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TRAVAUX PRATIQUES IV
LOW TEMPERATURE AC
SUSCEPTIBILITY OF LiErF_4

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1 INTRODUCTION

Quantum magnetism is a field which is concerned with spin configurations and dynamics on a variety of systems. Research is carried out on materials which are believed to be model systems which can confirm or challenge current theories. Although there are many different possible systems which can be chosen, one of the large groups of materials is rare earth compounds. These compounds are of interest due to the large total angular momentum of many rare earth metals (e.g. Holmium has total angular momentum $J = 8$).

One particularly special rare earth compound is Lithium Holmium Fluoride (LiHoF_4), which has been demonstrated to be a model ferromagnet and when LiYF_4 is doped with holmium the compound can become a spin glass. Due to the success of LiHoF_4 , another compound which has potential to give useful insights into quantum magnetic theory is Lithium Erbium Fluoride (LiErF_4), as Erbium is Holmium's heavier neighbour in the periodic table. LiErF_4 has been shown to have a phase change from a paramagnetic state to an anti-ferromagnet using AC susceptibility [1] and more recently using neutron diffraction techniques [2]. In this report, LiErF_4 is studied once again using AC susceptibility in the presence of magnetic fields to define a phase diagram separating the antiferromagnetic and paramagnetic states.

2 QUANTUM SUSCEPTIBILITY MEASUREMENTS

To measure the susceptibility of a material at low enough energies that the quantum effects can be seen is not a trivial task. Apart from the measurement itself, we require extremely low temperatures (several hundred milliKelvin) and a way to amplify the extremely weak signal while minimising noise.

2.1 AC SUSCEPTOMETRY

The interest in measuring the susceptibility is that it allows the determination of the magnetisation M via the application of an AC magnetic field H_{ac} . The AC susceptibility is given by:

$$\chi = \frac{dM}{dH_{ac}}$$

In an AC susceptibility measurement, a current is passed through a set of coils, inducing an ac magnetic field. The moment of the sample is changed due to the magnetic field, allowing for the dynamics of the system to be studied. Measurements of complex susceptibility gives an insight into the phase transition in LiErF_4 , as there is a peak in the real susceptibility signal (χ') at the transition.

AC susceptometers consist of 3 sets of coils, the Primary coil and two secondary coils. The primary coil carries the current used to create the AC magnetic field which the sample reacts to. One of the secondary coils is designed to detect the voltage induced by the response of the sample and the other secondary coil is used to cancel out the voltage induced by the original AC field. This is done by connecting the two secondary coils in opposite directions (i.e. so that one has right hand turns and the other has left hand turns) so that the induced voltages in both coils from the original AC field cancel out [4].

2.2 DILUTION REFRIGERATION

As has previously been mentioned, quantum effects are generally seen at extremely low temperatures. Achieving these temperatures is not a trivial task, and requires very complex apparatus. Pumping on liquid helium 4 allows us to get down to temperatures around 1.3 K and pumping on helium 3 allows us to get down to a few hundred mK. Getting to lower temperatures however requires a dilution refrigerator. The basic principle of a dilution refrigerator is to pump a mixture of liquid helium 3 and helium 4, allowing for temperatures to be lowered to a few mK for the most powerful refrigerators. Figure 1 shows the schematic of a dilution refrigerator.

A dilution fridge is operated inside a cryostat filled with liquid helium, as this cools it down to 4.2 K and also reduces the amount of heating from the surroundings. The “cold part” of the dilution fridge is inside the IVC (**I**nn**V**acuum **C**hamber), which is at very high vacuum, so there are no particles which can transfer heat from the outer surroundings. A very brief description of a dilution fridge while running is as follows:

- Practically all the ^4He is condensed inside the condenser line, mixing chamber and still.
- The gas leaves the IGH (**I**ntelligent **G**as **H**andler) and passes through liquid nitrogen then liquid helium cold traps ensuring no contaminants, which could block many of the thin capillaries, enter into the fridge
- The ^3He Circulates through the dilution refrigerator as follows
 - The gas enters into the condensing line where it passes through the 1K pot (which is cooled by pumping on liquid ^4He), pre-cooling (~ 1.5 K) and condensing it
 - The liquid passes through the heat exchanger, where it is further cooled, and arrives at the mixing chamber
 - In the mixing chamber there is a phase separation between the ^4He and ^3He , the phase dilute in ^3He sits below the concentrated phase (rich in ^3He).

