

Experimental report

08/02/2016

Proposal: 5-42-380

Council: 10/2014

Title: Susceptibility of spin ice, Ho₂Ti₂O₇

Research area: Physics

This proposal is a new proposal

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Samples: Ho₂Ti₂O₇

Instrument	Requested days	Allocated days	From	To
IN12	4	0		
D10	3	3	03/08/2015	06/08/2015

Abstract:

An interesting paper published by Pomaranski et al. in Nature Physics 9, 353 (2013) demands further refinement of the microscopic spin hamiltonian of spin ice, Dy₂Ti₂O₇ and Ho₂Ti₂O₇ (DTO,HTO). In pursuing this project we have discovered a discrepancy between the susceptibility of HTO measured in the static approximation by neutron scattering and that measured on spherical crystals using bulk magnetisation. This experiment is designed to identify the origin of this difference by testing the validity of the static approximation, possible breakdown of the fluctuation-dissipation theorem and possible crystal shape-dependent physics in this system. The outcome will be a deeper understanding of the physics of the Coulomb phase in spin ice and an insight into Pomaranski's result.

Preliminary result on the experiment entitled “Susceptibility of spin ice, $\text{Ho}_2\text{Ti}_2\text{O}_7$ “ #5-42-380

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The experiment suffered significant technical problems, as described below. This is a preliminary report, written to support a continuation proposal.

The aim of the experiment was to directly test the fluctuation-dissipation theorem in a spherical crystal of $\text{Ho}_2\text{Ti}_2\text{O}_7$ by measuring both $\chi(0,T)$ via diffuse scattering and field-induced magnetization, $M(T)$ via a Bragg peak, and then comparing the two. Spin ice lends itself well to such studies as there are zone centres with zero nuclear contribution (e.g. 002 or 420), but minimization of background is essential. The original proposal requested 3 days on D10 in 2-axis mode with vertical field cryo-magnet ($T > 1.8$ K) to measure zero field diffuse and field-induced Bragg intensities at different zone centres, at 8 temperatures (1.8-20 K) and 4 fields (0-0.1 T), giving $\chi(0,T)$ and $M(T)$ as described.

The vertical field cryo-magnet we originally requested (field strength 0-1 T) malfunctioned and temperature could not be stabilized below 10K. We had to change to a different cryo-magnet (field up to 10 T) that did not allow the use of the analyser and had a rather large frozen field. Base temperature could then be reached, but temperature stability problems continued through out the rest of the experiments (temperature fluctuations were of the order of few tenths of a kelvin), undermining the accuracy of the measurements. We nevertheless identified 420 as the best zone centre for our purpose, and tried to run the experiment with the bigger cryo-magnet and without analyzer, performing omega scans around the 420 Bragg peak at different temperatures (1.8-10 K) and different applied field (0-2 T). However, lack of time, caused by the various problems, did not allow us to collect a full set of data, and the results reported below are only preliminary.

First, in the current configuration (analyser not mounted) the background was too large to measure the diffuse scattering - the analyser would have helped here as it would have removed the nuclear spin incoherent scattering of holmium. Also our spherical crystal may have been too small – we address this problem in the continuation proposal.

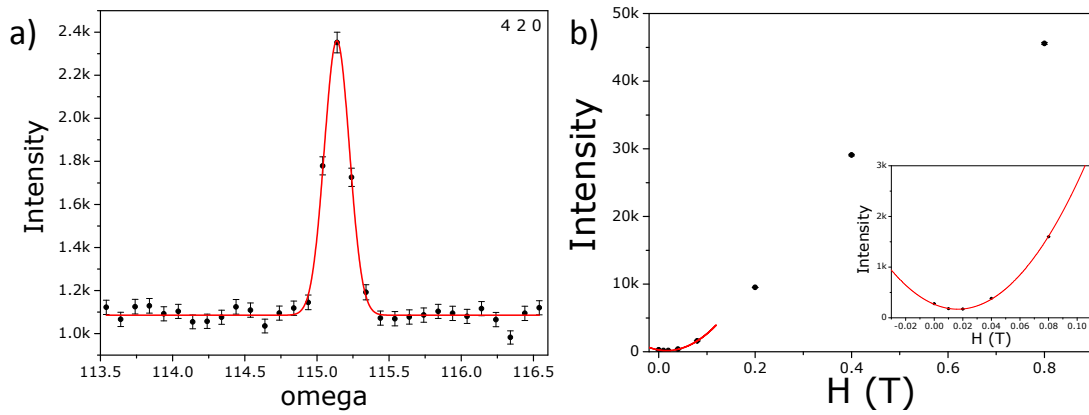


Figure 1: **a)** Omega scan around 420 Bragg peak at nominally zero applied field and 1.9K. Red line is a fit to a Gaussian function. **b)** 420 Bragg peak intensity as a function of applied field: the fit at low H provides a value of remanence field in the coils $H_0 = 0.0165$ T and the presence of a spurious peak $I_0 = 170$.

Further, the large “frozen field” in the 10 T magnet complicated the analysis of the experimental data – it meant we had to search for zero field. Figure 1a reports one of the omega scan with related fitting; figure 1b shows how the intensity of the Bragg peak changes as a function of nominal applied field. Preliminary data shown in figure 2, confirm that it is possible to follow the field induced magnetization $M(T)$ via a Bragg peak and that these data seem to be in good agreement with the bulk magnetometry data performed on the same crystal [1]; however, our limited data is hardly publishable, and the experiment needs to be repeated.

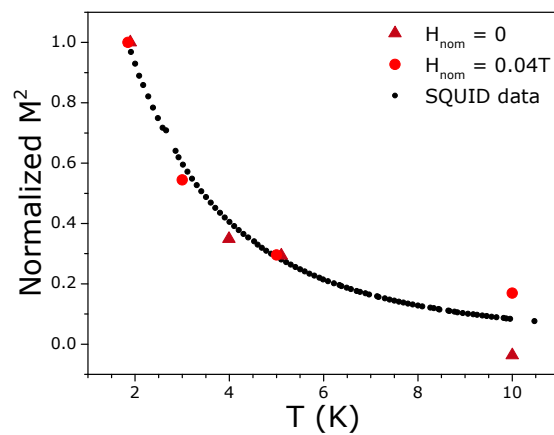


Figure 2: Normalised value of M^2 versus T obtained at two different field values as indicated in the legend. Black data are obtained from SQUID measurement on the same sample [1].

[1] Bovo et al., JPCM, 25, 386002 (2013).