Experimental report

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Proposal: 6-02-627 **Council:** 4/2021

Title: Structural characterization of the molecular interactions in concentrated aqueous azole solutions

Research area: Chemistry

This proposal is a resubmission of 6-02-621

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Samples: 1,2,4-Triazole-d3

Instrument	Requested days	Allocated days	From	To
D16	3	3	04/10/2021	07/10/2021
D4	2	0		

Abstract:

Azoles constitute an interesting class of heteroaromatic molecules because of the presence of two different structural patterns, such as p electron networks and hydrogen-bond donor and acceptor groups.
Azoles are exposed to water solvent in a large number of biological systems and these hydration sites give high solubility to these compounds in water. The determination of the structure of aqueous solutions of azoles is therefore of great interest, although hardly straightforward. Hydration is in fact a complex phenomenon and the use of a combined approach, both theoretical and experimental, is required to completely describe its structural and dynamical aspects. Spectroscopic, scattering and diffraction techniques provide qualitative information on intermolecular interactions. A particular problem is the insensitivity of X-ray scattering and X-ray spectroscopy techniques to hydrogen atoms. Neutron diffraction is sensitive to scattering from all constituent atoms, C, N, O and H. We would like to use this technique to examine the solvation structure and the nature of the intermolecular interactions of 4.5M and 7M aqueous 1,2,4-triazole solutions.

Structural characterization of the molecular interactions in concentrated aqueous azole solutions.

Abstract.

The triazole nucleus is one of the most important and well known heterocycles which is a common and integral feature of a variety of natural products and medicinal agents. The basic heterocyclic rings present in the various medicinal agents is 1,2,4-triazole. A large volume of research has been carried out on triazole and their derivatives. Neutron diffraction is sensitive to scattering from all constituent atoms, C, N, O and H. Here we propose to investigate the solvation structure and the nature of the intermolecular interactions of aqueous azole solutions by means of high energy neutron diffraction and atomic pair distribution function (PDF) data analysis.

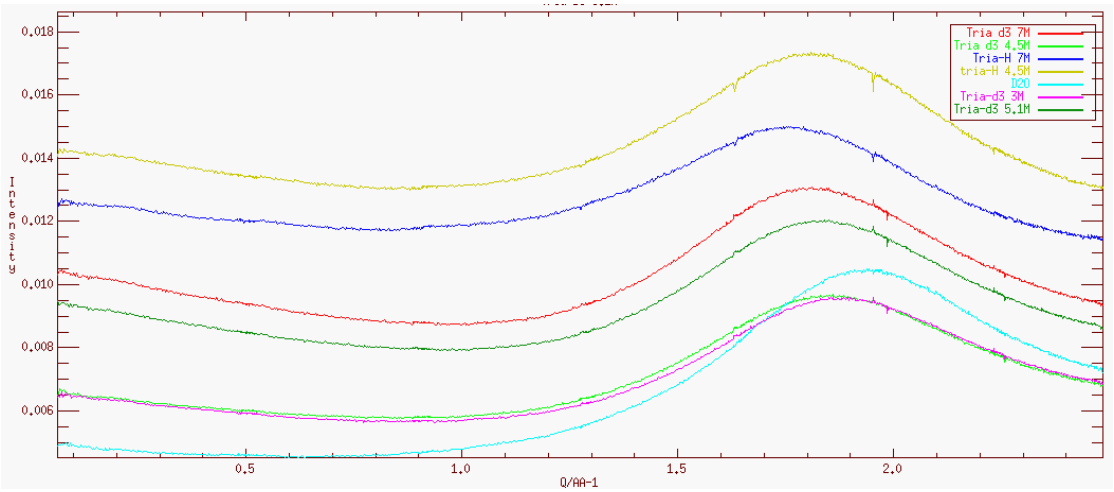
Experimental details.

About 500 mg of 1,2,4-Triazole-d3 (four sample fractions) were loaded into four 5 mm diameter vanadium cells containing D2O and we have obtained the following solutions respectively:
7M, 4.5M, 5.1M and 3M. Similar solutions in H2O were prepared for 123-Triazole-h3. All the measurements were performed at room Temperature.

Preliminary results.

In this experiment we performed a feasibility study to check the possibility to reconstruct the structure factor, S(Q), of deuterated triazoles. The characteristics of D16 made it possible to explore a Q_{max}=2.5 $Å^{-1}$

The structure factor, S(Q), of triazoles is reported, as S(Q):



This is a preliminary result: our goal is to measure, in both D16 and D4 instrument, the solvation structure and the nature of the intermolecular interactions of aqueous triazole solutions. The unique resolution and Q-range characteristics of D16 will allow us to study the intermolecular bonding, while D4 yields the necessary information of the interatomic arrangement.
In the next round we will present a continuation to perform measurements on D4, in order to obtain the entire range of Q (Qmax = 23.5 $Å^{-1}$). By Fourier transformation of S(Q) we will extract the pair distribution functions. These will be compared with the MD models.