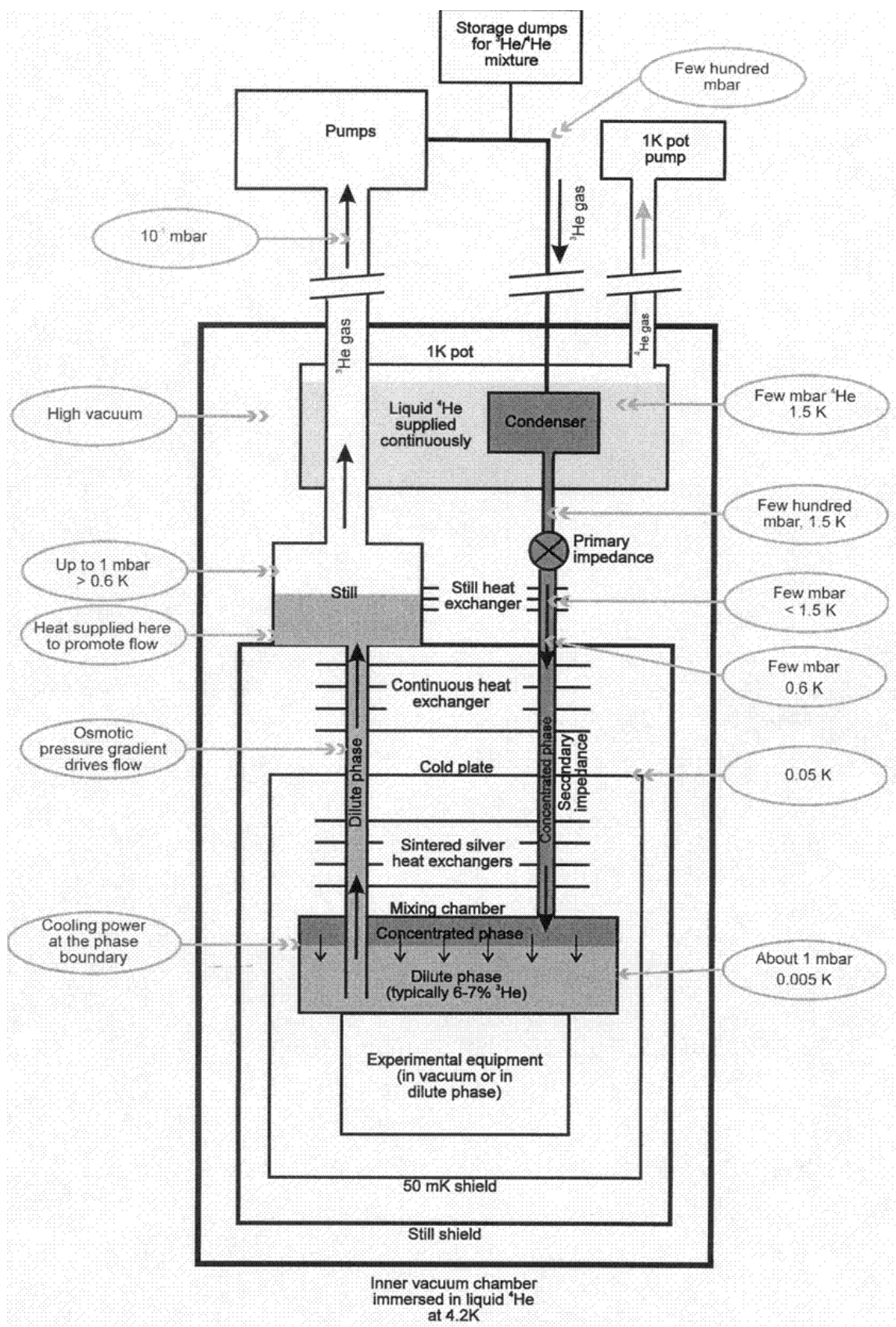


Figure 1: Schematic representation of a dilution fridge [3]

- Osmotic gradient pressure drives the flow of the dilute phase up through the heat exchanger and into the still where it is pumped back to the start of the circuit
- The phase separation inside the mixing chamber, coupled with the pumping of the dilute phase is what cools the mixing chamber down to ~ 70 mK (base temperature typically achieved).
- In general the rate of flow of the ^3He through the system is what determines the cooling power and the base temperature.

It is worthwhile noting that although the running procedure of a dilution fridge seems fairly straightforward, it is difficult to have the dilution fridge consistently achieve specified base temperatures and cooling powers. Some of the problems which were frequently encountered are:

- Unstable 1K pot temperature, causing the mixing chamber temperature to fluctuate
- Blockages in the cold traps or the capillaries leading to higher base temperatures and lower cooling power
- Base temperature rises significantly over extended periods of time (cause unknown)

In addition to the problems with keeping the fridge running smoothly at low base temperatures, care must also be taken with the measurements and diagnostics of the sample. As the temperatures are extremely low, it only takes a few μW of heat to change the temperature of the sample significantly. The first problem this causes is in thermometry, as resistance is used to determine the temperature. This problem is fairly well solved using devices known as resistance bridges, which allow accurate measurements of resistance using extremely small current. The second problem is that sending a large signal into the susceptometer (and thus sample) will cause heating. Therefore very small signals are used and we need a way to measure signals which are extremely small (even compared to the background).

2.3 LOCK-IN AMPLIFIER TECHNIQUE

In quantum AC susceptibility measurements, an extremely small AC voltage must be measured and be extracted from a possible noisy background. This is done by using a lock-in amplifier. A lock-in amplifier does this with the aid of a reference channel which oscillates at the same frequency as the signal channel. The lock-in generates the oscillating signal which is used to create the AC field to excite the sample and then measures the

resulting induced voltage. As the induced voltage is at the same frequency, the lock-in can filter out all other frequencies (by using a Fourier Transform like procedure). The lock-in also changes the phase at which it is measuring to increase the signal to a maximum. The fact that both the signal amplitude and phase are measured allows for the full complex susceptibility to be measured.

One must take care in the measurement of the real and imaginary channels, as the experimental set-up introduces a frequency dependent dephasing term. This means that before meaningful measurements can be taken, the internal phase must be set to a value which gives the correct real and imaginary channels and not a combination of both in each channel. One way to do this is to connect a 1Ω resistance in parallel to the measured signal, allowing us to measure the current directly. In this configuration, the lock-in can be told to “auto-phase”, meaning that it adjusts its internal phase so that all the channel is imaginary. Adding 90° to this phase gives the real and imaginary parts correctly.

2.4 LOW TEMPERATURE THERMOMETRY

Measuring the temperature at extremely low temperatures is a difficult task. Even once the technical problem not generating heat during the measurement has been worked out, the choice of the sensor becomes a problem. This is because normally the temperature scale which is of interest is ~ 50 mK - 1K and that there are not a large number of materials which show large changes in their electrical properties during this range. It is possible to buy chips rated down to these temperatures from cryogenics companies, however the high cost makes them generally only accessible for mounting on permanent set-ups. The problem that exists is that in general the sample will not be at the same temperature as the mixing chamber, so it would be beneficial to have thermometers on the sample.

