal superposition in the light of isomorph theory is due to hydrogen-bonding or methyl-group rotation. This will be investigated using partly deuterated dipropylene glycol to mask methyl-group rotation.

Dynamics of glycerol rich water-glycerol mixtures, 0.4 protonated sample

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This experiment used simultaneous dielectric spectroscopy (Novocontrol alpha/beta analyzer) and quasi elastic neutron scattering (IN16B). The aim was to study the dynamics of glycerol water mixtures in the glycerol rich region. This experiment focused on the moderately glycerol rich region (glycerol molar ratio = 0.4). The sample being fully protonated.

Experiment on IN16B

On IN16B, we performed 2 cooling ramp isobar at 0.4 K/min (Patm and 2 kbar) and 4 isotherms from atmospheric pressure to 2.5 kbar (310 K, 297 K, 284 K, 271 K) with fixed window scan at 0 and 3 μeV. We also did 5 full QENS spectrum at 5 state points (310 K Patm, 310 K 2.5 kbar, 297 K Patm, 297 K 2.5 kbar and lastly 40 K Patm). During the 1st day, 2 cooling ramp isobar at 0.4 K/min (Patm and 2 kbar), of pure glycerol and 0.3 molar ratio were done.

Technical problems

At the start of the experiment, just after the needed cryostat was put in place, during heating to 310 K, the heating part of the cryostat burned and a new cryostat needed to be put in place until repair, which was done in 24 h. The replacement cryostat could not accept our pressure cell, so an atmospheric pressure isobar was done on pure glycerol and 0.3 molar ratio water glycerol in an 0.1 mm thick standard cylinder sample holder. The last day, during the 4thisotherm, at 271 K. when the pressure when to 2 kbar, the pressure vessel failed and pressure liquid polluted the sample. Background was done then while preparing for a new sample for a last 2 kbar isobar, which gave mixed results due to a temperature sensor not well in contact with the cell.

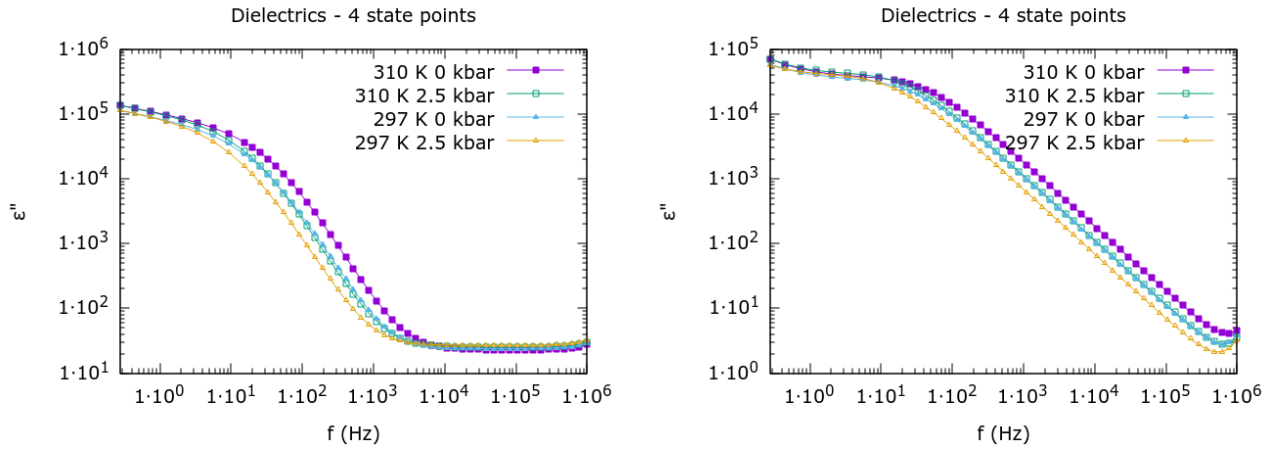

Figure 1: Dielectrics spectroscopy of glycerol water 0.4 molar ratio taken during the 4 state points QENS full spectrum.

Results

For the 4 state points where we were trying to look for a possible isomorph, 310 K 2.5 kbar and 297 K Patm do overlap very well in the QENS spectra, figure 2 left, and in the dielectric signal, either on the real part or in the conduction band, figure 1. We can see on the left the real part of ϵ, where under $\sim$5000 Hz electrode polarization comes into effect. On the right side is the imaginary part of ϵ, where nearly only conduction can be seen, with on the righ, the flattening is from the electrode polarization.The effect of pressure, in the range from Patm to 2.5 kbar, on the dynamics, does seem to be linear. The effect of applying 2.5 kbar has the same effect on dynamics as cooling down 13 K. This effect does seem to hold also at lower temperature than room temperature, as can be seen in the elastic and inelastic fixed window scans in figure 3.

