ESRF	**Experiment title: Resonant x-ray magnetic scattering studies of spin ice and kagome ice**	**Experiment number**:
Beamline: BM28	**Date of experiment**: from: 30/11/2005 to: 6/12/2005	**Date of report**: 27/8/2013
Shifts: 18	**Local contact(s)**: D Mannix	*Received at ESRF:*

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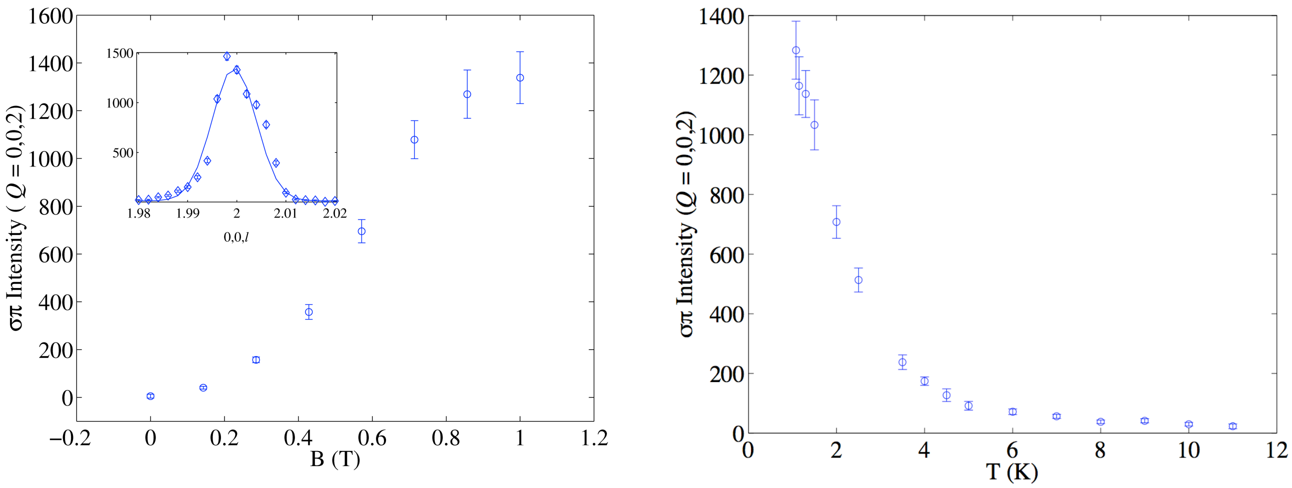
Report: The purpose of this experiment was to make the first resonant x-ray scattering investigations of the spin ice $Ho_2Ti_2O_7$. A spin ice is a material in which the spin configurations can be mapped to proton configurations in water ice, such that the two have identical degenerate manifolds governed by an ice rule. The magnetic version is found in the non-collinear Ising ferromagnet on the pyrochlore lattice. Strong local anisotropy constrains the spins to point in or out of the tetrahedra, along the <111> directions, and ferromagnetic coupling means that the local groundstate on a tetrahedron has two spins pointing in and two pointing out ($Ho_2Ti_2O_7$ is an effective ferromagnet, with the dominant interaction being the dipolar interaction), which is an ice rule constraint. In the case of spin ice, much has been learnt about the ice rule correlations by the measurement of diffuse neutron scattering, but this is thought to be immeasurable by magnetic x-ray scattering experiments.

However, the strong single-ion anisotropy means that a variety of partially ordered states can be obtained in applied field as different numbers of spins on a given tetrahedron are oriented parallel or perpendicular to the field. The diffuse scattering of zero field is modified into stronger, sharper features in these phases. We aimed to study the case of field applied along the [110] direction, in which two spins have a large component parallel to the field and form chains running parallel to the field (α-chains), while two spins per tetrahedron are perpendicular to the field, and form chains running perpendicular to the field (β-chains). Application of a field in this direction results firstly in magnetic Bragg scattering from the α-chains as they are driven into order. This scattering is at k=(0,0,0), but may be accessible in an x-ray scattering experiment since the pyrochlore has some systematically forbidden positions, such as (0,0,2). Ordering of the α-chains constrains the β-chains to a higher degree of order, and sharply peaked diffuse scattering appears at positions such as (0,0,3).