The method chosen to try to measure the temperature of the sample was to use RuO_2 resistors (which are used in most electronic devices as they have very stable resistances at room temperature). These resistors show an exponential increase in resistance below $\sim 1\text{K}$, allowing for sensitive measurements to be made. It should be noted that when these resistors are wired up, 4 wires are used to make the connection instead of the normal 2 as would be used for room temperature measurements. This effectively puts the resistance of the wires in parallel to the resistor, meaning that the effect of the resistance of the wires becomes negligible and doesn't affect the total resistance. The RuO_2 resistors can be bought very cheaply in bulk and give a means of having a relative temperature scale on the sample (or absolute temperature scale if they have been calibrated). For the experiments presented in this paper, the resistors were not calibrated and thus the resistors allowed us to see if the temperature of the sample was stabilised, but not know the exact temperature.

3 EXPERIMENTAL SETUP

One of the most important pieces of apparatus is the susceptometer, which is made by CMR and has all three coils in a concentric configuration. All the coils were made with $25\mu\text{m}$ copper wire which comes to a total thickness of $36\mu\text{m}$ when the insulation is taken into account. The specifications for the coils are given below

Coil winding	Inner diameter [mm]	Outer Diameter [mm]	Length [mm]	Number of turns	300K Resistance $\text{k}\Omega$
Primary	4.5	5.6	10	3867	2.45
1 st secondary	2.6	3.8	10	5273	2.104
2 nd secondary	3.8	4.5	10	2974	1.615

Table 1: Specifications of the Susceptometer

3.1 SAMPLE PREPARATION

The preparation of the sample is extremely important for the measurements of susceptibility. There are two factors which must be taken into account when the sample is being prepared and mounted: there needs to be a good thermal contact and a structural strength is required. A good thermal contact is needed so that the temperature measured on the mixing chamber can be considered the same as the sample temperature. The strength of the sample holder is needed as quite often large magnetic fields are used which can exert a torque on the sample, causing it to move.

The thermal contact is ensured in a number of ways. The original idea was to simply attach the sample to a sapphire wafer (sapphire is used as it has a relatively good thermal conductivity but is an insulator) between 0.33 mm and 1 mm thick and 1 mm wide. The sample was glued on with GE varnish and the sapphire wafer was glued onto a copper strip which was in turn screwed onto the copper block shown in Figure 2. It was found that for the LiErF_4 crystal that this configuration did not give a good enough thermal contact, with low temperature thermalisation taking around three hours (i.e. when cooling down, the sample reaches base temperature three hours after the mixing chamber).

For the results taken, the sample was prepared as follows. A crystal of LiErF_4 was cut to dimensions of 1.3 x 1.3 x 10 mm and then a RuO_2 thermometer was glued onto one of the ends. The thermometer was soldered up with four $50\mu\text{m}$ copper wires which were carefully run along the length of the crystal. The crystal and these four wires were then wrapped with a $50\mu\text{m}$ copper wire which was designed to give a good thermal contact between the copper cold-finger (attached to the mixing chamber). A 1.9 mm diameter hole was drilled into a Teflon block 2 cm thick and was filled with liquid Stycast (an epoxy which is commonly used at low temperatures) into which the sample was placed (with the

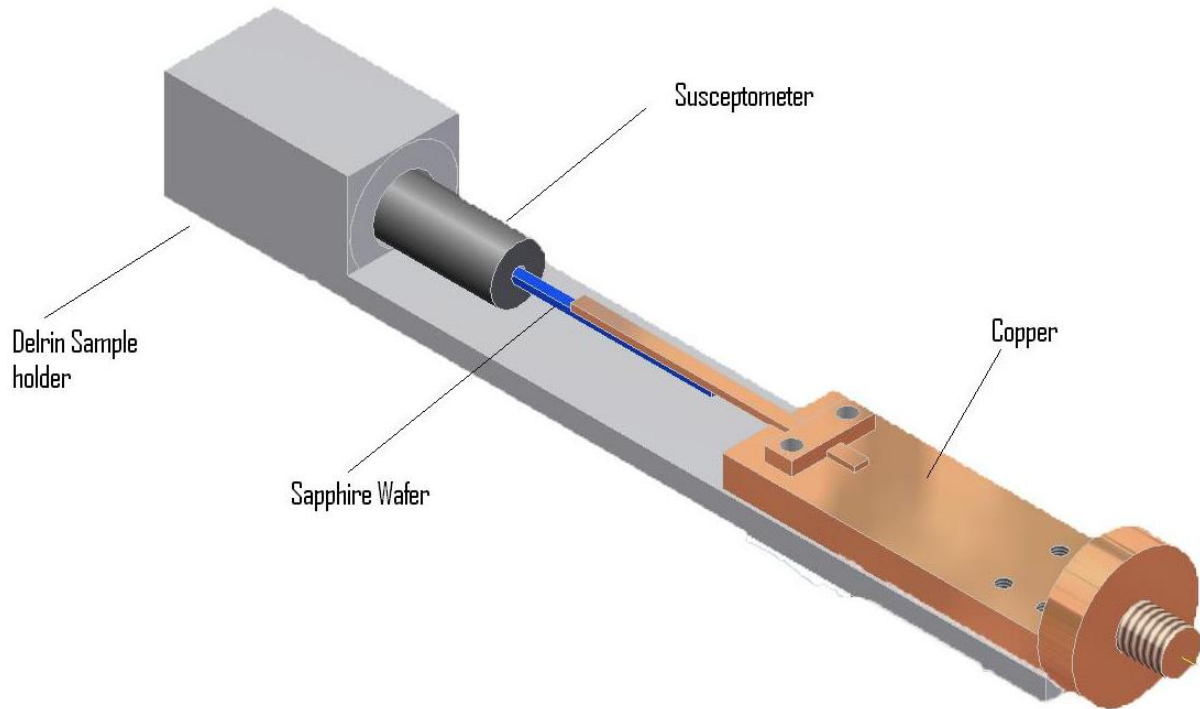


Figure 2: Sample holder

thermometer going in first). A sapphire wafer was placed behind the sample, giving the rigidity required. Once the Stycast had dried, the sample was pushed out of the Teflon block and a thin strip of copper was glued to the sapphire and was then mounted into the sample holder shown.