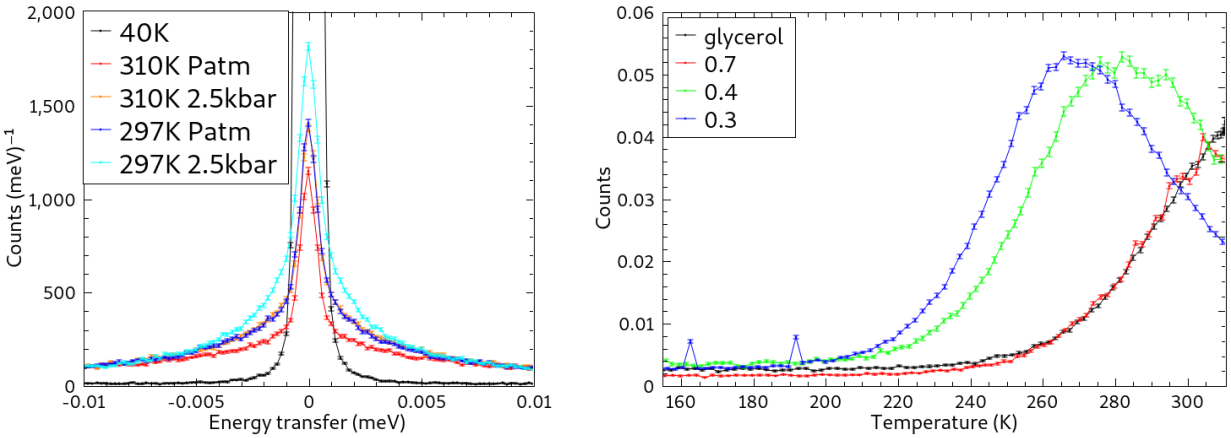

Figure 2: left: QENS full spectrum done at 310 and 297 K, and Patm and 2.5 kbar. in black taken at 40 K for instrument resolution. Right, 3 μeV fixed window scan of glycerol and 0.3, 0.4 and 0.7 glycerol molar ratio in water. using non pressur cells

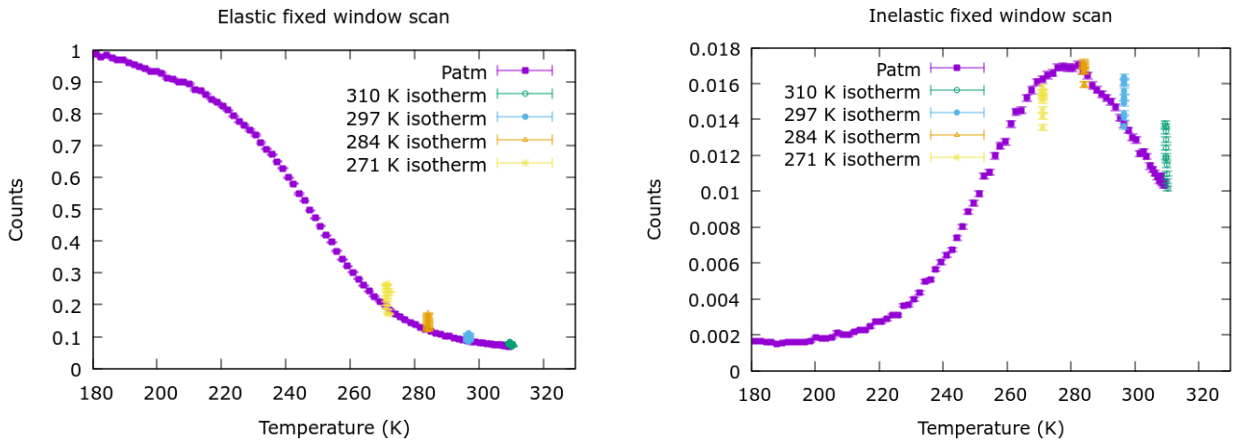

Figure 3: Fixed window scan. Elastic on the left and inelastic at 3 μeV on the right

The next step is to look into a deuterated sample of 0.4 molar ratio glycerol water to separate the dynamics of each constituent. will be done during experiment 6-05-1008 as well as in the concentrated glycerol rich region.