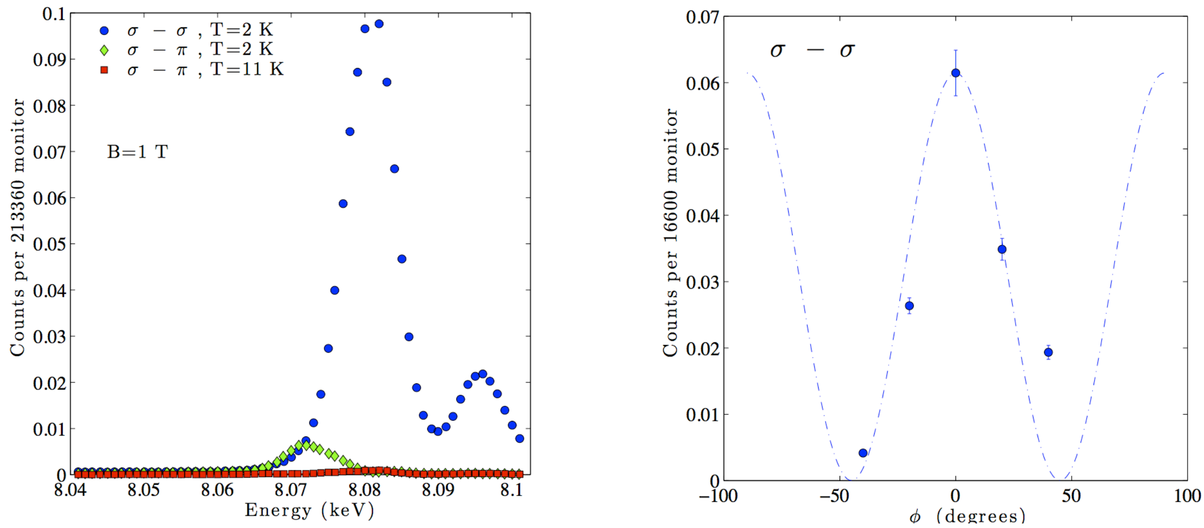
Using four-circle geometry, the ^{3}He Joule-Thompson refrigerator, and magnet at XMAS gave us access to the necessary phase space to study these processes. We used a graphite analyzer crystal. The sample was a small tile of $Ho_2Ti_2O_7$ with the [001] direction specular, and the

[110] direction parallel to the field. The sample was glued to a standard XMAS copper stub, and then sputtered with aluminium to try to enhance thermal contact across the surface.

We could not observe the diffuse signal in zero field, but found we could observe a resonant, field-induced intensity at (0,0,2) in the σ-π channel. This signal disappeared at higher temperatures (above 5 K in 1 T). We experienced some difficulties with the cryostat which required warming up and cooling back down to clear blockages several times during the experiment which hampered our progress. However, when the cryostat was working well, we found we could measure the magnetic signal without need for attenuation. We also found an enormous resonant signal in the σ-σ channel, which was independent of temperature and field. A short azimuthal scan suggests it has four-fold symmetry. Perhaps, given the combination of symmetry-forbidden Bragg position and highly anisotropic crystal field at the rare earth site, the appearance of such a signal is not a complete surprise. However, it is not otherwise known, and may provide a means to study subtle magnetostructural effects in other pyrochlores.



Left: Field dependence of σ-π intensity at (0,0,2), inset shows an example of a magnetic peak. Right: Temperature dpendence of the σ-π intensity at (0,0,2) in 1 T.



Left: Energy scans at (0,0,2) show that the magnetic signal in the σ- σ channel is dwarfed by a resonant signal in the σ-π channel. Right: Azimuthal scan of the σ- σ signal.