3.2 MOUNTING OF THE SAMPLE HOLDER

The mounting of the susceptometer and the connection of the sample to the mixing chamber is not a trivial task. The sample sits inside a sample holder as is shown in Figure 2. The basic idea is that the susceptometer is held in a piece of plastic and the prepared sample is inserted into the susceptometer. A sapphire connection to the cold temperature copper is used as this should reduce the background susceptibility due to the fact that sapphire is an insulator. The copper is then connected to the mixing chamber via the cold finger (a long copper rod). Additionally, the entire sample holder as seen in Figure 2 is sitting inside a stainless steel can. This can is filled with helium gas, which at such low temperatures becomes a super-fluid, and thus should give a very significant thermalisation power. In its present state, the effectiveness of this can is questionable as it is uncertain if the super-fluid helium is contained in the can, or if it leaks out.

Another important part in the mounting of the sample in the sample holder is making all the electrical connections. All the connections made used 4-pin Lemo connectors to

facilitate the mounting procedure. The thermometer wires were soldered to a connector and an extension cable was made for the susceptometer (as it finishes in a female Lemo connector). These two connectors were plugged into feed-throughs on the top of the helium can. From there, more extensions were made up to the mixing chamber where there was a 24-pin connector onto which the thermometer wires were soldered. The susceptometer connector was plugged into a connector attached to the inner cores of 4 stainless steel coaxial cables which were already installed on the dilution fridge.

3.3 PREPARATION OF THE DILUTION FRIDGE

Once the sample has been mounted and all connections are checked, the task of preparing the dilution fridge and cooling down to base temperature can begin. The first thing which is done is that the IVC is sealed up using an Indium wire seal. The idea behind this is that a piece of indium wire is clamped down between two pieces of metal, giving an extremely good vacuum seal. With the IVC sealed, it needs to be pumped out so that there will be no gas to exchange heat between the mixing chamber and the liquid helium in the cryostat. The IVC is normally vacuum pumped for an hour or so and then a small volume of helium (exchange) gas is introduced (a few cubic centimeters is normally sufficient).

Before being lowered into the cryostat, a sliding seal is attached to the dilution fridge. The sliding seal is simply a hollow cylinder which forms a seal at the top of the cryostat and allows for the dilution fridge to be lowered into the cryostat. The sliding seal performs two tasks: the first is that it stops the top of the cryostat from freezing and secondly it stops the loss of helium gas to the atmosphere and instead diverts it to the recovery system. The lowering of the dilution fridge is done slowly, so that not much liquid helium in the cryostat is boiled off. If lowered slowly, the temperature inside the IVC is normally at 4.2 K by the time the fridge is completely lowered. Once at this temperature all the exchange gas in the IVC is absorbed by the sorb (a piece of carbon material with a very large surface area).

With the mixing chamber at 4.2 K the condensation of the mixture can begin. First, all of the tubes (1K pot line, still line and condenser line) are connected to the IGH and the still and condenser lines are checked for leaks (as the mixture is very expensive). At the same time, if it has not already been done, the helium 3 and helium 4 pumps are turned on. The helium 3 pump is the pump which circulates the mixture and pumps on the still line. The helium 4 pump pumps on the 1K pot. Before condensing can begin, the 1K pot is cooled down by opening a needle valve. Once the 1K pot is cold, the mixture is released slowly into the system and begins to condense. Once the pressure in the mixture dump has decreased sufficiently, the valve on the still line is slowly opened and the mixture begins to circulate. The temperature then begins to decrease towards

base temperature (we normally need to heat the still with 5mW of power to achieve a good base temperature).

3.4 LOCK-IN CONFIGURATION

The lock-in amplifier was first connected with a 1Ω resistor in parallel to the signal channel so that the phase could be set correctly. This was done at a frequency of 990 Hz and therefore all scans would be done at the same frequency. As the resistor in parallel is 1Ω the measurement gives directly the current. The specifications of the susceptometer give the magnetic field generated per mA of current in the wires, thus by measuring the current, it is possible to know and adjust the excitation field. The oscillator voltage was adjusted so that the field was 1 Oe.

4 RESULTS & DISCUSSION

The experiments carried out consist of ramping a DC field at constant temperature and ramping the temperature at constant DC field.

4.1 DC FIELD SCANS

The phase diagram of LiErF_4 is given for the temperature and the DC field. Inside the cryostat there is a 9T superconducting magnet which is used to create a DC magnetic field. In a DC field scan the sample is taken to the desired temperature and we wait for it to stabilise at the temperature. The field is then ramped in both directions across the phase transition while the susceptibility is measured. At the phase transition, we see a peak in the χ' signal; the position of the peak is used to create a point on the phase diagram. A field scan taken at 100 mK is shown in Figure 3, the peak at the transition field can be easily seen at around 0.34 T.

