Experimental report

13/05/2019

Proposal: 6-02-587 **Council:** 4/2018

Title: Contribution of intramolecular motions to the global molecular dynamics of cumene

Research area: Soft condensed matter

This proposal is a continuation of 6-05-961

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Samples: d12-cumene (C9D12)
d7-cumene (C6H5CD(CD3)2)
Cumene (C9H12)

Instrument	Requested days	Allocated days	From	To
IN1 LAG	2	0		
IN5	2	0		
IN16B Si 111 BATS	2	0		
IN6-SHARP	2	1	23/10/2018	24/10/2018
D7	2	2	24/09/2018	26/09/2018

Abstract:

Cumene (isopropyl benzene) has been studied with various different techniques on different timescales in a large part of the (T,P)-phase diagram and has shown astonishingly simple dynamic behaviour. The proposed experiment aims to find and study the contribution of intramolecular motions to cumene's global molecular dynamics, containing center-of-mass translation, whole-molecule libration and rotation - both controlled by van der Waals potentials, and methyl-group rotation and libration - controlled by internal molecular potentials, using partly deuterated samples to complement previous studies on fully protonated cumene and additionally, fully deuterated cumene to obtain insight into the collective dynamics.

Beamtime report from D7 and IN6, experiment 6-02-587 24-26 September 2018 (D7) and 23-24 October 2018 (IN6)

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Sample: Fully protonated (H12) and fully deuterated (D12) isopropyl benzene (cumene).

Normal cylindrical aluminium cells of 0.2 mm thickness.

D7 – probing structure

Power: 52 MW
Wavelength: 3.1 Å corresponding to $\Delta E \approx 0.5$ meV.

Measuring only z-polarisation, 1:1 spinflip, 2θ-scan in $80.5° \geq 2\theta \geq 75.5$ with $\Delta\theta = 0.5°$ and counting time 245 s.

Both the fully deuterated and fully protonated cumene were measured at D7 probing the coherent and incoherent part of the structure factor, $S(Q)$. Each sample was measured at 5, 100, 127 (T_g), 160, 170, 180 and 200 K (Fig. 1). The sample was quenched from room temperature to 5 K to enter the glassy state, the sample was then heated up to the next temperature, followed by 30 min waiting time for thermal equilibrium. The sample is seen to crystallise when heated from the glass transition temperature at 127 K to 160 K. The melting temperature of cumene is at 177 K, and the sample are seen to move into the liquid state again at 180 K.

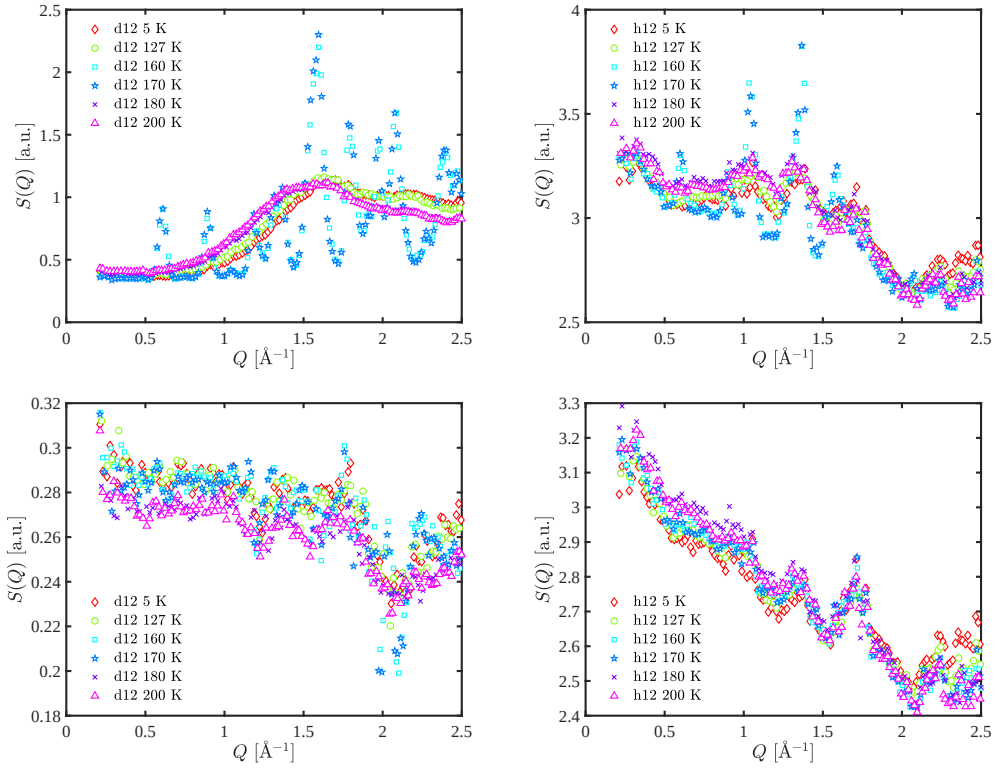

Figure 1: Coherent (top) and incoherent (bottom) part of the structure factor from D7 of deuterated (left) and protonated (right) cumene.

IN6 – probing dynamics

Power: 35 MW
Wavelength: 5.12 Å corresponding to $\Delta E \approx 0.1$ meV.

The same samples in the same sample cells etc., was measured at IN6. Unfortunately, there was a problem with the cryostat (missing o-ring for sealing the vacuum) over night when measuring the deuterated sample, which resulted in useless data of that sample.

The fully protonated cumene data was measured at 200 K, supercooling to 5 K, followed by heating to 100, 127 and 180 K. Because of the strongly scattering sample (and in an effort to squeesh in both samples in 24 h), the counting time for each spectrum was set to 1.5 h. Measuring also during the somewhat slow cooling on IN6, we are able to extract the mean-squared displacement (MSD) from the elastic signal and an inelastic fixed energy similar to IN16B (Fig. 2).

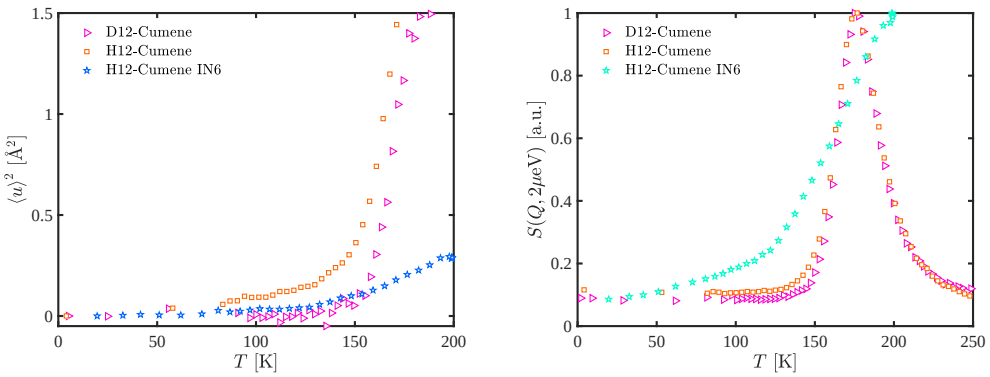

Figure 2: The MSD (right) from the fully protonated sample from the Q-dependence of the elastic signal at IN6 is here shown with the MSDs from IN16B. The inelastic fixed energy scan in temperature is shown from IN6 ($\Delta E \approx 0.2$ meV) along with the IN16B data ($\Delta E \approx 2$ µeV) for comparison scaled to the maximum within the temperature range.