Experimental report

17/03/2021

Proposal:	**TEST-3101**	**Council:**	10/2019
Title:	Structure of highly concentrated electrolytes		
Research area:			

This proposal is a new proposal

Main proposer:	**Filippa LUNDIN**
Experimental team:	Filippa LUNDIN
Local contacts:	Viviana CRISTIGLIO

Samples: (acetonitrile)n : Lithium bis (trifluoromethanesulfonyl)imide n=2,5,20 and pure

Instrument	Requested days	Allocated days	From	To
D16	2	2	24/09/2020	26/09/2020

Abstract:

Structure of Highly Concentrated Electrolytes

Experimental report

Virtual beamtime 24-26/9 - 2020

Proposal number: TEST-3101
Main proposer: Filippa Lundin
Co-proposer: Aleksandar Matic
Local contact: Viviana Cristiglio
Instrument: D16

Highly concentrated electrolytes show, unlike most liquids, order on the nanoscale, due to charge correlations[1]. Previous SAXS experiments we have performed on $(\text{acetonitrile})_n$-LiTFSI electrolytes (where n=2, 5, 20 and pure acetonitrile, denoted as AN2, AN5, AN20 and AN) have shown both the molecular peak found in all liquids (~1.5 $Å^{-1}$), as well as a pre-peak (~0.8-1 $Å^{-1}$) confirming the existence of some ordering on intermediate length scales, see figure 1 (left). Recently we performed QENS experiments at IN5 and IN16B to also investigate the dynamics on the length scales of these correlations. However, when the data from the two instruments was summed into S(Q,0) only the molecular peak was seen and not the pre-peak.

To investigate this further SANS measurement were performed in a TEST-experiment (by the local contact) on these electrolytes at D16 using deuterated acetonitrile and varying salt concentrations. The measurements were performed in the Q-range of 0.03-1.85 $Å^{-1}$. The results are shown in figure 1 (right) and confirm the lack of a pre-peak in agreement with the S(Q,0) data from the QENS experiments. Furthermore, the molecular peak shifts to lower Q with increasing salt concentration, reflecting that the average nearest neighbour distance is increased as the number of large anions is increased in the electrolyte with the addition of salt. Detailed analysis is underway, but we tentatively assign the absence of the pre-peak to cancelling of partial correlations due to the negative scattering length of the lithium ions.

This data will be part of a future publication on the structure and dynamics in highly concentrated electrolytes.

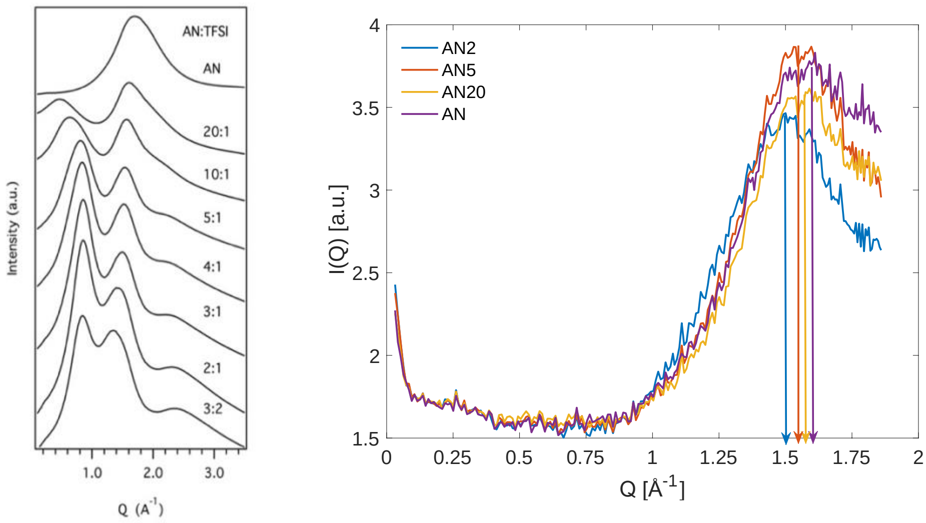

Figure 1 Left: SAXS patterns for different salt concentrations. Right: SANS patterns for different salt concentrations. The arrows indicate the peak position of each pattern. The data has been normalized to the lower Q-values and binned in Q.

References

1 L. Aguilera, S. Xiong, J. Scheers and A. Matic, *J. Mol. Liq.*, 2015, **210**, 238–242.