While doing the field scans, an interesting but unwanted effect was observed. There exists an effect known as the magnetocaloric effect. This result of this effect is that ramping the magnetic field can cause materials to heat up or cool down. In the case of LiErF_4 , we found that if the field was ramped too quickly the temperature of the sample would change. There are in fact two effects to take into account, the effect on the copper and the effect on the LiErF_4 . In copper, increasing the magnetic field causes the copper to heat up. What was observed on the sample thermometer was that initially the sample warmed up slightly as the field began to ramp up. As the transition approached, the sample would start to cool down, passing the stable temperature before the ramp started. For this reason the rate of scan was reduced from an initial rate of 0.01 T / min to 0.0005 T /min. The difference in the field and which the transition takes place is non-negligible

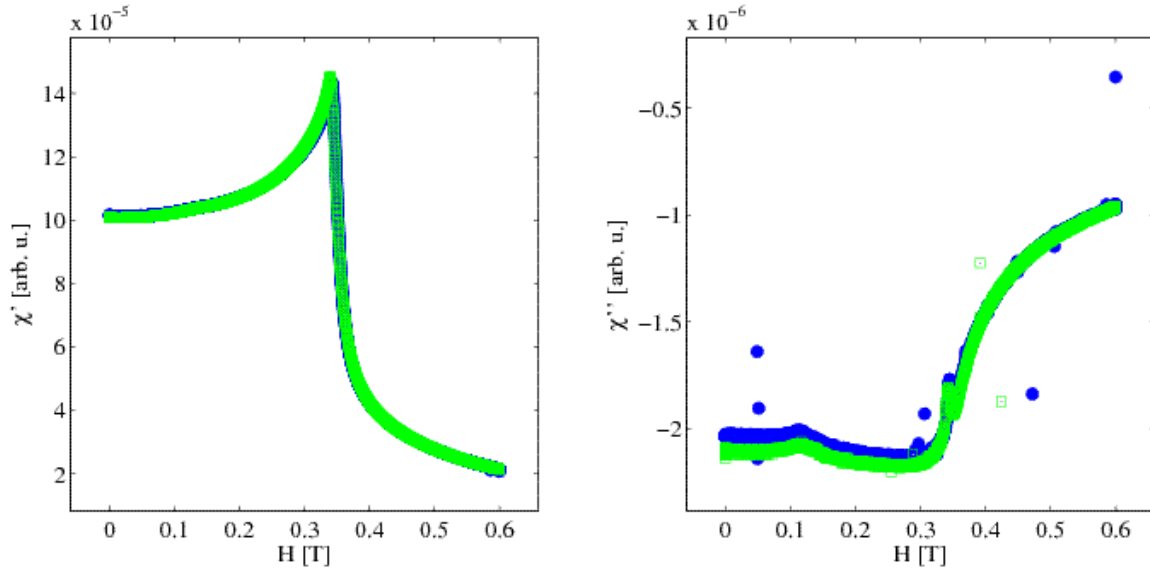


Figure 3: Typical χ curves for a DC field scan. Blue - Ramp up. Green - Ramp down

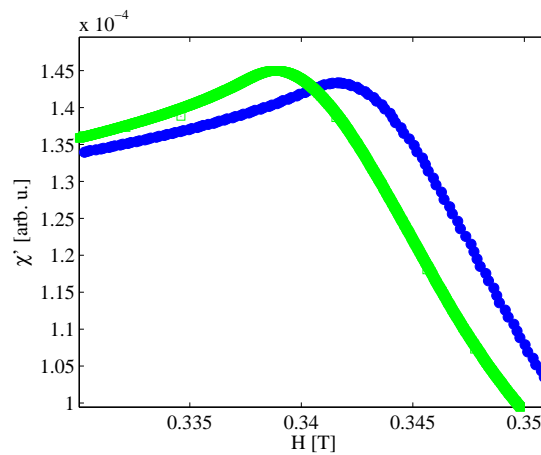


Figure 4: Comparison of 0.01T/min (blue) and 0.0005T/min (green) ramp up at 100 mK

as can be seen in Figure 4, which compares a fast and slow ramp up at 100mK. By comparing peaks of fast and slow ramps taken at different temperatures, the magnitude of the magnetocaloric effect was estimated to correspond to a temperature change of ~ 20 -30mK.

In addition to creating the phase diagram a full range field scan was taken at base temperature (70mK) to see if there are other phase changes or any interesting physics which presents itself at higher fields. As time is always to some extent an issue, the scan was made with a rate of 0.02 T/min. The resulting curves did not show any interesting features apart from the phase transition at ~ 0.35 T.

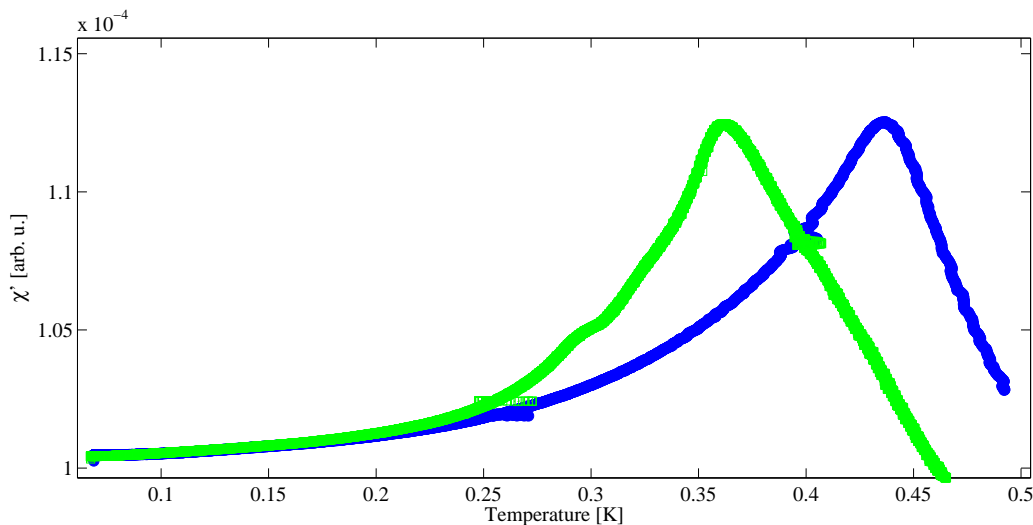


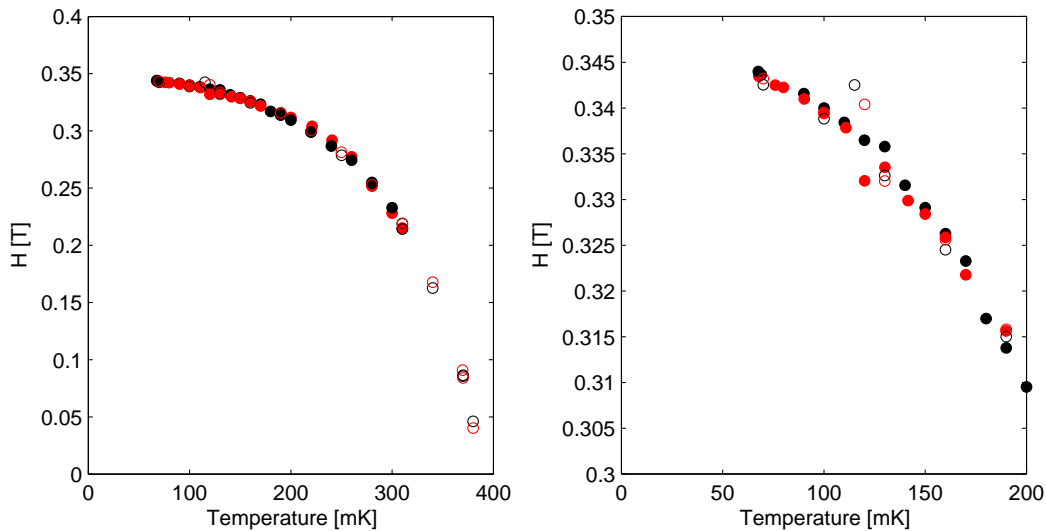
Figure 5: Comparison of 0.1 mK/s (blue) and 0.02 mK/s (green) temperature scans at zero field

4.2 TEMPERATURE SCANS

Although in theory doing a temperature scan at different fields can give the phase diagram, in practice, this doesn't give very good results. The reason why this is the case is that thermalisation of the sample and the mixing chamber takes a long time. In general, it helps by scanning up in temperature, as when the temperature increases, so does the thermal conductivity. Even doing ramp up temperature scans, the scans took a considerable amount of time and the data was not as accurate as for the field scans.

The reason for the loss of accuracy is that the temperature of the sample wasn't known. Although a thermometer was attached to the sample, it wasn't calibrated, thus we could only take values of its resistance and not convert them into temperature. This was useful in the sense that it was possible to see when the sample had thermalised or if there was a sudden temperature change in the sample. Unfortunately, there was always a difference between the sample and the mixing chamber temperature which was hard to quantify. As an example of the difference in the heating rates, Figure 5 shows a comparison between two scans taken at zero field, ramping from 70 mK to 500mK with two different heating rates. The blue curve corresponds to a heating rate of 0.1mK/s and the green curve to 0.02mK/s.

The fast scan took nearly 1.5 hours, which is an acceptable amount of time if many scans need to be taken, whereas the slow scan took almost 7 hours. To make the phase diagram, at least 10 points are needed, and it is desirable to have as many as possible. In this case it is clear that doing slow scans would take far too long and doing fast scans would give incredibly bad data. For this reason the phase diagram used only field scans taken at different temperatures.

Figure 6: Phase diagram of LiErF_4

4.3 LiErF_4 PHASE DIAGRAM

The main objective of this experiment was to get an accurate phase diagram of LiErF_4 , as previous phase diagrams had rather large errors, and didn't use the same experimental procedure to obtain all of the diagram. The phase diagram obtained can be seen in Figure 6.

The phase diagram was made using 4 different data sets. First of all both ramp up scans in H and ramp down scans in H at the specified temperatures were used. The ramp up scans are given by the red points and the down scans by the black points. In general both the up and down scans gave very similar values of $H_c(T)$, the critical field for a given temperature. It was however noted that the down scans may give slightly better results, as the sample appeared to have a larger magnetocaloric effect on the ramp up. In addition to the up and down scans, two different scanning rates were used. The solid points correspond to the slow scan at a rate of 0.0005 T /min and the hollow points correspond to the fast scan at 0.01 T/min. As has previously been mentioned, the slow scans are more accurate, as the temperature variation due to the magnetocaloric effect is greatly reduced.

The phase diagram obtained is very similar to the previous phase diagram as given in [2]. The values previously obtained from neutron diffraction data indication a zero temperature critical field $H_c(0)$ of 0.4 ± 0.02 T and the zero field critical temperature (known as the Néel temperature) $T_N(0)$ of 375 ± 5 mK. In comparison, the results obtained by extension of the phase diagram give $H_c(0) = 0.355 \pm 0.005$ T and $T_N(0) = 385 \pm 5$ mK. The critical temperature of both methods give almost the same result, however there is a reasonably large difference in the critical fields. This is most likely due to the fact that

the determination of the critical field at a given temperature using neutron scattering has a very large error compared to using the AC susceptibility. Another factor to take into account is that the phase diagram presented here has a very large number of points, reducing the error in estimating the critical field at zero temperature, whereas the previous work used less than 10 points for the entire phase diagram.

4.4 POSSIBLE NUCLEAR MOMENT INTERACTION

The most interesting result was, as is often the case, discovered by accident. While creating the phase diagrams using the field scans, two different graphics were plotted at the same time. One graphic was a superposition of all the different scans taken, and the other one was the phase diagram. The reason both graphs were plotted was to see if the routine used to extract the maxima to be used in the phase diagrams was working correctly. It was discovered that at $H_c(T) \sim 0.33$ T (which corresponds to a temperature of roughly 120mK) there was a peak in the signal strength at H_c as can be seen in Figure 7.

It was decided that this was an interesting effect to study as it could be due to the nuclear moment interacting with the electronic moments (although calculations have shown that this should happen at much lower temperatures). For this reason, many more field scans were taken around this region to verify that the apparent dip seen was reproducible. The dip was indeed reproducible as can be seen in Figure 8 and Figure 9. These two figures show χ' at the critical field (i.e. the maximum susceptibility for each scan) as a function of temperature and critical field ($H_c(T)$) respectively.

In these figures, a peak is clearly seen in the region expected. The difference between the red circles and black squares is that the squares came from fast scans and the circles came from slow scans. As can be seen, the variation isn't that large, although a smoother curve is seen when using only the slow scans (as is expected due to a smaller temperature variation during the scan).

Both figures in essence show the same thing, although the curves obtained as a function of $H_c(T)$ are likely to be more accurate. The reason for the improved accuracy is that even though the temperature of the sample isn't exactly known and is varying slightly (due to the magnetocaloric effect), the critical field is found by finding the peak position as a function of field. This implies that even if the temperature is unknown, the field at which the transition takes place is always well defined.

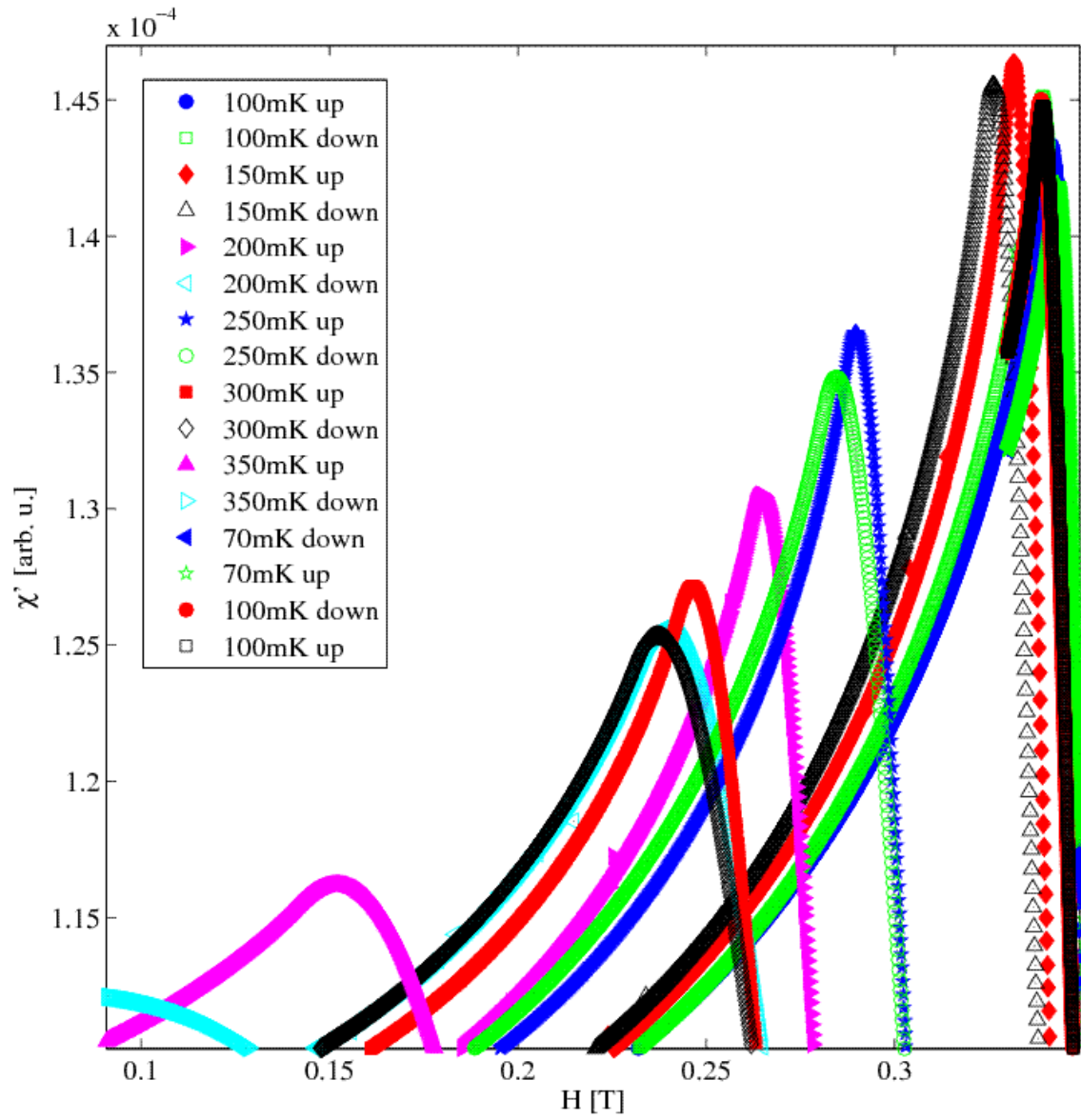
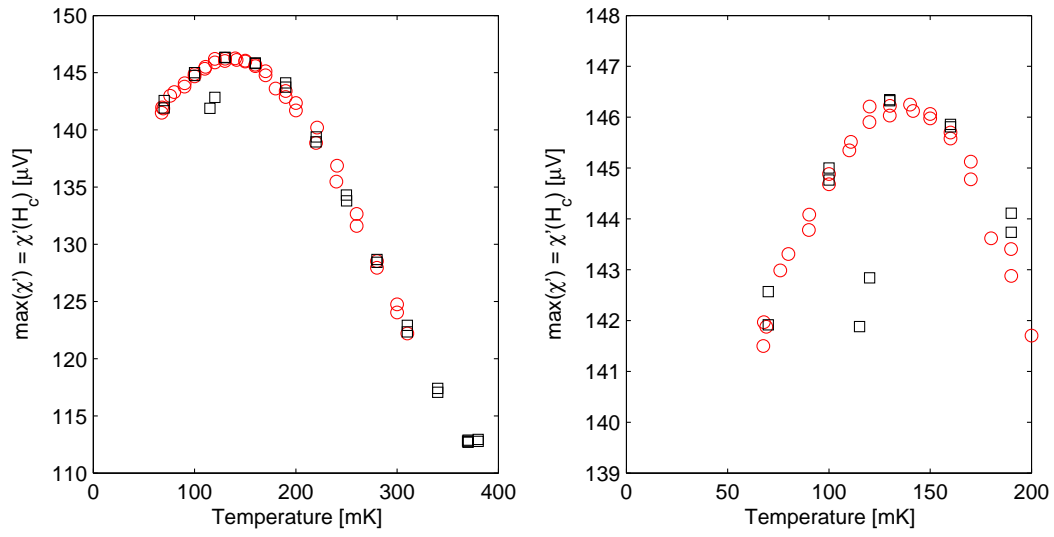
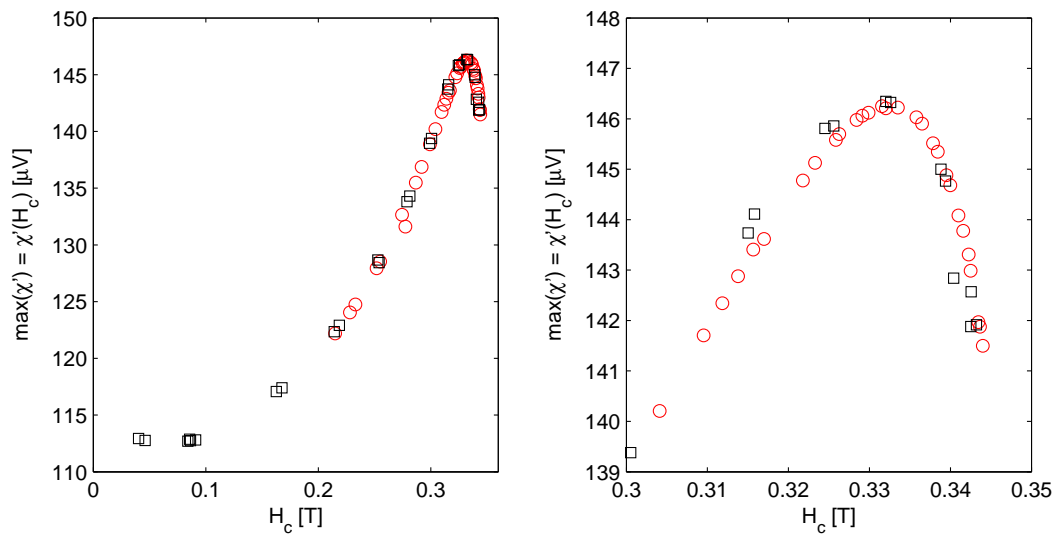


Figure 7: Field scans at many different temperatures showing a peak in the signal strength at H_c

Figure 8: Scans of Maximum χ' measured for each field scan as a function of TemperatureFigure 9: Scans of Maximum χ' measured for each field scan as a function of H_c (T)

5 CONCLUSIONS & FUTURE WORK

Probably the most important result from the experiments discussed in this report is the production of the phase diagram, which corresponds well to previous studies. The importance for this is that, although not directly mentioned in the context of the report, it is the first verifiable realisation of a completely working quantum susceptibility undertaken at LQM using this apparatus. There have been many different problems, ranging from badly mounted samples to bad thermalisation to problems with the dilution fridge to problems with the acquisition of the signal of the lock-in amplifier. This work demonstrates that the lab is now in a position where interesting physics of quantum magnetic systems can be observed at temperatures lower than 1K.

The conclusions from the data itself does however show that more work is needed in certain areas. First and foremost, it would be extremely beneficial to have a system of calibrating thermometers on the samples. If this could be done with acceptable accuracy, virtually all the problems due to thermalisation would be eliminated. The reason behind this is that a rapid thermalisation is only necessary for two things. The first is to get to the base temperature quickly, which as measurements don't normally require going down to the lowest possible temperature, isn't always necessary. The second reason why good thermalisation is currently needed is because we assume the mixing chamber temperature to be the sample temperature in our measurements. If we had an accurate reading of the temperature then this last condition isn't required. More work on thermalisation would also help, as it decreases the dependence on sample thermometry and increases the speed at which measurements can be taken.

The phase diagram was found to be in general agreement with previous experiments, and thus further investigation probably wouldn't give many insights into the magnetic system. One point which could be looked at is the critical fields at even lower temperatures, as there is evidence to suggest that there is a bump in the phase diagram as the temperature approaches absolute zero. This of course requires either a more powerful dilution fridge, or optimisations of the current fridge.

The final and most interesting result is of the dip in maximum susceptibility at low temperatures. This is a very interesting point, as it suggests that the actual physics of the system is not in accord with the current theory. The effects observed as seen in Figure 8 and Figure 9 should definitely be investigated in further detail. An example of further work which could be carried out on this effect is to see if the dip is only at $H_c(T)$ or if the entire field-curve has a lower susceptibility. One way to probe this effect would be to do a zero-field temperature scan and see if there is also a dip at lowest temperatures. The main problem with doing these kinds of experiments is that currently we are unable to achieve a stable base temperature below 70mK, and thus we don't have a large range of

temperatures available where the effect can be observed.

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